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JIS K 6220-2 : 2001

(JRMA/JSA)

**Rubber compounding ingredients—
Test methods—Part 2 : Organic
accelerators and vulcanizing agents**

ICS 83.040.20

Descriptors : gums, factice, chemical analysis and testing

Reference number : JIS K 6220-2 : 2001 (E)

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Foreword

This translation has been made based on the original Japanese Industrial Standard established by the Minister of Economy, Trade and Industry through deliberations at the Japanese Industrial Standards Committee according to the proposal of establishing a Japanese Industrial Standard from The Japan Rubber Manufacturers Association (JRMA)/the Japanese Standards Association (JSA), with a draft of Industrial Standard based on the provision of Article 12 Clause 1 of the Industrial Standardization Law.

By this establishment, **JIS K 6202 : 1979** and **JIS K 6203 : 1979** have been withdrawn and replaced with this Standard.

JIS K 6220 consists of the following three parts with the general title *Rubber compounding ingredients—Test methods*.

Part 1 : General

Part 2 : Organic accelerators and vulcanizing agents

Part 3 : Antidegradants

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Rubber compounding ingredients— Test methods—Part 2 : Organic accelerators and vulcanizing agents

Introduction This Japanese Industrial Standard has been prepared on the basis of the first edition of **ISO 11235 Rubber compounding ingredients—Sulfenamide accelerators—Test methods** published in 1999, with some modifications in the technical contents.

Furthermore, portions sidelined or dot-underlined in this Standard are the modified or added matters of the original International Standard. The table of modification with the explanation is shown in Annex 4.

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 to establish appropriate safety and health practices.

1 Scope This Standard specifies the test methods for physical and chemical properties of the organic accelerators and vulcanizing agents used as rubber compounding ingredients.

Remarks : The International Standard corresponding to this Standard is as follows.

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**.

ISO 11235 : 1999 *Rubber compounding ingredients—Sulfenamide accelerators—Test methods* (MOD)

2 Normative references The following standards contain provisions which, through reference in this Standard, constitute provisions of this Standard. If the indication of the year of publication is given to these referred standards, only the edition of indicated year constitutes the provision of this Standard but the revision and/or amendment made thereafter are not applied. The normative references without the indication of the year of publication apply limiting only to the most recent edition (including amendments).

JIS K 0050 General rules for chemical analysis

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

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

JIS K 8001 General rule for test methods of reagents

JIS K 8101 Ethanol (99.5)

JIS K 8180 Hydrochloric acid

JIS K 8322 Chloroform

JIS K 8355 *Acetic acid*

JIS K 8464 *Cyclohexane*

JIS K 8622 *Sodium hydrogen carbonate*

JIS K 8680 *Toluene*

JIS K 8839 *2-Propanol*

JIS K 8891 *Methanol*

JIS K 8913 *Potassium iodide*

JIS K 8951 *Sulfuric acid*

JIS Z 8401 *Guide to the rounding of numbers*

JIS Z 8801-1 *Test sieves—Part 1 : Test sieves of metal wire cloth*

ISO 6472 : 1994 *Rubber compounding ingredients—Abbreviations*

ISO 6472 : 1994/Amd. 1 : 1999 *Rubber compounding ingredients—Abbreviations
Amendment 1 : New materials*

ISO/TR 9272 : 1986 *Rubber and rubber products—Determination of precision for test
method standard*

3 Definitions For the main terms used in this Standard the definitions in **JIS K 0211** apply.

4 Classification of test The classification of test shall be as follows.

- a) Purity of sulfenamide organic accelerators
- b) Purity of peroxyketal compounds
- c) Purity of benzoyl peroxides
- d) Insoluble matter content of sulfenamide organic accelerators
- e) Melting point
- f) Volatile content
- g) Particle size distribution in wet system
- h) Residue on sieve in wet system
- i) Ash content

5 Sampling method The sampling method shall be in accordance with the method specified in clause 6 of **JIS K 6220-1**.

6 Abbreviations and names of chemical components for organic accelerators and vulcanizing agents The abbreviations and names of chemical components for organic accelerators and vulcanizing agents to be used in this Standard have been prepared based on **ISO 6472** and **ISO 6472 : 1994/Amd. 1** and specified in Table 1 by adding the abbreviations and names of chemical components which are in common use in Japan.

- Remarks 1 The abbreviations and names of chemical components which are not mentioned in **ISO 6472** and **ISO 6472 : 1994/Amd. 1** have been dot-underlined.
- 2 In this table, when two abbreviations without underlines appear in succession, the first abbreviation stands for that mainly used in North America and the second abbreviation stands for that mainly used in Europe.

Table 1 Abbreviations and names of chemical components for organic accelerators and vulcanizing agents

Classification	Abbreviation	Name of chemical components
Organic accelerator	Aldehyde-ammonia	HMT, <u>H</u>
		Hexamethylenetetramine
	Aldehyde-amine	BA, <u>BAA</u>
		Butylaldehyde-aniline condensate Reaction product of <i>n</i> -butylaldehyde and aniline
	Guanidine	DPG
		Diphenylguanidine <u>1,3-diphenylguanidine</u>
	DOTG	Di- <i>o</i> -tolylguanidine <u>1,3-di-<i>o</i>-tolylguanidine</u>
	Thiourea	ETU, <u>EU</u>
		Ethylenethiourea <u>2-mercaptoimidazoline</u>
		DETU, <u>DEU</u>
		1,3-diethylthiourea <u>Diethylthiourea</u>
	DBTU	1,3-dibutylthiourea <u>Dibutylthiourea</u>
	TMU	<u>Trimethylthiourea</u>
	Thiazole	MBT
		2-mercaptobenzothiazole
		MBTS
		Benzothiazole disulfide Benzothiazyl disulfide <u>Dibenzothiazyl disulfide</u>
		ZMBT, <u>ZnMBT</u>
		Zinc 2-mercaptobenzothiazole
	CMBT	<u>Cyclohexylamine salt of 2-mercaptobenzothiazole</u>
	MBDS, MDS, <u>MDB</u>	2-morpholinodithio-1,3-benzothiazole 4-morpholino-2-benzothiazyl disulfide <u>2-(4'-morpholinodithio) benzothiazole</u>
	Sulfenamide	CBS
		<i>N</i> -cyclohexylbenzothiazole-2-sulfenamide <i>N</i> -cyclohexylbenzothiazyl sulfenamide <u><i>N</i>-cyclohexyl-2-benzothiazyl sulfenamide</u>
	TBBS, <u>BBS</u>	<i>N</i> -tert-butylbenzothiazole-2-sulfenamide <i>N</i> -tert-butylbenzothiazyl sulfenamide <u><i>N</i>-tert-butylbenzothiazyl-2-sulfenamide</u>
	DCBS	<i>N,N</i> -dicyclohexylbenzothiazole-2-sulfenamide <i>N,N</i> -dicyclohexylbenzothiazyl sulfenamide

Table 1 (concluded)

Classification		Abbreviation	Name of chemical components
	Sulfenamide	DIBS, <u>DPBS</u>	<i>N,N</i> -diisopropylbenzothiazole-2-sulfenamide <i>N,N</i> -diisopropylbenzothiazyl sulfenamide
	Thiuram	TMTM	Tetramethylthiuram monosulfide
		TMTD	Tetramethylthiuram disulfide
		TETD	Tetraethylthiuram disulfide
		TBTD	Tetrabutylthiuram disulfide
		DPTH DPTT	Dipentamethylenethiuram hexasulfide Dipentamethylenethiuram tetrasulfide
	Dithiocarbamate	SEDC, SDEC, <u>NaEDC</u>	Sodium diethyldithiocarbamate
		SBDC, SDBC, <u>NaBDC</u>	Sodium dibutyldithiocarbamate <u>Sodium di-<i>n</i>-butyldithiocarbamate</u>
		ZMDC, ZDMC, <u>ZnMDC</u>	Zinc dimethyldithiocarbamate
		ZEDC, ZDEC, <u>ZnEDC</u>	Zinc diethyldithiocarbamate
		ZBDC, ZDBC, <u>ZnBDC</u>	Zinc dibutyldithiocarbamate <u>Zinc di-<i>n</i>-butyldithiocarbamate</u>
		ZPMDC, ZPMC, <u>ZnPDC</u>	Zinc pentamethylenedithiocarbamate
		ZEPDC, ZEPC, <u>ZnEPDC</u>	Zinc ethylphenyldithiocarbamate
		TeEDC, TeDEC	Tellurium diethyldithiocarbamate
		CuMDC, CuDMC	Copper dimethyldithiocarbamate
		<u>FeMDC</u>	<u>Ferric dimethyldithiocarbamate</u>
		PPDC	Piperidinium pentamethylenedithiocarbamate Piperidine pentamethylenedithiocarbamate
		<u>PMPDC</u>	<u>Pipecolin methylpentamethylenedithiocarbamate</u>
	Xanthate	ZIX, <u>ZnPX</u>	Zinc isopropylxanthate
		ZBX, <u>ZnBX</u>	Zinc butylxanthate
Organic vulcanizing agents	Organic peroxide	<u>DTBPC</u>	<u>1,1-bis(tert-butylperoxy) cyclohexane</u>
		<u>DBPMC</u>	<u>1,1-bis (tert-butylperoxy)-2-methylcyclohexane</u>
		DBPC	1,1-bis(tert-butylperoxy)-3,3,5-trimethylcyclohexane 1,3,3-trimethyl-5,5-di-tert-butylperoxy cyclohexane
		<u>DBPB</u>	<u>2,2-bis(tert-butylperoxy) butane</u>
		BPV	<i>n</i> -butyl bis(4,4-tert-butylperoxy) valerate 3,3-bis(tert-butylperoxy) butane carboxylic acid- <i>n</i> -butyl ester
		BPO	Benzoylperoxide Dibenzoylperoxide
	Dioxime	BQD, PBQD, <u>QO</u>	<i>p</i> -benzoquinone dioxime
		DBQD, <u>BQO</u>	<i>p,p'</i> -dibenzoyl- <i>p</i> -benzoquinone dioxime Quinone dioxime dibenzoate

7 Methods for determination of purity of sulfenamide organic accelerators

7.1 Method to determine the purity by reduction with 2-mercaptobenzothiazole (hereafter referred to as "MBT".)

7.1.1 Purpose This test method specifies the procedures to determine the purity of sulfenamide organic accelerators; CBS, DCBS and TBBS, and to determine free amine.

7.1.2 Principle of test method After neutralizing the free amine, the sulfenamide is reduced by means of a solution of MBT. An excess of hydrochloric acid is added and the unreacted hydrochloric acid is then titrated with sodium hydroxide by using either of the following two methods:

Method A: Potentiometric titration

Method B: Titration using an indicator

7.1.3 Instruments and apparatus The instruments and apparatus shall be as follows.

- a) **Mortar and pestle**
- b) **Pipette**, 25 cm³ in capacity.
- c) **Burette**, 25 cm³ in capacity, graduated in 0.05 cm³.
- d) **Beaker**, 250 cm³ in capacity.
- e) **Volumetric flask**
- f) **Water bath**, capable of maintaining the temperature at (55 ± 2) °C.
- g) **Magnetic stirrer**
- h) **pH meter**, capable of measuring to the unit of 0.1 pH (or 10 mV).
- i) **Chemical balance**, capable of weighing to the nearest 0.1 mg.

7.1.4 Reagents The reagents of recognized analytical grade shall be used for the reagents. Distilled water or water of equivalent purity (specified in 7.3 of **JIS K 0050**) shall be used for the water.

7.1.4.1 Reagents required for Method A and Method B

- a) **MBT**, not less than 99.0 % in purity.
- b) **Ethanol**, specified in JIS K 8101.
- c) **Toluene**, specified in JIS K 8680.
WARNING: Toluene is a toxic and inflammable solvent.
- d) **0.1 mol/dm³ hydrochloric acid**, specified in JIS K 8001.
- e) **0.5 mol/dm³ hydrochloric acid**, specified in JIS K 8001.
- f) **0.5 mol/dm³ sodium hydroxide solution**, specified in JIS K 8001.
- g) **Bromophenol blue solution** (hereafter referred to as "indicator"), specified in JIS K 8001.

7.1.4.2 Preparation of reagent for Method A

- a) **MBT 40 g/dm³ solution** Weigh a suitable amount of MBT to the nearest 0.1 g in a suitable volumetric flask, dissolve in ethanol by adding to an appropriate quantity. Dissolve it completely at not higher than 57 °C in temperature, cool it to room temperature and add ethanol to the marked line. Prepare this solution when used.

7.1.4.3 Preparation of reagent for Method B

- a) **Ethanol/toluene mixed solution** Use the mixed solution of ethanol and toluene in the volume ratio 5 : 3.
- b) **MBT 40 g/dm³ solution** Weigh a suitable amount of MBT to the nearest 0.1 g in a suitable volumetric flask, add the mixed solution of ethanol and toluene of appropriate quantity to dissolve. Dissolve it completely at a temperature not higher than 57 °C, cool it to room temperature and add the mixed solution of ethanol and toluene to the marked line. Prepare this solution when used.

7.1.5 Procedure of test method The procedure of test method shall be as follows.

7.1.5.1 Method A

- Grind the sample and weigh out approximately 2 g of the powder to the nearest 0.1 mg. However, for TBBS, weigh out approximately 1.6 g to the nearest 0.1 mg, and transfer it into a beaker.
- Add 50 cm³ of ethanol, and stir until dissolved. If needed, heat the solution to a temperature not higher than 55 °C. A slight turbidity may be neglected.
- Cool it to room temperature, add 3 drops of indicator, and titrate the free amine with 0.1 mol/dm³ hydrochloric acid until blue-green colour vanishes. Make the titration amount at this time V_1 .
- Add 50 cm³ of previously prepared MBT solution [7.1.4.2 a)] and immediately weigh out and add accurately 25 cm³ of 0.5 mol/dm³ hydrochloric acid by using a pipette.
- Stir the solution in a water bath maintained at (55 ± 2) °C for exactly 5 min by a magnetic stirrer.
- Titrate potentiometrically the unreacted hydrochloric acid with the 0.5 mol/dm³ sodium hydroxide solution.
- With continuous stirring, add the sodium hydroxide solution, and record the indicated potential difference corresponding to the titration amount of the sodium hydroxide solution or the pH.

The end point of titration shall be the point of inflection⁽¹⁾ of the titration curve. The titration amount at this time shall be V_2 .

Note (1) Draw two tangents on the slant of 45° in angle to the titration curve, and the reading on the horizontal axis corresponding to the intersection of the parallel line in equal distance from these lines with the titration curve shall be the inflection point (see Fig. 1).

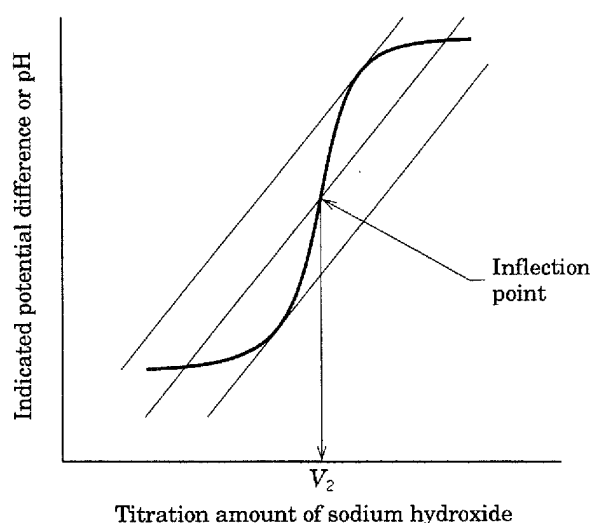


Fig. 1 Example of method for obtaining inflection point

7.1.5.2 Method B

- Grind the sample and weigh out approximately 2 g of the powder to the nearest 0.1 mg. However, for TBBS, weigh out approximately 1.6 g to the nearest 0.1 mg, and transfer it into a beaker.
- Add 50 cm³ of the mixed solution of ethanol/toluene, and stir until dissolved. If needed, heat the solution to a temperature not higher than 55 °C. A slight turbidity may be neglected.
- Cool it to room temperature, add 3 drops of indicator, and titrate the free amine with 0.1 mol/dm³ hydrochloric acid until blue-green colour vanishes. Make the titration amount at this time V_1 .
- Add 50 cm³ of previously prepared MBT solution [7.1.4.3 b)] immediately add accurately 25 cm³ of 0.5 mol/dm³ hydrochloric acid with a pipette.
- Stir the solution in a water bath maintained at (55 ± 2) °C for exactly 5 min.
- Add 3 drops of indicator, and titrate the unreacted hydrochloric acid with 0.5 mol/dm³ sodium hydroxide until the colour of blue-green changes to blue. The titration amount at this time shall be V_2 .

7.1.6 Calculation

7.1.6.1 Free amine The free amine content (%) shall be calculated according to the following formula.

$$F = \frac{V_1 \times C_1}{10 \times m_1} \times M_1$$

where, F : free amine content (%)

V_1 : amount of 0.1 mol/dm³ hydrochloric acid required for the titration of sample (cm³)

C_1 : concentration of hydrochloric acid used for titration (mol/dm³)

m_1 : mass of sample (g)

M_1 : molecular weight of amine corresponding to sample
(see Table 2)

Table 2 Molecular weight of amine corresponding to sample

Class of sulfenamide	Molecular weight of amine corresponding to sample
CBS	99.18
DCBS	181.32
TBBS	73.14

7.1.6.2 Purity The purity (%) shall be calculated according to the following formula.

$$P_1 = \frac{(25 \times C_2) - (V_2 \times C_3)}{10 \times m_2} \times M_2$$

where, P_1 : purity (%)

C_2 : concentration of hydrochloric acid added (mol/dm³)

C_3 : concentration of sodium hydroxide solution used for titration (mol/dm³)

V_2 : amount of 0.5 mol/dm³ sodium hydroxide solution required for the titration of sample (cm³)

m_2 : mass of sample (g)

M_2 : molecular weight of sulfenamide in sample (see Table 3)

Table 3 Molecular weight of sulfenamide in sample

Class of sulfenamide	Molecular weight of sulfenamide in sample
CBS	264.41
DCBS	346.58
TBBS	238.37

7.2 Method to determine purity by high performance liquid chromatography (HPLC)

7.2.1 Purpose This test method specifies the procedure to determine the purity of sulfenamide organic accelerators (CBS, TBBS, DIBS and DCBS) not lower than 80 % in purity.

7.2.2 Principle of test method The sample is dissolved in acetonitrile and this sample solution is analyzed by high performance liquid chromatography (HPLC). The peak area of analyte is calculated from the obtained chromatogram by chromatograph data processing apparatus and the purity is obtained by external standard calibration method.

7.2.3 Instruments and apparatus The instruments and apparatus shall be as follows.

7.2.3.1 High performance liquid chromatography, consisting of the following.

- a) **Pump**, having an excellent accuracy of definite flow rate and being capable of regulating a flow rate.
- b) **Sample injector**, capable of injecting 10 mm³ or smaller quantity of sample solution.
- c) **Column container**, capable of containing the columns of necessary length and having a temperature control mechanism to maintain at (35 ± 1) °C.
- d) **Column**, consisting of C18 (ODS) reversed-phase material with spherical, totally porous monomolecular 5 µm particles capable of providing 40 000 theoretical plates per metre (a minimum of 10 000 plates is required for this analysis).
- e) **Detector**, of variable wavelength type UV-detector.
- f) **Data processing apparatus and recorder** Data processing apparatus, capable of representing chromatogram, retention time, peak area value, quantitative value, etc., shall be used.

7.2.3.2 Analytical balance, capable of weighing to the nearest 0.1 mg.

7.2.4 Reagents The reagents shall be as follows.

- a) **Glacial acetic acid**, reagent.
- b) **Acetonitrile**, HPLC grade.

WARNING : Acetonitrile is a toxic and inflammable solvent.

- c) **Methanol**, HPLC grade.
- d) **Pure water**, HPLC grade.

7.2.5 Reference material The reference material is used to determine the correction factor of analyte.

7.2.6 Procedure of test method

7.2.6.1 Chromatographic conditions

- a) **Mobile phase and flow rate** The chromatographic conditions for the mobile phase composition and the flow rate are determined according to the column chosen. For the mobile phase, glacial acetic acid is added to the mixed solution of HPLC grade acetonitrile and HPLC grade pure water so as to become 0.001 mol/dm³ of concentration. The composition and flow rate of mobile phase are different depending on the sample.

HPLC grade methanol and HPLC grade pure water are used for the mobile phase to separate MBTS impurity from DIBS analyte well.

An example of the mobile phase composition and the flow rate of sample shall be shown in Table 4.

- b) **Capacity factor and resolution** For the analysis of sulfenamide organic accelerators, the mobile phase composition and the flow rate shall be adjusted so that the capacity factor (k') of analyte becomes 4 to 6 and the resolution (R_s) of analyte and MBTS impurity become at least 2.

In the case of using different column, the mobile phase to fit it shall be used.

Table 4 Mobile phase composition and flow rate of sample (example)

Class of sulfenamide	Mobile phase ⁽²⁾			Flow rate cm ³ /min
	Pure water (%)	Acetonitrile (%)	Methanol (%)	
DCBS	5	95	0	2.5
CBS	20	80	0	2.0
DIBS	15	0	85	1.0
TBBS	30	70	0	1.7

Note ⁽²⁾ That contains 0.001 mol/dm³ of glacial acetic acid.

- c) **Calculation of capacity factor k'** The capacity factor, k' , shall be obtained according to the following formula.

$$k' = \frac{t_A - t_S}{t_S}$$

where, k' : capacity factor

t_A : retention time of analyte

t_S : retention time of solvent

- d) **Calculation of resolution R_s** The resolution, R_s , shall be obtained according to the following formula.

$$R_s = 2 \times \frac{t_2 - t_1}{b_2 + b_1}$$

where, R_s : resolution

t_1, t_2 : retention times of analyte and impurity

b_1, b_2 : width at 10 % of each peak height of analyte and impurity

7.2.6.2 Detecting wave length The detecting wave length of detector shall be 275 nm.

7.2.6.3 Sensitivity of data processing apparatus The full-scale sensitivity shall be adjusted to 1 AU (absorbance unit). However, when the confirmation of peak of impurity is required, the sensitivity shall be raised.

7.2.7 Preparation of reference solution

- a) **Preparation of reference material** The reference material to be used shall be purified by recrystallization of the sulfenamide organic accelerators according to the following operations.

- 1) Add 100 g of sulfenamide organic accelerator to 200 cm³ of analytical reagent grade toluene to dissolve at 50 °C or lower in temperature. Add 2 g of activated carbon to it and stir for 30 min.
- 2) Filter this solution by gravity as it is warm, cool it in an ice-acetone bath and vacuum filter the precipitated crystals.

Repeat this recrystallization.

- 3) Dissolve the crystals from toluene recrystallization in methanol at 50 °C or lower in temperature, cool it in an ice-acetone bath and vacuum filter the precipitated crystals.
- 4) Dry the recrystallized matter in a vacuum oven maintained at 50 °C.
- 5) Estimate the purity of reference material by HPLC analysis and thermal analysis, and repeat the recrystallization operations mentioned above until disturbance material has disappeared.

Remarks: Store the reference material at 5 °C or lower, and evaluate its purity by carrying out the HPLC analysis every 90 days.

- b) **Preparation of reference solution** Weigh out approximately 20 mg of the reference material of sulfenamide organic accelerator to the nearest 0.1 mg into a 100 cm³ volumetric flask and dissolve it with acetonitrile. Dilute it with acetonitrile to give a maximum absorbance (peak height) between 0.4 AU and 0.8 AU, and adjust the concentration of reference solution (at this time, the working curve shall be linear). Analyze it within 4 h after the preparation of reference solution.

7.2.8 Sample analysis The procedure of sample analysis shall be as follows.

- a) Weigh out approximately 20 mg of well-blended sample to the nearest 0.1 mg into a 100 cm³ volumetric flask. Dissolve it in acetonitrile (an ultrasonic bath is recommended), add acetonitrile to the marked line.
Adjust the concentration of this sample solution by serial dilution to give a maximum absorbance with 10 % of the reference solution, and analyze the solution within 4 h.
- b) When solid content is contained in the sample solution, filter the solution with a chemically resistant filter paper with a normal pore size less than or equal to 0.5 µm, and analyze the solution within 4 h.
- c) Inject the reference solution and the sample solution into a high performance liquid chromatograph, and obtain a chromatogram and the peak area value.

7.2.9 Calculation

7.2.9.1 Correction factor The correction factor (R_f), shall be calculated according to the following formula.

$$R_f = \frac{C_0}{A_0} \times P_0$$

where, R_f : response factor

A_0 : peak area value of analyte

C_0 : concentration of reference solution (mg/cm³)

P_0 : purity of reference solution (%)

7.2.9.2 Purity of sample The purity (%) shall be calculated according to the following formula.

$$P_2 = R_f \times \frac{A_1}{C_1}$$

where, P_2 : purity (%)

A_1 : peak area value of analyte

C_1 : concentration of sample solution (mg/cm³)

R_f : correction factor

7.3 Expression of test results The test results shall be rounded off according to **JIS Z 8401** and expressed to the first decimal place.

7.4 Test report The test report shall include the following information.

- a) Name and number of this Standard
- b) Record of sample
- c) Test condition
- d) Test results

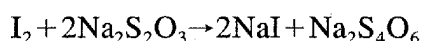
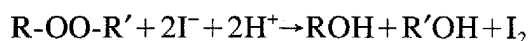
8 Method to determine the purity of peroxyketal organic peroxide

8.1 Purpose This test method specifies the procedure for the purity of the following group of peroxyketal organic peroxides used for rubber compounding ingredients.

Example of applicable peroxyketal peroxide

- 1,1 bis (tert-butyl peroxy) cyclohexane
- 1,1 bis (tert-butyl peroxy)-2-methylcyclohexane
- 1,1 bis (tert-butyl peroxy)-3,3,5-trimethylcyclohexane
- 2,2 bis (tert-butyl peroxy) butane
- n*-butyl-4,4-di (tert-butyl peroxy) valerate

8.2 Principle of test method The amount of activated oxygen is obtained by titrating the iodine freed by the oxidation-reduction reaction of organic oxide with sodium thiosulfate according to a series of following reaction.



On the other hand, the amount of activated oxygen of tert-butylhydroperoxide of impure matter is obtained by utilizing oxidization reaction of Ce^{4+} , and the purity is obtained by dividing the value subtracted of that by the theoretical amount of activated oxygen.

8.3 Determination of activated oxygen amount

8.3.1 Instruments and apparatus The instruments and apparatus shall be as follows.

- a) **One mouth, interchangeable ground joint (T29/32) flat bottom pear-shaped flask**, 300 cm³ in capacity.
- b) **Hot plate**
- c) **Reflux condenser**, of top open, with interchangeable ground joint (T29/32) of male mouth.
- d) **Burette**, 25 cm³ in capacity and 0.1 cm³ in minimum graduation.
- e) **Volumetric cylinder**, 50 cm³ and 10 cm³ in capacity.
- f) **Komagome pipette**, two pieces, 2 cm³ in capacity.
- g) **Chemical balance**, capable of weighing to the nearest 0.1 mg.

8.3.2 Reagents The reagents shall be as follows.

- a) **2-Propanol**, specified in **JIS K 8839**.
- b) **Acetic acid**, specified in **JIS K 8355**.
- c) **Sodium hydrogen carbonate**, specified in **JIS K 8622**.
- d) **Saturated solution of potassium iodide** Add approximately 250 cm³ of water to approximately 500 g of potassium iodide specified in **JIS K 8913**, warm it at approximately 40 °C, dissolve it, then cool to room temperature and preserve it in dark place.
- e) **Conc. hydrochloric acid**, specified in **JIS K 8180**.
- f) **0.1 mol/dm³ sodium thiosulfate solution**, specified in **JIS K 8001 4.5 (21.2)**.

8.3.3 Procedure of test method The procedure of test method shall be as follows.

- a) Weigh out approximately 0.5 mmol of sample into a 300 cm³ flat bottom pear-shaped flask, and weigh it to the nearest 0.1 mg.
- b) Add 30 cm³ of 2-propanol.
- c) Add 2 cm³ of acetic acid using a Komagome pipette.
- d) Add 1.5 g of sodium hydrogen carbonate.
- e) Add 2 cm³ of potassium iodide saturated solution using a Komagome pipette.
- f) Fix a reflux condenser to flat bottom pear-shaped flask and boil it on a hot plate gently for 5 min.
- g) Add approximately 8 cm³ of conc. hydrochloric acid from the upper part of reflux condenser, and boil it for 3 min.
- h) Detach the flat bottom pear-shaped flask.
- i) Add approximately 1.5 g of sodium hydrogen carbonate and approximately 50 cm³ of water, immediately titrate with 0.1 mol/dm³ sodium thiosulfate solution until the colour of iodine has disappeared.
- j) Repeat the operation mentioned above twice.