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Rubber compounding ingredients —  
Organic chemicals — Part 5:  
Test methods of organic peroxides

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## Foreword

This Japanese Industrial Standard has been established by the Minister of Economy, Trade and Industry through deliberations at the Japanese Industrial Standards Committee according to the proposal for establishment of Japanese Industrial Standard submitted by The Japan Rubber Manufacturers Association (JRMA)/Japanese Standards Association (JSA) with a draft being attached, based on the provision of Article 12, paragraph (1) of the Industrial Standardization Act.

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**JIS K 6220** series consists of the following 5 parts under the general title *Rubber compounding ingredients — Organic chemicals —*:

*Part 1 : General*

*Part 2 : Sulfenamide accelerators*

*Part 3 : p-Phenylenediamine antidegradants (PPDs)*

*Part 4 : Abbreviated terms*

*Part 5 : Test methods of organic peroxides*

# Rubber compounding ingredients — Organic chemicals — Part 5 : Test methods of organic peroxides

## Introduction

This Japanese Industrial Standard has been prepared based on **ISO 14932** : 2012, Edition 1, with some modifications and additions of the technical contents to meet the local needs and conditions in Japan.

The vertical lines on both sides and dotted underlines indicate changes from the corresponding International Standard. A list of modifications with the explanations is given in Annex JA.

## 1 Scope

This Standard specifies methods for the determination of the content of the organic peroxides used as rubber vulcanizing agents.

**NOTE 1** Organic peroxides are generally diluted with inert solvents or inorganic fillers for explosion protection and are also treated as master batches or the like. Some of them exist as an undiluted single chemical, in which case the term “content” has the same meaning as assay.

**NOTE 2** The International Standard corresponding to this Standard and the symbol of degree of correspondence are as follows.

ISO 14932 : 2012 *Rubber compounding ingredients — Organic vulcanizing agents — Determination of organic peroxide content* (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**.

**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.

## 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 0050 *General rules for chemical analysis*

JIS K 0123 *General rules for gas chromatography / mass spectrometry*

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

JIS K 0510 *High purity dodecane*

JIS K 1106 *Liquid carbon dioxide*

JIS K 1107 *Nitrogen*

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

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

JIS K 8001 *General rules for test methods of reagents*

NOTE Corresponding International Standard : ISO 6353-1 *Reagents for chemical analysis — Part 1 : General test methods*

JIS K 8180 *Hydrochloric acid (Reagent)*

JIS K 8355 *Acetic acid (Reagent)*

JIS K 8622 *Sodium hydrogen carbonate (Reagent)*

JIS K 8680 *Toluene (Reagent)*

JIS K 8839 *2-Propanol (Reagent)*

JIS K 8891 *Methanol (Reagent)*

JIS K 8913 *Potassium iodide (Reagent)*

JIS K 8951 *Sulfuric acid (Reagent)*

JIS P 3801 *Filter paper (for chemical analysis)*

JIS Z 8401 *Rounding of numbers*

### 3 Terms and definitions

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

#### 3.1

##### **number of -O-O- bond**

number of -O-O- bonds in a molecule of an organic peroxide

#### 3.2

##### **active oxygen of an organic peroxide**

oxygen-centred radicals, liberated by an organic peroxide, capable of initiating vulcanization of rubber compounds

#### 3.3

##### **total amount of active oxygen**

sum of the amount of active oxygen of an organic peroxide and the amount of active oxygen of *tert*-butyl hydroperoxide (TBHP) as an impurity

### 3.4

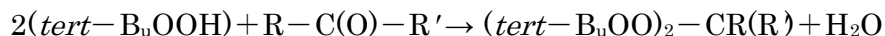
#### theoretical amount of active oxygen

calculated amount of active oxygen when the content of an organic peroxide is 100 %

### 3.5

#### peroxyketal

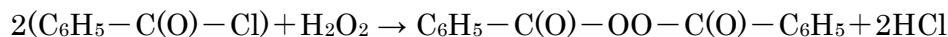
peroxide obtained by the reaction of a ketone with TBHP as shown in the following equation :



### 3.6

#### diacyl peroxide

peroxide obtained by the reaction of benzoyl chloride with hydrogen peroxide as shown in the following equation:

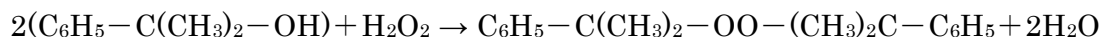


### 3.7

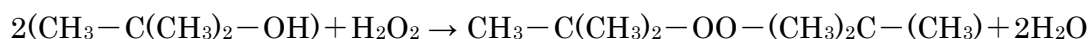
#### dialkyl peroxide

peroxide obtained by the reaction in presence of sulfuric acid as shown in the following equations :

- a) peroxide obtained by the reaction of a benzyl alcohol with hydrogen peroxide in presence of sulfuric acid as shown in the following equation :



- b) peroxide obtained by the reaction of a *tert*-butyl alcohol with hydrogen peroxide in presence of sulfuric acid as shown in the following equation :



## 4 Abbreviated terms and chemical names related to organic peroxides

For the purpose of this Standard, the abbreviated terms and chemical names related to organic peroxides given in JIS K 6220-4 apply.

## 5 General

General requirements of chemical analysis are provided in JIS K 0050.

## 6 Sampling

Sampling procedure is detailed in JIS K 5600-1-2.

## 7 Groups and test methods of organic peroxides

### 7.1 Pretreatment of test samples

Test samples of organic peroxides shall be the organic peroxides themselves or those

diluted with solvents. In the case where the organic peroxides are in a powder form diluted with inorganic fillers, they shall be used as they are. If the organic peroxides are in a granular form diluted with inorganic fillers, they shall be ground in a mortar into fine powder using a muddler. Furthermore, if the organic peroxides are in a solid form diluted with inorganic fillers and EPDM or other rubber ingredients, they shall be pretreated in accordance with Annex E and the content shall be determined.

When the pretreatment is carried out, a specified amount of the test sample shall be obtained from the solution after the pretreatment (extraction). Each test result shall be corrected using the factor obtained from Equation (E.1).

Although some of the single body organic peroxides are in a solid form, they need not be ground in advance, and shall be dissolved in a solvent specified by the test procedure (for example, 2-propanol, toluene, etc.)

## 7.2 Groups and test methods

The test methods for respective groups of organic peroxides are shown in Table 1.

For Method D, either Method D-1 or Method D-2 may be used.

**Table 1 Groups of organic peroxides and applicable test methods**

| Group                                                  | Abbreviated terms and chemical names                                                                                                                                              | Test method             | Test method                  | Applicable clause     |
|--------------------------------------------------------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-------------------------|------------------------------|-----------------------|
| • Peroxyketal                                          | • DTBPC : 1,1-bis( <i>tert</i> -butylperoxy)cyclohexane<br>• DBPB : 2,2-di( <i>tert</i> -butylperoxy)butane<br>• BPV : <i>n</i> -butyl bis(4,4- <i>tert</i> -butylperoxy)valerate | Titration               | Method A                     | Clause 8 and Annex A  |
| • Diacyl peroxide                                      | • BPO : benzoyl peroxide<br>• pMBPO : di(4-methylbenzoyl) peroxide                                                                                                                | Titration               | Method B                     | Clause 9 and Annex B  |
| • Dialkyl peroxide                                     | • di( <i>tert</i> -butylperoxyisopropyl)benzene<br>• DCP : dicumyl peroxide<br>• TBCP : <i>tert</i> -butyl cumyl peroxide                                                         | Titration               | Method C                     | Clause 10 and Annex C |
|                                                        | • DMBHa : 2,5-dimethyl-2,5-di ( <i>tert</i> -butylperoxy) hexane                                                                                                                  | Method GC <sup>a)</sup> | Method D (Method D-1 or D-2) | Clause 11 and Annex D |
| Note <sup>a)</sup> Abbreviation of gas chromatography. |                                                                                                                                                                                   |                         |                              |                       |

## 8 Determination of peroxyketal content (Method A)

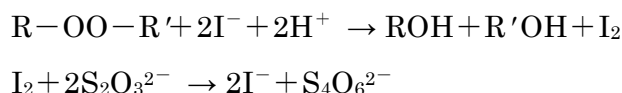
### 8.1 General

Method A is the procedure for determining the content of peroxyketals (DTBPC, DBPB, BPV, etc.) used as rubber organic vulcanizing agents. The titration technique used in this method determines the total amount of peroxyketal including impurities (TBHP)(total amount method), and requires subtraction of TBHP obtained by the impurity content measuring method (HPO method) to determine the content of the peroxyketal.

As TBHP exists only in trace amount, a simplified method regarding the TBHP content as zero may be used [hereafter referred to as the simplified method, see 8.4 and Equation (5)].

## 8.2 Principle

Peroxyketals react with iodide in an acetic acid-hydrochloric acid medium, liberating an equivalent amount of iodine which is titrated with a standard sodium thiosulfate solution :



Peroxyketals may contain traces of *tert*-butyl-hydro-peroxide (TBHP) as an impurity. If this is the case, the total amount of active oxygen of a peroxyketal is first obtained by the method (A.4) given in Annex A using iodometry.

Then the amount of active oxygen of TBHP is obtained by the TBHP content determination method (HPO method) (A.5) given in Annex A. The amount of active oxygen of the peroxyketal alone can then be obtained by subtraction, and the content of the peroxyketal is obtained by dividing the value by the theoretical amount of active oxygen of the peroxyketal.

## 8.3 Total amount determination of active oxygen

### 8.3.1 Procedure

The procedure for total amount determination of active oxygen shall be as follows.

- A weighed peroxyketal test sample ( $m_1$ ) is dissolved in an aqueous solution acidified with acetic acid and hydrochloric acid containing potassium iodide. Titrate the freed iodine with sodium thiosulfate of standard concentration and determine the volume required to complete the titration ( $V_1$ ).
- Repeat the same procedure without the peroxyketal as a blank test and determine the volume of sodium thiosulfate required to complete the titration ( $V_{b1}$ ).

### 8.3.2 Total amount of active oxygen

Calculate the total amount of active oxygen of a peroxyketal by the following equation :

$$A_{\text{O,kt}} = \frac{0.000\,80 \times (V_1 - V_{b1}) \times f_1}{m_1} \times 100 \quad \dots\dots\dots (1)$$

where,  $A_{\text{O,kt}}$  : total amount of active oxygen of the peroxyketal (%)  
 $V_1$  : volume of sodium thiosulfate solution used for the test ( $\text{cm}^3$ )  
 $V_{b1}$  : volume of sodium thiosulfate solution used for the blank test ( $\text{cm}^3$ )  
 $f_1$  : factor of 0.1 mol/L sodium thiosulfate solution  
 $m_1$  : mass of the test sample (g)  
0.000 80 : factor ( $\text{g}/\text{cm}^3$ ) obtained as follows :

$$0.000\ 80 = \frac{15.999\ 4}{2} \times 0.1 \times \frac{1}{1\ 000} \dots\dots\dots (2)$$

where, 15.999 4: atomic weight of oxygen  
0.1 : normality of the sodium thiosulfate solution (mol/L)

### 8.3.3 Calculation of theoretical amount of active oxygen

Calculate the theoretical amount of active oxygen,  $A_{T,kt}$ , expressed as a percentage, by the following equation.

The number of -O-O- bond, molecular mass and theoretical amount of active oxygen of peroxyketals are shown in Table 2.

$$A_{T,kt} = \frac{n_1 \times 15.999\ 4}{M_1} \times 100 \dots\dots\dots (3)$$

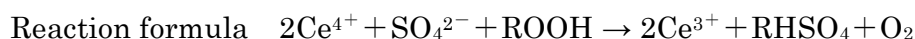
where,  $n_1$ : number of -O-O- bond of the peroxyketal (see Table 2)  
 $M_1$ : molecular mass of the peroxyketal (see Table 2)

**Table 2 Number of -O-O- bond, molecular mass and theoretical amount of active oxygen of peroxyketals**

| Peroxyketal | $n_1$ | $M_1$  | $A_{T,kt}$ |
|-------------|-------|--------|------------|
| DTBPC       | 2     | 260.38 | 12.29      |
| DBPB        | 2     | 234.34 | 13.65      |
| BPV         | 2     | 334.46 | 9.57       |

### 8.4 Impurity (TBHP) content determination (HPO method)

HPO method determines the amount of impurities (TBHP) in a peroxyketal by utilizing oxidation by  $Ce^{4+}$  under acidic condition and from the amount of active oxygen in TBHP [ $C_{HPO}$  (%) ] as shown in the following reaction :



See Annex A (HPO method) for the detailed procedure.

Alternatively, a simplified method where the TBHP content is regarded as zero and the total amount of organic peroxide is taken as the amount of peroxyketal [see Equation (5)] may be used

### 8.5 Calculation of peroxyketal content

#### 8.5.1 For untreated test samples

If the test sample was not pretreated, calculate the content of the peroxyketal,  $P_{kt}$ , expressed as a percentage, using the following equation :

$$P_{kt} = \frac{A_{O,kt} - C_{HPO} \times 0.177\ 5}{A_{T,kt}} \times 100 \dots\dots\dots (4)$$

where,  $A_{O,kt}$ : total amount of active oxygen (%)  
 $C_{HPO}$ : TBHP content (see Annex A) (%)

0.177 5 : theoretical amount of active oxygen of TBHP, divided by 100

$A_{T,kt}$ : theoretical amount of active oxygen of the peroxyketal obtained by Equation (3) (%)

If adopting the simplified method, use Equation (5) instead to calculate the content of the peroxyketal,  $P_{kt}$  (%).

$$P_{kt} = \frac{A_{O,kt}}{A_{T,kt}} \times 100 \quad \dots\dots\dots (5)$$

### 8.5.2 For pretreated test samples

If the test sample was pretreated, calculate  $P_{kt}$  according to Equation (4) or (5) and obtain the corrected peroxyketal content,  $P_{kt1}$ , expressed as a percentage, to the nearest 0.1 % according to Equation (6) which is Equation (E.1) with  $C_p$  replaced with  $P_{kt}$ .

$$P_{kt1} = \frac{m_t}{m_{s1}} \times \frac{P_{kt}}{100 - P_{kt}} \times 100 \quad \dots\dots\dots (6)$$

where,  $m_t$  : mass of toluene (g)  
 $m_{s1}$  : mass of the mixed test sample before extraction (g)  
 $P_{kt}$  : peroxyketal content before correction (%)

## 8.6 Expression of results

The test results shall be rounded to one decimal place according to Rule A of JIS Z 8401.

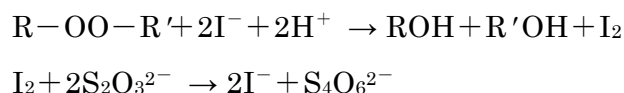
## 9 Determination of diacyl peroxide content (Method B)

### 9.1 General

Method B is the procedure for determining the content of diacyl peroxides such as BPO used as rubber organic vulcanizing agents.

### 9.2 Principle

Diacyl peroxides react with iodide in a solvent medium, liberating an equivalent amount of iodine which is titrated with a standard sodium thiosulfate solution as shown in the following equation (see Annex B). See Annex B for the detailed procedure.



The content of the diacyl peroxide is obtained by dividing the amount of active oxygen measured by the theoretical amount of active oxygen.

### 9.3 Determination of amount of active oxygen

#### 9.3.1 Procedure

A weighed diacyl peroxide test sample ( $m_2$ ) is dissolved in dilute acetic acid containing potassium iodide. Titrate the freed iodine with sodium thiosulfate of standard concentration and determine the volume required to complete the titration ( $V_2$ ). Repeat the same procedure without the diacyl peroxide as a blank test and determine the volume

of sodium thiosulfate required to complete the titration ( $V_{b2}$ ).

### 9.3.2 Calculation of amount of active oxygen

Calculate the amount of active oxygen of a diacyl peroxide,  $A_{O,da}$ , expressed as a percentage, by the following equation :

$$A_{O,da} = \frac{0.000\ 80 \times (V_2 - V_{b2}) \times f_1}{m_2} \times 100 \quad \dots\dots\dots (7)$$

where,  $V_2$  : volume of sodium thiosulfate solution used for the test ( $\text{cm}^3$ )  
 $V_{b2}$  : volume of sodium thiosulfate solution used for the blank test ( $\text{cm}^3$ )  
 $f_1$  : factor of 0.1 mol/L sodium thiosulfate solution  
 $m_2$  : mass of the test sample (g)  
0.000 80 : factor obtained by Equation (2) ( $\text{g/cm}^3$ )

### 9.3.3 Calculation of theoretical amount of active oxygen

Calculate the theoretical amount of active oxygen of a diacyl peroxide,  $A_{T,da}$ , expressed as a percentage, by the following equation.

The number of -O-O- bond, molecular mass and theoretical amount of active oxygen of diacyl peroxides are shown in Table 3.

$$A_{T,da} = \frac{n_2 \times 15.999\ 4}{M_2} \times 100 \quad \dots\dots\dots (8)$$

where,  $n_2$  : number of -O-O- bond of the diacyl peroxide (see Table 3)  
 $M_2$  : molecular mass of the diacyl peroxide (see Table 3)  
15.999 4 : atomic weight of oxygen

**Table 3 Number of -O-O- bond, molecular mass and theoretical amount of active oxygen of diacyl peroxides**

| Diacyl peroxide | $n_2$ | $M_2$  | $A_{T,da}$ |
|-----------------|-------|--------|------------|
| BPO             | 1     | 242.23 | 6.61       |
| pMBPO           | 1     | 270.28 | 5.92       |

## 9.4 Calculation of diacyl peroxide content

### 9.4.1 For unpretreated test samples

Calculate the diacyl peroxide content,  $P_{da}$ , expressed as a percentage, by the following equation :

$$P_{da} = \frac{A_{O,da}}{A_{T,da}} \times 100 \quad \dots\dots\dots (9)$$

where,  $A_{O,da}$  : active oxygen (%)  
 $A_{T,da}$  : theoretical amount of active oxygen of the diacyl peroxide obtained by Equation (8) (%)

### 9.4.2 For pretreated test samples

If the test sample was pretreated, calculate  $P_{da}$  according to Equation (9) and obtain the corrected diacyl peroxide content,  $P_{da1}$ , expressed as a percentage, to the nearest 0.1 % according to Equation (10) which is Equation (E.1) with  $C_p$  replaced with  $P_{da}$ .

$$P_{da1} = \frac{m_t}{m_{s1}} \times \frac{P_{da}}{100 - P_{da}} \times 100 \quad \dots\dots\dots (10)$$

where,  $m_t$  : mass of toluene (g)  
 $m_{s1}$  : mass of the mixed test sample before extraction (g)  
 $P_{da}$  : diacyl peroxide content before correction (%)

## 9.5 Expression of results

The test results shall be rounded to one decimal place according to Rule A of JIS Z 8401.

## 10 Determination of dialkyl peroxide content (Method C)

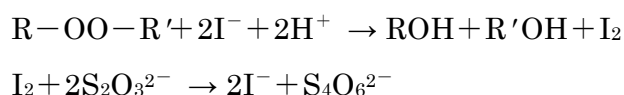
### 10.1 General

Method C is the procedure for determining the content of dialkyl peroxides such as DCP used as rubber organic vulcanizing agents by titration. See Annex C for the detailed procedure.

Where the material under test is DMBHa, the determination should be performed by gas chromatography according to the procedure given in Clause 11.

### 10.2 Principle

Dialkyl peroxide react with iodide in a solvent medium, liberating an equivalent amount of iodine which is titrated with a standard sodium thiosulfate solution as shown in the following equation.



The content of the dialkyl peroxide is obtained by dividing the amount of active oxygen measured by the theoretical amount of active oxygen, and then multiplying the result with the correction factor ( $f_p$ ) in Table 4.

### 10.3 Determination of amount of active oxygen

#### 10.3.1 Procedure

The dialkyl peroxide sample ( $m_3$ ) is refluxed in an inert atmosphere with acetic acid and a specified amount of water containing sodium iodide. Water is added to the reaction mixture to prevent side reactions taking place between iodide and decomposition products of the dialkyl peroxide. After refluxing for 30 min, the reaction mixture is cooled to room temperature to prevent side reactions between the liberated iodine and decomposition products of the dialkyl peroxide and to avoid loss of iodine through volatilization. After dilution with water, the liberated iodine is titrated with a standard sodium thiosulfate solution ( $V_3$ ).

Repeat the same procedure without the dialkyl peroxide as a blank test and determine the volume of the standard sodium thiosulfate required to complete the titration ( $V_{b3}$ ).

### 10.3.2 Calculation of amount of active oxygen

Calculate the amount of active oxygen of a dialkyl peroxide,  $A_{O,AA}$ , expressed as a percentage, by the following equation :

$$A_{O,AA} = \frac{0.000\ 80 \times (V_3 - V_{b3}) \times f_1}{m_3} \times 100 \dots\dots\dots (11)$$

where,  $V_3$  : volume of sodium thiosulfate solution used for the test ( $\text{cm}^3$ )  
 $V_{b3}$  : volume of sodium thiosulfate solution used for the blank test ( $\text{cm}^3$ )  
 $f_1$  : factor of 0.1 mol/L sodium thiosulfate solution  
 $m_3$  : mass of the test sample (g)  
0.000 80 : factor obtained by Equation (2) ( $\text{g}/\text{cm}^3$ )

### 10.3.3 Calculation of theoretical amount of active oxygen

Calculate the theoretical amount of active oxygen of a dialkyl peroxide,  $A_{T,AA}$ , expressed as a percentage, by the following equation.

The number of -O-O- bond, molecular mass, theoretical amount of active oxygen and correction factor of dialkyl peroxides are shown in Table 4.

$$A_{T,AA} = \frac{n_3 \times 15.999\ 4}{M_3} \times 100 \dots\dots\dots (12)$$

where,  $n_3$  : number of -O-O- bond of the dialkyl peroxide  
 $M_3$  : molecular mass of the dialkyl peroxide (see Table 4)  
15.999 4 : atomic weight of oxygen

**Table 4 Number of -O-O- bond, molecular mass, theoretical amount of active oxygen and correction factor of dialkyl peroxides**

| Dialkyl peroxide                                                                                               | $n_3$ | $M_3$ | $A_{T,AA}$ | $f_p$                      |
|----------------------------------------------------------------------------------------------------------------|-------|-------|------------|----------------------------|
| Di( <i>tert</i> -butylperoxyisopropyl)benzene                                                                  | 2     | 338.5 | 9.45       | $\frac{100}{87.9} = 1.138$ |
| DCP                                                                                                            | 1     | 270.4 | 5.92       | $\frac{100}{93} = 1.075$   |
| TBCP                                                                                                           | 1     | 208.3 | 7.68       | $\frac{100}{91} = 1.099$   |
| NOTE The correction factor $f_p$ is the empirically determined deviation from the theoretical value (100/100). |       |       |            |                            |

## 10.4 Calculation of dialkyl peroxide content

### 10.4.1 For unpretreated test samples

Calculate the dialkyl peroxide content,  $P_{AA}$ , expressed as a percentage, by the fol-