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INTERNATIONAL STANDARD



2522

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## Tritolyl phosphate for industrial use — Determination of apparent free phenols content — Volumetric method

*Tritolyl phosphate à usage industriel — Dosage des phénols libres apparents — Méthode volumétrique*

First edition — 1974-04-01

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UDC 661.634 : 547.533 : 547.56 : 543.241

Ref. No. ISO 2522-1974 (E)

**Descriptors :** tritolyl phosphate, chemical analysis, determination of content, phenols, volumetric analysis.

## FOREWORD

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Draft International Standards adopted by the Technical Committees are circulated to the Member Bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 2522 was drawn up by Technical Committee ISO/TC 47, *Chemistry*, and circulated to the Member Bodies in September 1971.

It has been approved by the Member Bodies of the following countries :

|                     |                       |                |
|---------------------|-----------------------|----------------|
| Austria             | Ireland               | Spain          |
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No Member Body expressed disapproval of the document.

# Tritolyl phosphate for industrial use – Determination of apparent free phenols content – Volumetric method

## 1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a volumetric method for the determination of the apparent free phenols content of tritolyl phosphate [(CH<sub>3</sub>C<sub>6</sub>H<sub>4</sub>)<sub>3</sub>PO<sub>4</sub>] for industrial use.

NOTE – This method determines only those phenols capable of reacting at the 2,4 and 6 positions (–OH = 1).

## 2 PRINCIPLE

Extraction of free phenols from a test portion with sodium hydroxide solution. Bromination of these phenols with potassium bromide-bromate solution in an acid medium and determination of the excess bromine, after reaction with potassium iodide, by titration with standard volumetric sodium thiosulphate solution.

## 3 REAGENTS

Distilled water, or water of equivalent purity, shall be used in the test.

### 3.1 Chloroform.

**3.2 Hydrochloric acid**,  $\rho$  approximately 1,19 g/ml, about 38 % (m/m) solution, or approximately 12 N.

**3.3 Sodium hydroxide**, 25 g/l solution.

**3.4 Potassium iodide**, 100 g/l solution.

### 3.5 Potassium bromide-bromate solution.

Dissolve 0,6 g of potassium bromate and 3,0 g of potassium bromide in water and dilute to 1 000 ml.

**3.6 Sodium thiosulphate**, 0,02 N standard volumetric solution.

**3.7 Starch paste**, 2,5 g/l solution.

## 4 APPARATUS

Ordinary laboratory apparatus and :

**4.1 Iodine flask**, 500 ml.

## 5 PROCEDURE

### 5.1 Test portion

Weigh, to the nearest 0,01 g, 10,0 g of the laboratory sample.

### 5.2 Blank test

Carry out a blank test at the same time as the analysis, substituting 50 ml of water for the 50 ml of filtered extract but otherwise proceeding exactly as described in 5.3 and 5.4.

### 5.3 Preparation of the test solution

Shake the test portion (5.1) vigorously for 3 min with 50 ml of the sodium hydroxide solution (3.3) in a 100 ml one-mark volumetric flask. Dilute to the mark with water and mix. Filter the aqueous layer through a wet filter paper (more than once if necessary) until it is clear.

#### 5.4 Determination

Transfer 50,0 ml of the filtered extract (5.3) to the flask (4.1) and add 20,0 ml of the potassium bromide-bromate solution (3.5). Acidify with 5 ml of the hydrochloric acid solution (3.2), stopper immediately, shake the mixture thoroughly, fill the cup top with water and allow to stand for 15 min.

Raise the stopper carefully, add 10 ml of the potassium iodide solution (3.4), shake and wash down the stopper and walls of the flask. If there is a white precipitate, also add 1 ml of the chloroform (3.1). Titrate with the standard volumetric sodium thiosulphate solution (3.6). Near the end point, shake the flask well and use the starch paste (3.7) as indicator. The end point corresponds to the disappearance of the blue coloration.

#### 6 EXPRESSION OF RESULTS

Apparent free phenols content, expressed as a percentage by mass of phenol, is given by the formula

$$\frac{9}{50} \times 31,3 \times 10^{-6} (V_1 - V_2) \times 100 = 0,005\ 643 \times (V_1 - V_2)$$

where

$V_1$  is the volume, in millilitres, of the standard volumetric sodium thiosulphate solution (3.6) used in the blank test;

$V_2$  is the volume, in millilitres, of the standard volumetric sodium thiosulphate solution (3.6) used for the determination;

$31,3 \times 10^{-6}$  is the mass, in grams, of phenol corresponding to 1 ml of the 0,02 N standard volumetric sodium thiosulphate solution;

$\frac{9}{50}$  is the ratio between the volume of the test solution (90 ml) and the aliquot portion taken for the titration (50 ml), divided by the mass (10 g) of the test portion.

#### 7 TEST REPORT

The test report shall include the following particulars :

- a) the reference of the method used;
- b) the results and the method of expression used;
- c) any unusual features noted during the determination;
- d) any operation not included in this International Standard, or regarded as optional.

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#### ANNEX

This document forms part of the following series on methods of test for tritolyl phosphate for industrial use :

ISO 2520 – *List of methods of test.*

ISO 2521 – *Determination of acidity to phenol red – Volumetric method.*

ISO 2522 – *Determination of apparent free phenols content – Volumetric method.*