



JAPANESE
INDUSTRIAL
STANDARD

Translated and Published by
Japanese Standards Association

JIS K 6220-3 : 2018

(JRMA/JSA)

**Rubber compounding ingredients—
Organic chemicals—Test methods—
Part 3: *p*-Phenylenediamine
antidegradants (PPDs)**

ICS 83.040.20

Reference number : JIS K 6220-3 : 2018 (E)

PROTECTED BY COPYRIGHT



Documents

Document Sharing Hub

14 S

Date of Establishment: 2001-01-20

Date of Revision: 2018-02-20

Date of Public Notice in Official Gazette: 2018-02-20

Investigated by: Japanese Industrial Standards Committee
Standards Board for ISO area

JIS K 6220-3:2018, First English edition published in 2018-06

Translated and published by: Japanese Standards Association
Mita MT Building, 3-13-12, Mita, Minato-ku, Tokyo, 108-0073 JAPAN

In the event of any doubts arising as to the contents,
the original JIS is to be the final authority.

© JSA 2018

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from the publisher.

Printed in Japan

PROTECTED BY COPYRIGHT

Contents

	Page
Introduction	1
1 Scope	1
2 Normative references	1
3 Terms and definitions	2
4 Abbreviations and names of chemical components	2
5 Sampling	2
6 Method for determination of purity	2
6.1 General	2
6.2 Method for determination of purity by gas chromatography (GC)	2
6.3 Method for determination of purity by high performance liquid chromatography (HPLC)	7
Annex A (informative) Precision of method for testing purity of <i>p</i> -phenylenediamine antidegradants (PPDs)	12
Annex JA (informative) Gas chromatogram of <i>p</i> -phenylenediamine antidegradants (PPDs)	14
Annex JB (informative) Representative qualitative characteristic values of antidegradants	18
Annex JC (informative) Comparison table between JIS and corresponding International Standard	20

Foreword

This Japanese Industrial Standard has been revised by the Minister of Economy, Trade and Industry through deliberations at the Japanese Industrial Standards Committee as the result of proposal for revision of Japanese Industrial Standard submitted by The Japan Rubber Manufacturers Association (JRMA)/Japanese Standards Association (JSA) with the draft being attached, based on the provision of Article 12 Clause 1 of the Industrial Standardization Law applicable to the case of revision by the provision of Article 14. Consequently **JIS K 6220-3**:2001 is replaced with this Standard.

This **JIS** document is protected by the Copyright Law.

Attention is drawn to the possibility that some parts of this Standard may conflict with patent rights, applications for a patent after opening to the public or utility model rights. The relevant Minister and the Japanese Industrial Standards Committee are not responsible for identifying any of such patent rights, applications for a patent after opening to the public or utility model rights.

JIS K 6220 series consists of the following three parts under the general title “*Rubber compounding ingredients*”:

JIS K 6220-1 *Rubber compounding ingredients—Organic chemicals—Test methods—Part 1: General*

JIS K 6220-2 *Rubber compounding ingredients—Organic chemicals—Test methods—Part 2: Sulfenamide accelerators*

JIS K 6220-3 *Rubber compounding ingredients—Organic chemicals—Test methods—Part 3: p-Phenylenediamine antidegradants (PPDs)*

JIS K 6220-4 *Rubber compounding ingredients—Organic chemicals—Part 4: Abbreviated terms*

Rubber compounding ingredients— Organic chemicals—Test methods— Part 3: *p*-Phenylenediamine antidegradants (PPDs)

Introduction

This Japanese Industrial Standard has been prepared based on **ISO 11236:2017**, Edition 2, with some modifications of the technical contents.

The dotted underlines indicate changes from the corresponding International Standard. A list of modifications with the explanations is given in Annex JC.

1 Scope

This Standard specifies the test methods for the purity of *p*-phenylenediamine antidegradants (hereafter referred to as PPDs) used as a rubber compounding ingredient.

WARNING Persons using this Standard should be familiar with normal laboratory practice. This Standard does not purport to address all of the safety problems, if any, associated with its use.

It is the responsibility of the user of this Standard to establish appropriate safety and health practices.

NOTE : The International Standard corresponding to this Standard and the symbol of degree of correspondence are as follows.

ISO 11236:2017 *Rubber compounding ingredients—*p*-Phenylenediamine antidegradants (PPDs)—Test methods* (MOD)

In addition, symbols which denote the degree of correspondence in the contents between the relevant International Standard and **JIS** are IDT (identical), MOD (modified), and NEQ (not equivalent) according to **ISO/IEC Guide 21-1**.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this Standard. The most recent editions of the standards (including amendments) indicated below shall be applied.

JIS K 0211 *Technical terms for analytical chemistry (General part)*

JIS K 5600-1-2 *Testing methods for paints—Part 1: General rules—Section 2: Sampling*

NOTE : Corresponding International Standard: ISO 15528 *Paints varnishes and raw materials for paints and varnishes—Sampling*

JIS K 6220-4 *Rubber compounding ingredients—Organic chemicals—Part 4: Abbreviated terms*

JIS K 8032 *Acetonitrile (Reagent)*

JIS K 8109 2-Aminoethanol (Reagent)

JIS Z 8401 Guide to the rounding of numbers

3 Terms and definitions

For the purpose of this Standard, the terms and definitions given in **JIS K 0211** apply.

4 Abbreviations and names of chemical components

For the purpose of this Standard, the abbreviations and names of chemical components of PPDs given in **JIS K 6220-4** apply.

5 Sampling

Sampling shall be carried out in accordance with **JIS K 5600-1-2**.

6 Method for determination of purity

6.1 General

Purity can be determined by gas chromatography (GC) or high performance liquid chromatography (HPLC).

6.2 Method for determination of purity by gas chromatography (GC)

6.2.1 Outline

In this method, sample is analysed by a gas chromatograph (GC). From the chromatogram obtained, the peak area of the analyte is calculated using a data processing system. Then, the purity is determined by the area percentage method. The following two measuring methods are available depending on the column to be used.

- a) **Method A**, using a packed column.
- b) **Method B**, using a capillary column.

6.2.2 Method A

6.2.2.1 Instrument and apparatus

The instrument and apparatus shall be as follows.

- a) **Gas chromatograph**, a high-quality temperature-programmable apparatus equipped with a thermal conductivity detector (TCD).

NOTE : Although a TCD is recommended, a flame ionization detector (FID) may also be used if appropriate adjustment is made for the flow rate of gas and the injection volume of sample.

- b) **Packed column**, as specified below.
 - 1) **Column**, made of glass with a dimension of 1.8 m × 3.54 mm (inside diameter).
 - 2) **Liquid phase**, of methyl silicone (100 % purity in mass fraction) at the liquid phase impregnation rate of 10 % in mass fraction.

- 3) **Support**, of acid-washed and silanized diatomite with a particle size of 0.15 mm to 0.18 mm (100 to 80 mesh).
- 4) **Pretreatment** Condition the packed column with a helium flow of 20 cm³/min by programming from room temperature to 350 °C at the rate of 2 °C/min to 3 °C/min and by holding at 350 °C overnight for pre-burning with the detector disconnected. Refrain from injecting helium into the TCD and leave the power of the TCD off.
- 5) **Theoretical plate number** The number of theoretical plates, N , shall be determined by Formula (1).

When the 6PPD peak is measured under the chromatographic conditions stated in Table 1, a minimum of 5 000 theoretical plates shall be required.

$$N = 5.5 \times \left[\frac{X(R)}{Y(0.5)} \right]^2 \dots\dots\dots (1)$$

where, N : number of theoretical plates

$X(R)$: the length, on the chart, of the retention time from the injection point to the peak (mm)

$Y(0.5)$: peak width at half height (mm)

- c) **Data processing system**, capable of indicating chromatogram, retention time, peak area, peak height, etc.
- d) **Volumetric flask**, capacity 10 cm³.
- e) **Mortar and pestle**
- f) **Balance**, capable of weighing to the nearest 1 mg.
- g) **Microsyringe**, of sufficient capacity.

6.2.2.2 Sample preparation

Sample shall be prepared as follows before weighing.

- a) Grind the sample, if solid, in a mortar so as to ensure homogeneity.
- b) When solid and liquid PPDs are mixed, warm the sample at temperatures not causing decomposition to melt with occasional stirring.

NOTE : Representative qualitative characteristic values of antidegradants are shown in Annex JB.

6.2.2.3 GC conditions

- a) GC conditions shall be in accordance with Table 1.

Table 1 GC conditions for Method A

Helium flow rate (cm ³ /min)	50
Injection-port temperature (°C)	300
Initial column temperature (°C)	100
Heating rate (°C/min)	8
Final temperature (°C)	350
Detector temperature (°C)	350
Detector	TCD

6.2.2.4 Test procedure

The test procedure shall be as follows.

- a) Weigh out about 2.5 g to 3.0 g of sample into a 10 cm³ volumetric flask, dissolve with methylene chloride and take this as sample solution.
- b) When the apparatus satisfies the conditions given in Table 1 for starting analysis, inject 5.0 mm³ of sample solution into a gas chromatograph with a microsyringe and initiate the measurement. An autosampler may be used for injection.

NOTE 1 When a small-diameter column is used, the flow rate of gas and the injection volume of sample can be adjusted in proportion to the cross-sectional area of the column.

- c) When the test is complete, analyse the chromatogram.
- d) Conduct the test twice on the same sample.

NOTE 2 Examples of gas chromatogram obtained by Method A are shown in Annex JA.

6.2.3 Method B

6.2.3.1 Instrument and apparatus

The instrument and apparatus shall be as follows.

- a) **Gas chromatograph**, a high-quality temperature-programmable apparatus equipped with a FID and as specified below.
 - 1) When a capillary column of 0.25 mm in inside diameter is used, a split injection system is required.
 - 2) When a wide-bore capillary column of 0.53 mm in inside diameter is used, a cold on-column injector is recommended.
- b) **Capillary column** Use either standard capillary column or wide-bore capillary column indicted below.
 - 1) **Standard capillary column**, fused-silica capillary column of 30 m × 0.25 mm (inside diameter) which is internally coated with methyl silicone of 0.25 µm in film thickness.

- 2) **Wide-bore capillary column**, fused-silica capillary column of 15 m × 0.53 mm (inside diameter) which is internally coated with 5 % phenyl silicone, HP-5 or equivalent, of 3.0 µm in film thickness.
- c) **Data processing system**, capable of indicating chromatogram, retention time, peak area, peak height etc.
- d) **Volumetric flask**, capacity 10 cm³.
- e) **Mortar and pestle**
- f) **Balance**, capable of weighing to the nearest 1 mg.
- g) **Microsyringe**, of sufficient capacity.

6.2.3.2 Sample preparation

Sample shall be prepared in accordance with 6.2.2.2.

6.2.3.3 GC conditions

GC conditions shall be in accordance with Table 2.

Table 2 GC conditions for Method B

		Standard capillary column	Wide-bore capillary column	
Column size [length (m) × inside diameter (mm)]		30 × 0.25	15 × 0.53	
Stationary phase		Bonded methyl silicone	Bonded 5 % phenyl silicone	
Film thickness (µm)		0.25	3.0	
Carrier gas		Helium	Helium	
Linear velocity, 100 °C (m/s)		0.34	—	
Flow rate (cm ³ /min)		1.0	30	
Head pressure, gauge (kPa)		60	—	
Detector		FID	FID	
Detector temperature (°C)		300	300	
Injection-port temperature (°C)		300	Oven tracking	
Hydrogen flow rate ^{a)} (cm ³ /min)		30	30	
Air flow rate ^{a)} (cm ³ /min)		300	300	
Make up gas		Nitrogen or helium	Nitrogen or helium	
Make up gas flow rate ^{a)} (cm ³ /min)		29	10	
Split ratio		180 : 1	—	
Column temperature programme		Setting 1	Setting 2	Setting 3
	Initial temperature (°C)	42	35	240
	Programme rate (°C/min)	9	15	8
	Final temperature (°C)	300	240	290
	Retention time at final temperature (min)	22	3	17