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International Standard



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## Aromatic hydrocarbons — Determination of sulphur content — Pitt-Ruprecht reduction and spectrophotometric method

*Hydrocarbures aromatiques — Dosage du soufre — Méthode par réduction et spectrophotométrie de Pitt-Ruprecht*

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## 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 5282 was developed by Technical Committee ISO/TC 78, *Aromatic hydrocarbons*, and was circulated to the member bodies in September 1979.

It has been approved by the member bodies of the following countries :

Austria	Italy	Romania
Czechoslovakia	Korea, Rep. of	South Africa, Rep. of
France	Libyan Arab Jamahiriya	United Kingdom
Germany, F. R.	Netherlands	USSR
Hungary	Philippines	
India	Poland	

No member body expressed disapproval of the document.

# Aromatic hydrocarbons — Determination of sulphur content — Pitt-Ruprecht reduction and spectrophotometric method

## 1 Scope and field of application

This International Standard specifies a method for the determination of the sulphur content of aromatic hydrocarbons boiling below 200 °C.

The method is directly applicable to sulphur contents of 0,1 to 30 mg/kg; higher contents may be determined by suitable dilution of the sample.

Alkylsulphones, arylsulphonic acids and arylsulphonates all give incomplete recovery of sulphur.

## 2 Reference

ISO 1995, *Aromatic hydrocarbons — Sampling*.

## 3 Principle

Reduction of the sulphur compounds to nickel sulphide with Raney nickel.

Stripping of the hydrogen sulphide in hydrochloric acid medium and absorption in an alkaline zinc acetate solution. Formation of methylene blue with *N,N*-dimethyl-*p*-phenylenediamine sulphate and iron(III) ammonium sulphate.

Determination of the absorbance of the blue coloured solution at a wavelength of 667 nm.

## 4 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

### 4.1 Glycol-potassium hydroxide solution.

Dissolve 4 g of potassium hydroxide in 100 ml of ethylene glycol.

### 4.2 Sodium hydroxide solution, $c(\text{NaOH}) = 2,5 \text{ mol/l}$ .

### 4.3 Hydrochloric acid solution, $c(\text{HCl}) = 5 \text{ mol/l}$ .

### 4.4 Zinc acetate [zinc di(acetate)], 10 g/l solution.

**4.5 Propan-2-ol**, of such quality that the use of different quantities for the blank test (for example 25 and 50 ml) should give no difference in absorbance. Otherwise, the blank test shall be carried out using sulphur-free toluene, instructions for the preparation of which are given in the annex.

### 4.6 Raney nickel.

Raney nickel consists of a mixture of  $50 \pm 5 \%$  nickel and  $50 \pm 5 \%$  aluminium.

The reducing power of the Raney nickel shall be checked regularly, for example by means of a standard sample.

### 4.7 Sulphur, standard solution.

Weigh, to the nearest 1 mg, about 320 mg of thiophene (purity  $> 99 \%$ ), transfer quantitatively to a 500 ml one-mark volumetric flask containing 250 ml of the propan-2-ol (4.5), dilute to the mark with the propan-2-ol and mix.

Pipette 25,0 ml of this solution into a second dry 500 ml one-mark volumetric flask, dilute to the mark with the propan-2-ol and mix.

1 ml of this standard solution contains  $3,81 \times 10^{-5} m$  mg of S, where  $m$  is the actual mass, in milligrams, of thiophene taken.

### 4.8 Iron(III) ammonium sulphate [ammonium iron bis(sulphate)] solution.

Dissolve 120,6 g of iron(III) ammonium sulphate dodecahydrate  $[\text{FeNH}_4(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}]$  in 750 ml of water; while cooling and stirring, add 27 ml of sulphuric acid ( $\rho = 1,84 \text{ g/ml}$ ), dilute with water to 1 000 ml and mix.

### 4.9 *N,N*-dimethyl-*p*-phenylenediamine sulphate (4-amino-*N,N*-dimethylaniline sulphate) solution.

Dissolve 930 mg of *N,N*-dimethyl-*p*-phenylenediamine sulphate in 75 ml of water; while cooling and stirring, add 187 ml of sulphuric acid ( $\rho = 1,84 \text{ g/ml}$ ), dilute with water to 1 000 ml and mix.