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Second edition
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Rubber compounding ingredients — *p*-Phenylenediamine antidegradants (PPDs) — Test methods

Ingrédients de mélange du caoutchouc — Antidégradants du type p*-phénylènediamine (PPDs) — Méthodes d'essai*

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 3, *Raw materials (including latex) for use in the rubber industry*.

This second edition cancels and replaces the first edition (ISO 11236:2000), which has been technically revised. It also incorporates the Technical Corrigendum ISO 11236:2000/Cor.1:2006.

The main changes compared to the previous version are as follows:

- removal of DTPD (*N*,*N*′-ditolyl-*p*-phenylenediamine), which is widely acknowledged to be hazardous to health and environment;
- transfer in an informative Annex A of the precision and bias for the determination of purity by gas chromatography by procedure A, for the determination of ash and for the determination of volatile matter;
- addition of a Bibliography.

Rubber compounding ingredients — *p*-Phenylenediamine antidegradants (PPDs) — Test methods

WARNING — Persons using this document should be familiar with normal laboratory practice. This document does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

1 Scope

This document applies to a variety of substituted *p*-phenylenediamine antidegradants (PPDs) used in the rubber industry. The three general classes of PPDs are dialkyl, alkyl-aryl and diaryl, which are used to impart ozone resistance to rubber.

The following test methods are of greatest significance in assessing the purity of production PPDs, and hence their suitability for use in rubber. They are specified in this document as follows:

- determination of purity by gas chromatography (GC);
- determination of purity by high-performance liquid chromatography (HPLC);
- determination of ash;
- determination of volatile matter.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 1042, *Laboratory glassware — One-mark volumetric flasks*

ISO 1772, *Laboratory crucibles in porcelain and silica*

ISO 15528, *Paints, varnishes and raw materials for paints and varnishes — Sampling*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at http://www.iso.org/obp
- IEC Electropedia: available at http://www.electropedia.org/

3.1
area normalization
method of calculating the percent composition by measuring the area of each component peak observed in a chromatogram and dividing the area of the peak by the total peak area for all the components observed

3.2
lot sample
production sample representative of a standard production unit

3.3
test portion

actual material used in the analysis

Note 1 to entry: The test portion shall be representative of the lot sample.

4 Abbreviated terms

PPDs	substituted *p*-phenylenediamine antidegradants

The following abbreviations, taken from ISO 6472, are used in the text.

77PD	*N,N′*-bis(1,4-dimethylpentyl)-*p*-phenylenediamine
IPPD	*N*-isopropyl-*N’*-phenyl-*p*-phenylenediamine
6PPD	*N*-1,3-dimethylbutyl-*N′*-phenyl-*p*-phenylenediamine

5 Use and classification

PPDs represent the primary additive used in tyres and other mechanical rubber goods to impart ozone protection and to improve resistance to fatigue cracking. PPDs are also used as antioxidants in a number of applications.

Although all PPDs exhibit similar performance characteristics, particular types are frequently preferred for certain end-use conditions, for example, the type and degree of flexing experienced by the rubber article.

PPDs are classified into the following types.

a) Type I: *N,N′*-dialkyl PPDs (see Figure 1)

R and R’ are secondary alkyl groups, usually C_6 or larger. These materials are generally liquids at ambient conditions and consist for the most part (>90 %) of a single chemical component.

b) Type II: *N*-alkyl-*N′*-aryl PPDs (see Figure 2)

R is a secondary alkyl group and R′ is an aryl substituent (usually phenyl). These materials generally consist of a single component or a mixture containing two or more major components (mainly isomers).

c) Type III: *N*, *N′*-diaryl PPDs (see Figure 3)

R and R′ can be the same aryl group or different (usually phenyl or *p*-tolyl) and can be single components or mixtures of three or more isomers. This type of PPDs is generally solid at ambient conditions.

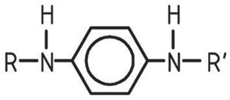


Figure 1 — Type I: *N,N′*-dialkyl *p*-phenylenediamines

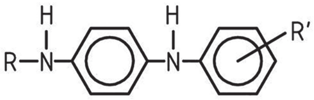


Figure 2 — Type II: *N*-alkyl-*N′*-aryl *p*-phenylenediamines

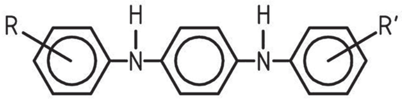


Figure 3 — Type III: *N*,*N*′-diaryl *p*-phenylenediamines

6 Sampling and repeat determinations

Carry out sampling in accordance with ISO 15528. To ensure homogeneity, blend at least 250 g of the lot sample thoroughly prior to removing any test portions.

If the difference between the results of duplicate determinations exceeds the repeatability given for the method concerned, repeat the test. If no repeatability figure is given for a particular method, report the results of both determinations.

7 Determination of purity by gas chromatography (GC)

7.1 General

This method is designed to assess the relative purity of production PPDs by determining the purity of type I, II and III PPDs using temperature-programmed gas chromatography with either a packed column (procedure A) or a capillary column (procedure B). Quantification is achieved by area normalization using a peak integrator or chromatography data system.

Since the results are based on area normalization, the method assumes that all components are eluted from the column and each component has the same detector response. Although this is not strictly true, the errors introduced are relatively small and much the same for all samples. Thus, they can be ignored, since the intent of the method is to establish relative purity.

Although trace amounts of “low boilers” are present in production samples, they are disguised by the solvent peak when using packed columns (procedure A).

7.2 Interference

Utilizing the chromatographic conditions prescribed, there are no significant co-eluting peaks. However, degradation of column performance could result in interference problems. Thus, when using the packed column, it is essential that the total system be capable of 5 000 theoretical plates. Evaluation of system efficiency is described in 7.3.2.

7.3 Apparatus

7.3.1 Gas chromatograph.

7.3.1.1 Procedure A

Any high-quality temperature-programmed gas chromatograph equipped with a thermal-conductivity detector and a peak integrator or chromatography data system is sufficient for this analysis.

Although a thermal-conductivity detector is recommended, a flame-ionization detector (FID) can be used if appropriate adjustment is made for flow rate and sample size. This will probably involve using a smaller-diameter column, in which case, the adjustment in flow rate and injection volume shall be proportional to the cross-sectional area of the column.

7.3.1.2 Procedure B

Any high-quality temperature-programmed gas chromatograph with a flame-ionization detector and equipped for capillary columns is suitable. When utilizing standard capillary columns (0,25 mm), a split injection system is required. However, a “cold on-column” injector is preferred for the wide-bore (0,53 mm) capillaries. The FID shall have sufficient sensitivity to give a minimum peak-height response of 30 µV for 0,1 mass % of 6PPD when operated at the stated conditions. Background noise at these conditions shall not exceed 3 µV.

7.3.2 Gas-chromatographic columns.

7.3.2.1 Procedure A

Use a glass column with the following dimensions: 1,8 m × 6,4 mm outside diameter × 4 mm inside diameter. The glass column shall be packed with 10 % methyl silicone fluid (100 %) on a 0,15/0,08 mm (80/100 mesh) acid-washed and silanized diatomite support.

Condition the column with a helium flow of approximately 20 cm^3/min by programming from ambient temperature to 350 °C at the rate of 2 °C/min to 3 °C/min and holding at 350 °C overnight with the detector disconnected.

When using a packed column, a minimum of 5 000 theoretical plates, as measured from the analyte peak, under the chromatographic conditions stated in Table 1, is required for analysis. The number of theoretical plates, N, is determined by Formula (1).

$$N = 5{,}5\left[\frac{X(\mathrm{R})}{Y(0{,}5)}\right]^2 \tag{1}$$

where

$X(\mathrm{R})$ is the retention time measured from the injection point to the apex of the 6PPD peak (adjust the attenuation to keep peak on scale), in mm;

$Y(0{,}5)$ is the 6PPD peak width at half height, in mm.

7.3.2.2 Procedure B

Use one of the following:

- a 30 m × 0,25 mm fused-silica capillary internally coated to a film thickness of 0,25 µm (bonded) with methyl silicone (column 1), or
- a 15 m × 0,53 mm fused-silica (megabore) capillary coated with a 3,0 µm bonded film of 5 % phenyl silicone, HP-5 or equivalent (column 2).

7.3.3 Integrator/data system.

Use an integrator/data system capable of determining the relative amount of each component by integration of the detector output as a function of time. When using capillary columns (procedure B), the equipment shall be capable of integrating at a sufficiently fast rate so that narrow peaks (1 s peak width) are accurately measured.

7.3.4 Volumetric flask, capacity 10 cm^3, meeting the requirements of ISO 1042.

7.3.5 Mortar and pestle.

7.3.6 Precision balance, accurate to ±1 mg or better.

7.3.7 Syringe, of suitable size (see relevant procedure).

7.4 Calibration and standardization

Chromatograms from typical PPDs run on the packed columns in accordance with the prescribed procedure are given in Figures 4 and 5.

NOTE When using the conditions described for procedure A (packed column), the detector response for injections of 500 µg to 5 000 µg of 6PPD was found to be somewhat nonlinear. However, over the more limited range 750 µg to 2 500 µg, the response was nearly linear. It is important that the samples be prepared so that 1 250 µg to 1 500 µg injections are made.

7.5 Procedure

7.5.1 Sample preparation

To ensure homogeneity, grind lot samples of PPDs with a mortar and pestle prior to weighing out the test portion. In the case of liquid PPDs where partial crystallization might have occurred resulting in fractionation, melt the lot sample, with occasional stirring, prior to weighing out the test portion. Care should be taken that the sample does not decompose during the process. For example, in the case of 6PPD, it is recommended to melt the sample in an oven at 50 °C to 60 °C.

7.5.2 Procedure A

7.5.2.1 Use the following chromatographic conditions:

— helium flow rate	50 cm^{3}/min;
— injection-port temperature	300 °C;
— initial column temperature	100 °C;
— heating rate	8 °C/min;
— final temperature	350 °C;
— detector temperature	350 °C;
— detector attenuation	8.

7.5.2.2 Weigh a 2,5 g to 3,0 g test portion (to the nearest milligram) into a 10 cm^{3} volumetric flask, dilute to volume with methylene chloride and shake well to dissolve.

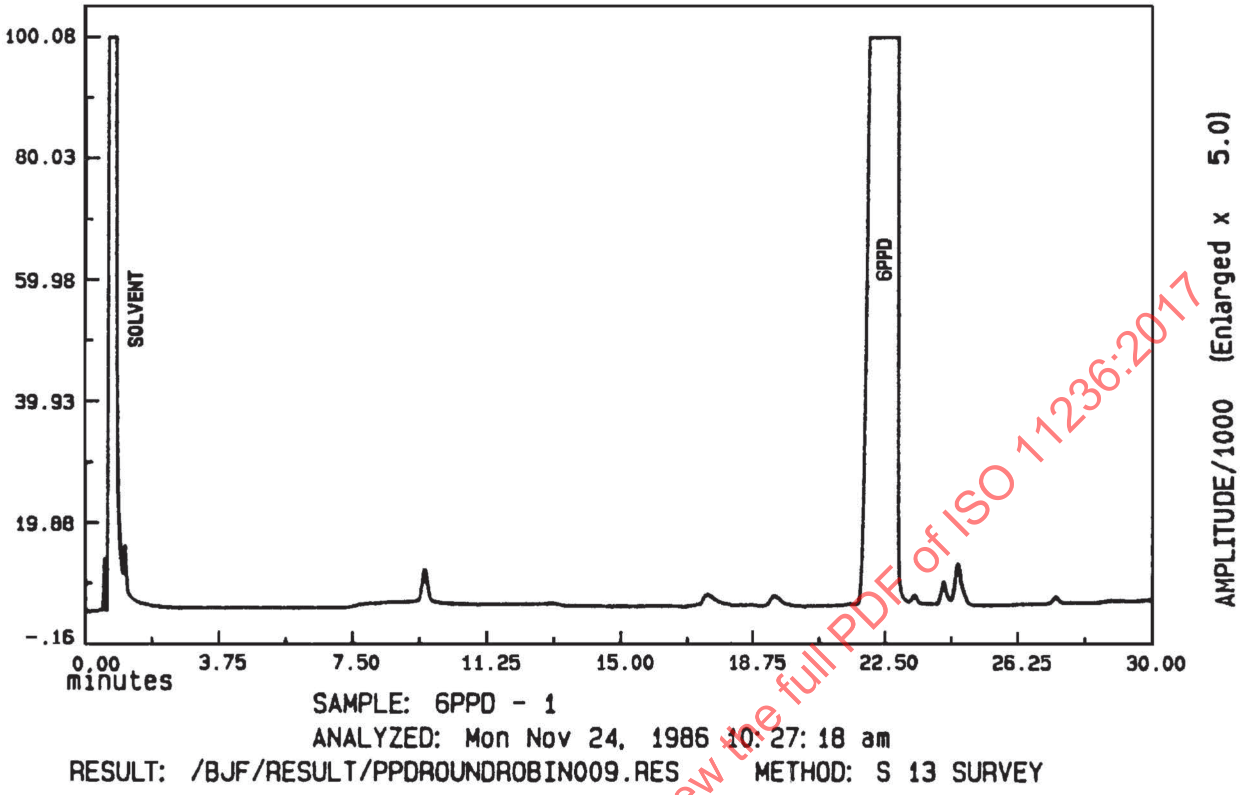
7.5.2.3 When the instrument has equilibrated at the initial conditions described above, inject 5,0 mm^{3} (µl) of sample solution (see 7.5.2.2) and initiate the temperature programme and data collection.

Sample size and carrier-gas flow rates shall be adjusted according to the cross-sectional area of the column utilized. For example, if a nominally 3,2 mm outside diameter column (1,87 mm inside diameter) is used rather than a 6,4 mm outside diameter column (3,54 mm inside diameter), the adjustment would be as follows: the ratio of the cross-sectional areas is $(3{,}54/1{,}87)^2$, which equals 3,6. Thus, the sample size and helium-carrier flow rate shall be decreased by this factor, i.e. the sample size to 5/3,6 or 1,4 mm^{3} (µl) and the flow rate to 50/3,6 or 14 cm^{3}/min.

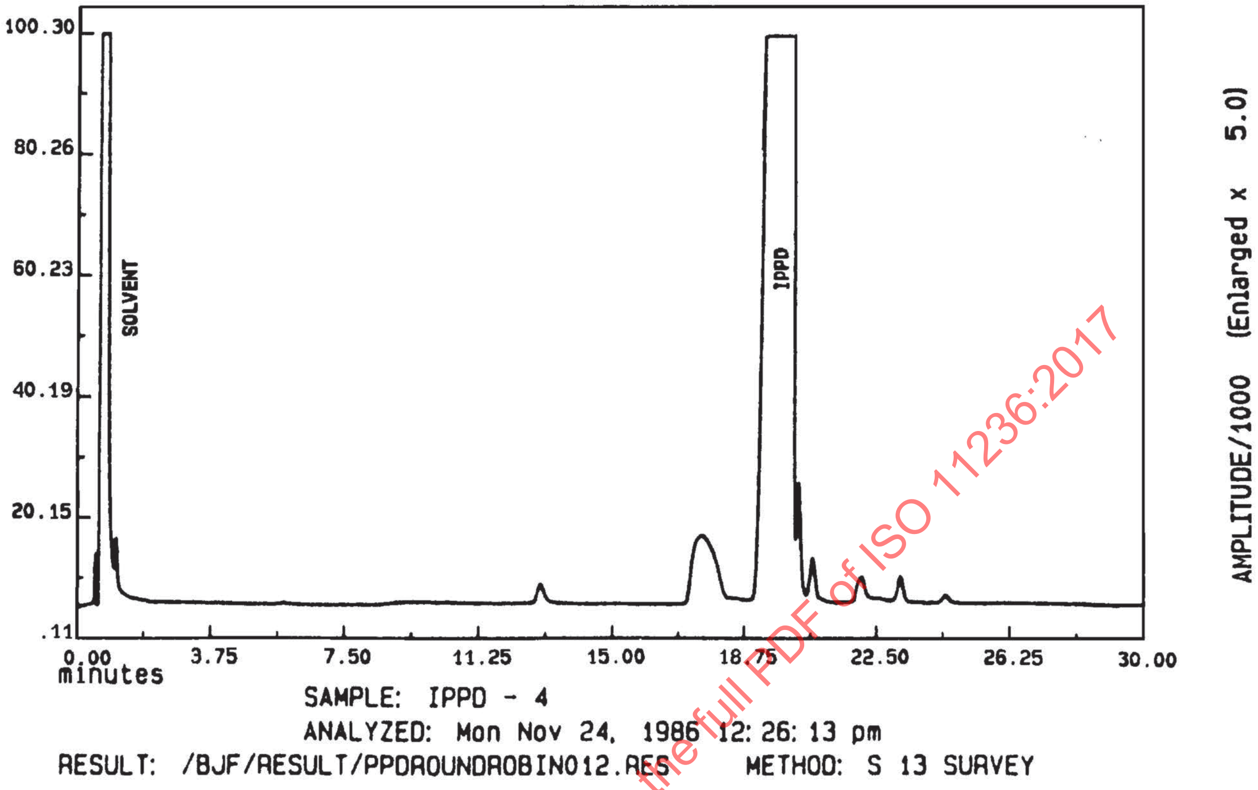
7.5.2.4 When the run is complete, inspect the chromatogram and output data for proper appearance and peak identification (see Figure 4).

7.5.2.5 Repeat the run described in 7.5.2.3 on the same sample.

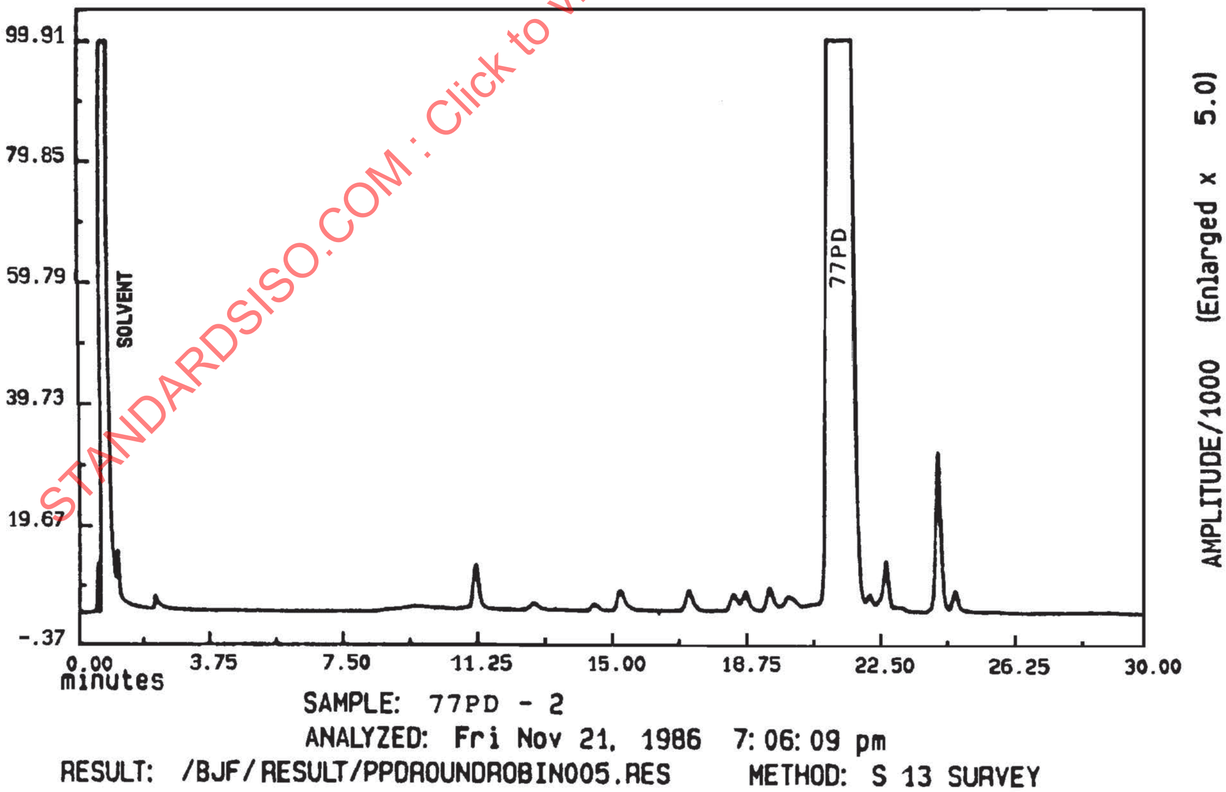
7.5.2.6 Typical chromatograms obtained by procedure A for 6PPD, IPPD and 77PD are shown in Figures 4 a) to 4 c), respectively.



a) 6PPD



b) IPPD



c) 77PD

Figure 4 — Chromatograms obtained using procedure A

7.5.3 Procedure B

7.5.3.1 The suggested operating conditions for the analysis using a capillary column are given in Table 1. Column 1 is a standard capillary and column 2 a megabore capillary.

Table 1 — Chromatographic conditions in procedure B

	Column 1	Column 2	
Column size	30 m × 0,25 mm	15 m × 0,53 mm	
Stationary phase	Bonded methyl silicone	Bonded 5 % phenyl silicone	
Film thickness	0,25 µm	3,0 µm	
Carrier gas	Helium	Helium	
Linear velocity at 100 °C	0,34 m/s	NA	
Flow rate	1,0 cm^3/min	30 cm^3/min	
Head pressure, gauge	60 kPa	NA	
Detector	FID	FID	
Detector temperature	300 °C	300 °C	
Injection-port temperature	300 °C	Oven tracking	
Hydrogen flow rate[a]	30 cm^3/min	30 cm^3/min	
Air flow rate[a]	300 cm^3/min	300 cm^3/min	
Make-up gas	Nitrogen or helium	Nitrogen or helium	
Make-up gas flow rate[a]	29 cm^3/min	10 cm^3/min	
Split ratio	180:1	No split	
Column-temperature programme		Ramp A	Ramp B
Initial temperature	42 °C	35 °C	240 °C
Programme rate	9 °C/min	15 °C/min	8 °C/min
Final temperature	300 °C	240 °C	290 °C
Time at final temperature	22 min	3 min	17 min
Injection volume	0,4 mm^3 (µl)	1 mm^3 (µl)	
Solvent	Methylene chloride	Methylene chloride	
Test-portion concentration	10 mg/cm^3	3 mg/cm^3	

[a] Consult the manufacturer's manual for optimum flow rates on different instruments.

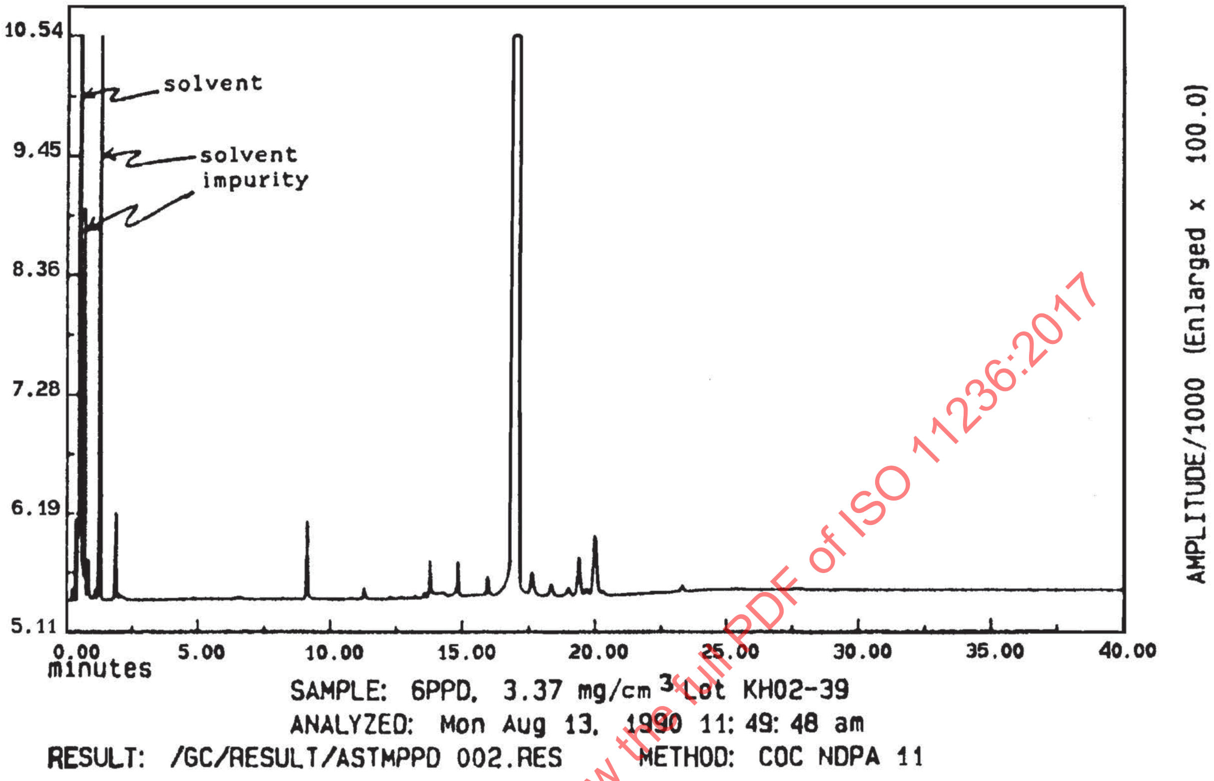
7.5.3.2 Prepare the test portion in accordance with Table 1.

7.5.3.3 When the instrument has equilibrated at the initial conditions given in Table 1, inject the indicated amount of diluted test portion and immediately start the recorder, integrator and column-temperature programming sequence.

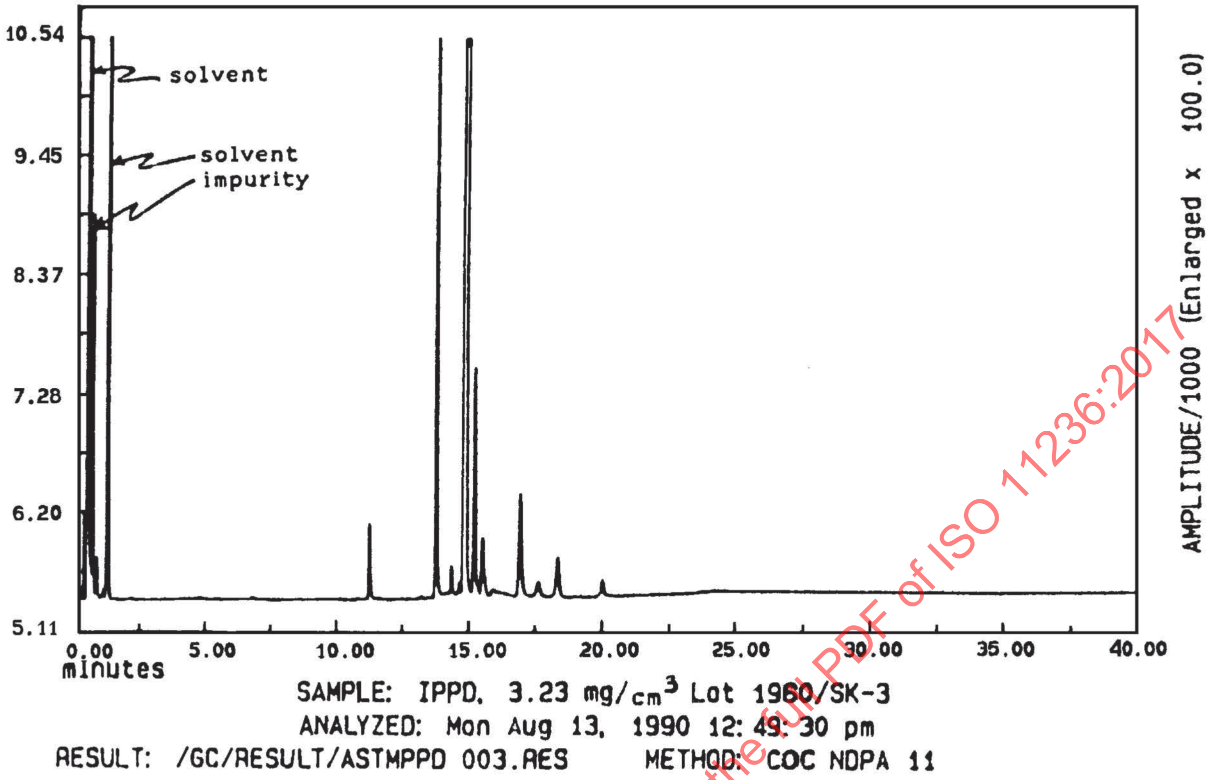
7.5.3.4 When the analysis is complete, inspect the chromatogram and output data for proper appearance and peak identification.

7.5.3.5 Repeat the run described in 7.5.3.3 on the same sample.

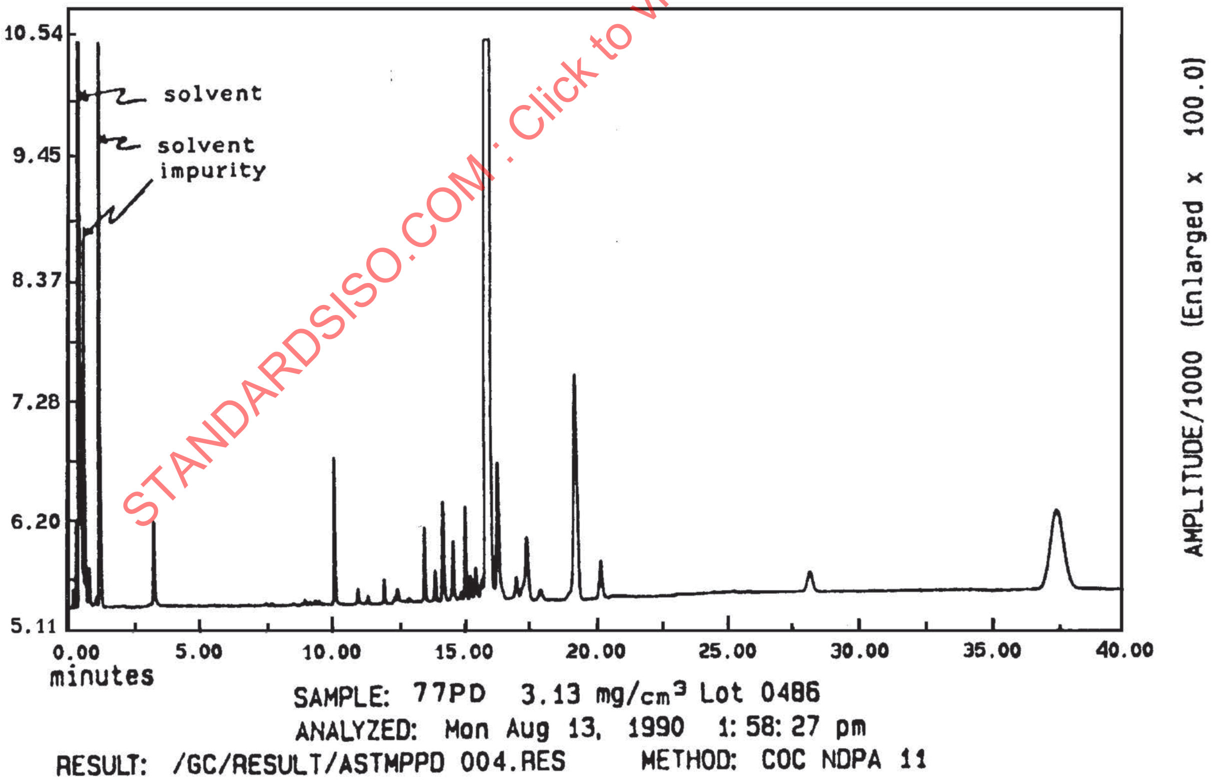
7.5.3.6 Typical chromatograms obtained by procedure B using a megabore capillary for 6PPD, IPPD and 77PD are shown in Figures 5 a) to 5 c), respectively.



a) 6PPD



b) IPPD



c) 77PD

Figure 5 — Chromatograms obtained using procedure B

7.6 Calculation

For each run, calculate the relative percent peak area, P_A, for PPDs and the other identified components according to Formula (2).

$$P_A = \frac{A_C}{A_T} \times 100 \qquad (2)$$

where

A_C is the area count for the component in question;

A_T is the total area count for all the components.

7.7 Precision and bias

See Annex A for the precision and bias data for determination of purity by gas chromatography by procedure A.

7.8 Test report

The test report shall include the following information:

a) a reference to this document, i.e. ISO 11236;

b) all details necessary for complete identification of the sample;

c) the procedure used (A or B);

d) the result obtained from each of the two individual injections, and the mean value, expressed to the nearest 0,1 %;

e) the combined area of all the unidentified peaks, expressed as “percent other”;

f) details of any deviations from the procedure specified, as well as details of any unusual incidents likely to have affected the results;

g) the date of the test.

8 Determination of purity by high-performance liquid chromatography (HPLC)

8.1 General

This method determines the purity of Type I, II and III PPDs by reverse-phase HPLC with UV detection. Quantification is achieved by using an external standard.

8.2 Apparatus

8.2.1 Precision balance, accurate to ±0,1 mg.

8.2.2 Shaker or **ultrasonic bath**.

8.2.3 Volumetric flask, capacity 100 cm^3, according to ISO 1042.

8.2.4 Screw-cap vials, capacity 125 cm^3.

8.2.5 Syringe, capacity 2 cm^3.

8.2.6 Mortar and pestle.

8.2.7 High-performance liquid chromatograph.

Any high-quality HPLC apparatus equipped with an autosampler or loop injector, a variable-wavelength UV detector and a peak integrator or chromatography data system is suitable for this analysis.

8.2.8 HPLC columns.

- Precolumn: C_{18} silica, diameter 35 µm to 50 µm.
- Analytical column: C_{18} silica, 3 µm to 5 µm, 150 mm × 4,6 mm.

8.3 Reagents

8.3.1 HPLC-grade solvents.

8.3.2 Base salt.

8.3.3 Water, with a resistivity of >200 MΩ·m (e.g. double-distilled).

8.3.4 PPDs working standard, the purity of which is ensured by the use of an acceptable purification technique.

8.4 Procedure

8.4.1 Chromatographic conditions

Use the following conditions.

— Eluent flow rate	1 cm^3/min
— Column	C_{18} 150 mm × 4,6 mm
— Injection volume	20 mm^3 (µl)
— Detector wavelength	290 nm
— Capacity factor k'	$3 < k' < 15$
— Test portion	20 mg
— Solvent	for example, CH_3CN
— Eluent	for example, CH_3CN 65 % water 35 % ethanolamine 0,1 g/l

Samples shall be stored in the dark at a temperature below 5 °C.

8.4.2 Preparation of test solutions

8.4.2.1 Using a mortar and pestle, grind at least 5 g of the lot test sample of the PPDs to be analysed.

8.4.2.2 Perform the procedure described in 8.4.2.3 to 8.4.2.4 on duplicate test portions taken from the ground lot sample (8.4.2.1).

8.4.2.3 Into a tared 125 cm^3 screw-cap vial, weigh, to the nearest 0,15 mg, an amount (m_2) of the ground lot sample, so as to obtain a suitable detector response (see 8.4.5.3).

8.4.2.4 Add 100 cm^3 of a suitable solvent (for example, CH_3CN for 6PPD) to each vial, cap the vials and dissolve the test portion by shaking or using an ultrasonic bath.

8.4.3 Preparation of reference solutions

Using the PPDs working standard, prepare reference solutions in the same manner as the test solutions were prepared in 8.4.2.

8.4.4 Preparation of eluent

8.4.4.1 The eluent is a solvent or mixture of solvents which may or may not contain a base salt (see 8.4.1). Its composition is dictated by the capacity factor k' and resolution R of the apparatus (see 8.4.5.5).

8.4.4.2 Degas the eluent with helium or nitrogen before use.

8.4.5 HPLC analysis

8.4.5.1 Rinse and condition the precolumn at 25 °C ± 1 °C using the proper eluent for the product being analysed (for example, CH_3CN, water and ethanolamine for 6PPD).

8.4.5.2 Using the eluent flow rate and detection wavelength given in 8.4.1, inject the reference solutions as follows.

a) Manual method: using a 2 cm^3 syringe, make duplicate injections, each about 100 mm^3 (µl), of each of the reference solutions into the sample loop (i.e. a total of four injections).

b) Automatic method: programme the autosampler to make duplicate injections, each about 100 mm^3 (µl), of each of the reference solutions (i.e. a total of four injections).

8.4.5.3 Calculate the response factors, R_F, and their mean, $\overline{R_F}$, and check them according to Formulae (3) and (4).

$$R_F = \frac{m_1}{A} \tag{3}$$

where

m_1 is the mass, in milligrams, of working standard in 100 cm^3 of reference solution;

A is the area of the peak produced by the working standard.

$$P_{disp} = \frac{W \times 100}{\overline{R_F}} \tag{4}$$

where

P_{disp} is the percentage dispersion;

W is the range between a pair of duplicate determinations of the response factor;

$\overline{R_F}$ is the mean response factor.

8.4.5.4 Inject the test solutions as in 8.4.5.2.

8.4.5.5 Validate the chromatographic conditions by calculating the capacity factor, k', and resolution, R, according to Formulae (5) and (6).

$$k' = \frac{t_1 - t_0}{t_0} \tag{5}$$

where

t_1 is the retention time of the analyte;

t_0 is the column dead time.

The calculated value of k' shall be between 3 and 15.

$$R = 2\frac{|t_1 - t_2|}{tw_1 + tw_2} \tag{6}$$

where

t_1 is the retention time of the analyte;

t_2 is the retention time of a non-retained component;

tw_1, tw_2 are the corresponding peak widths at 10 % of peak height.

The calculated value of R shall be above 1.

8.5 Calculation

Calculate the PPDs content, P_{PPDs}, in percent, according to Formula (7).

$$P_{\text{PPDs}} = \overline{R_{\text{F}}} \times \frac{A}{m_0} \times \frac{V_{\text{e}}}{V_{\text{r}}} \times 100 \times K \tag{7}$$

where

$\overline{R_{\text{F}}}$ is the mean response factor;

m_0 is the mass of the test portion, in milligrams;

V_{e} is the volume of the test solution, in cubic centimetres;

V_{r} is the volume of the reference solution, in cubic centimetres;

A is the area of the component peak;

K is the working-standard purity correction factor.

8.6 Precision and bias

No precision and bias data are currently available for this method.

8.7 Test report

The test report shall include the following information:

a) a reference to this document, i.e. ISO 11236;

b) all details necessary for complete identification of the sample;

c) the results obtained from the two individual determinations and their average, reported to the nearest 0,1 %;

d) details of any deviations from the procedure specified as well as details of any unusual incidents likely to have affected the results;

e) the date of the test.

9 Determination of ash

9.1 General

The ash is determined by heating a known mass of product over a gas burner or by equivalent method, to volatilize/pyrolyse the organic material, followed by ignition of the remaining carbonaceous material in a muffle furnace. The amount of ash remaining is expressed as a percentage of the original material.

The ash produced by a material is made up of all the components that remain after combustion, irrespective of chemical form. In effect, the determination measures inorganic impurities that can remain in the product at low levels following the manufacturing process.

Such impurities in accelerators or antidegradants can affect the performance of these additives in rubber if critical levels are exceeded.

9.2 Apparatus

9.2.1 Muffle furnace, capable of temperature regulation to ±25 °C between 500 °C and 800 °C.

9.2.2 Laboratory gas burner, or equivalent system.

9.2.3 Laboratory fume hood.

9.2.4 Porcelain crucible, high form, 15 cm^3 capacity, meeting the requirements of ISO 1772.

9.2.5 Clay triangle.

9.2.6 Steel crucible tongs.

9.2.7 Heat-resistant gloves.

9.2.8 Desiccator.

9.2.9 Analytical balance, accurate to ±0,1 mg.

9.3 Procedure

9.3.1 Heat the 15 cm^3 crucible in the muffle furnace at 750 °C ± 25 °C for 30 min.

9.3.2 Transfer the crucible to the desiccator, allow to cool to room temperature and weigh to the nearest 0,1 mg (m_1).

9.3.3 Weigh a nominally 2 g test portion to the nearest 0,1 mg into the crucible (m_2). Place the crucible in the clay triangle, and carefully heat the crucible and contents with the gas burner until all volatile material and pyrolysis products have been removed (gases may flame) and the residue has been carbonized. The pre-ignition temperature shall not exceed the ignition temperature.

9.3.4 Transfer the crucible to the muffle furnace and heat for 2 h at 750 °C ± 25 °C.

9.3.5 Carefully transfer the crucible containing the ash to the desiccator, allow to cool to room temperature, and reweigh to the nearest 0,1 mg (m_3).

Repeat the procedure on a second test portion.

9.4 Calculation

Calculate the ash, A, in percent, to the nearest 0,01 % according to Formula (8).

$$A = \left[\frac{m_3 - m_1}{m_2 - m_1}\right] \times 100 \tag{8}$$

where

m_1 is the mass of the crucible, in grams;

m_2 is the mass of the crucible plus test portion, in grams;

m_3 is the mass of the crucible plus ash, in grams.

9.5 Precision and bias

See Annex A for the precision and bias data for determination of ash.

9.6 Test report

The test report shall include the following information:

a) a reference to this document, i.e. ISO 11236;

b) all details necessary for complete identification of the sample;

c) the results obtained from the two individual determinations and their average, reported to the nearest 0,01 %;

d) details of any deviations from the procedure specified as well as details of any unusual incidents likely to have affected the results;

e) the date of the test.

10 Determination of volatile matter

10.1 General

A test portion of the antidegradant is weighed before and after heating for 3 h at 70 °C. The measured difference in mass is the volatile-matter content.

The quantity of volatile matter in PPDs can affect the performance of these antidegradants in rubber if it exceeds critical levels.

10.2 Apparatus

10.2.1 Weighing bottle, low form.

10.2.2 Air-circulation oven, capable of being maintained at a temperature of 70 °C ± 2 °C.

10.2.3 Desiccator.

10.2.4 Analytical balance, accurate to ±0,1 mg.

10.3 Preparation of sample

To ensure homogeneity, blend at least 250 g of the lot sample thoroughly prior to removing the test portion.

10.4 Procedure

10.4.1 Perform the following procedure (10.4.2 to 10.4.5) on duplicate test portions.

10.4.2 Dry a clean weighing bottle and stopper (with the stopper removed) for 30 min in the oven at 70 °C. Place the bottle and stopper in the desiccator and allow them to cool to room temperature. Weigh the bottle with its stopper to the nearest 0,1 mg (m_1).

10.4.3 Weigh a nominally 5 g test portion into the weighing bottle to the nearest 0,1 mg (m_2).

10.4.4 Place the weighing bottle containing the test portion, and the stopper (with the stopper removed), in the oven, equilibrated at 70 °C ± 2 °C, for 3 h.

10.4.5 After the heating period, replace the stopper and transfer the bottle to the desiccator for a period of time sufficient for the assembly to equilibrate at room temperature. Reweigh the bottle to the nearest 0,1 mg (m_3).

10.5 Calculation

Calculate the volatile-matter content, C_{vm}, in percent, to the nearest 0,1 % according to Formula (9).

$$C_{\text{vm}} = \left[\frac{m_2 - m_3}{m_2 - m_1} \right] \times 100 \qquad (9)$$

where

m_1 is the mass of the weighing bottle and stopper, in grams;

m_2 is the mass of the weighing bottle, stopper and test portion before heating, in grams;

m_3 is the mass of the weighing bottle, stopper, and test portion after heating, in grams.

10.6 Precision and bias

See Annex A for the precision and bias data for determination of volatile matter.

10.7 Test report

The test report shall contain the following information:

a) a reference to this document, i.e. ISO 11236;

b) all details necessary for complete identification of the sample;

c) the results obtained from the two individual determinations and their average, reported to the nearest 0,01 %;

d) details of any deviations from the procedure specified as well as details of any unusual incidents likely to have affected the results;

e) the date of the test.