

Report 144

# Rubber Curing Systems

R.N. Datta

Volume 12, Number 12, 2002

# **RAPRA REVIEW REPORTS**

A Rapra Review Report comprises three sections, as follows:

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| Source of original article | Item 1<br>Macromolecules<br>33, No.6, 21st March 2000, p.2171-83<br>EFFECT OF THERMAL HISTORY ON THE RHEOLOGICAL<br>BEHAVIOR OF THERMOPLASTIC POLYURETHANES<br>Pil Joong Yoon; Chang Dae Han<br>Akron,University   | Authors and affiliation                    |
|----------------------------|--|--|
|                            | The effect of thermal history on the rheological behaviour of ester- and ether-based commercial thermoplastic PUs (Estane 5701, 5707 and 5714 from B.F.Goodrich) was investigated. It was found that the injection moulding temp. used for specimen preparation had a marked effect on the variations of dynamic storage and loss moduli of specimens with time observed during isothermal annealing. Analysis of FTIR spectra indicated that variations in hydrogen bonding with time during isothermal annealing very much resembled variations of dynamic storage modulus with time during isothermal annealing. Isochronal dynamic temp. sweep experiments indicated that the thermoplastic PUs exhibited a hysteresis effect in the heating and cooling processes. It was concluded that the microphase separation transition or order-disorder transition in thermoplastic PUs could not be determined from the isochronal dynamic temp. sweep experiment. The plots of log dynamic storage modulus versus log loss modulus varied | Abstract                                   |
| Location —                 | with temp. over the entire range of temps. (110-190C) investigated. 57 refs.<br>GOODRICH B.F.<br>USA<br>Accession no.771897  | Companies or<br>organisations<br>mentioned |
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# Rubber Curing Systems

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(Flexsys BV)

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# **1** Introduction

Crosslinking or curing, i.e., forming covalent, hydrogen or other bonds between polymer molecules, is a technique used very widely to alter polymer properties. The first commercial method of crosslinking has been attributed to Charles Goodyear (a.1) in 1839. His process, heating rubber with sulfur, was first successfully used in Springfield, Massachusetts, in 1841. Thomas Hancock used essentially the same process about a year later in England. Heating natural rubber with sulfur resulted in improved physical properties. However, the vulcanisation time was still too long (>5 h) and the vulcanisates suffered from disadvantages, e.g., ageing properties.

Since these early days, the process and the resulting vulcanised articles have been greatly improved. In addition to natural rubber, many synthetic rubbers have been introduced over the years. Furthermore, many substances other than sulfur have been introduced as components of curing (vulcanisation) systems.

The accelerated sulfur vulcanisation of general purpose diene rubbers (e.g., natural rubber (NR), Styrene butadiene rubber (SBR), and butadiene rubber (BR)) in the presence of organic accelerators and other rubbers, which are vulcanised by closely related technology (e.g., ethylene-propylene-diene terpolymer (EPDM) rubber, butyl rubber (IIR), halobutyl rubber (XIIR), nitrile rubber (NBR)) comprises more than 90% of all vulcanisations.

#### 1.1 Conventional Vulcanisation, Semi-Efficient Vulcanisation and Efficient Vulcanisation

Over the years three special types of cure systems have been developed. They are:

- efficient vulcanisation (EV) systems,
- semi-efficient vulcanisation (SEV) systems and
- conventional vulcanisation (CV) systems.

EV systems are those where a low level of sulfur and a correspondingly high level of accelerator or sulfurless curing are employed in vulcanisates for which an extremely high heat and reversion resistance is required. In the conventional curing systems, the sulfur dosage is high and correspondingly the accelerator level is low. The CV systems provide better flex and dynamic properties but worse thermal and reversion resistance. For optimum levels of mechanical and dynamic properties of vulcanisates with intermediate heat, reversion, flex and dynamic properties, the so-called SEV systems with an intermediate level of accelerator and sulfur are employed. The levels of accelerator and sulfur in CV, SEV and EV systems are shown in **Table 1**.

| Table 1 | e 1 CV, SEV and EV vulcanisation systems |                         |              |
|---------|--|-------------------------|--------------|
| Туре    | Sulfur<br>(S, phr)                       | Accelerator<br>(A, phr) | A/S<br>ratio |
| CV      | 2.0-3.5                                  | 1.2-0.4                 | 0.1-0.6      |
| SEV     | 1.0-1.7                                  | 2.4-1.2                 | 0.7-2.5      |
| EV      | 0.4-0.8                                  | 5.0-2.0                 | 2.5-12       |

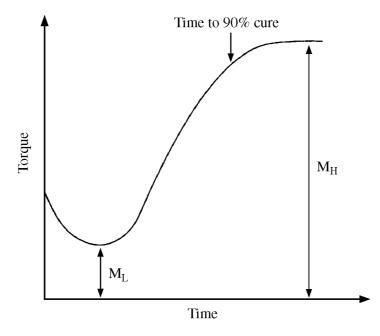
Many studies have documented both the advantages (increased age resistance), and the disadvantages (impaired fatigue resistance) of EV and SEV systems. The worse fatigue resistance correlates to lower amounts of polysulfidic crosslinks in the network. The CV systems provide higher amounts of poly- and disulfidic crosslinks and higher proportions of sulfidic and non-sulfidic modifications. This combination provides high flex fatigue resistance but at the expense of heat and reversion resistance. The vulcanisate structures and properties for CV, SEV and EV systems are shown in **Table 2**.

It is evident that there are trade-offs in the use of efficient vulcanisation systems. Besides the technical trade-off of improved ageing but inferior fatigue resistance, there are cost considerations – a 10% increase might be expected.

#### 1.2 Measuring Cure

The vulcanisation characteristics of a rubber are usually followed using a rheometer. In one version of such a device the sample of rubber is enclosed within a heated chamber. Vulcanisation is measured by the increase in the torque required to maintain a given amplitude of oscillation at a given temperature. The torque is proportional to low strain modulus of elasticity. The torque is plotted against time to give a so-called rheometer chart, rheograph or cure curve. A typical cure curve in shown in **Figure 1**.

| Table 2 Vulcanisate structures and properties |         |        |      |  |  |  |
|---|---------|--------|------|--|--|--|
| Factures                                      | Systems |        |      |  |  |  |
| Features                                      | CV      | SEV    | EV   |  |  |  |
| Poly- and disulfidic crosslinks (%)           | 95      | 50     | 20   |  |  |  |
| Monosulfidic crosslinks (%)                   | 5       | 50     | 80   |  |  |  |
| Cyclic sulfide (conc.)                        | High    | Medium | Low  |  |  |  |
| Non-sulfidic (conc.)                          | High    | Medium | Low  |  |  |  |
| Reversion resistance                          | Low     | Medium | High |  |  |  |
| Heat ageing resistance                        | Low     | Medium | High |  |  |  |
| Fatigue resistance                            | High    | Medium | Low  |  |  |  |
| Heat build up                                 | High    | Medium | Low  |  |  |  |
| Tear resistance                               | High    | Medium | Low  |  |  |  |
| Compression set (%)                           | High    | Medium | Low  |  |  |  |



**Figure 1** A typical cure curve

The curve exhibits a number of features which are used to compare cure:

- Maximum torque, M<sub>H</sub>
- ts2, T2 or T5: There is a delay or induction time before the torque or resistance value begins to rise. Because the onset of this rise is difficult to determine precisely, it is normal to note the point

at which the torque rises to a prescribed level above minimum. Suitably chosen, this provides a measure of the scorch time at the curing temperature.

ts2 or T2: The time to reach a 2 unit increase in torque above minimum. This is another way to express scorch safety. ts2 is defined as the time to achieve 2% cure above minimum.

T5: The scorch time at lower temperature is of importance too. This can be obtained by using a Mooney Viscometer at lower temperature. A Mooney Viscometer is also used to measure the viscosity of the compounds (important for dictating injection-moulding behaviour). The viscometer is also used to assess the tendency to scorch, and sometimes the rate of cure of a compound. A useful estimate of scorch behaviour is represented by T5, the time taken from the beginning of the warm-up period to that at which the Mooney value rises five units above the minimum value.

• T90: The most useful information obtained from the rheometer curve is T90, which is defined as the time to achieve 90% cure. Mathematically, T90 is the time for the torque to increase to:

 $90/100(M_{\rm H} - M_{\rm L}) + M_{\rm L}$ 

• Cure rate: A rise in the value of torque with time, the slope of the curve, gives the measure of cure rate. Sometimes cure rates of various cure systems are compared with T90–ts2 data.

#### **1.3 Test Equipment and Conditions**

Later in this review many results are presented from tests on different cure systems carried out by the author. Conditions and equipment used are now briefly described. Cure characteristics were determined using an MDR 2000EA rheometer. Test specimens were vulcanised by compression molding in a Fontyne TP-400 press at temperatures and times indicated.

Stress-strain properties were determined according to ISO 37, tear strength according to ISO 34/1, DIN abrasion ISO 4649, fatigue to failure ASTM 4482/85 and hardness according to ISO 48. Ageing of the test specimens was carried out in a ventilated air oven at 100 °C for 3 days (ISO 188). Heat build up and permanent set after dynamic loading were determined using a Goodrich Flexometer (Load 11 kg or 22 kg; stroke 0.445 cm, frequency 30 Hz, start temperature 100 °C) according to ISO 4666/3-1982. Dynamic mechanical analysis was carried out using a RDA-700 (prestrain 0.75%, frequency 15 Hz and temperature 60 °C) according to ASTM D 2231.

Vulcanisate network structure was determined by equilibrium swelling in toluene using the method reported by Ellis and Welding. The volume fraction (Vr) obtained was converted into the Mooney-Rivlin elastic constant (C1) and finally into the concentration of chemical crosslinks. The proportions of mono-, di-, and polysulfidic crosslinks in the vulcanisates were determined using thiol amine chemical probes. Following the cleavage of the poly- and disulfidic crosslinks, the samples were treated with methyl iodide to distinguish carbon-carbon based crosslinks from monosulfidic crosslinks.

The brass coated steelcord used in adhesion tests was of a 3+9x 0.22+1 construction with a Cu content of 63%. The rubber to metal adhesion characteristics were determined according to ASTM 2229-85. The wire adhesion data quoted are averages of 10 individual tests. Wire adhesion samples were aged under the following conditions:

- Heat aged 3 days at 105 °C in the presence of air
- Steam aged 2 days at 121 °C
- Salt aged 7 days at 25 °C in a 10% solution of NaCl.

#### 2 Curing Systems

Curing systems can be classified into four categories. Guidelines, and examples of selecting different curing systems for crosslinking are reviewed in this section.

#### 2.1 Sulfur Curing Systems

Initially, vulcanisation was accomplished by heating elemental sulfur at a concentration of 8 parts per hundred parts of rubber (phr) for 5 h at 140 °C. The addition of zinc oxide reduced the time to 3 h. Accelerators in concentrations as low as 0.5 phr have since reduced times to 1-3 min. As a result, elastomer vulcanisation by sulfur without accelerator is no longer of commercial significance. An exception is the use of about 30 or more phr of sulfur, with little or no accelerator, to produce moulded products of a hard rubber called ebonite.

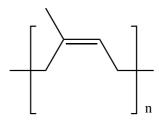
#### 2.1.1 Accelerators

Organic chemical accelerators were not used until 1906, 65 years after the Goodyear-Hancock development of unaccelerated vulcanisation, when the effect of aniline on sulfur vulcanisation was discovered by Oenslayer (405, a.2).

Aniline, however, is too toxic for use in rubber products. Its less toxic reaction product with carbon disulfide, thiocarbanilide, was introduced as an accelerator in 1907. Further developments led to guanidine accelerators (a.3). Reaction products formed between carbon disulfide and aliphatic amines (dithiocarbamates) were first used as accelerators in 1919 (a.4). These were and still are the most active accelerators in respect to both crosslinking rates and extent of crosslink formation. However, most dithiocarbamate accelerators give little or no scorch resistance and therefore cannot be used in all applications.

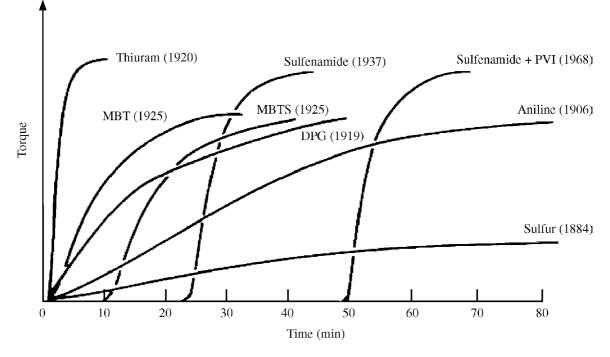
The first delayed action accelerators were introduced in 1925 with the development of 2-mercaptobenzothiazole (MBT) and 2-mercaptobenzothiazole disulfide (or 2,2'-dithiobisbenzothiazole) (MBTS) (a.5-a.7). Even more delayed action and yet faster-curing vulcanisation became possible in 1937 with the introduction of the first commercial benzothiazolesulfenamide accelerator (a.8, a.9). Further progress was made in 1968 with the introduction (a.10) of pre-vulcanisation inhibitor (PVI), *N*-cyclohexylthiophthalimide (CTP), which can be used in small concentrations together with benzothiazole sulfenamide accelerators. The history of the progress toward faster vulcanisation with better control of premature vulcanisation or scorch is illustrated by **Figure 2**.

Accelerated sulfur vulcanisation is the most widely used method. This method is useful to vulcanise NR, SBR, BR, IIR, NBR, chloroprene rubber (CR), XIIR and EPDM rubber. The reactive moiety present in all these rubbers is:



There are a wide variety of accelerators available to the compounder; including accelerator blends these number well over 100. Accelerators or combinations of accelerators are now available to enable vulcanisation to be efficiently carried out over a wide range of time/temperature/thickness from a few seconds at 200 °C to a few hours at room temperature.

Accelerators may be classified in several ways: (a) inorganic or organic, (b) acidic or basic, (c) by chemical type, or (d) by speed of the cure, giving rise to the terms slow, medium, semi-ultra and ultra.





Crosslinking activities of different accelerators in NR at 140 °C. The approximate year of commercial introduction is given in parentheses (405)

Functionally, accelerators can also be classified into two broad categories:

- Primary accelerators: Primary accelerators are mercapto based accelerators, generally efficient and confer good processing safety to the rubber compounds, exhibiting a broad vulcanisation plateau with relatively low crosslink density. Examples are sulfenamides and thiazoles. Primary accelerators provide considerable scorch delay, medium fast cure, and good modulus development.
- Secondary accelerators: Some rubber compounds use only one accelerator but most contain two, a

primary at about 1 phr and a secondary (or booster) at 0.1 to 0.5 phr. These combinations cause faster vulcanisation than each product separately and a considerable activation of cure which is positive for the general property spectrum of the vulcanisates. Examples are guanidines, thiurams, dithiocarbamates, dithiophosphates, etc. Secondary accelerators are usually scorchy, and very fast cure.

In order to rationalise the extensive range of accelerator materials it is useful to classify them in terms of their generic chemical structure listed next, some examples are shown in **Figure 3**. The structures of commonly used accelerators are given in **Table 3**.

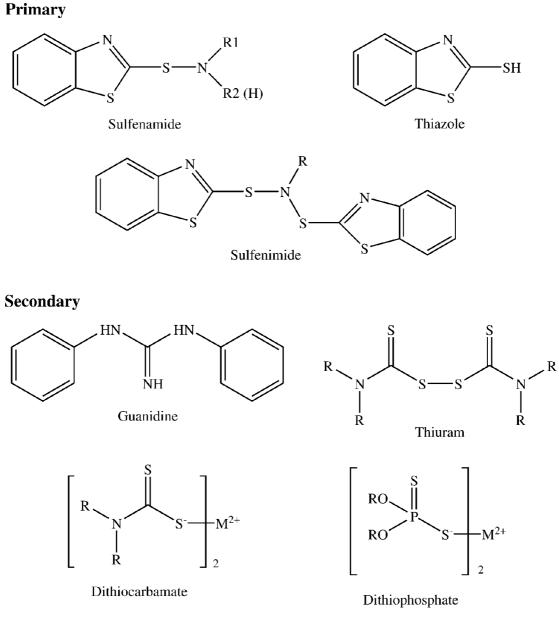


Figure 3 Chemical structure of accelerators

| Table 3 Accelerators for sulfur vulcanisation |              |   |  |  |  |  |
|---|--------------|---|--|--|--|--|
| Compound                                      | Abbreviation | Structure   |  |  |  |  |
| Benzothiazole                                 |              |   |  |  |  |  |
| 2-mercaptobenzothiazole                       | MBT          | SH SH   |  |  |  |  |
| 2,2'-dithiobenzothiazole                      | MBTS         |   |  |  |  |  |
| Benzothiazolesulfenamide                      |              |   |  |  |  |  |
| N-cyclohexylbenzothiazole-2-sulfenamide       | CBS          | S-NH-   |  |  |  |  |
| N-butylbenzothiazole-2-sulfenamide            | TBBS         | S-NH-   |  |  |  |  |
| 2-morpholinothiobenzothiazole                 | MBS          | S S S S S S S S S S S S S S S S S S S   |  |  |  |  |
| N-dicyclohexylbenzothiazole-2-sulfenamide     | DCBS         |   |  |  |  |  |
| Thiuram                                       |              |   |  |  |  |  |
| tetramethylthiuram monosulfide                | TMTM         | (CH <sub>3</sub> ) <sub>2</sub> —N S N—(CH <sub>3</sub> ) <sub>2</sub>                                |  |  |  |  |
| tetramethylthiuram disulfide                  | TMTD         | $\left[\begin{array}{c} \\ (CH_3)_2 \\ \end{array} \\ N \\ \end{array} \\ S \\ \end{array} \right]_2$ |  |  |  |  |

| Table 3 Acce                 | lerators for sulf | fur vulcanisation  |
|------------------------------|-------------------|--|
| Compound                     | Abbreviation      | Structure  |
| Thiuram (Continued)          |                   |  |
| tetraethylthiuram disulfide  | TETD              | $\begin{bmatrix} & & S \\ & & & & \\ & & & \\ & & & & \\ $  |
| tetrabenzylthiuram disulfide | TBzTD             | $\left[ \left[ \begin{array}{c} \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\$  |
| Dithiocarbamate              |                   |  |
| zinc dimethyldithiocarbamate | ZDMC              | $\begin{bmatrix} & & \\ (CH_3)_2 & & \\$  |
| zinc diethyldithiocarbamate  | ZDEC              | $\begin{bmatrix} S \\ Et_2 \\ N \\ S \\ 2 \end{bmatrix}_2 Zn^{2+}$   |
| zinc dibenzyldithiocarbamate | ZBEC              | $ \begin{bmatrix} & & \\ &$ |
| Guanidines                   |                   |  |
| diphenyl guanidine           | DPG               | H H H H H H H H H H H H H H H H H H H  |
| di-o-tolylguanidine          | DOTG              | H H H<br>N NH  |

Classification of accelerators by chemical structure:

Class 1 Sulfenamides

- Class 2 Thiazoles Class 3 Guanidines
- Class 4 Thiurams Class 5 Dithiocarbamates
- Class 6 Dithiophosphates
- Class 7 Aldehyde-amine (no longer in use) Class 8 Sulfenimide (new)

**Table 4** provides comparisons of the different classes of accelerators based on their rates of vulcanisation. The secondary accelerators are seldom used alone, but generally are found in combination with primary accelerators to gain faster cures.

**Figure 4** compares five different classes of accelerators in NR compounds. Thiurams and dithiocarbamates are fast but relatively scorchy. Thiazoles (e.g., MBTS) and guanidines (e.g., DPG) are scorchy, slow accelerators and seldom used alone. On the other hand sulfenamides (e.g., TBBS) exhibit excellent scorch safety and moderate cure rate with a good torque development.

| Table 4 Comparison of different classes of accelerators (a.11) |                     |  |  |  |  |
|--|---------------------|--|--|--|--|
| Class  | Vulcanisation rate  |  |  |  |  |
| Aldehyde-amine   | Slow                |  |  |  |  |
| Guanidines   | Medium              |  |  |  |  |
| Thiazoles  | Semi-fast           |  |  |  |  |
| Sulfenamides   | Fast-delayed action |  |  |  |  |
| Sulfenimides   | Fast-delayed action |  |  |  |  |
| Dithiophosphates   | Fast                |  |  |  |  |
| Thiurams   | Very fast           |  |  |  |  |
| Dithiocarbamates   | Very fast           |  |  |  |  |

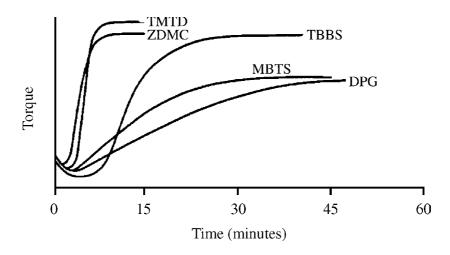
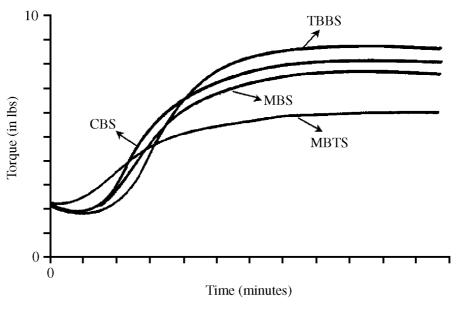


Figure 4 Comparison of accelerators/sulfur (0.5/2.5 phr) in NR (a.11)





Comparison of primary accelerators/sulfur (0.5/2.5 phr) in NR at 144 °C (a.11)

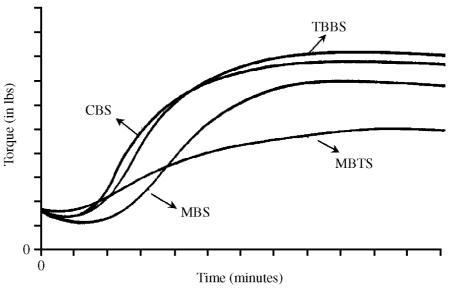


Figure 6

Comparison of primary accelerators/sulfur (1.2/1.5 phr) in SBR (a.11)

A comparison of cure between different primary accelerators in NR and SBR is shown in **Figures 5** and 6 respectively. The thiazole based accelerators show different cure patterns in different rubbers. For example, TBBS shows better scorch than CBS and MBS in NR, whereas MBS shows improved scorch compared with TBBS and CBS in SBR.

A large number of secondary accelerators can be used with primary accelerators, thereby providing a great deal of flexibility in processing and curing properties. **Tables 5 and 6** (a.11) examine some of the more commonly used secondary accelerators and their effect with sulfenamide accelerator, TBBS. Seven different secondary accelerators are evaluated with TBBS in both

| Table 5 Comparis                         | Table 5 Comparison of secondary accelerators in NR (a.11) |           |           |            |            |         |      |      |
|--|---|-----------|-----------|------------|------------|---------|------|------|
| Ingredients/stocks                       | 1   | 2         | 3         | 4          | 5          | 6       | 7    | 8    |
| Sulfur                                   | 2.5   | 2.5       | 2.5       | 2.5        | 2.5        | 2.5     | 2.5  | 2.5  |
| MBTS                                     | 1.2   | 0         | 0         | 0          | 0          | 0       | 0    | 0    |
| DPG                                      | 0.4   | 0         | 0         | 0          | 0          | 0       | 0    | 0    |
| TBBS                                     | 0   | 0.6       | 0.6       | 0.6        | 0.6        | 0.6     | 0.6  | 0.6  |
| TMTD                                     | 0   | 0.4       | 0         | 0          | 0          | 0       | 0    | 0    |
| ТМТМ                                     | 0   | 0         | 0.4       | 0          | 0          | 0       | 0    | 0    |
| TETD                                     | 0   | 0         | 0         | 0.4        | 0          | 0       | 0    | 0    |
| ZDMC                                     | 0   | 0         | 0         | 0          | 0.4        | 0       | 0    | 0    |
| ZDEC                                     | 0   | 0         | 0         | 0          | 0          | 0.4     | 0    | 0    |
| ZDBC                                     | 0   | 0         | 0         | 0          | 0          | 0       | 0.4  | 0    |
| DOTG                                     | 0   | 0         | 0         | 0          | 0          | 0       | 0    | 0.4  |
| Mooney scorch at 120 °C, T5 (minutes)    | 7.2   | 16.8      | 21.5      | 23.5       | 13.7       | 16.7    | 20.2 | 21.7 |
| Rheometer at 145 °C                      |   | 1         |           |            | 1          |         |      | 1    |
| T90 (minutes)                            | 9.2   | 7.5       | 9.5       | 9.8        | 7.0        | 7.8     | 9.3  | 16.5 |
| Modulus at 100% elongation (MPa)         | 2.6   | 3.4       | 3.5       | 3.0        | 3.1        | 2.9     | 2.8  | 2.8  |
| Elongation at break (%)                  | 550   | 450       | 430       | 480        | 470        | 500     | 510  | 510  |
| Formulation: NR 100; FEF Black 40; aroma | tic oil 10.   | 0; zinc o | xide 5.0; | stearic ac | cid 1.5; 6 | PPD 2.0 |      |      |

| 1<br>1.8<br>1.2 | <b>2</b><br>1.8                                    | <b>3</b>  | 4   | 5   | 6   | 7   |
|-----------------|--|---|---|---|---|---|
|                 | 1.8  | 1.8   |   |   |   | 1 .   |
| 1.2             |  | 1.0   | 1.8   | 1.8   | 1.8   | 1.8   |
|                 | 0  | 0   | 0   | 0   | 0   | 0   |
| 0.4             | 0  | 0   | 0   | 0   | 0   | 0   |
| 0               | 0.5  | 0.5   | 0.5   | 0.5   | 0.5   | 0.5   |
| 0               | 0.3  | 0   | 0   | 0   | 0   | 0   |
| 0               | 0  | 0.3   | 0   | 0   | 0   | 0   |
| 0               | 0  | 0   | 0.3   | 0   | 0   | 0   |
| 0               | 0  | 0   | 0   | 0.3   | 0   | 0   |
| 0               | 0  | 0   | 0   | 0   | 0.3   | 0   |
| 0               | 0  | 0   | 0   | 0   | 0   | 0.3   |
| 10.4            | 12.3   | 22.0  | 14.5  | 13.2  | 18.7  | 24.4  |
|                 | 1  |   |   | 1   |   |   |
| 9.2             | 7.4  | 9.3   | 8.6   | 8.7   | 12.0  | 21.3  |
| 2.0             | 2.1  | 2.1   | 2.0   | 1.9   | 1.9   | 1.6   |
| 450             | 430  | 420   | 430   | 470   | 470   | 500   |
|                 | 0<br>0<br>0<br>0<br>0<br>10.4<br>9.2<br>2.0<br>450 | 0         0.3           0         0           0         0           0         0           0         0           0         0           0         0           0         0           0         0           0         0           10.4         12.3           9.2         7.4           2.0         2.1           450         430 | $\begin{array}{c ccccccccccccccccccccccccccccccccccc$ | $\begin{array}{c ccccccccccccccccccccccccccccccccccc$ | $\begin{array}{c ccccccccccccccccccccccccccccccccccc$ | $\begin{array}{c ccccccccccccccccccccccccccccccccccc$ |

NR and SBR compounds with MBTS/DPG as the control. The secondary accelerators also change the network structures. The polysulfidic crosslinks are converted to monosulfidic exhibiting heat stability.

# 2.1.2 Sulfur Donors

Aside from the sulfur itself, sulfur bearing compounds that liberate sulfur at the vulcanisation temperature can be used as vulcanising agents. A few sulfur donors are given in Table 7, which includes some compounds like dithiodimorpholine (DTDM), which can directly substitute sulfur. Others, like TMTD, can act simultaneously as vulcanisation accelerators. The amount of active sulfur, as shown in Table 7, is different for each compound. Sulfur donors may be used when a high amount of sulfur is not tolerated in the compounding recipe, for example, high temperature vulcanisation of rubber. They are used in EV and SEV systems. Sulfur donors are used to generate a network capable of resistance to degradation on exposure to heat. Generally sulfur donors convert intially formed polysulfides to monosulfides which is characteristic for EV or SEV systems.

A sulfur donor system in NR (DTDM 1.5; CBS 1.5; TMTD 0.5) yields a stable network structure with a contribution of 80% mono- and disulfidic crosslinks at the optimum cure at 143 °C or 183 °C curing (411).

*cis*-Isoprene rubber cured with bis(diisopropyl) thiophosphoryl disulfide (DIPDIS) at 160 °C, produces a predominantly monosulfidic network structure (412). Similar work on heat resistant network structures has been carried out on other synthetic rubbers. For example, a sulfurless system using 1 phr TBBS, 2.0 phr DTDM and 0.4 phr TMTD in SBR gives the best ageing resistance (409).

Although ultra accelerators or sulfur donors can be used together with primary accelerators to improve cure rate as well as heat resistance (393, 404, 413), their use is restricted because of concerns over the carcinogenic nature of the *N*-nitrosamines (NA) formed from the parent amines (397). Accelerators derived from secondary amines, for example MBS, TMTD, TETD, TMTM, and OTOS fall into this category. The combination of a sulfenamide, such as CBS or TBBS, and a thiuram, such as TMTD or TETD, shows high cure rates but suffers from adverse effects on scorch resistance and vulcanisate

| Table 7 Sulfur donors  |  |                         |              |  |  |  |  |  |
|--|--|-------------------------|--------------|--|--|--|--|--|
| Material   | Structure  | Active<br>sulfur<br>(%) | M.P.<br>(°C) |  |  |  |  |  |
| TMTD   | $\begin{bmatrix} & & & \\ (CH_3)_2 & & N & \\ & & & & \\ & & & \\ & & & & \\$ | 13.3                    | 124          |  |  |  |  |  |
| 4,4'-dithiodimorpholine<br>(DTDM)                                      | o N S S N O  | 13.6                    | 125          |  |  |  |  |  |
| 2-morpholino-dithio-benzothiazole<br>(MBSS)                            | s—s—s—o  | 28.4                    | 129          |  |  |  |  |  |
| caprolactam disulfide<br>(CLD)   | N-S-S-N  | 28.8                    | 131          |  |  |  |  |  |
| Dipentamethylenethiuram tetrasulfide<br>(DPTT)                         | $S_{4}$ $N$  | 16.6                    | 120          |  |  |  |  |  |
| N-oxydiethylene thiocarbamyl-N'-<br>oxydiethylenesulfenamide<br>(OTOS) | S S S S S S S S S S S S S S S S S S S  | 12.9                    | 136          |  |  |  |  |  |
| TBzTD*   | $\left[ \begin{array}{c} \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\$   | 5.8                     | 124          |  |  |  |  |  |
| *TBzTD acts as sulfur donor only at ver                                | y high concentration   |                         |              |  |  |  |  |  |

dynamic properties (119, a.12), as well as the nitrosamine issue. The solution to this has been to introduce nitrosamine safe ultra accelerators such as TBzTD (396, a.13).

Unlike TMTD, TBzTD is unique since use of a small amount of TBzTD (0.1-0.2 phr) with a sulfenamide system does not influence the processing characteristics, but does improve cure rate and dynamic properties. Datta and co-workers (4) investigated the effects of using a small amount of TBzTD in NR formulations cured at 150 °C for 90 minutes. **Table 8** lists the formulations used, **Table 9** illustrates the processing and cure characteristics of the different rubbers, **Table 10** shows the vulcanisate properties, **Table 11** indicates the crosslink types found in network studies and **Table 12** gives the viscoelastic properties of the different formulations.

| Table 8 Formulations |     |     |     |     |     |  |  |
|----------------------|-----|-----|-----|-----|-----|--|--|
| Ingredients/stocks   | 1   | 2   | 3   | 4   | 5   |  |  |
| NR SMR CV            | 100 | 100 | 100 | 100 | 100 |  |  |
| N-330                | 50  | 50  | 50  | 50  | 50  |  |  |
| Zinc oxide           | 5   | 5   | 5   | 5   | 5   |  |  |
| Stearic acid         | 2   | 2   | 2   | 2   | 2   |  |  |
| Santoflex 6PPD       | 2   | 2   | 2   | 2   | 2   |  |  |
| Santocure CBS        | 0.6 | 0.6 | 0.6 | 0.6 | 0.6 |  |  |
| Perkacit TBzTD       | -   | 0.1 | 0.2 | -   | -   |  |  |
| Perkacit TMTD        | -   | -   | -   | 0.1 | 0.2 |  |  |
| Sulfur               | 2.3 | 2.3 | 2.3 | 2.3 | 2.3 |  |  |

| Table 9 Processing and cure characteristics             |              |                       |                       |                      |                      |  |  |  |
|---|--------------|-----------------------|-----------------------|----------------------|----------------------|--|--|--|
| Properties  | 1<br>Control | 2<br>0.1 phr<br>TBzTD | 3<br>0.2 phr<br>TBzTD | 4<br>0.1 phr<br>TMTD | 5<br>0.2 phr<br>TMTD |  |  |  |
| Processing data   |              |                       |                       |                      |                      |  |  |  |
| Mooney viscosity<br>ML(1+4) 100 °C<br>(Mooney unit, MU) | 51           | 52                    | 52                    | 53                   | 51                   |  |  |  |
| Mooney scorch, 121                                      | °C           |                       |                       |                      |                      |  |  |  |
| T5 (minutes)  | 30           | 30                    | 27                    | 22                   | 18                   |  |  |  |
| Rheometer at 150 °C                                     | 2            |                       |                       |                      |                      |  |  |  |
| M <sub>L</sub> (Nm)                                     | 0.20         | 0.20                  | 0.20                  | 0.21                 | 0.20                 |  |  |  |
| $M_{\rm H}M_{\rm L}({\rm Nm})$                          | 1.66         | 1.82                  | 1.90                  | 1.85                 | 2.03                 |  |  |  |
| ts2 (minutes)   | 3.8          | 3.7                   | 3.4                   | 3.0                  | 2.5                  |  |  |  |
| T90 (minutes)   | 10.0         | 7.3                   | 5.8                   | 6.2                  | 4.6                  |  |  |  |
| Cure rate (T90-ts2)                                     | 6.2          | 3.6                   | 2.4                   | 3.2                  | 2.1                  |  |  |  |

| Table 10 Vulcanisate properties (cure: 150 °C/T90)   |              |                       |                       |                      |                      |  |
|--|--------------|-----------------------|-----------------------|----------------------|----------------------|--|
| Properties   | 1<br>Control | 2<br>0.1 phr<br>TBzTD | 3<br>0.2 phr<br>TBzTD | 4<br>0.1 phr<br>TMTD | 5<br>0.2 phr<br>TMTD |  |
| Hardness (IRHD)                                      | 72           | 74                    | 75                    | 74                   | 75                   |  |
| Modulus at 100% elongation (MPa)                     | 2.9          | 3.6                   | 3.8                   | 3.8                  | 4.2                  |  |
| Modulus at 300% elongation (MPa)                     | 14.0         | 15.7                  | 16.4                  | 16.0                 | 17.5                 |  |
| Tensile strength (MPa)                               | 27.7         | 28.1                  | 28.7                  | 29.0                 | 28.3                 |  |
| Elongation at break (%)                              | 530          | 500                   | 505                   | 525                  | 480                  |  |
| Compression set (%)<br>(100 °C/72 h)<br>(70 °C/24 h) | 55<br>37     | 53<br>32              | 50<br>29              | 49<br>33             | 45<br>29             |  |
| Fatigue to failure (kc) (100% extension)             | 143          | 142                   | 142                   | 122                  | 122                  |  |
| Abrasion loss (mm <sup>3</sup> )                     | 104          | 101                   | 94                    | 93                   | 91                   |  |

| Table 11 Distribution of crosslink types (cure: 150 °C/T90)       |              |                       |                       |                      |                      |  |  |
|---|--------------|-----------------------|-----------------------|----------------------|----------------------|--|--|
| Crosslink Concentration*  | 1<br>Control | 2<br>0.1 phr<br>TBzTD | 3<br>0.2 phr<br>TBzTD | 4<br>0.1 phr<br>TMTD | 5<br>0.2 phr<br>TMTD |  |  |
| Total   | 5.20         | 5.41                  | 5.62                  | 5.86                 | 6.25                 |  |  |
| Poly-sulfidic   | 4.15         | 2.34                  | 2.41                  | 2.30                 | 2.33                 |  |  |
| Di-sulfidic   | 0.88         | 0.78                  | 0.60                  | 0.58                 | 0.55                 |  |  |
| Mono-sulfidic   | 0.17         | 2.29                  | 2.61                  | 2.98                 | 3.37                 |  |  |
| *Expressed in gram moles/gram rubber hydrocarbon x10 <sup>5</sup> |              |                       |                       |                      |                      |  |  |

| Table 12 Vulcanisate viscoelastic properties (cure: 150 °C/T90) |              |                       |                       |                      |                      |  |  |
|---|--------------|-----------------------|-----------------------|----------------------|----------------------|--|--|
| Properties  | 1<br>Control | 2<br>0.1 phr<br>TBzTD | 3<br>0.2 phr<br>TBzTD | 4<br>0.1 phr<br>TMTD | 5<br>0.2 phr<br>TMTD |  |  |
| Storage modulus (E') (MPa)                                      | 7.9          | 8.6                   | 8.9                   | 8.7                  | 9.0                  |  |  |
| Loss modulus (E'') (MPa)  | 1.13         | 1.19                  | 1.21                  | 1.17                 | 1.12                 |  |  |
| Tan delta   | 0.143        | 0.139                 | 0.137                 | 0.135                | 0.125                |  |  |
| Conditions: temperature 60 °C, frequency 15 Hz and strain 2%    |              |                       |                       |                      |                      |  |  |

# **2.2 Cures for Speciality Elastomers**

The curing systems used to vulcanise speciality elastomers such as EPDM, CR, IIR and NBR are different than those used to cure NR, SBR, BR and its blends. The former elastomers are less unsaturated and therefore need a high ratio of accelerator to sulfur.

# 2.2.1 Cure Systems for EPDM

EPDM vulcanisates exhibit some unique properties such as ozone, heat, light, weathering and chemical resistance (a.14). Because of this attractive combination of properties, EPDM has taken over a wide variety of applications. EPDM has relatively low unsaturation and therefore requires complex cure systems to achieve the desired properties. Nearly every conceivable combination of curing ingredients has been evaluated in various EPDM polymers over the years (a.15), five systems are described in **Table 13**. In all cases, nitrosamine free or safe alternatives are looked for. For System 1, the following alternative was suggested:

| System 1 | NA free alternative |
|----------|---------------------|
| S 1.5    | S 1.3               |
| MBT 0.5  | MBT 0.75            |
| TMTD 1.5 | CBS 3.8             |

Comparative properties of the system 1 EPDM and the NA free alternative are listed **in Table 14**.

| Table 13 Cure systems in EPDM   |   |   |  |  |
|---|---|---|--|--|
| Systems (phr) Merits Drawbacks  |   |   |  |  |
| System 1<br>S 1.5<br>TMTD 1.5<br>MBT 0.5  | Low cost  | Bloom   |  |  |
| System 2<br>(Triple 8)<br>S 2.0<br>MBT 1.5<br>Tellurium diethyl dithiocarbamate (TDEC) 0.8<br>Dipentamethyl thiuram tetrasulfide (DPTT) 0.8<br>TMTD 0.8 | Excellent physical properties and fast cure               | Scorchy and expensive                             |  |  |
| System 3<br>S 0.5<br>ZDBC 3.0<br>ZDMC 3.0<br>DTDM 2.0<br>TMTD 3.0   | Excellent compression set and good heat ageing resistance | Bloom and very high cost                          |  |  |
| System 4<br>S 2.0<br>MBTS 1.5<br>ZDBC 2.5<br>TMTD 0.8   | Non-blooming  | Cure relatively slow and<br>worse compression set |  |  |
| System 5<br>(2121 system)<br>ZBPD 2.0<br>TMTD 1.0<br>TBBS 2.0<br>S 1.0  | Fast cure and good physical properties                    | Bloom   |  |  |

| Properties            | System 1 | NA free alternative |
|-----------------------|----------|---------------------|
| Mooney scorch @121 °C |          |                     |
| T5 (minutes)          | 12       | 19                  |
| T35 (minutes)         | 15       | 29                  |
| Compression set (%)   |          |                     |
| 72 h/23 °C            | 6        | 8                   |
| 72 h/70 °C            | 24       | 23                  |
| 72 h/100 °C           | 63       | 65                  |

Base formulation: Keltan 314, 25; Keltan 4903F 75; N-539 110; N-772 60; whiting 30; Sunpar 2280, 85; Escorez 1102, 5; Struktol WB 212, 2; wax 4000, 3; Caloxol W5, 6.4; stearic acid 1.5; zinc oxide 5.

| Table 15 Properties of system 2 EPDM and the NA free alternative |    |    |  |  |  |  |
|--|----|----|--|--|--|--|
| Properties Triple 8 Alternative                                  |    |    |  |  |  |  |
| Mooney scorch @121 °C  |    |    |  |  |  |  |
| T5 (minutes)   | 5  | 5  |  |  |  |  |
| T35 (minutes)  | 8  | 8  |  |  |  |  |
| Compression set (%)  |    |    |  |  |  |  |
| 72 h/23 °C   | 5  | 9  |  |  |  |  |
| 72 h/70 °C   | 33 | 35 |  |  |  |  |
| 72 h/100 °C  | 70 | 80 |  |  |  |  |

System 2, the 'Triple 8' system can be replaced by an NA-safe alternative as shown next. The comparative properties are listed in **Table 15**.

| System 2 (Triple 8) | NA free alternative |
|---------------------|---------------------|
| S 2.0               | S 1.5               |
| MBT 1.5             | ZMBT 2.0            |
| TDEC 0.8            | ZBEC 0.5            |
| DPTT 0.8            | ZBPD (75) 2.0       |
| TMTD 0.8            |                     |

System 5 (2121 system) can be replaced by an NA-safe alternative:

| System 5 (2121 system) | NA free alternative |  |  |
|------------------------|---------------------|--|--|
| ZBPD (75) 2.0          | ZBPD (75) 2.5       |  |  |
| TMTD 1.0               | 0                   |  |  |
| TBBS 2.0               | TBBS 2.0            |  |  |
| S 1.0                  | S 1.2               |  |  |

Recently an EPDM formulation has been developed based on DTDM, TMTD, ZDBC and DPTT called the '2828' system, it comprises:

| Curatives | phr |
|-----------|-----|
| DTDM      | 2.0 |
| TMTD      | 0.8 |
| ZDBC      | 2.0 |
| DPTT      | 0.8 |

The beauty of this system is having low compression set and non-bloom. Comparative data obtained by Flexsys for different formulations are listed in **Tables 16 and 17**.

| Table 16 Curing systems for low compression set |      |      |          |      |  |
|---|------|------|----------|------|--|
| Curatives/stocks                                | 1    | 2    | 3 (2828) | 4    |  |
| Sulfur  | 0.5  | 0.5  | 0        | 0.5  |  |
| DTDM  | 2.0  | 1.7  | 2.0      | 0    |  |
| TMTD  | 3.0  | 2.5  | 0.8      | 1.0  |  |
| ZDMC  | 3.0  | 2.5  | 0        | 0    |  |
| ZDBC  | 3.0  | 2.5  | 2.0      | 0    |  |
| DPTT  | 0    | 0    | 0.8      | 0    |  |
| CBS   | 0    | 0    | 0        | 2.0  |  |
| ZBPD (75)                                       | 0    | 0    | 0        | 3.2  |  |
| Properties                                      |      |      |          |      |  |
| Mooney viscosity at 135 °C                      |      |      |          |      |  |
| ML (1+4) (MU)                                   | 20.0 | 21.4 | 21.2     | 20.5 |  |
| T5 (min)  | 14.2 | 13.7 | 16.4     | 14.2 |  |

2,2,4-trimethyl-1,2-dihydroquinoline, polymerised (TMQ), 2.0

| Table 17 Properties of EPDM formulations for low compression set |      |      |          |      |  |
|--|------|------|----------|------|--|
| Properties   | 1    | 2    | 3 (2828) | 4    |  |
| Rheometer, 160 °C  |      |      | - I      |      |  |
| M <sub>H</sub> -M <sub>L</sub> (Nm)                              | 2.19 | 2.13 | 1.76     | 1.41 |  |
| ts2 (minutes)  | 5.0  | 4.8  | 6.7      | 5.2  |  |
| T90 (minutes)  | 11.2 | 11.2 | 15.2     | 11.2 |  |
| Physical properties (Cure: 160 °C/T90)                           |      |      |          |      |  |
| Hardness (Shore A)   | 70   | 69   | 70       | 66   |  |
| Modulus at 100% elongation (MPa)                                 | 3.6  | 2.7  | 2.3      | 1.9  |  |
| Modulus at 300% elongation (MPa)                                 | 6.8  | 6.4  | 5.7      | 4.7  |  |
| Tensile strength (MPa)   | 8.8  | 8.3  | 7.8      | 6.5  |  |
| Elongation at break (%)  | 550  | 545  | 670      | 640  |  |
| Aged 70 h/121 °C   |      |      | - I I    |      |  |
| Hardness (Shore A)   | 72   | 73   | 70       | 71   |  |
| Modulus at 100% elongation (MPa)                                 | 3.4  | 3.2  | 2.5      | 2.8  |  |
| Modulus at 300% elongation (MPa)                                 | 8.2  | 7.8  | 6.5      | 6.7  |  |
| Tensile strength (MPa)   | 9.1  | 8.9  | 7.9      | 7.8  |  |
| Elongation at break (%)  | 435  | 425  | 560      | 450  |  |
| Compression set  |      | 1    |          |      |  |
| 70 h/121 °C (%)  | 57   | 58   | 60       | 76   |  |

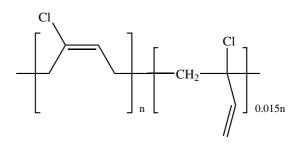
# 2.2.2 Cure Systems for Nitrile Rubber

Cure systems for nitrile rubber are somewhat analogous to those of NR, SBR or BR except that magnesium carbonate (MC) treated sulfur is usually used to aid sulphur dispersion into the polymer (a.12). Common accelerator systems include thiazole, thiuram, thiazole/thiuram, or sulfenamide/ thiuram types. Examples of these systems are shown in **Table 18**.

As operating requirements for nitrile rubber become more stringent, improved ageing and set properties become important. In order to address those requirements, formulations with sulfur donor systems (as a partial or total replacement of rhombic sulfur) have been proposed. These are summarised in **Tables 19 and 20**. The advantages of these systems are improved set and ageing resistance while adequate processing safety and fast cures are maintained.

# 2.2.3 Cure Systems for Polychloroprene

Chlorobutadiene or chloroprene rubbers (CR), also called neoprene rubbers, are usually vulcanised by the action of metal oxides. The crosslinking agent is usually zinc oxide in combination with magnesium oxide (a.16). CR can be vulcanised in the presence of zinc oxide alone, but magnesium oxide is necessary to confer scorch resistance. The reaction may involve the allylic chlorine atom, which is the result of the small amount of 1,2 polymerisation (a.17).



| Curatives/stocks                              | 1    | 2    | 3    |
|---|------|------|------|
| MC treated sulfur                             | 1.5  | 1.5  | 1.5  |
| TMTM  | 0.4  | 0    | 0    |
| MBTS  | 0    | 1.5  | 0    |
| TBBS  | 0    | 0    | 1.2  |
| TMTD  | 0    | 0    | 0.1  |
| Processing and curing properties              |      |      | I    |
| Mooney scorch at 121 °C, T5 (minutes)         | 6.8  | 8.1  | 5.7  |
| Rheometer at 160 °C, T90 (minutes)            | 8.7  | 15.2 | 4.7  |
| Physical properties (Cure: 160 °C/90 minutes) |      |      | I    |
| Hardness (Shore A)                            | 73   | 71   | 75   |
| Modulus at 100% elongation (MPa)              | 4.2  | 3.6  | 5.0  |
| Tensile strength (MPa)                        | 16.3 | 16.2 | 17.3 |
| Elongation at break (%)                       | 380  | 475  | 355  |
| Heat ageing at 100 °C/72 h                    |      |      | I    |
| Hardness (Shore A)                            | 80   | 78   | 82   |
| Tensile strength retention (%)                | 85   | 68   | 57   |
| Compression set                               |      | 1    | 1    |
| 100 °C/22 h (%)                               | 31   | 50   | 55   |

| Table 19 High sulfur versus low sulfur cure systems for nitrile rubber |      |      |  |
|--|------|------|--|
| Curatives/stocks   | 1    | 2    |  |
| MC treated sulfur  | 1.5  | 0.3  |  |
| MBT  | 1.5  | 0    |  |
| TBBS   | 0    | 1.0  |  |
| TMTD   | 0    | 1.0  |  |
| Mooney scorch at 135 °C  |      | 1    |  |
| T5 (minutes)   | 8.1  | 8.1  |  |
| Rheometer at 160 °C  |      | I    |  |
| T90 (minutes)  | 15.2 | 10.2 |  |
| Physical properties (Cure: 160 °C/T90)                                 |      | 1    |  |
| Hardness (Shore A)   | 71   | 69   |  |
| Modulus at 100% elongation (MPa)                                       | 3.6  | 3.1  |  |
| Tensile strength (MPa)   | 16.2 | 15.1 |  |
| Elongation at break (%)  | 475  | 485  |  |
| Heat ageing at 100 °C/70 h)  |      | I    |  |
| Hardness (Shore A)   | 78   | 74   |  |
| Tensile strength retention (%)   | 68   | 89   |  |
| Compression set  |      | 1    |  |
| 100 °C/22 h (%)  | 50   | 24   |  |
|  |      |      |  |

| Table 20 Sulfurless cure systems for nitrile rubber |      |      |      |      |
|---|------|------|------|------|
| Curatives/stocks                                    | 1    | 2    | 3    | 4    |
| MC treated sulfur                                   | 1.5  | 0    | 0    | 0    |
| MBTS  | 1.5  | 0    | 0    | 0    |
| TBBS  | 0    | 1.0  | 3.0  | 2.0  |
| TMTD  | 0    | 1.0  | 3.0  | 2.0  |
| DTDM  | 0    | 1.0  | 1.0  | 2.0  |
| Mooney scorch at 135 °C                             |      |      |      |      |
| T5 (minutes)  | 8.1  | 10.7 | 7.0  | 7.9  |
| Rheometer at 160 °C                                 |      |      |      |      |
| T90 (minutes)                                       | 15.2 | 14.7 | 12.5 | 13.3 |
| Physical properties (Cure: 160 °C/T90)              |      |      |      | 1    |
| Hardness (Shore A)                                  | 71   | 68   | 71   | 73   |
| Modulus at 100% elongation (MPa)                    | 3.6  | 3.1  | 4.3  | 5.7  |
| Tensile strength retention (MPa)                    | 16.2 | 15.4 | 16.3 | 16.7 |
| Elongation at break (%)                             | 475  | 485  | 360  | 290  |
| Heat ageing at 100 °C/70 h                          | -    |      |      |      |
| Hardness (Shore A)                                  | 78   | 73   | 75   | 78   |
| Tensile strength retention (%)                      | 68   | 87   | 89   | 83   |
| Compression set                                     |      |      |      |      |
| 100 °C/22 h (%)                                     | 50   | 22   | 13   | 12   |

Most accelerators used in the sulfur vulcanisation of other high diene rubbers are not applicable to the metal oxide vulcanisation of CR. An exception is the use of a so-called mixed curing system for CR, in which metal oxide and accelerated sulfur vulcanisation are combined. Along with the metal oxides, TMTD, DOTG and sulfur are used. This is a good method to obtain high resilience and dimensional stability.

The accelerator that has been widely used with metal oxide cures is ethylene thiourea (ETU) or 2-mercaptoimidazoline. Further extensive use of ETU in vulcanisation of CR is restricted because it is a suspected carcinogen. The related compound, thiocarbanilide, used formerly as an accelerator for sulfur vulcanisation, has been revived for CR vulcanisation; other substitutes for ETU have been proposed (401, 406).

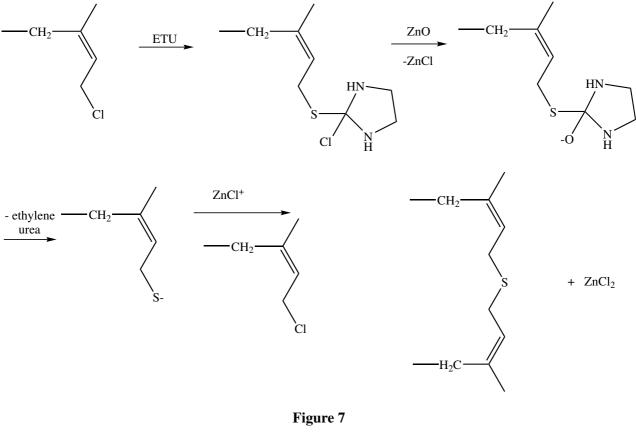
The following mechanism for ETU acceleration has been proposed (a.18), **Figure 7**.

Recipes for metal oxide vulcanisation of CR are given in **Table 21**. In one case, calcium stearate was used instead of magnesium oxide to obtain better ageing characteristics (a.19).

| Table 21 Vulcanisation systems for CR (phr) |     |     |   |  |  |  |  |
|---|-----|-----|---|--|--|--|--|
| Curatives/Stock123                          |     |     |   |  |  |  |  |
| Zinc oxide                                  | 5   | 5   | 5 |  |  |  |  |
| Magnesium oxide                             | 4   | 0   | 4 |  |  |  |  |
| Calcium stearate                            | 0   | 5.5 | 0 |  |  |  |  |
| Stearic acid                                | 0   | 0   | 1 |  |  |  |  |
| ТМТМ  | 0   | 0   | 1 |  |  |  |  |
| DOTG  | 0   | 0   | 1 |  |  |  |  |
| ETU   | 0.5 | 0.5 | 0 |  |  |  |  |
| Sulfur                                      | 0   | 0   | 1 |  |  |  |  |

#### 2.2.4 Cure Systems for Butyl and Halobutyl Rubber

Isobutylene-based elastomers include butyl rubber, the copolymer of isobutylene and isoprene, halogenated butyl rubber, star-branched versions of these polymers and the terpolymer isobutylene-*para*-methylene styrene-bromo-*para*-methyl styrene (BIMS). A number of reviews on isobutylene-based elastomers are available (395, a.20, a.21).



CR crosslinking

Polyisobutylene and butyl rubber have the good chemical resistance expected of saturated hydrocarbons. Oxidative degradation is slow and the material may be further protected by antioxidants, for example hindered phenols.

#### 2.2.4.1 Butyl Rubber

In butyl rubber, the hydrocarbon group positioned alpha to the C-C double bond permits vulcanisation into a crosslinked network with sulfur and organic accelerators (408). The low degree of unsaturation requires the use of ultra accelerators, such as thiuram or dithiocarbamate. Phenolic resins, bisazoformates (a.22), and quinone derivatives can also be employed. Vulcanisation introduces a chemical crosslink every 250-carbon atoms along the polymer chain, producing a molecular network. The number of sulfur atoms per crosslink is between one and four or more (a.23).

Butyl rubber is used in tyre tube applications among others. A typical formulation (mix 01) is shown in **Table 22.** The performance needs for inner tubes are:

- Low air permeability
- Improved heat resistance
- Low tension set

| Table 22 Model formulation of butyl rubber(inner tubes) |     |  |  |  |
|---|-----|--|--|--|
| Ingredients/stock 01                                    |     |  |  |  |
| IIR Polysar 301   | 100 |  |  |  |
| N-660   | 70  |  |  |  |
| Zinc oxide  | 5   |  |  |  |
| Stearic acid  | 1   |  |  |  |
| Paraffinic Oil Sunpar 2280                              | 28  |  |  |  |
| Perkacit MBTS   | 0.5 |  |  |  |
| Perkacit TMTD   | 1.0 |  |  |  |
| Sulfur  | 2.0 |  |  |  |

- Improved heat ageing properties
- Good physical properties and retention of the properties.

One investigation suggested (19) adding 0.75 phr of antireversion chemical, 1,3-bis(citraconimidomethyl) benzene (Perkalink 900) for improving heat resistance (mix 02). It is known that Perkalink 900 provides heat resistance to sulfur cured diene elastomers. The effect of Perkalink 900 on IIR is shown in **Figure 8**. It is clear that Perkalink 900 has a positive effect on the torque retention, which can be translated into better heat resistance.

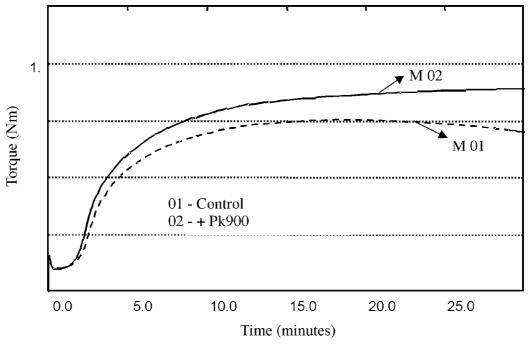


Figure 8

Cure characteristics of the mixes 01 (Control) and 02 (+ Perkalink 900) at 170 °C

The cure data are shown in **Table 23**. The data presented clearly show that Perkalink 900 does not influence the processing characteristics. Although a longer T90 seems to be negative, raising the temperature by 5 or 10  $^{\circ}$ C will neutralise this effect with the additional advantage of higher cure rate and stabilisation of tan delta characteristics.

| Table 23 Cure characteristics of mixes 01and 02 at 170 °C |       |       |  |  |
|---|-------|-------|--|--|
| Properties/stocks 01 02                                   |       |       |  |  |
| M <sub>H</sub> -M <sub>L</sub> (Nm)                       | 0.78  | 0.95  |  |  |
| Scorch safety, ts2 (minutes)                              | 2.6   | 2.3   |  |  |
| Optimum cure time, T90 (minutes)                          | 9.5   | 11.9  |  |  |
| Tan δ (30'-T90)   | 0.112 | 0.056 |  |  |
| M <sub>L</sub> (1+4) @ 100 °C (MU) 55 53                  |       |       |  |  |

**Table 24** presents some data concerning physical properties of the vulcanisates cured at 170°C for T90 and 30 minutes. A careful comparison of the data clearly shows the decline in the properties of the control, whereas a higher retention of vulcanisate properties is observed for the vulcanisate containing Perkalink 900.

**Table 25** presents the relevant data concerning air permeability. It is evident that Perkalink 900 does not have any negative influence on air permeability.

Sulfur crosslinks have limited stability at elevated temperatures and can rearrange to form new crosslinks. This results in poor permanent set and creep for vulcanisates when exposed for long periods of time at high temperatures. Resin cure systems provide C-C crosslinks and heat stability. Alkyl phenol formaldehyde derivatives are usually employed for tyre bladder applications. A typical vulcanisation system is shown in **Table 26** (mix 03). Perkalink 900 has been added at 0.5 phr (mix 04)

| Table 24 Properties of butyl rubber mixes 01 and 02 before and after ageing(cure 170 °C/T90 and 30 minutes, in the parentheses) |             |             |             |             |  |  |  |
|---|-------------|-------------|-------------|-------------|--|--|--|
| Properties         01         01 (aged)         02         02 (aged)  |             |             |             |             |  |  |  |
| Hardness (Shore A)  | 47 (45)     | 50 (48)     | 48 (48)     | 50 (50)     |  |  |  |
| Modulus (MPa) at  |             |             |             |             |  |  |  |
| 50% elongation  | 0.94 (0.87) | 0.82(0.72)  | 0.94 (0.90) | 0.90(0.88)  |  |  |  |
| 100% elongation   | 1.62 (1.5)  | 1.27 (1.01) | 1.60 (1.58) | 1.63 (1.60) |  |  |  |
| 300% elongation   | 4.9 (4.6)   | 4.2 (3.2)   | 5.2 (5.2)   | 4.8 (4.7)   |  |  |  |
| Tensile strength (MPa)  | 11.2 (10.0) | 8.8 (6.2)   | 11.6 (11.0) | 11.1 (9.5)  |  |  |  |
| Elongation at break (%)   | 630 (615)   | 590 (530)   | 640 (625)   | 620 (510)   |  |  |  |
| Tension set (%)   | 7.1         | 12.5        | 5.7         | 6.2         |  |  |  |

| Table 25 Air permeability in ml (STP)/m <sup>2</sup><br>in 24 h at 1 bar (cure: 170 °C/30') |      |      |  |  |  |
|---|------|------|--|--|--|
| Samples12(Control)(+ Perkalink 900)   |      |      |  |  |  |
| A1  | 87   | 74   |  |  |  |
| A2  | 84   | 90   |  |  |  |
| A3 87 91  |      |      |  |  |  |
| Thickness (mm)  | 0.53 | 0.55 |  |  |  |
| Average   | 86   | 85   |  |  |  |

| Table 26 Model formulation of butyl rubber(bladder) application |     |  |  |  |
|---|-----|--|--|--|
| Ingredients/stock 03  |     |  |  |  |
| Butyl 268   | 100 |  |  |  |
| Neoprene W  | 5   |  |  |  |
| N-330   | 50  |  |  |  |
| Castor oil  | 5   |  |  |  |
| Zinc oxide  | 5   |  |  |  |
| Resin SP 1045   | 10  |  |  |  |

| Table 27 Cure characteristics of the butyl rubber mixes obtained at 190 $^\circ 	ext{C}$ |      |      |      |  |  |  |  |
|--|------|------|------|--|--|--|--|
| Properties/stocks030405  |      |      |      |  |  |  |  |
| M <sub>H</sub> -M <sub>L</sub> (Nm)  | 1.1  | 1.0  | 1.2  |  |  |  |  |
| M <sub>L</sub> (Nm)  | 0.25 | 0.24 | 0.24 |  |  |  |  |
| ts2 (minutes)  | 1.7  | 1.7  | 1.7  |  |  |  |  |
| T90 (minutes)  | 27.4 | 26.9 | 26.5 |  |  |  |  |
| Mooney scorch, T5 (minutes) at 150 °C  | 13.7 | 14.1 | 14.3 |  |  |  |  |

| Table 28 Physical properties of the butyl rubber formulations (cure: 190 °C/T90+2') |      |          |      |  |
|---|------|----------|------|--|
| Properties/stocks   | 03   | 04       | 05   |  |
| At room temperature   |      |          |      |  |
| Hardness (Shore A)  | 65   | 65       | 65   |  |
| Modulus at 100% elongation (MPa)  | 1.7  | 1.6      | 1.7  |  |
| Modulus at 300% elongation (MPa)  | 5.2  | 5.0      | 5.5  |  |
| Tensile strength (MPa)  | 13.0 | 13.2     | 13.5 |  |
| Elongation at break (%)   | 670  | 680      | 660  |  |
| 300% Tension set (%)  | 12.5 | 10.0     | 9.1  |  |
| At 190 °C   |      | · I      |      |  |
| Modulus at 100% elongation (MPa)  | 1.7  | 1.6      | 1.8  |  |
| Modulus at 300% elongation (MPa)  | 5.3  | 4.8      | 5.8  |  |
| Tensile strength (MPa)  | 6.1  | 7.0      | 7.7  |  |
| Elongation at break (%)   | 350  | 370      | 380  |  |
| Aged 2 d/177 °C; Pulled at 190 °C   |      | <u> </u> |      |  |
| Modulus at 100% elongation (MPa)  | 3.1  | 3.2      | 3.4  |  |
| Tensile strength (MPa)  | 4.2  | 5.0      | 6.2  |  |
| Elongation at break (%)   | 210  | 220      | 230  |  |
| 200% Tension set (%)  | 28   | 20       | 18   |  |

and 0.75 phr (mix 05). The comparative properties of these formulated rubbers are summarised in **Tables 27 and 28**.

# 2.2.4.2 Halobutyl Rubber

Halobutyl rubber (XIIR) vulcanisates are generally acoustic loss materials having essentially the same physical and dynamic mechanical properties as regular butyl rubber. They have the advantage of rapid rate of cure with reduced curative levels, cure compatibility with other polymers and good cure adhesion to themselves and other elastomers (394, 415, a.24-a.26). This combination of properties makes them potentially superior base elastomers for the manufacture of a variety of mechanical goods ranging from heavy duty shock absorbers to smaller sound and vibration damping mountings, hoses, belting and many other tyre and non-tyre applications. One of the primary applications for XIIR is in the inner liners for tyres. The type and level of a suitable chemical crosslinking system for compounding must be selected very carefully to achieve the desired combination of properties and service life. Several crosslinking systems are available, one of them is crosslinking via bis-maleimides (HVA-2) (400). Unfortunately crosslinking with HVA-2 does not generate sufficient advantage with respect to heat generation and set characteristics. The use of antireversion agent, 1,3 bis(citraconimidomethyl) benzene (Perkalink 900) is being explored in this application to bring the desired properties. The model formulations are shown in **Table 29**. The cure data and physicomechanical properties of these vulcanisates are tabulated in **Table 30**. The data clearly show that Perkalink 900 acts as an efficient crosslinker in XIIR. This can be explained via the reaction mechanism as shown in **Figure 9**.

| Table 29 Model formulations for halobutyl rubber (inner liners) |             |     |     |     |  |  |
|---|-------------|-----|-----|-----|--|--|
| Ingredients/stocks  | 06 07 08 09 |     |     |     |  |  |
| CIIR Polysar 1240   | 100         | 100 | 100 | 100 |  |  |
| N-550   | 55          | 55  | 55  | 65  |  |  |
| Zinc oxide  | 5           | 5   | 5   | 5   |  |  |
| Stearic acid  | 1           | 1   | 1   | 1   |  |  |
| Paraffinic oil  | 10          | 10  | 10  | 10  |  |  |
| HVA-2   | 0           | 3   | 0   | 0   |  |  |
| Perkalink 900   | 0           | 0   | 2   | 2   |  |  |

| Table 30 Properties of the vulcanisates (cure: 160 °C/T90) |      |      |      |      |  |
|--|------|------|------|------|--|
| Properties/stocks  | 06   | 07   | 08   | 09   |  |
| Cure data at 160 °C  |      | 1    |      | 1    |  |
| M <sub>H</sub> M <sub>L</sub> (Nm)                         | 0.5  | 1.5  | 1.1  | 1.5  |  |
| ts2 (minutes)  | 4.2  | 2.2  | 4.0  | 2.6  |  |
| T90 (minutes)  | 9    | 16   | 26   | 18   |  |
| Cure: 160 °C/T90   |      |      |      |      |  |
| Hardness (IRHD)  | 50   | 69   | 62   | 71   |  |
| Modulus at 100% elongation (MPa)                           | 1.5  | 4.5  | 2.8  | 5.3  |  |
| Modulus at 300% elongation (MPa)                           | 3.9  | 11.5 | 9.4  | 11.5 |  |
| Tensile strength (MPa)                                     | 12.2 | 12.1 | 14.1 | 13.5 |  |
| Elongation at break (%)                                    | 500  | 210  | 310  | 260  |  |
| Tear strength (kN/m)                                       | 34   | 20   | 24   | 28   |  |
| Compression set (%)  |      |      |      | 1    |  |
| 72 h/room temperature                                      | nm   | 4.2  | nm   | 4.5  |  |
| 24 h/100 °C  | nm   | 11   | nm   | 6    |  |
| Heat build up at 100 °C                                    |      |      | 1    |      |  |
| ΔT in 25'  | 60   | 30   | 18   | 20   |  |
| *nm = not measured   |      | 1    | 1    | 1    |  |

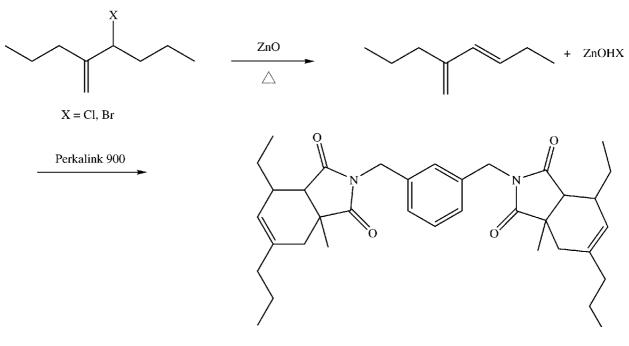


Figure 9

Reaction of Perkalink 900 with XIIR in the presence of zinc oxide

It can be concluded from this study that Perkalink 900 can be used as a crosslinker in XIIR and could provide additional advantages such as better high temperature compression set and lower heat built up in the Goodrich Flexometer test over HVA-2.

#### 2.3 Peroxide Cure Systems

Crosslinking with peroxides has been known since 1915 when Ostromyslenski disclosed that natural rubber could be transformed into a crosslinked state with dibenzoyl peroxide (a.27). However, little interest in peroxide crosslinking evolved until the development of fully saturated ethylene-propylene copolymers in the early 1970s.

The use of peroxides for the crosslinking of elastomers is limited to those that are stable during storage, safe to handle during processing but, on the other hand, decompose sufficiently fast at cure temperatures. In order to meet these requirements peroxides containing tertiary carbon atoms are most suitable, whilst peroxy groups bonded to primary and secondary carbon atoms are less stable. Organic peroxides that are suitable for crosslinking elastomers are shown in **Figure 10**. Peroxides containing more than one peroxy group are also suitable (**Figure 11**).

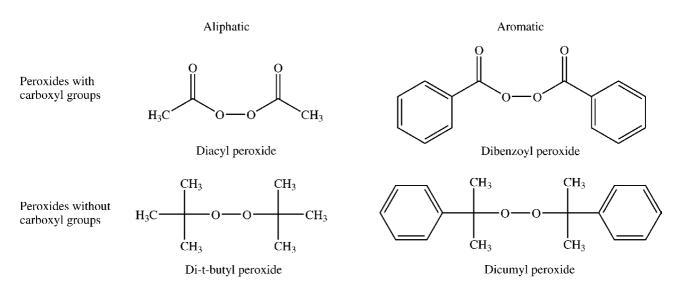
In addition to the symmetrical peroxides, asymmetrical peroxides are also in use, for example *tert*-butyl perbenzoate, *tert*-butylcumyl peroxide and some polymeric peroxides (403).

A further limitation with regard to the suitability of peroxides concerns the efficiency of crosslinking. Higher efficiencies are observed for those peroxides that form one of the radicals shown in **Figure 12** during homolytic decomposition (a.28).

The thermal stability of peroxides can be expressed in terms of their half-life ( $t_{1/2}$ ). Half-life values can be estimated in solution utilising the technique of differential thermal analysis. These values, or more precisely the temperatures at which their half-life is equivalent to 6 minutes, provide an indication of practical vulcanisation temperatures (see **Table 31**) (381).

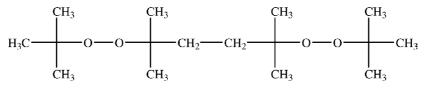
There are a number of advantages, listed below, associated with the peroxide vulcanisation of elastomers:

- scorch free storage of compounds,
- possibility to apply high vulcanisation temperatures without reversion,
- simple compound formulation,
- low compression set even at high cure temperatures,
- good electrical properties of vulcanisates,
- good high temperature vulcanisate stability,
- no discoloration of compounds.

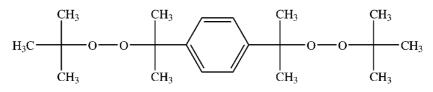


# Figure 10

Organic peroxides for crosslinking elastomers



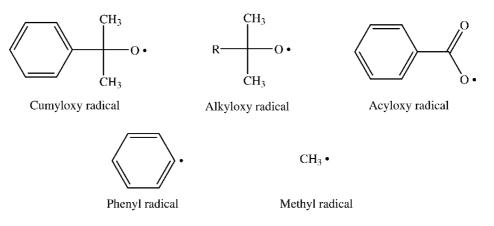
2,5-Bis(tert-butylperoxy)-2,5 dimethyl hexane



1,4-Bis(tert-butylperoxy isopropyl) benzene

#### Figure 11

Peroxides containing more than one peroxy group





Radicals formed during the homolytic cleavage of peroxides that are most effective in producing crosslinks

| Table 31 Typical crosslinking temperatures of crosslinking peroxides based on their half-life |  |   |  |  |
|---|--|---|--|--|
| Class   | Example  | Temperature<br>(°C)<br>t <sub>1/2</sub> = 6 min | Typical<br>crosslinking<br>temperature<br>(°C) |  |
| Dialkyl peroxide  | 2,5-Bis(tert-butylperoxy) 2,5-dimethyl-3-hexyne        | 173   | 190  |  |
|   | 2,5-Bis(tert-butylperoxy) 2,5-dimethyl hexane          | 159   | 180  |  |
| Alkyl-aralkyl peroxide  | Bis(tert-butylperoxy isopropyl) benzene                | 160   | 180  |  |
|   | Tert-butylcumyl peroxide                               | 160   | 180  |  |
| Diaralkyl peroxide  | Dicumyl peroxide                                       | 155   | 170  |  |
| Peroxy ketals   | Butyl 4,4-bis (tert-butyl peroxy) valerate             | 143   | 160  |  |
|   | 1,1-Bis(tert-butyl peroxy) 3,3,5-trimethyl cyclohexane | 129   | 150  |  |
| Peroxy ester  | Tert-Butylperoxy benzoate                              | 146   | 140  |  |

There are, however, some drawbacks compared to sulfur vulcanisation:

- limited compounding flexibility due to the reaction of peroxides with other compounding ingredients; for example with antioxidants, plasticisers and resins,
- sensitivity of vulcanisation reactions to oxygen,
- lack of flexibility in regulating scorch and optimum cure time,
- inferior tensile, tear and flex properties,
- inferior abrasion resistance,
- odours of peroxide decomposition products,
- generally higher cost.

A large variety of polymers can be crosslinked by peroxides but the reaction rates and mechanism of different polymers with peroxides vary considerably. Some polymers are readily crosslinked by peroxides while others suffer degradation (6). Polymers that can be effectively crosslinked by peroxides include:

Natural rubber Styrene butadiene rubber Polybutadiene Polyisoprene Nitrile rubber Halogenated nitrile rubber Ethylene propylene rubber

#### EPDM

Ethylene-vinyl acetate Acrylonitrile butadiene styrene Silicones Fluorocarbon elastomers Acrylic elastomers Polyurethanes Polyurethanes Polyethylene Chlorinated polyethylene Chlorosulfonated polyethylene Poly(vinyl chloride)

Polymers that cannot be effectively crosslinked with peroxides are:

Polyisobutylene rubber Butyl rubber Halobutyl rubber Polyepichlorohydrin Polypropylene Polypropylene oxide

Of the polymers that can be crosslinked, the crosslink efficiency varies considerably. In general the relative efficiency of peroxide vulcanisation of polymers (a.29, a.30) is:

BR>NR and SBR>NBR>CR>EPDM

Peroxide crosslinking of the more highly unsaturated polymers is more efficient due to the higher concentration of allylic hydrogens. These are readily abstracted and efficiently converted to crosslink.

| 2.3.1 Peroxide Vulcanisation of EPDM  | Allylic type:      | triallyl cyanurate (TAC)<br>triallyl isocyanurate (TAIC)  |
|---|--------------------|---|
| Peroxide vulcanisation of EPDM is growing in<br>popularity because of enhanced ageing resistance. A<br>comparison of sulfur and peroxide cure systems for | Methacrylate type: | zinc dimethacrylate   |
| EPDM is shown in <b>Table 32</b> (381).   | Acrylate type:     | ethylene glycol diacrylate<br>zinc diacrylate   |
| Apart from peroxide type and the amount of peroxide<br>incorporated in compounds, the efficiency of<br>crosslinking depends on coagents. The commercially | Polymeric coagents | liquid 1,2-polybutadiene resin  |
| <ul><li>important ones are:</li><li>Maleimide type: <i>N,N´</i>-phenyl maleimide</li></ul>  | and chemical resi  | weathering resistance, good heat<br>stance, good low temperature<br>tanding electric properties, make |

| Ingredients                      | Sulfur cure | Di-Cup cure | Vul-Cup cure |
|----------------------------------|-------------|-------------|--------------|
| Nordel 1040                      | 100         | 100         | 100          |
| HAF black                        | 50          | 50          | 50           |
| Zinc oxide                       | 5           | 5           | 5            |
| Stearic acid                     | 1           | 0           | 0            |
| Sulfur                           | 1.5         | 0           | 0            |
| ТМТМ                             | 1.5         | 0           | 0            |
| MBT                              | 0.5         | 0           | 0            |
| Di-Cup 40KE*                     | 0           | 6.6         | 0            |
| Vul-Cup 40KE*                    | 0           | 0           | 4.1          |
| AgeRite Resin D                  | 0.5         | 0.5         | 0.5          |
| Cure temperature (°C)            | 160         | 171         | 177          |
| Cure time (minutes)              | 20          | 20          | 20           |
| Physical properties              |             | 1           | 1            |
| Modulus at 100% elongation (MPa) | 2.2         | 1.8         | 1.9          |
| Modulus at 200% elongation (MPa) | 6.1         | 4.9         | 5.3          |
| Tensile strength (MPa)           | 17.9        | 17.2        | 16.7         |
| Elongation at break (%)          | 400         | 375         | 375          |
| Hardness (Shore A)               | 68          | 62          | 60           |
| Aged properties (70 h/150 °C)    |             |             |              |
| Modulus at 100% elongation (MPa) | 5.4         | 1.7         | 2.1          |
| Modulus at 200% elongation (MPa) | 12.8        | 4.7         | 5.2          |
| Tensile strength (MPa)           | 15.0        | 17.4        | 16.7         |
| Elongation at break (%)          | 220         | 400         | 350          |
| Hardness (Shore A)               | 78          | 58          | 60           |
| Compression set (%)              |             | •           | •            |
| (70 h/150 °C)                    | 77          | 21          | 19           |

EPDM rubber preferred for a great number of specific applications. For many years peroxide-cured EPDM based compounds have been applied, e.g., for window seals, automotive hoses, steam hoses, conveyer belts, roof sheeting, tank lining, roll coverings, mouldings, and last but not least, for electrical insulation and jacketing compounds. Two formulation types (403) are illustrated in **Tables 33 and 34**.

| Table 33 Pilot recipe for automotive radiatorhose (steam cure: 180 °C/20')   |      |  |
|--|------|--|
| Properties   |      |  |
| Hardness (Shore A)   | 60   |  |
| Tensile strength (MPa)   | 13.3 |  |
| Modulus at 100% elongation (MPa)   | 3.0  |  |
| Elongation at break (%)  | 340  |  |
| Change in elongation at break after ageing (%)   |      |  |
| 168 h/140 °C   | -4   |  |
| 168 h/150 °C   | -12  |  |
| 168 h/160 °C   | -21  |  |
| Extractables, 168 h/100 °C   |      |  |
| water glycol (g/100 g rubber)  | 0.05 |  |
| Composition: Vistalon 7500, 100; N-550 100; Flexon<br>815, 45; TMQ 1; zinc salt of 4- and 5-methyl-2-<br>mercapto-benzimidazole (ZMMBI) 3; coagent ethylene<br>dimethacrylate (EDMA) 0.5, Perkadox 14-40, 7. |      |  |

| Table 34 Pilot recipes for injection<br>moulded article   |      |     |
|---|------|-----|
| Properties  | 01   | 02  |
| Butylated hydroxy toluene (BHT) (phr)   | 0    | 0.4 |
| Scorch time, T2 (minutes)   |      |     |
| (120 °C)  | 5.8  | 9.1 |
| (150 °C)  | 2.1  | 2.5 |
| Cure time, T90 (minutes) (150 °C)   | 8.4  | 9.6 |
| Torque increase (Nm) (150 °C)   | 4.4  | 4.0 |
| Mould temperature (°C)  | 180  | 200 |
| Cure time (minutes)   | 3.0  | 0.5 |
| Hardness (Shore A)  | 67   | 65  |
| Tensile strength (MPa)  | 12.1 | 9.0 |
| Elongation at break (%)   | 250  | 230 |
| Compression set, 24 h/100 °C (%)  | 26   | 22  |
| Compositions: Keltan 578, 100, N-770, 70; N-550, 70;<br>Sunpar 150, 70; stearic acid, 0.5; Perkalink 400, 1;<br>BHT, as indicated; Trigonox 2940, 11.4. |      |     |

# 2.4 Sulfur Free Curing Systems

Some special vulcanising agents can cure diene rubbers such as NR, SBR and BR. These are now discussed.

# 2.4.1 Phenolic Curatives, Benzoquinone Derivatives and Bismaleimides

Diene rubbers can be vulcanised by the action of phenolic compounds like phenol-formaldehyde resin (5-10 phr). Resin cured NR offers good set properties and low hysteresis (a.31).

Resin curing of SBR and BR imparts excellent cut growth and abrasion resistance. Resin cured nitrile rubber shows high fatigue life and high relaxation, while resin-cured butyl rubber shows outstanding ozone and age resistance (409).

A high diene rubber can also be vulcanised by the action of a dinitrosobenzene, made *in situ* by the oxidation of a quinonedioxime (**Figure 13**) (402, a.32-a.35) incorporated into the rubber together with the vulcanising agent lead peroxide.

Another vulcanising agent for diene rubbers is *m*-phenylenebismaleimide. A catalytic free-radical source such as dicumyl peroxide or benzothiazyldisulfide (MBTS) is commonly used to initiate the reaction (a.36). Phenolic curatives, benzoquinonedioxime, and *m*-phenylenebismaleimide are particularly useful where thermal stability is required.

# 2.4.2 Vulcanisation by Triazine Accelerators

Logothetis (399) describes the use of triazine accelerators in the vulcanisation of nitrile and fluoro elastomers. The triazine accelerators are more effective than the thiazole accelerators and produce highly reversion resistant vulcanisates.

# 2.4.3 Urethane Crosslinkers

Natural rubber can be crosslinked by a blocked diphenyl methane diisocyanate to produce urethane crosslinks. The crosslinking agent dissociates into two quinonedioxime molecules and one diphenyl methane diisocyanate. The quinone reacts with the rubber via a nitroso group and forms crosslinks via a diisocyanate group. The performance of this system in NR is characterised by excellent age resistance and outstanding reversion resistance.

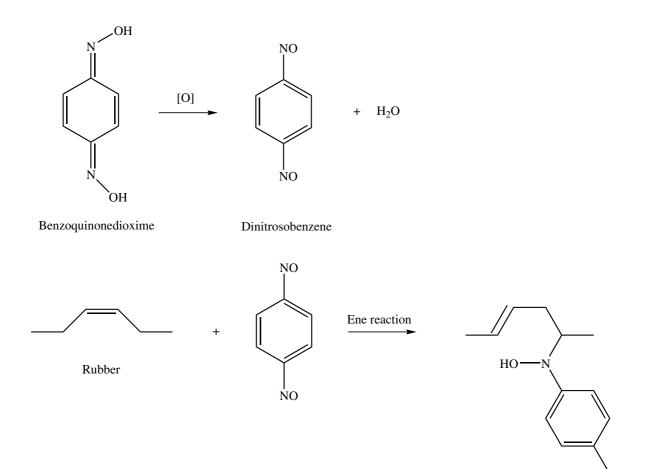


Figure 13 Vulcanisation by benzoquinonedioxime

Further variation of the structure of nitroso diisocyanate and compounds yielded NOVOR 924. A NR vulcanisate containing NOVOR 924 is more reversion resistant than any EV system (Baker in 398).

#### 2.4.4 Other Crosslinking Agents (a.37)

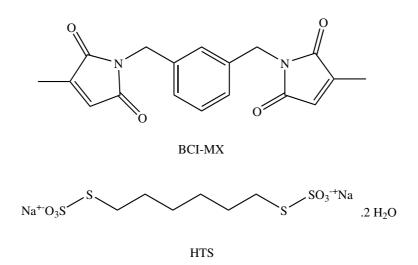
There exist a considerable number of compounds containing labile chlorine which bring about sulfurless vulcanisation at levels of approximately 3 phr. Basic chemicals such as lead oxides and amines are needed. It may be assumed that diene rubbers are crosslinked by such systems through the formation of C-C links; this would mean, initially, hydrogen chloride is split off and later neutralised by the base. Examples of chemicals that act in this manner are:

2,4-dichlorobenzotrichloride (a.38) hexachloro-*p*-xylene (414) chloranil (416) bis-chloromethylxylene (410) benzene disulfochloride (407) hexachlorocyclopentadiene (a.39) trichloromethane sulfochloride (a.39) trichloromelamene (a.39)

Apart from the list above some N-bearing molecules are described in the literature (a.37).

#### **2.5 New Developments**

Maintaining properties and performance throughout a rubber product's service life is directly related to maintaining the integrity of the vulcanisate structure under both thermal and thermal oxidative conditions. Historically, this has been achieved by reducing the sulfur content in the crosslinks by using efficient or semi-efficient vulcanisation (SEV) systems. However, as with many changes in rubber compounding, there is a trade-off, which, in this case, is a reduction in performance in dynamic fatigue and tear resistance. Two additives have allowed compounders to forget this compromise, namely hexamethylene-1,6-bisthiosulfate (HTS) (260, a.40, a.41), a post vulcanisation stabiliser and 1,3-



**Figure 14** Structures of HTS and BCI-MX

bis(citraconimidomethyl) benzene (BCI-MX, Perkalink 900) (26, 157, 181, 210, 260, 263, 284, 289, 309, 392, a.42-a.44), an antireversion agent. The structures of HTS and BCI-MX are shown in **Figure 14**.

# 3 Some Practical Examples with Varying Cure Systems

Good compounding means formulas are developed that are environmentally safe, factory processable, provide a satisfactory service life, and are cost competitive to other compounds used in the same applications. Costs are always a major concern and constantly increasing environmental safety regulations most not be overlooked. In this section, the focus is on cure systems and formulations of practical interest. This provides a guideline for compounding in various applications.

# 3.1 Tyres

# 3.1.1 Tread

The tread is probably the most critical component of the tyre determining the final performance. It is also the thickest component of the tyre and it contributes most of the energy losses that in turn will cause a rise in the tyre's running temperature and an increase in fuel consumption for the vehicle. Tread is also responsible for the safety component of the tyre and its surface is designed to provide good grip in all conditions of dry, wet, ice or snow but with minimum noise generation. Trying to balance the three main apparently conflicting needs of wear, wet grip and rolling resistance, together with many other performance requirements leads to a wide range of tread formulations covering several natural and synthetic rubbers combined with different ratios of alternative filler types. Compounds need good flexibility, thermal resistance and abrasion resistance. The cure system should be CV/SEV. Accelerators used are sulfenamide class (TBBS, CBS, MBS).

A typical formulation for a radial type truck tread is shown in **Table 35**. For improving heat resistance, the use of Perkalink 900 is recommended (289). The recommended loading for this cure system is 0.5 phr.

| Table 35 Radial truck tread formulation |     |
|---|-----|
| Ingredients                             | phr |
| NR                                      | 100 |
| N-234                                   | 55  |
| Zinc oxide                              | 5   |
| Stearic acid                            | 2   |
| Aromatic oil                            | 8   |
| 6PPD                                    | 2   |
| TMQ                                     | 1.5 |
| TBBS                                    | 1.5 |
| PVI                                     | 0.1 |
| Sulfur                                  | 1.5 |

A typical formulation for a bias tyre truck tread is shown in **Table 36**.

A typical formulation for a passenger tyre tread is shown in **Table 37**. To raise the curing temperature, the use of antireversion chemical, Perkalink 900 has been recommended (119).

Silica as a reinforcing filler is being used more extensively in the tyre industry to provide improved tear resistance and decreased rolling resistance. In

| Table 36 Bias truck tread (Lug) formulation |     |  |
|---|-----|--|
| Ingredients                                 | phr |  |
| NR, RSS                                     | 100 |  |
| Renocit 11                                  | 0.1 |  |
| N-330                                       | 50  |  |
| Aromatic oil                                | 8   |  |
| Zinc oxide                                  | 5   |  |
| Stearic acid                                | 2   |  |
| TMQ   | 1   |  |
| 6PPD  | 2   |  |
| MBS   | 0.6 |  |
| PVI   | 0.2 |  |
| Sulfur                                      | 2.3 |  |
| Perkalink 900                               | 0.5 |  |

| Table 37 Conventional passenger treadformulation |      |  |
|--|------|--|
| Ingredients                                      | phr  |  |
| SBR 1712   | 82.5 |  |
| BR CB 29   | 55   |  |
| N-220  | 70   |  |
| Zinc oxide                                       | 3    |  |
| Stearic acid                                     | 2    |  |
| 6PPD   | 1    |  |
| TMQ  | 2    |  |
| Microcrystalline wax                             | 3    |  |
| TBBS   | 1    |  |
| Sulfur   | 2    |  |

order to maximise these benefits the silane coupling agent bis-(3-triethoxysilylpropyl) tetrasulfide (TESPT) is often employed, functioning by chemically bonding the filler particles to the elastomer network. In addition, this coupling agent may act as a delayed action sulfur donor, providing some degree of reversion resistance via the formation of sulfidic crosslinks of low sulfur rank. A passenger tread containing silica needs a cure system based on sulfenamide/sulfur (SEV/CV system) and guanidine activators (DPG).

With the advent of 'Green tyres' the use of silica has significantly increased. A typical 'Green tyre' formulation for passenger tread is shown in **Table 38**. In order to achieve high temperature cure, the use of Perkalink 900 has been reported (26).

Off-the-road (OTR) tread compounds suffer from the worst chipping and chunking. In order to improve resistance, the use of silica together with black is recommended. A typical formulation is shown in **Table 39**. The off-the-road (OTR) tread cure system is an SEV system based on sulfenamide accelerator. Silica delays the cure and hence a cure activator (such as DPG) is required to accelerate the curing process.

In order to improve reversion resistance, the use of Perkalink 900 has been recommended as documented by Datta and co-workers (26).

| Table 38 Green tyre formulation – passenger tread |     |  |
|---|-----|--|
| Ingredients                                       | phr |  |
| SBR Cariflex S1215                                | 75  |  |
| BR Buna CB 10                                     | 25  |  |
| Silica, Perkacil KS 408                           | 80  |  |
| Zinc oxide  | 3   |  |
| Stearic acid                                      | 2   |  |
| Aromatic oil                                      | 34  |  |
| Coupling agent, TESPT                             | 6.4 |  |
| Santoflex 6PPD                                    | 2   |  |
| Wax PEG 4000                                      | 3   |  |
| Perkacit TBBS                                     | 1   |  |
| Perkacit DPG                                      | 2   |  |
| Sulfur  | 2   |  |

| Table 39 Model OTR tyre tread formulation |     |  |
|---|-----|--|
| Ingredients                               | phr |  |
| NR SMR 10                                 | 100 |  |
| Carbon black N-220                        | 40  |  |
| Silica, Perkasil KS 408                   | 20  |  |
| Zinc oxide                                | 5   |  |
| Stearic acid                              | 2   |  |
| Aromatic oil                              | 3   |  |
| Resin cumarone                            | 3   |  |
| Coupling agent, TESPT                     | 3.0 |  |
| TMQ                                       | 1.5 |  |
| 6PPD                                      | 2.5 |  |
| Wax Sunolite 240                          | 1.0 |  |
| CBS                                       | 1.4 |  |
| DPG                                       | 1   |  |
| Sulfur                                    | 1.4 |  |

Aircraft tread requires a high modulus, better abrasion resistance and low heat generation. The above requirements could be satisfied by using a CV/SEV cure system with DCBS as accelerator and insoluble sulfur (IS) as crosslinker. For achieving high modulus, HMT and a resorcinol system is used. In order to reduce the heat generation, the use of Perkalink 900 has been suggested. A typical formulation for an aircraft tyre tread recipe is shown in **Table 40**.

| Table 40 Aircraft tyre tread formulation      |     |  |
|---|-----|--|
| Ingredients                                   | phr |  |
| NR  | 100 |  |
| N-330   | 50  |  |
| Zinc oxide                                    | 5   |  |
| Stearic acid                                  | 2   |  |
| 6PPD  | 1.1 |  |
| TMQ   | 1.0 |  |
| Resorcinol                                    | 1.5 |  |
| MC Wax  | 1.0 |  |
| Aromatic oil                                  | 3.0 |  |
| Hexamethylene tetramine<br>(HMT) Cohedur H 30 | 0.8 |  |
| DCBS  | 1.1 |  |
| PVI   | 0.2 |  |
| IS OT 20                                      | 2.2 |  |

# 3.1.2 Tread Base or Sub Tread

A natural rubber rich undertread layer can enhance the adhesion between belt or cap-ply and tread whilst a thicker subtread compound may be included to offer some additional benefits of low hysteresis for car tyres and low heat generation for truck tyres within the bulk of a thick section. The cure system needs better flexibility and low heat generation. Typically the cure system will be based on CV/SEV. A general composition for a tyre tread base is depicted in **Table 41**.

| Table 41 Tyre tread base formulation |     |  |
|--------------------------------------|-----|--|
| Ingredients                          | phr |  |
| NR                                   | 100 |  |
| N-550                                | 45  |  |
| Zinc oxide                           | 5   |  |
| Stearic acid                         | 2   |  |
| Aromatic oil                         | 10  |  |
| 6PPD                                 | 2.5 |  |
| Microcrystalline wax                 | 2   |  |
| CBS                                  | 0.9 |  |
| PVI                                  | 0.1 |  |
| Sulfur                               | 1.5 |  |
| Perkalink 900                        | 0.5 |  |

# 3.1.3 Belts

In radial tyres, sets of belts or breakers made from brass coated steel cords are layed at alternate bias angles to provide a trellising effect, to stiffen the area under the tread and also to prevent growth under inflation or high speed rotation. These belts provide a rigid support to the tread offering a more controlled contact with the road. They therefore play an important role in the wear of tyres and the handling and stability of vehicles. The design of the belt package should give a smooth ride with minimum energy loss, but with sufficient stiffness to prevent undue movement within the contact area causing irregular or rapid tyre wear.

The cure package is composed of a slow accelerator (DCBS, or TBSI) with a high level of sulfur for improving the bond strength between the rubber and brass layer. A typical formulation is shown in **Table 42**.

| Table 42 Typical formulation for type belts |      |  |
|---|------|--|
| Ingredients                                 | phr  |  |
| NR  | 100  |  |
| N-326                                       | 45   |  |
| Silica                                      | 15   |  |
| Zinc oxide                                  | 8    |  |
| Stearic acid                                | 1.2  |  |
| Tackifier SP 1068                           | 2    |  |
| Cobalt salt (NAPCO 105)                     | 0.75 |  |
| Resorcinol                                  | 2    |  |
| Hexamethoxymethylmelamine (HMMM)            | 3    |  |
| TMQ   | 1    |  |
| 6PPD  | 1    |  |
| DCBS  | 1    |  |
| PVI   | 0.1  |  |
| OT 20                                       | 5    |  |
| Duralink HTS                                | 1.5  |  |

Addition of Duralink HTS (hexamethylene-1,6bis(thiosulfate), disodium salt dihydrate) improves adhesion characteristics under ageing environments (157). Formulations without H/R bonding systems are also available (157).

# 3.1.4 Sidewall

The sidewall rubber provides protection for the body plies and in the case of heavy-duty truck tyres may be of significant gauge in areas where abrasion or damage from kerb strikes is a likely occurrence. Sidewall compounds cover the thinnest part of the tyre where most flexing occurs as the tyre deflects. They therefore need to have a high degree of flex resistance and good dynamic properties, as well as excellent age resistance. Sidewall compounds contain additional ingredients to prevent oxidative or ozone attack since this part of the tyre will be particularly exposed to the sun and the elements. The cure system should be CV or between CV/SEV. A typical formulation is shown in **Table 43**.

# 3.1.5 Carcass

The carcass or body ply of the tyre is made up of fabric yarns, typically of steel, nylon, rayon or

| Table 43 Sidewall    |     |  |
|----------------------|-----|--|
| Ingredients          | phr |  |
| NR                   | 50  |  |
| BR                   | 50  |  |
| N-550                | 50  |  |
| Naphthenic oil       | 6   |  |
| Zinc oxide           | 4   |  |
| Stearic acid         | 1   |  |
| TMQ                  | 1.5 |  |
| 6PPD                 | 2.5 |  |
| Microcrystalline wax | 2.0 |  |
| CBS                  | 1.0 |  |
| Sulfur               | 1.2 |  |

polyester, twisted into parallel weftless cord layers known as plies. These plies are loaded with natural rubber based compound loaded with adhesion promoters to generate a bond between the cord surface and other tyre components.

The cure system of choice for a carcass compound is based on a scorch resistant cure package. Care must also be taken that the necessary high loadings of sulfur or other additives do not bloom to the surface of the calendered sheet since good tack levels must be maintained throughout the building stage. For this reason, thermally stable, insoluble sulfur and bloom resistant adhesion additives are required. Because of the importance of adhesion to brass plated steel cord, the cure system suitable for this application is based on a slow accelerator such as DCBS with a high amount of insoluble sulfur (generally 4-5 phr). A typical formulation for a truck radial carcass compound is shown in **Table 44**.

A passenger tyre carcass formulation is shown in **Table 45**. In this application, adhesion to fibres is a consideration. The cure system suitable for this application is based on TBBS/MBTS/IS.

# 3.1.6 Bead

The bead is usually a wound hoop of high strength monofilament steel wire coated with rubber, providing the tyre with a secure fitment to the wheel rim such that it does not move or dislodge as the vehicle undergoes severe manoeuvres. The bead and apex compounds

| Table 44 Truck radial carcass formulation |      |  |
|---|------|--|
| Ingredients                               | phr  |  |
| NR SMR L                                  | 100  |  |
| N-339                                     | 55   |  |
| Aromatic oil                              | 3.0  |  |
| Zinc oxide                                | 8.0  |  |
| Stearic acid                              | 0.5  |  |
| TMQ                                       | 1.0  |  |
| 6PPD                                      | 2.0  |  |
| Wingstay L                                | 1.0  |  |
| NAPCO105 (10%Co)                          | 0.63 |  |
| D-HTS                                     | 1.5  |  |
| DCBS                                      | 1.1  |  |
| Insoluble sulfur OT 20                    | 5.0  |  |

| Table 46 Tyre bead formulation |     |  |
|--------------------------------|-----|--|
| Ingredients                    | phr |  |
| NR                             | 100 |  |
| N-330                          | 65  |  |
| Zinc oxide                     | 10  |  |
| Stearic acid                   | 2   |  |
| Aromatic oil                   | 8   |  |
| TMQ                            | 1   |  |
| Resorcinol                     | 3.5 |  |
| HMMM (0.65%)                   | 3   |  |
| PVI                            | 0.1 |  |
| CBS                            | 1.0 |  |
| IS OT 20                       | 6.5 |  |

| Table 45 Passenger tyre carcass formulation |      |  |
|---|------|--|
| Ingredients                                 | phr  |  |
| NR  | 60   |  |
| BR  | 20   |  |
| SBR   | 27.5 |  |
| N-660                                       | 40   |  |
| Zinc oxide                                  | 3    |  |
| Stearic acid                                | 2.0  |  |
| Processing oil                              | 13   |  |
| TMQ   | 0.5  |  |
| Octylated diphenylamine                     | 1    |  |
| Resin                                       | 1.0  |  |
| MBTS  | 0.1  |  |
| TBBS  | 0.7  |  |
| IS OT 20                                    | 2.8  |  |
| PVI   | 0.1  |  |

require high modulus and good adhesion to steel wires. High modulus is achieved by using resorcinol/HMMM or phenol formaldehyde resins. The cure system is based on sulfenamide with a high amount of insoluble sulfur. A typical cure package is shown in **Table 46**.

# 3.1.7 Apex

The apex or filler insert components provide the gradual shape and stiffness reduction from the rigid bead coil to the flexible mid-sidewall of the tyre. These components need to be very hard to provide good vehicle handling and to reduce the risk of flexural fatigue at component endings. High loadings of filler or reinforcing resins make such components difficult to process and notoriously dry, lacking in tack. Tackifying additives and process aids may therefore be required to help in the tyre manufacturing process. The cure system is shown in the formulation tabulated in **Table 47**. TBzTD is used to obtain cure activation.

# 3.1.8 Cap-Ply

Cap-plies are more commonly used on high performance car tyres. Having a circumferential cord direction they provide an additional contractive force. They are also found more often now in normal performance tyres where they can act as a barrier layer between the tread and the casing to restrict migration of chemicals from the tread into the belt. Cap-ply requires high modulus and good adhesion behaviour. The cure system used to achieve the target properties is based on a combination of CBS and DCBS with a high amount of insoluble sulfur. A resorcinol/HMMM system is used to obtain the required high modulus. A typical formulation of a cap-ply (passenger) compound is shown in **Table 48**.

| Table 47 Tyre apex formulation |     |  |
|--------------------------------|-----|--|
| Ingredients                    | phr |  |
| NR                             | 100 |  |
| N-351                          | 55  |  |
| Zinc oxide                     | 10  |  |
| Stearic acid                   | 2   |  |
| SP 6700 Resin                  | 2   |  |
| Phenol formaldehyde resin      | 10  |  |
| Bonding agent                  | 2   |  |
| 6PPD                           | 2   |  |
| TMQ                            | 1   |  |
| TBBS                           | 0.6 |  |
| TBzTD                          | 0.2 |  |
| PVI                            | 0.2 |  |
| IS OT 20                       | 5.0 |  |

| Table 48 Passenger tyre cap-ply formulation           |      |  |
|---|------|--|
| Ingredients   | phr  |  |
| NR  | 80   |  |
| BR  | 20   |  |
| Mineral oil   | 7    |  |
| N-326   | 55   |  |
| Silica  | 7.5  |  |
| Mixture of N,N'-diaryl-p-<br>phenylenediamines (DTPD) | 1    |  |
| TMQ   | 1    |  |
| Resorcinol  | 1.25 |  |
| НМММ  | 1.5  |  |
| CBS   | 0.4  |  |
| DCBS  | 1.0  |  |
| ZnO   | 8    |  |
| Zinc stearate   | 1.0  |  |
| IS OT 20  | 4.5  |  |

# 3.1.9 Inner Liner

The inner liner forms the vital internal membrane which hold the inflation medium at an elevated pressure within the structure of the tyre. In early days the liner was a separate tube of natural or butyl or more particularly, halobutyl rubber compound as an integral part of the tyre structure. Adhesion levels of butyl compounds can be critically low requiring an insulating or barrier layer of a natural rubber compound to act as an interface between the liner and the casing. Zinc oxide with MBTS and a small amount of sulfur is used to crosslink halobutyl rubber. A typical formulation of an inner liner is documented in **Table 49**.

| Table 49 Inner liner formulation |      |  |
|----------------------------------|------|--|
| Ingredients phr                  |      |  |
| Chlorobutyl 1065                 | 100  |  |
| N-660                            | 60   |  |
| Naphthenic oil                   | 8    |  |
| Stearic acid                     | 2    |  |
| Phenolic resin tackifier         | 4    |  |
| Homogenisers                     | 7    |  |
| Magnesium oxide                  | 0.15 |  |
| Zinc oxide                       | 3    |  |
| Sulfur                           | 0.5  |  |
| MBTS                             | 1.5  |  |

# **3.2 Industrial Rubber Products**

This section provides information on the property requirements and the best cure systems in compounds typical of some industrial rubber products (IRP). Examples of industrial rubber products and typical polymers used are shown in **Table 50**. In a recent study the effect of an antireversion agent (Perkalink 900) was also reported (a.45).

| Table 50 Examples of industrial rubberproduct applications |                 |  |  |
|--|-----------------|--|--|
| Application Polymer  |                 |  |  |
| Conveyor belt cover  | NR              |  |  |
| Engine mount   | NR              |  |  |
| Tank pad   | NR/SBR/BR blend |  |  |
| Oil seal   | NBR             |  |  |

# 3.2.1 Conveyor Belt Cover - NR

The key performance properties of conveyor belts, particularly belt cover compounds, are flex resistance, abrasion resistance and low heat build up. In order to achieve better flex resistance, a conventional cure package is recommended. However, such a system suffers from the adverse effects of heat generation. In a recent study, the addition of Perkalink 900 was recommended, the effect is shown in a typical, NR based belt cover formulation in **Table 51**.

| Table 51 Conveyor belt cover formulations |         |                 |  |
|---|---------|-----------------|--|
| Ingredients                               | Control | + Perkalink 900 |  |
| NR  | 100     | 100             |  |
| N330                                      | 45      | 45              |  |
| Aromatic oil                              | 4       | 4               |  |
| ZnO                                       | 5       | 5               |  |
| Stearic acid                              | 2       | 2               |  |
| 6PPD                                      | 1       | 1               |  |
| CBS                                       | 0.5     | 0.5             |  |
| Sulfur                                    | 2.5     | 2.5             |  |
| Perkalink 900                             | -       | 1               |  |

Cure characteristics for these two formulations are shown in **Figure 15**. The antireversion agent has no effect on scorch resistance and time to optimum cure. The beneficial effect becomes apparent when reversion occurs, as observed in the control compound: the compound containing the antireversion agent maintains a torque level close to the maximum.

The antireversion effect is also evident in the stability of vulcanisate properties following overcure or air ageing at 100 °C. Tensile, flex/cut growth characteristics and abrasion resistance are superior for the compound containing the antireversion agent. **Figure 16** shows the improvement in tensile strength of aged compounds containing Perkalink 900. **Figure 17** illustrates the superior flex/cut growth properties of compound containing Perkalink 900. **Figure 18** shows an improvement in abrasion resistance of formulations containing Perkalink 900.

A major benefit imparted by the antireversion agent concerns heat build up under dynamic conditions as encountered during the service of conveyor belts. Both at optimum cure and overcure the antireversion agent is extremely effective in reducing heat build up as determined by the Goodrich Flexometer test (**Figure 19**). The control compound, containing no antireversion agent, exhibits a significantly higher heat build up. This

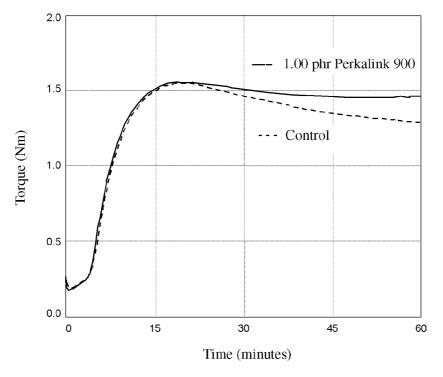


Figure 15

Cure characteristics of conveyor belt cover formulations at 150 °C

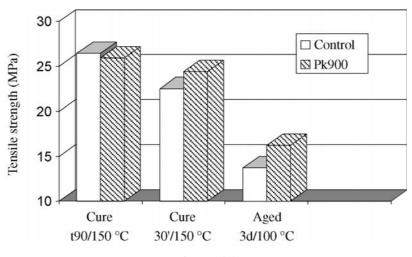


Figure 16

Tensile strength of conveyor belt cover formulations

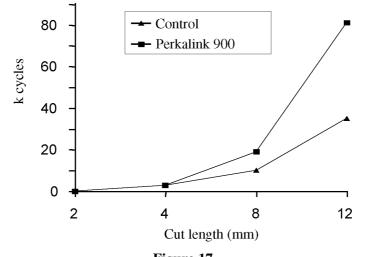


Figure 17 De Mattia flex/cut growth of conveyor belt cover formulation (cure 30'/150 °C)

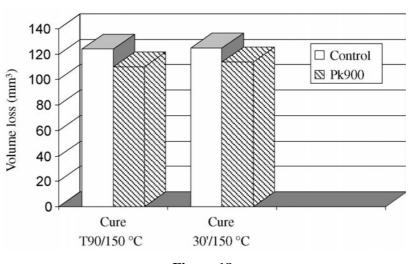


Figure 18 Abrasion loss (DIN) of conveyor belt cover formulations

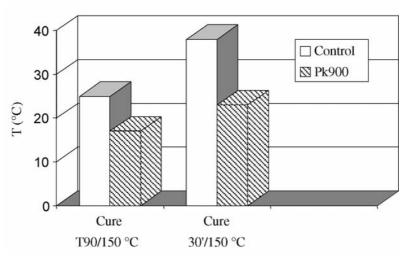


Figure 19

Goodrich Flexometer heat build up at 100 °C after 30 minutes – tests on conveyor belt cover compounds (Load 108 N, Stroke 4.45 mm, Frequency 30 Hz)

suggests that rolling energy losses can be reduced through the use of Perkalink 900, an important consideration in today's energy conscious environment.

# 3.2.2 Engine Mount - NR

The essential performance properties for an engine mount are low heat build up and low dynamic compression set. In order to achieve the desired properties, a cure system comprising of sulfenamide (moderate loading, 1.2-1.7 phr) with sulfur (2-2.5 phr) is recommended. A small amount of ultra accelerator might be necessary to generate a crosslink network capable of improving dynamic compression set as well as heat build up. In spite of this modified cure system (CBS/TMTD/S), the properties are still not optimum, particularly with regard to heat build up. The addition of an antireversion agent might provide advantage and with this in mind the effect of Perkalink 900 has been studied. The formulations are shown in **Table 52**. These have been determined for a typical engine mount compound based on natural rubber, with and without Perkalink 900.

The engine mount formulation contains a cure package tending to an SEV system; this will provide a greater degree of reversion resistance compared to a CV cure system.

The cure data show that Perkalink 900 does not affect scorch time or time to optimum cure, this is illustrated in **Table 53**. Tensile data following

| Table 52 Engine mount formulations |         |               |  |
|------------------------------------|---------|---------------|--|
| Ingredients                        | Control | Perkalink 900 |  |
| NR                                 | 100     | 100           |  |
| N-660                              | 42      | 42            |  |
| Zinc oxide                         | 5       | 5             |  |
| Stearic acid                       | 2       | 2             |  |
| Oil                                | 9       | 9             |  |
| MC wax                             | 2       | 2             |  |
| 6PPD                               | 2       | 2             |  |
| TMQ                                | 2       | 2             |  |
| Resin                              | 2       | 2             |  |
| Santocure CBS                      | 2.5     | 2.5           |  |
| Perkacit TMTD                      | 0.2     | 0.2           |  |
| Sulfur                             | 2.25    | 2.25          |  |
| Perkalink 900                      | -       | 0.75          |  |

overcure (at 150 °C and 170 °C) and ageing at 100 °C indicate a trend of improved strength (**Figure 20**).

The important vulcanisate properties demanded by this application, low heat build up and low dynamic set, have been determined in the Goodrich Flexometer test. The compound containing the antireversion agent exhibits a marked decrease in heat build up and dynamic permanent set (**Figures 21 and 22**).

| Table 53 Cure characteristics at 150 °C, (170 °C) and Mooney scorch |             |               |
|---|-------------|---------------|
|   | Control     | Perkalink 900 |
| ML (Nm)   | 0.17 (0.16) | 0.16 (0.15)   |
| MH-ML torque (Nm)   | 1.49 (1.35) | 1.43 (1.34)   |
| ts2 (minutes)   | 4.1 (1.1)   | 4.1 (1.1)     |
| T90 (minutes)   | 8.7 (2.5)   | 8.4 (2.4)     |
| Mooney scorch, T5, 121 °C   | 25.6        | 25.4          |

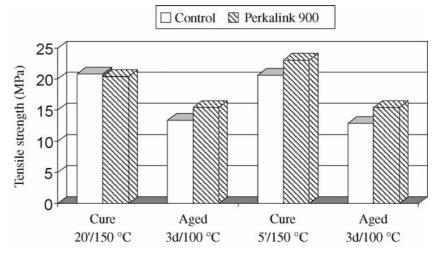
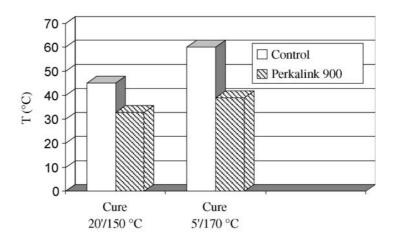


Figure 20

Tensile strength tests on engine mount compounds





Goodrich Flexometer heat build up at 100 °C after 30 minutes – tests on engine mount compounds (Load 108 N, Stroke 4.45 mm, Frequency 30 Hz)

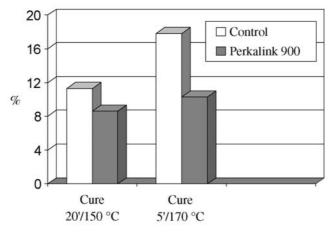


Figure 22

Goodrich Flexometer Dynamic Permanent Set at 100 °C after 30 minutes – tests on engine mount compounds (Load 108 N, Stroke 4.45 mm, Frequency 30 Hz)

Even at the increased cure temperature of 170 °C the compound containing the antireversion agent is little changed with regard to heat build up and dynamic set compared to the lower cure temperature. The control compound on the other hand exhibits both increased heat build up and dynamic set at the higher cure temperature. This suggests that increasing the cure temperature as a means of increasing productivity would have no detrimental effect on compound performance in this application.

# 3.2.3 Tank Pad – NR/SBR/BR Blend

The required properties for a tank pad are heat stability, low heat generation, improved flex and good compression set. This can be achieved by selecting a cure package between SEV and towards an EV cure. A combination of a sulfenamide with a thiazole is suitable. Generally the sulfenamide level should be low and the thiazole accelerator should be high. This is to allow a reaction between the sulfenamide and the thiazole accelerator so that the intermediate can function as an efficient accelerator. The sulfur loading is generally high to address the improved flex requirements. Because of high sulfur loadings, the system suffers from the adverse effects of heat and reversion resistance.

Perkalink 900 is active in compounds based on blends of NR with the synthetic elastomers SBR and BR. An evaluation in a tank pad formulation has provided evidence of reduced heat build up on overcure; overcure is a common problem in the manufacture of these relatively bulky components. The control and test formulations, in which two levels of the antireversion agent have been evaluated, are listed in **Table 54**. Cure characteristics are given in **Table 55**.

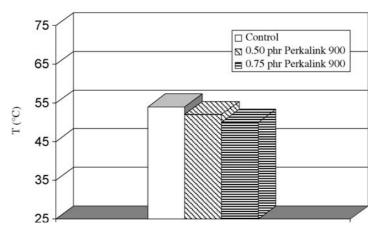
| Table 54 Tank pad formulations |      |      |      |
|--------------------------------|------|------|------|
| Ingredients                    | 1    | 2    | 3    |
| NR                             | 60   | 60   | 60   |
| SBR 1500                       | 20   | 20   | 20   |
| BR Buna CB10                   | 20   | 20   | 20   |
| N-220                          | 70   | 70   | 70   |
| Zinc oxide                     | 4    | 4    | 4    |
| Stearic acid                   | 2    | 2    | 2    |
| Oil                            | 40   | 40   | 40   |
| Santoflex 6PPD                 | 1    | 1    | 1    |
| MC wax                         | 3    | 3    | 3    |
| Santocure TBBS                 | 1    | 1    | 1    |
| Perkacit MBTS                  | 2    | 2    | 2    |
| Santogard PVI                  | 0.15 | 0.15 | 0.15 |
| Sulfur                         | 2    | 2    | 2    |
| Perkalink 900                  | -    | 0.5  | 0.75 |

| Table 55 Cure characteristics of tank pad<br>formulations at 155 °C |      |      |      |
|---|------|------|------|
|   | 1    | 2    | 3    |
| ML (Nm)   | 0.18 | 0.18 | 0.18 |
| MH-ML torque (Nm)   | 1.37 | 1.33 | 1.31 |
| ts2 (minutes)   | 3.4  | 3.6  | 3.7  |
| T90 (minutes)   | 7.2  | 7.3  | 7.3  |

Although little advantage is observed with regard to vulcanisate physical properties (**Table 56**), a benefit is apparent in terms of reduced heat build up as shown in **Figure 23**. In addition, the antireversion agent provides

greater thermal stability in terms of blow-out resistance (**Figure 24**). This observation is of important practical significance since it is not uncommon for tank pads to blow-out during service.

| Table 56 Vulcanisate properties of tank pad formulations (cure 30'/155 °C) |      |      |      |
|--|------|------|------|
| Properties   | 1    | 2    | 3    |
| Modulus at 300% elongation (MPa)   | 9.9  | 9.9  | 9.9  |
| Tensile strength (MPa)   | 17.4 | 17.8 | 18.0 |
| Elongation at break (%)  | 475  | 470  | 470  |
| Tear strength (kN/m)   | 67   | 75   | 72   |
| Abrasion loss (mm <sup>3</sup> )   | 88   | 75   | 72   |





Goodrich Flexometer heat build up at 100 °C after 60 minutes – tests on tank pad formulations (Load 108 N, Stroke 4.45 mm, Frequency 30 Hz)

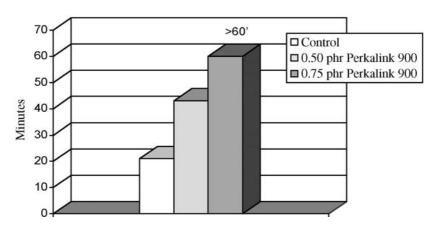


Figure 24

Goodrich Flexometer blow-out at 100 °C – tests on tank pad formulations (Load 216 N, Stroke 4.45 mm, Frequency 30 Hz, test time 60')

# 3.2.4 Oil Seal - NBR

The general requirement for oil seal applications is oil resistance, low compression set, high elongation and good heat stability. In order to achieve this, a cure system comprising of MBTS/TMTD and sulfur is preferred. TMTD is used in a small amount so that elongation is not negatively affected. In order to keep high elongation, the amount of sulfur should not be too low. Compression can be negatively affected if the sulfur loading is too high. The heat stability can be improved by incorporating antireversion agent Perkalink 900. Test and typical formulations for an NBR based oil seal are listed in **Table 57**. The comparative cure data are given in **Table 58**.

| Table 57 NBR based oil seal formulations |         |                |
|--|---------|----------------|
| Ingredients                              | Control | +Perkalink 900 |
| NBR                                      | 100     | 100            |
| N-550                                    | 60      | 60             |
| ZnO                                      | 5       | 5              |
| Stearic acid                             | 1       | 1              |
| DOP                                      | 15      | 15             |
| TMQ                                      | 2       | 2              |
| MBTS                                     | 1       | 1              |
| TMTD                                     | 0.1     | 0.1            |
| Sulfur                                   | 1.5     | 1.5            |
| Perkalink 900                            | -       | 0.75           |

| Table 58 Cure characteristics at 170 °C ofNBR oil seal formulations |         |                 |
|---|---------|-----------------|
|   | Control | + Perkalink 900 |
| M <sub>H</sub> -M <sub>L</sub> torque (Nm)                          | 1.36    | 1.39            |
| ts2 (minutes)   | 0.9     | 0.9             |
| T90 (minutes)   | 1.9     | 2.1             |

On extended overcure, which can be considered as an anaerobic ageing process, the compound containing the antireversion agent exhibits a higher tensile strength as shown in **Table 59** together with improved physical properties.

Both dynamic permanent set and oil swell resistance are improved on addition of Perkalink 900, indicative of the greater thermal and mechanochemical strength of the crosslinks formed from Perkalink 900 compared to sulfidic crosslinks. Data are shown in **Figures 25 and 26**.

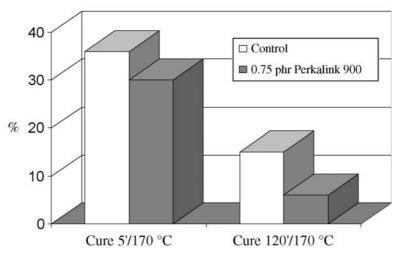
# **4** Concluding Remarks

Cure systems play an important role in achieving the performance requirements of rubber articles. Furthermore, these performance requirements are becoming more and more demanding due to the severity of service conditions, health and safety concerns and quality expectations.

Today, more and more know how is available to design a cure system capable of meeting these demanding requirements. New developments in materials allow compounders to reformulate a cure system capable of providing improved performance. Antireversion chemicals constitute a class of such materials, and can improve service performance. Health and safety issues, for example the concerns about the possible carcinogenicity of *N*-nitrosamines, have also lead to the introduction of new rubber additives.

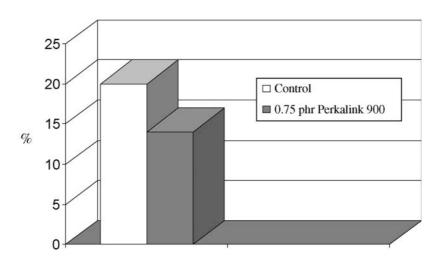
Cure systems, however, paint only part of the picture with regard to the manufacture of rubber articles; polymers and fillers are equally important in meeting performance requirements. In addition, compound mixing and processing play a critical role in achieving the final vulcanisate properties.

| Table 59 Physical properties of NBR oil seal formulations - cure 5'/170 $^\circ$ C (and 120'/170 $^\circ$ C) |             |                 |
|--|-------------|-----------------|
| Properties   | Control     | + Perkalink 900 |
| Modulus at 300% elongation (MPa)   | 12.9 (13.6) | 13.3 (14.0)     |
| Tensile strength (MPa)   | 19.0 (18.2) | 20.4 (21.1)     |
| Elongation at break (%)  | 530 (470)   | 560 (470)       |
| Tear strength (kN/m)   | 62 (55)     | 65 (60)         |





Goodrich Flexometer Dynamic Permanent Set at 100 °C after 120 minutes – tests on NBR oil seal formulations (Load 108 N, Stroke 4.45 mm, Frequency 30 Hz)





Swelling in ASTM oil #3, 168 hours at 150 °C (cure 5'/170 °C) – tests on NBR oil seal formulations

# References

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| Abbreviations and Acronyms |  |  |
|----------------------------|--|--|
| 6PPD                       | N-1,3-dimethylbutyl-N´-phenyl-p-phenylenediamine             |  |
| BCI-MX                     | 1,3-bis(citraconimidomethyl) benzene                         |  |
| BHT                        | butylated hydroxy toluene                                    |  |
| BIMS                       | isobutylene-para-methylene styrene-bromo-para-methyl styrene |  |
| BR                         | butadiene rubber   |  |
| CBS                        | N'-cyclohexylbenzothiazole-2-sulfenamide                     |  |
| CIIR                       | chlorobutyl rubber   |  |
| CLD                        | caprolactam disulfide  |  |
| CR                         | chloroprene rubber   |  |
| СТР                        | N-cyclohexylthiophthalimide                                  |  |
| CV                         | conventional vulcanisation                                   |  |
| DCBS                       | N-dicyclohexylbenzothiazole-2-sulfenamide                    |  |
| DIPDIS                     | bis(diisopropyl)thiophosphoryl disulfide                     |  |
| DOP                        | di(2-ethylhexyl)phthalate                                    |  |
| DOTG                       | di-o-tolylguanidine  |  |
| DPG                        | diphenyl guanidine   |  |
| DPTT                       | dipentamethylenethiuram tetrasulfide                         |  |
| DTDM                       | 4,4 <sup>-</sup> -dithiodimorpholine                         |  |
| DTPD                       | mixture of N,N'-diaryl-p-phenylenediamines                   |  |
| EDMA                       | coagent ethylene dimethacrylate                              |  |
| EPDM                       | ethylene-propylene-diene terpolymer                          |  |
| ETU                        | ethylene thiourea  |  |
| EV                         | efficient vulcanisation                                      |  |
| HMMM                       | hexamethoxymethylmelamine                                    |  |
| НМТ                        | hexamethylene tetramine                                      |  |
| H/R                        | HMT or HMMM and resorcinol                                   |  |
| HTS                        | hexamethylene-1,6-bis(thiosulfate)                           |  |
| HVA-2                      | bis-maleimides   |  |
| IIR                        | butyl rubber   |  |
| IRP                        | industrial rubber products                                   |  |
| IS                         | insoluble sulfur   |  |
| IS OT 20                   | insoluble sulfur oil treated 20%                             |  |
| MBS                        | 2-morpholinothiobenzothiazole                                |  |
| MBSS                       | 2-morpholino-dithio-benzothiazole                            |  |
| MBT                        | 2-mercaptobenzothiazole                                      |  |
| MBTS                       | 2,2 <sup>-</sup> -dithiobenzothiazole                        |  |

| Abbreviations and Acronyms (Continued) |  |  |
|--|--|--|
| МС                                     | magnesium carbonate                                      |  |
| MU                                     | Mooney unit  |  |
| NA                                     | N-nitrosamines   |  |
| NBR                                    | nitrile rubber   |  |
| NR                                     | natural rubber   |  |
| OTOS                                   | N-oxydiethylene thiocarbamyl-N´-oxydiethylenesulfenamide |  |
| OTR                                    | off-the-road   |  |
| phr                                    | parts per hundred parts of rubber                        |  |
| PVI                                    | pre-vulcanisation inhibitor                              |  |
| S                                      | sulphur  |  |
| SBR                                    | styrene butadiene rubber                                 |  |
| SEV                                    | semi-efficient vulcanisation                             |  |
| t_                                     | half-life  |  |
| TAC                                    | triallyl cyanurate                                       |  |
| TAIC                                   | triallyl isocyanurate                                    |  |
| TBBS                                   | N-butylbenzothiazole-2-sulfenamide                       |  |
| TBSI                                   | N-tert-butyl-2-benzothiazolesulfenimide                  |  |
| TBzTD                                  | tetrabenzylthiuram disulfide                             |  |
| TDEC                                   | tellunium diethyldithiocarbamate                         |  |
| TESPT                                  | bis-(3-triethoxysilylpropyl) tetrasulfide                |  |
| TETD                                   | tetraethylthiuram disulfide                              |  |
| TMQ                                    | 2,2,4-trimethyl-1,2-dihydroquinoline, polymerised        |  |
| TMTD                                   | tetramethylthiuram disulfide                             |  |
| ТМТМ                                   | tetramethylthiuram monosulfide                           |  |
| XIIR                                   | halobutyl rubber   |  |
| ZBEC                                   | zinc dibenzyldithiocarbamate                             |  |
| ZBPD                                   | zinc o,o-dibutylphosphorodithioate                       |  |
| ZDBC                                   | zinc dibutyldithiocarbamate                              |  |
| ZDEC                                   | zinc diethyldithiocarbamate                              |  |
| ZDMC                                   | zinc dimethyldithiocarbamate                             |  |
| ZMMBI                                  | 5-methyl-2-mercapto-benzimidazole                        |  |

# Abstracts from the Polymer Library Database

#### Item 1

Journal of Applied Polymer Science 83, No.5, 31st Jan.2002, p.1119-27 A COMPARISON OF TETRAETHYLTHIURAM AND TETRAMETHYLTHIURAM DISULFIDE VULCANISATION. II. REACTIONS IN RUBBER Gradwell M H S; Grooff D Port Elizabeth,University

In studying the reactions of tetraethylthiuram disulphide (TEDT) with polyisoprene under vulcanisation conditions, polyisoprene samples compounded with different combinations of TETD, sulphur, and ZnO were heated to different degrees of vulcanisation in a differential scanning calorimeter. The crosslink density values of the compounds obtained were determined by swelling, and the extractable residual curing agents and reaction products were analysed by HPLC. The TETD and also tetramethylthiuram disulphide (TMTD) induced crosslinking in the absence of added sulphur. When sulphur was present TETD sulphides were formed immediately before crosslinking started, and the TETD polysulphides were the initial crosslinking agents. The easy reaction between TETD and zinc oxide to give zinc diethyldithiocarbamic acid produced much higher crosslink densities. The higher activity between TETD and zinc oxide as compared with TMTD and zinc oxide failed to produce any appreciable changes in the vulcanisate. 21 refs.

SOUTH AFRICA Accession no.842758

# Item 2 Elastomery 5, No.5, 2001, p.13-28 Polish CANCEROGENIC N-NITROSAMINES IN THE RUBBER INDUSTRY Kleps T; Pysklo L

Instytut Przemyslu Gumowego Stomil Piastow

The current state of knowledge on N-nitrosamines formation in production processes in the rubber industry is presented. Taking into account that N-nitrosamines are toxic and carcinogenic, or probably carcinogenic for human beings, their emission creates threat for environment and health particularly in the case of rubber goods intended for direct contact with foodstuffs and humans, especially children (soothers, toys etc.) The origin of N-nitrosamines formation in the rubber industry is described. Raw materials, mainly vulcanisation accelerators, which can cause N-nitrosamines formation, are characterised and necessary measures to eliminate their emission or reduce concentration are presented. The best way is to apply 'safe accelerators' which are not derivatives of secondary amines. The most selective and sensitive method of N-nitrosamines determination and identification is the GC/TEA technique. By means of this method, very small quantities of N-nitrosamines and their precursors can be identified with accuracy and precision required in standards and EU directives. This is very important from the point of view of human health. Information on legislation concerning N-nitrosamines is also included. 63 refs.

EASTERN EUROPE; POLAND *Accession no.839942* 

# Item 3

Polymer Plastics Technology and Engineering 40, No.5, 2001, p.635-52 EFFECT OF FILLERS AND VULCANISING SYSTEMS ON THE PHYSICOMECHANICAL AND ELECTRICAL PROPERTIES OF EPDM VULCANISATES Ismail M N; Turky G M

Egypt, National Research Centre

The results are reported of a study of the effects of fillers and vulcanising agents on the physicomechanical and dielectric properties of EPDM. Fillers employed were kaolin, quartz, PVC and talc and the vulcanising agents were TMTD and sulphur/N-cyclohexyl-2-benzothiazyl sulphenamide. The permittivity and dielectric loss versus temperature for the vulcanisates are illustrated and the effects of thermal ageing on the physicochemical properties of the vulcanisates are discussed. 25 refs. EGYPT

Accession no.839169

# Item 4

# Kautchuk und Gummi Kunststoffe 54, No.11, 2001, p.612/7 BALANCED CROSSLINK NETWORK CREATED IN NATURAL RUBBER BY USING SULPHENAMIDE/TETRABENZYL THIURAM DISULPHIDE CURE SYSTEM Datta R; Oude Egbrink B; Ingham F; Mori T Flexsys BV

An investigation was carried out into the use of a lower amount of tetrabenzyl thiuram disulphide in NR formulations to achieve a faster cure rate without adversely affecting scorch resistance and flex related properties. Vulcanisate properties were correlated with the fine structure of the vulcanisates through a network study and vulcanisate properties at a cure temperature of 150C and cure times of 60 and 90 minutes tabulated. It was found that a combination of the above accelerator and a sulphenamide provided efficient vulcanisation with little or no loss in scorch safety and generated a crosslink network containing a more balanced distribution of polysulphidic and monosulphidic crosslinks compared to a compound containing only the sulphenamide or a combination of a sulphenamide and TMTD. 15 refs. EUROPEAN COMMUNITY: EUROPEAN UNION:

NETHERLANDS; WESTERN EUROPE Accession no.837197

# Item 5

**Rubber World** 225, No.2, Nov. 2001, p.41-4 **COMPARING CURING SYSTEMS: PEROXIDE-COAGENT VERSUS SULPHUR-ACCELERATOR IN POLYISOPRENE** 

McElwee C B; Lohr J E Sartomer Co.

The development of a new generation of peroxideinitiated solid functional coagents is reported along with the results of a study carried out to compare these cure systems with sulphur and peroxide cure systems in polyisoprene formulations. Data are provided on the cure characteristics, compression set, tensile properties, hardness, heat ageing, flexural properties, tan delta, complex modulus and lap shear adhesion of the formulations and the benefits of the new systems are highlighted.

USA Accession no.837116

Item 6

**Rubber Chemistry and Technology** 74, No.3, July/Aug. 2001, p.451-92 **PEROXIDE VULCANIZATION OF ELASTOMERS** Dluzneski P R

GEO Speciality Chemicals Inc.

Details are given of the competing chemical reactions involved in the peroxide vulcanisation of elastomers. The effect of various factors including the type of polymer, type and concentration of peroxide, cure temperature, and the presence of additives such as co-agents and antidegradants are discussed. 100 refs.

USA Accession no.836938

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# Item 7

*Rubber World* 224, No.5, Aug. 2001, p.34-7 **CHEMISTRY OF PEROXIDE VULCANIZATION** Dluzneski P R Hercules Inc.

Brief details are given of the chemistry involved in the vulcanisation of elastomers and plastics using peroxides as an alternative to sulphur. Data are presented for the peroxide curing of EPDM.

USA Accession no.835538

Item 8

*Polymer Bulletin* 47, No.2, Oct.2001, p.159-66 **NOVEL REAGENTS FOR HEAT-ACTIVATED POLYMER CROSSLINKING** Huffman B S; Schultz R A; Schlom P J National Starch & Chemical Co.

The dipolar cycloaddition of nitrile oxides with species containing unsaturation is a very attractive means of forming crosslinks in polymer systems, since the reaction is rapid and yields thermally stable links. Latent nitrile oxide precursors can be prepared by reaction of an isocyanate and an alkyl nitroacetate. These precursors release alkanol and carbon dioxide when heated to liberate the highly reactive nitrile oxide species. An improved synthetic procedure is developed to afford novel crosslinking agents based on difunctional, trifunctional and aliphatic precursors. Application of these agents to polymer crosslinking is demonstrated. 9 refs.

Accession no.835040

Item 9

# Polymers & Polymer Composites 9, No.7, 2001, p.459-468 EFFECT OF THE VULCANIZING SYSTEM OF CURE AND MECHANICAL PROPERTIES OF NBR/EPDM BLENDS Oliveira M G; Soares B G Rio de Janeiro,Universidade Federal

The effect is investigated of the choice of vulcanising system on the curing parameters and mechanical properties of NBR/EPDM blends. The curing characteristics of the blends were shown to be affected by the accelerator type and the sulphur concentration. Sulphur/benzothiazyldisulphide and single and binary accelerator systems based on tetramethyl thiuram disulphide were investigated. Higher thermal stability was achieved in blends containing higher levels of sulphur, due, it is thought, to increased crosslinking in the EPDM phase. Results of dynamic mechanical investigations showed differences in Tgs, which increased with increasing the sulphur contents in both vulcanising systems. 27 refs.

BRAZIL

Accession no.834298

Item 10 Gummi Fasern Kunststoffe 38, No.4, April 2001, p.330-3 German EFFECT OF CBS SURFACE TREATMENT ON ITS REACTIVITY IN RUBBER VULCANISATION

# Vidal E; Borros S; Agullo N; van Ooij W J

Plasma polymerisation of appropriate monomers onto the surface of accelerator particles, in the present case cyclohexylbenzothiazole sulphenamide (CBS), allow the accelerating effect during the vulcanisation of rubber compounds to be controlled. Using squalene as a model substance for rubber, the effect of CBS modified with three different monomers on scorch time and crosslink density was studied. 21 refs. Articles from this journal can be requested for translation by subscribers to the Rapra produced International Polymer Science and Technology.

Accession no.833608

#### Item 11

Nippon Gomu Kyokaishi 74, No.3, 2001, p.105-9 Japanese STUDIES OF VULCANISING AGENTS AND ACCELERATORS. IX. SULPHYLIMINES AS PREVULCANISATION INHIBITORS

Akiba M; Ishida T; Tagami T

A report is presented on the use of sulphylimines with various substituents as scorch inhibitors in sulphur and peroxide curing of natural rubber and EPDM. 8 refs. Articles from this journal can be requested for translation by subscribers to the Rapra produced International Polymer Science and Technology.

Accession no.832996

Item 12

*Plastics, Rubber and Composites* 30, No.6, 2001, p.270-4 **STUDIES ON XANTHATE-ZINC DIETHYL DITHIOCARBAMATE ACCELERATOR COMBINATION IN NATURAL RUBBER** Palaty S; Joseph R

Cochin, University of Science & Technology

Zinc salts of ethyl isopropyl, and butyl xanthates are prepared in the laboratory. The synergistic effect of these xanthates with zinc diethyl dithiocarbamate (ZDEC) on the vulcanisation of NR is studied at different temperatures. The amount of the three xanthates and ZDEC in the compounds is optimised by varying the amount of ZDEC from 0 to 1.25 pphr and the xanthates from 0 to 1.5 pphr. The cure characteristics are also studied. The rubber gum compounds with the three xanthate accelerators and ZDEC are cured at various temperatures from 60 to 150 deg.C. The sheets are moulded and properties such as tensile strength, tear strength, crosslink density, elongation at break, compression set, heat build-up, abrasion resistance and flex resistance are evaluated. The properties show that the zinc xanthate ZDEC accelerator system has a positive synergistic effect on the curing and mechanical properties of NR compounds. 18 refs.

INDIA Accession no.831736

# Item 13

*Journal of Applied Polymer Science* 81, No.6, 8th August 2001, p.1439-43 EFFICIENCY OF SOME DINITRODIAMINES AND DINITRODIAMIDES IN IMPROVING DYNAMIC PROPERTIES OF VULCANISED RUBBER

Klasek A; Filipovicova E; Spacek J Zlin, Tomas Bata University; Brno, Technical University

The effect of dinitrodiamine and dinitrodiamide coupling agents on the dynamic properties of natural rubber and butadiene rubber based compounds was investigated as a way of improving dynamic properties. The vulcanisation characteristics, tear strength, impact resilience, and gel content of the compounds were measured and it was concluded that only the dinitrodiamines increased the impact strength. The carbon gel content assessment indicated that the dinitrodiamides did not influence the dynamic properties of the vulcanised rubber as they did form bonds between the rubber and carbon black. Full details of the compounds prepared and test results are given. 12 refs.

CZECH REPUBLIC

Accession no.830240

Item 14

RUBBERCHEM 2001 - THE INTERNATIONAL RUBBER CHEMICALS, COMPOUNDING AND MIXING CONFERENCE. Proceedings of a conference held Brussels, 3rd-4th April 2001. Shawbury, Rapra Technology Ltd., 2001, Paper 11, pp.3. 012 **STABILIZATION OF DYNAMIC PROPERTIES FOLLOWING AGEING BY USING 1,3 BIS(CITRACONIMIDOMETHYL) BENZENE** Datta R N; Huntink N Flexsys BV (Rapra Technology Ltd.)

The effect of the above antireversion agent (Perkalink 900) in truck (skim) as well as passenger tyre (tread) compounds (NR and SBR) was studied with respect to the stabilisation of dynamic properties. The physicomechanical and viscoelastic properties were studied and the crosslink density and distribution of crosslink types measured. The results obtained indicated that Perkalink 900 stabilised the changes in viscoelastic properties as observed during oxidation ageing process. The network studies suggested that the compound formed crosslinks at optimum cure when the system contained higher levels of sulphur as in skim compound. In SBR tread compounds, the C-C crosslinks were introduced during the ageing process indicating that Perkalink 900 was reactive under service conditions. 2 refs.

BELGIUM; EUROPEAN COMMUNITY; EUROPEAN UNION; NETHERLANDS; UK; WESTERN EUROPE

Accession no.829482

# Item 15

RUBBERCHEM 2001 - THE INTERNATIONAL RUBBER CHEMICALS, COMPOUNDING AND MIXING CONFERENCE. Proceedings of a conference held Brussels, 3rd-4th April 2001. Shawbury, Rapra Technology Ltd., 2001, Paper 9, pp.6. 012 **COMPARISON OF RUBBER CURING SYSTEMS** - **PEROXIDE-COAGENT VS SULFUR-ACCELERATOR IN POLYISOPRENE(IR)** Amara Y Cray Valley France

(Rapra Technology Ltd.)

A new generation of peroxide-initiated solid functional coagents for curing of polyisoprene is introduced. The physical properties obtained using these SARET coagents were shown to be virtually identical to the classic sulphur-accelerator cure systems. In addition, the flexural properties obtained using the new system greatly surpassed those of the sulphur system, while still maintaining the superior ageing characteristics of a peroxide-coagent system. This combination of properties opened up a wide range of applications previously unavailable for peroxide-coagent curing of elastomers. 3 refs.

BELGIUM; EUROPEAN COMMUNITY; EUROPEAN UNION; FRANCE; UK; WESTERN EUROPE

Accession no.829480

# Item 16

*Journal of Applied Polymer Science* 81, No.11, 12th Sept.2001, p.2587-96 **INFLUENCE OF PENDANT GROUPS ON THE TENSILE PROPERTIES OF POLYISOPRENE VULCANIZATES** 

van der Merwe M J; Gradwell M H S; McGill W J Port Elizabeth,University

The importance of stress induced crystallisation in promoting the tensile properties of polyisoprene gum vulcanisates is demonstrated by results of tensile and X-ray diffraction studies on compounds prepared with different accelerator and curative formulations that contribute different pendant side groups. Compounds were tested at slow and fast extension rates on a tensile test machine and those exhibiting stress induced crystallinity had higher tensile strength at higher rates of extension. These included compounds cured with tetramethylthiuram disulphide (TMTD) and zinc dimethyldithiocarbamate (ZDC). Compounds cured with mercaptobenzthiazole (MBTS) exhibited reverse behaviour with a higher ultimate tensile strength (UTS) being achieved at low extension rates, addition of zinc stearate altered the cure behaviour, allowing similar behaviour to TMTD and ZDC. Comparison with peroxide cure systems which exhibit high degrees of stress-induced crystallisation was made, but due to several reasons which were discussed the UTS in this case is much lower. Tensile behaviour of each

vulcanisation system is discussed in terms of structural restraints and crystallisation. 34 refs. SOUTH AFRICA Accession no.829358

# Item 17

Journal of Applied Polymer Science 81, No.11, 12th Sept.2001, p.2565-72 CRYSTALLIZATION OF VULCANIZATES. I. LOW-TEMPERATURE CRYSTALLIZATION AS A FUNCTION OF EXTENT OF CURE FOR POLYISOPRENE VULCANIZED WITH TETRAMETHYLTHIURAM DISULFIDE/ SULFUR AND 2-BISBENZTHIAZOLE-2,2'-DISULFIDE/SULFUR

van der Merwe M J; Gradwell M H S; McGill W J Port Elizabeth,University

A density column at -25 degrees centigrade was used to study the crystallisation behaviour of polyisoprene gum compounds cured with either tetramethylthiuram disulphide (TMTD and sulphur, or mercaptobenzthiazole sulphenamide (MBTS) and sulphur. The amounts of accelerator and sulphur were maintained constant at 4 and 3 phr respectively but the cure time was varied from 2.5 to 35 minutes at 130 degrees centigrade. Compounds were mixed in a miniature batch mixer. Both rate and degree of crystallisation decrease with an increase in the cure time of the sample, and the induction period prior to crystallisation commencing increased. This behaviour was explained by residual pendant groups on the polymer chain and to a lesser degree by crosslink formation. The bulkier MBTS pendant groups showed more prominent changes than did TMTD, and did not crystallise at all once a gel was formed during vulcanisation. Extraction of the unbound curatives resulted in samples with traces of remaining solvent which masked any expected effects. 43 refs.

SOUTH AFRICA

Accession no.829356

*Item 18 China Synthetic Rubber Industry* 24, No.5, 2001, p.294-7 Chinese **APPLICATION OF ZINC METHACRYLATE IN NBR** 

Zhaohui C; Dizhen W South China, University of Technology

The influence of Zn(MAA)2 on the vulcanisation properties, mechanical properties and heat-air ageing properties of sulphur and peroxide curing system of NBR is studied. The results show that mechanical properties and heat-air ageing properties of NBR vulcanisate are enhanced by adding Zn(MAA)2. Optimum curing time is prolonged in sulphur curing system and shortened in peroxide curing system by adding Zn(MAA)2. DSC indicates that the curing mechanisms of Zn(MAA)2 in two curing systems are different. 8 refs. CHINA

Accession no.829001

# Item 19

Kautchuk und Gummi Kunststoffe 54, Nos.7-8, 2001, p.372-6 EFFECT OF 1,3 BIS(CITRACONIMIDOMETHYL)BENZENE IN BUTYL, HALOBUTYL AND NBR COMPOUNDS Datta R N; Talma A G Flexsys BV

The above compound (Perkalink 900) was shown to be an effective antireversion agent for sulphur-cured diene elastomers. The performance advantages of using Perkalink 900 in butyl rubber (inner tubes, bladders), halobutyl rubber (inner liners) and nitrile rubber were demonstrated, the aim being to improve the heat resistance of the compounds. A mechanistic interpretation of the chemistry underlying the crosslinking in butyl rubber and halobutyl rubber was provided. 15 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; NETHERLANDS; WESTERN EUROPE

Accession no.827645

#### Item 20

*Journal of Polymer Engineering* Vol.21 No.4, July-Aug. 2001, p.341-7 **CURE CURVE WITH TWO PLATEAUS: THE RESULT OF INDIVIDUAL VULCANISATION REACTIONS** 

Duchacek V; Duskova M Prague,Institute of Chemical Technology; Prague,Institut fur Makromoleculare Chemie

The vulcanisation of natural rubber with the efficient accelerator N-cyclohexyl-2'benzothiazolesulphenamide, in the presence of zinc oxide, stearic acid, and 4,4'-dithiobismorpholine (DTBM), was studied with a disc oscillating curemeter to determine the causes of the pronounced plateaus in the cure curves observed with this and similar systems. It was determined that this behaviour was not caused by the formation of an inhibitory by-product but was due to the occurrence of at least two distinct crosslinking reactions. DTBM was shown to act as a sulphur donor and not, as initially suspected, as an inhibitor. 14 refs.

CZECH REPUBLIC

Accession no.826306

#### Item 21

BLOWING AGENTS AND FOAMING PROCESSES CONFERENCE 2001. Proceedings from a conference held Frankfurt, 13th-14th March 2001. Shawbury, Rapra Technology Ltd., 2001, Paper 27. 012

#### **OPTIMISATION OF CROSSLINKING AND GAS LIBERATION IN CELLULAR RUBBER** Vinje K

SINTEF Materials Technology (Rapra Technology Ltd.)

Geflex AS in Norway is the only manufacturer of cellular rubber insulation in Scandanavia. Its product Glavaflex is due to be launched on the world market, and is to be reformulated to meet the requirements of the global market for thermal conductivity, resistance to moisture vapour diffusion, and fire rating. SINTEF is working with varying compounds of synthetic rubber to optimise the crosslinking and gas liberation of Glavaflex. To stop vapour transmission, the rubber is made with closed cells filled with a gas made from a precursor which is decomposed when exposed to elevated temperatures. The effect of the selection of components in the rubber blend on the final properties of the product is examined, and in particular, the choice of vulcanisation chemicals and blowing agents are studied in order to produce a cellular material with a high degree of closed cells and with a low density. Nitrile rubbers blended with PVC were used as the base polymer.

#### GEFLEX AS

EUROPEAN COMMUNITY; EUROPEAN UNION; NORWAY; SCANDINAVIA; UK; WESTERN EUROPE Accession no.826303

Accession no.82030

#### *Item 22*

# International Polymer Science and Technology 28, No.6, 2001, p.T/42-4 INVESTIGATION OF THE CONTACT RESISTANCE AND SPECIFIC RESISTIVITY OF ELECTRICALLY CONDUCTIVE RUBBERS PRODUCED BY THIURAM VULCANISATION IN A RUBBER-METAL COUPLE

Shkapov D V; Kornev A E; Os'kin V M Lomonosov Academy of Fine Chemical Technology

The choice of a vulcanising system for electrically conductive rubbers is discussed, where the rubber is in contact with metal substrates, in order to improve the stability of the contact resistance at the rubber/metal boundary over a long service time. The aim of this work is to investigate the influence of the composition of a thiuram vulcanising system and the formulation of a conductive rubber system, on the contact resistance and volume resistivity of conductive compounds based on polyisoprene. The vulcanising group of the rubbers investigated contained: tetramethylthiuram disulphide, Ncyclohexyl-2-benzthiazyl sulphenamide, zinc oxide, and stearic acid. For comparison, a rubber produced using a vulcanising group of the same composition but without stearic acid, was also investigated. Results are discussed. 5 refs. (Article translated from Kauchuk i Rezina, No.6, 2000, p.32) RUSSIA

Accession no.826051

Item 23

*Revista de Plasticos Modernos* 80, No.533, Nov.2000, p.554-60 Spanish

# SOME ASPECTS OF THE CROSSLINKING REACTION OF NATURAL RUBBER WITH DIPENTAMETHYLENETHIURAM TETRASULPHIDE

Gonzalez L; Rodriguez A; del Campo A; Marcos A Instituto de Ciencia y Tecnologia de Polimeros

A study was made of the mechanism of the vulcanisation of NR compounds in the presence of the sulphur donor dipentamethylenethiuram tetrasulphide and in the presence or absence of zinc oxide and stearic acid. The density and distribution of crosslinks were determined, and the mechanical properties of the vulcanisates investigated. 14 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; SPAIN; WESTERN EUROPE

Accession no.825305

Item 24

Revista de Plasticos Modernos 80, No.533, Nov.2000, p.538-46 Spanish VULCANISATION SYSTEMS FOR CARBOXYLATED NITRILE RUBBERS (XNBR) Ibarra L; Alzorriz M

Instituto de Ciencia y Tecnologia de Polimeros

The structure and vulcanisation mechanisms of carboxylated rubbers in general are discussed, and results are presented of a study of the vulcanisation of carboxylated nitrile rubber using four different curing systems. The crosslink density, tensile and dynamic mechanical properties, stress relaxation and tear strength of vulcanised samples were investigated. 22 refs. EUROPEAN COMMUNITY; EUROPEAN UNION; SPAIN; WESTERN EUROPE

Accession no.825302

# Item 25

International Journal of Polymeric Materials 49, No.2, 2001, p.191-204 COMPARISON EFFECTS OF PALM OIL FATTY ACID AND STEARIC ACID ON DYNAMIC PROPERTIES, CURING CHARACTERISTICS AND MECHANICAL PROPERTIES OF CARBON BLACK FILLED EPOXIDIZED NATURAL RUBBER COMPOUNDS

Ismail H; Kamal S K; Mark S E Universiti Sains Malaysia

The effects of palm oil fatty acid and stearic acid on dynamic properties, curing characteristics and mechanical properties of carbon black-filled epoxidised NR compounds were studied. It was found that the scorch time and cure time increased with increasing concentrations of both acids. The maximum elastic torque increased with acid concentration, while the minimum elastic torque showed a decreasing trend. The results also indicated that the decrease in viscous torque and tan delta was significant with increasing acid concentration. For tensile modulus, hardness, maximum torque-minimum torque and swelling index, results indicated that both acids had some effect on crosslink density. Tensile and tear strengths, however, passed through a maximum as the concentrations of both acids increased. 12 refs.

MALAYSIA

Accession no.825132

Item 26

Kautchuk und Gummi Kunststoffe 54, No.6, 2001, p.308/14 EFFECT OF 1,3 BIS(CITRACONIMIDOMETHYL)BENZENE IN SILICA CONTAINING COMPOUNDS Datta R N; Hondeveld M G J

Flexsys BV

The effect of the antireversion agent 1,3 bis(citraconimidomethyl)benzene(Perkalink 900) was investigated in silica-containing off-the-road(OTR) and 'green tyre' compounds. It was shown that, by using Perkalink 900, it was possible to reduce the level of silane coupling agent, bis(3-triethoxysilylpropyl)tetrasulphide, both in OTR and in 'green tyre' compounds, providing either equivalent or better performance characteristics such as improved reversion resistance, lower heat buildup and enhanced blow-out times. The polymer-filler and filler-filler interaction parameters remained virtually unchanged, indicating no detrimental effect on reinforcement characteristics. Network studies indicated that Perkalink 900 provided better heat resistance because of the incorporation of thermally stable as well as flexible C-C crosslinks during the reversion regime. 24 refs. EUROPEAN COMMUNITY; EUROPEAN UNION; NETHERLANDS; WESTERN EUROPE

Accession no.825066

Item 27

**Polymer Testing** 20, No.6, 2001, p.607-14 **COMPARISON OF CETYLTRIMETHYLAMMONIUM MALEATE AND SULPHENAMIDE AS AN ACCELERATOR IN CARBON BLACK FILLED NATURAL RUBBER COMPOUNDS** 

Ismail H; Ahmad Z; Ishak Z A M Universiti Sains Malaysia

The effects of using cetyltrimethylammonium maleate(CTMAM) and N-cyclohexyl-2-benzothiazole-2-sulphenamide(CBS) as accelerators in carbon black-filled NR compounds were studied. The results obtained showed that cure time and scorch time were faster for CTMAM

than for CBS. CTMAM could function in its own right as an accelerator for sulphur vulcanisation, without the presence of zinc oxide and stearic acid as activators. The use of CTMAM in the absence of zinc oxide and stearic acid gave good tensile and tear strengths, tensile modulus, rebound resilience and hardness. 9 refs.

MALAYSIA

Accession no.824980

# Item 28

159th ACS RUBBER DIVISION MEETING - SPRING 2001. Held Rhode Island. 24th-27th April 2001. Akron,Oh., 2001, Paper No.37, pp.26. 012 HIGH MOLECULAR WEIGHT TETRA ALKYL THIURAM DISULFIDE(TATD) IN TIRE COMPOUNDS TO INCREASE CURE RATE WITHOUT SACRIFICING SCORCH SAFETY AND DYNAMIC PROPERTIES

Hong S W; Hannon M J; Kounavis J; Greene P K Uniroyal Chemical Co. (ACS,Rubber Div.)

The use of TATD in various tyre compounds was evaluated with the aim of increasing cure rate and maintaining properties. Comparison was made with the use of tetrabenzylthiuram disulphide(TBzTD). TATD provided equivalent scorch time to TBzTD in NR compounds, lower modulus and higher elongation. Tetramethylthiuram disulphide gave the fastest cure, but worst scorch safety, lowest elongation and higher modulus. TBzTD provided equivalent scorch time to the control, but reduced elongation and increased modulus. TATD had similar properties to the control with faster cure. Abrasion resistance with TBzTD and TATD was much better than the control compound without a secondary accelerator. 4 refs.

USA

Accession no.824952

#### Item 29

159th ACS RUBBER DIVISION MEETING -SPRING 2001. Held Rhode Island. 24th-27th April 2001.

# Akron,Oh., 2001, Paper No.35, pp.38. 012 CARBASULFANES AS CROSSLINKER FOR DIENE RUBBER - A MODEL COMPOUND STUDY

Hahn J; Palloch P; Walter E; Thelen N Cologne,University (ACS,Rubber Div.)

The potential of polycarbasulphanes (Sx(CH2)y)p as crosslinking agents was investigated by studying the structures of the crosslinks generated by these polymers in dependence on the parameters x (x = 2-4) and y (y = 1-3, 6). Model compound vulcanisation in combination with reversed-phase high performance liquid chromatography, NMR spectroscopy and mass spectrometry were used to identify the structure of the crosslinks. As a complex variety of hybrid bridges must be expected, the simple model olefin 2,3-dimethyl-2-butene, which did not further increase the complexity of the vulcanisation products, was selected. 20 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; GERMANY; USA; WESTERN EUROPE Accession no.824951

# Item 30

159th ACS RUBBER DIVISION MEETING - SPRING 2001. Held Rhode Island. 24th-27th April 2001. Akron,Oh., 2001, Paper No.34, pp.12. 012 **COMPARISON OF RUBBER CURING SYSTEMS: PEROXIDE-COAGENT VERSUS SULFUR-ACCELERATOR IN POLYISOPRENE** McElwee C B; Lohr J E Sartomer Co.Inc. (ACS,Rubber Div.)

A new generation of peroxide-initiated solid functional coagents was introduced for curing of synthetic polyisoprene elastomers. The resulting physical properties were shown to be similar to those of classical sulphuraccelerator cure systems. In addition, the flexural properties obtained by using the new system were found greatly to surpass those of the sulphur system, while still maintaining the superior ageing characteristics. The combination of properties obtained provided the opportunity of use in a wide range of applications previously unavailable to peroxide-coagent curing of elastomers.

USA

Accession no.824950

#### Item 31

159th ACS RUBBER DIVISION MEETING - SPRING 2001. Held Rhode Island. 24th-27th April 2001. Akron,Oh., 2001, Paper No.33, pp.27. 012 **MODIFYING THE PERFORMANCE OF SULFENAMIDE ACCELERATORS WITH THIURAMS** Chasar D W Goodrich B.F.,Co. (ACS,Rubber Div.)

Cure-Rite IBM and Cure-Rite IBT from B.F.Goodrich, a thiuram monosulphide and disulphide, respectively, based upon diisobutylamine, are new low nitrosamineproducing accelerators. These additives, together with other commercial thiuram compounds, were compared as co-accelerators for benzothiazole sulphenamides in SBR/polybutadiene and NR compounds. The effects that these thiurams had on the scorch safety and cure rates during curing and on the reversion resistance of the vulcanisates were demonstrated. Cure-Rite IBM excelled in improving the scorch safety of the benzothiazole while improving the cure rates. Cure-Rite IBT appeared to be a suitable replacement for the more expensive benzyl thiurams. Both new materials improved reversion resistance and blow-out properties in NR. Differences in the types of sulphur crosslinks developed by the various thiurams during vulcanisation are discussed. 6 refs.

USA

Accession no.824949

# Item 32

159th ACS RUBBER DIVISION MEETING - SPRING 2001. Held Rhode Island. 24th-27th April 2001. Akron,Oh., 2001, Paper No.30, pp.18. 012 **COAGENT SELECTION FOR WIRE AND CABLE APPLICATIONS** Costin R

Sartomer Co.Inc. (ACS,Rubber Div.)

The effect of Type I and Type II coagents on the properties of peroxide-cured EPDM, EPM and chlorinated PE(CPE) compounds was studied. The coagents contributed to the crosslink network by forming more C-C bonds relative to the peroxide control. In addition, compounds cured with peroxides and coagents were superior to sulphur-cured compounds in many properties, including heat aged performance, fluid resistance and electrical properties. Commercially-available coagents were compared in wire and cable formulations based on the above polymers. In the EPDM and EPM formulations, the liquid high 1,2vinyl polybutadiene(PBD) coagents often exceeded the performance of the trimethylolpropane trimethacrylate. The PBD-modified compounds particularly excelled in heat aged, water resistance and electrical properties. All the coagents improved performance of the CPE compound. The liquid maleinised PBD coagent was found to be particularly effective in CPE, resulting in the best physical properties and good electrical properties. 7 refs. USA

Accession no.824946

#### Item 33

159th ACS RUBBER DIVISION MEETING - SPRING 2001. Held Rhode Island. 24th-27th April 2001. Akron,Oh., 2001, Paper No.21, pp.22. 012 EFFECTS OF NBR POLYMER VARIATIONS ON COMPOUND PROPERTIES Del Vecchio R J; Ferro E B

Technical Consulting Services; Corry Rubber Corp. (ACS,Rubber Div.)

Using a designed experiment, variations in Mooney viscosity, acrylonitrile content, polymerisation method and cure system (sulphur or low sulphur) were investigated in relation to their effects on processing characteristics and standard physical properties, including heat ageing, oil resistance, compression set and stress relaxation. The use of a specialised polymeric additive (polyoctenylene) was also examined and testing of dynamic properties reported. Conclusions were drawn about the existence and level of effects of the control factors on the various properties. 1 ref.

USA

Accession no.824939

# Item 34

International Polymer Science and Technology 28, No.5, 2001, p.T/29-30 EVALUATION OF THE EFFICIENCY OF A BINARY SYSTEM OF ACCELERATORS CONSISTING OF PHOSPHONIUM SALT AND METAL DIPHENYLDITHIOPHOSPHATE Ovcharov V I; Okhtina O V

An evaluation is reported into the effect of using a binary system of accelerators consisting of a cation-active phosphonium salt and an anion-active metal dithiophosphate. The studies were carried out using model unsaturated elastomer compositions based on butadienemethylstyrene rubber. Also investigated was the efficiency of this system on the action of the thiazole accelerator dibenzothiazolyl disulphide. A synergistic effect was noted in relation to the process of sulphur vulcanisation, the degree of crosslinking, and the elastic strength properties. The proposed system of secondary accelerators was shown to provide a higher degree and rate of vulcanisation of elastomer compositions in combination with scorch resistance, than the systems of the individual accelerators. 7 refs. (Article translated from Kauchuk i Rezina, No.5, 2000, p.41)

RUSSIA

Accession no.824314

Item 35

# Journal of Applied Polymer Science 81, No.4, 25th July 2001, p.800-8 NR-EPDM COVULCANIZATION: A NOVEL APPROACH

Arup Kumar Ghosh; Subhas Chandra Debnath; Nityananda Naskar; Dipak Kumar Basu Indian Association for the Cultivation of Science

An elastomer blend consisting of highly unsaturated natural rubber (NR) and ethylene propylene diene rubber (EPDM) of low unsaturation was co-vulcanised using bis(diisopropyl)thiophosphoryl disulphide (DIPDIS) as a novel coupling agent/accelerator. The blend vulcanisates obtained showed improved physical properties, which was further improved by two-stage vulcanisation combined with judicious selection of the NR to EPDM ratio, which gave a coherent and homogeneous blend. The cure-rate mismatch problem was solved by formation of rubberbound intermediates and a multifunctional rubber additive, i.e. DIPDIS, which restricted the curative migration from lower to highly unsaturated rubber. The blend morphology as revealed by scanning electron microscopy indicated significant improvement in physical properties, especially of two-stage vulcanisates. 47 refs. INDIA

Accession no.823805

# Item 36

Rubber and Plastics News 30, No.23, 11th June 2001, p.23/34 CURE, ANTIDEGRADANT USE BETTER HOSE, BELT COMPOUNDS Sinha D P; Jablonowski T; Ohm B; Vanis V

Uniroyal Chemical

Rubbers used in hoses and belting are reviewed and several examples of cure systems and antidegradant packages for use in hose and belting compositions, which can increase heat resistance, improve flex fatigue and provide ozone resistance are presented and discussed. 22 refs. (2001 meeting of the All India Rubber Industries Assn.)

USA

Accession no.823139

#### Item 37

# Journal of Applied Polymer Science 81, No.3, 18th July 2001, p.710-8 EFFECT OF CROSSLINK STRUCTURES ON DYNAMIC MECHANICAL PROPERTIES OF NATURAL RUBBER VULCANATES UNDER DIFFERENT AGING CONDITIONS

Ruliang Fan; Yong Zhang; Chen Huang; Yinxi Zhang; Yongzhong Fan; Kang Sun

Shanghai, Jiao Tong University

The dynamic mechanical properties of rubber vulcanisate is an important property for dynamic applications including tyres. In this study the three main types of sulphur vulcanised natural rubbers were investigated to assess how the differing crosslink structures affect the dynamic mechanical properties of both filled and unfilled compounds before and after ageing. The total crosslink densities of conventional, semi-efficient and efficient curing systems were controlled to roughly the same level by changing both the accelerators and ratio of sulphur level to accelerator. All compounds were aged under aerobic and anaerobic conditions at various temperature and samples. Aged samples were measured for multiwave compression and static and dynamic stress using dynamic mechanical thermal analysis. The main conclusions were that using the conventionally cured compound showed the greatest change in dynamic mechanical properties after aerobic heat ageing with aerobic ageing causing more dynamic mechanical property changes than anaerobic ageing in all three compounds. Complete details of the preparation and test procedures are given and the results fully discussed. 25 Refs.

CHINA Accession no.821954 Item 38

Kautchuk und Gummi Kunststoffe 54, No.5, 2001, p.256-9 IMPROVED RUBBER PROPERTIES BY USING N-T-BUTYL-2-BENZOTHIAZOLE SULFENIMIDE (SANTOCURE TBSI) Datta R N; de Hoog A J Flexsys BV

The use of SANTOCURE TBSI primary amine-based accelerator in a NR/SBR 70/30 tyre compound requiring long scorch safety combined with a moderately slow cure rate was studied. The properties of the vulcanisates examined included cure and processing characteristics, physical properties after overcure and ageing, tear strength, compression set, heat build-up and crosslink density distribution. Attempts were made to correlate these properties with the network structure. 11 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; NETHERLANDS; WESTERN EUROPE

Accession no.820513

# Item 39

*Kautchuk und Gummi Kunststoffe* 54, No.5, 2001, p.250-5

German

INFLUENCE OF NOVEL CARBASULPHANE CROSSLINKS ON THE PROPERTY PROFILE OF DIENE-NATURAL RUBBER VULCANISATES Buding H; Jeske W; Weidenhaupt H J Bayer AG

Using 2,3-dimethylbutene-2 as a model, studies of the crosslink structure showed that 1,6-bis(N,Ndibenzylthiocarbamoyldithio)hexane (Vulcuren VP KA 9188 from Bayer) formed carbasulphane crosslinks. The thiol-amine swelling technique for determination of crosslink density and sulphur crosslink structure was used to show that Vulcuren resulted in short sulphur bridges in comparison with a semi-efficient vulcanisation(semi-EV) system. Alteration of the crosslink structure and the crosslink density after overcuring was less for the compound with Vulcuren. The performance of the bifunctional crosslinker was investigated in terms of static and dynamic properties in a carbon black-filled NR compound in comparison with a conventional and semi-EV vulcanisation system and also in a silica-filled S-SBR/ polybutadiene compound. The good performance of the Vulcuren-containing system could be seen by comparing the rheometer curves. The reversion tendency was almost zero, while the rheometer torque of the other reference compounds decreased significantly. 16 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; GERMANY; WESTERN EUROPE

Accession no.820512

Item 40

IRC 2001. Proceedings of a conference held Birmingham, 12th.-14th. June.

Birmingham, IOM Communications, 2001, p.496-518. 012 NOVOR 950 - THE CURE SYSTEM FOR HIGH PERFORMANCE

Metherell C Tun Abdul Razak Research Centre (Institute of Materials)

Novor 950 is a diisocyanate crosslinker for natural rubber, which imparts a combination of physical properties, which cannot be achieved by other means, it is claimed. The paper reviews the mechanism of reaction of Novor 950 with rubber and shows how reversion resistant formulations can be obtained. A cost effective series of mixed sulphur/Novor 950 systems of high performance can be obtained which possess fast curing characteristics, coupled with high reversion resistance. In particular, the two or three fold increase of fatigue life on high temperature heat ageing is noted. Another feature of Novor 950 vulcanisates is that it is almost impossible to detect the type of crosslinking system by chemical analysis; hence Novor 950 is an 'invisible' crosslinking system which cannot be reconstructed by means of reverse engineering of a product containing Novor 950. The product is reported to have become established in the European automotive field where the advantages of improved heat and flex fatigue resistance provide benefits in under-the-bonnet high temperature applications. 3 refs. EUROPEAN COMMUNITY; EUROPEAN UNION; UK; WESTERN EUROPE

Accession no.820081

Item 41

Blowing Agents '99. Proceedings of a conference held at the conference centre UMIST, UK, 9th.-10th December 1999. Shawbury, 1999, Paper 7, p.29-36. 012 **OPTIMISATION OF THE PRODUCTION OF** 

# FOAMED RUBBER GOODS WITH A LAB

**SCALE METHOD** Fuchs E; Reinartz K S Bayer AG

(Rapra Technology Ltd.)

Factors affecting the production of foamed rubber articles are examined, with respect to optimisation of the cell structure and surface appearance. Experiments are described, which demonstrate the importance of investigating the decomposition reaction of the blowing agent and the crosslinking reaction. It is shown that these reactions interfere strongly with each other, thus emphasising the need for careful selection of the blowing agents and crosslinking agents. This paper describes experiments with an EPDM-based formulation to investigate the different interactions caused by the ingredients in the his formulation, carried out on a laboratory scale. In additions, some results for formulations based on polychloroprene are discussed. 5 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; GERMANY; UK; WESTERN EUROPE

Accession no.818860

#### *Item 42 Industria della Gomma* 44, No.7, Sept.2000, p.44-7 Italian **FIGHTING REVERSION** De Tuoni E

An examination is made of the range of rubber chemicals produced by Flexsys, including accelerators, anti-reversion agents and antidegradants. Particular attention is paid to Perkalink 900, an anti-reversion agent based on biscitraconimidomethylbenzene. Reference is made to investments by the Company in environmental protection, the construction of a plant in Antwerp for the production of intermediates for antiozonants, and the acquisition of Kali Chemie's activities in the production of insoluble sulphur.

FLEXSYS; KALI CHEMIE AG BELGIUM; EUROPEAN COMMUNITY; EUROPEAN UNION; GERMANY; UK; USA; WESTERN EUROPE; WORLD Accession no.818376

# Item 43

Rubber Chemistry and Technology 74, No.1, March/April 2001, p.28-43 REVERSION STABLE NETWORKS WITH POLYSULPHIDE POLYMERS AS VULCANISATION AGENTS Hahn J; Palloch P; Thelen N; Weidenhaupt H J Cologne, University; Bayer AG

To study the network structure generated by the polysulphide ((CH2)2S4)p as a crosslinking agent, model compound vulcanisation with 2,3-dimethyl-2-butene as the model alkene is carried out. It is found that the polysulphide polymer generates hybrid general constitution Sn-(CH2CH2-Sm)k (n=1-4, m=1-4, k=1-6). To a lesser extent, the polysulphide behaves as a sulphur donor generating conventional sulphur bridges Sx (x=1-5). Polysulphidic bridges (x=4,5) are only detected in the very early stage of network formation. The process of network formation and the development of the bridges during prolonged heating is monitored by 1H NMR spectroscopy and compared with that of a sulphur cure. In the case of the polysulphide polymer cure the early network contains a relatively high amount of monomer and disulphide bridges and a stable network is formed after at least 60 min of heating. This effect results from the higher thermal stability of hybrid bridges in comparison to conventional sulphur bridges. The higher stability of the hybrid bridges also leads to a suppression of the formation of 3,4-dimethylthiophene which is identified in the sulphur cure as a product of reversion processes. Thus, the increased reversion stability, of polysulphide polymer cures, which has been claimed earlier, can be related to the formation and the increased stability of the hybrid bridges. 19 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; GERMANY; WESTERN EUROPE

Accession no.818143

Item 44

# Journal of Applied Polymer Science 80, No.12, 20th June 2001, p.2292-9 COMPARISON OF TETRAETHYL- AND TETRAMETHYLTHIURAM DISULFIDE VULCANIZATION. I. REACTIONS IN THE ABSENCE OF RUBBER Gradwell M H S; Grooff D

Port Elizabeth, University

The reactions of tetraethylthiuram di- and monosulphide (TETD and TETM, respectively) were investigated in the absence of rubber and compared with those reported previously for tetramethylthiuram compounds. The reactions of TETM, TETD and zinc diethyldithiocarbamic acid with zinc oxide and sulphur were investigated by DSC and the reaction products analysed by highperformance liquid chromatography. TETM was shown to be more stable at vulcanisation temps. but also less reactive with sulphur than tetramethylthiuram disulphide(TMTD). The reactions of TETD were very similar to those of TMTD, the TETD reacting more slowly than the TMTD to form analogous products. In the presence of zinc oxide, the formation of the zinc compound of TETD occurred readily. TMTD did not react readily with zinc oxide. The reaction of TETD with sulphur led to the formation of polysulphidic accelerator species, although the concentrations formed in the absence of rubber were significantly lower than that formed by the corresponding TMTD system. 23 refs.

SOUTH AFRICA

Accession no.817865

#### Item 45

Rubber World 224, No.2, May 2001, p.29-30 DISPELLING ORGANIC PEROXIDES' MYTHS AND LEGENDS Eilo G W Hercules Inc.

Organic peroxides have been used to crosslink elastomers and plastics for over 50 years. The organic peroxides utilised by the rubber industry react very predictably. Most are stable at room temperature and will decompose based on their half-life temperature curves. They can represent a severe hazard, however, if they are stored or used improperly. These issues are reviewed in detail. 4 refs. USA

Accession no.816766

Item 46 Kautchuk und Gummi Kunststoffe 54, No.3, 2001, p.122-6 ELECTROCHEMICAL EVIDENCE OF TRANSITORY IONIC SPECIES IN THE VULCANIZATION OF NATURAL RUBBER WITH SULFUR. 2-

#### MERCAPTOBENZOTHIAZOLE AND ZINC OXIDE: THE ACTIVATOR EFFECT OF STEARIC ACID Hummel K: Rodriguez E LS

Hummel K; Rodriguez F J S Graz, Technische Universitat

Continuous low level current measurements are used to investigate the vulcanisation of natural rubber with sulphur, 2-mercaptobenzothiazole and zinc oxide in the presence and absence of stearic acid. At elevated temperatures, the mixtures showed increased conductivity, which was attributed to the presence of ions. Current curve maxima, characteristic of transitory ionic species are detected. Possible ions are discussed in connection with references in the literature. For the mixture NR/S/MBT/ ZnO/StAc, the comparison with the rheometer measurements demonstrates a relationship between the formation of ionic intermediates and crosslinking of the polymer. The suitability of the electrochemical method for the investigation of ionic species in crosslinking reactions is demonstrated. 20 refs.

AUSTRIA; EUROPEAN UNION; WESTERN EUROPE *Accession no.815292* 

#### Item 47

Kautchuk und Gummi Kunststoffe 54, No.3, 2001, p.84/91 EFFECTS OF SULFUR ACCELERATORS ON THE PERFORMANCE OF EPDM/SBR BLENDS Zhao J; Ghebremeskel G; Peasely J Ameripol Synpol Corp.

The role of accelerators in the curing and mechanical properties of EPDM/SBR blends is investigated. In this study, blend properties were optimised by selecting accelerators with a shorter scorch time and a faster cure rate in the EPDM phase than in the SBR phase. The accelerators investigated included a sulphenamide-based accelerator and a system composed of a combination of a thiuram and thiazole-based accelerator. By use of techniques described in this work, sulphur vulcanisates with compound properties comparable to those cured with a cure system composed of peroxide and sulphur coagent were obtained. The effects of the accelerators in final compound properties, ozone resistance, heat ageing, and compression set. 13 refs.

USA

Accession no.815286

Item 48 China Rubber Industry 48, No.3, 2001, p.145-8 Chinese EFFECT OF POLYOLS ON PHYSICAL PROPERTIES OF SILICA-FILLED NR COMPOUND

Zhang Yan-mei; Weng Guo-wen; Qian Chun-ming

Xuzhou, School of Chemical Technology

The effect of type and level of polyol on the properties of silica-filled NR compound was investigated. The test results showed that the vulcanisation of silica-filled NR compound was accelerated by polyols, the diglycol being the most active, giving the fastest vulcanisation and good physical properties, followed by triethanolamine, glycol, polyglycol and glyceryl alcohol. There was an optimum level for each polyol. 1 ref. CHINA

Accession no.813827

# Item 49

China Rubber Industry 48, No.3, 2001, p.133-7 Chinese EFFECTIVE FACTORS ON HEAT AGING PROPERTIES OF IIR VULCANIZATE

Guo Jian-hua; Luo Quan-kun South China,University of Technology

The effect of different curing systems and other additives on the heat ageing properties of butyl rubber vulcanisate was investigated. The test results showed that the physical property retention of butyl rubber cured with resin after 160C heat ageing for 24 h was superior to that of those cured with sulphur or sulphur donor. The optimum heat ageing properties of butyl rubber vulcanisate were obtained when about 5 to 10 phr of resin, 5 to 9 phr of tin(II) chloride and 5 phr of zinc oxide were used and the heat ageing properties were further improved by adding ISAF/GPF and ricinus oil. 9 refs.

CHINA

Accession no.813824

#### Item 50

*Elastomery* 4, No.6, 2000, p.24-36 Polish

# TECHNICAL ASPECTS OF USING INSOLUBLE SULPHUR

Pysklo L; Glijer T

Instytut Przemyslu Gumowego Stomil Piastow

Physicochemical properties of insoluble sulphur (IS) grades applied by the Polish rubber industry are tested. Effects of some ingredients of rubber compounds, viscosity of compounds and processing temperature on IS reversion are also evaluated. It is found that Polsinex 33 has the best properties of all Polish IS grades. It has better reversion resistance and dispersibility than Flexsys' Crystex OT33. To assure maximum IS stability during processing, it is necessary to eliminate from rubber compounds ingredients of strong basic character. In the case of using the antioxidant TMQ and sulphenamide accelerators, only products containing minimum quantity of free amines should be applied. The sequence of addition of ingredients is also important. It is observed that addition

of zinc oxide in the final stage of mixing together with IS causes reduction of IS reversion. During processing, IS reverts faster in compounds of high viscosity. These results should help rubber technologists to better utilise advantages of IS based mainly on its non-blooming behaviour and to reduce to the lowest level IS reversion during processing. 12 refs.

EASTERN EUROPE; POLAND *Accession no.811180* 

# Item 51

Patent Number: US 6140427 A1 20001031 VULCANIZATION ACCELERATORS SUITABLE FOR THE INTRODUCTION OF POLAR SUBSTITUENTS

Scholl T; Weidenhaupt H-J; Kelbch S; Engels H-W Bayer AG

Vulcanization accelerators of given formula are particularly suitable for the production of vulcanised rubbers modified with lateral polar groups.

EUROPEAN COMMUNITY; EUROPEAN UNION; GERMANY; USA; WESTERN EUROPE

Accession no.809220

#### Item 52

Journal of Applied Polymer Science 79, No.6, 7th Feb.2001, p.1074-83 NEW MECHANISM FOR REACTION OF AMINES WITH ZINC DITHIOCARBAMATES Dirksen A; Nieuwenhuizen P J; Hoongenraad M; Haasnoot J G; Reedijk J Leiden,University

Based on work with model compounds, a new mechanism is proposed for the reactions between bis(dithiocarbamato)zinc(II) (ZDAC) and amines. These reactions are an integral part of the reactions involved in the curing of unsaturated rubbers, especially polybutadiene, and are shown to be catalysed by elemental sulphur. In this proposed mechanism, the formation of an amine- dithiocarbamic intermediate is suggested, from which two known products of the reaction of ZDAC with primary amines, 1,1,3-trisubstituted and 1,3-disubstituted thioureas can be formed. With secondary amines a mixture of zinc dithiocarbamates is found and thiourea products are not formed. 13 refs

EUROPEAN COMMUNITY; EUROPEAN UNION; NETHERLANDS; WESTERN EUROPE Accession no.808782

Item 53

China Synthetic Rubber Industry 24, No.2, 2001, p.82-6 Chinese VULCANISATION CHARACTER OF TRANS-1,4-POLYISOPRENE AND MECHANICAL

#### **PROPERTIES OF VULCANIZED TPI**

Zuwei M; Baochen H; Jingshe S; Wei Y; Mingdong W Qingdao,Institute of Chemical Technology; Beijing,Research & Design Inst.of Rubber Ind.

The nature of the vulcanisation of trans-1,4-polyisoprene (TPI) and the effect of the degree of vulcanisation on the mechanical properties of vulcanised TPI were studied. The results showed that among three conventional types of accelerator used, (M, CZ, and TT), the vulcanisation curve of TPI with CZ as accelerator was found to be the most suitable. Vulcanised TPI was found to change suddenly from a hard material to a soft elastic rubber when the sulphur content increased from 4 phr to 5 phr. In addition, the addition of carbon black influenced the tensile strength advantageously. 5 refs.

CHINA

Accession no.808743

#### Item 54

**Polimeros: Ciencia e Tecnologia** 11, No.1, Jan./Mar.2001, p.9-15 Portuguese **EVALUATION OF PHENOLIC RESIN VULCANIZATION OF NITRILE RUBBER (NBR) THROUGH DETERMINATION OF THE CURE CONSTANT** 

Furtado A M; Peres A C C; Nunes R C R; Visconte L L Y; Furtado C R G

UFRJ; Rio de Janeiro, Universidade do Estado

The cure process involves reactions with multiple mechanisms, and its investigation can be followed by using different techniques. This work compares two methods in which rheometric parameters are used to calculate the cure rate related to the cure of nitrile rubber with phenolic resin and having polychloroprene and zinc oxide as the activators. A complete factorial (2 to the power of 3 + central point) experimental design was used as a base to prepare the formulations and to analyse the results. The results obtained identified the influence of the temperature, the amount of each component in the formulation, and the suitability of the evaluation methods for the cure process of nitrile rubber with phenolic resin. 8 refs.

BRAZIL

Accession no.808736

# *Item 55* Patent Number: US 6162875 A1 20001219 **LOW NITROSAMINE SYSTEMS FOR RUBBER VULCANIZATION** Virdi R S Robinson Brothers Ltd.

A metal dialkyldithiocarbamate or other such compound including a group of the formula R2NCS- or R2NS- is useful as a rubber accelerator/vulcanising agent, wherein each R is C5-18 branched alkyl. EUROPEAN COMMUNITY; EUROPEAN UNION; UK; USA; WESTERN EUROPE Accession no.808437

#### Item 56

Revue Generale des Caoutchoucs et Plastiques 77, No.786, May 2000, p.52-8 French VULCANISATION: IDENTIFICATION AND MECHANISMS OF REACTION

Plessier L; Lemaitre T; Triquenaux O; Gomez E Ifoca; LRCCP

Results are presented of a study in which the sulphur vulcanisation of polyisoprene was carried out at 150C and gas chromatography/mass spectrometry and HPLC were used to determine the chemical nature of intermediates and accelerator residues formed during the vulcanisation reaction. 15 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; FRANCE; WESTERN EUROPE Accession no.803898

#### Item 57

**Polymer Plastics Technology and Engineering** 39, No.5, 2000, p.817-27 **STYRENE-BUTADIENE RUBBER/EPOXIDISED NATURAL RUBBER BLENDS: THE EFFECT OF VULCANISATION SYSTEM ON BLENDS PROPERTIES** 

Ismail H; Suzaimah S Universiti Sains Malaysia

The dynamic properties, curing characteristics and swelling behaviour of blends cured using two types of vulcanisation systems, i.e. conventional vulcanisation (CV) and semi-efficient vulcanisation (semi-EV) are investigated. Results indicate that the maximum elastic torque and the torque difference (maximum elastic torque minus minimum elastic torque) increase with the increasing epoxidised NR (ENR) composition in the blend. However, a reverse trend is observed for tan delta and viscous torque. At a similar blend ratio the CV system gives a higher maximum elastic torque and torque difference but a lower tan delta and viscous torque than the semi-EV system. The scorch time, t2 and cure time, t90, decrease with increasing ENR composition in the blends. Semi-EV system blends exhibit shorter t2 and t90 than CV system blends. Degree of swelling decreases with increasing ENR in the blends and the CV system blends show better oil-swelling resistance. 24 refs. MALAYSIA

Accession no.802533

#### Item 58

*European Rubber Journal* 183, No.2, Feb.2001, p.20-1 WHERE IS ZINC GOING?

# White L

In rubber, zinc is typically used as zinc oxide at levels of around 2%. This adds up to 100 kt of zinc oxide a year used in the rubber sector in Western Europe. Environmentalists have raised concerns about zinc's potential ability to harm certain small aquatic species. This is currently causing a reassessment of its industrial uses, with manufacturers looking at ways of reducing zinc use. In rubber, zinc primarily acts as an activator. In addition, it is added to peroxide-cured materials to improve heat ageing, it acts as a crosslinking agent for rubbers containing halogen or carboxyl groups, and it can lower heat buildup and abrasion in tyre compounds. WESTERN EUROPE-GENERAL

Accession no.801940

# Item 59

Journal of Applied Polymer Science 78, No.13, 20th Dec.2000, p.2302-10 VULCANIZATION OF CHLOROBUTYL RUBBER. II. A REVISED CATIONIC MECHANISM OF ZNO/ZNCL2 INITIATED CROSSLINKING

Hendrikse K G; McGill W J Port Elizabeth,University

The vulcanisation of chlorinated poly(isoprenecoisobutylene) was studied when accelerated by ZnO/ ZnCl2. Both ZnCl2 and conjugated diene butyl units on the polymer chain are precursors to crosslinking and a revised cationic mechanism is proposed to account for crosslinking. Zn2OCl2 catalyses dehydrohalogenation, and the formation of catalytic amounts of Zn2OCl2 by reaction of ZnCl3- with ZnO, followed by H+ abstraction to give Zn2OCl2 and HCl is necessary in overall crosslinking reaction. The abstraction by ZnOCl2 of HCl in a reaction results in Zn(OH)Cl and ZnCl2. Zn(OH)Cl remains in the polymer as an extractable salt, and 50 % of the chlorine in the rubber is extracted as ZnCl2 when compounds reach their equilibrium crosslink density. The maximum crosslink density is only a quarter that theoretically possible, as the crosslinking restricts chain movement and reduces chance meetings. Zinc stearate promotes dehydrohalogenation, ZnCl2, being the only chloro-zinc salt formed. Where there is insufficient ZnO to trap all the chlorine present in the rubber reversion occurs. Trapping of the HCl by ZnO prevents reversion. 9 refs.

SOUTH AFRICA Accession no.800139

#### Item 60

*Journal of Applied Polymer Science* 79, No.1, 3rd Jan.2001, p.1-8 **STUDIES ON BINARY ACCELERATOR SYSTEM IN SULFUR VULCANIZATION OF NATURAL RUBBER**  Susamma A P; Varghese T; Mini E; Kuriakose A P Cochin,University of Science & Technology

The synergistic activity of binary accelerator systems in rubber vulcanisation is well understood. In this regard thiourea and its derivatives are important secondary accelerators. The proposal is that thiourea containing binary systems of rubber vulcanisation proceed by a nucleophilic reaction mechanism. Extensive investigations have been conducted of amidinothioureas (ATUs), which are derivatives of thiourea, as secondary accelerators. One of this study's objectives was to obtain further evidence in support of the theory of the nucleophilic reaction mechanism in such binary systems. In the current investigation phenyl substituted ATU was used as a secondary accelerator together with mercaptobenzothiazyl disulphide, tetramethyl thiuram disulphide, or cyclohexyl benzothiazyl sulphenamide in the sulphur vulcanisation of natural rubber. The results revealed an appreciable reduction in the cure time for the mixes containing the ATU compared to the reference mixes. These results are evidence supporting a nucleophilic reaction mechanism in the vulcanisation reaction being considered. Comparatively better, tensile properties and good retention of these properties after ageing were also shown by these vulcanisates. The optimum dosages of the secondary accelerator required for these vulcanisation reactions were also derived. 12 refs.

Accession no.798770

#### Item 61

*European Rubber Journal* 182, No.12, Dec.2000, p.11 SEC TO SELL NEW FKM CURING AGENT White L

In this article we are told that SEC Gummimischungen GMBH of Germany, a specialty compound distributor, has developed a hexamethylene diamine carbamate curing agent, "HMDC-Batch", which is designed for curing fluoroelastomers and ethylene acrylic elastomers.

SEC GUMMIMISCHUNGEN GMBH; DUPONT DOW ELASTOMERS; PMG SPA; US,FOOD & DRUG ADMINISTRATION; INTERBUSINESS EASTERN EUROPE; EASTERN EUROPE-GENERAL; EUROPEAN COMMUNITY; EUROPEAN UNION; FAR EAST; FRANCE; GERMANY; HUNGARY; ITALY; LATIN AMERICA; SOUTH AMERICA; SPAIN; UK; USA; WESTERN EUROPE

Accession no.798729

#### Item 62

# *Journal of Applied Polymer Science* 78, No.8, 21st Nov.2000, p.1495-504 **STRUCTURE PROPERTIES OF PURIFIED NATURAL RUBBER**

Suchiva K; Kowitteerawut T; Srichantamit L Mahidol University Purified natural rubber (PNR) is of interest because of its potentially lower toxicological effects than whole natural rubber (WNR) due particularly to the reduction in protein content. Improved dynamic mechanical properties have also been reported. Comparison of both gum and filled compounds, vulcanised using conventional cure systems (CV) and efficient vulcanisation systems (EV) prepared from PNR and WNR indicate that generally properties of the PNR are poorer than WNR. The exception is in the flex cracking resistance and the heat build up in the filled samples where PNR shows an improvement. Using the EV cure system on filled PNR gives properties almost comparable to WNR. A study of the distribution of the types of sulphur crosslink in both pNR and WNR vulcanisates indicates a more uniform distribution of monosulphidic (S), disulphidic (S2) and polysulphidic (Sx) crosslinks in the PNR samples (38.7/25/36.4 in PNR compared to 64.1/29.7/6.6 in WNR respectively). This is believed to be the reason for the better dynamic properties of PNR vulcanisates. 7 refs

THAILAND

Accession no.796699

Item 63

Journal of Applied Polymer Science 78, No.10, 5th Dec.2000 p.1769-75 XANTHATE ACCELERATORS FOR LOW TEMPERATURE CURING OF NATURAL RUBBER Palaty S; Joseph R

Cochin,University of Science & Technology

Laboratory preparation of zinc salts of ethyl, isopropyl, and butyl xanthates was carried out. These were purified by reprecipitation and characterised by IR, NMR, and thermogravimetric analysis techniques and melting points determined. The rubber compounds with different xanthate accelerators were cured at temperatures from 30 to 150 degrees Celsius. Moulding of the sheets was performed and properties such as tensile strength, tear strength, crosslink density, elongation at break, and modulus at 300 percent elongation were calculated. The results revealed that all three xanthate accelerators are effective for room temperature curing. 12 refs.

INDIA

Accession no.794915

#### Item 64

China Synthetic Rubber Industry 23, No.6, 2000, p.375 MECHANICAL PROPERTIES AND CROSSLINK DENSITY OF ZINC DIMETHACRYLATE REINFORCED EPDM

Zonglin P; Yong Z; Yinxi Z Shanghai,Jiao Tong University

The mechanical properties and crosslink density is studied of peroxide cured EPDM containing zinc dimethacrylate powder metal salts as reinforcing agent. High hardness and strength are imparted and high elongation is retained. Optimum levels are indicated for a balance of properties. 2 refs.

CHINA Accession no.794764

#### Item 65

*China Synthetic Rubber Industry* 23, No.6, 2000, p.357-61 Chinese

APPLICATION OF CROSSLINKING COAGENTS MPBM AND TMPTA IN HNBR

Xiang T; Jun Y; Dizhen W; Dongshan L South China, University of Technology

The effect is studied of the use of bis-functional crosslinking coagent, N,N'-m-benzyl bismaleimide (MPBM) and tri-functional crosslinking coagent trimethylpropane triacylate (TMPTA) on the properties of hydrogenated nitrile rubber, (HNBR). The results showed that MPBM and TMPTA could decrease the Tg of HNBR more than a half and one-third respectively, and increase the crosslinking density. FTIR analysis confirmed that MPBM and TMPTA both joined the crosslinking reaction. Small amounts of MPBM improved the heat resistance of HNBR, and TMPTA was shown to improve ozone resistance and maintain heat resistance. 5 refs.

CHINA

Accession no.794758

Item 66

158th. ACS Rubber Division Meeting - Fall 2000. Conference preprints. Cincinnati, Oh., 17th.-19th. Oct. 2000, paper 98 **NEW CABOT CARBON BLACK FOR IMPROVED PERFORMANCE IN PEROXIDE CURED INJECTION MOLDED COMPOUNDS** Bussolari S; Laube S Cabot Corp. (ACS,Rubber Div.)

Details are given of Spheron 4000, a furnace black developed by Cabot for use in peroxide cured injection moulded compounds. The product solves the problems normally associated with injection moulding peroxide cured systems, namely those relating to flow, and for which processing aides are often used, which can lead to an increase in mould fouling and consequently downtime. Lowering the viscosity of the compound through the use of a filler is an alternative to using processing aids. Cabot has developed Spheron 4000 furnace black with a surface area of about 32, which is demonstrated to improve flow properties and reduce mould fouling.

USA

Accession no.794196

*Item 67* 158th. ACS Rubber Division Meeting - Fall 2000. Conference preprints. Cincinnati, Oh., 17th.-19th. Oct. 2000, paper 97 **PREDISPERSIONS OF PEROXIDES AND COAGENTS** Kellgren J; Johansson A H

Rhein Chemie Corp. (ACS,Rubber Div.)

Advantages are examined of a peroxide/coagent cure system over the conventional sulphur curing system. However, the physical forms of peroxides/coagents can present handling problems leading to inaccurate dosages and variations in the physical properties of the final product. A comparison was made of t90 values for 40 batches of compound mixed with powdered peroxides versus 40 batches mixed with predispersed peroxides. Improved batch-to-batch consistency was found for the predispersed peroxides. It was also found that the average t90 was shorter for the batches mixed with predispersed peroxides, suggesting that more peroxide actually ended up in the rubber. The critical nature of the accurate dosing of peroxides and coagents was also demonstrated with data showing the variation in 100% modulus as a function of coagent concentration, and reinforcing the advantages offered by the use of peroxides in predispersed form. USA

Accession no.794195

#### Item 68

158th. ACS Rubber Division Meeting - Fall 2000. Conference preprints.

Cincinnati, Oh., 17th.-19th. Oct. 2000, paper 91 EFFECT OF CBS SURFACE TREATMENT ON ITS REACTIVITY IN RUBBER VULCANIZATION

Vidal E; Borros S; Agullo N; van Ooij W J Ramon Llull,University; Cincinnati,University (ACS,Rubber Div.)

Surface modification of sulphenamide accelerators is studied with respect to the influence of such modification on rubber scorch time. Cyclohexylbenzotriazole sulphenamide, (CBS) was surface treated by a proprietary plasma process, and using the model compound vulcanisation approach with squalene as the model molecule for natural rubber, the influence of the surface treatment on the reactivity of the accelerator was studied. Results of tests indicate that the film formed on the accelerator surface had changed the scorch time of the CBS, but without changing its crosslinking properties. 21 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; SPAIN; USA; WESTERN EUROPE

Accession no.794190

# Item 69

158th. ACS Rubber Division Meeting - Fall 2000. Conference preprints.

# Cincinnati, Oh., 17th.-19th. Oct. 2000, paper 79 MONTE CARLO SIMULATION OF THE PEROXIDE VULCANISATION OF ETHYLENE ELASTOMERS Johnston R T

DuPont Dow Elastomers (ACS,Rubber Div.)

A simulation program is used to investigate the curing of ethylene elastomers with peroxide curing agents. Simulation and experimental results are compared for several peroxide containing systems, and overall peroxide efficiencies and crosslinking/scission ratios are obtained. The study involves the use of dicumyl peroxide crosslinking of ethylene-octene copolymers from DuPont Dow Elastomers, with special emphasis on their rubber elastic and sol-gel properties. In addition, a new approach for the analysis of crosslinking behaviour and prediction of modulus is described. 19 refs. USA

Accession no.794179

#### Item 70

158th. ACS Rubber Division Meeting - Fall 2000. Conference preprints. Cincinnati, Oh., 17th.-19th. Oct. 2000, paper 77 **COAGENT SELECTION FOR PEROXIDE-CURED ELASTOMERS** Costin R; Nagel W; McElwee C B Sartomer Co.Inc. (ACS,Rubber Div.)

Peroxide-coagent curing systems are examined as alternatives to sulphur-accelerator curing. The peroxidecoagent systems are shown to provide greater versatility and improved cure performance and coagents which serve as adhesion promoters are also available. Advantages and disadvantages of the two cure systems are discussed. 8 refs.

USA

Accession no.794178

#### Item 71

158th. ACS Rubber Division Meeting - Fall 2000. Conference preprints. Cincinnati, Oh., 17th.-19th. Oct. 2000, paper 76 SAFE USE OF ORGANIC PEROXIDES FOR CROSSLINKING

Wakker J P; de Groot J J Akzo Nobel (ACS,Rubber Div.)

The properties of organic peroxides used of the crosslinking of elastomers are discussed from a safety point of view, with particular emphasis on its stability and decomposition effects. Differences between pure peroxides and peroxide formulations are shown. Classification of organic peroxides and their hazard types is briefly discussed, and general requirements for storage and handling of typical crosslinking peroxides and formulations are discussed. 9 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; NETHERLANDS; USA; WESTERN EUROPE Accession no.794177

### Item 72

158th. ACS Rubber Division Meeting - Fall 2000. Conference preprints. Cincinnati, Oh., 17th.-19th. Oct. 2000, paper 75 **CHEMISTRY OF VULCANISATION OF RUBBER** Dluzneski P R Hercules Inc. (ACS,Rubber Div.)

The advantages are outlined of the use of peroxide vulcanising agents in place of sulphur curing for elastomers. The chemistry of both types of vulcanisation systems is explained. Since the chemical mechanisms of each are very different, rubber compounders, experienced in the use of sulphur are reported to often find difficulties in formulating new products which make use of peroxide curatives. An overview is presented of the types of peroxides available and their properties. A description is included of the chemistry by which peroxides vulcanise rubber, followed by a discussion of the potential chemical interferences which may face rubber compounders converting from sulphur vulcanisation to peroxide curing. USA

Accession no.794176

#### Item 73

158th. ACS Rubber Division Meeting - Fall 2000. Conference preprints. Cincinnati, Oh., 17th.-19th. Oct. 2000, paper 73 **DISPELLING THE MYTHS AND LEGENDS SURROUNDING ORGANIC PEROXIDES** Eilo G W Hercules Inc.

(ACS,Rubber Div.)

Common myths and legends surrounding the use of organic peroxides as crosslinking agents in the rubber industry are discussed. An overview of regulatory requirements is included, as well as fundamental precautions for safe storage and handling. Aspects considered include shipping, fire hazards, health hazards, decomposition, storage and handling. The chemicals are claimed to be predictably reactive, and safe to use if they are not overheated, are stored in approved packages, kept away from incompatible materials, and spillages are cleaned up. 6 refs.

#### USA

Accession no.794174

#### Item 74

158th. ACS Rubber Division Meeting - Fall 2000. Conference preprints. Cincinnati, Oh., 17th.-19th. Oct. 2000, paper 54

#### PACKAGING IS KEY TO SUCCESS

Garrison T; Zeitler J Rhein Chemie Corp. (ACS,Rubber Div.)

The Bag-in-a-Bag technology from Rhein Chemie is described, together with a discussion on the advantages of using cure-blends. The cure-blends are customised multi-ingredient blends, which are designed to be quickly incorporated into rubber formulations by taking advantage of dispersion technology and/or the natural 'wetting out' properties of some chemicals to promote the incorporation of others. The blend components are provided in preweighed custom batch inclusion bags. Potential advantages and disadvantages associated with cure-blend chemistry are identified.

USA

Accession no.794159

#### Item 75

*International Polymer Science and Technology* 27, No.9, 2000, p.T/18-20

VULCANISATION OF AN ETHYLENE-PROPYLENE-DIENE RUBBER MODIFIED WITH DICHLOROCARBENE, AND ITS COVULCANISATION WITH CIS-1,4-POLYISOPRENE

Vinogradov A A; Kogan L M; Smirnov V P Russia,Scientific Research Institute for Synthetic Rubber

A study is made of the vulcanisation of an EPDM rubber extensively modified with dichlorocarbene, and its covulcanisation with cis-polyisoprene. On the basis of the structure of modified units, and taking into account the possibility of their transformations during heating, certain oxides and methylene-bis(maleimide) were tested as vulcanising agents. In addition, the self-vulcanisation of a modified EPDM without the addition of any vulcanisation agents was also studied. The principle of selective crosslinking of a dichlorocarbene-modified EPDM is established, based on the Diels-Alder reaction using the units containing a conjugated diene structure that are formed during heating under vulcanisation conditions. Strong dienophiles such as the bis(maleimides) are shown to be suitable for use as vulcanising agents, since they selectively crosslink the chlorine-containing EPDM phase in blends with cispolyisoprene, and thereby improve the covulcanisability of the given rubbers. 5 refs. Translation of Kauchuk i Rezina, No.2, 2000, p.4. RUSSIA

Accession no.791811

#### Item 76

*International Polymer Science and Technology* 27, No.9, 2000, p.T/15-7 **STUDY OF THE VULCANISATION OF SKF-26** 

#### FLUORINE-CONTAINING RUBBER BY A COMPOSITION OF 1,6-BIS(FURFURYLIDENE)HEXAMETHYLENEDIIMINE AND POLYPHOSPHITE

Gaidadin A N; Kutsov A N; Ogrel' A M; Tuseev A P Volgograd,State Technical University; OOO Intovelast

A new structure-forming system of the aminophosphite type has been developed for the vulcanisation of SKF-26 fluorine-containing rubber, which consists of a combination of bifurgin and polyethylene glycol phosphite, - an oligomer of the polyhydrophosphoryl compound type. The effectiveness of the selected system for the production of rubbers with improved hydrolytic resistance, thermal stability, and dynamic load resistance is investigated. 4 refs. Translation of Kauchuk i Rezina, No.2, 2000, p.2

RUSSIA

Accession no.791810

#### Item 77

Industria della Gomma 44, No.4, May 2000, p.16-8 Italian 1999/45/CE DIRECTIVE AND THE RUBBER INDUSTRY Garlanda B

The 1999/45/CE European Union directive relating to substances hazardous to the health or the environment is examined. Its implications for chemicals used in the rubber industry are briefly discussed, and classifications are presented for a number of accelerators.

EU; EUROPEAN COMMUNITY; EUROPEAN UNION; WESTERN EUROPE-GENERAL

Accession no.790897

# Item 78

Patent Number: US 6072009 A1 20000606 METHODS FOR VULCANIZING ELASTOMERS USING MONOMERIC DISTILLATE BY-PRODUCT Phillips C F; Conte A J

Arizona Chemical Co.

The specification discloses a method for vulcanising an elastomer using as the cure promoter the monomeric distillate by-product from the clay-based dimerisation of an unsaturated fatty acid mixture, preferably a tall oil fatty acid mixture. The distillate provides a cost-effective alternative to conventional fatty acid promoters.

Accession no.790762

#### Item 79

*Kautchuk und Gummi Kunststoffe* 53, Nos.7/8, July/Aug.2000, p.419/25 **IMPROVEMENT OF THE CROSSLINKING OF POLYCHLOROPRENE** 

# Fuchs E; Reinartz K S Bayer AG

Vulcanisation of polychloroprene is usually performed by using metal oxides and derivatives of thiourea, mainly ethylene thiourea (ETU). These vulcanisates are known to have excellent physical properties, good ageing resistance and very good sealing properties that are based on the low compression and tension set. A substitution of ETU is necessary due to its assessment as a potential carcinogen. Laws or regulations have already been issued in several European countries. 3-Methyl-thiazolidinethione-2 (MTT) is an alternative crosslinking agent for polychloroprene, which permits economical and ecological optimisation of processing. In the presence of MTT, the amount and the activity of zinc oxide and magnesium oxide also show significant influences on the crosslinking characteristics. This is another way in which the vulcanisation rate can be influenced. The physical properties, e.g. tensile strength, depend only marginally on the vulcanisation system. Furthermore, significant advantages in the compression set and the ageing behaviour are found. 4 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; GERMANY; WESTERN EUROPE

Accession no.789810

#### Item 80

## Journal of Applied Polymer Science 78, No.2, 10th Oct.2000, p.304-10 EFFECT OF VULCANISATION SYSTEMS AND ANTIOXIDANTS ON DISCOLOURATION AND DEGRADATION OF NATURAL RUBBER LATEX THREAD UNDER UV RADIATION

Kurian J K; Peethambaran N R; Mary K C; Kuriakose B India,Rubber Research Institute

The effect of accelerator combinations and antioxidants on UV radiation degradation of NR latex thread with a conventional and efficient vulcanisation system is presented. Zinc diethyl dithiocarbamate (ZDEC), zinc dibutyl dithiocarbamate (ZDBC), zinc mercaptobenzothiazole (ZMBT) and tetramethyl thiuram disulphide (TMTD) are used as accelerators. The antioxidants used are reaction products of butylated p-cresol and dicyclopentadiene (Wingstay-L), tris-nonylated phenyl phosphite (Crystol EPR 3400), styrenated phenol (SP) and polymerised 1,2-dihydro 2,2,4-trimethyl quinoline (HS). The thread samples are exposed to UV radiation and the appearance and physical properties of the thread are examined. The results indicate that the threads having MBC + ZDEC and ZDBC + ZMBT combinations as accelerators are more resistant to UV radiation than the thread having the ZDEC + ZMBT combination. The antioxidants Wingstay-L and SP+HS are effective in retaining the physical properties of the thread after UV exposure, and Crystol EPR 3400 is better in reducing discolouration. 14 refs.

INDIA

## Item 81

IRC 2000. Conference proceedings. Helsinki, Finland, 13th-15th June 2000, paper 73 **IMPROVED FORM OF INSOLUBLE SULPHUR** Oude edgbrink B J G M; Ingham F A A; To B H Flexsys America LP (Nordic Council of Rubber Technology)

Insoluble sulphur is a high molecular weight polymeric form of sulphur and, as the name implies, is insoluble in rubber. This feature provides the rubber compounder with the freedom to formulate compounds containing high levels of sulphur but with no detrimental effect on green compound tack. The use of ordinary rubber makers sulphur (RMS) in such applications would lead to sulphur bloom on the surface of uncured components due to its relatively low solubility in elastomers; around 1 phr at room temperature in NR-based compounds. Insoluble sulphur, however, is sometimes difficult to disperse in rubber compounds. During the mixing process agglomeration can occur leading to poorly dispersed material. Cool processing temperatures aid in obtaining good dispersion. High mixing temperature, however, reduces the viscosity of the compound and results in lower effective shear forces, unfavourable towards good dispersion. Additives such as oil and silica can improve dispersion significantly and even further improvements can be achieved through the use of pre-dispersed masterbatches. However, the preparation of pre-dispersed masterbatches and silica containing grades involves additional costs. A new grade of insoluble sulphur has been developed, making use of a special additive package that leads to enhanced dispersion and reduced incorporation time. This new product also provides superior flow characteristics, allowing ease of handling. The special additive package also provides increased thermal stability of the insoluble sulphur, ensuring enhanced bloom resistance following mixing and processing.

USA

Accession no.787105

#### Item 82

IRC 2000. Conference proceedings. Helsinki, Finland, 13th-15th June 2000, paper 22 PASSENGER CAR TREAD COMPOUNDS WITH IMPROVED PERFORMANCE BY USING VULCUREN TRIAL PRODUCT KA 9188 - A NEW BIFUNCTIONAL CROSSLINKER Jeske W; Dr.Buding; Dr. Weidenhaupt

Bayer AG (Nordic Council of Rubber Technology)

Most passenger car tyre tread compounds are based on SBR or SBR/BR blends and crosslinked with a cure system containing sulphur and various accelerators. Synthetic rubber vulcanisates are not as sensitive to reversion as NR vulcanisates but nevertheless they are subject to alterations due to changes in the crosslink structure during vulcanisation at high temperatures or during ageing. Such alterations should be avoided as the tyre performance could be negatively affected. The effectiveness of 1,6-bis(N,Ndibenzylthiocarbamoyldithio)hexane, a bifunctional crosslinker, is investigated in passenger car tread compounds. It is found that it can be used to advantage in cure systems for SBR-based vulcanisates. The vulcanisate properties, dynamic behaviour at optimum cure and the retention of properties after ageing can be considerably improved.

EUROPEAN COMMUNITY; EUROPEAN UNION; GERMANY; WESTERN EUROPE

Accession no.787059

Item 83

IRC 2000. Conference proceedings. Helsinki, Finland, 13th-15th June 2000, paper 11 EPDM COOLANT HOSES BASED ON PEROXIDE VULCANISATION TECHNOLOGY Vroomen G DSM Elastomers BV (Nordic Council of Rubber Technology)

In line with major automotive requirements calling for reduced energy consumption of cars, automotive bodies and engines are becoming increasingly compact to minimise air resistance. Further, automotive engines are designed to operate at higher temperatures in order to increase fuel efficiency. These two factors lead to a steady and continuous increase of the temperatures under the bonnet. Similarly, the operational temperatures of the cooling circuits in these cars have increased. Temperatures occurring under the bonnet in the circumference of the radiator hose are commonly 110-125 deg.C, with peak temperatures as high as 150 deg.C locally. The temperatures of the cooling liquid of the operating car can range between 90-110 deg.C. EPDM has established itself as the most preferred elastomer for radiator coolant hoses for cars (regular, not Turbo charged engines). It combines good temperature and ozone resistance with sufficient oil and chemical resistance for this application. Coolant hoses on basis of sulphur cured EPDM are well suited for the conditions mentioned above and, if properly designed and manufactured, perform well over the whole lifetime of a car: ten years or 250,000 km. 9 refs. EUROPEAN COMMUNITY; EUROPEAN UNION; NETHERLANDS; WESTERN EUROPE Accession no.787052

#### Item 84

IRC 2000. Conference proceedings. Helsinki, Finland, 13th-15th June 2000, paper 10 EFFECT OF PEROXIDE CURATIVES ON THE ELECTRO CHEMICAL DEGRADATION BEHAVIOUR OF HOSE COMPOUNDS Bennett M; Nijhof L

Avon Rubber plc; Akzo Nobel Polymer Chemicals BV

(Nordic Council of Rubber Technology)

Over the last years the automotive industry has made considerable changes in engine constructions. Typical effects of these changes are weight reduction of the engine, higher temperatures under the bonnet, more efficient fuel consumption and increased lifetime of components used in the engine compartment. As a consequence rubber articles, e.g. used in coolant systems, have to be reformulated to meet current specifications. New requirements, with respect to zinc sedimentation, heat ageing and low compression set at elevated temperature, have been set by the automotive industry. Also the fact that certain authorities demand to work with nitrosamine safe cure systems has made that the majority of current crosslink systems, for radiator hoses, are based upon organic peroxides. A relatively new phenomenon associated with all these changes is electrochemical degradation (ECD) of the coolant hose, which manifests as cracks and excessive swellof the rubber article. The effect of peroxide curatives on the ECD behaviour of hose compounds is evaluated. Special attention is paid to the type of peroxide, amount of peroxide and cure conditions. The relation between conductivity and ECD is evaluated for the compounds investigated. 6 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; NETHERLANDS; UK; WESTERN EUROPE

Accession no.787051

Item 85

IRC 2000. Conference proceedings. Helsinki, Finland, 13th-15th June 2000, paper 7 **"SELECTING ACRYLIC COAGENTS FOR HOSE APPLICATIONS"** Costin R; Nagel W Sartomer Co.Inc. (Nordic Council of Rubber Technology)

Most rubber products have traditionally been cured with sulphur-accelerator cure systems. In recent years, automotive specifications for various rubber products have changed, requiring longer product life in terms of wear and heat ageing. Sulphur cure systems suffer from embrittlement due to poor heat age resistance and, therefore, do not meet many of the higher performance requirements. Peroxide-coagent cure systems are available that do provide the improvement needed in heat ageing while maintaining the desirable sulphur cure properties of good dynamic performance, tensile strength and wear. In addition, by selecting the right coagent, excellent adhesive properties can be achieved between rubber and reinforcement without the use of external bonding agents. A review of coagents appropriate for peroxide-cured hose applications is presented. 9 refs.

USA Accession no.787048

#### Item 86

Journal of Applied Polymer Science 77, No.12, 19th Sept.2000, p.2732-9 N,N'-PENTAMETHYLENETHIURAM DISULPHIDE- AND N,N'-PENTAMETHYLENETHIURAM HEXASULPHIDE-ACCELERATED SULPHUR VULCANISATION. II. RELATIVE STABILITY OF N,N'-PENTAMETHYLENETHIURAM DISULPHIDE AND TETRAMETHYLENETHIURAM DISULPHIDE WITH RESPECT TO AMINE FORMATION Reyneke-Barnard C P; Gradwell M H S; McGill W J Port Elizabeth,University

The heat stability of N,N'-dipentamethylenethiuram disulphide was studied and compared with that of TMTD with respect to thiourea formation. The agent responsible for degradation was investigated and identified as alkyldithiocarbamic acid, which, upon decomposition, produced amine. The effectiveness of zinc compounds, such as ZnO and zinc bis(pentamethylenedithiocarbamate), in preventing degradation was also examined. 22 refs.

SOUTH AFRICA

Accession no.784354

Item 87

Indian Journal of Natural Rubber Research 11, Nos.1 & 2, 1998, p.50-7 STUDIES ON THE DEGRADATION OF NATURAL RUBBER CURED BY ACCELERATED LOW SULPHUR AND URETHANE CROSSLINKING SYSTEMS Alex R; Farid A S India,Rubber Research Institute; North London,University

Changes in molecular level network structure are investigated for NR cured by low sulphur (EV) and urethane (Novor) systems by measurement of continuous and intermittent stress relaxation. NR cured by the urethane system show more crosslinking and scission reactions at temperatures between 100 and 130 deg.C but lower crosslinking and scission reactions at temperatures between 150 and 160 deg.C compared to NR cured by low sulphur system. At temperatures between 100 and 130 deg.C, NR vulcanised by low sulphur system shows lower permanent set and at temperatures between -150 and 160 deg.C higher permanent set, as compared to NR vulcanised by the urethane system. The ageing of urethane cured NR perhaps results in the occurrence of intermolecular interactions such as hydrogen bonding. A lower value of activation energy of stress relaxation shown by urethane-cured NR indicates that the mechanism of degradation is not identical to that of low sulphur cured NR.

EUROPEAN COMMUNITY; EUROPEAN UNION; INDIA; UK; WESTERN EUROPE

## Item 88

Journal of Polymer Science: Polymer Physics Edition 38, No.11, 1st June 2000, p.1417-23 SOLID-STATE NMR STUDIES OF CIS-1,4-POLYISOPRENE CROSSLINKED WITH DICUMYL PEROXIDE IN THE PRESENCE OF TRIALLYL CYANURATE Sung Joon Oh; Koenig J L

Case Western Reserve University

Solid-state techniques such as direct-polarisation(DP), cross-polarisation(CP) and proton T2 experiments were used to study the network of dicumyl peroxide(DCP)/ triallyl cyanurate(TAC) crosslinked cis-1,4-polyisoprene. Line broadening and cis/trans isomerisation of mobile carbons were observed in the DP experiments. Rigid carbons of network structures was observed with the CP technique. Motional heterogeneity was examined by proton T2 relaxation experiments. Decreases in long T2 values from the mobile non-network structures and short T2 values from the rigid network structures were observed with an increase in peroxide or coagent concentration. The percentage of short T2 in T2 relaxation, which was related to network density, was observed to increase with peroxide and coagent addition. 28 refs. USA

Accession no.781016

*Item 89 Revue Generale des Caoutchoucs et Plastiques* 77, No.783, Feb.2000, p.46/53 French **DOSING OF N-NITROSAMINES** Khalfoune H; Aubin C; Gomez E LRCCP; IFOCA

Consideration is given to the toxicity of nitrosamines formed during rubber vulcanisation in the presence of certain accelerators, the mechanisms by which they are formed, and French, German and European Union regulations relating to nitrosamines in the workplace atmosphere and in rubber products. Methods used in the sampling and analysis of nitrosamines are also described. 6 refs.

EU; EUROPEAN COMMUNITY; EUROPEAN UNION; FRANCE; GERMANY; WESTERN EUROPE; WESTERN EUROPE-GENERAL

Accession no.780260

#### Item 90

*International Polymer Science and Technology* 27, No.4, 2000, p.T/52-T/57. (Translation of Kauchuk i Rezina, No.6, 1999, p.30)

HEXACHLORO-P-XYLENE IN VULCANISATES: EXPERIENCE AND FUTURE PROSPECTS Kandyrin K L

Lomonosov Institute of Fine Chemical Technology

Hexachloroparaxylene (HCPX) as a polyfunctional ingredient in rubber formulations is discussed, with

reference to its benefits and shortcomings, in the form of a literature review. The product is non-toxic, has good solubility in rubbers, and improves several parameters of rubber mixes and vulcanisates, being particularly effective as a component of vulcanising and modifying systems. It is currently being used in significant quantities only in tyre rubbers for the coating of polyamide cord, but if its noted shortcomings are overcome, it is argued that it could also be used for high-modulus rubbers in the mechanical rubber goods industry. 70 refs.

RUSSIA

Accession no.779709

Item 91

*International Polymer Science and Technology* 27, No.4, 2000, p.T/17-T/21. (Translation of Kauchuk i Rezina, No.6, 1999, p.2)

# VULCANISATION OF POLYCHLOROPRENE IN THE PRESENCE OF AEROSIL FINE SILICA MODIFIED WITH AMMONIUM ETHYLENE BIS(DITHIOCARBAMATE) Nosnikov A F; Ebich Y R

Ukraine, State Chemico-Technological University

The successful use of modified aerosil as the vulcanising agent for polychloroprenes has made it possible to replace known vulcanising agents such as metal oxides, and to also improve the processing properties of rubber mixes, and the service properties and mechanical properties of rubber goods based on Nairit polychloroprene rubbers of all types and grades. Aerosil was modified with ammonium ethylene bisdithiocarbamate. Crosslinking kinetics are studied. 7 refs.

RUSSIA

Accession no.779700

#### Item 92

Nippon Gomu Kyokaishi

73, No.5, 2000, p.259-66 Japanese

## STUDIES OF VULCANIZING AGENTS AND VULCANIZATION ACCELERATORS. PART VIII. VULCANISATION OF HALOGEN-CONTAINING RUBBER WITH CYCLOHEXASULPHIDE Ishida T; Kondo T; Imai S; Samejima H; Akiba M

7-n-Butylamino-7-phenyl-8-thioxo-1,2,3,4,5,6hexathiocane (BPTH) derivative obtained from psubstituted acetophenone, n-butyl or n-propylamine and sulphur was tested as a vulcanising agent for NR, CR and halogen-containing rubber. Data are given on the physical properties of the rubbers. 12 refs. Articles from this journal can be requested for translation by subscribers to the Rapra produced International Polymer Science and Technology. *Accession no.779417* 

*Item 93* Patent Number: EP 1016691 A1 20000705

# CURING OF BUTYL RUBBER WITH A PHENOLIC RESIN

Hopkins W; von Hellens C W Bayer Inc.

Butyl rubber formulations are cured with polycyclic phenol-formaldehyde resins having dimethylene ether bridges and methylene bridges, the molar ratio of dimethylene ether bridges to methylene bridges being less than about 2.5:1. They display improved heat ageing characteristics.

CANADA; EUROPEAN COMMUNITY; EUROPEAN UNION; WESTERN EUROPE-GENERAL

Accession no.778603

## Item 94

*China Rubber Industry* 47, No.6, 2000, p.331-4

Chinese

# EFFECT OF TMTD ON HEAT AND OXYGEN AGEING OF CLAY-FILLED NR COMPOUND

Yang D; Ao N; Yang S

Zhanjiang,Ocean University; South China,Tropical Agricultural Product Processing Research Institute; South China,Teachers' Training University

The effect is examined of tetramethyl thiuram disulphide (TMTD) on the heat ageing and oxidation of clay-filled NR, with reference to the plasticity retention index of NR, using thermal analysis and scanning electron microscopy test methods. The results showed that heat and oxygen resistant properties could be obtained when the clay-filled natural rubber compound was cured by semi-effective or effective curing systems, with 1.5 phr or 3.0 phr of TMTD. 3 refs.

CHINA Accession no.778522

Item 95

*Elastomery* 4, No.1, 2000, p.23-7 Polish **INVESTIGATION OF THE INFLUENCE OF STEARIC ACID ESTERS ON ELASTOMERS PROPERTIES** 

Kleps T; Rajkiewicz M; Berek I Instytut Przemyslu Gumowego Stomil Piastow

The influence of stearic acid methyl ester on crosslinking of elastomer compounds and elastomer properties has been studied. The purpose of the research is to recognise the possibility of application of stearic acid esters which have been obtained according to Polish technology from waste technical fats as the crosslinking activator of diene elastomers. 7 refs.

EASTERN EUROPE; POLAND *Accession no.777204* 

## Item 96

Kautchuk und Gummi Kunststoffe 53, No.3, March 2000, p.131-6 SOME CONTRIBUTIONS TO THE UNDERSTANDING OF THE ACCELERATING SYSTEM DURING NATURAL RUBBER VULCANISATION Borros S; Agullo N Sarria,Institut Quimic

A method based on the modelling approach has been developed for the study of NR vulcanisation using two different accelerating systems. Squalene is chosen as the model compound. The main reaction paths followed by sulphenamide (N-cyclohexylamine-2-benzothiazole, CBS) during the first stages of the process are studied. In all cases the active role of ZnO and the olefinic chain is established. 17 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; SPAIN; WESTERN EUROPE

Accession no.776693

#### Item 97

West Conshohocken, PA, 1999, pp.4. NALOAN. ASTM D 4818-. CLASSIFICATION FOR RUBBER COMPOUNDING MATERIALS -VULCANIZATION ACCELERATORS American Society for Testing & Materials

ASTM D 4818-

Version 89(R1999). Photocopies and loans of this document are not available from Rapra. USA

Accession no.774062

### Item 98

West Conshohocken, PA, 1999, pp.2. NALOAN. ASTM D 4572-. TEST METHOD FOR RUBBER CHEMICALS - WET SIEVE ANALYSIS OF SULFUR American Society for Testing & Materials

American Society for Testing & Materials ASTM D 4572-

Version 89(R1999). Photocopies and loans of this document are not available from Rapra. USA

Accession no.774058

## Item 99

Journal of Applied Polymer Science 76, No.9, 31st May 2000, p.1413-21 BENZOTHIAZOLE-ACCELERATED SULPHUR VULCANISATION. V. 2-BISBENZOTHIAZOLE-2,2'-DISULPHIDE/ZNO AND 2-BISBENZOTHIAZOLE-2,2'-DISULPHIDE/(2-MERCAPTOBENZOTHIAZOLE)ZINC(11) AS ACCELERATORS FOR 2,3-DIMETHYL-2-BUTENE

# Morgan B; McGill W J Port Elizabeth, University

The results are reported of a study of accelerated sulphur vulcanisation using the above compounds. 2,3-Dimethyl-2-butene, was utilised as a model compound for polyisoprene and residual curatives, intermediates and products produced by heating the mixes for various times at 150C in sealed tubes were analysed using highperformance liquid chromatography. A synergistic effect was observed between bis(2-mercaptobenzothiazole) zinc(II) and 2-bisbenzothiazole-2,2'-disulphide. 23 refs. SOUTH AFRICA

Accession no.772406

## Item 100

Journal of Applied Polymer Science 76, No.9, 31st May 2000, p.1377-85 BENZOTHIAZOLE-ACCELERATED SULPHUR VULCANISATION. 1. 2-MERCAPTOBENZOTHIAZOLE AS ACCELERATOR FOR 2,3-DIMETHYL-2-BUTENE Morgan B; McGill W J

Port Elizabeth, University

A study was made of 2-mercaptobenzothiazoleaccelerated sulphur vulcanisation using 2,3-dimethyl-2-butene as a model compound for polyisoprene. Highperformance chromatography was employed to analyse heated mixtures containing the curatives only and the curatives and the model compound for residual curatives, intermediates and reaction products. The data obtained indicated that the accelerator formed polysulphidic species in the presence of sulphur, which react with the model compound to form polysulphidic hydrogenterminated pendent groups, and that crosslinking occurred slowly as a result of the interaction of polythiol pendent groups. 25 refs.

SOUTH AFRICA *Accession no*.772402

#### Item 101

*International Polymer Science and Technology* 26, No.10, 1999, p. T/8-11. (Translation of Muanyag es Gumi, NO.6, 1999, p.A9) **SULPHENIMIDE VULCANISATION ACCELERATORS** Kosar K

Sulphenimide accelerators are proposed as a replacement for sulphenamide-based vulcanisation accelerators derived from secondary amines. Disadvantages of using secondary amine derivatives include health hazards associated with the production of N-nitrosomorpholine during its use. Sulphenimide are primary amine derivatives, and offer no possibility for the formation of hazardous N-nitrosoamine. Results are examined of trials which compare the effects of the two types of vulcanisation accelerators, with respect to mechanical properties, adhesion, and vulcanising properties of the rubber compounds. 2 refs.

EASTERN EUROPE; HUNGARY *Accession no.771193* 

#### Item 102

*European Rubber Journal* 182, No.5, May 2000, p.25 **NEW CURING AGENT FROM BAYER** Shaw D

A novel curing agent called Vulcuren has been developed by Bayer, which is claimed will improve quality and eventually reduce costs in the moulding of large rubber products and tyres. Vulcuren creates crosslinks in diene rubbers in which the crosslink is made of carbon rather than sulphur atoms. Benefits reported, include a reduction in reversion, and an ability to control the length of the crosslink in order to achieve the desired dynamic properties. The product is currently undergoing lab-scale trials.

#### BAYER AG

EUROPEAN COMMUNITY; EUROPEAN UNION; GERMANY; WESTERN EUROPE

Accession no.771086

#### Item 103

Rubberchem '99. Conference proceedings. Antwerp, Belgium, 22nd-23rd Nov.1999, paper 21 BALANCED NETWORK WITH SULPHENAMIDE/TETRABENZYL THIURAM DISULPHIDE (TBZTD) Datta R; Mori T Flexsys BV

(Rapra Technology Ltd.)

The major objective of the tyre industry is to improve productivity and to extend tyre life. Tyre durability requires heat resistance, antiflex properties, antioxidation and antiozonation. A proper balance of network is important for flex and heat ageing properties. The use of binary system of accelerators such as sulphenamide and commonly used thiuram such as MTD improves cure efficiency but suffers from the adverse effect of scorch and flex properties. The possibility of an N-nitrosamine safe thiuram such as tetrabenzyl thiuram disulphide (TBzTD) is explored to obtain improvement in cure efficiency and a balanced network. The effect of lower amount of TBzTD is studied in sulphenamide cure to obtain better balance of performance characteristics. A crosslink study is done to correlate the properties to the fine structure of the network.

EUROPEAN COMMUNITY; EUROPEAN UNION; NETHERLANDS; WESTERN EUROPE Accession no.769263

# *Item 104* Rubberchem '99. Conference proceedings. Antwerp, Belgium, 22nd-23rd Nov.1999, paper 20 **CURRENT THEORIES ON THE ACTION OF THIURAM DISULPHIDE ACCELERATORS**

Hendrikse K G Port Elizabeth, University (Rapra Technology Ltd.)

Thiuram (tetramethylthiuram disulphide (TMTD), tetraethylthiuram (TETD) and N,N'-pentamethylenethiuram disulphide (CPTD)) and sulphur are heated isothermally in the absence and presence of polyisoprene. In the absence of rubber, TMTD/sulphur mixes, heated at 130-150 deg.C rapidly form a series of TMTPs. The concentrations TMTPs of lower sulphur rank increase most rapidly, indicating that sulphur atoms are added to the accelerator sequentially. 2,3-Dibutyl-2-butene (TME) is used as a model for polyisoprene in studying the formation of pendent groups that act as precursors to crosslink formation during sulphur vulcanisation. TME is heated at 130 deg.C with TMTD/sulphur in sealed tubes for various times, after which the mixture is analysed by HPLC. The rate at which the TMTPs bound to the network cannot be accounted for in terms of a free radical mechanism, and a concerted mechanism is proposed. Dimethyldithiocarbamic acid (Hdmtc), a product of pendent group is shown to play an essential role in the crosslinking process and its removal from the system during vulcanisation severely impedes crosslinking. Crosslinking is shown to occur between thiuram and thiol terminated pendent groups, the latter are formed when sulphonated Hdmtc reacts with the polymer chain. TMTD does not decompose to tetramethylthiourea (TMTU) at vulcanisation temperatures as has been suggested. Instead it results from the attack of dimethylamine, released on decomposition of Hdmtc on TMTD. 34 refs.

SOUTH AFRICA

Accession no.769262

#### Item 105

Rubberchem '99. Conference proceedings. Antwerp, Belgium, 22nd-23rd Nov.1999, paper 16 **NEW DEVELOPMENTS IN CURING HALOGEN-CONTAINING POLYMERS** Ohm R F Vanderbilt R.T.,Co.Inc. (Rapra Technology Ltd.)

Di- and poly-functional mercaptans have been used for many years to crosslink polymers with labile halogens (chlorine or bromine). 2,5-Dimercapto-1,3,4-thiadiazole (DMTD) is one such crosslinker that displays relatively fast cures but poor scorch safety. Improved scorch safety can be achieved by derivatising one or both mercapto groups. Several derivatives of DMTD have been developed recently for this purpose. Proper selection of the thiadiazole derivative and associated accelerator is often a key to successful use in a given halogen-containing polymer compound. The various thiadiazoles and other additives that crosslink halogencontaining polymers are reviewed. 3 refs.

USA

Accession no.769258

## Item 106

Rubberchem '99. Conference proceedings. Antwerp, Belgium, 22nd-23rd Nov.1999, paper 15 **PEROXIDE CROSSLINKING OF SATURATED POLYMERS** Bekendam G Akzo Nobel NV (Rapra Technology Ltd.)

Peroxide crosslinking of elastomers dates back nearly eight decades. It was not until the development of fully saturated ethylene propylene copolymers in the early 1970s that technical interest grew in organic peroxides. This new interest in peroxides helped foster the development of new types of peroxides and their formulations. These peroxides overcame many of the drawbacks in thermal stability, crosslink efficiency, handling and safety at that time. In recent years new technology has been introduced which allows the production of polyolefin polymers (POE). Precise and predictable molecular control is a distinguishing feature of this technology. These new polyolefin elastomers are said to bridge the gap between thermoplastics and elastomers. They have the processing characteristics of thermoplastics and the performance of elastomers. Another group of saturated polymers, which has become widely recognised for its excellent properties, is the hydrogenated nitrile polymers (HNBR). This class of polymers in combination with peroxides opens the possibility to enter more flexible applications where heat resistance is a must. Both classes of polymers are evaluated from a peroxide point of view. First a comparison with a selection of peroxides is made in order to find out which of these is the most efficient, both for POEs and for HNBR polymers. A comparison, using the selected peroxide, is made between some of the most commonly used coagents (allylic types, methacrylate types, bismaleimide and acrylates).

EUROPEAN COMMUNITY; EUROPEAN UNION; NETHERLANDS; WESTERN EUROPE Accession no.769257

# Item 107

Rubberchem '99. Conference proceedings. Antwerp, Belgium, 22nd-23rd Nov.1999, paper 13 **SELECTING ACRYLIC TYPE COAGENTS FOR HOSE AND BELT APPLICATIONS** Costin R; Nagel W; Younger J Sartomer Co.Inc.; Cray Valley Functional Additives; Totalfina Oil,Gas & Chemicals (Rapra Technology Ltd.)

Hose and belt applications have traditionally been cured with sulphur-accelerator cure systems. In recent years, automotive hose and belt specifications have changed, requiring longer product life in terms of wear and heat ageing. Sulphur cure systems suffer from embrittlement due to poor heat age resistance. Efforts have been made to develop improved cure systems. Peroxide-coagent cure systems do provide the improvement needed in heat ageing while maintaining the desirable sulphur cure properties of good dynamic properties, tensile strength and wear. In addition, by selecting the right coagent, excellent adhesive properties can be achieved between rubber and reinforcement without the use of external bonding agents. A review of these coagents appropriate for peroxide cured hoses and belts is presented. 6 refs. EUROPEAN COMMUNITY; EUROPEAN UNION; FRANCE; WESTERN EUROPE

Accession no.769255

#### Item 108

Rubberchem '99. Conference proceedings. Antwerp, Belgium, 22nd-23rd Nov.1999, paper 12 **BETTER VULCANISATE PROPERTIES USING N-NITROSAMINE FREE CURING SYSTEMS** Schuch A Rhein Chemie Rheinau GmbH

(Rapra Technology Ltd.)

It has been shown that almost 60% of common curing agents are critical with regard to their toxicological potential and should be substituted by harmless accelerators. Generally dithioates can replace many critical curing agents. In some applications like OTOS in NR, the first steps toward this substitution are promising. The results on ageing show a good consistency of elasticity but there is still room for improvements in terms of consistency of tensile data. The substitution of MBS by dithioates in SBR results in improved dynamic behaviour and higher heat stability of the network at similar other physical properties. Even the substitution of TMTM by dithioates in coloured blends gives colour stability, faster curing speed and handling advantages. 6 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; GERMANY; WESTERN EUROPE

Accession no.769254

#### Item 109

Rubberchem '99. Conference proceedings. Antwerp, Belgium, 22nd-23rd Nov.1999, paper 10 **IMPROVED FORM OF INSOLUBLE SULPHUR** Ingham F A A; To B H Flexsys BV; Flexsys America LP (Rapra Technology Ltd.)

Insoluble sulphur is a high molecular weight polymeric form of sulphur and is insoluble in rubber. This feature provides the rubber compounder with the freedom to formulate compounds containing high levels of sulphur but with no detrimental effect on green compound tack. The use of ordinary rubber makers sulphur (RMS) in such applications would lead to sulphur bloom on the surface of uncured components due to its relatively low solubility in elastomers; around 1 phr at room temperature in natural rubber based compounds. Insoluble sulphur, however, is sometimes difficult to disperse in rubber compounds. During mixing agglomeration can occur leading to poorly dispersed material. Cool processing temperatures will aid in obtaining good dispersion. A high mixing temperature on the other hand reduces the viscosity of the compound and results in lower effective shear forces, unfavourable towards good dispersion. Additives such as oil and silica can improve dispersion significantly and even further improvements can be achieved through the use of predispersed masterbatches. However, the preparation of predispersed masterbatches and silica containing grades involves additional costs. A new grade of insoluble sulphur has been developed making use of a special additive package that leads to enhanced dispersion and reduced incorporation time. This new product provides superior flow characteristics allowing ease of handling. The special additive package also provides increased thermal stability of the insoluble sulphur, ensuring enhanced bloom resistance following mixing and processing.

EUROPEAN COMMUNITY; EUROPEAN UNION; NETHERLANDS; USA; WESTERN EUROPE Accession no.769252

#### Item 110

Journal of Applied Polymer Science 75, No.6, 7th Feb.2000, p.735-9 IMPROVEMENT OF FILLER-RUBBER INTERACTION BY THE COUPLING ACTION OF VEGETABLE OIL IN CARBON BLACK REINFORCED RUBBER Kundu P P

Sant Longowal, Institute of Engineering & Technology

Small amounts of vegetable oil added to a natural rubber/ polybutadiene blend compound during mixing has a very significant effect on both vulcanisation and physical properties. Some of the effects may be from a lower crosslink density, or plasticisation. 14 refs.

INDIA

Accession no.769014

Item 111 China Rubber Industry 47, No.2, 2000, p.90-3 Chinese APPLICATION OF CURING AGENT DL-268 TO NR/SBR COMPOUND Pang J

Double Happiness Tyre Industries Corp.Ltd.

Addition of curing agent DL-268 to a natural rubber styrene butadiene rubber blend was investigated. Reversion and thermooxidatively aged physical properties were examined. Adhesion between the compound and tyre cord was found to be improved, but fatigue heat build-up was decreased.

CHINA

Accession no.768048

## Item 112

**Polymers for Advanced Technologies** 11, No.1, Jan.2000, p.1-8 **EVALUATION OF THE PROPERTIES OF SOME NITRILE-BUTADIENE RUBBER/ POLYCHLOROPRENE MIXES AND VULCANIZATES** 

Abdel-Bary E M; von Soden W; Helaly F M Mansoura, University; Ulm, University; Egypt, National Research Centre

Nitrile rubber was blended with polychloroprene(CR) in a weight ratio of 1:1. The vulcanising systems in the blend formulations were varied to obtain non-crosslinked CR embedded in vulcanised nitrile rubber and noncrosslinked nitrile rubber embedded in vulcanised CR. The effects of these two different phases on the rheological and mechanical characteristics were evaluated. The dynamic compliance of the blends was also measured over wide ranges of frequency and temp. It was found that the mechanical and rheological properties of the vulcanised blends depended on the type of vulcanising system, its concentration and the presence of reinforcing filler. The mechanical properties of the blend containing Ncyclohexyl-2-benzthiazyl sulphenamide/sulphur as vulcanising system suitable for nitrile rubber were higher than those of the blend containing non-sulphur vulcanising system (zinc oxide/magnesium oxide and ethylene thiourea) suitable for CR. The rubbers in the blend were incompatible as two Tgs were observed. 14 refs. EGYPT; EUROPEAN COMMUNITY; EUROPEAN UNION; GERMANY; WESTERN EUROPE

Accession no.766102

#### Item 113

**Polymer International** 49, No.1, Jan.2000, p.115-21 **VULCANIZATION OF CARBOXYLATED NITRILE RUBBER(XNBR) BY A MIXED ZINC PEROXIDE-SULPHUR SYSTEM** 

Ibarra L; Alzorriz M Instituto de Ciencia y Tecnologia de Polimeros

Vulcanisation of XNBR with a mixed system based on zinc peroxide and sulphur accelerators was shown to produce materials with favourable mechanical properties because of the formation of ionic aggregates which gave the vulcanised compounds a certain rigidity. These properties were drastically reduced by the effect of saturated ammonia vapour which plasticised the ionic aggregates. This plasticisation, however, was reversible and the aggregates could be regenerated by addition of a solvent, which resulted in recovery and even improvement of the original properties such as stress at constant deformation, TS, crosslink density and storage modulus. The temps. of the two transitions observed in the relaxation spectra, the Tg of the polymer and the ionic transition corresponding to ionic aggregates, were displaced to higher values when ionic structures were regenerated. 7 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; SPAIN; WESTERN EUROPE

Accession no.766016

#### Item 114

Rubber Chemistry and Technology 72, No.2, May-June 1999, p.334-42 STUDIES OF PEROXIDE CURING OF CIS-1,4-POLYISOPRENE/DIALLYL PHTHALATE BLENDS BY SPECTROSCOPIC TECHNIQUES Sung Joon Oh; Koenig J L Case Western Reserve University,Dept.of

Macromolecular Science

Differential scanning calorimetry (DSC), solid state nuclear magnetic resonance (NMR), Fourier transform infrared spectroscopy (FTIR) and equilibrium swelling techniques were used to study the peroxide curing of cis-1,4-polyisoprene with addition levels of coagent diallyl phthalate from 0 to 20 percent by weight. It is postulated that, although reaction kinetics are controlled by rate of peroxide decomposition, the coagent catalyses the curing reactions and undergoes both copolymerisation and homopolymerisation during the reaction. Chemically bound coagent domains, which are phase separated are suggested to cause a decrease in segmental mobility with increase in crosslink density. 20 refs

USA

Accession no.763828

#### Item 115

**Rubber Chemistry and Technology** 72, No.2, May-June 1999, p.318-33 **INSIGHTS INTO SULFUR VULCANISATION FROM QSPR QUANTITATIVE STRUCTURE-PROPERTY RELATIONSHIPS STUDIES** 

Ignatz-Hoover F; Katritzky A R; Lobanov V S; Karelson M

Flexsys America; Florida, University; Tartu, University

Studies on the vulcanisation of a black and oil filled styrenebutadiene rubber compound accelerated by a number of different sulphenamide and sulphenimide compounds were made using a conventional curemeter operated to normal ASTM standards. The vulcanisation reactions were also studied using different modelling software, CODESSA software for deriving quantitative structure-property relationships and MOPAC software for semiempirical molecular orbital calculations which together yielded excellent correlation to onset of cure and maximum cure rate. The Quantity Structure-Activity Relationships indicated support for previously proposed reaction mechanisms, but a new structure for the sulphurating intermediate is indicated and proposed 62 refs

ESTONIA; USA

Accession no.763827

Item 116

**Rubber Technology International** 1999, p.62-5 **ACCELERATING THE CURE PROCESS** Dzikowicz R T Vanderbilt R.T.,Co.Inc.

The SETSIT line of ultra accelerators is described. It includes proprietary water-soluble, water-miscible activated dithiocarbamates which are easy to use and freeze-thaw stable. Three new products are announced in the range of liquid accelerators for latex, and characteristics of the 6 available SETSIT grades are described, with property and performance data. All grades can be rapidly incorporated into latex without undue mechanical shear. By eliminating the accelerator from the masterbatch dispersion, a greater flexibility is achieved for adjusting the level of ingredients, and consequently larger dispersion batches are possible. Further advantages of their use are discussed. 3 refs.

USA

Accession no.763109

#### Item 117

Polymer Degradation and Stability 67, No.2, Feb.2000, p.319-23 COMPARISON OF THERMAL STABILITY OF SULPHUR, PEROXIDE AND RADIATION CURED NBR AND SBR VULCANISATES Ahmed S; Basfar A A; Abdel Aziz M M

Saudi Arabia, Institute of Atomic Energy Research

The merits of radiation curing with gamma rays over the conventional processes of sulphur and peroxide curing were evaluated by comparing the heat stability of sulphur- and peroxide-cured NBR and SBR with their radiation cured counterparts. The vulcanisates were obtained by blending the elastomers with fillers, antioxidants and accelerators, followed by vulcanisation at 150 to 160C. Blends of the same elastomers with appropriate coagent types were also cured by gamma radiation and the influence of the variation of the amount of coagent and other additives on the heat stability of the formulations assessed. The radiation cured samples exhibited better heat stability than the sulphur-and peroxide-cured samples. 11 refs.

SAUDI ARABIA

Accession no.760992

#### Item 118

Hazards in the European Rubber Industry. Conference proceedings. Manchester, 28th-29th Sept.1999, paper 17

#### NITROSAMINE CONTROL AT SOURCE

Scott K; Willoughby B G Rapra Technology Ltd.

Some N-nitrosamines have been classified as possible human carcinogens by the International Agency for Research on Cancer and are recognised potential byproducts of rubber vulcanisation. For this reason the rubber industry has faced increasingly stringent requirements for the control of N-nitrosamines. Much work has been carried out on the possibilities of reformulation; however the factors influencing Nnitrosamine formation have remained obscure. Nnitrosamine-free formulations are now in use, but for many rubber compounders there are no easy options for the replacement of basic ingredients such as carbon black and tetramethylthiuram disulphide (TMTD). This has led to a study with the primary objective of investigating the key influences controlling N-nitrosamine formation when these two components are present in a rubber vulcanisate. Some of the published information about N-nitrosamine formation in rubber vulcanisates and its prevention is reviewed. The findings of a Rapra study on nitrosamine formation in TMTD/carbon black-containing vulcanisates are reviewed. 49 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; UK; WESTERN EUROPE Accession no.760322

Item 119

Kautchuk und Gummi Kunststoffe 52, No.11, Nov.1999, p.758-62 HIGH TEMPERATURE CURING OF PASSENGER TYRES BY USING 1,3 BIS-(CITRACONIMIDOMETHYL)BENZENE Datta R N; Ingham F A A Flexsys BV

The effect of the above compound (Perkalink 900), as an anti-reversion agent, in a passenger tyre tread based on SBR/BR blends was investigated with the aim of increasing cure temp. such that cure times can be reduced, thus improving productivity. It was found that this compound reacted by scavenging dienes and trienes in the reverted network, thereby compensating for a reduced level of sulphidic crosslinks observed at increased cure temps. It did not affect compound cure characteristics or compound viscosity. 15 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; NETHERLANDS; WESTERN EUROPE

Accession no.759951

#### Item 120

Macromolecules 32, No.22, 2nd Nov.1999, p.7504-8 VULCANIZATION OF BUTADIENE RUBBER BY MEANS OF CYCLIC DISULFIDES. I. A 2D NMR STUDY ON THE CROSS-LINK STRUCTURE OF A BR MODEL COMPOUND VULCANIZATE Seyger R M; Hulst R; van Duynhoven J P M; Winters R; van der Does L; Noordermeer J W M; Bantjes A Twente, University; Unilever Research

The vulcanisation of cis,cis,cis-1,5,9-cyclodecatriene (as a model compound for true polybutadiene vulcanisates) by means of 1-oxa-4,5-dithiacycloheptane, zinc dimethyl dithiocarbamate and 1,12-diaminododecane was studied using several one-dimensional and two-dimensional NMR techniques in order to determine crosslinking sequence and overall molecular structure. As a result of the relatively high solubility of the products, typical solution 2D NMR pulse sequences could be applied, offering a framework of reference for the subsequent solid state NMR studies on true vulcanisates. 20 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; NETHERLANDS; WESTERN EUROPE

Accession no.759204

Item 121

156th ACS Rubber Division Meeting - Fall 1999. Conference preprints. Orlando, Fl., 21st-23rd Sept.1999, paper 38 **NOVOR 950: THE HIGH PERFORMANCE CROSSLINKER FOR THE 21ST CENTURY** Metherell C Rubber Consultants (ACS,Rubber Div.)

Novor 950 is an isocyanate crosslinker for NR imparting a combination of physical properties which cannot be achieved by other means. The mechanism of reaction of Novor 950 with rubber is reviewed, and it is shown how reversion resistant formulations can be obtained. A cost effective series of mixed sulphur/Novor 950 systems of high performance can be obtained which possess fast curing characteristics, coupled with high reversion resistance. One of the most interesting features of most Novor 950 cure systems is the two- or three-fold increase of fatigue life on ageing. A feature of Novor 950 vulcanisates is that it is almost impossible to detect the type of crosslinking system by chemical means, hence Novor 950 is an 'invisible crosslinking system'. A competitor is most unlikely to be able to reconstruct the cure system by reverse engineering of a product containing Novor 950. Novor 950 has become established in the European automotive field where the advantages of improved heat and flex fatigue resistance are much sought after due to increasing under-the-bonnet temperatures. 3 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; UK; WESTERN EUROPE

Accession no.758335

# Item 122

156th ACS Rubber Division Meeting - Fall 1999. Conference preprints. Orlando, Fl., 21st-23rd Sept.1999, paper 37

# NEW BIFUNCTIONAL CROSSLINKER FOR DIENE RUBBER

Jeske W; Buding H; Weidenhaupt H-J Bayer AG

(ACS,Rubber Div.)

The reduction or elimination of reversion in vulcanisates, particularly NR vulcanisates, by the incorporation of specific additives or crosslinkers is a general demand of the rubber processing industry. Reversion is the anaerobic ageing of a polysulphidic rubber network which takes place not only at high vulcanising temperatures and during long vulcanisation times, but also in use if the vulcanisate is subject to dynamic stress. Reversion reduces the crosslink density of the vulcanisate that causes a deterioration in the mechanical and dynamic vulcanisate properties. The performance of 1,6-bis(N,N'-dibenzylthiocarbamoyldithio)-hexane is investigated as a bifunctional crosslinker for truck tyre compounds in comparison with conventional and semiefficient vulcanising systems. The different technological profiles of the vulcanisates obtained are discussed in terms of static and dynamic properties before and after ageing. It is found that vulcanisates with 1,6-bis(N,N'-dibenzylthiocarbamoyldithio)-hexane as the crosslinker have excellent reversion and heat stability with no deterioration in the dynamic properties. 3 refs. EUROPEAN COMMUNITY; EUROPEAN UNION; GERMANY; WESTERN EUROPE

Accession no.758334

#### Item 123

156th ACS Rubber Division Meeting - Fall 1999. Conference preprints.

# Orlando, Fl., 21st-23rd Sept.1999, paper 28 ANTIDEGRADANT SYSTEM DEVELOPMENT FOR EPDM PRODUCED VIA CONSTRAINED GEOMETRY CATALYST TECHNOLOGY

Buckley T M; Vara R G; Ohm R F DuPont Dow Elastomers LLC; Vanderbilt R.T.,Co.Inc. (ACS,Rubber Div.)

The components of a sulphur cure system are examined to optimise compression set and high temperature ageing for sealing applications. The trends observed are extended by eliminating elemental sulphur and 2mercaptobenzothiazole (MBT), in order to further improve compression set and high temperature ageing. In addition, examples of various types of antidegradants are screened, including carbon radical traps, alkoxy radical traps, peroxide decomposers and antioxidant synergists, to determine their effect on compression set and high temperature ageing. 7 refs.

USA

Accession no.758326

Item 124

156th ACS Rubber Division Meeting - Fall 1999. Conference preprints.

## Orlando, Fl., 21st-23rd Sept.1999, paper 26 EFFECT OF CURE SYSTEM ON NATURAL RUBBER BONDING

Halladay J R; Krakowski F J Lord Corp. (ACS,Rubber Div.)

It is perceived within the rubber industry that the choice of cure system may have an influence on the ease of bonding of general-purpose elastomers. The effect on bonding of varying sulphur level combined with accelerator level and type in a NR formulation is investigated using designed experiments. Bond integrity is evaluated using ASTM D 429 Method B and buffer specimens (proposed Method F) with various commercial solvent based and aqueous adhesives. The results contradict some conventional wisdom with respect to bonding. 13 refs.

USA

Accession no.758324

#### Item 125

156th ACS Rubber Division Meeting - Fall 1999. Conference preprints. Orlando, Fl., 21st-23rd Sept.1999, paper 21 ACTION OF N-(CYCLOHEXYLTHIO)PHTHALIMIDE AS A PREVULCANISATION INHIBITOR OF 2-BISBENZOTHIAZOLE-2,2'-DISULPHIDE ACCELERATED SULPHUR VULCANISATION Gradwell M H S; Stephenson N R Port Elizabeth,University (ACS,Rubber Div.)

Prevulcanisation inhibitors (PVI) provide an important delay in the curing reactions that occur in accelerated sulphur vulcanisation. The reactions of Ncyclohexylthiophthalimide (CTP) are investigated in the presence and absence of rubber. The CTP is shown to react very readily with 2-mercaptobenzothiazole (MBT) and this reaction has previously been credited with the delay observed. However, recent studies have shown that MBT is not formed until crosslinking has started. In a 2bisbenzothiazole-2,2'-disulphide (MBTS) accelerated sulphur cure, CTP is shown not to delay the onset of crosslinking, but once crosslinking begins, with the concurrent formation of MBT, the CTP reacts with the MBT inhibiting further polysulphide formation, pendent group formation and thus crosslinking. Crosslinking continues after the CTP has been consumed. 25 refs.

SOUTH AFRICA

Accession no.758319

#### Item 126

**Polymers & Polymer Composites** 7, No.6, 1999, p.431 **PROPERTIES AND STRUCTURES OF PEROXIDE-CURED NBR CONTAINING MAGNESIUM METHACRYLATE**  Xingheng Yuan; Zongling Peng; Yong Zhang; Yinxi Zhang

Shanghai, Jiao Tong University

The mechanical properties and cross-linked network structure of a peroxide-cured nitrile rubber containing magnesium methacrylate are investigated and discussed. Some typical properties of the material are compared with those of carbon black reinforced nitrile rubber. 8 refs. CHINA

Accession no.758187

Item 127

Rubber World 221, No.3, Dec.1999, p.18/69 EFFECT OF CURE SYSTEM ON NR BONDING Halladay J R; Krakowski F J Lord Corp.

Much literature has been published on bonding rubber to metal and testing rubber to metal bonds. This article continues the bond investigations by concentrating on the influence of sulphur and accelerator choices in natural rubber. To determine the effect of sulphur, two levels were chosen. Four different accelerators, and two solvent-based and two aqueous adhesive systems were chosen. The two test methods used for comparison were ASTM D 429 Method B and the proposed Method F buffer specimens. 13 refs. USA

Accession no.758078

#### Item 128

Applied Spectroscopy 74, No.11, 9th Dec.1999, p.2756-63 STUDIES ON DYNAMIC AND STATIC CROSSLINKING OF ETHYLENE VINYL ACETATE AND ETHYLENE PROPYLENE DIENE TERCOPOLYMER BLENDS Mishra S; Baweja B; Chandra R North Maharashtra,University; Delhi,College of Engineering

Both mechanical (torque rheometer) and thermal (differential scanning calorimetry) tests were used to study the effect of the blend ratio of ethylene vinyl acetate (EVA) and ethylene propylene diene terpolymer (EPDM) on peroxide vulcanisation. Static cure in the calorimeter was noted to be much slower than dynamic cure using the torque rheometer. Increasing the EVA/EPDM ratio reduces optimum cure time and energy consumption during cure, indicating increased rate of cure. Increasing the EVA content was found to decrease the activation energy. 19 refs.

INDIA

Accession no.756017

#### Item 129

Innovations in Rubber. Conference proceedings. Shawbury, 14th May 1999, paper 3

## METHACRYLATE COAGENTS REVISE THE PERFORMANCE OF PEROXIDE CURED ELASTOMERS

Younger J Cray Valley SA (Rapra Technology Ltd.)

Recent developments in the use of a range of methacrylate coagents to modify the performance of peroxide vulcanised elastomers are summarised. Peroxide vulcanisation has successfully been used in the past to improve heat resistance, but there have been a number of adverse comparisons with sulphur vulcanisation, most notably in tear strength and dynamic performance. The current status is reported, as is the improvement of the adhesion of many elastomers to a range of metals and fibres.

EUROPEAN COMMUNITY; EUROPEAN UNION; FRANCE; WESTERN EUROPE

Accession no.755788

#### Item 130

Tyretech '99. Conference proceedings. Prague, 27th-28th Sept.1999, paper 13 USE OF VARIOUS ACCELERATORS AND ANTIDEGRADANTS IN RADIAL TYRE COMPONENTS FOR IMPROVED PERFORMANCE Hong S W

Uniroyal Chemical Co.Inc. (Rapra Technology Ltd.; European Rubber Journal)

Pneumatic passenger radial tyres usually contain a variety of rubber compositions, each designed for a specific function requiring different physical properties. The method of construction for a pneumatic tyre also varies between tyre companies. Usually, the simplest construction using the minimum number of components is the most favourable approach. However, durability, handling, riding, dry/wet traction, rolling resistance, flex fatigue resistance and speed rated performance, force tyre companies to develop more sophisticated constructed tyres with various antidegradants to improve performance. Internal tyre components such as apex, carcass, wire breaker, cushion, base tread and bead compounds are protected by antioxidants and should be cured faster than the outside components such as the sidewall, chafer and cap tread compounds, which are protected by antiozonants and also should be vulcanised without severe reversion due to over curing. Tyre durability is directly related to the antidegradants and the stable crosslink system. Therefore, it is important to select the proper antidegradants and accelerators for the optimum performance for each tyre component. The correct solution and/or amount of antidegradants and accelerators to improve the performance of tyres are described. Longer lasting tyres and lower production cost of tyres are the primary goals of the tyre producers. 10 refs.

USA

Accession no.755782

#### Item 131

Tyretech '99. Conference proceedings. Prague, 27th-28th Sept.1999, paper 5 NEW CURING SYSTEMS FOR THE TYRE INDUSTRY

Schuch A; Fruh T Rhein Chemie Rheinau GmbH (Rapra Technology Ltd.; European Rubber Journal)

For technical and environmental reasons, the tyre industry is interested in finding substitutes for hazardous materials. In the field of accelerators, dithioates could replace many critical curing agents. In SBR it has been shown that dithioates do not simply substitute MBS, they also improve heat build-up and provide higher network stability. The results were related to literature and a recent patent. The possible technical background of the patent claim is explained by a simplified model. The advantages of dithioates, like reversion resistance and reduced heat build-up, are even more obvious in NR than in SBR and indicate improvements in thick and/or dynamically loaded tyre elements and their production. 10 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; GERMANY; WESTERN EUROPE

Accession no.755775

#### Item 132

*Industria della Gomma* 43, No.8, Oct.1999, p.17-20 Italian **POLYFUNCTIONAL PRODUCT WITH PERMANENT ANTIOXIDANT ACTION** Datta R; Ingham F; Mignanelli E Flexsys BV

An examination is made of the antioxidant and antiozonant effects of Q-Flex QDI, a quinone diimine produced by Flexsys, in sulphur vulcanised unsaturated rubbers. It is also shown that this additive acts as a scorch retarder and viscosity modifier in NR compounds, and that it modifies the viscoelastic properties of NR compounds resulting in reduced rolling resistance of tyre treads. 5 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; NETHERLANDS; WESTERN EUROPE Accession no.754684

#### Item 133

Rubber Chemistry and Technology 72, No.1, March-April 1999, p.65-73 2-T-BUTYLBENZOTHIAZOLE SULFENAMIDE ACCELERATED SULFUR VULCANISATION OF POLYISOPRENE

Gradwell M H S; van der Merwe M J Port Elizabeth,University

The crosslinking reactions of TBBS (tert-butyl benzothiazyl sulphenamide) with polyisoprene were studied using differential scanning calorimetry for TBBS

and polyisoprene alone, with the addition of zinc oxide, with the addition of sulphur and with the addition of both sulphur and zinc oxide. With TBBS alone crosslinking occurred at a very high temperature. With TBBS and ZnO, crosslinking followed a similar method but at a slightly lower temperature and resulted in higher crosslink densities With TBBS and sulphur, and with the addition of ZnO crosslinking started at a much lower temperature. Formation of MBT and MBTS during the reaction was noted in each case, but the reaction mechanisms proposed indicate that the delayed action of TBBS is due to benzothiazole terminated pendant groups rather than the formation of the butylamine salt of MBT as postulated by others.

SOUTH AFRICA Accession no.753123

Item 134

## **Rubber Chemistry and Technology** 72, No.1, March-April 1999, p.27-42 **HOMOGENEOUS ZINC(II) CATALYSTS IN ACCELERATED VULCANIZATION III. DEGRADATION MODES OF MONO- AND DI-SULFIDIC CROSS-LINKS**

Nieuwenhuizen P J; van Veen J M; Haasnoot J G; Reeduk J

Leiden, Institute of Chemistry; Leiden, University

Reversion mechanisms in diene rubbers were examined by studying the reaction products from degradation of mono and disulphidic crosslinks in model compounds. No evidence was found to support the theory that these crosslinks degrade to form cyclic disulphides and zinc sulphide. Studies were based on the model compound vulcanisation of dimethylbutene which allows two isomeric crosslink attachments. Degradation of monosulphidic crosslinks resulted in olefins and a 1,3dithiin which was postulated to result from dimerisation via a Diels-Alder reaction of unstable unsaturated thioaldehydes. Degradation of disulphidic crosslinks were shown to be catalysed by zinc diethyl dithiocarbamate (ZDEC) (and presumably other zinc complexes) via a previously unpostulated mechanism to result in a 1,2dithia-3-cyclopentene. Attempts to reduce the degradation reactions by introducing nitrogen containing ligands (to complex with the zinc dithiocarbamate), dienophiles (to scavenge dienes and trienes formed during vulcanisation reaction) or dipolarophiles (to react with 1,3 dipolar species) did not prove successful. Typical vulcanisation temperatures of 140 degrees C were used for this work rather than higher temperatures commonly used previously. 35 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; NETHERLANDS; WESTERN EUROPE Accession no.753121

Item 135 **Rubber Chemistry and Technology** 

#### 72, No.1, March-April 1999, p.15-26 HOMOGENEOUS ZINC(II) CATALYSTS IN ACCELERATED VULCANIZATION II. (POLY)OLEFIN OXIDATION, DEHYDRATION, AND REACTION WITH ANTI-REVERSION COAGENTS

Nieuwenhuizen P J; Haasnoot J G; Reeduk J Leiden, University

Reversion mechanisms in diene rubbers were studied. Using model 1,5-polyolefins in the presence of dioxygen at temperatures of 140 degree C and above, and via allylic alcohols or hydroperoxides which dehydrate when catalyzed by ZDMC bis(dimethyldithiocarbamato) zinc(II) or ZMBT bis(mercaptobenzothiazolato)zinc(II), a new route for the formation of dienes and trienes has been indicated. A 1,2-dehydration route has been observed exclusively in this process with no evidence of 1,4dehydration. A complete reaction between squalene and NBCI (N-benzylcitraconimide, a model compound for the anti-reversion agent BCIMB (1,3-bis(citraconimidomethyl)benzene) has been observed in the absence of vulcanisation chemicals at a temperature of 170 degrees C which appears to follow the same mechanism. From the evidence of these two reactions it would appear that oxidation of the rubber backbone followed by dehydration may account for the presence in vulcanised rubber of dienes and trienes. This is in contrast to accepted belief that the presence of dienes and trienes in a cured rubber is attributed only to reactions involving the breaking of crosslinks. 32 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; NETHERLANDS; WESTERN EUROPE Accession no.753120

Item 136

## Journal of Polymer Engineering 19, No.1, 1999, p.13-26 EFFECT OF OLIGOAMIDE PHOSPHATE AND ITS AMMONIUM DERIVATIVE IN DIBENZOTHIAZOLYL DISULPHIDE-ACCELERATED NATURAL RUBBER COMPOUNDS Vladkova T G

Sofia, University of Chemical Technology & Metallurgy

The modifying and accelerating effects of oligoamidephosphate and its ammonium derivative on the properties of dibenzothiazolyldisulphide-accelerated sulphur-vulcanised natural rubber was investigated. Both materials enhanced the fatigue resistance of the rubber, the number of cycles to failure being increased by at least one order of magnitude. The ammonium derivative also provided an additional accelerated effect, being most effective when added in the second preparation stage. 11 refs.

BULGARIA; EASTERN EUROPE

#### Item 137 Kautchuk und Gummi Kunststoffe 52, No.9, Sept.1999, p.576/85 NEW BINARY ACCELERATOR SYSTEM FOR SULPHUR VULCANIZATION OF NATURAL RUBBER

Aprem A S; Mathew G; Joseph K; Thomas S

A new binary accelerator system uses 1-phenyl-2-4dithiobiuret (DTB) as a secondary accelerator for 2-(4morpholinothio)-benzothiazole (MBS) for the sulphur vulcanisation of natural rubber. Particular reference is made to the processing characteristics, mechanical properties and swelling behaviour. It was found that that irrespective of the concentration of DTB, all the cure reactions followed first order kinetics. Mechanical properties of the system were analysed for different DTB loading, and the strain crystallising nature of NR was found to be not affected by DTB. Based on processing characteristics, mechanical properties and swelling behaviour, the optimum dosage of DTB was found out. 24 refs.

INDIA

Accession no.751767

#### Item 138

Journal of Applied Polymer Science 74, No.6, 7th Nov.1999, p.1371-9 ROLE OF DIMETHYLDITHIOCARBAMIC ACID IN ACCELERATED SULFUR VULCANIZATION Shelver S R; Shumane M; Gradwell M H S; McGill W J Port Elizabeth,University

The vulcanisation of polyisoprene/tetramethylthiuram disulphide (TMTD)/sulphur compounds was carried out under a variety of conditions. The dimethyldithiocarbamic acid (Hdmtc) liberated during the reaction released dimethylamide which attacked the TMTD forming tetramethylthiourea (TMTU). Hdmtc was shown to play a vital role in the crosslinking process in polyisoprene/ TMTD/sulphur formulations and its removal from the system during vulcanisation severely retarded crosslinking. The importance of the thio pendent groups and polysulphidic thiuram groups is discussed and their role in the mechanism examined. Data to support the proposed reaction mechanism is given. Hdmtc accelerated sulphur vulcanisation and acted as a catalyst for the reaction, by its regeneration during the crosslinking process.

SOUTH AFRICA *Accession no.751564* 

Item 139

**Polymer International** 48, No.9, Sept.1999, p.855-60 **EFFECT OF SOME CURATIVES ON THE PROPERTIES OF ETHYLENE PROPYLENE DIENE RUBBER/POLYETHYLENE BLENDS**  Abdel-Bary E M; von Soden W; Pechhold W Mansoura, University; Ulm, University

A series of EPDM/PE blends was prepared containing different weight fractions of PE up to 0.66. The blends were vulcanised with a sulphur system, N-cyclohexyl-2benzthiazole sulphenamide/sulphur(CBS/S), and a nonsulphur system, dicumyl peroxide. The concentration of the latter was changed from 1.5 phr to 6 phr calculated on the total weight of the blend composition. The maximum torque obtained from rheographs for blends vulcanised with the CBS/S system decreased markedly with increasing PE concentration in comparison with those vulcanised with peroxide. The elastic modulus obtained from the stress-strain diagrams at 110C demonstrated the role played by the crosslinking of PE, the modulus for blends vulcanised by peroxide being higher than for samples vulcanised with CBS/S. The values of elastic modulus of both samples were, however, practically the same at room temp. and reached more than 40 MPa, depending on composition. The TS at room temp. strongly increased with increasing weight fraction of PE. It was also confirmed that the m.p. of the crystalline phase of PE decreased with increasing crosslink density of PE. The shear modulus obtained from dynamic mechanical measurements was in agreement with that obtained from static mechanical measurements. 12 refs.

EGYPT; EUROPEAN COMMUNITY; EUROPEAN UNION; GERMANY; WESTERN EUROPE

Accession no.751066

#### Item 140

155th ACS Rubber Division Meeting, Spring 1999. Conference Preprints.

Chicago, Il., 13th-16th April 1999, Paper 69, pp.36 OPTIMIZATION OF THE PRODUCTION OF EPDM SPONGE RUBBER SEALS FOR THE AUTOMOTIVE INDUSTRY

Krusche A; Haberstroh E IKV

(ACS,Rubber Div.)

A study was made of relationships between compound rheological properties, microwave vulcanisation parameters and accelerators on the quality of extruded EPDM foam seals for the automotive industry. The influence of these factors on cell size and structure, density and mechanical properties was investigated. Correlations were found between the chemical composition of the compound, variations in processing parameters and the quality of the finished product. 12 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; GERMANY; USA; WESTERN EUROPE

Accession no.749881

*Item 141* 155th ACS Rubber Division Meeting, Spring 1999. Conference Preprints.

# Chicago, Il., 13th-16th April 1999, Paper 63, pp.25 DIFFERENCES AMONG THIURAM ACCELERATORS THAT CAN AFFECT TIRE DURABILITY

Chasar D W Goodrich B.F.,Co. (ACS,Rubber Div.)

Two thiuram accelerators developed by Goodrich, Curerite IBM tetraisobutylthiuram monosulphide and Curerite IBT tetraisobutylthiuram disulphide, were evaluated along with other commercial thiurams as co-accelerators for benzothiazole sulphenamides in the vulcanisation of NR and SBR/polybutadiene tyre compounds. The effects of these thiurams on scorch safety and cure rates during vulcanisation and on the reversion resistance of vulcanisates were investigated, and differences in the types of sulphur crosslinks developed by the various thiurams were examined. 6 refs.

USA

Accession no.749876

#### Item 142

155th ACS Rubber Division Meeting, Spring 1999. Conference Preprints. Chicago, Il., 13th-16th April 1999, Paper 53, pp.16 **SELECTING ACRYLIC TYPE COAGENTS FOR HOSE AND BELT APPLICATIONS** Costin R; Nagel W Sartomer Co.Inc. (ACS,Rubber Div.)

Sartomer's Saret zinc diacrylate and zinc dimethacrylate coagents were evaluated in the peroxide vulcanisation of EPDM automotive belt and hose compounds. Cure characteristics and effects on mechanical properties, heat and water resistance and adhesion of EPDM to metal and synthetic fibre reinforcements and to other rubbers were investigated. The results of this study and other experiments with Saret coagents in rubber formulations are discussed. 6 refs.

USA

Accession no.749866

#### Item 143

155th ACS Rubber Division Meeting, Spring 1999. Conference Preprints.

Chicago, Il., 13th-16th April 1999, Paper 52, pp.36 CURE SYSTEMS AND ANTIDEGRADANT PACKAGES FOR HOSE AND BELT POLYMERS Ohm R F; Callais P A; Palys L H

Vanderbilt R.T.,Co.Inc.; Elf Atochem North America Inc.

## (ACS,Rubber Div.)

Types of rubbers used in the manufacture of hoses and belts for the automotive industry are reviewed, and the improvement of heat resistance, flexural fatigue and other mechanical properties in these applications through the selection of curing systems and antioxidants is discussed. 26 refs. USA

Accession no.749865

#### Item 144

155th ACS Rubber Division Meeting, Spring 1999. Conference Preprints. Chicago, II., 13th-16th April 1999, Paper 16, pp.53 **STUDY OF EPDM/SBR BLENDS** Zhao J; Ghebremeskel G; Peasely J Ameripol Synpol Corp. (ACS,Rubber Div.)

The influence of mixing techniques, sulphur, peroxide and coagent curing systems, carbon black levels and oil content on the performance of EPDM/SBR blends was investigated. The mechanical properties, heat ageing and ozone resistance of the blends were studied in comparison with 100% EPDM compounds. 15 refs. USA

Accession no.749833

#### Item 145

#### Macromolecular Chemistry & Physics 200, No.8, Aug.1999, p.1965-74 PHOTOCROSSLINKING OF FUNCTIONALIZED RUBBERS, VIII. THE THIOL-POLYBUTADIENE SYSTEM

Decker C; Nguyen Thi Viet T Haute-Alsace,Universite

The light-induced crosslinking of styrene-b-butadiene-bstyrene block copolymer, using a trifunctional thiol and phosphine oxide photoinitiator, was studied using infrared spectroscopy, and solubility and hardness measurements. Under intense illumination in air at ambient temperature, the curing proceeded extensively within less than 1 s. The relative reactivity of the thiyl radicals towards the polybutadiene vinyl and butene double bonds, and the competition between homo- and copolymerisation for the propagating alkyl radical, were determined. The crosslinking process was little affected by an increase of the SBS vinyl content from 8 to 59%, as it mainly enhanced intramolecular reactions. It was concluded that the thiol/ene polymerisation was much more effective in crosslinking thermoplastic SBS elastomers than was the copolymerisation of the polybutadiene double bonds with a diacrylate monomer. 16 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; FRANCE; WESTERN EUROPE

Accession no.749667

*Item 146 Industria della Gomma* 43, No.4, May 1999, p.39-44

#### Italian

# ORGANIC PEROXIDES AS AN ALTERNATIVE TO VULCANISATION WITH SULPHUR

The advantages and limitations of sulphur and peroxide vulcanisation are discussed. The Luperox range of organic peroxides produced by Elf Atochem and their applications in rubber vulcanisation and as crosslinking agents for polyolefins are reviewed. A number of commercial developments by the Company in this field are examined, and the activities of its plant in Gunzburg in Germany are described.

ELF ATOCHEM SA; ELF ATOCHEM DEUTSCHLAND GMBH EUROPEAN COMMUNITY; EUROPEAN UNION; FRANCE; GERMANY; WESTERN EUROPE; WORLD

Accession no.745792

#### Item 147

Rubber Chemistry and Technology 71, No.5, Nov.-Dec.1998, p.1073-86 COMPARATIVE STUDIES ON THE CROSSLINKING REACTION OF BIS-MALEIMIDES AND BIS-CITRACONIMIDES IN SQUALENE AND NATURAL RUBBER Datta R N; Talma A G; Schotman A H M Flexsys BV; Akzo Nobel Central Research

A study was conducted to differentiate between the crosslinking reactions of bismaleimides and biscitraconimides in squalene as well as in NR. Bismaleimides were found to participate in the crosslinking reaction in the absence of curing compounds such as accelerators and sulphur. Biscitraconimides took part in the crosslinking reaction when polysulphidic crosslinks were under the process of degradation. Sulphur and accelerator were required for the formation of polysulphidic crosslinks and hence the presence of sulphur and accelerator were essential for biscitraconimide crosslinking. The differences in the reactivity and the chemistry of crosslinking are studied and discussed. Some application data are provided in order to elaborate on the differences. 17 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; NETHERLANDS; WESTERN EUROPE Accession no.744902

# Item 148

Journal of Applied Polymer Science 73, No.8, 22nd Aug.1999, p.1509-19 DIELECTRIC RELAXATION AND MECHANICAL INVESTIGATION OF ETHYLENE PROPYLENE DIENE MONOMER RUBBER WITH SOME CROSSLINKING ADDITIVES

Abd-El-Messieh S L; El-Sabbagh S; Abadir I F Egypt,National Research Centre; Cairo,University

The permittivity and dielectric loss of ethylene propylene diene rubber, mixed with zinc chloride and ammonium

iodide in different proportions, were measured over the frequency range 100 Hz to 10 MHz, to provide information on the dielectric properties of the crosslinking process. The dielectric data are discussed in terms of the different relaxation mechanisms present in the system. Thermal ageing and mechanical properties were also investigated. 10 refs.

# EGYPT

Accession no.744176

## Item 149

Journal of Applied Polymer Science 73, No.6, 8th Aug. 1999, p.927-33 EFFECT OF CROSSLINKING TYPE ON THE PHYSICAL PROPERTIES OF CARBOXYLATED ACRYLONITRILE BUTADIENE ELASTOMERS Ibarra L

Madrid, Instituto de Ciencia y Tec.de Polim.

The vulcanisation of carboxylated NBR with curing agents such as zinc peroxide, sulphur and a zinc peroxide-sulphur mixed system was studied. The properties of the vulcanised compounds were dependent on the type of crosslinking produced, i.e. ionic or covalent, rather than the crosslinking density. Ionic crosslinks gave rise to greater stress relaxation, relaxation rates and a greater generation of heat. In the relaxation spectra, tan delta versus temp., two transitions appeared. Those occurring at the lower temp. corresponded to the polymer Tg, while the transition occurring at the higher temp. was associated with ionic structures. The properties of the vulcanised compounds with ionic crosslinks decreased significantly after treatment with ammonia, which acted as a plasticiser of the ionic aggregates formed. The effect of ammonia disappeared on expansion in solvent, which resulted in the recovery of the original crosslinks, producing a value of volume fraction of swollen rubber in equilibrium close to the original value and the reappearance of the ionic transition. 19 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; SPAIN; WESTERN EUROPE

Accession no.743241

# Item 150

**Progress in Rubber and Plastics Technology** 15, No.2, 1999, p.95-112 **PEROXIDE VULCANISATION OF RUBBER** Ogunniyi D s Ilorin,University

A review of the peroxide vulcanisation of elastomers is presented. The generalised mechanism of crosslinking reaction is discussed while network structure is related to vulcanisate properties. Emphasis is placed on elastomers where only the peroxide system is suitable or where it has a comparable advantage over other systems. In addition, the uses of coagents in peroxide vulcanisation are discussed while an attempt to design a delayed-action peroxide system is mentioned. 40 refs. NIGERIA

Accession no.741974

#### Item 151

## *Kautchuk und Gummi Kunststoffe* 52, No.6, June 1999, p.438-45 **IDENTIFICATION OF THE INTERMEDIATES. SULPHUR VULCANISATION OF NATURAL RUBBER**

Rodriguez S; Masalles C; Agullo N; Borros S; Comellas L; Broto F Ramon Llull,University

This paper deals with the sulphur-accelerated vulcanisation of natural rubber using the model compound vulcanisation approach. Squalene was the model molecule chosen, and a curing system containing a combination of two different accelerator types, namely sulphenamide and thiuram types. Such a combination is known to give a synergistic effect in both the processing and final properties. The vulcanisation process was characterised using HPLC, GPC and MS and three families of intermediate compounds that act as sulphurating agents were identified, which helps to explain the synergistic effects of the combination of accelerators of different types. 31 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; SPAIN; WESTERN EUROPE

Accession no.739860

#### Item 152

Journal of Applied Polymer Science 73, No.5, 1st Aug.1999, p.813-8 CURE CHARACTERISTICS AND PHYSICOMECHANICAL PROPERTIES OF NATURAL RUBBER MODIFIED WITH PHOSPHORYLATED CASHEW NUT SHELL LIQUID PREPOLYMER - A COMPARISON WITH AROMATIC OIL

Menon A R R; Pillai C K S; Nando G B Kerala,Regional Research Laboratory; Indian Institute of Technology

NR (ISNR 5 grade) was modified with 10 phr each of phosphorylated cashew nut shell liquid(PCNSL) prepolymer and an aromatic oil plasticiser (spindle oil) in a typical semi-efficient vulcanisation system. Despite the lower chemical crosslink density, the PCNSL modified NR vulcanisates showed higher TS, EB, thermal stability and resistances to fatigue failure and thermooxidative decomposition, as compared with the vulcanisate containing the same dosage of spindle. 12 refs. INDIA

Accession no.738377

#### Item 153

**Polymer Degradation and Stability** 64, No.2, May 1999, p.197-205 **INFLUENCE OF CURE AND CARBON BLACK ON THE HIGH TEMPERATURE OXIDATION OF NATURAL RUBBER. I. CORRELATION OF PHYSICO-CHEMICAL CHANGES** 

Edge M; Allen N S; Gonzalez-Sanchez R; Liauw C M; Read S J; Whitehouse R B Manchester, Metropolitan University; Cabot Corp.

The thermooxidative stability of NR was studied at 150C as a function of the cure process (sulphur, sulphurless or peroxide curing) and the properties of the carbon black filler (five different carbon blacks were used). The thermooxidation was followed by iodometric analysis of hydroperoxide levels, FTIR spectroscopy and differential thermal analysis. The results were discussed and they showed that the type of curing system had an effect on the degradation profile, but the surface reactivity and structure of the carbon black had a controlling role in each system.13 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; UK; USA; WESTERN EUROPE

Accession no.737341

#### Item 154

#### Journal of Applied Polymer Science 72, No.8, 23rd May 1999, p.999-1006 EFFECT OF CARBOXYLIC ACIDS ON 2-BISBENZOTHIAZOLE-2,2'-DISULFIDE- AND TETRAMETHYLTHIURAM DISULFIDE-ACCELERATED SULFUR VULCANIZATION. I. REACTION BETWEEN THE ACIDS AND ACCELERATORS McGill W J: Shelver S R

Port Elizabeth, University

Benzoic and stearic acids were shown to react with 2bisbenzothiazole-2,2'-disulphide(MBTS) and its polysulphides(MBTP) at vulcanisation temps. to form acid-accelerator complexes 2-benzoylthiobenzothiazole(BzM) and stearic thiobenzothiazole(StM), respectively. At higher temps., MBTS, MBTP, BzM and StM were decomposed by the acids. Analogous reactions were observed with tetramethylthiuram disulphide and its polysulphides(TMTP). Lower amounts of acidaccelerator complexes of 2-benzoyldimethyldithiocarbamate and stearic dimethyldithiocarbamate were formed, but TMTP were more susceptible to decomposition at higher temps. The degradation of MBTP and TMTP by carboxylic acids would reduce the concentration of polysulphide accelerator available in compounds and should have a negative effect on vulcanisation. 21 refs. SOUTH AFRICA

Item 155 Revue Generale des Caoutchoucs et Plastiques No.773, Dec.1998, p.75-7 French STRENGTHENING LEGISLATION ON POLLUTION IN THE WORKPLACE Aubin C LRCCP

A survey is made of French and European Union legislation relating to hazardous chemicals in the workplace, and particular attention is paid to regulations concerning exposure to chemicals in the rubber industry. The carcinogenicity of nitrosamines formed during vulcanisation processes is examined, and methods used in their detection are discussed. 2 refs.

EU; EUROPEAN COMMUNITY; EUROPEAN UNION; FRANCE; WESTERN EUROPE; WESTERN EUROPE-GENERAL

Accession no.736308

## Item 156

**Rubber and Plastics News** 28, No.19, 19th April 1999, p.14-5 **VARIOUS VULKALENT E ROLES EXAMINED** Moakes C A Bayer Corp.

The primary function of Vulkalent E is as a scorch retarder in sulphur vulcanisation of unsaturated rubbers. This paper examines the accelerating affect of this chemical, particularly in mercapto accelerated sulphur vulcanisation where it can replace other nitrosamine forming, secondary accelerators. This accelerating effect is combined with its delaying effect on the onset of cure. Examples are shown of its utility in improving crosslink density and hence compression set resistance as well as increasing modulus. Vulkalent E does not form carcinogenic nitrosamine during vulcanisation.

USA

Accession no.735636

#### Item 157

Kautchuk und Gummi Kunststoffe 52, No.5, May 1999, p.322/8 STEEL CORD ADHESION. EFFECT OF 1,3-BIS(CITRACONIMIDOMETHYL)BENZENE AND HEXAMETHYLENE-1,6-BIS(THIOSULPHATE), DISODIUM SALT, DIHYDRATE

Datta R N; Ingham F A A Flexsys BV

Results of studies are examined regarding factors affecting the adhesion of steel cord to rubber. The adhesion is balanced with improved aged compound characteristics and reduced compound heat build-up through the use of 1,3-bis(citraconimidomethyl)benzene, and hexamethylene-1,6-bis(thiosulphate) disodium salt dihydrate. This combination of chemicals not only improves the properties of steel cord skim compounds, but also can be used effectively to replace the bonding system based on resorcinol and hexamethoxy-methylmelamine, it is found. 19 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; NETHERLANDS; WESTERN EUROPE Accession no.735014

#### Item 158

Latex Ltd.

Kautchuk und Gummi Kunststoffe 52, No.5, May 1999, p.316-9 EFFECT OF THE VULCANISING SYSTEM ON THE QUALITY OF NR LATEX CONDOMS Maret J; Mathew M; Francis D J; Sahayadasan J Kottayam,Rubber Research Institute; Cochin,University of Science & Technology; Hindustan

The quantity of sulphur and the type of accelerators used in the vulcanisation of natural rubber condoms was found to affect the tensile properties, burst volume and burst pressure of condoms. Different accelerator combinations were studied, including S-5-ZMBT and NTP-ZMBT combinations, which were found to impart high mechanical strength. The S-ZMBT system was found to give maximum ageing resistance to condoms, compared to other accelerator combinations. Mechanical strength of condoms was found to increase with an increase in the concentration of sulphur, but was accompanied by a decrease in ageing resistance. 7 refs. INDIA

Accession no.735013

#### Item 159

Journal of Applied Polymer Science 72, No.4, 25th April 1999, p.487-92 RUBBER SEED OIL: A MULTIPURPOSE ADDITIVE IN NR AND SBR COMPOUNDS Nandanan V; Joseph R; George K E

Cochin, University of Science & Technology

Rubber seed oil was used as a multipurpose plasticiser and fatty acid component of the activator in NR and SBR compounds. The oil, when substituted for conventional plasticiser, imparted good mechanical properties to the vulcanisates, and improved ageing resistance, reduced cure time, increased abrasion resistance and flex resistance, and reduced blooming. 17 refs. INDIA

Accession no.734903

#### Item 160

International Rubber Exhibition and Conference 1999. Conference proceedings.

Manchester, 7th-10th June 1999, Materials paper 10. HIGH TEMPERATURE CURING OF PASSENGER TYRES BY USING 1,3 BIS-(CITRACONIMIDOMETHYL) BENZENE Ingham F A A; Datta R N Flexsys BV (Crain Communications Ltd.)

The tyre industry is continually striving to improve productivity whilst maintaining a high level of product quality and product performance. Reduced tyre cure time will aid in improving productivity, and one way to achieve this is by increasing cure temperatures. However, the use of higher cure temperatures may give rise to the onset of cure reversion leading to a deterioration of physical properties and, possibly, reduced tyre performance. 1,3 bis-(citraconimidomethyl)benzene (Perkalink 900), an extremely effective anti-reversion agent, maintains the physical properties of vulcanisates that are overcured. Laboratory results obtained in a passenger tread compound are reported that demonstrate the feasibility of improving productivity by increasing cure temperature. 14 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; NETHERLANDS; WESTERN EUROPE

Accession no.734471

#### Item 161

International Rubber Exhibition and Conference 1999. Conference proceedings.

Manchester, 7th-10th June 1999, Materials paper 2. **IMPROVED RUBBER PROPERTIES BY USING** N-T-BUTYL-2-BENZOTHIAZOLE **SULPHENAMIDE** 

de Hoog A J; Datta R N Flexsys BV (Crain Communications Ltd.)

TBSI, N-t-butyl-2-benzothiazole-sulphenimide, is a primary amine-based accelerator which is finding a growing market in applications requiring long scorch safety combined with a moderately slow cure rate, providing good properties, including improved heat build up. Recently investigations have been carried out demonstrating the advantages in typical tyre compounds. Physical and dynamic mechanical properties in various compounds are summarised. Attempts are made to correlate these properties with the network structure. 4 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION: NETHERLANDS; WESTERN EUROPE

Accession no.734465

#### Item 162

International Rubber Exhibition and Conference 1999. Conference proceedings.

Manchester, 7th-10th June 1999, Environment paper 4. NITROSAMINE FREE CURING SYSTEMS FOR **MODERN RUBBER COMPOUNDS** 

Schuch A; Fruh T Rhein Chemie GmbH (Crain Communications Ltd.)

The international discussion about carcinogenic Nnitrosamines generated during or after vulcanisation of rubber products started in the late 1970s. It reached its

climax in 1994 when the German government passed a law limiting nitrosamines to a maximum concentration of 2,5 mug per cubic meter of air. International discussion lost momentum because other countries did not follow the German initiative. The topic was more an intellectual than a real problem until recently, when the automotive industry demanded nitrosamine-free products. With increasing sensibility regarding environmental aspects, there is a growing list of curing agents (and additives) that may no longer be used in the new future. It is shown that the substitution of curing agents with a high toxicological potential does not necessarily mean that compounding is getting more difficult or that the products show a reduced performance. 2 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; GERMANY; WESTERN EUROPE

Accession no.734448

Item 163

Luntai Gongye 19, No.5, 1999, p.274-7 Chinese STUDY ON IMPROVING AGEING PROPERTIES **OF NR TUBE** Rui C

Hangzhou China Strategy Rubber Corp.Ltd.

The effect of sulphur variation on the thermal ageing properties of a natural rubber inner tube was investigated under laboratory conditions. The results showed that the thermal ageing properties of the tube improved significantly by reducing the sulphur level. The test result in pilot production showed that the semi-efficient curing system comprising sulphur 0.9 phr, accelerator 2.28 phr, was the optimal choice. This result was also confirmed by the physical properties and results of storage tests on 9.00-20 finished tube.

CHINA

Accession no.734332

#### Item 164

International Polymer Science and Technology 25, No.10, 1998, p.23-5 **REACTION OF SULPHENAMIDE** ACCELERATORS WITH SULPHUR UNDER **TEMPERATURE CONDITIONS MODELLING** THE VULCANISATION SCHEDULE

Tarasov D V; Vishnyakov I I; Grishin B S

Results are reported of a study of the thermochemical processes occurring under temp. conditions similar to vulcanisation schedules with N-oxydiethylene-2benzthiazyl sulphenamide and N-cyclohexyl-2benzthiazyl sulphenamide and their mixtures with sulphur. Experiments were carried out using a chromato-mass spectrometer and a TGA-DTA thermal analyser. The formation of high molec.wt. polysulphides in the thermolysis products of sulphenamides with sulphur at

vulcanisation temps. was established. 3 refs. (Full translation of Kauch.i Rezina, No.5, 1991, p.39) RUSSIA

Accession no.733369

#### Item 165

Rubber Chemistry and Technology 71, No.4, Sept-Oct. 1998, p.766-77 NITROSAMINE FORMATION IN RUBBER. II. INFLUENCE OF CURE Willoughby B G; Scott K W Rapra Technology Ltd.

Details are given of nitrosamine formation in twenty-nine different EPDM cures. Both ingredient and processrelated effects were investigated. Two different levels of tetramethylthiuram disulphide and carbon black were variables in this study, as was the type of carbon black used. Batch effects with respect to both polymer and carbon black were also studied as was the presence or absence of mercaptobenzothiazole. 14 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; UK; WESTERN EUROPE

Accession no.730170

#### Item 166

International Polymer Science and Technology 25, No.9, 1998, p.T/22-7 INFLUENCE OF TEMPERATURE ON NETWORK FORMATION IN POLYISOPRENES Sokolova L V; Shershnev V A Moscow.Lomonosov Institute

NR and synthetic cis-1,4-polyisoprene were vulcanised by quinol ether at temperatures of 87C and 143C for 1h under 20MPa. The degree of crosslinking was varied by the amount of quinol ether. The vulcanisates were studied by swelling in various solvents, uniaxial elongation and relaxation experiments, and EB measurements. Variations in Mooney-Rivlin parameters and swelling are discussed in terms of differences between NR and synthetic polyisoprene, degree of crosslinking, orientation, uneven distribution of crosslinks and ordering. It is shown that supermolecular formations have a considerable influence on the process of network formation in polyisoprenes. 25 refs.

RUSSIA

Accession no.729162

#### Item 167

International Polymer Science and Technology 25, No.9, 1998, p.T/19-21 INFLUENCE OF A DITHIOPHOSPHATE-POLYQUATERNARY AMMONIUM SALT BINARY SYSTEM ON THE SULPHUR VULCANISATION OF SKI-3 POLYISOPRENE RUBBER

Ovcharov V I; Fomina A V Ukraine,State Chemico-Technological University The polyquaternary ammonium salt and sodium diisobutyldithiophosphate were used together and separately to accelerate the sulphur vulcanisation of polyisoprene. Kinetic curves showed that vulcanisation in the presence of the polyquaternary salt alone resulted in a considerable induction period, a slow rate and a low degree of vulcanisation, so that the polyquaternary salt alone can be considered to have low effectiveness as an accelerator. The sodium diisobutyldithiophosphate was an accelerator and its accelerating effect was enhanced by the presence of the polyquaternary salt. Use of both substances accelerated the formation of sulphide complexes and the crosslink formation. Thus it is established that the binary system of sodium diisobutyldithiophosphate and polyquaternary ammonium salt exhibit synergism in relation to different stages of sulphur vulcanisation, the degree of crosslinking and the elastic strength properties. The polyquaternary salt is a co-accelerator. 8 refs.

#### UKRAINE

Accession no.729161

#### Item 168

*International Polymer Science and Technology* 25, No.7, 1998, p.67-70 **WAYS OF SOLVING THE PROBLEM OF NITROSOAMINES IN THE PRODUCTION OF TYRES AND MECHANICAL RUBBER GOODS** Novopol'tseva O M; Novakov I A; Krakshin M A Volgograd,State Technical University

The carcinogenicity of nitrosamines, compounds which are formed during vulcanisation by the reaction of amines produced during the thermal decomposition of certain vulcanisation accelerators with nitrosing agents, is discussed. Methods of eliminating conditions where nitrosamines may be formed and enter the human body are considered. Particular attention is paid to the elimination of the possibility of formation of nitrosamines and/or the creation of conditions for reactions of nitrosamines with certain reactants with the formation of non-toxic compounds and to the replacement of vulcanisation accelerators and other rubber mix ingredients that are capable of forming nitrosamines with amine-free compounds or with compounds containing primary amines instead of secondary amines. 30 refs. (Full translation of Kauch.i Rezina, No.2, 1998, p.5) RUSSIA

Accession no.727056

Item 169 Elastomery 2, No.6, 1998, p.10-7 Polish DITHIOATES ARE MORE THAN JUST NITROSAMINE FREE Schuch A; Fruh T

### Rhein-Chemie AG

Concerns relating to nitrosamine forming accelerators and sulphur donors has led to an interest in vulcanising agents based on dithioates. In addition to the environmental advantages afforded by such products, dithioates are also shown to provide reversion resistance in NR and SBR, and are known to protect against oxygen ageing. Due to their non-polar characteristics, dithioates demonstrate good solubility in non-polar rubbers such as EPDM, and can prevent blooming. Details are also given of Rhenocure SDT, sulphur donor based on a dithioate structure. 10 refs. EUROPEAN COMMUNITY; EUROPEAN UNION; GERMANY; WESTERN EUROPE

Accession no.726332

#### Item 170

### *Polymer Plastics Technology and Engineering* 38, No.1, 1999, p.127-36 **NEW RUBBER INGREDIENT BASED ON PALM OIL FATTY ACID**

Ismail H; Ng C T; Ishiaku U S; Ishak Z A M Universiti Sains Malaysia

The potential use of palm oil fatty acid additive (POFA) as a new rubber ingredient in carbon black-filled NR compounds is examined. It is found that the cure time (t90) and scorch time decrease with increasing POFA concentration. The Mooney viscosity of the rubber compounds also decreases with increasing POFA concentration. The mechanical properties of the carbon black-filled NR compounds are enhanced by the addition of POFA, especially at a concentration of 2 phr. Results of the swelling measurement and scanning electron microscopy indicate that POFA has some effect on crosslink density and also improved filler dispersion. 9 refs.

MALAYSIA

Accession no.724278

#### Item 171

Tech XXI. Conference proceedings. Orlando, Fl., 6th-8th May,1998, p.25-35. 6A7 **HIGH PERFORMANCE UV CURABLE PRESSURE SENSITIVE ADHESIVES BASED ON KRATON LIQUID POLYMERS** Erickson J R; Mancinelli P A Shell Chemical Co. (Pressure Sensitive Tape Council)

Recent developments in Kraton Liquid Polymer Technology have led to new developmental raw materials useful in the preparation of 100% solids UV cure high performance pressure-sensitive adhesives. The compounding of a mixture of Kraton L-207 (hydrogenated butadiene-isoprene copolymer) and Kraton L-1203 (hydrogenated polybutadiene) with UV curing systems (mixing of UV curer requires sonication) and tackifiers is described in detail. A wide range of tack, peel (on steel, glass and various polymers), room temperature shear and elevated temperature shear properties were measured and compared with acrylic transfer tapes and solvent acrylic pressure-sensitive adhesives. Resistance to humidity, chemicals and UV light is also shown.

USA

Accession no.724121

#### Item 172

*Gummi Fasern Kunststoffe* 51, No.11, Nov.1998, p.906-11

#### German

ENVIRONMENTAL HAZARDS BY DUMPING RUBBER WASTE IN UNSEALED LANDFILL SITES Buettner R

Thueringisches Institut fuer Textil- & Kunststoff-Forschung eV

Results are presented of an investigation of the effect of leaching of rubber vulcanisation accelerators and antioxidants into the environment at unsealed dump sites in Germany. 7 refs. Articles from this journal can be requested for translation by subscribers to the Rapra produced International Polymer Science and Technology. EUROPEAN COMMUNITY; EUROPEAN UNION; GERMANY; WESTERN EUROPE

Accession no.723327

### Item 173

*China Rubber Industry* 46, No.2, 1999, p.71-4 Chinese

# CURVE EQUATIONS FOR DESCRIBING FATIGUE LIFE OF NR/BR BLENDS CURED WITH CONVENTIONAL, SEMI-EFFICIENT OR EFFICIENT CURING SYSTEM

Qiu Yiming; Xiao Jianbin; Fu Zheng; Liang Guoliang; Ji Xinsheng

Qingdao,Institute of Chemical Technology

The fatigue life of NR/polybutadiene(BR) vulcanisates cured with conventional, semi-efficient and efficient curing systems and possessing the same apparent crosslink density was investigated. The fatigue parameters of the three vulcanisates were compared with each other and curves of equations for describing the fatigue life of the vulcanisates cured with three different curing systems were fitted to the exponential function model. The equations with maximum relative error only up to 6.25% and correlation factors greater than 0.9535 were suitable for describing the dynamic fatigue process of NR/BR vulcanisates cured with different curing systems. 12 refs. CHINA

Accession no.716401

Item 174

*International Polymer Science and Technology* 25, No.6, 1998, p.39-42

## INFLUENCE OF ESTERS OF DITHIOPHOSPHORIC ACID ON THE SULPHUR VULCANISATION OF POLYISOPRENE

Ovcharov V I

Ukraine, State Chemico-Technological University

Details are given of the effectiveness of new derivatives of dithiophosphoric acid as accelerators of the sulphur vulcanisation of polyisoprene. The dependence of the activity of the accelerators on their structure is discussed. 7 refs. Translated from Kauch.i.Rezina, 1, 1998, p.30 UKRAINE

Accession no.714287

## Item 175

*Kauchuk i Rezina (USSR)* No.2, 1997, p.46-8 Russian

## NEW VULCANISATION ACCELERATOR FOR BUTADIENE-STYRENE RUBBERS

Yuldashev D Ya; Yusupbekov A Kh; Ibadullaev A; Negmatov S S

Uzbek Republic Technological Complex "Fan va Tarakkiet"

The study relates to the effect of a new class of donor-acceptor vulcanisation accelerators, namely phosphatised alkylamides of fatty acids, on the rate of vulcanisation of butadienestyrene rubber and on the physical and mechanical properties of the vulcanisates. 7 refs. Articles from this journal can be requested for translation by subscribers to the Rapra produced International Polymer Science and Technology.

RUSSIA

Accession no.714163

# Item 176

Kauchuk i Rezina (USSR)

No.2, 1997, p.40-2 Russian

# DYNAMIC VULCANISATION OF ELASTOMER COMPOUNDS BASED ON SKI-3 AND SKF-32

Petrova N N; Khodzhaeva I D; Adrianova O A; Cherskii I N

Yakutsk,Institute of Non-metallic Materials; Moscow,Institute of Fine Chemical Technology

The authors examined the possibility of using a nontraditional method of vulcanisation of compounds based on cis-1,4-polyisoprene and fluororubber under dynamic conditions with the application of shear forces. Vulcanising systems based on a sulphur-containing system and copper salicylal iminate were employed. Data are given on the effect of the method of vulcanisation on the physical and mechanical properties of the vulcanisates. 6 refs. Articles from this journal can be requested for translation by subscribers to the Rapra produced International Polymer Science and Technology. RUSSIA

Accession no.714161

Item 177

*Kauchuk i Rezina (USSR)* No.1, 1998, p.38-40

Russian

SELECTING A VULCANISING AGENT FOR ROOFING MATERIALS BASED ON SYNTHETIC RUBBER AND TYRE PRODUCTION WASTE

Glebov E N; Shumilin Yu F; Molodyka A V; Kinash T A

Voronezh, State Technological Academy

The possibility was investigated of using rubber waste consisting of polyisoprene, butadiene-methylstyrene and polybutadiene with machine oils and carbon black for the production of roofing materials. Mechanical properties of the resulting materials are listed. 6 refs. Articles from this journal can be requested for translation by subscribers to the Rapra produced International Polymer Science and Technology.

RUSSIA

Accession no.713885

Item 178

Kauchuk i Rezina (USSR) No.5, 1997. p.27-34 Russian EFFECT OF SULPHUR-, PHOSPHORUS- AND NITROGEN-CONTAINING ORGANIC ACCELERATORS ON SULPHUR VULCANISATION OF RUBBERS AND THE PROPERTIES OF THE VULCANISATES Ovcharov V I

Using various classes of organic compounds containing different functional groups and substituents, the authors examined the relationship between their structure and the kinetic parameters of sulphur vulcanisation, the mechanism of sulphur vulcanisation of unsaturated rubbers in the presence of these organic compounds, and studied the effectiveness of binary systems of accelerators. The compounds studied were phosphorylated dithiocarbamates and dithiocarbamates, derivatives of dithiophosphoric and dithiophosphonic acids, phosphorylated thioamides and thioamides, and phosphorylated thioureas and thioureas. 50 refs. Articles from this journal can be requested for translation by subscribers to the Rapra produced International Polymer Science and Technology.

RUSSIA

Accession no.713252

*Item 179 Kauchuk i Rezina (USSR)* No.5, 1997, p.10-2 Russian POLYMERIC AQUEOUS DISPERSION VULCANISING AGENTS FOR GENERAL-PURPOSE LATICES AND RUBBERS Averko-Antonovich I Yu; Smirnov A A; Fomina L I Kazan,State Technological University

The authors describe the use of Tiotex dispersion, a stable aqueous dispersion of polysulphide polymer, as a vulcanising agent for use with latex compositions. The effect of this vulcanising agent on the crosslinking of compositions based on butadiene-styrene, butadienevinylidene chloride, butadiene-vinylidene chloride carboxylated and butadiene-acrylonitrile carboxylated latices is described. Physico-mechanical properties of films made from latices and of the vulcanisates are given. 5 refs. Articles from this journal can be requested for translation by subscribers to the Rapra produced International Polymer Science and Technology. RUSSIA

Accession no.713245

#### Item 180

IRC '98. Conference Proceedings. Paris, 12th-14th May 1998, p.259-60. 012 French

#### **STUDY OF THE INFLUENCE OF VULCANISATION ON N-NITROSAMINE FORMATION MECHANISMS IN RUBBERS** Amelot V; Derouet D; Brosse J C; Aubin C; Gomez E Maine,Universite; LRCCP; IFOCA

(AFICEP; Societe de Chimie Industrielle)

The methodology used in a study of nitrosamine formation in vulcanisation processes accelerated by tetraethylthiuram disulphide is described. This involved the characterisation of nitrosable species and the mechanisms of their formation, and studies of the influence of vulcanisation reactions and curing systems on the formation of nitrosable species. The results of the study are summarised.

EUROPEAN COMMUNITY; EUROPEAN UNION; FRANCE; WESTERN EUROPE

Accession no.710619

#### Item 181

*Gummi Fasern Kunststoffe* 49, No.11, Nov.1996, p.892-9 German

# **CROSSLINK COMPENSATION: A NEW WAY TO COUNTERACT REVERSION**

Datta R N; Talma A G; Wagenmakers J C; Seeberger D Flexsys BV

The basic chemistry of crosslinking and reversion is discussed, and a new concept is proposed to counteract reversion, based on the use of 1,3-bis(citraconimidomethyl)benzene (BCI-MX, tradename Perkalink 900). The effect of BCI-MX in natural rubber compounds with regard to suppression of reversion, hysteresis and dynamic properties is investigated. A theory on the mechanism of reversion prevention is proposed. 22 refs. Articles from this journal can be requested for translation by subscribers to the Rapra produced International Polymer Science and Technology.

EUROPEAN COMMUNITY; EUROPEAN UNION; GERMANY; WESTERN EUROPE

Accession no.710108

### Item 182

Nippon Gomu Kyokaishi 70, No.1, 1997, p.43-9 Japanese KINETIC STUDY OF THE PEROXIDE CROSSLINKING PROCESS OF RUBBERS USING AN OSCILLATING RHEOMETER Sato T

Nippon Zeon Co.Ltd.

A formula is proposed for analysis of the kinetics of peroxide crosslinking of rubber, showing the relation between the rate of crosslinking and the initial peroxide concentration. Calculated values for the coefficients A and B for SBR, BR, MBR and HNBR with varying peroxide concentrations showed that the crosslinking mechanism varies according to the type of rubber. 11 refs. Articles from this journal can be requested for translation by subscribers to the Rapra produced International Polymer Science and Technology.

JAPAN

Accession no.710060

#### Item 183

*Gummi Fasern Kunststoffe* 49, No.12, Dec.1996, p.984-95 German **REVERSION RESISTANCE OF NATURAL RUBBER MIXES WITH THE USE OF DITHIOPHOSPHATE CURING SYSTEMS** Graf H-J; Johansson A H

Rhein Chemie Corp

Curing systems consisting of sulphur, MBTS and dithiophosphate comparison systems which permit amines to be split off are studied with regard to thermal ageing properties, and show considerably less reversion. 9 refs. Articles from this journal can be requested for translation by subscribers to the Rapra produced International Polymer Science and Technology.

EUROPEAN COMMUNITY; EUROPEAN UNION; GERMANY; WESTERN EUROPE

Accession no.710022

#### Item 184

Technical Rubber Goods - Part of our Everyday Life. Conference proceedings. Puchov, 23rd-24th May 1996, p.279-87. 4 **REVERSION RESISTANCE TO NR WITH SPECIAL DITHIOPHOSPHATE CURE SYSTEMS** Graf H-J; Fruh T Rhein Chemie Rheinau GmbH

## (Matador AS)

NR cured with common accelerators shows reversion if heat aged. Addition of an organic acid in addition to the use of stearic acid decreases the tendency toward reversion. Different accelerators cause different degrees of crosslink degradation as seen with a rheometer or evidenced by lower modulus values upon heat ageing. It can be concluded that amines available during vulcanisation as decomposition products from accelerators will catalyse this reversion. If that is truly the case, then much less reversion can be expected if an accelerator system is used that does not form amines during curing. Standard, semi-efficient and efficient cure systems are developed using sulphur, zinc dithiophosphate and dithiophosphate tetrasulphide accelerator systems. After heat ageing, the retention of modulus and all other physical properties are much better than the control. After ageing for seven days at 90 deg.C, vulcanisates cured with the efficient cure system without free sulphur display outstanding reversion resistance. 9 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; GERMANY; WESTERN EUROPE

Accession no.708378

#### Item 185

Technical Rubber Goods - Part of our Everyday Life. Conference proceedings.

Puchov, 23rd-24th May 1996, p.83-105. 4 **PERKALINK 900 - A NEW CHEMICAL FOR REVERSION RESISTANT COMPOUNDING** Datta R N; van der Heide F R; Hellendoorn R Flexsys BV (Matador AS)

The phenomenon of reversion is associated with decrease in crosslink density and formation of main-chain modifications. The consequence of degradation of network and the formation of sulphidic and non-sulphidic modifications during reversion is explained. Emphasis is placed on a chemical, 1,3-bis(citraconimidomethyl) benzene (Perkalink 900), which counteracts reversion by a process called crosslink compensation. Not only does Perkalink 900 add crosslinks lost during reversion, it also eliminates dienes and trienes form the network. The mechanism of Perkalink 900 crosslinking is discussed. Based on the above studies, the advantage of using Perkalink 900 in NR-based compounds is examined. Key applications and guidelines for optimal use are outlined. 25 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; NETHERLANDS; WESTERN EUROPE Accession no.708359

# Item 186

Kauchuk i Rezina (USSR) No.3, 1996, p.35-6 Russian

## HEALTH AND TOXICOLOGY STUDIES OF ELASTOMER COMPOSITIONS CONTAINING **DINITRILE OXIDE**

Vlasyuk M G; Boiko V V; Prokof'eva L G

A method and results are described of the toxicity index of compositions of SKI-3 polyisoprene and SKN-18 butadiene-acrylonitrile rubbers containing mesitylene dinitrile oxide as a curing agent. 5 refs. Articles from this journal can be requested for translation by subscribers to the Rapra produced International Polymer Science and Technology.

RUSSIA Accession no.708284

Item 187

Nippon Gomu Kyokaishi 69, No.4, 1996, p.280-289 Japanese ENVIRONMENTALLY FRIENDLY CURING **SYSTEMS** Mori T; Ito I Flexsys KK

The mechanism of formation of nitrosamines is described, and the use of secondary sulphenamide and thiuram curing systems and sulphur-donor systems is discussed. 23 refs. Articles from this journal can be requested for translation by subscribers to the Rapra produced International Polymer Science and Technology. JAPAN

Accession no.707019

#### Item 188

Nippon Gomu Kyokaishi 69, No.4, 1996, p.271-279 Japanese CHEMISTRY OF CROSSLINKING Ontsuka H; Akiba M Incorporated Chemicals Inspection Association

Sulphur crosslinking, and the crosslink structure of chloroprene rubber, other halogenated rubbers and other types of rubbers are described. 39 refs. Articles from this journal can be requested for translation by subscribers to the Rapra produced International Polymer Science and Technology.

JAPAN

Accession no.707018

Item 189 Nippon Gomu Kyokaishi 69, No.3, 1996, p.223-228 Japanese SULPHUR CURING OF NATURAL RUBBER USING A COMBINATION OF TOTD AND CBS Otomo S; Kobayashi Y; Yamamoto Y Ouchi Shinko Kagaku

The activity of tetrakis(2-ethylhexyl)thiuramdisulphide (TOTD) as a curing accelerator was evaluated. Used with CBS for sulphur curing of natural rubber, TOTD showed superior curing characteristics such as good scorch resistance and a faster curing rate than other thiuramdisulphides. 5 refs. Articles from this journal can be requested for translation by subscribers to the Rapra produced International Polymer Science and Technology. IAPAN

Accession no.707014

# *Item 190 Kauchuk i Rezina (USSR)* No.1, 1996, p. 36-39 Russian HEAT- AND OIL-RESISTANT RUBBERS FOR HOSES

Murav'eva L V; Saraeva V P; Bebikh G F

Studies are carried out with the aim of devising new vulcanisates based on butyl acrylate rubber and combinations of this with fluoro rubber, having high resistance to heat and oils, to be used in hoses required to operate at temperatures in excess of 200 C. A wide range of vulcanising agents was investigated, and data are given on the physico-mechanical properties of the vulcanisates before and after ageing. 4 refs. Articles from this journal can be requested for translation by subscribers to the Rapra produced International Polymer Science and Technology. RUSSIA

Accession no.706872

# Item 191

IRC '98. Conference proceedings. Paris, 12th-14th May 1998, p.333-4. 012 **ADVANTAGES OF DITHIOPHOSPHATES IN THICK SECTION ARTICLES AND DYNAMICALLY HIGH LOADED PARTS** Fruh T; Schulz H Rhein Chemie Rheinau GmbH

(AFICEP; Societe de Chimie Industrielle)

Cure systems based on dithiophosphates offer a variety of advantages to the user. Due to their chemical structure, dithiophosphates cannot generate N-nitrosamines. Furthermore, they give superior vulcanisate properties compared to traditional curing systems. In NR or blends with NR they lead to extraordinary reversion stability. The unique network structure leads to very good dynamic properties and a low heat build-up. These properties make dithiophosphates in general ideal candidates for thick section articles and dynamically high loaded parts. This is true for tyres as well as technical rubber goods. Examples for important applications are presented. 6 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; GERMANY; WESTERN EUROPE

Accession no.706000

#### Item 192

IRC '98. Conference proceedings. Paris, 12th-14th May 1998, p.261-2. 012 THE USE OF VULKALENT E AS A REPLACEMENT FOR NITROSAMINE FORMING SECONDARY ACCELERATORS IN SULPHUR VULCANISATION Moakes C A Bayer AG

(AFICEP; Societe de Chimie Industrielle)

The primary function of Vulkalent E (N phenyl N(tri chloromethyl sulphenyl) benzenesulphonamide) is as a scorch retarder in sulphur vulcanisation of unsaturated rubbers. The accelerating affect of this chemical, particularly in mercapto accelerated sulphur vulcanisation where it can replace other, nitrosamineforming, secondary accelerators, is examined. This accelerating effect is combined with its delaying effect on the onset of cure.

EUROPEAN COMMUNITY; EUROPEAN UNION; GERMANY; WESTERN EUROPE

Accession no.705989

#### Item 193

Kauchuk i Rezina (USSR) No.6, 1995, p.15-6 Russian USE OF SULPHO-DERIVATIVES OF 2,6-DI-TERT-BUTYLPHENOL IN THE PREPARATION OF EMULSION RUBBERS

Averko-Antonovich I Yu; Samuilov Ya D; Krokhin V V; Bukharov S V

The authors tested the effects of derivatives of 2,6-ditert-butylphenol in which the sulphur is present in the form of mono-, di-, poly- and hydrosulphide groups as molecular mass regulators for emulsion butadiene-styrene rubbers. 2,6-di-tert-butyl-4-mercaptophenol is recommended as giving latices with good physical properties and polymers with fragments of chemically combined antioxidant. 6 refs. Articles from this journal can be requested for translation by subscribers to the Rapra produced International Polymer Science and Technology. RUSSIA

Accession no.704985

#### Item 194

*Nippon Gomu Kyokaishi* 68, No.7, 1995, p.489-95 Japanese

# CROSSLINKING OF EPDM AND NBR/EPDM BLENDS WITH POLYFUNCTIONAL ALLYL AND/OR VINYL COMPOUNDS

Onouchi Y; Inagaki S; Okamoto H; Furukawa J Aichi,Institute of Technology

Crosslinking promoters consisting of polyfunctional allyl and/or vinyl compounds were prepared by ring-opening

addition of allylglycidyl ether or glycidyl methacrylate and polyfunctional alcohols or carboxylic acids. The effect of these on pure EPDM and NBR/EPDM blends was examined, and data are given on the crosslinking effect and mechanical properties. 2 refs. Articles from this journal can be requested for translation by subscribers to the Rapra produced International Polymer Science and Technology.

JAPAN

Accession no.704950

## Item 195

Patent Number: US 5777012 A 19980707 POLYSULPHIDES OF N-METHYLPYRROLIDINONE

Wideman L G; Futamura S Goodyear Tire & Rubber Co.

These are added to rubber to improve hysteresis, accelerate cure rate and improve filler dispersion. USA

Accession no.704870

# Item 196

*Kauchuk i Rezina (USSR)* No.2, 1995, p.35-37

#### Russian INFLUENCE OF MESITYLENE DINITRILE OXIDE ON THE PROCESSING PROPERTIES OF ELASTOMER COMPOSITIONS Daile VV/c Crimer LV

Boiko V V; Grinev I V

The effect of mesitylene dinitrile oxide (MDNO) as a lowtemperature vulcanising agent for unsaturated elastomers is investigated. For the purpose of predicting the processing properties of elastomer compositions when mesitylene dinitrile oxide is introduced, the authors examined the change in Mooney viscosity of polyisoprene (SKI-3), butadiene-styrene (SKS-30ARKM-15), butadiene-nitrile (SKN-18) and polybutadiene rubbers. 7 refs. Articles from this journal can be requested for translation by subscribers to the Rapra produced International Polymer Science and Technology. RUSSIA

Accession no.704334

# Item 197

**Polymer Plastics Technology and Engineering** 37, No.4, Nov.1998, p.483-94 **PALM OIL FATTY ACID AS AN ACTIVATOR IN NATURAL RUBBER GUM COMPOUNDS** Ismail H; Ruhaizat T A Universiti Sains Malaysia

The potential of palm oil fatty acid as an activator in NR gum compounds is studied. The curing characteristics and mechanical properties of NR gum compounds with palm oil fatty acid as an activator are

not much different compared to commercial stearic acid, especially at concentrations below 3 phr. Incorporation of both acids increases the scorch and cure times, t90, and the MHR - ML (maximum torque - minimum torque) passes through a maximum value and decreases slightly with increasing amounts of acids. Tensile modulus and hardness for stearic acid increase up to 5 phr and then decrease with increasing acid concentration. However, for palm oil fatty acid, these properties show an optimum value at 3 phr and then start to decrease. The fracture properties also pass through a maximum as the concentration of both acids increase. 15 refs.

MALAYSIA

Accession no.704284

Item 198

# Polymer Plastics Technology and Engineering 37, No.4, Nov.1998, p.469-81 EPOXIDISED NATURAL RUBBER COMPOUNDS: EFFECT OF VULCANISATION SYSTEMS AND FILLERS

Ismail H; Ishiaku U S; Arinab A R; Ishak Z A M Universiti Sains Malaysia

The effects of various vulcanisation systems and fillers in epoxidised NR (ENR) at 50% epoxidation (ENR 50 compounds) are examined. ENR compounds cured with the semi-EV (efficient vulcanisation) system show the shortest cure and scorch times but the highest torque values. White rice husk ash (WRHA) filled compounds have the shortest cure and scorch times followed by carbon black and silica. The presence of a silane coupling agent, gamma-mercaptopropyl trimethoxysilane, enhances the curing characteristics but the trend is similar. ENR compounds cured with the semi-EV system give the best overall mechanical properties. A significant enhancement of the mechanical properties is observed in the presence of a silane coupling agent, especially for WRHA- and silica-filled compounds. 17 refs.

MALAYSIA

Accession no.704283

#### Item 199

Nippon Gomu Kyokaishi 68, No.6, 1995, p.430-31 Japanese NOCCELER-DZ DELAYED ACTION VULCANISATION ACCELERATOR (3)

Test results are given on the mechanical properties of rubber containing Nocceler-DZ and on the coupling effect of this accelerator in rubbers containing cobalt naphthenate. Articles from this journal can be requested for translation by subscribers to the Rapra produced International Polymer Science and Technology. IAPAN

## Item 200 Kauchuk i Rezina (USSR) No.3, 1995, p.13-4 Russian VULCANISATION OF BUTYL RUBBER AT MODERATE TEMPERATURE Shilov I B; Khlebov G A; Baranov L A

A new rubber formulation has been developed based on BK-1675 butyl rubber with a new vulcanising agent based on quinol ether for rapid vulcanisation in hot water. Mechanical and physico-chemical properties of the vulcanisate are listed. 4 refs. Articles from this journal can be requested for translation by subscribers to the Rapra produced International Polymer Science and Technology. RUSSIA

Accession no.704138

Item 201

Nippon Gomu Kyokaishi 68, No.3, 1995, p.197-201 Japanese PERFORMANCE OF BISAMINE DISULPHIDES AND THIURAM DI- AND POLYSULPHIDE DERIVATIVES AS NON-NITROSAMINE VULCANISING AGENTS Watanabe T; Yamaguchi R Yokohama,University; Yanai Chemical Industries KK

For the purpose of developing non-nitrosamine type vulcanising agents, bicyclic amide disulphides, nonnitrosamine bisamine disulphides and non-nitrosamine thiuram di- and polysulphide derivatives were synthesised and their performance in sulphur and EV vulcanisation were investigated. 8 refs. Articles from this journal can be requested for translation by subscribers to the Rapra produced International Polymer Science and Technology. JAPAN

Accession no.703574

# Item 202

154th ACS Rubber Division Meeting - Fall 1998. Conference preprints. Nashville, Tn., 29th Sept.-2nd Oct.1998, paper 25. 012 **THIURAM CHARACTERISATION IN EPDM** Smith R S; De Hoog A J Flexsys America LP; Flexsys BV (ACS,Rubber Div.)

A range of thiuram secondary accelerators is evaluated in an ENB-based EPDM compound and in a new technology EPDM compound. The recipes used are fairly basic, utilising CV and EV vulcanisation systems. The rheology is tested on an MDR rheometer and the compound viscosity and scorch tested on a Mooney viscometer. Stress-strain properties are established after cure at 160 deg.C and after ageing at 100 deg.C. In addition, tear properties and compression set values at various temperatures are determined. The observed differences between the various thiurams and their different behaviour in the two EPDM types are discussed. EUROPEAN COMMUNITY; EUROPEAN UNION; NETHERLANDS; USA; WESTERN EUROPE Accession no.701862

#### Item 203

154th ACS Rubber Division Meeting - Fall 1998. Conference preprints. Nashville, Tn., 29th Sept.-2nd Oct.1998, paper 17. 012 **USE OF TWO ANTI-REVERSION AGENTS TO GREATLY ENHANCE COMPOUND REVERSION RESISTANCE** Kuebler T P

Kumho Technical Center (ACS,Rubber Div.)

Aircraft tyres can endure severe service which builds up extreme heat causing reversion of the compounds leading to possible tyre failure. Development of high reversion resistant aircraft compounds is an important goal. Details are given of the development work carried out in which the combination of two anti-reversion agents - 1,3-bis citraconimidomethyl benzene (BCI-MX), Perkalink 900, and a mixture of zinc salts of one or more C8-C10 aliphatic carboxylic acids, Activator 73 - used in an aircraft carcass compound provide a synergistic improvement in reversion resistance. Data are presented which show the reversion resistance improvement with each of the two subject chemicals separately and together, the work done to arrive at the most effective concentration levels, and other changes made to maintain physical properties. Reversion resistance is measured using the BFG Flexometer operated under blowout conditions as described in ASTM D623 and the Oscillating Disk Rheometer. As a result, US Patent 5,623,007 is granted on April 22, 1997. USA

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Accession no.701855

#### Item 204

ITEC '96 Select. Conference proceedings. Akron, Oh., 1996, p.101-6. 6T **PEROXIDE-CURED COMPOSITIONS CAN CAUSE CHANGE IN SCORCH TIME** Class J B Hercules Inc. (Rubber & Plastics News)

Peroxide vulcanisation of tyre compounds is examined and compared to sulphur vulcanisation in terms of curing mechanism and compounding considerations. The elimination of cure reversion with peroxides will allow tyres to be cured at higher temperatures, resulting in improved productivity, and the excellent aging properties expected for peroxide cures are claimed to be suitable for high mileage tyres. In addition, reduced compression set can improve the running performance of tyres. USA

# Item 205

ITEC '96 Select. Conference proceedings. Akron, Oh., 1996, p.37-42. 6T IMPROVING TRUCK TIRE PERFORMANCE THROUGH CROSSLINK COMPENSATION

Kumar N R; Chandra A K; Mukhopadhyay R Singhania Elastomer & Tire Research Institute (Rubber & Plastics News)

The effect of Perkalink 900 anti-reversion agent is studied in tread cap and base compounds of truck tyres, and the advantages it affords regarding the performance of truck tyres are discussed. Its use is shown to bring about a reduction or elimination of the reversion process when determined from the cure characteristics in a rheometer, and a higher retention of modulus and tensile strength after extended cure and hot air aging.14 refs.

FLEXSYS BV

EUROPEAN COMMUNITY; EUROPEAN UNION; INDIA; NETHERLANDS; USA; WESTERN EUROPE

Accession no.701529

Item 206 ITEC '96 Select. Conference proceedings. Akron, Oh., 1996, p.33-6. 6T CHEMICAL REVERSION RESISTANCE IMPROVED BY TWO NEW APPROACHES Data R; Helt W F Flexsys Rubber Chemicals

(Rubber & Plastics News)

The use and benefits are described of two new rubber chemicals which have been introduced to the rubber industry to provide unique approaches to the problem of reversion and associated changes in rubber tyre compound performance. The products are Duralink HTS and Perkalink 900. They are claimed to improve the thermal aging resistance of sulphur based vulcanisates, the former participating in the vulcanisation reaction to form thermally stable and flexible hybrid crosslinks, and the latter, reacting during the reversion process to replace degraded sulphur crosslinks with thermally stable and long lasting carbon-carbon crosslinks, which improves dynamic mechanical properties, while maintaining other performance characteristics.

USA

Accession no.701528

# Item 207

China Rubber Industry 45, No.10, 1998, p.587-9 Chinese ACCELERATION OF EPDM CROSSLINKING WITH TMPTA

Wang Shifeng; Wang Dizhen; Chen Shangan South China, University of Technology

The effect of TMPTA co-curing agent on the curing behaviour, physical properties and ageing properties of

peroxide-cured EPDM was investigated. The results obtained showed that the viscosity of the mix decreased, the optimum cure time shortened and the physical properties and ageing properties were improved by addition of TMPTA. 5 refs.

CHINA

Accession no.701004

#### Item 208

Kauchuk i Rezina (USSR) No.5, 1994, p.18-20 Russian USE OF THE PRODUCT ELASTID AS AN EFFECTIVE MODIFYING ADDITIVE FOR VULCANISATES WITH SULPHUR VULCANISING SYSTEMS Romanova T V; Ushmarin N F; Fomin A G;

Gorelik R A

The product studied is obtained as a by-product of nylon 6 manufacture and consists of a mixture of epsiloncaprolactam, low-molecular cyclic and linear polyamides and products of their thermo-oxidative degradation and interaction with sodium hydroxide or other neutralising agents. Data are given on the effect of this secondary vulcanising accelerator on the vulcanisation properties and physico-mechanical properties of various rubbers. 3 refs. Articles from this journal can be requested for translation by subscribers to the Rapra produced International Polymer Science and Technology. RUSSIA

Accession no.700417

Item 209

*Kauchuk i Rezina (USSR)* No.5, 1994, p.16-8 Russian **NEW LOW-TEMPERATURE VULCANISING AGENT FOR ELASTOMERIC COATINGS** Boiko V V; Shirokova S Ya; Malaya N D; Medvedeva V I

The authors describe promising vulcanising agents for unsaturated elastomers, consisting of sterically hindered dinitrile dioxides of aromatic dicarboxylic acids, particularly mesitylene dinitrile dioxide (MDNO), which interacts with diene elastomers at 25-30 C to form a threedimensional network. This compound was studied as a vulcanising agent for elastomeric coatings for the development of a method of low-temperature vulcanisation of rubberised fabrics. It was found that this method of vulcanisation helps to reduce the energy costs in the preparation of rubberised materials. 6 refs. Articles from this journal can be requested for translation by subscribers to the Rapra produced International Polymer Science and Technology.

RUSSIA

# Item 210

## Kautchuk und Gummi Kunststoffe 51, No.10, Oct.1998, p.662/9 IMPROVING TREAD WEAR WITHOUT INCREASING HEAT BUILD-UP BY USING 1,3-BIS(CITRACON-IMIDOMETHYL)BENZENE Datta R N; Ingham F A A

The anti-reversion agent 1,3-bis(citraconimidomethyl) benzene (Perkalink 900) reduces heat build-up in vulcanisates under dynamic conditions. It functions by a crosslink compensation mechanism that maintains compound physical and dynamic properties on overcure or during service. The conventional approaches to improving tread wear - partial replacement of NR with BR, increased carbon black level or use of higher reinforcing blacks - lead to increased heat build-up. This has limited the application of these approaches in practice. However, by incorporating Perkalink 900 together with these compound modifications the increased heat build up can be prevented, thus allowing improvements in tread wear to be realised. This is shown by laboratory data. 27 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; NETHERLANDS; WESTERN EUROPE Accession no.699766

Item 211

*Elastomery* 2, No.1, 1998, p.27-33 Polish **RESEARCH DEVELOPMENT IN FIELD OF IMPROVEMENT OF ECOLOGY IN RUBBER INDUSTRY. I. ELIMINATION OF CAUSES OF N-NITROSAMINES FORMATION** Kleps T; Rajkiewicz M

STOMIL

Areas in which improvements have been attained in rubber manufacture with regard to ecology at the workplace and in contact with rubber goods are described. Some of the problems related to the carcinogenicity of N-nitrosamines formed during rubber processing are discussed. Methods proposed by several authors for elimination of the problems arising from N-nitrosamines are considered. The main trend is the use of 'safe accelerators' in place of the traditional amine accelerators used for diene rubber vulcanisation. New accelerators and chemicals for rubber processing are proposed. 31 refs. EASTERN EUROPE; POLAND

Accession no.699064

#### Item 212

*Chimica e l'industria* 79, No.5, June 1997, p.597-603 **FREE RADICAL POLYMERIC PHOTOINITIATORS FOR UV CURING** Angiolini L; Carlini C

## Bologna, Universita; Pisa, University

The photoinitiating activity of polymeric photoinitiators capable of promoting UV curing reactions is examined, and the advantages of such initiators over low molecular weight products are discussed. Polymeric photoinitiators bearing hydroxyalkylphenone, benzoin, benzophenone and diarylketone/amine moieties are reviewed. 34 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; ITALY; WESTERN EUROPE

Accession no.698927

#### Item 213

Patent Number: US 5736615 A 19980407 **RUBBER VULCANISATION COMPOSITION** D'Sidocky R M; Maly N A Goodyear Tire & Rubber Co.

Vulcanisates having superior physical properties and reversion resistance are produced using tetrabenzylthiuram disulphide in combination with a bismaleimide compound and a sulphenamide compound. USA

Accession no.694956

#### Item 214

Polymer Testing '97. Day 3: Chemical Analysis. Conference proceedings. Shawbury, 7th-11th April 1997, Paper 1. 9T **ELASTOMERS: SULPHUR OR PEROXIDE CROSSLINKED? AN ANALYTICAL APPROACH** Affolter S EMPA (Rapra Technology Ltd : Plastics & Pubber Weekly:

(Rapra Technology Ltd.; Plastics & Rubber Weekly; European Plastics News)

An overview of analytical tools for the characterisation of elastomeric materials, and especially crosslinking systems, is presented. It is based on available literature, experience and work in the laboratories. An attempt is made to establish the crosslinking system and the agents used for crosslinking elastomeric materials with high certainty. Analytical tools and their usefulness are discussed, and an attempt to answer the sulphur versus peroxide crosslinking question is presented. 25 refs.

SWITZERLAND; WESTERN EUROPE Accession no.694709

#### Item 215

**Rubber Chemistry and Technology** 71, No.2, May/June 1998, p.310-22 **NITROSAMINE FORMATION IN RUBBER. I. INFLUENCE OF MIXING HISTORY** Willoughby B G; Scott K W RAPRA Technology Ltd.

N-Nitrosodimethylamine formation was studied in a range of TMTD-accelerated, carbon black-filled rubber formulations including EPDM, SBR, NR and NBR rubbers. Two levels of TMTD and carbon black were variables in this study, as was the type of carbon black used (N330 and N550). The use of statistical experimental design showed that reliable data on nitrosamine formation in vulcanisates could be obtained by paying strict attention to process history. For the SBR, NR and NBR rubbers, a correlation was observed between the temp. reached on mixing and the level of nitrosamine generated on subsequent vulcanisation. For these rubbers, the effect of this process history outweighed that of any ingredient variable. While allowing new insights to be gained on nitrosamine formation in rubber, these results allow development of a self-consistent mechanism for nitrosation processes where the reactions in diene rubbers mirrored those in air. 14 refs. EUROPEAN COMMUNITY; EUROPEAN UNION; UK; WESTERN EUROPE

Accession no.692441

#### Item 216

#### *Revue Generale des Caoutchoucs et Plastiques* No.760, June/July 1997, p.83-6 French

# FLAME RETARDANT PROPERTIES OF EPDM COMPOUNDS

Krans J; Eichler H J Martinswerk GmbH; DSM Elastomers Europe

Results are presented of a study of the influence of different fillers, plasticiser oils and curing systems on the limiting oxygen index and smoke emission of EPDM compounds, and of flammability tests undertaken on roof coverings based on three different compounds. 5 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; GERMANY; NETHERLANDS; WESTERN EUROPE

Accession no.691336

#### Item 217

#### Patent Number: US 5710210 A 19980120 PROCESS FOR POLYMER REACTIONS WITH FUNCTIONALISED PEROXIDES Stein D L

Elf Atochem North America Inc.

Novel functionalised peroxides are described which may be used as crosslinking, grafting and curing agents, initiators for polymerisation reactions and as monomers for condensation polymerisations to form peroxy-containing polymers, which in turn can be used to prepare block and graft copolymers. The compounds, as represented by a given formula, may be prepared by reacting hydroxy-containing tertiary hydroperoxides with diacid halides, phosgene, dichloroformates, diisocyanates, anhydrides and lactones to form the functionalised peroxides. These reaction products may be further reacted, if desired, with dialcohols, diamines, aminoalcohols, epoxides, epoxy alcohols, epoxy amines, diacid halides, dichloroformates and diisocyanates to form additional functionalised peroxides.

USA

Accession no.688647

#### Item 218

### Patent Number: US 5684202 A 19971104 TERTIARY AMINES, A PROCESS FOR THEIR PREPARATION AND THEIR USE AS HARDENING ACCELERATORS Meier H M; Fischer W; Clemens H

Bayer AG

The present invention relates to novel tertiary amines derived from bisphenols, diepoxides and aromatic secondary amines, to a process for their preparation and to their use as hardening accelerators for ethylenically unsaturated cold hardenable acrylic and polyester resins. EUROPEAN COMMUNITY; EUROPEAN UNION; GERMANY; WESTERN EUROPE

Accession no.678064

#### Item 219

# Patent Number: US 5684091 A 19971104 SULPHUR VULCANISABLE RUBBER COMPOUND

Maly N A; McGilvrey J R Goodyear Tyre & Rubber Co.

This invention relates to the discovery that the combination of tetrabenzylthiuram disulphide, cashew nut oil modified novolak-type phenolic resin, a bismaleimide compound and a sulphenamide compound provides for excellent vulcanisation of rubbers and results in desirable final rubber vulcanisate physical properties in the absence of generating undesirable nitrosamines and fumes during processing and cure.

USA

Accession no.678025

#### Item 220

## *Journal of Macromolecular Science B* B37, No.2, 1998, p.135-42 **MECHANISM OF SULPHUR BEHAVIOUR IN RUBBER COMPOUNDS** Jurkowski B; Jurkowska B

Poznan, University of Technology

Details are given of the use of sulphur compounds in the vulcanisation process of rubbers. Emphasis is given to ways of overcoming surface bloom during storage. 20 refs.

EASTERN EUROPE; POLAND *Accession no.676729* 

#### Item 221

*International Polymer Science and Technology* 24, No.10, 1997, p.T/60-1 **NEW VULCANISATION ACCELERATOR FOR BUTADIENE-STYRENE RUBBERS** 

Yuldashev D Ya; Yusupbekov A Kh; Ibadullaev A; Negmatov S S Uzbek Fan Va Tarakkiet Technological Complex The effect of a new class of accelerators of the donoracceptor type - phosphated alkylamides of fatty acids on the kinetics of SBR and properties of the vulcanisates were studied, and compared with mixes of the same composition, but containing Altax (MBTS) as the accelerator. 7 refs. Translation of Kauchuk i Rezina, No.2, 1997, p.46

#### RUSSIA

Accession no.676672

### Item 222

International Polymer Science and Technology 24, No.11, 1997, p.T/53-4 COPOLYMERS OF GASEOUS SULPHUR AND DICYCLOPENTADIENE AS VULCANISING AGENTS FOR UNSATURATED RUBBERS

Okhotina N A; Liakumovich A G; Samuilov Y D; Avramenko Y P; Maksimov T V; Filippova A G; Yamalieva L N; Mannanova F G Kazan,State Technological University

A study was made of the conditions of copolymerisation of gaseous sulphur with dicyclopentadiene and its use as a vulcanising agent in the production of unsaturated rubbers. The mechanism of the copolymerisation is explained, and properties of rubber mixes of metal-cord breaker containing sulphur Kristeks and copolymeric sulphur are tabulated. 3 refs. (Full translation of KIZ, No.3, 1997, p.33)

Accession no.676327

#### Item 223

Patent Number: EP 829508 A1 19980318 SULPHUR VULCANISABLE RUBBER CONTAINING HYDRATED THIOSULPHATE D'Sidocky R M; Cowling T B Goodyear Tire & Rubber Co.

The vulcanisation rate of a sulphur rubber composition is increased by heating the rubber composition to a temperature ranging from 100 to 200C. The rubber composition contains a sulphur vulcanisable rubber, a sulphenamide compound and a hydrated thiosulphate, the latter two compounds significantly increasing the vulcanisation rate of the rubber.

USA

Accession no.672221

## Item 224

**Rubber Chemistry and Technology** 70, No.3, July/Aug.1997, p.368-429 **THIURAM- AND DITHIOCARBAMATE-ACCELERATED SULFUR VULCANIZATION FROM THE CHEMIST'S PERSPECTIVE; METHODS, MATERIALS AND MECHANISMS REVIEWED** 

Nieuwenhuizen P J; Reedijk J; Van Duin M; McGill W J

Leiden, University; DSM Research; Port Elizabeth, University

Research methodologies for the investigation of the mechanism of vulcanisation are described and the reactivity of thiuram and dithiocarbamate chemicals is discussed. The mechanism and chemistry of both thiuram- and dithiocarbamate-accelerated sulphur vulcanisation are then considered. The integration of the original mechanistic ideas from the 1960s and the results obtained in the past three decades are shown to lead to a more balanced appraisal of events during vulcanisation. Proposals for future research are made. 262 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; NETHERLANDS; SOUTH AFRICA; WESTERN EUROPE Accession no.671090

#### Item 225

*Revue Generale des Caoutchoucs et Plastiques* No.752, Sept.1996, p.69-72 French

LATEST GENERATION OF ACCELERATORS Rebours B; Krans J DSM France: DSM Electomete Europa

DSM France; DSM Elastomers Europe

Studies were made of nitrosamine formation by a number of accelerators in EPDM compounds, and of the effects of nitrosamine-free accelerators on cure characteristics and heat resistance and compression set of vulcanisates. The results showed the possibility of reducing cure times and compression set values through the use of combinations of different nitrosamine-free accelerators. EUROPEAN COMMUNITY; EUROPEAN UNION; FRANCE; NETHERLANDS; WESTERN EUROPE Accession no.670803

Item 226

#### Kautchuk und Gummi Kunststoffe

## 51, No.2, Feb.1998, p.83-7 NEW INTERPRETATION OF CROSSLINKING MECHANISM OF NR WITH DICUMYL PEROXIDE

Gonzalez L; Rodrigez A; Marcos A; Chamorro C

The peroxide curing of NR is described. At high temperature and high DCP loading, changes in the crosslinking mechanism are observed. The results confirm that the crosslink efficiency might be less than 1. Under these conditions both mechanisms, abstraction of an allylic hydrogen and addition to the double bond can take place. H1 NMR, fracture analysis and tensile stress data reveal that the compound with 3 phr DCP crosslinked at 116 deg.C presents a non-uniform distribution of the crosslinks in the network. 21 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; SPAIN; WESTERN EUROPE

# Item 227

*China Rubber Industry* 45, No.2, 1998, p.116-8

#### Chinese

#### **PROGRESS IN SULPHUR CURING OF RUBBER** Yuhua Y: Hai Z

12 refs. CHINA Accession no.670178

## Item 228

*China Rubber Industry* 45, No.2, 1998, p.67-71 Chinese

# STUDY ON NR REVERSION. I. CONVENTIONAL SULPHUR CURING SYSTEM Xiangfu Z; Yinxi Z; Yutang Z; Miao D

Shanghai, Jiao Tong University

A study is made of NR reversion. The inhibition effect of some commercial anti-recovery agents, such as, DHTS, HVA-2 and Si69, on NR reversion is investigated by determining curing curve, physical properties and swelling index of the anti-recovery agent-containing compounds. The test results show that HVA-2 is the most effective anti-recovery agent, DHTS is the second, and Si69 is the poorest; HVA-2 and DHTS improve the thermo-oxidative stability. It is found by analysing the curing curve that the sulphur curing reaction obeys the first order reaction low. 2 refs.

Accession no.670163

# Item 229

Rubber World 217, No.3, Dec.1997, p.39/44 EFFECT OF CROSSLINKING SYSTEMS ON FATIGUE LIFE Sommer J G

Elastech Inc.

This article describes the effect of changes in the crosslinking system on the fatigue life and other properties in natural rubber suspension bushings. The effect of sulphur/accelerator ratio was determined for several properties that included fatigue life, crystallisation, friction and creep. 5 refs.

USA

Accession no.668230

# Item 230

Patent Number: EP 823453 A1 19980211 RUBBER COMPOUND HAVING IMPROVED REVERSION RESISTANCE

D'Sidocky R M; Maly N A; Wideman L G Goodyear Tire & Rubber Co.

This is obtained using a combination of a bismaleimide compound and a bis benzothiazolyldithio end capped

compound, which provides for excellent vulcanisation of the rubber. USA Accession no.667925

# Item 231

**Rubber India** 49, No.11, Nov.1997, p.19-24 **THIADIAZOLES FOR CROSSLINKING HALOGEN-CONTAINING POLYMERS** Ohm R F Vanderbilt R.T.,Co.Inc.

2,4-Dimercapto-1,3,4-thiadiazole and its derivatives can be used to crosslink a wide variety of halogen-containing polymers. Successful use of the thiadiazole in a given polymer is seen to depend on the particular derivative and any associated accelerator. A review is presented of the various thiadiazoles for crosslinking halogencontaining polymers. 3 refs.

DUPONT DOW ELASTOMERS LLC USA

Accession no.666970

Item 232

**Rubber India** 49, No.11, Nov.1997, p.11-5 **OPTIMIZING TIRE COMPOUND REVERSION RESISTANCE WITHOUT SACRIFICING PERFORMANCE CHARACTERISTICS** Datta R N; Helt W F; Flexsys B V

The optimising of a tyre compound to improve reversion resistance is discussed with reference to the use of two new rubber chemicals developed by Flexsys. They are 1,3 bis(citraconimidomethyl benzene) (BMI-MX) and hexamethylene-1,6-bisthiosulphate disodium salt dihydrate (HTS), which are claimed to provide two different ways to improve the thermal ageing resistance of sulphur-based vulcanisates as compared to existing technology. Each functions by uniquely different mechanisms of action and each provides different levels of performance improvements. A comparison is made of HTS and BCI-MX to conventional cure systems and semiefficient cure systems to illustrate the potential benefits of these approaches to address reversion in tyre compounds. 5 refs.

FLEXSYS INDIA Accession no.666969

Item 233 European Polymer Journal 34, No.1, Jan.1998, p.51-7 EFFECT OF ACCELERATOR/SULPHUR RATIO ON THE SCORCH TIME OF EPOXIDIZED NATURAL RUBBER(ENR) Sadequl A M; Ishiaku U S; Ismail H; Poh B T Universiti Sains Malaysia

The Mooney scorch time of ENR was studied with SMR L NR as a reference. The effect of sulphur to accelerator ratio on scorch behaviour in the presence of the more common types of accelerators was investigated. Mooney scorch time was studied by using the Mooney Shearing Disk viscometer in the temp. range 100-180C. The Mooney scorch time for SMR L was longer than that for ENR, irrespective of the sulphur/accelerator ratio. This was attributed to the activation of double bonds by the adjacent epoxide groups in ENR. The accelerator dependence of Mooney scorch time of ENR and NR compounds tended to become less significant as temp. increased, while the influence of temp. decreased from the slow through the medium fast to the ultrafast accelerators. The Mooney scorch times of ENR and NR compounds depended on accelerator concentration, with the effect being more significant at lower curing temps. 22 refs.

MALAYSIA

Accession no.666730

# Item 234

**Polymer** 39, No.1, 1998, p.109-15 **RHEOLOGICAL PROPERTIES OF EPDM COMPOUND. EFFECT OF BLOWING AGENT, CURING AGENT AND CARBON BLACK FILLER** 

Guriya K C; Bhattachariya A K; Tripathy D K Indian Institute of Technology

Details are given of the rheological properties of EPDM compounds in extrusion containing a blowing agent. The cell morphology development and rheological properties were studied for gum and carbon black filled systems with variation of the blowing agent, extrusion temperature and shear rate. The effect of the curing agent and blowing agent on rheology was also studied. 15 refs.

INDIA

Accession no.663331

*Item 235 Elastomery* No.4, 1997, p.22-6 Polish **MODERN, NON-DUSTING CHEMICAL ADDITIVES FOR RUBBER** 

Rajkiewicz M; Berek I; Wilkonski P

Some modern forms of curing accelerators were investigated. The technology for the production of granulated composites of accelerators with a polymer binder is discussed. The effectiveness of granulated and powdered accelerators in rubber mixes was compared. 9 refs.

EASTERN EUROPE; POLAND

Accession no.663233

#### Item 236

International Polymer Science and Technology 24, No.6, 1997, p.T/22-3 USE OF TALL OIL FATTY ACIDS FOR THE SYNTHESIS OF HIGHER C18 FATTY ACIDS AS ADDITIVES TO VULCANISATES Chernova I K; Bychkov B N; Filimonova E I; Solov'ev V V Yaroslavl',State Technical University

The synthesis of higher C18 fatty acids (oleic, stearic) using a renewable, inedible feedstock, i.e. tall oil fatty acids (a by-product of wood processing of paper and pulp plants), is described. The effect on this process of amount of catalyst, hydrogen pressure, temp. and time of reaction is considered. The fatty acids are of use as activators of rubber mix vulcanisation. 1 ref. (Full translation of Kauch.i Rezina, No.6, 1996, p.39)

CIS; COMMONWEALTH OF INDEPENDENT STATES Accession no.663031

#### Item 237

*International Polymer Science and Technology* 24, No.6, 1997, p.T/20-1

INFLUENCE OF THE PHYSICOCHEMICAL CHARACTERISTICS OF STEARIC ACID ON THE PROPERTIES OF VULCANISATES

Pankratov V A; Lukanicheva V Y; Emel'yanov D P; Shashina I A

Yaroslavl', State Technical University; Yaroslavl Tyre Works

The relationship between the properties of vulcanisates and the iodine number of stearic acid in unfilled rubber mixes based on polyisoprene rubber and containing Sulphenamide Ts as the accelerator was investigated, using samples of stearic acid with different physicochemical characteristics. The presence of unsaturated fragments in stearic acid was found to be an undesirable factor. The unsaturation of stearic acid should be monitored carefully to avoid possible scorching of the mixes and lowering of the physicomechanical properties of the finished products. Stearic acid in rubber mixes acts as a disperser and a secondary activator. 3 refs. (Full translation of Kauch.i Rezina, No.6, 1996, p.37)

CIS; COMMONWEALTH OF INDEPENDENT STATES Accession no.663030

#### Item 238

*International Polymer Science and Technology* 24, No.6, 1997, p.T/16-9

METAL POLYSULPHIDES AS PROMISING FEEDSTOCK FOR THE PRODUCTION OF RUBBER MIX COMPONENTS

Maslosh V Z; Mikulenko L I; Strubchevskaya E M Rubezhnoe,Institute of Applied Chemistry

The suitability of alkali and alkaline earth metal sulphides and polysulphides as the initial feedstock for the production of rubber mix components was investigated. Tabulated data are presented on the effect of zinc sulphide on the properties of vulcanisates based on polyisoprene, nitrile rubber and a silicone rubber, the results of tests of a tread mix for radial lorry tyres with activator KV-2 (zinc sulphide-based) and the results of tests of thiazole and sulphenamide accelerators. 2 refs. (Full translation of Kauch.i Rezina, No.6, 1996, p.34)

CIS; COMMONWEALTH OF INDEPENDENT STATES; UKRAINE

Accession no.663029

#### Item 239

*International Polymer Science and Technology* 24, No.6, 1997, p.T/13-5

# STUDY OF THE ACCELERATOR DISULFAL' MG IN VULCANISATE FORMULATIONS FOR MECHANICAL RUBBER GOODS

Boiko V V; Gusarov B P; Boyarskaya N A; Medvedeva V I

Kiev, Elastic Scientific Research Institute

The possibility was demonstrated of using the accelerator Disulfal' MG as a replacement for the Altax or Sulphenamide Ts traditionally used in formulations of vulcanisates for mechanical rubber goods, provided that the appropriate adjustment was made to the composition of the vulcanising group and the vulcanisation time. 2 refs. (Full translation of Kauch.i Rezina, No.6, 1996, p.33)

CIS; COMMONWEALTH OF INDEPENDENT STATES; UKRAINE

Accession no.663028

#### Item 240

*International Polymer Science and Technology* 24, No.6, 1997, p.T/10-2

## PROPERTIES OF NATURAL AND GASEOUS GROUND SULPHUR FROM DIFFERENT PRODUCERS

Yalovaya L I; Frolikova V G Moscow,Scientific Research Institute of the Tyre Industry

The properties of natural ground sulphur from Turkmen and Ukraine are discussed and the production of ground sulphur from gaseous feedstock is described. The vulcanisation activity of sulphur from various producers is compared in model vulcanisates based on polyisoprene rubber and the physicomechanical properties of the vulcanisates are tabulated. (Full translation of Kauch.i Rezina, No.6, 1996, p.31)

RUSSIA

Accession no.663027

# Item 241

*Journal of Applied Polymer Science* 66, No.7, 14th Nov.1997, p.1231-6

## CURE CHARACTERISTICS OF ETHYLENE PROPYLENE DIENE RUBBER-POLYPROPYLENE BLENDS. I. CALCULATION OF STATE OF CURE IN BLENDS CONTAINING CONVENTIONAL SULFUR CURING SYSTEM UNDER VARIABLE TIME-TEMPERATURE CONDITIONS

Sengupta A; Konar B B Calcutta,University

Blends of EPDM and PP were prepared in which the rubber phases were compounded with an optimum sulphur curing system. The equivalent cure time and state of cure of the blends were studied by rheometry at temperatures between 120C and 190C. The activation energies of the crosslinking reaction were very similar for virgin EPDM and for the blends containing different proportions of EPDM and PP. However, there were differences in the induction period and the curing rate constant for the different blends. The temperature conversion factor of the blends was discussed. 18 refs.

INDIA

Accession no.660937

#### Item 242

Patent Number: EP 767205 A1 19970409 VULCANIZATION FREE OF REVERSION OF NATURAL AND SYNTHETIC RUBBER BY ACCELERATION WITH DITHIOPHOSPHATE Schafer V; Graf H-J; Schulz H; Steger L Rhein-Chemie Rheinau GmbH

Rubber mixtures which can be vulcanised without reversion are disclosed, containing natural and/or synthetic rubber together with (based on the amount of rubber) 0.1-10 wt.% of a dithiophosphoric acid polysulphide with 4-12C alkyl groups, 0.5-8 wt.% metal alkyl dithiophosphate and/or mercaptobenzthiazole and optionally conventional additives.

EUROPEAN COMMUNITY; EUROPEAN UNION; GERMANY; WESTERN EUROPE

Accession no.660529

#### Item 243

152nd ACS Rubber Division Meeting, Fall 1997. Conference Preprints. Cleveland, Oh., 21st-24th Oct.1997, Paper 108, pp.30. 012 **TETRAISOBUTYLTHIURAM MONOSULFIDE: SOME FURTHER APPLICATIONS OF THIS UNIQUE RETARDER/KICKER** Chasar D W Goodrich B.F.,Co. (ACS,Rubber Div.)

Tetraisobutylthiuram monosulphide (TiBTM) was used as a cure retarder and secondary accelerator (kicker) with benzothiazole sulphenamide accelerators in the vulcanisation of NR and SBR/polybutadiene compounds. Increases in cure temperature, various levels of sulphur and the presence of silica did not affect the ability of TiBTM to enhance the cure rates and scorch times of the sulphenamide accelerators. Trends in the mechanical properties of vulcanisates were similar to those occurring in straight sulphenamide cured systems. The reversion resistance of NR vulcanisates was improved by TiBTM. 6 refs.

#### USA

Accession no.659551

## Item 244

152nd ACS Rubber Division Meeting, Fall 1997. Conference Preprints. Cleveland, Oh., 21st-24th Oct.1997, Paper 107, pp.26. 012 FORMULATING FOR OPTIMIZED PROPERTIES USING REVERSION RESISTANCE CHEMICALS Ignatz-Hoover F; Maender O; Datta R N

Flexsys America LP; Flexsys BV (ACS,Rubber Div.)

A combination of two anti-reversion agents, biscitraconimidomethylbenzene and hexamethylene-1,6bisthiosulphate disodium dihydrate, was used to improve the reversion resistance and tensile and tear properties of sulphur vulcanised NR compounds. Experimental design and response surface methodology were used to arrive at a formulation providing an optimum balance of properties for tyre applications. Vulcanisates having excellent retention of modulus on overcure or high temperature cure were obtained. Tear strength was maintained even at high retention of modulus in room temperature and hot tear tests. 5 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; NETHERLANDS; USA; WESTERN EUROPE Accession no.659550

## Item 245

152nd ACS Rubber Division Meeting, Fall 1997. Conference Preprints.

Cleveland, Oh., 21st-24th Oct. 1997, Paper 106, pp.45. 012 IMPROVED REVERSION RESISTANCE BY USING DITHIOPHOSPHATE VULCANIZATION SYSTEMS IN THICK-SECTION MOLDED PARTS BASED ON NR

Johansson A H; Frueh T

Rhein Chemie Corp.; Rhein Chemie Rheinau GmbH (ACS,Rubber Div.)

Rhenocure SDT (Rhein Chemie), a dialkyl dithiophosphate polysulphide sulphur donor, was evaluated in curing systems for the vulcanisation of thick NR parts. It was found that a curing system based on a dithiophosphate accelerator and the dithiophosphate sulphur donor was superior to a system based on a sulphenamide and dithiodimorpholine. A substantially higher amount of short sulphur bridges was generated, leading to improved reversion resistance and superior heat ageing properties. The dithiophosphate system allowed higher curing temperatures and shorter cure times while maintaining the desired mechanical properties. 5 refs. EUROPEAN COMMUNITY; EUROPEAN UNION; GERMANY; USA; WESTERN EUROPE Accession no.659549

#### Item 246

IRC '97. Conference proceedings. Kuala Lumpur, 6th-9th Oct.1997, p.382-6. 012 NEW CURING SYSTEM OF EPOXIDISED NATURAL RUBBER (ENR) BY DI-O-TOLYLGUANIDINE SALT OF DICATECHOL BORATE

Nishi K; Kodama S; Kawasaki H Okayama Prefecture Industrial Technology Center (Rubber Research Institute of Malaysia)

The curing behaviour of epoxidised NR (ENR) with diorthotolylguanidine salt of dicatechol borate (DTGS), and the physical properties of the vulcanisate, are investigated. The vulcanisate, which shows better tensile properties, lower resilience and better performance of compression set than peroxide-cured ENR, is obtained without other curing agent. The vulcanisate containing high amount of DTGS is found to be more rigid than that of the sulphurcured ENR at room temperature, because its glass transition temperature (Tg) is higher than room temperature. This suggests that the crosslinking reaction involving the ringopening reaction of epoxy group in the rubber leads to an increase in the Tg of the vulcanisate. 6 refs.

JAPAN

Accession no.658907

## Item 247

IRC '97. Conference proceedings. Kuala Lumpur, 6th-9th Oct.1997, p.379-81. 012 **ROLE OF STEARIC ACID IN ACCELERATED SULPHUR VULCANISATION** McGill W J; Mallon P E; Shelver S R Port Elizabeth, University (Rubber Research Institute of Malaysia)

It is shown that the role of zinc stearate in vulcanisation formulations is to promote the reaction of accelerator terminated polysulphidic pendant groups with neighbouring polymer chains, thereby resulting in higher crosslink densities. Cyclisation reactions may also be reduced, thus increasing stress induced crystallisation under load and thereby further enhancing the physical properties of the vulcanisate. 6 refs.

SOUTH AFRICA

Accession no.658906

*Item 248* IRC '97. Conference proceedings. Kuala Lumpur, 6th-9th Oct.1997, p.65-70. 012

## EFFECT OF DICUMYL PEROXIDE ON MECHANICAL STABILITY TIME OF CONCENTRATE LATEX

Dalimunthe R PT Perkebunan Nusantara XIII (Rubber Research Institute of Malaysia)

It has previously been shown that a hydrophobic monomer, styrene, added to NR latex for modification purposes by means of graft copolymerisation, decreases the colloidal stability of latex. Reports on the preparation of prevulcanised latex show that low molecular weight hydrophobic vulcanising agents have also been added to the latex. An attempt has been made to avoid the possibility of formation of volatile N-nitrosamines as well as nitrosatable amines in the rubber products. However, no report of the effect of a hydrophobic vulcanising agent upon the colloid stability of latex has been published, despite the fact that such vulcanising agents may decrease or increase the colloidal stability of latex, in turn affecting the crosslinking reaction during the preparation of the prevulcanised latex. Dicumyl peroxide is one hydrophobic vulcanising agent that can be used for the preparation of prevulcanised latex in the absence of conventional accelerators. It is shown to decrease significantly the mechanical stability time of latex brought about by swelling of rubber particles. However, the solution of dicumyl peroxide in palm kernel oil increases significantly the MST of latex brought about by hydration stabilisation effect of palm kernel oil. 7 refs.

INDONESIA

Accession no.658867

## Item 249

IRC '97. Conference proceedings. Kuala Lumpur, 6th-9th Oct.1997, p.29-39. 012 SYNTHESIS AND CHARACTERISATION OF MACROMONOMER CROSSLINKER: TOWARDS STUDY OF EFFECT OF CROSSLINKER ON LATEX FILM FORMATION Ghazaly H M; Daniels E S; Dimonie V L; Klein A; El-Aasser M S Rubber Research Institute of Malaysia; Lehigh,University (Rubber Research Institute of Malaysia)

A macromonomer crosslinker with a number average molecular weight, Mn~3300 +/- 100 g/mol (obtained from gel permeation chromatography) is synthesised from a hydroxy-terminated, saturated ethylene-butylene polymer by reaction with acrylic acid. Preservation of the double bonds is achieved by inhibiting the reaction utilising methyl hydroquinone with nitrobenzene and oxygen or t-butylcatechol and oxygen. Purification of the reacted mixture with aqueous sodium hydroxide or solid sodium bicarbonate gives similar products. Characterisation of the macromonomer crosslinker and comparison with the diol precursor are made using dilute solution viscosity, gel permeation chromatography, Fourier transform IR spectroscopy and 1H NMR spectroscopy. The crosslinking ability is verified by the formation of an insoluble gel of the macromonomer in solution homopolymerisations and also in solution copolymerisations with a primary monomer, n-butyl methacrylate in the presence of 2,2'-azobis(isobutyro-nitrile) (AIBN) at 700 deg.C. 17 refs.

MALAYSIA; USA Accession no.658863

## Item 250

Journal of Applied Polymer Science 66, No.6, 7th Nov.1997, p.1093-9 REACTIONS OF 2-(4-MORPHOLINOTHIO)-AND 2-(4-MORPHOLINODITHIO)BENZO-THIAZOLE(MOR AND MDB) IN THE PRESENCE OF POLYISOPRENE Gradwell M H S; Hendrikse K G; McGill W J Port Elizabeth,University

MOR and MDB were reacted, in combination with sulphur and zinc oxide, in the presence of polyisoprene. Samples were heated in a DSC at 2.5C/min and characterised by swelling experiments. The products formed at various temps. were analysed by highperformance liquid chromatography. Crosslinking only occurred once all the benzothiazole sulphenamide had been consumed, the onset of vulcanisation being characterised by a significant increase in 2mercaptobenzothiazole concentration. Crosslinking occurred earlier in all corresponding MDB formulations. Higher crosslink densities were recorded with addition of zinc oxide. The delayed action experienced in MOR systems was attributed to an exchange reaction between benzothiazole-terminated pendent groups and MOR and not due to the stability of the disulphide, MDB. 18 refs. SOUTH AFRICA

Accession no.658671

## Item 251

IRC '97. Conference proceedings. Kuala Lumpur, 6th-9th Oct.1997, p.427-35. 012 IMPROVING TREADWEAR WITHOUT INCREASING HEAT BUILD-UP BY USING 1,3-BIS(CITRACONIMIDOMETHYL)BENZENE (PERKALINK 900) Datta R N; Ingham F A A

Flexsys BV

(Rubber Research Institute of Malaysia)

The anti-reversion agent 1,3-bis(citraconimidomethyl) benzene (Perkalink 900) reacts by a unique crosslink compensation mechanism. In doing so, the crosslink density of vulcanisates can be maintained during overcure or during ageing as encountered during service life. As a result, the mechanical properties are stabilised, thereby controlling heat build-up. The anti-reversion effect of Perkalink 900 can be applied in tread compounds in which improved abrasion resistance is sought through the incorporation of an increased level of black or addition of a polymer of lower Tg (e.g. polybutadiene). Under normal circumstances, excessive heat build-up during service conditions may prohibit the application of this compound modification. However, heat build-up can be controlled by incorporating Perkalink 900, thereby achieving the desired effect of improved tread wear with no penalty of increased heat build-up. The effect of Perkalink 900 on model NR tread compounds is described. Cure characteristics, mechanical, dynamic mechanical and flex properties are illustrated together with laboratory abrasion data. 11 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; NETHERLANDS; WESTERN EUROPE

Accession no.658319

## Item 252

IRC '97. Conference proceedings. Kuala Lumpur, 6th-9th Oct.1997, p.408-12. 012 SULPHUR/DICYCLOPENTADIENE COPOLYMERS AS VULCANISING AGENTS FOR RUBBER

Liakumovich A G; Samuilov G D; Ohotina N A; Filippova A G

Kazan, State Technological University (Rubber Research Institute of Malaysia)

The copolymerisation of sulphur with dicyclopentadiene (DCPD) in the presence of an agent which regulates the structure of the copolymer is studied. Sulphur/DCPD copolymers are tested as vulcanising agents in wire coating stock. 12 refs.

RUSSIA

Accession no.658317

## Item 253

IRC '97. Conference proceedings. Kuala Lumpur, 6th-9th Oct.1997, p.403-7. 012 BIS(DIALKYL)THIOPHOSPHORYL SULPHIDE AS ACCELERATOR

Kodama S; Nishi Kawasaki H Edited by: Okayama,Industrial Technology Center; Nippon Shokusei Co.Ltd. (Rubber Research Institute of Malaysia)

The curing acceleration effect of bis(dialkyl) thiophosphoryl sulphide (DaPDS) are investigated. The order of acceleration effect is ethyl, butyl, 2-ethylhexyl. Ethyl and butyl derivatives indicate the function of a sulphur donor. It is assumed that the active species is the zinc salt of each DaPDS. In contrast with the conventional curing system, curing of the DaPDS/S curing system is activated by silica. Physical properties of silica-filled vulcanisates are superior to those of conventional curing systems. 4 refs.

JAPAN Accession no.658316

#### Item 254

IRC '97. Conference proceedings. Kuala Lumpur, 6th-9th Oct.1997, p.393-402. 012 USING DITHIOPHOSPHATE VULCANISATION SYSTEMS TO ACHIEVE REVERSION RESISTANCE IN THICK-SECTION MOULDED PARTS BASED ON NR Frueh T; Issel H M; Dinges U

Rhein Chemie Rheinau GmbH; Bayer AG (Rubber Research Institute of Malaysia)

The use of accelerator systems offering protection against reversion ensures that thick section parts have a more homogeneous crosslink density and therefore have more uniform properties over the cross section of a product. This is investigated experimentally as well as computationally based on mathematical models. It is shown that vulcanisation systems containing dithiophosphates in combination with mercaptobenzothiazole accelerators do not produce any signs of reversion in NR at temperatures up to 190 deg.C. Different vulcanisation systems can be used in thicksection articles, to demonstrate that network structure and density over the cross-section of an article vary depending on the applied vulcanisation system. Curing efficiency is considerably affected by the impact of reversion. Vulcanisation systems based on dithiophosphates show less fluctuation in network structure and density over the cross-section of the article. A comparison of different methods shows that dithiophosphate-cured elastomers perform particularly well in terms of reversion resistance as indicated by only slight variations of modulus 300, tensile strength, hardness shore A and compression set after prolonged curing at 170 and 180 deg.C. 11 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; GERMANY; WESTERN EUROPE

Accession no.658315

## Item 255

152nd ACS Rubber Division Meeting, Fall 1997.
Conference Preprints.
Cleveland, Oh., 21st-24th Oct.1997, Paper 28, pp.29.
012
VULKALENT E: AN EXAMINATION OF ITS

## USEFULNESS AS RETARDER AND ACCELERATOR OF SULFUR VULCANIZATION Moakes C A Bayer AG

(ACS,Rubber Div.)

A study is made of the retarding and accelerating activity of Bayer's Vulkalent E, a sulphonamide derivative, in the vulcanisation of a number of rubber compounds, and of its effects on the mechanical properties of vulcanisates. It is shown that this additive acts as an effective retarder of scorch in several cure systems, and that in most cases it combines the function of scorch retarding at both processing and moulding temperatures with secondary acceleration, which can give improved modulus and compression set values.

EUROPEAN COMMUNITY; EUROPEAN UNION; GERMANY; USA; WESTERN EUROPE Accession no.658282

*Item 256* **Rubber Technology International** 1997, p.64-8 **THIADIAZOLES FOR CROSSLINKING HALOGEN-CONTAINING POLYMERS** Ohm R F Vanderbilt R.T.,Co.

Di- and poly-functional mercaptans have been used for many years to crosslink polymers with mobile halogens (chlorine or bromine). 2,5-Dimercapto-7,3,4-thiodiazole (DMTD) is one such crosslinking agent that displays relatively fast cures but poor scorch safety. Improved scorch safety can be achieved by derivatising one or both mercapto groups. Several derivatives of DMTD have been developed in recent years for this purpose. Proper selection of the thiodiazole derivative and associated accelerator is often a key to successful use in a given halogen-containing polymer compound. 3 refs. USA

Accession no.657643

## Item 257

Patent Number: US 5616279 A 19970401 RUBBER VULCANISATION COMPOSITION CONTAINING TETRABENZYLTHIURAM DISULPHIDE, A BISMALEIMIDE, A SULPHENAMIDE COMPOUND AND SULPHUR, A SULPHUR DONOR OR MIXTURES THEREOF D'Sidocky R M; Maly N A

Goodyear Tire & Rubber Co.

Rubber vulcanisates obtained using the above compounds exhibit superior physical properties and reversion resistance.

USA

Accession no.657314

## Item 258

*International Polymer Science and Technology* 24, No.4, 1997, p.T/1-11

#### **REVERSION RESISTANCE OF NATURAL RUBBER MIXES WITH USE OF DITHIOPHOSPHATE CURING SYSTEMS**

Graf H J; Johansson A J Rhein-Chemie Rheinau GmbH; Rhein Chemie Corp.

NR cured with standard accelerators shows signs of reversion when exposed to heat ageing. The addition of an organic acid in addition to stearic acid decreases this tendency towards reversion. Different types of accelerator cause different degrees of crosslink degradation which may be determined by a rheometer or by modulus measurements after heat ageing. It appears that amines, which are formed during the vulcanisation process as the decomposition products of accelerators, are able to catalyse this reversion. If this is the case, the reversion should be much reduced by the use of accelerators which do not release amines during the curing process. 9 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; GERMANY; USA; WESTERN EUROPE Accession no.657011

Item 259 **Rubber and Plastics News** 26, No.23, 30th June 1997, p.30-2 **ACRYLIC COAGENTS FOR RADIATOR HOSE USE** Burak L; Nagel W

Sartomer Co.

It is explained that acrylic coagents can improve processability and vulcanisation of elastomers used in automotive applications, and that "Saret" coagents from Sartomer Co. of the USA provide scorch-retarding properties along with improved radiator hose properties for all curing situations. This article describes tests comparing several coagents in ethylene propylene diene monomer radiator hose formulations. Results are presented and discussed.

USA

Accession no.653452

Item 260

**Rubber World** 216, No.5, Aug.1997, p.24-7 **OPTIMISING TYRE COMPOUND REVERSION RESISTANCE WITHOUT SACRIFICING PERFORMANCE CHARACTERISTICS** Datta R N; Helt W F Flexsys BV

Reversion is the thermal degradation of sulphidic crosslinks leading to a reduction of crosslink density, a change in the distribution of crosslink types and an introduction of main-chain modifications. It leads to a decline in compound physical properties such as modulus, tensile and elongation, and in performance characteristics such as tear, fatigue and hysteresis. Over the last few years, Flexsys has introduced two new rubber chemicals which provide two different ways to improve the thermal ageing resistance of sulphur-based vulcanisates as compared to existing technology. 5 refs.

BELGIUM; EUROPEAN COMMUNITY; EUROPEAN UNION; WESTERN EUROPE

Accession no.652374

*Item 261* Patent Number: EP 794219 A1 19970910

#### DIHYDROPYRIDINES AS VULCANISATION ACCELERATORS FOR NATURAL OR SYNTHETIC RUBBERS

Broussard F; Adovasio M; Roncalli J Great Lakes Chemical Italia Srl

#### A formula for these accelerators is given.

EUROPEAN COMMUNITY; EUROPEAN UNION; ITALY; WESTERN EUROPE Accession no.651281

Item 262

*China Rubber Industry* 44, No.8, 1997, p.462-7 Chinese

## HIGH-TEMPERATURE STABLE INSOLUBLE SULFUR IS-HS

Pu Qijun

Beijing, Research & Design Inst. of Rubber Ind.

New types of insoluble sulphur, IS-HS, are described. The preparation of insoluble sulphur and the physicochemical causes of its reversion to soluble sulphur are discussed based on sulphur chemistry and the polymerisation mechanism of rhombic sulphur. The stability of new IS-HS products which have been treated with high-temp. stabiliser (PEF) is shown to reach over 90% after exposure at 105C for 25 min. The stability of conventional insoluble sulphur is only about 57%. The effect of basic compounds on the stability is considered. The effect of amines on reversion is shown to decrease in the order diphenylguanidine accelerators, sulphenamides, thiurams, thiazoles, ketoamine antioxidants, terephthalic diamines, naphthylamines. Commonly-used bi-, tri- or tetravalent metal oxides have no significant effect on the stability. A method for predicting the possibility of blooming of IS-containing compounds in the formula design is proposed. CHINA

Accession no.650489

## Item 263

Rubber Chemistry and Technology 70, No.1, March/April 1997, p.129-45 BISCITRACONIMIDES AS ANTI-REVERSION AGENTS FOR DIENE RUBBERS: SPECTROSCOPIC STUDIES ON CITRACONIMIDE-SQUALENE ADDUCTS Datta R N; Schotman A H M; Weber A J M; van Wijk F G H; van Haeren P J C; Hofsraat J W; Talma A G; Bovenkamp-Bouwman A G V D Flexsys BV; Akzo Nobel Central Research

The mechanism of anti-reversion activity of biscitraconimides during rubber vulcanisation was investigated. The products were identified as Diels-Alder adducts of conjugated polyenes formed during reversion with citraconimide. The NMR spectroscopic assignments of the products are discussed. 16 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; NETHERLANDS; WESTERN EUROPE Accession no.649667

## Item 264

## Rubber Chemistry and Technology 70, No.1, March/April 1997, p.120-8 VULCANISATION OF ULTRASONICALLY DEVULCANISED SBR ELASTOMERS Levin V Y; Kim S H; Isayev A I Akron,University

Sulphur cured SBR elastomers devulcanised by high power ultrasonic irradiation, were revulcanised using the same curing conditions as for virgin SBR. Crosslink density and gel fraction of virgin, devulcanised, and revulcanised SBR were investigated. 21 refs.

USA

Accession no.649666

## Item 265

## **Rubber Chemistry and Technology** 70, No.1, March/April 1997, p.106-19 **SULPHUR VULCANISATION OF SIMPLE MODEL OLEFINS. V. DOUBLE BOND ISOMERISATION DURING ACCELERATED SULPHUR VULCANISATION AS STUDIED BY MODEL OLEFINS**

Versloot P; Haasnoot J G; Nieuwenhuizen P J; Reedijk J; van Duin M; Put J Leiden,Institute of Chemistry; DSM

The sulphur vulcanisation of unsaturated rubber was

studied with the use of various olefins as simple, lowmolecular models. The position of the double bond was determined by crosslink formation mechanisms and isomerisation. 21 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; NETHERLANDS; WESTERN EUROPE Accession no.649665

## Item 266

*Iranian Polymer Journal* 6, No.2, April 1997, p.97-104 **EFFECT OF PALM OIL FATTY ACID ON CURING CHARACTERISTICS AND MECHANICAL PROPERTIES OF CALCIUM CARBONATE-FILLED NATURAL RUBBER COMPOUNDS** 

Ismail H; Ruhaizat T A Universiti Sains Malaysia

Results obtained from studies of the above showed that the scorch time and t90 increased with increasing concentration of palm oil fatty acid, while the maximum torque-minimum torque passed through a maximum value and decreased with acid concentration. Mechanical properties such as tensile modulus, hardness, TS and tear strength followed a similar trend. Reversion studies indicated that incorporation of palm oil fatty acid improved the resistance to reversion of the NR compounds. 16 refs. MALAYSIA

Accession no.648250

## Item 267

*Journal of Materials Science* 32, No.14, 15th July 1997, p.3717-25 EFFECT OF 1,3-BIS(CITRACONIMIDOMETHYL)BENZENE ON THE AEROBIC AND ANAEROBIC AGEING OF DIENE RUBBER VULCANIZATES

Kumar N R; Chandra A K; Mukhopadhyay R Hari Shankar Singhania Elastomer & Tyre Res.Inst.

The effect of the above antireversion agent (Perkalink 900) was investigated in diene rubber vulcanisates in truck-tyre tread-cap and tread-base compounds. Cure characteristics were studied at 141 and 193C. Retention of physical properties after anaerobic and aerobic ageing was found to be higher for the compounds with Perkalink 900. Heat build-up characteristics measured using a Goodrich Flexometer and Martin's ball fatigue tester were found to be lower for the Perkalink 900 compounds. Dynamic mechanical properties (tan delta) were reduced for the compounds containing Perkalink 900 even after extended cure. Fatigue to failure properties and abrasion resistance remained unaffected with the addition of Perkalink 900 in the compounds. The observed improvements in tyre-tread compound performance supported the previously-published mechanism of Perkalink 900. 19 refs.

FLEXSYS BV INDIA

Accession no.647749

## Item 268

Journal of Applied Polymer Science 65, No.3, 18th July 1997, p.499-505 THERMOMECHANICAL STUDY OF MOLECULAR AND TOPOLOGICAL STRUCTURE OF DIFFERENT KINDS OF SULFUR

Olkhov Y; Kurkowski B Russian Academy of Sciences; Poznan, University of Technology

A new method based on thermomechanical analysis of solid (not dissolved) polymer was used to study the topology and molecular structure of different kinds of sulphur (mineral and two polymeric types), commonly used as vulcanising agents for diene rubbers. It was found that a significant difference existed in their MWDs and in their degrees of crystallinity. Both mineral and polymeric sulphurs had a polymeric nature. 9 refs.

EASTERN EUROPE; POLAND; RUSSIA

Accession no.645667

Item 269 Luntai Gongye 17, No.7, 1997, p.400-404 Chinese COMPARISON BETWEEN PROPERTIES OF ACCELERATOR NS MADE IN CHINA AND IMPORTED ONE Xunning J; Fushui Y; Jing X

Rongcheng Guotai Tire Corp.Ltd.

A comparative test is carried out on the chemicophysical nature of accelerator NS made in China and an imported one, and their in-rubber physical properties and curing characteristics. The results show that the chemicophysical nature and the properties in service of accelerator NS made in China are similar to those of the import and it is suitable to replace the latter. It is concluded that the accelerator NS made in China needs to be improved by the following measures: to lower the ash content and to increase the purity; to develop the granular product; and to decrease the cost and to increase the stability of product quality. CHINA

Accession no.645250

Item 270

Patent Number: US 5594052 A 19970114 SULPHUR VULCANISABLE RUBBER CONTAINING SODIUM THIOSULPHATE PENTAHYDRATE

D'Sidocky R M; Futamura S Goodyear Tire & Rubber Co.

A method is disclosed for increasing the rate of vulcanisation of a sulphur rubber composition comprising heating a sulphur vulcanisable rubber composition to a temperature ranging from 100-200C; the rubber composition contains a sulphur vulcanisable rubber, a sulphenamide compound and a hydrated thiosulphate. Addition of the hydrated thiosulphate to a sulphur vulcanisable rubber and a sulphenamide compound significantly increases the rate of vulcanisation of the rubber.

USA

Accession no.642847

## Item 271

Patent Number: EP 785229 A1 19970723 SCORCH RESISTANT PEROXIDIC COMPOSITIONS

Sempio C; Angeloni G; Cesana M; Abrigo L Elf Atochem Italia Srl

Peroxidic compositions, which are useful for curing elastomers and polyolefins, comprise (A) a peroxide in the form of a powder, granules or a masterbatch and (B) + (C) an inhibitor derived from hydroquinone and a curing promoter in powder, granule or masterbatch form. EUROPEAN COMMUNITY; EUROPEAN UNION; ITALY;

EUROPEAN COMMUNITY; EUROPEAN UNION; ITALY; WESTERN EUROPE

Item 272

## *Journal of Applied Polymer Science* 64, No.13, 27th June 1997, p.2623-30 **SOME INVESTIGATIONS ON STRUCTURE-PROPERTY RELATIONSHIP IN XNBR VULCANIZATES IN THE PRESENCE OF BIS(DIISOPROPYL**

THIOPHOSPHORYL)DISULFIDE

Biswas T; Naskar N; Basu D K Indian Association for the Cultivation of Science

A new type of sulphidic crosslink arising from reaction between carboxylated nitrile rubber(XNBR) and bis(diisopropyl)thiophosphoryl disulphide was shown to be capable of improving the mechanical properties of the XNBR vulcanisates. It was also found that these sulphidic crosslinks in combination with metallocarboxylate crosslinks produced a significant effect in XNBR vulcanisation. Some investigations were carried out using a methyl iodide probe to elucidate the formation of chemical linkages during cure. Attempts were also made to establish the relationship between the structure and physical properties of the XNBR vulcanisates. 18 refs. INDIA

Accession no.640206

## Item 273

151st ACS Rubber Division Meeting, Spring 1997, Conference Preprints. Anaheim, Ca., 6th-9th May 1997, Paper 92, pp.38. 012 **NEW PEROXIDE FORMULATIONS FOR CROSSLINKING CHLORINATED POLYETHYLENE, SILICONE, FLUOROELASTOMER AND POLYETHYLENE CO- AND TERPOLYMER TYPE ELASTOMERS** Palys L H; Callais P A; Novits M F; Moskal M G Elf Atochem North America Inc. (ACS.Rubber Div.)

The crosslinking performance of scorch resistant, high performance (HP) peroxide formulations was examined and compared with that of conventional organic peroxides in a variety of elastomers, including EPDM, ethyleneoctene copolymers, chlorinated and chlorosulphonated PE, silicone rubber, fluoroelastomers and hydrogenated nitrile rubber. It was shown the HP peroxide systems gave increased processing time, i.e. a longer scorch time during the earlier stages of the cure profile followed by a conventional crosslinking step. Moving die rheometer and Mooney viscometer data indicated that HP formulations provided a wider processing window with the potential for improved productivity for all the elastomers studied. Tensile properties and compression set were equivalent to or better than those of elastomers crosslinked with conventional peroxides. 25 refs.

USA

Accession no.639069

#### Item 274

#### *Rubber World* 216, No.2, May 1997, p.40/50 ACCELERATOR ACTIVITY OF TBAB IN NR VULCANISATION

Purohit V; Chandra A K; Mukhopadhyay R; Mathur R Hari Shankar Singhania Elastomer & Tyre Res.Inst.; Sukhadia,University

Activity of quaternary ammonium salts in acceleration of two phase organic reactions is well documented. In order to study the behaviour of variations of these salts in vulcanisation of NR, triethylbenzylammonium chloride (TBAB) was selected for estimation of its accelerator activity. The cure characteristics and physical properties for several mixes revealed that TBAB possesses accelerator activity comparable with N-oxybenz thiazyl sulphenamide. 9 refs.

INDIA

Accession no.638596

Item 275

Patent Number: US 5569716 A 19961029 RUBBER COMPOSITION

Okamoto K; Inoue K; Kutuno T Idemitsu Petrochemical Co.Ltd.

A rubber composition is claimed composed of a hydrocarbon rubber, a vulcanising agent and/or crosslinking agent, and a hydrogenated petroleum resin having a bromine number not greater than 10 (g/100 g). The rubber composition exhibits improved processability over hydrocarbon rubber used alone but has no bad influence on the physical properties and heat resistance of its vulcanisate. It is free from any trouble involved in crosslinking with a peroxide which is often used for EPR and EPDM. Therefore, it will find general use in the rubber industry.

JAPAN

Accession no.638171

Item 276

Kautchuk und Gummi Kunststoffe 50, No.5, 1997, p.386-91 German ISOTHIOCYANATES FORMED DURING ACCELERATED VULCANISATION. I. THIURAM- AND DITHIO-CARBAMATE-ACCELERATORS

Kretzschmar H-J; Weigel H

Reaction products formed during accelerated vulcanisation were analysed by gas chromatography combined with mass spectrometry. The reduction of hazardous components of fumes by small changes to either the formulation or processing conditions are discussed. 6 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; GERMANY; WESTERN EUROPE

## *Item 277 Rubber World* 215, No.6, March 1997, p.33-8 *IMPROVED CURING SYSTEM FOR CHLORINE-CONTAINING POLYMERS* Ohm R F; Taylor T C

Vanderbilt R.T., Co.Inc.; DuPont Dow Elastomers

This article describes the performance of a new curative for polychloroprene, a derivative of 2,5-dimercapto-1,3,4-thiadiazole (DMTD). This curative provides long Mooney scorch times, rapid cure rate resulting in short cure times and an exceptionally flat cure plateau. Other performance characteristics are presented, including the effect of other additives in a typical polychloroprene formulation. 5 refs.

USA

Accession no.636321

*Item 278 Tire Technology International* 1997, p.120-22 FACTORS AFFECTING STABILITY OF SULFENAX CBS

Istrochem AS, Rubber Chemical Div.

CBS accelerators are one of the substitutes for morpholintype sulphenamide accelerators. This comprehensive article supplies details of ongoing research into factors that can affect the thermal stability of this type of accelerator during storage and transportation and identifies the most important influences that need to be taken into account.

SLOVAK REPUBLIC; SLOVAKIA

Accession no.636138

## Item 279

*Tire Technology International* 1997, p.114-17 **POLSINEX - AN INSOLUBLE SULPHUR WITH HIGH THERMAL STABILITY** Glijer T; Pysklow L; Parasiewicz W Stomil Rubber Research Institute

This comprehensive article supplies detailed results of a study evaluating a new insoluble sulphur, Polsinex, compared to conventional currently available insoluble sulphur. Information is included on the procedures of the study and focuses on the findings, which show that Polsinex exhibits high thermal stability and very good dispersibility. 7 refs.

EASTERN EUROPE; POLAND *Accession no.636136* 

## Item 280

151st ACS Rubber Division Meeting, Spring 1997, Conference Preprints. Anaheim, Ca., 6th-9th May 1997, Paper 17, pp.29. 012

## **EFFECT OF PRECIPITATED SILICAS IN TRUCK TIRE TREAD APPLICATIONS** Bice J A E; Patkar S D; Okel T A

PPG Industries Inc. (ACS,Rubber Div.)

The relative influence of various silicas and their levels on the balance of compound cure and mechanical and dynamic properties in an NR truck tyre tread formulation was investigated. The degree to which this balance was affected by the perturbation of the cure system by silica surface area and loading was examined, and the relative influence of silane coupling agent and CBS accelerator level was determined. Increasing the accelerator level with increasing silica surface area and content diminished the effect of the coupling agent on cure related compound properties over the range studied. 23 refs.

USA

Accession no.636031

## Item 281

151st ACS Rubber Division Meeting, Spring 1997, Conference Preprints. Anaheim, Ca., 6th-9th May 1997, Paper 16, pp.21. 012 **DEVELOPMENTS IN IMPROVED PERFORMANCE BROMOBUTYL RUBBERS** Sumner A J M; Kelbch S A; Verbiest A Bayer AG (ACS,Rubber Div.)

The processing and properties of tyre inner liner compounds based on bromobutyl rubbers with different levels of modification (of unidentified nature) were studied in comparison with a standard inner liner compound. Tests undertaken with the Bayer Tear Analyser showed a strong reduction in crack growth rate with increased modification, especially after ageing. Modulus was reduced with increasing modification, and scorch time increased without seriously affecting cure rate. Cured adhesion to an NR carcass remained at a high level over much of the range, and permeability was not affected. The low temperature properties of the more modified polymers showed a small improvement. The effects of using TMTD as a secondary accelerator on cut growth and modulus were also investigated. 3 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; GERMANY; USA; WESTERN EUROPE

Accession no.636030

## Item 282

151st ACS Rubber Division Meeting, Spring 1997, Conference Preprints. Anaheim, Ca., 6th-9th May 1997, Paper 7, pp.27. 012 EPDM/HIGH DIENE RUBBER BLENDS FOR IMPROVED TIRE SIDEWALL APPEARANCE: UNDERSTANDING THE CHARACTERISTICS OF EACH COMPONENT Ferrandino M P; Hong S W

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Uniroyal Chemical Co. (ACS,Rubber Div.)

Tyre sidewall formulations based on blends of EPDM, NR and polybutadiene were optimised by examining the cure characteristics and mechanical properties of each polymer component and by mixing and curing system studies on the blends. Polymer blending with a homogeniser in an extra preblend step gave good dynamic ozone resistance at the expense of cut growth resistance. Preblending in the early stages of a two-step standard mixing procedure resulted in compounds with good ozone and cut growth resistance. Cut growth resistance could also be improved by preblending carbon black in the NR phase. The use of a sulphenamide/sulphur/peroxide cure system improved the covulcanisation and increased the reversion resistance, resulting in improved ozone and cut growth resistance. 10 refs.

USA

Accession no.636021

## Item 283

151st ACS Rubber Division Meeting, Spring 1997, Conference Preprints. Anaheim, Ca., 6th-9th May 1997, Paper 6, pp.23. 012 IMPROVED ELASTOMER BLEND COMPOUNDS FOR TIRE SIDEWALLS McElrath K O: Tisler A L

Exxon Chemical Co. (ACS,Rubber Div.)

Tyre sidewall compounds based on blends of NR, polybutadiene and a brominated isobutylene-pmethylstyrene elastomer (BIMS) were prepared, and the effects of mixing conditions, curing systems and BIMS structure on compound properties were examined. A high degree of BIMS phase dispersion, necessary for the suppression of crack growth, could be achieved by a remill type mix with high mixing temperatures in both the first and second passes. Further improvements could be obtained through structural variations of BIMS. In particular, raising the p-methylstyrene level while lowering the bromo-p-methylstyrene level seemed to give a better balance of total properties. Fast cures could be obtained regardless of the BIMS structure by variations in the curing systems. 5 refs.

USA

Accession no.636020

## *Item 284 Kautchuk und Gummi Kunststoffe* 50, No.4, April 1997, p.274/81 **REVISITING CONVENTIONAL CURE** Datta R N; Talma A G; Wagenmakers J C

The phenomenon of reversion which occurs in a NR compound when vulcanised using a conventional cure (CV) system is well known. Reversion leads to the

impairment of vulcanisate properties such as modulus, compression set and heat generation. The effects of a new chemical 1,3 bis(citraconimidomethyl)benzene (BCI-MX) are discussed with respect to its mechanism of action and applications. The use of this chemical will allow rubber compounders to take advantage of CV cure systems with regard to flex/fatigue properties whilst improving reversion resistance. 18 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; NETHERLANDS; WESTERN EUROPE Accession no.635688

Item 285

*International Polymer Science and Technology* 23, No.12, 1996, p.T/25-8 **VULCANISATION ACTIVITY OF A NUMBER OF DITHIOCARBAMIC ACID DERIVATIVES** Ovcharov V I; Ranskii A P; Nenashev G O

Technology has been developed for producing organic dithiocarbamate compounds from waste agrochemicals. The accelerators used in the sulphur vulcanisation of rubber mixes based on diene rubbers were metal dithiocarbamates isolated from pesticides unfit for direct application. The investigation was carried out on diethyldithiocarbamates of copper, zinc, nickel, and cobalt, copper dimethyldithiocarbamate and copper cyclohexyldithiocarbamate. An investigation of metal dithiocarbamates as individual vulcanisation accelerators was carried out in unfilled model polyisoprene composites. 9 refs. Translation of Kauchuk i Rezina, No.3, 1996, p.15

RUSSIA Accession no.635170

Item 286

Polymer Bulletin 38, No.5, May 1997, p.545-9 CROSSLINKING OF ETHYLENE PROPYLENE DIENE MONOMER (EPDM) BY BUCKMINSTERFULLERENE Jiang A; Hamed G R Akron,University

An EPDM (ethylene-propylene-ethylidene norbornene terpolymer) rubber was dissolved in o-dichlorobenzene with 1 phr of carbon-60. When cast onto a glass slide and dried, an insoluble film exhibiting reversible elasticity forms. 8 refs.

USA Accession no.634080

Item 287

Patent Number: WO 9534602 A1 19951221 SECONDARY ACCELERATORS FOR THE SULFUR-VULCANIZATION OF RUBBER De Block R F; Datta R N; Hovius H W

## Akzo Nobel NV

Secondary accelerators of a given formula, which are useful in combination with a primary accelerator selected from sulphenamides and thiazoles, in the sulphurvulcanisation of rubber compositions, are disclosed. Also disclosed are sulphur-vulcanised rubber compositions which are vulcanised with a primary accelerator and in the presence of one or more of the disclosed compounds. The present approach achieves a clear improvement in the vulcanisation time as compared to a system without a secondary accelerators, the scorch time is improved by these secondary accelerators.

EUROPEAN COMMUNITY; EUROPEAN UNION; NETHERLANDS; WESTERN EUROPE

Accession no.633871

#### Item 288

## Patent Number: WO 9534601 A1 19951221 IMPROVED CURE PARAMETERS IN RUBBER VULCANIZATION Datta R N; Hondeveld M G J

Akzo Nobel NV

A vulcanised rubber composition is disclosed which comprises the vulcanisation reaction product of 100 pbw of at least one natural or synthetic rubber; 0.1-25 pbw of sulphur and/or a sufficient amount of a sulphur donor to provide the equivalent of 0.1-25 pbw of sulphur; 10-100 pbw of at least one silica filler; 0.1-5 pbw of at least one anti-reversion coagent, comprising at least two groups selected from citraconimide and/or itaconimide groups; and a sufficient amount of at least one cure-improving resin to significantly increase the delta torque of the rubber composition during the vulcanisation process, compared to the delta torque of a similar rubber composition in the absence of the cure-improved resin. Also disclosed is a process for the vulcanisation of this rubber composition, and articles of manufacture comprising the vulcanised rubber composition. The invention is useful for the partial or complete replacement of silane coupling agents in silica-filled rubber compositions.

EUROPEAN COMMUNITY; EUROPEAN UNION; NETHERLANDS; WESTERN EUROPE Accession no.633870

## Item 289

Rubber Chemistry and Technology 69, No.5, Nov-Dec.1996, p.727-41 STUDIES ON A NEW ANTIREVERSION AGENT FOR SULFUR VULCANIZATION OF DIENE

## RUBBERS

Schotman A H M; van Haeren P J C; Weber A J M; van Wijk F G H; Hofstraat J W; Talma A G; Steenbergen A; Datta R N

Akzo Nobel Central Research; Flexsys BV

The mechanism of biscitraconimides (BCI) as antireversion agents for sulphur vulcanised diene rubber

was investigated choosing squalene as a model substrate. Curing of squalene with sulphur in the presence of accelerator, activator and BCI at 170 deg. C gave BCI bridged adducts. The products were identified by spectroscopic methods as being results of a Diels-Alder reaction of BCI (dienophile) with conjugated polyenes that are formed as a result of reversion. The rate at which the sulphur network degrades is comparable to the rate at which BCI adduct is formed. As a result of this, a nearly constant crosslink density is obtained after optimum cure time (t90). Based on this concept, the advantage of using 1,3-bis-(citraconimidomethyl) benzene (BCI-MX), a representative of the BCI-class, in NR based compounds is examined and the influence of BCI-MX on the cure characteristics, mechanical properties, flexural fatigue properties, heat build-up characteristics and dynamic mechanical properties discussed. 26 refs. EUROPEAN COMMUNITY: EUROPEAN UNION:

EUROPEAN COMMUNITY; EUROPEAN UNION NETHERLANDS; WESTERN EUROPE

Accession no.633312

## Item 290

## China Synthetic Rubber Industry 20, No.2, 1997, p.120 APPLICATION OF BISMALEIMIDE IN EPDM PEROXIDE CURING SYSTEM Peng Z; Hong F; Dizhen W; Dongshan L

South China, University of Technology

An attempt is made to improve the curing and heat resistance of EPDM by loading with N,N-metaphenyl bismaleimide (MPBM). Meanwhile, the negative effect of MPBM should be avoided. It is shown that a little MPBM can significantly shorten curing time and speed up the curing reaction, and that the crosslink density of EPDM peroxide curing vulcanisate can be enhanced significantly. Heat and ageing resistance of EPDM peroxide curing vulcanisates are also improved, demonstrated by the compression values listed. CHINA

Accession no.632010

## *Item 291 Kautchuk und Gummi Kunststoffe* 50, No.3, 1997, p.216-25 German **ELASTOMERS: SULPHUR- OR PEROXIDE-CROSSLINKED? CHEMICAL-ANALYTICAL STUDY** Affolter S

The chemical determination of sulphur gives a first impression of the vulcanisation system of crosslinked elastomers. A more precise characterisation is obtained by means of chromatographic methods, i.e. by identifying the products which originated from thermal degradation of the crosslinking reagents (sulphur, sulphur donors, peroxides). The vulcanisation additives used (accelerator, retarder) can be determined on the basis of the degradation products of sulphur-linked material. Evolved gas analysis (EGA) and pyrolysis gas chromatography with adequate detectors (mass spectrometer, flame emission detector) prove to be very successful as chemical-analytical methods for such investigations. 43 refs.

SWITZERLAND; WESTERN EUROPE

Accession no.631938

## Item 292

Journal of Polymer Science : Polymer Physics Edition 35, No.5, 15th April 1997, p.725-34 SORPTION AND DIFFUSION OF ALIPHATIC HYDROCARBONS INTO CROSSLINKED NATURAL RUBBER

Unnikrishnan G; Thomas S Mahatma Gandhi,University

The sorption and transport of four aliphatic hydrocarbons (n-hexane, n-heptane, n-octane and n-nonane) into NR crosslinked using conventional, efficient, dicumyl peroxide(DCP) and mixed sulphur/peroxide vulcanisation systems were investigated at temp. of 28 to 60C. The NR vulcanised by DCP exhibited the lowest penetrant uptake of the systems studied. It was observed that the kinetics of liquid sorption in every case deviated from the regular Fickian trend, characteristic of sorption of liquids by rubbers. The diffusion coefficient, activation energy of sorption, enthalpy, entropy and rubber-solvent interaction parameter were evaluated for the four systems from the swelling data. 30 refs.

INDIA

Accession no.631112

## Item 293

*Kobunshi Ronbunshu* 54, No.2, 1997, p.108-14 Japanese

## STRUCTURAL CHARACTERIZATION BY PYROLYSIS GAS CHROMATOGRAPHY OF ETHYLENE-PROPYLENE-DIENE RUBBERS CURED WITH SULPHUR/THIAZOLE ACCELERATOR

Okumoto T; Yamada T; Tsuge S; Ohtani H Toyota Technical Institute; Toyoda Gosei Co.Ltd.; Nagoya,University

EPDM rubbers cured with sulphur and accelerators were characterised by high resolution pyrolysis gas chromatography with both a flame ionisation detector and a sulphur-selective flame photometric detector. For curing with sulphur and 2-mercaptobenzothiazole accelerator, two types of sulphur-containing products, such as cyclopentathiophene and benzothiazole, were typically observed in the pyrogram. The former was supposed to be formed from sulphur bridges to ethylidene norbornene residue in the polymer chain through cyclisation, while the latter was considered to be derived from the pendant accelerator residue. The vulcanisation process for EPDM rubber was examined on the basis of the relationships between the peak intensities of the characteristic products and cure time. 18 refs.

JAPAN

Accession no.631060

#### Item 294

Rubber and Plastics News 26, No.13, 10th Feb.1997, p.13-14 APPROACHES TO IMPROVE REVERSION RESISTANCE Datta R N; Helt W F Flexsys BV Edited by: Herzlich H

Reversion and the associated changes in rubber compound performance properties is reported to continue to be a major concern for manufacturers of thick rubber products. It is becoming increasingly difficult to balance the need for improved product service life, particularly as service temperatures rise, with increased product productivity and using higher curing and/or processing temperatures without realising some loss in physical performance characteristics through the cross section of these thick rubber articles. During the last few years two new rubber chemicals - 1,3-bis (citraconimidomethyl) benzene (Perkalink 900) and hexamethylene-1,6-bisthiosulphate disodium salt dihydrate (Duralink HTS) - have been introduced to the rubber industry to provide unique approaches for improving reversion resistance without sacrificing performance properties. The use and benefits of these materials in thick rubber articles are described, providing a comparison with traditional approaches utilising lower sulphur/higher accelerator levels.

USA

Accession no.630975

## Item 295

## *International Polymer Science and Technology* 23, No.11, 1996, p.T/78-83 **MELAMINE IN THE RUBBER INDUSTRY** Afanas'ev S V

A survey is made of applications of melamine derivatives in the rubber industry, with particular reference to their uses as adhesion promoters, accelerators and scorch retarders. 43 refs. (Translation of Kauchuk i Rezina, No.4, 1996, p.45).

RUSSIA Accession no.629932

*Item 296 Rubber Chemistry and Technology* 69, No. 4, Sept.-Oct.1996, p.577-90 **STRENGTH OF SULFUR-LINKED ELASTOMERS** 

## Chun H; Gent A N Akron,University,Inst.of Polym.Engineering

Fracture energies were determined for tearing through a sheet of a polysulphide elastomer, and for peeling apart two sheets bonded together with sulphur interlinks. Measurements were made over wide ranges of rate of crack propagation and test temperature. By shifting curves at various temperatures along the rate axis, using shift factors aT calculated from the "universal" form of the WLF equation, master curves were obtained for tear and peel energy vs. rate of tear or peel at Tg about -55 deg C. These master curves of strength vs. effective rate of crack propagation at Tg were closely similar to those obtained previously for several hydrocarbon elastomers; BR, SBR and EPR; interlinked with C-C bonds. The question then arises ; Why are sulphur-vulcanised elastomers stronger in common experience than peroxide-cured ones? Possible reasons are discussed. 20 refs.

USA

Accession no.628821

## Item 297

*International Polymer Science and Technology* 23, No.10, 1996, p.T/55-T/56 **QUALITY OF VULCANISATES CONTAINING DIFFERENT GRADES OF ZINC WHITE** Shutilin Yu F; Pugach I G; Karmanova O V

Zinc white samples containing 86%, 89% and 97.5% zinc oxide were compared with a high purity sample (99.7% zinc oxide) in black-filled polyisoprene and polybutadiene mixes. Each mix contained 2 phr sulphur and 6 phr of zinc white. A masterbatch containing all the components except zinc white and stearin were prepared in a laboratory internal mixer. The white and stearin were then introduced into the mill by two methods, as powder or a melt (melted together for 24h at 70C). High purity white showed comparatively high activity in the vulcanisation process, with optimum vulcanisation 1.5-2 minutes earlier than with other types of white. With an increase in the amount of impurities in the white, the content of free sulphur hardly changed in vulcanisates based on polybutadiene, but increased in vulcanisates based on polyisoprene. TS, EB and tear strength decreased with an increasing content of mineral impurities in zinc white introduced in powder form. The use of melts made it possible to improve the quality of the vulcanisates to the level of vulcanisates with high purity white. 7 refs.

RUSSIA

Accession no.628032

## Item 298

*International Polymer Science and Technology* 23, No.10, 1996, p.T/27-T/29 **PROBLEM OF CARCINOGENIC N-NITROSAMINES AT ENTERPRISES OF THE RUBBER AND TYRE INDUSTRY** 

## Krivosheeva L V; Sokol'skaya N N; Khesina A Ya Russian Academy of Sciences

Original and literature data are given for the presence of N-nitrosamines in rubber industry working environments (tyre and mechanical rubber products factories), in finished product stores, in the finished products (catheters, gloves and teats) and in compounding ingredients (curing agents, blowing agents and accelerators). Tetramethylthiuram disulphide contained up to 22868 micrograms/ kg of N-nitrosodimethylamine. The nitrosamines sought were N-nitrosodimethylamine, N-nitrosodiethylamine, Nnitrosodipropylamine, N-nitrosodibutylamine, Nnitrosopiperidine, N-nitrosopyrrolidine and N-nitrosomorpholine. 16 refs.

RUSSIA

Accession no.628023

## Item 299

# *International Polymer Science and Technology* 23, No.10, 1996, p.T/22-T/24

## FEATURES OF THE USE OF DISULFAL MG IN TYRE VULCANISATES

Boguslavskaya E D; Snegur S A; Boguslavskaya K V; Litvin B L

Dnepropetrovsk, Scientific Res.Inst.for Large-Size Tyres

Disulfal MG is a disulphide that contains a benzothiazolyl group and a morpholyl group. It is produced from di(2benzothiazolyl) disulphide and dithiomorpholine. A study was made of Disulfal MG's effectiveness in comparison with traditional sulphenamide accelerators (Sulphenamide Ts and Sulphenamide M) in tread, breaker and coating rubber mixes (polyisoprene and blends) for lorry, underground, agricultural, roadbuilding, and large and extra-large tyres. Data are shown for cure characteristics, mechanical properties (tensile and tear) and adhesion to impregnated textile cord. It was concluded that Disulfal MG could replace the traditional sulphenamide accelerators without adversely affecting the mechanical properties of the vulcanisates. Disulfal MG is produced in granulated form with a binder to prevent dust formation. UKRAINE

Accession no.628021

## Item 300

Journal of the Adhesive and Sealant Council. Volume 1. Fall 1996. Conference proceedings. San Francisco, Ca. 3rd-6th Nov.1996, p.69-81. 6A1 ECONOMICAL, LOW NITROSAMINE ULTRA ACCELERATORS Ferradino A G Vanderbilt R.T.,Co.Inc. (US,Adhesives & Sealants Council)

The issue of nitrosamine formation from the decomposition of accelerators based on secondary amines during vulcanisation at elevated temperatures is

being actively addressed by the rubber industry. R.T. Vanderbilt has developed two new commercially available ultra accelerators that provide an excellent balance of low toxicity, low nitrosamine formation, good technical properties, and cost effectiveness. The technology was discovered by B.F. Goodrich and is patented. The unique combination of properties offered by the new ultra accelerators based on diisobutylamine is discussed. 3 refs.

## GOODRICH B.F.,CO. USA

Accession no.626517

## Item 301

Journal of Applied Polymer Science 63, No.10, 7th March 1997, p.1353-9 APPLICATIONS OF AN AZIDE SULPHONYL SILANE AS ELASTOMER CROSSLINKING AND COUPLING AGENT

Gonzalez L; Rodriguez A; de Benito J L; Marcos-Fernandez A

Instituto de Ciencia y Tecnologia de Polimeros

N-3-triethoxysilylpropyl-N'-p-benzene sulphonyl azide urea was synthesised and evaluated as a coupling agent in unfilled and silica filled SBR compounds and in unfilled EPM compounds. The mechanical properties and crosslink density of vulcanisates were investigated. The trialkoxysilane moiety of the coupling agent was capable of reacting with the filler surface, and the azide group reacted with the polymer matrix. In addition, alkoxy groups hydrolysed in the presence of moisture to form silanols which subsequently condensed into Si-O-Si and formed an intermolecular crosslink. 12 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; SPAIN; WESTERN EUROPE

Accession no.624426

## Item 302

*Industria della Gomma* 39, No.10, Oct.1995, p.16-20 Italian **ELECTROCHEMICAL DEGRADATION OF EPDM COOLER HOSES** 

Vroomen G; Verhoef H DSM Elastomers Europe; DSM Research BV

Results are presented of studies by DSM of the mechanism of electrochemical degradation of EPDM automotive cooler hoses in contact with water/ethylene glycol coolants, and of the cracking occurring after different exposure times. The resistance to electrochemical corrosion was influenced by the composition of the EPDM compounds. Vulcanisates in which electrical conductivity was reduced by partly replacing carbon black with calcium carbonate were resistant for longer periods than vulcanisates filled with carbon black alone. The degree of crosslinking and type of curing agent used also influenced the resistance of the vulcanisates to degradation. 3 refs. EUROPEAN COMMUNITY; EUROPEAN UNION; NETHERLANDS; WESTERN EUROPE Accession no.621093

## Item 303

Journal of Applied Polymer Science 63, No.2, 10th Jan.1997, p.233-7 KINETIC AND RHEOMETRIC INVESTIGATION OF HEXAMETHYLOL MELAMINE DERIVATIVE ON THE CURE OF NATURAL RUBBER COMPOUNDS Konar B B Calcutta,University

Hexamethylol melamine (HMM) was characterised by FTIR spectroscopy. The optimum dose of HMM in the presence of a sulphur accelerator in a NR-filled compound was determined. The cure rate and kinetics of crosslink formation indicated that HMM enhanced the first-order rate of vulcanisation in an NR compound. The results revealed good mechanical properties of an HMM-based NR carbon black-filled vulcanisate. 15 refs.

INDIA

Accession no.618929

Item 304

*China Rubber Industry* 43, No.11, 1996, p.658-62 Chinese

## ACTIVATION OF CRUMB RUBBER WITH RD-F MECHANICAL-CHEMICAL METHOD

Wen Z; Suhe Z; Guochun B; Yanhao Z; Yanhui Z; Qiuhua L

Beijing, University of Chemical Technology

An activator is uniformly applied to the surface of crumb rubber with a high speed agitator, the strong shearing and fine grinding effect is applied to the crumb rubber by an open mill to facilitate the bonding between activator and crumb rubber. The activator is composed of curing agent, peptiser, antioxidant and phenolic resin. The curing agent is used to increase the crosslink density at the interface between crumb rubber and rubber matrix; the antioxidant is used to increase the dynamic properties of the compound; the phenolic resin is used to increase the adhesion at the interface between crumb rubber and rubber matrix. The RD-F activator system includes antioxidant RD 1, phenolic resin 1,420 oil 1.5, accelerator H 0.4, sulphur 0.5, accelerator CZ 0.5, vinyl tar 8. The wear resistance of tyre tread can be significantly improved by using the crumb rubber activated with the RD-F activator system. CHINA

## Item 305

## Journal of Natural Rubber Research 11, No.2, 1996, p.69-76 ACCELERATOR SYSTEM FOR TRANSPARENT LATEX PRODUCTS Jose L; Joseph R

Cochin,University of Science & Technology

The accelerator combination TMTD/thiocarbanilide was examined in NR latex. The transparency of latex films prepared with TMTD/thiocarbanilide combination was found to be superior compared to those films prepared with ZDC/TMTD system. The mechanical properties and ageing resistance of the vulcanisates were compared with those containing ZDC/TMTD accelerator combination. Vulcanisates prepared with TMTD/thiocarbahilide showed faster curing rates, higher tensile properties and better ageing resistance in comparison to ZDC/TMTD. 9 refs. INDIA

Accession no.617430

## Item 306

*Revue Generale des Caoutchoucs et Plastiques* No.744, Oct.1995, p.87-90 French **DETERMINING THE FREE SULPHUR** 

CONTENT OF VULCANISED RUBBERS

Barbe G; Karnatak A; Gomez E IFOCA

Results are presented of experiments in which HPLC/UV spectrophotometry and gas chromatography with flame ionisation detection were used to determine the free sulphur content of polyisoprene vulcanisates. Detection was enhanced by reacting sulphur with triphenylphosphine to form triphenylphosphine monosulphide. 24 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; FRANCE; WESTERN EUROPE Accession no.616939

## Item 307

*Industria della Gomma* 39, Nos.7/8, July/Aug.1995, p.16-20 Italian **REVERSION RESISTANCE THROUGH CROSSLINK COMPENSATION** Datta R N; De Haag A J; Wilbrink J H Flexsys BV

The use of Perkalink 900 biscitraconimidomethylbenzene (Flexsys) for increasing the reversion resistance of rubber compounds through crosslink compensation is discussed. The effects of this additive on the cure characteristics of an NR compound and on the mechanical and dynamic mechanical properties of the vulcanisates are examined. EUROPEAN COMMUNITY; EUROPEAN UNION; NETHERLANDS; WESTERN EUROPE

Accession no.616888

#### Item 308

## *European Polymer Journal* 32, No.12, Dec.1996, p.1395-401 CHARACTERISATION OF THE NATURAL RUBBER VULCANISATES OBTAINED BY DIFFERENT ACCELERATORS

Travas-Sejdic J; Jelencic J; Bravar M; Frobe Z Zagreb,University; Zagreb,Institute of Medical Research

This study investigates how to use accelerators zinc mercaptobenzothiazole, diphenylguanidine, and their combination as a substitute for commonly used zinc diethyl dithiocarbamate in the vulcanisation of NR latex, as dithiocarbamates, being secondary amines, produce harmful N-nitrosamines. Network structures of vulcanisates obtained by these accelerators were characterised through total crosslink density and the distribution of crosslink types, and were related with physical and mechanical properties. 19 refs. CROATIA

Accession no.616796

## Item 309

*Kautschuk und Gummi Kunststoffe* 49, No.10, Oct.1996, p.671/7 **NEW RUBBER CHEMICAL FOR IMPROVED REVERSION RESISTANCE** Datta R N; Wagenmakers J C

Flexsys bv

The concept of rubber reversion protection by using biscitraconimidomethylbenzene, trade name Perkalink 900, is discussed. The advantages of using this chemical in NR based adhesion compounds was examined. The effects of its use on adhesion to textiles, cure characteristics physical, mechanical and thermal properties are discussed. 14 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; NETHERLANDS; WESTERN EUROPE Accession no.614192

## Item 310 **Rubber and Plastics News** 26, No.7, 4th Nov.1996, p.14-6 **ECONOMICAL, LOW-NITROSAMINE ULTRA ACCELERATORS** Ferradino A; Zukowski R

Vanderbilt R.T.,Co.Inc.

This article places under the spotlight two new ultra accelerators for rubber curing, which reduce the exposure to mutagenic and suspected-carcinogenic airborne nitrosamines. The accelerators, Isobutyl Zimate and Isobutyl Tuads, from RT Vanderbilt Co. Inc. of the USA, provide an excellent balance of low-toxicity, low nitrosamine levels, good properties, and favourable economics.

GOODRICH B.F. USA Accession no.612205

## Item 311

150th ACS Rubber Division Meeting. Fall 1996. Conference Preprints. Louisville, Ky., 8th-11th Oct.1996, Paper 89, pp.14. 012 **TETRAISOBUTYLTHIURAM MONOSULPHIDE: HOW CAN IT BE BOTH A CURE RETARDER AND ACCELERATOR WITH BENZOTHIAZOLE SULPHENAMIDES?** Chasar D W

Goodrich B.F.,Co. (ACS,Rubber Div.)

A study was made of the mechanisms by which tetraisobutylthiuram monosulphide (TiBTM) acts both as a retarder and a secondary accelerator in curing systems containing benzothiazole sulphenamides as primary accelerators. Model reactions between TiBTM and TBBS, CBS or MBTS were followed by HPLC to identify intermediates formed between TiBTM and its various analogues and the primary accelerators. Some of these intermediates were independently synthesised and evaluated as retarders and secondary accelerators in SBR/ polybutadiene compounds. The isobutyl groups were responsible for imparting scorch delay characteristics to one of these intermediates, while another intermediate was most likely responsible for the secondary accelerator effect. 14 refs.

USA

Accession no.611844

## Item 312

150th ACS Rubber Division Meeting. Fall 1996. Conference Preprints. Louisville, Ky., 8th-11th Oct.1996, Paper 88, pp.32. 012 **NEW PEROXIDE CROSSLINKING FORMULATIONS FOR METALLOCENE BASED POLYETHYLENE-OCTENE COPOLYMERS** Palys L H; Callais P A; Novits M F; Moskal M G Elf Atochem North America Inc. (ACS,Rubber Div.)

The crosslinking performance of conventional peroxides and high performance (HP) Luperox peroxides (Elf Atochem North America) was evaluated in several grades of Engage ethylene-octene copolymer elastomers (Du Pont Dow Elastomers) with different octene contents. Confirming previous data for EPDM, the HP peroxides at reduced concentrations provided a significantly longer scorch time compared to standard peroxides at the same curing temperature. Increasing the curing temperature for an HP formulation by 10C provided equivalent scorch time protection while reducing the cure time by 50%, thus doubling productivity. Despite being used at reduced concentrations, the HP peroxides gave equivalent or improved mechanical properties, particularly lower compression set. 13 refs.

DUPONT DOW ELASTOMERS LLC USA Accession no.611843 *Item 313* 150th ACS Rubber Division Meeting. Fall 1996. Conference Preprints. Louisville, Ky., 8th-11th Oct.1996, Paper 79, pp.26. 012 **NEW COAGENT FOR IMPROVED EPDM PROPERTIES** Nagel W Sartomer Co.Inc.

PRO-1276 (Sartomer Co.), a cyclohexane dimethanol diacrylate coagent, was evaluated in the peroxide vulcanisation of radiator hose formulations based on three EPDM grades varying in diene type and content, molecular weight and MWD. Key radiator hose properties, including high modulus and tear strength, low compression set and good heat ageing performance, were achieved for all three EPDM grades. Several alternative coagents studied gave properties close to those obtained using PRO-1276, and offered secondary properties desirable in radiator hoses and other applications. 4 refs. USA

Accession no.611835

(ACS,Rubber Div.)

#### Item 314

## *Rubber Technology International* 1996, p.56-9 **SULPHENAMIDE ACCELERATOR**

CBS type accelerators are one of the substitutes for morpholine type sulphenamide accelerators and have grown in importance in recent years. This comprehensive article reviews the latest generation derivatives born out of an ongoing development programme by Slovakian producer Istrochem.

ISTROCHEM AS SLOVAK REPUBLIC; SLOVAKIA Accession no.610796

*Item 315 Rubber Technology International* 1996, p.39-47 **CURATIVES FOR NATURAL RUBBER** Warren P A; Rende V A Lord Corp.

A rubber curative based on adducts of quinone dioxime and toluene diisocyanate has been developed that offers improved reversion resistance and heat ageing properties compared to conventional cure systems. This comprehensive article supplies a detailed examination of the use of these adducts as curatives for natural rubber and their effect on bondability.

USA

Accession no.610793

Item 316 Journal of Applied Polymer Science

## 62, No.7, 14th Nov.1996, p.1057-66 TETRAMETHYLTHIURAM DISULPHIDE(TMTD) AND 2-MERCAPTOBENZOTHIAZOLE(MBT) AS BINARY ACCELERATORS IN SULPHUR VULCANISATION. III. VULCANISATION OF POLYISOPRENE IN THE ABSENCE OF ZINC OXIDE

Giuliani B V K M; McGill W J Port Elizabeth,University

Polyisoprene was vulcanised with the binary accelerator system TMTD-MBT in the absence of zinc oxide. Samples were heated in a DSC at a programmed rate, the reaction was stopped at points along the thermal curve and the system was analysed. Extractable curing agents and reaction intermediates were analysed by high-performance liquid chromatography and the crosslink density of samples measured by swelling. Two crosslinking reaction sequences were identified, the first being initiated by polysulphides of the mixed accelerator N,N-dimethyldithiocarbamylbenzothiazole disulphide and the second by MBT. All the TMTD was consumed in the first reaction sequence. The synergism of the reaction is discussed in terms of recent studies detailing a reaction mechanism for TMPTDaccelerated vulcanisation. 17 refs.

SOUTH AFRICA

Accession no.610748

Item 317

IRC '96. International Rubber Conference. Conference proceedings.

## Manchester, 17th-21st June 1996, paper 75. 012 NITROSAMINES IN RUBBER - LEGISLATIVE TRENDS

Loadman M J R Malaysian Rubber Producers' Research Assn. (Institute of Materials)

Before considering any legislative trends relating to nitrosamine regulations, it is helpful to know what legislation currently exists and how this position has evolved. An insight is provided into the thinking which has produced the current situation, with the aim of applying it to a future scenario. 22 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; UK; WESTERN EUROPE; WORLD

Accession no.610139

## Item 318

IRC '96. International Rubber Conference. Conference proceedings.

Manchester, 17th-21st June 1996, paper 40. 012 **REVISITING THE CONVENTIONAL CURE: NOVEL WAY TO CONTROL HEAT BUILD-UP** Datta R N; Talma A G; Wagenmakers J C Flexsys BV (Institute of Materials) The phenomenon of reversion which occurs in a NR compound when vulcanised using a conventional cure (CV) system is well known. Reversion leads to the impairment of vulcanisate properties such as modulus, compression set and heat generation. Efficient vulcanisation (EV) systems significantly reduce reversion and heat generation, and improve ageing resistance of NR vulcanisates. The improvement obtained, however, is achieved at the expense of flex and related strength properties. This is an important consideration when rubber is used under dynamic conditions. As a compromise between CV and EV, the industry has adopted the so-called semi-efficient vulcanisation (SEV) system. For years, developments have been directed to enhance reversion resistance without sacrificing flex and dynamic properties. The effects of a new chemical, 1,3 bis(citraconimidomethyl)benzene (BCI-MX), are discussed, with respect to its mechanism of actions and applications. The use of this chemical will allow rubber compounders to take advantage of the CV cure system with regard to flex/fatigue properties whilst improving reversion resistance. 17 refs. EUROPEAN COMMUNITY: EUROPEAN UNION:

EUROPEAN COMMUNITY; EUROPEAN UNION NETHERLANDS; WESTERN EUROPE Accession no.610056

#### Item 319

IRC '96. International Rubber Conference. Conference proceedings. Manchester, 17th-21st June 1996, paper 37. 012 **RESISTANCE TO REVERSION OF NATURAL RUBBER WITH THE HELP OF THIOPHOSPHATE CURE SYSTEMS** Graf H-J; Johansson A H

Rhein Chemie Rheinau GmbH; Rhein Chemie Corp. (Institute of Materials)

NR cured with common accelerators shows reversion if heat aged. Addition of an organic acid in addition to the use of stearic acid decreases the tendency toward reversion. Different accelerators cause different degrees of crosslink degradation as seen with a rheometer or evidenced by lower modulus values upon heat ageing. It can be concluded that amines available during vulcanisation as decomposition products from accelerators will catalyse this reversion. If that is truly the case, then much less reversion can be expected if an accelerator system is used that does not form amines during curing. Standard, semi-efficient and efficient cure systems were developed using sulphur, zinc dithiophosphate and dithiophosphate tetrasulphide accelerator systems. After heat ageing, the retention of modulus and all other physical properties were much better than the control. After ageing seven days at 90 deg.C, vulcanisates cured with the efficient cure system without free sulphur displayed outstanding reversion resistance. 9 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; GERMANY; USA; WESTERN EUROPE

## Item 320

## *China Synthetic Rubber Industry* 19, No.5, 1996, p.317-8 Chinese **TECHNICAL ADVANCES OF SAFE VULCANISATION ACCELERATOR** Fan Y

China, Ministry of Chemical Industry

The classification, new varieties and technical advances of safe vulcanisation accelerators are given 5 refs. CHINA

Accession no.609957

## Item 321

China Synthetic Rubber Industry 19, No.5, 1996, p.290-2 Chinese

## IDENTIFICATION OF SULPHENAMIDE ACCELERATORS IN VULCANISATE

Zhou N; Zhou Q

Beijing, Research & Design Inst. of Rubber Ind.

The benzothiazole and residual amine of sulphenamide accelerators in vulcanisates were determined by a coupling technique. 5 refs.

CHINA Accession no.609947

## Item 322

*Polymer Testing* 15, No.6, 1996, p.507-15 **EFFECT OF SULPHUR ON BOTH THERMAL** 

## AND DIELECTRIC PROPERTIES OF PRE-COMPRESSED FEF BLOCK-LOADED SBR VULCANISATES

Badawy M M Cairo,University

Thermal and dielectric properties of conductive SBR loaded with different concentrations of sulphur were measured. The effect of hydrostatic pressure on dielectric properties and pre-compression is discussed. 20 refs. EGYPT

Accession no.609915

## Item 323

150th ACS Rubber Division Meeting. Fall 1996. Conference Preprints. Louisville, Ky., 8th-11th Oct.1996, Paper 17, pp.37. 012 STABLE CROSSLINK SYSTEMS AND A REACTIVE PLASTICISER FOR IMPROVED WEAR RESISTANCE OF TRUCK TREADS Hong S W; Ferrandino M P; Sanders J A

Uniroyal Chemical Co.Inc. (ACS,Rubber Div.)

Two approaches were investigated for obtaining higher modulus NR truck tyre treads without sacrificing processing characteristics. A stable crosslinking system was developed using tetrabenzylthiuram disulphide as a secondary accelerator with N-t-butyl benzothiazole sulphenamide, and oil was replaced with liquid polyisoprene as a reactive plasticiser. Tread wear resistance was predicted using DIN abrasion tests. The optimised cure system provided approximately 14% better wear resistance than the conventional system, and the reactive plasticiser improved wear resistance by around 10%. 4 refs.

## USA

Accession no.609318

## Item 324

150th ACS Rubber Division Meeting. Fall 1996. Conference Preprints. Louisville, Ky., 8th-11th Oct.1996, Paper 8, pp.20. 012 **EPICHLOROHYDRIN IN FUEL HOSE** Cable C; Smith C T Zeon Chemicals Inc. (ACS,Rubber Div.)

Polyepichlorohydrin (ECO) fuel hose compounds were vulcanised using lead-free curing systems based on a triazine, a thiadiazole and a bisphenol/phosphonium salt/ acid acceptor package. Permeation results showed the advantages of using ECO elastomers as a support layer for fluoroelastomers in fuel hose construction. The adhesion to fluoroelastomers of ECO compounds vulcanised with the alternative curing systems was evaluated. 4 refs.

USA

Accession no.609309

## Item 325

150th ACS Rubber Division Meeting. Fall 1996. Conference Preprints. Louisville, Ky., 8th-11th Oct.1996, Paper 6, pp.24. 012 **HEAT RESISTANT CURING SYSTEM FOR HALOGEN-CONTAINING POLYMERS** Musch R; Schubart R; Sumner A

Bayer AG (ACS,Rubber Div.)

Rhenogran GE 1449 (Rhein Chemie), an alkaline organic compound with amidine structure acting as a heat stabiliser and as a HCl trap during vulcanisation, was used to replace magnesium oxide and thiuram in the vulcanisation of polychloroprene and halobutyl rubbers. The vulcanisates showed unusually high resistance to elevated temperatures and dynamic stress combined with improved scorch safety and mechanical properties. When used to replace magnesium oxide in a blend of sulphur modified polychloroprene and chlorobutyl rubber, the additive gave improved ageing resistance and cut growth performance. 5 refs.

RHEIN CHEMIE RHEINAU GMBH EUROPEAN COMMUNITY; EUROPEAN UNION; GERMANY; USA; WESTERN EUROPE

## Item 326

150th ACS Rubber Division Meeting. Fall 1996. Conference Preprints. Louisville, Ky., 8th-11th Oct.1996, Paper 5, pp.32. 012 **REVERSION RESISTANT CURES FOR RUBBER COMPOUNDS BASED ON BIMS (BROMINATED ISOBUTYLENE-PARAMETHYLSTYRENE)** McElrath K O; Measmer M B Exxon Chemical Co.

(ACS,Rubber Div.)

Brominated isobutylene-paramethylstyrene copolymer compounds were vulcanised using a variety of curing systems. Cure characteristics, reversion resistance and vulcanisate mechanical properties were investigated, and the effects of stearic acid variations in a zinc oxide/zinc diethyldithiocarbamate curing system, glycols, hydrotalcite and copolymer composition were evaluated. Studies of high severity heat ageing showed that certain composition and curing system combinations could yield compounds retaining some mechanical properties even up to 70 hours at 200C. 6 refs.

USA

Accession no.609306

## Item 327

150th ACS Rubber Division Meeting. Fall 1996. Conference Preprints.

## Louisville, Ky., 8th-11th Oct.1996, Paper 4, pp.19. 012 NEW APPROACHES TO IMPROVE REVERSION RESISTANCE OF THICK SECTIONED RUBBER ARTICLES

Datta R N; Helt W F Flexsys BV; Flexsys LP (ACS,Rubber Div.)

Biscitraconimidomethylbenzene and hexamethylene bisthiosulphate disodium dihydrate, alone and in combination, were evaluated as anti-reversion agents in thick sectioned NR articles cured at high and low temperatures. Effects on cure characteristics and the mechanical properties of vulcanisates were investigated. EUROPEAN COMMUNITY; EUROPEAN UNION; NETHERLANDS; USA; WESTERN EUROPE

Accession no.609305

Item 328

150th ACS Rubber Division Meeting. Fall 1996. Conference Preprints. Louisville, Ky., 8th-11th Oct.1996, Paper 2, pp.11. 012 ZINC SOAPS FOR IMPROVED VULCANISATES. II. Vander Kooi J

Struktol Co.of America (ACS,Rubber Div.)

The effects of an aryl zinc soap (Activator 73) on the curing and reversion kinetics of rubber compounds and the mechanical properties of vulcanisates are examined.

It is shown that this additive can improve the thermal stability of the sulphur network, allowing for the use of higher mould temperatures to decrease cure time and extensive overcures without loss of mechanical properties. Some applications of NR and NR/polybutadiene blends which take advantage of this improved thermal stability are described. 8 refs.

USA

Accession no.609303

## Item 329

International Polymer Science and Technology 23, No.5, 1996, p.T/70-2 VULCANISATION CHARACTERISTICS OF MIXES BASED ON SYNTHETIC POLYISOPRENE AND CONTAINING BIOLOGICALLY ACTIVE COMPOUNDS Goncharova Y E; Sakharova E V; Potapov E E

Results are presented of an investigation of the influence of phospholipids and/or proteins (components of NR) of different compositions on the kinetics of vulcanisation of mixes based on SKI-3 synthetic polyisoprene. Phospholipids, which are highly effective surface-active compounds, are shown to ensure better dispersion of the protein and components of the traditional vulcanising group in the elastomer matrix. Nitrogen-containing phospholipids also have a significant accelerating effect during sulphur vulcanisation. 7 refs. (Full translation of Kauch.i Rezina, No.6, 1995, p.17)

CIS; COMMONWEALTH OF INDEPENDENT STATES *Accession no.608104* 

## Item 330

China Rubber Industry 43, No.9, 1996, p.536-8 Chinese THERMAL STABILITY OF EPDM VULCANISATES Chen Y; Ma X

Beijing, Research & Design Inst. of Rubber Ind.

Comparisons are given of thermal stabilities of EPDM vulcanisation cured with peroxide, sulphur, sulphurcontaining compounds, and two different blend curing systems. 2 refs.

CHINA Accession no.606835

## Item 331

Patent Number: WO 9519390 A1 19950720 **RUBBER COMPOSITIONS COMPRISING A POLYMERIC AMINE CO-ACTIVATOR** Coran A Y; Tremont S J; Davis L H; Ignatz-Hoover F; McGrath M P Monsanto Co.

Vulcanisable rubber compositions are disclosed which contain a vulcanisation co-activator which is a polymeric

amine, i.e. a hydrocarbon polymer with pendant alkyl amine groups. The co-activators impart increased rates of vulcanisation with little decrease in scorch delay and very little increase in the modulus of the cured compositions. No increase in heat build-up or decrease in flex-fatigue life is shown. Typical polymers forming the backbone of the co-activators include EPDM rubber and polymers from butadiene or isoprene, with optional co-monomers.

#### USA

Accession no.606614

## Item 332

## Patent Number: EP 735083 A1 19961002 ENAMINES AS VULCANISATION ACCELERATORS FOR NATURAL OR SYNTHETIC RUBBERS

Broussard F; Adovasio M; Roncalli J; Taroni G; Callierotti C

Great Lakes Chemical Italia Srl

A general formula for these enamines is given. EUROPEAN COMMUNITY; EUROPEAN UNION; ITALY; WESTERN EUROPE

Accession no.606213

## Item 333

Journal of Applied Polymer Science 61, No.8, 22nd Aug.1996, p.1239-44 ELASTOMERS WITH TWO CROSSLINKING SYSTEMS OF DIFFERENT LENGTHS VIEWED AS BIMODAL NETWORKS Madkour T M; Hamdi M S

Helwan, University

Elastomers cured with two crosslinking systems such as sulphur and the polymerisation products of pbenzoquinone are shown to have much improved overall mechanical properties. Non-Gaussian behaviour of quinone polymer crosslinked elastomers viewed as bimodal networks was studied. The study focused on the effect of ageing time on the reduced stress values of the networks in relation to the elongation of the samples. The study is also extended to cover the possible effect of these bound antioxidants on the onset of the vulcanisation process and the hardness values of the elastomeric networks. 15 refs.

EGYPT

Accession no.604743

## Item 334

*Journal of Polymer Science : Polymer Physics Edition* 34, No.12, 15th Sept.1996, p.1997-2006 EFFECTS OF THE TYPE OF CROSSLINK ON VISCOELASTIC PROPERTIES OF NATURAL RUBBER

Hagen R; Salmen L; Stenberg B

Swedish Pulp & Paper Research Institute; Stockholm,University

The viscoelastic properties of various crosslinked NRs were studied by mechanical spectroscopy. The Tg was found to be dependent on both crosslink density and crosslink type. Higher values of Tg were obtained for sulphur-crosslinked NR than for peroxide-crosslinked NR at the same crosslink density. An increase in the accelerator level for the sulphur vulcanisate resulted in an increased crosslink density, but it had only a slight effect on the viscoelastic properties. Polysulphidic crosslinks, distinguished by carbon-13 NMR solid-state spectroscopy, were obtained at higher sulphur contents, i.e. lower accelerator/sulphur ratios, and they had a greater influence on Tg than monosulphidic and C-C crosslinks. The rubbery tensile modulus was found to be dependent on the crosslink density but almost independent of the crosslink type. The crosslink density estimated from modulus values was higher than estimates from swelling measurements or from stress-strain data, probably due to the time dependence of disentangling. 26 refs. SCANDINAVIA; SWEDEN; WESTERN EUROPE

Accession no.604383

## Item 335

## Journal of Applied Polymer Science 61, No.9, 29th Aug.1996, p.1515-23 SULPHUR VULCANISATION OF POLYISOPRENE ACCELERATED BY BENZOTHIAZOLE DERIVATIVES. IV. REACTION OF POLYISOPRENE WITH N-CYCLOHEXYLBENZOTHIAZOLE SULPHENAMIDE(CBS), SULPHUR AND ZINC OXIDE

Gradwell M H S; McGill W J Port Elizabeth,University

Polyisoprene was vulcanised using CBS, sulphur and zinc oxide by heating in a differential scanning calorimeter at a programmed rate to given temps. The decomposition of CBS in the rubber was studied and the formation of extractable reaction intermediates prior to and during crosslinking was investigated. The data were used to propose a mechanism for the delayed action observed with CBS and to explain the roles of sulphur, 2bisbenzothiazole-2,2'-disulphide, 2-mercaptobenzothiazole and zinc oxide in the reaction sequence. 24 refs. SOUTH AFRICA

Accession no.604288

## Item 336

Rubber Chemistry and Technology 69, No.2, May/June 1996, p.273-6 STYRENE-BUTADIENE RUBBER FILLED WITH FLUORINATED CARBON BLACK. II. EFFECT OF CURATIVE LEVEL Ames K; Gibala DS; Hamed G R

## Akron, University

The cure and tensile properties of sulphur vulcanised styrene-butadiene rubber filled with a conventional furnace black or a fluorinated black have been determined. Compositions with the fluorinated black and normal curative levels exhibit retarded cure compared to corresponding ones with the furnace black. This is due to a reaction between the sulphenamide accelerator and the fluorinated black. Notwithstanding, a fluoro-filled composition with no curatives substantially crosslinks when moulded at 150 deg.C. Thus, fluoro-black filled specimens have competing effects toward crosslinking. On the one hand, crosslinking is inhibited by reaction with the accelerator, while on the other, the fluoro-black itself can cause crosslinking. 2 refs.

USA

Accession no.603441

## Item 337

Rubber Chemistry and Technology 69, No.2, May/June 1996, p.180-202 NOVEL SULPHUR VULCANISATION ACCELERATORS BASED ON MERCAPTO-PYRIDINE, -PYRAZINE, AND -PYRIMIDINE Rostek C J; Lin H-J; Sikora D J; Katritzky A R; Kuzmierkiewicz W; Shobana N Monsanto Co.; Florida,University

Sulphur vulcanisation accelerators derived from 2mercaptobenzothiazole (MBT) have been a staple for the rubber processing industry for over 65 years. Most noteworthy are the sulphenamide derivatives, which provide various combinations of scorch delay and cure rate, depending upon the basicity and steric nature of the sulphenamide N-substituent(s). While the effect of the amine moiety on benzothiazole sulphenamide accelerator performance has been well documented, the effect of the S-linked heterocyclic moiety of the sulphenamide has been investigated to a much lesser extent. Thus, to gain a better understanding of the role of the lieterocycle, a variety of novel disulphides and sulphenamides based on pyridine is synthesised and tested. The performance of the pyridine based accelerators are compared to each other and to the various benzothiazole analogues. Until recently, the corresponding sulphenimides seem to have received little attention in the rubber industry. Sulphenimides based on pyridine, pyrazine and pyrimidine are therefore also synthesised and tested. The differences in preparation and vulcanisation activity for the benzothiazole sulphenimides and sulphenamides are described, as well as some rudimentary differences which have appeared in the early stages of mechanistic work. 35 refs.

USA

Accession no.603433

*Item 338 China Rubber Industry* 43, No.8, 1996, p.478-81

## Chinese

## VULCANISATION BEHAVIOUR OF M OR DM ACCELERATOR CONTAINING COMPOUNDS Xu J; Fu Y

Jiangsu Danhua Group Corp.; Beijing,Research & Design Inst.of Rubber Ind.

The vulcanisation behaviour of NR compounds containing M or DM accelerators was studied. Their effects on the processability and physical properties were investigated in detail. Data are given for tensile strength, modulus, tear resistance, rebound resilience, and heat ageing resistance. CHINA

Accession no.603235

Item 339

Kautchuk und Gummi Kunststoffe

49, No.7-8, July/Aug.1996, p.532-7 German

## ANALYSIS OF N-NITROSAMINES IN RUBBER AND VULCANISATES - INFLUENCE OF INHIBITORS

Blume A; Giese U; Schuster R H Deutsches Institut fuer Kautschuktechnologie eV

N-Nitrosamines are generally detected in rubber and vulcanisates by methanol extraction and subsequent detection by gas chromatography/thermal energy analysis (TEA). The use of ascorbic acid as an inhibitor of nitrosation during extraction has a negative effect on the quality of the analysis. Interferences in the chromatogram hinder the classification and determination of N-nitroso dimethylamine, N-nitroso diethylamine and N-nitroso dipropylamine. The mechanism of the ascorbic acid interference is considered and it is shown that alphahydroxy acids decay at the temp. of the chromatographic columns by generating carbon monoxide which will give a signal like NO at the TEA detector. The use of hydroquinone instead of ascorbic acid is shown to avoid this problem, thus significantly improving the determination of the above N-nitrosamines. 26 refs. EUROPEAN COMMUNITY; EUROPEAN UNION; GERMANY;

WESTERN EUROPE Accession no.602882

## Item 340

Patent Number: EP 726255 A2 19960814 **3-PYRIDAZINE DERIVATIVES AND THEIR USE IN RUBBER** Lin H-J; Rostek C J; Sikora D J Monsanto Co.

Thiols, disulphides and sulphenamides of certain pyridazines are effective accelerators in the sulphur vulcanisation of rubber. They exhibit improved rates of vulcanisation and states of cure compared with known sulphenamide accelerators.

USA Accession no.597919

## *Item 341 Rubber and Plastics News* 25, No.22, 20th May 1996, p.17-9 HYBRID CROSSLINKER IMPROVES NR PROPERTIES

Cole W F; Scheufler D Flexsys America LP; Clevite Elastomers Inc.

This comprehensive article supplies detailed information concerning the key issues regarding the future needs of the automotive industry. These include long term part endurance, fitness for use testing, and supplier partnering. The article describes the development and testing of a hybrid crosslinker designed to improve the properties of natural rubber.

Accession no.596105

#### Item 342

Plasty a Kaucuk 31, No.9, 1994, p.274-6 EPOXY OLIGOMERS WITH PEROXIDE GROUPS AS NEW VULCANISING AGENTS FOR ETHYLENE-PROPYLENE RUBBERS Bratychak M; Berezovskaya; Duchacek V

Lvov,Polytechnical Institute; Prague,Institute of Chemical Technology

The study shows that the epoxy oligomers with peroxide groups simultaneously act as vulcanising agents and modifiers of ethylene-propylene rubbers. They plasticise the rubber compounds and give heat-resistant vulcanisates in a similar way to organic peroxides but without releasing toxic reaction products. 7 refs. Articles from this journal can be requested for translation by subscribers to the Rapra produced International Polymer Science and Technology. CZECH REPUBLIC

Accession no.592342

## Item 343

Kauchuk i Rezina (USSR) No.4, 1994, p.16-19 Russian CROSSLINKING OF POLYBUTADIENE RUBBER WITH ETHERS OF 1,4-BENZOQUINONEDIOXIME

Avdeenko A P; Glinyanaya N M; Kalinichenko V N

The effect of mono- and bisethers of 1,4-benzoquinonedioxime as crosslinking agents for SKD polybutadiene rubber was studied. 23 refs. Articles from this journal can be requested for translation by subscribers to the Rapra produced International Polymer Science and Technology. RUSSIA

Accession no.592317

*Item 344 Polimeri* 15, No.4, 1994, p.119-22 **ULTRAVIOLET DEGRADATION OF NATURAL** 

## RUBBER LATEX FILMS VULCANISED WITH DIFFERENT ACCELERATORS

Travas-Sejdic J; Jelencic J; Bravar M Zagreb,University

Natural rubber latex films were prepared by sulphur vulcanisation. Four different accelerators were employed, diphenylguanidine, zinc 2-mercaptobenzothiazole, a combination of these, and zinc diethyldithiocarbamate. The chemical and physical changes occurring in the vulcanisates during UV ageing were studied. The results showed that different types of accelerators formed different crosslink structures. These structures influenced changes in the properties of the vulcanisates during photo-oxidation. 10 refs. Articles from this journal can be requested for translation by subscribers to the Rapra produced International Polymer Science and Technology. CROATIA

Accession no.592308

## Item 345

Kautchuk und Gummi Kunststoffe 49, No.5, May 1996, p.336-9 TWO-STEP VULCANISATIONS OF CARBON BLACK FILLED EPDM Verschuren LL M: Zeeman R: Van Der Does L: Banties

Verschuren J L M; Zeeman R; Van Der Does L; Bantjes A Townsend Engineering BV

Several mechanical properties of carbon black filled EPDM changed drastically by using a strain during the second step of a two-stage peroxide vulcanisation. EB decreased whereas tensile strength increased due to this vulcanisation method. Crosslinked rubbers with the same mechanical properties could be obtained as from a vulcanisation without strain, but with considerably less peroxide. 17 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; NETHERLANDS; WESTERN EUROPE

Accession no.592025

#### Item 346

International Polymer Science and Technology 22, No.7, 1995, p.T/21-3 INFLUENCE OF MESITYLENE DINITRILE OXIDE ON THE PROCESSING PROPERTIES OF ELASTOMER COMPOSITIONS Boiko V V; Grinev I V

To predict the processing properties of elastomer compositions when mesitylene dinitrile oxide was introduced, an investigation was made of the changes in viscosity of polyisoprene, SBR, butadieneacrylonitrile copolymers and BR with different mesitylene dinitrile oxide contents. Properties were compared with those of the initial elastomers plasticised for the same amount of time. The shear stress relaxation rate was determined. 7 refs.

## Item 347

149th ACS Rubber Division Meeting, Spring 1996. Conference preprints. Montreal, 5th-8th May 1996, paper 45, pp.21. 012 **IMPROVED CURING SYSTEM FOR CHLORINE-CONTAINING POLYMERS** Ohm R F; Taylor T C Vanderbilt P. T. Co. Inc. : DuPont Dow Elastomers

Vanderbilt R.T.,Co.Inc.; DuPont Dow Elastomers (ACS,Rubber Div.)

Curatives based on 1,3,4-thiadiazole have been used to crosslink chlorine-containing polymers for several years. Chlorinated polyethylene is the usual polymer cured by one such derivative. The cure behaviour and physical properties of a new class of 1,3,4-thiadiazole derivatives that is being developed specifically for polychloroprene (CR) are described. Polychloroprene curatives generally are slow curing and give a marching modulus. The new system offers an exceptionally flat cure plateau. At processing temperatures, the new derivatives give two to four times the scorch time of ethylene thiourea (ETU). At cure temperatures, shorter cure times than ETU are obtained. Typical of polychloroprene, metal oxides are used in this new system. The properties of suitable metal oxides are outlined. Also demonstrated is the optional use of coactivators. 5 refs.

USA

Accession no.591686

Item 348

149th ACS Rubber Division Meeting, Spring 1996. Conference preprints.

Montreal, 5th-8th May 1996, paper 29, pp.51. 012 RESISTANCE TO REVERSION OF NATURAL RUBBER WITH THE HELP OF THIOPHOSPHATE CURE SYSTEMS

Graf H-J; Johansson A H Rhein Chemie Rheinau GmbH; Rhein Chemie Corp. (ACS,Rubber Div.)

NR cured with common accelerators shows reversion if heat aged. Addition of an organic acid in addition to the use of stearic acid decreases the tendency towards reversion. Different accelerators cause different degrees of crosslink degradation as seen with a rheometer or evidenced by lower modulus values upon heat ageing. It can be concluded that amines available during vulcanisation as decomposition products from accelerators will catalyse this reversion. If that is the case, then much less reversion can be expected if an accelerator system is used that does not form amines during curing. Standard, semi-efficient and efficient cure systems are developed using sulphur, zinc dithiophospate and dithiophosphate tetrasulphide accelerator systems. After heat ageing the retention of modulus and all other physical properties are much better than the control. After ageing for seven days at 90C, vulcanisates cured with the efficient cure system without free sulphur display outstanding reversion resistance.

EUROPEAN COMMUNITY; EUROPEAN UNION; GERMANY; USA; WESTERN EUROPE Accession no.591670

## Item 349

149th ACS Rubber Division Meeting, Spring 1996. Conference preprints. Montreal, 5th-8th May 1996, paper 20, pp.26. 012 **PRECISION PRE-WEIGH OF THE FUTURE** Falco L; Johansson A Rhein Chemie Corp. (ACS,Rubber Div.)

Rhein Chemie has introduced the Burrito, a customised precision preweighed and predispersed cure package. The Burrito contains Rhenogran chemicals for rapid and uniform dispersion, which are weighed into separate pockets to prevent chemical interaction. A study is conducted to investigate the phenomena of eutectic behaviour. Eutectic behaviour occurs when chemicals in close proximity have a mutual solubility, whereby the melting point of the mixture is lower than the individual melting points of the chemicals. The basic problem associated with eutectic mixtures is the variable nature of the changes as a function of the heat history of the mixture, which can vary from shipment to shipment and even boxes within a shipment. The reactive and eutectic behaviour of most accelerators used in curing EPDM and the effects on cure rate, scorch safety and physical properties are presented.

USA

Accession no.591661

## Item 350

British Engel Elast Symposium 1995. Symposium Proceedings. Warwick, 21st-22nd June 1995, paper 5, pp.10. 831 ACCELERATOR SYSTEMS FOR IM COMPOUNDS - DEPENDENCE OF ECONOMICAL PROCESS DESIGN Graf H J

Rhein Chemie Rheinau GmbH (Engel Vertriebsges mbH)

Information is presented in some detail on factors affecting the economical production of injection moulded rubber compounds. The effect of additives such as fillers, plasticisers and crosslinking agents on the processing characteristics is discussed. A comparison is also made between the performance of four Rhenocure accelerator systems (AP 7, AP 2, AP 3 and AP 6) in NBR, NR and EPDM compounds. 10 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; GERMANY; WESTERN EUROPE Accession no.590901

*Item 351 Journal of Applied Polymer Science* 60, No.10, 6th June 1996, p.1569-74

## CURE CHARACTERISTICS OF UNACCELERATED SULPHUR VULCANISATION OF EPOXIDISED NATURAL RUBBER(ENR)

Poh B T; Chen M F; Ding B S Universiti Sains Malaysia

The curing characteristics of unaccelerated sulphur vulcanisation of ENR 25 and ENR 50 (25 and 50 mol % of epoxidation, respectively) were studied in the temp. range from 100-180C. The range of sulphur contents was 1.5 to 6.5 phr. The scorch time was determined by use of a Mooney Shearing Disk Viscometer, while the initial cure rate, maximum torque and reversion properties were obtained from the Moving Die Rheometer. The results showed that ENR 25 gave a longer scorch time than ENR 50, an observation similar to that in an accelerated system reported previously. For temps. below 120C, scorch time depended exponentially on sulphur content for both rubber. This dependence diminished, however, as temp. was increased. This observation was attributed to the availability of activated sulphur molecules for vulcanisation. The initial cure rate and maximum torque increased with increasing sulphur loading. ENR 50, however, exhibited higher value than ENR 25, suggesting faster cure in the former. For a fixed sulphur loading, reversion was a time- and temp.-dependent phenomenon. It decreased with increasing sulphur loading because of the increase of crosslink density for both rubbers studied. 13 refs.

MALAYSIA

Accession no.590588

## Item 352

Journal of Applied Polymer Science 60, No.9, 31st May 1996, p.1349-59 CURE SYNERGISM IN CARBOXYLATED NITRILE RUBBER(XNBR) VULCANISATION IN PRESENCE OF THIOPHOSPHORYL DISULPHIDES AND AMINE DISULPHIDE/ THIAZOLE ACCELERATORS Biswas T; Basu D K

Indian Association for the Cultivation of Science

The vulcanisation of XNBR containing synergistic combinations of accelerators with thiophosphoryl disulphide as one of the components was studied. Other accelerators used were 2-mercaptobenzothiazole, 2mercaptobenzothiazyl disulphide, bis(N-oxydiethylene) disulphide and N-oxydiethylene 2-benzothiazole sulphenamide(OBTS). The combinations of thiophosphoryl disulphides with OBTS exhibited the highest mutual activity as far as resulting physical properties were concerned. Structural characterisation of different thiophosphoryl disulphide-accelerated XNBR vulcanisates, including those formed from the synergistic combinations with OBTS, were studied using a methyl iodide probe. It was found that the amount of sulphidic crosslinks arising from the reaction between -COOH groups of XNBR and thiophosphoryl disulphides actually controlled the network structure and the physical properties of the vulcanisates. 29 refs.

#### INDIA

Accession no.590567

#### Item 353

## *International Polymer Science and Technology* 22, No.12, 1995, p.T/1-3 **INFLUENCE OF**

#### TRIETHYLALKYLAMMONIUM BROMIDES ON THE SULPHUR VULCANISATION OF DIENE RUBBERS

Pankratov V A; Romanova T V; Fomin A G; Fonskii D Y

Results are presented of investigations of the effect of cationic surfactants of the above type, at levels of 0.1 to 2.5 parts per 100 parts rubber, on the kinetics of sulphur vulcanisation of polyisoprene rubber and butadieneacrylonitrile rubber and on the properties of the vulcanisates. It is shown that these quaternary ammonium compounds shorten the process of sulphur vulcanisation of diene elastomers, increase the effectiveness of their crosslinking and improve the strength properties of vulcanisates, but also result in a reduction in the time before the start of vulcanisation, which can lead to scorching of the rubber mixes. 4 refs. (Full translation of Kauch.i Rezina, No.3, 1995, p.17)

CIS; COMMONWEALTH OF INDEPENDENT STATES; RUSSIA Accession no.589998

## Item 354

International Polymer Science and Technology 22, No.11, 1995, p.T34/5 VULCANISATION OF BUTYL RUBBER AT A MODERATE TEMPERATURE Shilov I B; Khlebov G A; Baranov L A

Investigations were carried out on vulcanisates based on butyl rubber, to create, by rapid vulcanisation in hot water, a corrosion resistant vulcanisate based on saturated rubbers, which is characterised by a longer guaranteed storage life. Vulcanising groups were found that would be highly active at 60 degrees C, but inactive at temperatures below 50 degrees C. 4 refs. Translated from Kauchuk i Rezina, No.3, 1995, p.13.

RUSSIA

Accession no.588417

## Item 355

Journal of Applied Polymer Science 60, No.6, 9th May 1996, p.845-55 STUDIES ON THE EFFECT OF THIURAM DISULPHIDE ON NR VULCANISATION ACCELERATED BY THIAZOLE-BASED ACCELERATOR SYSTEMS Debnath S C; Basu D K Indian Association for the Cultivation of Science

Investigations were carried out with the binary combinations of each of bis(N-methylpiperazino)thiuram disulphide, tetrabenzylthiuram disulphide, and tetramethylthiuram disulphide, separately with Ncyclohexyl-2-benzothiazole sulphenamide, 2mercaptobenzothiazole, and 2-mercaptobenzothiazyl disulphide in the combinations studied. 19 refs. INDIA

Accession no.588288

## Item 356

## Rubber World 213, No.5, Feb.1996, p.34-6 QUATERNARY AMMONIUM SALTS: NEW CURATIVES FOR VULCANISATION OF NATURAL RUBBER

Mathur R; Biswas A; Chandra A K; Mukhopadhyay R Sukhadia,University; Singhania Elastomer & Tyre Research Institute

This article describes preliminary work carried out to evaluate the efficiency of triethylbenzylammonium chloride (TEBA) in curing NR under different conditions. The study indicates that TEBA acts as a booster accelerator and produces a synergistic action with other primary accelerators to improve cure characteristics and physical properties. The scorch safety properties of TEBA were compared with other combinations of synergistic accelerators.9 refs.

INDIA

Accession no.587356

## Item 357

## *Journal of Applied Polymer Science* 60, No.3, 18th April 1996, p.439-47 **THIURAM ACCELERATED SULPHUR VULCANISATION. III. THE FORMATION OF CROSSLINKS** Geyser M; McGill W J

Port Elizabeth, University

2,3-Dimethyl-2-butene (TME) was used as a model for polyisoprene in studying the formation of crosslinks in tetramethylthiuram disulphide (TMTD)-accelerated sulphur vulcanisation. Mixtures of TME with TMTD, sulphur and zinc oxide were heated in sealed tubes at 130 C for various times and the contents analysed by HPLC. In the absence of zinc oxide crosslinking is delayed until most of the TMTD was consumed. Crosslinking resulted from the interaction of thiuram and dimethylammonium pendent groups, tetramethylthiourea being formed as a by-product. 21 refs.

SOUTH AFRICA

Accession no.586942

Item 358 Journal of Applied Polymer Science

## 60, No.3, 18th April 1996, p.425-30 THIURAM ACCELERATED SULPHUR VULCANISATION. I. THE FORMATION OF THE ACTIVE SULPHURATING AGENT Geyser M; McGill W J

Port Elizabeth, University

Mixtures of tetramethylthiuram disulphide(TMTD)/zinc oxide and TMTD/sulphur/zinc oxide were heated in a DSC to various temperatures. Zinc dimethyldithiocarbamate formed only in undried TMTD/zinc oxide mixes, the reaction being catalysed by water on the zinc oxide surface. The presence of zinc oxide delayed the decomposition of TMTD by absorbing thiuram sulphenyl radicals, which were needed to initiate tetramethylthiuram monosulphide and tetramethylthiuram polysulphide formation. 29 refs.

SOUTH AFRICA

Accession no.586940

## Item 359

Rubber and Plastics News 25, No.17, 11th March 1996, p.14-7 COAGENTS FOR RUBBER-TO-METAL ADHESION Costin R; Nagel W R

Sartomer Co.

Metallic coagent-peroxide systems were shown to provide the best properties of peroxide and sulphur cures, yielding high tensile and tear strength, good heat-aged properties with the benefit of good adhesion. The use of Saret metallic coagent systems, in particular, Saret 633, an anhydrous zinc diacrylate containing a non-nitroso scorch retarder and Saret 634, an anhydrous zinc dimethacrylate are demonstrated to improve performance properties for adhesive application. They eliminate the need for external adhesives and a separate curing step, developing bonds at the rubber-metal interface during the curing step, and producing crosslinks in the rubber during curing. USA

Accession no.586794

## Item 360

Rubber Chemistry and Technology 68, No.5, Nov-Dec.1995, p.794-803 SOLUBILITY OF SULPHUR AND DITHIOCARBAMATES IN NATURAL RUBBER Morris M D; Thomas A G Malaysian Rubber Producers' Research Assn.; London,University,Queen Mary & Westfield College

Immersion of 1 mm thick sheets of peroxide-vulcanised NR in both liquid and, more surprisingly, powdered solid curing agents was shown to be an adequate method for measuring solubilities. The same experimental method also enabled some diffusion data to be obtained. Solubilities of sulphur, zinc diethyldithiocarbamate and

zinc butyl dithiocarbamate were determined at various temps. between 23 and 135C. The experimental results were not in good agreement with the theory which had previously been found to describe the solubility of waxes in NR. This theory, which was a combination of the Flory-Huggins equation with an expression for the latent heat of fusion, was modified to take into account the dependence of the interaction parameter on temp. and on degree of swelling. The new expression gave much better agreement with the experimental results for all three compounds. 26 refs. (ACS Rubber Div., Spring Meeting, Philadelphia, May 1995)

EUROPEAN COMMUNITY; EUROPEAN UNION; UK; WESTERN EUROPE

Accession no.586556

## Item 361

Rubber Chemistry and Technology 68, No.5, Nov.-Dec.1995, p.746-56 SULPHUR/OLEFIN COPOLYMERS AS VULCANISING AGENTS FOR RUBBER Colvin H; Bull C Goodyear Tire & Rubber Co.

A suspension process for the preparation of sulphur/ dicyclopentadiene and sulphur/dicyclopentadiene/styrene copolymers, for use as vulcanising agents for rubber, was developed. The relationship between the sulphur/olefin copolymer and vulcanisate properties was investigated in terms of the structure of the copolymer and other reaction products. 21 refs.

USA

Accession no.586552

## Item 362

Rubber Chemistry and Technology 68, No.5, Nov.-Dec.1995, p.717-27 CHEMISTRY OF PHENOL-FORMALDEHYDE RESIN VULCANISATION OF EPDM. I. EVIDENCE FOR METHYLENE CROSSLINKS Van Duin M; Souphanthong A DSM Research; Taurus Hungarian Rubber Works

The reaction products in phenol-formaldehyde resin curing of EPDM, containing 2-ethylidene norbornene as the third monomer, were studied, using 2-ethylidene norbornane(ENBH) as a low molec.wt. model for EPDM. The reaction of ENBH and a resole resulted in scission of the dimethylene ether bridges, i.e. in degradation of the resole into mono-, bis- and terisooctylphenol units. These were consequently converted into products consisting of two ENBH molecules linked by mono-, bisand terisooctylphenyl units. The results supported the use of 2-hydroxymethylphenol(HMP) as a low molec.wt. model for the resole. The reaction products of ENBH with both the resole and HMP were shown to contain methylene-linked structures, as demonstrated by the formation of monoisooctylphenol crosslinks and the presence of residual unsaturation and hydroxyl groups, apart from chroman linked structure. 31 refs. EASTERN EUROPE; EUROPEAN COMMUNITY; EUROPEAN UNION; HUNGARY; NETHERLANDS; WESTERN EUROPE Accession no.586549

#### Item 363

IRC '95 Kobe International Rubber Conference. Conference proceedings. Kobe, 23rd-27th Oct.1995, p.555-8. 012 STUDY OF THE VULCANISATION MECHANISM OF DIFFERENT ELASTOMERS USING DSC AND FTIR

Gupta S D; Deuri A S; Mukhopadhyay R Hari shankar Singhania Elastomer & Tyre Res.Inst. (Japan,Society of Rubber Industry)

Accelerated sulphur vulcanisation of rubber proceeds through an intermediate complex formulation between accelerator and activator (zinc ion), the mechanism of which is yet to be fully understood. An attempt is made to understand the effect of different accelerators on complex formation by different analytical techniques. The results contribute to an elucidation of complex formation mechanism with different accelerators. 5 refs.

INDIA

Accession no.586268

#### Item 364

IRC '95 Kobe International Rubber Conference. Conference proceedings. Kobe, 23rd-27th Oct.1995, p.543-6. 012 **SUBSTITUTION OF TMTD IN DIENE RUBBER COMPOUNDS** Graf H-J; Issel H-M

Rhein-Chemie Rheinau GmbH (Japan,Society of Rubber Industry)

Thiurams are problematic from the point of ecology and health care. Changing from thiuram-based systems to alternative curing systems, especially TMTD, proves to be a very powerful accelerating sulphur donor which is hard to replace. Dithiophosphoryl combinations as alternatives for TMTD are described. 2 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; GERMANY; WESTERN EUROPE

Accession no.586265

## Item 365

IRC '95 Kobe International Rubber Conference.
Conference proceedings.
Kobe, 23rd-27th Oct.1995, p.539-42. 012
HIGH TEMPERATURE CURING OF NATURAL
RUBBER COMPOUNDS BY USING 1,3
BIS(CITRACONIMIDOMETHYL) BENZENE
Datta R N; Wilbrink J H
Flexsys BV
(Japan,Society of Rubber Industry)

The chemistry of reversion is reviewed. The concept of reversion protection by using a new chemical, 1,3 bis(citraconimidomethyl) benzene (BCI-MX), is discussed. Based on this concept, the possibility of high temperature cure is examined. 4 refs.

BELGIUM; EUROPEAN COMMUNITY; EUROPEAN UNION; WESTERN EUROPE

Accession no.586264

## Item 366

IRC '95 Kobe International Rubber Conference. Conference proceedings. Kobe, 23rd-27th Oct.1995, p.535-8. 012 **POLYCHLOROPRENE WITH IMPROVED HEAT STABILITY** 

Musch R Bayer AG (Japan,Society of Rubber Industry)

Polychloroprene (CR) is approaching its stress limit in several fields of application due to increased requirement profiles. It is therefore necessary to develop grades with improved temperature resistance and to optimise crosslinking systems. To this end, crosslinking studies on model compounds are described, the findings verified by means of compound studies and a new crosslinking system is developed. This results in CR vulcanisates with unusually high resistance to temperature loads and dynamic stressing. 3 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; GERMANY; WESTERN EUROPE

Accession no.586263

Item 367

IRC '95 Kobe International Rubber Conference. Conference proceedings. Kobe, 23rd-27th Oct.1995, p.531-4. 012

## ACRYLIC RUBBERS AND THEIR CURING SYSTEMS

Kawanaka T; Umetsu K; Hosoya K; Hashimoto K Nippon Zeon Co.Ltd. (Japan,Society of Rubber Industry)

Polyacrylic rubber (ACM) is a polymer consisting of various monomers, including acrylic esters and cure site monomers. There are various systems, eg ammonium benzoate for epoxy functional ACM and soap/sulphur for active chlorine ACM. Crosslinking of epoxy functional ACM with isocyanuric acid yields fast cure. The characteristics and cure mechanism for this system are presented. 5 refs.

JAPAN Accession no.586262

## Item 368

IRC '95 Kobe International Rubber Conference. Conference proceedings. Kobe, 23rd-27th Oct.1995, p.333-6. 012

## TETRAKIS (2-ETHYLHEXYL) THIURAMDISULPHIDE AS SECONDARY CURING ACCELERATOR

Otomo S; Kobayashi Y; Yamamoto Y Ouchi Shinko Chemical Ind.Co.Ltd. (Japan,Society of Rubber Industry)

Compared with common thiuram-type curing accelerators such as TMTD, TETD and TBTD, tetrakis(2ethylhexyl)thiuramdisulphide (TOTD) shows superior curing characteristics (good scorch safety and faster curing rate) as a secondary curing accelerator for NR sulphur vulcanisation using CBS, and gives the NR vulcanisate with excellent heat resistance because of the increase of thermal stable mono and disulphidic crosslinks. 4 refs.

JAPAN

Accession no.586091

## Item 369

Kautchuk und Gummi Kunststoffe 49, No.3, March 1996, p.206-9 INFLUENCE OF OLIGOAMIDEPHOSPHATES ON SOME CHARACTERISTICS OF ELASTOMER COMPOUNDS BASED ON NBR Arnaudova R; Vladkova T; Arnaudov M Sofia,Technological University; Sofia,University

The addition of oligoamidephosphates (OAPh) without reducing the quantity of the sulphenamide accelerator in NBR compounds offers additional acceleration of the curing process and gives good vulcanisates with good mechanical properties and ageing resistance. Vulcanisates accelerated with OAPh as sole accelerator is very slow, but it leads to vulcanisates with high tensile strength. The activity of OAPh in NBR compounds is similar to their activity in IR and SBR. 12 refs.

BULGARIA; EASTERN EUROPE

Accession no.586046

## Item 370

Euradh '94. Conference Proceedings. Mulhouse, 12th-15th Sept.1994, p.478-81. 9(12)4 **RUBBER/RUBBER ADHESION MECHANISMS. INFLUENCE OF CURATIVES MIGRATION** Giami S; Vallat M F; Schultz J; Daubourg N; Coupard A; Demuvnck M

Lab.de Recherches et de Controle du Caoutchouc et des Plastiques; Centre de Recherches sur Physico-Chimie Surf.Sol.; Centre de Recherches Michelin (Societe Francaise du Vide; Institute of Materials; Dechema Institut)

Two blends of isoprene rubber, filled with carbon black, had chemical compositions which differed only in the sulphur/accelerator ratio. After curing, the final mechanical stiffness was almost the same for each of the blends. After the determination of optimal compositions and vulcanisation conditions, the elastomer sheets were assembled. Peel tests showed that failure was interfacial in all cases and the reasons for this were discussed. 4 refs. EUROPEAN COMMUNITY; EUROPEAN UNION; FRANCE; WESTERN EUROPE

Accession no.584902

Item 371

148th ACS Rubber Division Meeting. Fall 1995. Conference Preprints. Cleveland, Oh., 17th-20th Oct.1995, Paper 110, pp.38.

012

## BLACK TYRE SIDEWALL COMPOUNDS BASED ON BROMINATED POLYISOBUTYLENE-CO-P-METHYLSTYRENE ELASTOMER AND ACCELERATED WITH 1,6-HEXAMETHYLENE BISSODIUM THIOSULPHATE

Ignatz-Hoover F; Newman N F Flexsys America LP; Exxon Chemical Co. (ACS,Rubber Div.)

The disodium salt of hexamethylene bisthiosulphate (HTS) was evaluated as an accelerator in the zinc oxide mediated vulcanisation of tyre sidewall compounds containing a brominated isobutylene-p-methylstyrene copolymer. Rheometer profiles of blends with polybutadiene cured with sulphenamide, sulphur, zinc oxide and HTS showed a characteristic delayed action fast cure kinetic profile, while the same blends cured without HTS exhibited undesirable cure characteristics, with long early cure and fast late cure. Many vulcanisation characteristics and vulcanisate properties were largely dominated by a single cure component. State of cure was dominated by sulphur level, scorch delay and early cure time by HTS loading, and the time to reach t90 by the sulphenamide level. Time of flight secondary ion mass spectroscopy imaging showed that HTS favourably affected the distribution of curatives in blends with SBR. 11 refs.

USA

Accession no.583033

#### Item 372

China Rubber Industry 43, No.2, 1996, p.95-8 Chinese COMPARISON BETWEEN PROPERTIES OF

RADIATION CURED SBR VULCANISATE AND SULPHUR CURED ONE Xu Yunshu; Youssef H A

7 refs. CHINA Accession no.580973

Item 373 China Rubber Industry

43, No.2, 1996, p.67-71 Chinese

## STUDY ON COMPRESSION SET OF NBR AT ELEVATED TEMPERATURE

Liu Yuzhen; Guo Yong; Li Zhenming; Xiao Jianbin Qingdao,Institute of Chemical Technology

The influence of different curing systems and inorganic fillers on the compression set of NBR N41 at 100 C was studied. The results show that DTDM EV system was the most effective in terms of minimum compression set of the vulcanisate; DCP was more effective; CZ EV system was poor; and sulphur curing system was the most poor. Among the inorganic fillers, the quartz filler was the most favourable to the minimum compression set of the vulcanisate, and the compression set would change little with the increase of the quartz filler level; the other favourable inorganic fillers were put in order of iron oxide red, super-fine calcium carbonate and silica 233N and 255N. 2 refs.

CHINA

Accession no.580967

#### Item 374

Tyretech '95. 21st Century Technology: Evolution or Revolution? Conference proceedings. Turin, 16th-17th Oct.1995, paper 17, pp.8. 6T **PERKALINK 900 NEW POSSIBILITIES FOR HIGH TEMPERATURE CURING OF NATURAL RUBBER COMPOUNDS** Datta R N; Wilbrink J H Flexsys BV (Rapra Technology Ltd.; European Rubber Journal)

This paper presents information in some detail on the use of a unique chemical to increase the curing temperature of natural rubber compounds. Developed by Flexsys BV of The Netherlands, Perkalink 900 1,3-bis(citraconimidomethyl)benzene is said to be effective in the improvement of reversion behaviour of systems containing diene rubbers. It is thought that using this curing agent will reduce mould times, thereby aiding productivity in tyre manufacturing. 10 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; NETHERLANDS; WESTERN EUROPE

Accession no.580914

#### Item 375

148th ACS Rubber Division Meeting. Fall 1995.
Conference Preprints.
Cleveland, Oh., 17th-20th Oct.1995, Paper 97, pp.31.
012
SELECTING THE OPTIMUM COAGENT FOR SEVERAL RUBBER APPLICATIONS
Costin R; Nagel W
Sartomer Co.Inc.
(ACS,Rubber Div.)

Saret acrylic and methacrylic acid salts (Sartomer) were compared with triallyl cyanurate as co-curing agents in the peroxide vulcanisation of EPDM compounds for a number of applications, including building profiles, radiator hose, moulded goods, cables, roll covers and sealants. Effects on curing characteristics, mechanical properties and rubberto-metal adhesion were studied. 1 ref.

USA

Accession no.580278

## Item 376

148th ACS Rubber Division Meeting. Fall 1995. Conference Preprints.

Cleveland, Oh., 17th-20th Oct.1995, Paper 95, pp.17. 012

## HYBRID, SEMI-EV CURE SYSTEM DESIGN FOR GREATER PHYSICAL AND DYNAMIC PROPERTIES STABILITY IN A NATURAL RUBBER BUSHING COMPOUND

Cole W F; Scheufler D

Flexsys America LP; Clevite Elastomers Inc. (ACS,Rubber Div.)

Optimised NR bushing compounds were developed using semi-efficient cure systems in which N-tert-butyl-2benzothiazole was used as the primary accelerator, zinc dibutyldithiophosphate as the secondary accelerator, Ncyclohexyl thiophthalimide as retarder, and hexamethylene bisthiosulphate disodium dihydrate as a stabilising hybrid crosslinker. These cure systems gave compounds with improved stability of mechanical and dynamic mechanical properties and shortened mould cycle times.

USA

Accession no.580276

## Item 377

148th ACS Rubber Division Meeting. Fall 1995. Conference Preprints.

Cleveland, Oh., 17th-20th Oct.1995, Paper 94, pp.15. 012

## TETRAISOBUTYLTHIURAM MONOSULPHIDE (TIBTM): A UNIQUE RETARDER/KICKER IN ONE MOLECULE

Chasar D W Goodrich B.F.,Co. (ACS,Rubber Div.)

Tetraisobutylthiuram monosulphide (TiBTM) was evaluated as a retarder and secondary accelerator for both N-t-butyl-2-benzothiazole sulphenamide and Ncyclohexyl-2-benzothiazole sulphenamide in SBR/ polybutadiene and NR compounds. TiBTM was also compared in these systems to the retarder N-cyclohexyl thiophthalimide (CTP) and the secondary accelerator tetramethylthiuram monosulphide (TMTM). When used at low levels with sulphenamide accelerators, TiBTM could extend the scorch time by 2-3 minutes and then increase the cure rate beyond that of the primary accelerator. The extent of these effects was influenced by the types of rubber and sulphenamide. At low levels TiBTM performed as an equivalent substitute for a CTP/ TMTM combination, giving equivalent or better vulcanisation and vulcanisate properties. 4 refs.

Accession no.580275

## Item 378

148th ACS Rubber Division Meeting. Fall 1995.
Conference Preprints.
Cleveland, Oh., 17th-20th Oct.1995, Paper 75, pp.21.
012
DCBS: A PREFERRED ACCELERATOR FOR
ADHESION COMPOUNDS AND OTHER TYRE

## **ADHESION COMPOUNDS AND OTHER TYRE APPLICATIONS** Kleiner T; Ruetz L

Bayer AG (ACS,Rubber Div.)

An examination is made of the performance of dicyclohexyl benzothiazole sulphenamide (DCBS) as an accelerator in tyre compounds. Cure characteristics, adhesion and flow properties are discussed, and it is shown that DCBS does not form carcinogenic N-nitrosamines. 8 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; GERMANY; USA; WESTERN EUROPE

Accession no.580258

## Item 379

148th ACS Rubber Division Meeting. Fall 1995. Conference Preprints. Cleveland, Oh., 17th-20th Oct.1995, Paper 68, pp.17. 012

## EFFECT OF 1,6-HEXAMETHYLENE BISSODIUM THIOSULPHATE ON CURE KINETICS OF ACCELERATED SULPHUR VULCANISATION

Ignatz-Hoover F; Dick J S; Maender O W Flexsys America LP (ACS,Rubber Div.)

Duralink HTS 1,6-hexamethylene bissodium thiosulphate (Flexsys America) was used as a post-vulcanisation stabiliser in the sulphur vulcanisation of an NR compound. The effects of this additive on the cure characteristics and on the tensile properties and tear strength of vulcanisates were investigated. The improved reversion resistance, network sulphur rank analysis on overcure and the stoichiometric dependence of activation energy on HTS level supported the conclusion that the active sulphurating species was altered. Improvements in tear strength at equal modulus were consistent with the formation of a bimodal network. 16 refs.

USA

Accession no.580251

Item 380

148th ACS Rubber Division Meeting. Fall 1995. Conference Preprints.

## Cleveland, Oh., 17th-20th Oct.1995, Paper 64, pp.21.012 REVERSION CHEMISTRY: COMPENSATION OF REVERSION AND APPLICATION POSSIBILITIES BY USING 1,3-BISCITRACONIMIDOMETHYLBENZENE

Talma A G; Datta R N; Schotman A H M; Helt W H Akzo Nobel Central Research; Flexsys America LP; Flexsys BV

(ACS,Rubber Div.)

The chemistry of the reversion of rubber vulcanisates is reviewed, and the application of Perkalink 900 1,3biscitraconimidomethylbenzene in the reversion protection of high sulphur NR compounds is described. It is shown that this chemical compensates the loss in crosslinks encountered during reversion by reacting with dienes and trienes formed on the rubber backbone. The effects on the mechanical properties and steel cord adhesion of NR vulcanisates are examined. 13 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; NETHERLANDS; USA; WESTERN EUROPE Accession no.580247

*Item 381* Elastomer Technology Handbook. Boca Raton, Fl., CRC Press, 1993, p.495-518 011 **PEROXIDE CROSSLINKING OF EPDM RUBBERS** 

Endstra W C; Wreesmann C T J Akzo Chemicals BV Edited by: Cheremisinoff N P (SciTech Technical Services)

Typical aspects of crosslinking of EPDM rubber compounds by organic peroxides are reviewed. Special attention is given to compounding considerations, as far as these deviate from general compounding principles applicable for conventional cure systems. Further processing characteristics of peroxide-containing EPDM compounds are discussed, and some pilot compound recipes for different applications are presented. 47 refs. EUROPEAN COMMUNITY; EUROPEAN UNION; NETHERLANDS; USA; WESTERN EUROPE

Accession no.579400

## Item 382

Elastomer Technology Handbook. Boca Raton, Fl., CRC Press, 1993, p.475-93 011 NATURE OF SULPHUR VULCANISATION

Kresja M R; Koenig J L Monsanto Chemical Co.; Case Western Reserve University Edited by: Cheremisinoff N P (SciTech Technical Services)

A detailed review is presented relating to unaccelerated and accelerated sulphur vulcanisation. Examples of accelerator systems are tabulated and reaction mechanisms are given. 94 refs. USA Accession no.579399

Item 383

Kauchuk i Rezina (USSR) No.3, 1994, p.20-4 Russian SALTS OF QUINONE-MONO- AND QUINONE-DIOXIMES: SYNTHESIS AND VULCANISING ACTIVITY IN SKD POLYBUTADIENE RUBBER Avdeenko A P; Glinyanaya N M; Kalinichenko V N

The authors expanded the range of salts of quinone-monoand quinone-dioximes usable for crosslinking of polybutadiene rubber, by synthesising lithium, sodium, potassium, copper, zinc and aluminium salts. Structures and colours of the salts are tabulated and data are given on the vulcanisation characteristics. 5 refs. Articles from this journal can be requested for translation by subscribers to the Rapra produced International Polymer Science and Technology. RUSSIA

Accession no.579168

*Item 384 Gummi Fasern Kunststoffe* 47, No.7, 1994, p.437-48 German **NITROSAMINE-FREE VULCANISING SYSTEMS FOR EPDM** Hofmann W; Diederichsen J Deutsche Oelfabrik

In the concluding part of this study, a critical discussion is presented of the legally permissible N-nitrosamine concentrations, nitrosamine-free vulcanising systems are studied and evaluated, the activation of zinc dithiophosphate is described, and the BG 187 systems are compared with other systems. 73 refs. Articles from this journal can be requested for translation by subscribers to the Rapra produced International Polymer Science and Technology.

EUROPEAN COMMUNITY; EUROPEAN UNION; GERMANY; WESTERN EUROPE

Accession no.579161

## Item 385

**Rubber Chemistry and Technology** 

68, No.4, Sept/Oct.1995, p.563-72 SULPHUR VULCANISATION OF SIMPLE MODEL OLEFINS. IV. VULCANISATIONS OF 2,3-DIMETHYL-2-BUTENE WITH TMTD AND ACTIVATED ZINC DITHIOCARBAMATE/ XANTHATE ACCELERATORS AT DIFFERENT TEMPERATURES

Versloot P; Haasnoot J G; Reedijk J; Van Duin M; Put J Leiden, University; DSM Research BV

The mechanism of the accelerated sulphur vulcanisation of rubber was studied using 2,3-dimethyl-2-butene (TME)

as a simple, low molecular model alkene. Vulcanisation experiments were carried out in the presence of a special accelerator system, i.e. Robac Gamma A1 and A2. 18 refs. EUROPEAN COMMUNITY; EUROPEAN UNION; NETHERLANDS; WESTERN EUROPE

Accession no.577587

#### Item 386

Journal of Applied Polymer Science 59, No.4, 24th Jan. 1996, p.639-45 RADIOISOTOPIC DETERMINATION METHODS OF SULPHUR DISPERSION AND SULPHUR BLOOMING IN RUBBER COMPOUNDS

Jurkowski B; Koczorowska E; Goraczko W; Manuszak J Poznan, University of Technology

The dispersion of sulphur in rubber compounds was studied using radioisotopes. The radioactivity of the sulphur-35 isotope introduced into rubber compounds was measured by volumetric and surface methods. These methods could be used to investigate physical and chemical processes in rubber and to optimise technical conditions of tyre production. It was found that the radioisotopic methods could be used in studies of sulphur blooming. Experimental data were obtained on the effect of mixing and the storage temps. of the rubber compound on these processes. Rubber compounds containing soluble sulphur, insoluble Crystex sulphur and T-accelerator were used for the studies. 20 refs.

EASTERN EUROPE; POLAND *Accession no.576748* 

## Item 387

Rubber World 213, No.2, Nov.1995, p.29/45 INNOVATIVE PEROXIDE AND COAGENT CURE SYSTEMS FOR USE WITH HNBR ELASTOMERS

Recchio M J; Bradford W G Zeon Chemicals Inc.

HNBR has been cured with peroxides since its commercial introduction in 1984. Coagents have been used in peroxide cures to improve processing, physical properties and increase cure speed. Recently, scorch inhibited versions of these materials have been introduced. This article examines the benefits and drawbacks of using scorch inhibited peroxides and coagents. Their effect on processing properties such as Mooney viscosity, scorch and cure rate are evaluated. Tradeoffs in physical properties such as heat resistance, compression set, elongation, tear, water and oil ageing are also evaluated. In the coagent study, the effect on the 10% modulus and the Young's modulus is investigated and the potential benefits discussed. 7 refs.

USA

Accession no.576479

Item 388

Journal of Applied Polymer Science 59, No.2, 10th Jan.1996, p.365-81 EFFECT OF FILLERS IN THE BINARY SYSTEMS CONTAINING TMTD-AMIDINOTHIOUREA AND MBTS-AMIDINOTHIOUREA IN NR VULCANISATION Mathew C; Mini V T E; Kuriakose A P; Francis D J; Amma M L G Cochin,University of Science & Technology; Rubber Research Institute of India

The effect of various fillers (carbon black, precipitated silica and china clay) on the sulphur vulcanisation of NR using amidinothiourea as the secondary accelerator was investigated. Mixing, curing and physical properties of the various vulcanisates were studied. Control mixes were also prepared and their properties compared with those of the new mixes. In order to study the variation in physical properties of the different vulcanisates, chemical crosslinks were also estimated using the equilibrium swelling method. 29 refs.

INDIA

Accession no.575777

Item 389

#### Journal of Applied Polymer Science 58, No.9, 28th Nov.1995, p.1433-40 EFFECT OF CARBON BLACK ON TETRAMETHYLTHIURAM DISULPHIDE ACCELERATOR SULPHUR VULCANISATION OF POLYISOPRENE MaGill W. J. Shalvar S. P.

McGill W J; Shelver S R Port Elizabeth,University

Cis-1,4-polyisoprene (Afprene) was vulcanised with the tetramethylthiuram disulphide/sulphur/zinc oxide system in the presence and absence of N330 HAF black. Crosslinking was carried out in a DSC at a programmed heating rate, the reaction stopped at points along the thermal curve, and the system analysed. Residual curatives and reaction intermediates were determined by HPLC and crosslink densities by swelling in benzene. Combinations of the powdered curatives were also heated with and without carbon black and analysed. It is shown that the step in the vulcanisation sequence, influenced by carbon black, is the formation of tetramethylthiuram polysulphides that act as the active sulphurating agent in vulcanisation. Carbon black catalyses their formation, and to a lesser extent, the formation of accelerator terminated polysulphidic pendent groups on the chain. 20 refs. SOUTH AFRICA

Accession no.571564

#### Item 390

*Journal of Applied Polymer Science* 58, No.12, 19th Dec.1995, p.2185-91 **SULPHUR VULCANISATION OF POLYISOPRENE** 

## ACCELERATED BY BENZOTHIAZOLE DERIVATIVES. I. COMPARISON OF SULPHUR AND 2-MERCAPTOBENZOTHIAZOLE ACCELERATED REACTIONS

Gradwell M H S; McGill W J Port Elizabeth,University

Polyisoprene compounds with sulphur and with sulphur and 2-mercaptobenzothiazole were vulcanised by heating in a DSC calorimeter at a programmed rate. The reaction was stopped at various temperatures along the thermogram and the product analysed by determining the crosslink density and crosslink type, and by determining the amount of extractable curing agents and soluble reaction products by HPLC. DSC cure curves and plots of crosslink density and extractable sulphur versus temperature for the unaccelerated and MBT accelerated compounds can be made to coincide by shifting them along the temperature axis. 31 refs.

SOUTH AFRICA

Accession no.571122

## Item 391

**Rubber and Plastics News** 25, No.5, 9th Oct.1995, p.91-2 **FUNDAMENTALS OF CROSSLINKING WITH PEROXIDES** Class J B

Hercules Inc.

Crosslinking elastomers with peroxides has been known for over 50 years. Serious interest in vulcanisation with peroxides began with the commercial introduction of dicumyl peroxide in the late 1950s. The major feature of this peroxide was its higher curing temperature, which has provided scorch time needed for many rubber manufacturing systems. The main features of peroxide vulcanisation are reviewed and the complications that can interfere with crosslinking performance are discussed. USA

Accession no.569323

## Item 392

Rubber World 212, No.5, Aug.1995, p.24/93 CHEMICAL FOR REVERSION RESISTANT COMPOUNDING Datta R N; Ivany M S Alma Nabal

Akzo Nobel

Reversion of sulphur-based crosslinks continues to a problem for NR compounders. Reversion is the thermal degradation of polysulphidic crosslinks leading to a reduction of crosslink density and an introduction of main chain modifications. In practice, reversion leads to a decline in compound physical properties. Reversion occurs when compounds are overcured or when vulcanisates are exposed to anaerobic ageing. Ideally, a system to address reversion would not affect compound properties except to eliminate reversion. Application of the new system would not alter scorch time, cure rate or optimum properties. Perkalink 900 from Akzo Nobel is a new chemical that addresses reversion without changing compound properties. Comprehensive experimental data and property data are presented. 17 refs.

EUROPEAN COMMUNITY; EUROPEAN UNION; NETHERLANDS; WESTERN EUROPE Accession no.563886

## Item 393

147th Meeting, Spring 1995, Conference Preprints. Philadelphia, Pa., 2nd-5th May 1995, Paper 29, pp.27. 012

## TETRABENZYLTHIURAM DISULPHIDE: A SECONDARY ACCELERATOR FOR STABLE CROSSLINK SYSTEMS IN TYRE APPLICATIONS

Ferrandino M P; Sanders J A; Hong S W Uniroyal Chemical Co.Inc. (ACS,Rubber Div.)

An examination is made of the characteristics obtained by vulcanisation with tetrabenzylthiuram disulphide (TBzTD)/sulphenamide accelerator systems, including reversion resistance, efficient vulcanisation with sufficient scorch delay, and increased nitrosamine safety. It is shown that the combination of properties imparted by these accelerator blends results from low sulphur rank crosslinks provided by the thiuram and the scorch safety provided by the sulphenamide. The advantages of these systems over sulphur donor/sulphenamide and lower molecular weight thiuram/sulphenamide systems are illustrated by the evaluation of blends of TBzTD and Nt-butyl-2-benzothiazole-2-sulphenamide in NR tyre compounds. 13 refs.

USA

Accession no.552365

## Item 394

Handbook of Elastomers. New York,Marcel Dekker Inc.,1988,p.443-84. 011 **HALOGEN-CONTAINING ELASTOMERS** Hertz D L SEALS EASTERN INC. Edited by: Bhowmick A K;Stephens H L

72 refs USA Accession no.406496

## Item 395

Sarnia,Ont., 1983, pp.29. 12ins. 9/6/87. 42C131D12 BUTYL AND THE HALOBUTYLS. PAPER PRESENTED TO THE EDUCATIONAL SYMPOSIUM AT THE ACS, RUBBER DIV.

## **MEETING, HELD HOUSTON, TEXAS, OCTOBER 24, 1983** Wilson G J ACS,RUBBER DIV.; POLYSAR LTD.

Butyl and halobutyl rubbers are considered with respect to their chemical structure, market size, manufacture and plant capacities. The production of both types of rubbers is described, with details of grades and properties. Compounding butyl and halobutyl rubbers is discussed and the use and effect of fillers, plasticisers, tackifiers and processing aids is examined. Vulcanising systems are also described and it is found that butyl rubber requires highly activated cure systems and halobutyl rubber can be cured by zinc oxide with other accelerators and curatives added at low levels for modification of cure rate or state.

CANADA Accession no.397195

Item 396

*Kautschuk und Gummi Kunststoffe* 42,No.1,Jan.1989,p.27-30 German **FORMATION OF NITROSOAMINES IN VULCANISATES** Seeberger D;Raabe G AKZO CHEMIE GMBH

A method for the isolation of volatile and less volatile nitrosoamines from vulcanisates was developed in which the released nitrosoamines can be related to vulcanisate surface area and vulcanisate formulation. Experimental results obtained are suitable for giving a more precise description of origin and formation of some important nitrosoamines in vulcanisates than was previously possible. 20 refs.

WEST GERMANY

Accession no.382048

## Item 397

Kautchuk und Gummi Kunststoffe 42,No.1,Jan.1989,p.22-6 German INFLUENCE OF RECENT LEGISLATION ON PRODUCT DEVELOPMENTS IN THE CHEMICAL INDUSTRY Lohwasser H BAYER AG

Recent West German regulations on health and environmental aspects pf product development are considered with special reference to their effects on the rubber industry. Particular mention is made of the TRGS 552 regulation on volatile carcinogenic nitrosoamines. The interpretation of this regulation is discussed as is the development of possible replacement materials. WEST GERMANY

Accession no.382047

## Item 398

Oxford, Oxford University Press, 1988, pp.xvii,1136. LS.56.25. 9ins. 10/6/88. 41C1 NATURAL RUBBER SCIENCE AND TECHNOLOGY Roberts A D (ed.)

MALAYSIAN RUBBER PRODUCERS' RESEARCH ASSN.

This comprehensive book deals in depth with the history of the rubber industry; biosynthesis of rubber; latex concentrates; technological processing of NR latex; rheology of raw rubber; compounding; injection moulding; compounding for tyres; blends of NR with thermoplastics; chemical modification of NR; non-sulphur vulcanisation; sulphur vulcanisation chemistry; oxidative ageing; graft copolymers from NR; strength properties of NR; friction and wear; diffusion of liquids and solids in rubber; low temperature crystallisation of NR; engineering uses of NR; vibration isolation and earthquake protection of buildings; physical testing and automation; chemical analysis; new applications of rubber.

UK

Accession no.359526

Item 399

IRC '85 Kyoto;International Rubber Conference Proceedings. Kyoto,Japan,15-18 Oct.1985,Paper IL-10,p.73-8. 012 **DEVELOPMENTS IN PERFLUOROCARBON ELASTOMERS** Logothetis A L DU PONT DE NEMOURS E.I.,&

CO.INC.,FABRIC.FIN.DEPT (Japan Society of Rubber Industry)

Copolymers of tetrafluoroethylene with perfluoroalkylvinyl ethers were the ultimate elastomers for fluid, chemical, thermal and oxidative resistance. Cure chemistry mainly determined tensile properties, compression set resistance and upper use-temp. The chemistry was described for a perfluoroelastomer using a nitrile-substituted perfluoroalkylvinyl ether as cure-site monomer. It cured by trimerisation of the nitriles to triazine functionality and was devoid of any C-H bonds. Physical properties and thermal resistance in air over 300C were excellent. Peroxide-curable perfluoroelastomers, using hydrocarbon crosslinkers, were limited in performance. 3 refs.

JAPAN; USA

Accession no.346078

*Item 400* 126th Meeting-Fall. Denver,Col.,October 1984,Paper 73,pp.26. 012 **CROSSLINKING EFFICIENCY OF SOME HALOBUTYL CURING REACTIONS** Vukov R;Wilson G J

## POLYSAR LTD. (ACS,Rubber Div.)

A study was made of the crosslinking efficiency of chlorobutyl and bromobutyl rubbers using as curing agents, zinc oxide, zinc ozide/MBTS/sulphur and zinc oxide/m-phenylene-bis-maleimide. The influence of the monodienophile, N-phenylmaleimide, on the degree of cure of a bromobutyl/zinc oxide/m-phenylene-bismaleimide compound was also investigated. The role of conjugated diene groups in the overall zinc oxide chemistry of the rubbers is examined. 11 refs. CANADA

Accession no.279234

## Item 401

**Rubber Chemistry and Technology** 57,No.1,March/April 1984,p.34-47 **CROSSLINKING OF HALOGEN-CONTAINING RUBBERS WITH TRIAZINE DITHIOLS** Mori K;Nakamura Y IWATE UNIVERSITY

Crosslinking of CR, chlorobutyl rubber, chlorinated acryl rubber, epichlorohydrin rubber, ethylene-epichlorohydrin copolymer rubber, PVC, chlorinated ethylene and vinylidene fluoride-hexafluoropropene copolymer rubbers in presence of metal compounds such as MgO was studied in detail to elucidate mechanism, variables affecting reaction, points of crosslinking and relative reactivity of rubbers. Control techniques using retarders or accelerators, depending on halogen activity, were proposed. 17 refs.

SANKYO KASEI CO.LTD. Japan

Accession no.253816

Item 402 **Rubber Chemistry and Technology** 56,No.5,Nov./Dec.1983,p.883-91 **QUINOID CURING OF BUTYL AND NATURAL RUBBERS** Com L. McChewy C. H.

Gan L M;Chew C H

Results are presented of an investigation of the vulcanisation of butyl and natural rubbers by pnitrosobenzene, p-quinone dioxime and p-quinone dioxime dibenzoate oxidised by various oxidants. Quinoid vulcanisation mechanisms are discussed. 28 refs. (ACS,Rubber Division, Toronto,Canada,May 1983).

MALAYSIA; SINGAPORE

Accession no.245718

Item 403

*Gummi, Asbest, Kunststoffe* 36,No.10,Oct.1983,p.534/8 German **EPDM GRADES WITH IMPROVED** 

#### APPLICATION BEHAVIOUR AT HIGH TEMPERATURES Allen R D

Ways of further improving the thermal stability of EPDM rubbers are discussed. Special emphasis is placed on the effects of polymer structure, crosslinking systems, zinc oxide content, addition of chlorosulphonated PE, antioxidants and coagents on performance characteristics of EPDM at high temps. 2 refs. (Paper presented at 5th Australasian Rubber Technology Convention 1980, PRI Australasian Section).

Accession no.244483

## Item 404

## **Rubber Chemistry and Technology** 56,No.2,May/June 1983,p.327-36 **STUDIES ON THE REACTION BETWEEN THIOCARBAMYLSULPHENAMIDES AND DIBENZOTHIAZYL DISULPHIDE** Adhikari B;Pal D;Basu D K;Chaudhun A K

The results of a study on the reaction between thiocarbamylsulphenamides and dibenzothiazyl disulphide in the solid phase carried out to get an insight into the mutual activity provided by this system in filled and gum NR vulcanisates are reported. Sulphenamide accelerators investigated included N,N-diethylthiocarbamyl- N'-cyclohexylsulphenamide, N-cyclopentamethylenethiocarbamyl-N'- cyclopentamethylenesulphenamide, N-cyclohexyl-2- benzothiazylsulphenamide and N-cyclopentamethylene-2- benzothiazylsulphenamide. Reaction products were identified by high- performance liquid chromatography. 17 refs. INDIA

Accession no.234864

*Item 405 Chemtech* 13,No.2,Feb.1983,p.106-16 **ART OF SULPHUR VULCANISATION** Coran A Y MONSANTO CO.

Research is described which has led to the development of a new class of additives that give precise control of delayed action in the sulphur vulcanisation of rubber. 23 refs. USA

Accession no.228754

## Item 406

## Rubber Chemistry and Technology 55,No.4,Sept./Oct.1982,p.949-60 DEVELOPMENT OF SYNERGISTIC CURING SYSTEMS FOR POLYCHLOROPRENE Kato H; Fujita H

Of the dihydric phenols, catechol exhibited a significant synergistic accelerating effect on joint use with TMTD in curing systems for polychloroprene. Ageing characteristics of the vulcanisates were improved when the aromatic nucleus of the catechol was bonded to sulphur. Dicatechol sulphides (XX-125) derived from catechol and sulphur chloride gave improved vulcanisate thermal stability. Curing properties of XX-125 seemed enhanced by use with TMTD, and a variety of accelerators was developed by blending XX-125 with TMTD at the weight ratio to obtain the desired cure rates and physical properties. These accelerators were suitable substitutes for 2-mercaptoimidazoline (NA-22) in curing polychloroprene. 8 refs.

Accession no.222561

## Item 407

Izvestiya Vysshikh Uchebnykh Zavedenii Khimiya i Khimicheskaya Teknologiya 23,No.5,1980,p.619-21 Russian INFLUENCE OF METAL OXIDES ON VULCANISATION OF RUBBERS BY BENZENE DISULPHOCHLORIDE (BD)

Chekanova A A; Kostrykina G I; Zakharov N D; Degtyarev E V

A study was made of the vulcanisation of SKMS-30 ARK (70:30 butadiene-styrene copolymer) and SKN-26M (butadiene-acrylonitrile copolymer, nominal AN content 27-30%) by BD, using ZnO, CaO or Al2O3 as activators. The activating capacity of the oxides was shown to depend on their reactivity to Cl in ionic or radical form, and on the role of vulcanisation of the metal chlorides formed in the reaction. 9 refs.

Accession no.168210

## *Item 408*

SCIENCE & TECHNOLOGY OF RUBBER; ED.BY F.R.EIRICH NEW YORK, ACADEMIC PRESS, 1978, p.292-338. R.ROOM. 012 VULCANISATION CORAN A Y

Vulcanisation of elastomers is discussed with special reference to the following types of curing: sulphur and accelerated sulphur vulcanisation; vulcanisation by phenolic resins, quinone derivatives and maleimides; vulcanisation by metal oxides; curing with peroxides; and crosslinking by chain extension reactions. Details on thermoplastic elastomers are included. 108 refs.

Accession no.134706

## Item 409

PLASTICS & RUBBER INSTITUTE. PROGRESS OF RUBBER TECHNOLOGY. VOL.41 LONDON,1978, p.61-95. R.ROOM. 012 **CURRENT STATUS OF ELASTOMER VULCANISATION** KIRKHAM M C The chemistry of vulcanisation and the technological properties of the vulcanisates obtained by the different cure systems available are discussed. Sulphur vulcanisation, crosslinking reactions involving free radicals, electron deficient species, ionic addition, ene-addition and dielsalder addition and vulcanisation by dehalogenation and dehydrohalogenation are described. 429 refs.

Accession no.132405

#### Item 410

*Izvestiya Vysshikh Uchebnykh Zavedenii Khimiya i Khimicheskaya Teknologiya* 21, No.3,1978, p.433-6 Russian **CAUSES OF LOW STRENGTH IN RUBBERS FROM SKI-3 WITH BISCHLOROMETHYLXYLENE** SAL'NIKOVA V I; CHEKANOVA A A; ZAKHAROV N D; KOSTRYKINA G I; LITVINOVA L T

The causes for the low strength values of ski-3 (cis-1,4polyisoprene) vulcanisates with bischloromethylxylene are studied. The basic reason for the low strength for nonsulphur vulcanisates in comparison with sulphurcontaining vulcanisates is the low degree of crosslinking and the high degree of non-uniformity in the vulcanisation network, and the resulting low rate of orientation crystallisation. 11 refs.

Accession no.123332

#### Item 411

Kauchuk i Rezina (USSR) No.9,1975, p.15-8 Russian EFFECT OF 'EFFECTIVE' CURING SYSTEMS AND CURING TEMPERATURE ON TYPE OF CROSSLINK AND PROPERTIES OF POLYISOPRENE RUBBER VULCANISATES SHAPKIN A N; TOLSTUKHINA F S; LAPSHOVA A A; FEDOROV A G

A study was made of the effect of 'effective' curing systems on the properties of  $sk_{1-3}$  vulcanisates, cured normally (at 143c) and at elevated temperatures (180c). The curing agents used were n,n'- dithiodimorpholine and n,n'- tetrathiodimorpholine. Differences in the type of crosslink formed at different temperatures by ordinary and 'effective' curing systems were investigated. The ratio between the different types of crosslink was correlated with the properties of the vulcanisates. The advantages of using the more effective sulphur donor (n,n'-tetrathiodimorpholine) are demonstrated. 9 refs.

Accession no.55324

*Item 412 Journal of Applied Polymer Science* 19, No.3, MARCH 1975, p.865-77

## BIS(DIISOPROPYL)THIOPHOSPHORYL DISULPHIDE IN CIS-1,4-POLYISOPRENE VULCANISATION REACTIONS. I. AS A SULPHUR DONOR

## PIMBLOTT J G; SCOTT G; STUCKEY J E

It was shown that the network structure consisted of polyand disulphidic crosslinks at early stages of cure, simplifying at optimum cure to monosulphic crosslinks. It is thought that pendent accelerator groups are bound to the rubber molecule at early stages of cure, but are subsequently replaced by cyclic sulphidic groups. The good thermal and thermal oxidative ageing behaviour of the vulcanisate was due to the formation of zinc diisopropyldithiophosphate in situ. 53 refs.

Accession no.50533

## Item 413

## PUBLN.BA 115 pp.4. 12ins. 2/10/74. 6B221 TIMING BELTS FOR EXACT SPEED DRIVES. WIGAN, C.1974 TBA INDUSTRIAL PRODUCTS LTD.,BELTING

DIV.; TURNER & NEWALL LTD.

The belts consist of a synthetic rubber backing, reinforced with glass cord tension members and protected by a nylon fabric coating on the teeth. The size range available is listed.

TURNER & NEWALL LTD.; TBA INDUSTRIAL PRODUCTS LTD., BELTING DIV. Accession no. 12097

## Item 414

Kauchuk i Rezina (USSR) 31, No.4,1972, p.20-2 Russian VULCANISATION OF RUBBERS WITH HEXACHLORO-P-XYLENE SHERSHNEV V A; GLUSHKO V V; DOGADKIN B A; KURMEEVA A I

Strongly basic amines and certain metal oxides are found to have an activating effect on the vulcanisation of rubbers by hexachloro-p-xylene. On the basis of chemical analysis of the vulcanisates, mechanisms of the reaction of the hexachloro-p-xylene with diene elastomers are suggested. The possibility is also established of vulcanisation of diene rubbers with hexachloro-p-xylene and sulphur. 3 refs.

Accession no.32510

Item 415

## *Journal of Applied Polymer Science* 16, No.5, May 1972, p.1191-212 EFFECT OF VULCANISATION ON LOW-STRAIN DYNAMIC PROPERTIES OF FILLED RUBBERS

PAYNE A R; WHITTAKER R E; SMITH J F

A change in the degree of vulcanisation in a carbon black filled rubber is shown not to affect the change of in-phase shear modulus with strain amplitude, provided the amount of filler is kept constant. Similar results are found with the change of out-of-phase modulus and phase angle with strain amplitude. A similar value of change of in-phase shear modulus with strain amplitude is found for a vulcanised and unvulcanised tyre tread rubber; this behaviour is attributed to the breakdown of the secondary three-dimensional carbon black aggregated network. Detailed consideration is given to other factors which contribute to the difference between the shear modulus of the gum rubber and the filled rubber when all the carbon black structure has been broken down. 58 refs.

Accession no.19395

#### Item 416

## *Vysokomolekulyarnye Soedineniya* 13B, No.9, Sept.1971, p.692-5 Russian **REACTION BETWEEN CHLORANIL AND UNSATURATED POLYMERS** VINOGRADOV P A; KASTORSKII L P; GAVSHINOVA K E; ARSEN'EVA N G

The results of an IR and chemical analysis of some rubbers cured with chloranil (tetrachloro-p-benzoquinone) are discussed. The compounds tested consisted of 10 pts. Chloranil and 0-5 pts. Zinc oxide to 100 pts. Rubber(cis-1,4-polybutadiene, sodium butadiene rubber and nitrile rubber). It is shown that, during curing, dehydrogenation is accompanied by addition of chloranil to the polymer molecules by a diels-alder and other reactions. 9 refs.

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