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INTERNATIONAL STANDARD 1897/III

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

Phenol, *o*-cresol, *m*-cresol, *p*-cresol, cresylic acid and xylenols for industrial use — Methods of test — Part III : Determination of neutral oils and pyridine bases

Phénol, o-crésol, m-crésol, p-crésol, acide crésylique et xylénols à usage industriel — Méthodes d'essai — Partie III : Dosages des huiles neutres et des bases pyridinées

First edition — 1977-04-15

UDC 661 : 547.562/563 : 543.8 **Ref. No. ISO 1897/III-1977 (E)**

Descriptors : phenols, phenol, cresols, xylenol, chemical analysis, determination of content, water, volumetric analysis, Karl Fischer reagent.

Price based on 5 pages

FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

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.

Prior to 1972, the results of the work of the technical committees were published as ISO Recommendations; these documents are in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 47, *Chemistry*, has reviewed ISO Recommendation R 1899-1971 and found it technically suitable for transformation. Number 1899, however, has been changed to 1897/III. International Standard ISO 1897/III therefore replaces ISO Recommendation R 1899-1971, to which it is technically identical.

ISO Recommendation R 1899 had been approved by the member bodies of the following countries :

Australia	Hungary	Romania
Belgium	India	South Africa, Rep. of
Chile	Israel	Spain
Czechoslovakia	Italy	Switzerland
Egypt, Arab Rep. of	Japan	Thailand
France	Netherlands	Turkey
Germany	New Zealand	United Kingdom
Greece	Poland	U.S.S.R.

No member body had expressed disapproval of the Recommendation.

The member body of the following country disapproved the transformation of the Recommendation into an International Standard :

Netherlands

Printed in Switzerland

1897 PT III-77 4851903 0007399 6

Phenol, *o*-cresol, *m*-cresol, *p*-cresol, cresylic acid and xylenols for industrial use — Methods of test — Part III : Determination of neutral oils and pyridine bases

1 SCOPE AND FIELD OF APPLICATION

This part of ISO 1897 specifies a volumetric method, after distillation, for the determination of neutral oils, and a titrimetric method, after distillation, for the determination of pyridine bases, in phenol, *o*-cresol, *m*-cresol, *p*-cresol, cresylic acid and xylenols for industrial use.

This document should be read in conjunction with part I (see the annex).

2 PRINCIPLE

Distillation of the neutral oils and pyridine bases from an aqueous alkaline solution of a test portion. Measurement of the volume of neutral oils collected. Titration of the pyridine bases present in the aqueous distillate and in the neutral oils with standard volumetric hydrochloric acid solution using methyl orange and xylene cyanol FF as indicator.

3 REAGENTS

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

3.1 Sodium chloride, 20 g/l solution.

3.2 Sodium hydroxide, 270 g/l solution.

3.3 Hydrochloric acid, approximately 0,1 N solution.

3.4 Hydrochloric acid, 1 N standard volumetric solution.

3.5 Phenolphthalein, 5 g/l ethanolic solution.

Dissolve 0,5 g of phenolphthalein in 100 ml of 95 % (*V/V*) ethanol, and make faintly pink by the addition of dilute sodium hydroxide solution.

3.6 Methyl orange and xylene cyanol FF, aqueous ethanolic solution.

Dissolve 1 g of methyl orange and 1,4 g of xylene cyanol FF in 500 ml of 50 % (*V/V*) ethanol.

4 APPARATUS

Ordinary laboratory apparatus, and

4.1 Distillation apparatus, as shown in figure 1 and including the following components :

4.1.1 *Distillation flask* (A), of borosilicate glass, capacity 1 000 ml.

4.1.2 *Splash head* (B), as shown in figure 2.

4.1.3 *Bubbler tube* (C), as shown in figure 3.

4.1.4 *Liebig condenser* (D), of borosilicate glass, as shown in figure 4.

4.1.5 *Separating funnel receiver* (E) : one of the two types shown in figure 5, according to whether the sample is expected to contain more than or less than 1 % (*m/m*) of neutral oil.

4.1.6 *Safety screen,* placed between the apparatus and the operator.

5 PROCEDURE

5.1 Test portion

Weigh into the flask (A), to the nearest 0,1 g, 100 g of the test sample.

5.2 Preparation of apparatus

Before commencing the test, it is essential to ensure that the receiver (E) and conical flask mentioned in 5.4 are scrupulously clean. Washing with detergent, followed by thorough rinsing first in tap water and then in distilled water, is usually adequate.

5.3 Distillation

Add to flask (A), containing the test portion (5.1), 170 ml of the sodium hydroxide solution (3.2), followed by 100 ml of water, using the same measuring cylinder, without

intermediate cleaning or rinsing, for both operations. Mix thoroughly. To prevent bumping, add a small fragment of porous porcelain. Measure 25 ml of the sodium hydroxide solution (3.2) into the inner bubbler tube (C). Fill the graduated portion of the receiver (E) with the sodium chloride solution (3.1) to prevent lodgement of the neutral oil. Assemble the apparatus as shown in figure 1, with the safety screen in position, and ensure that the condenser has a steady supply of water. Make certain that the hole in the bubbler tube is not blocked. Distil the material at such a rate that 100 ml of condensate is collected within 10 to 15 min. Ensure that the condensate runs down the inside surface of the receiver and not directly on to the liquid surface. Towards the end of the distillation turn off the water and drain approximately two-thirds of the condenser (D) to melt any condensed solids. Do not allow steam to issue from the end of the condenser at any time during the distillation.

5.4 Determination

5.4.1 *Neutral oils*

If necessary, warm the distillate in the receiver (E) just enough to liquefy any solids. Run the aqueous distillate into a 250 ml conical flask, cautiously, so that the neutral oil is retained in the graduated portion of the receiver. Note the volume of oil obtained.

5.4.2 *Pyridine bases*

Transfer the neutral oil in the receiver (E) to the aqueous distillate in the conical flask. Wash the receiver with two 10 ml portions of water and transfer the washings to the conical flask. Add 3 drops of the phenolphthalein solution (3.5).

If a colour develops, add the hydrochloric acid solution (3.3) drop by drop from a burette until the colour disappears. To this solution or, if no colour has developed, to the original solution, add 2 drops of the methyl orange and xylene cyanol FF solution (3.6). Titrate with the hydrochloric acid solution (3.4), shaking the mixture thoroughly after each addition of acid, until the colour changes sharply from green to magenta.

NOTE — It is desirable to clean the apparatus after each determination by distilling 400 ml of distilled water in the assembled apparatus, but without water flowing through the condenser.

6 EXPRESSION OF RESULTS

6.1 Neutral oils content

Report the volume, in millilitres, of neutral oils obtained (see 5.4.1) from a test portion of 100 g.

6.2 Pyridine bases content

The content of pyridine bases, expressed as a percentage by mass of pyridine (C_5H_5N), is given by the formula.

$$0{,}079 \times V$$

where V is the volume, in millilitres, of the hydrochloric acid solution (3.4) used for the titration (5.4.2).

NOTE — If the concentration of the standard volumetric solution used is not exactly as specified in the list of reagents, an appropriate correction should be made.

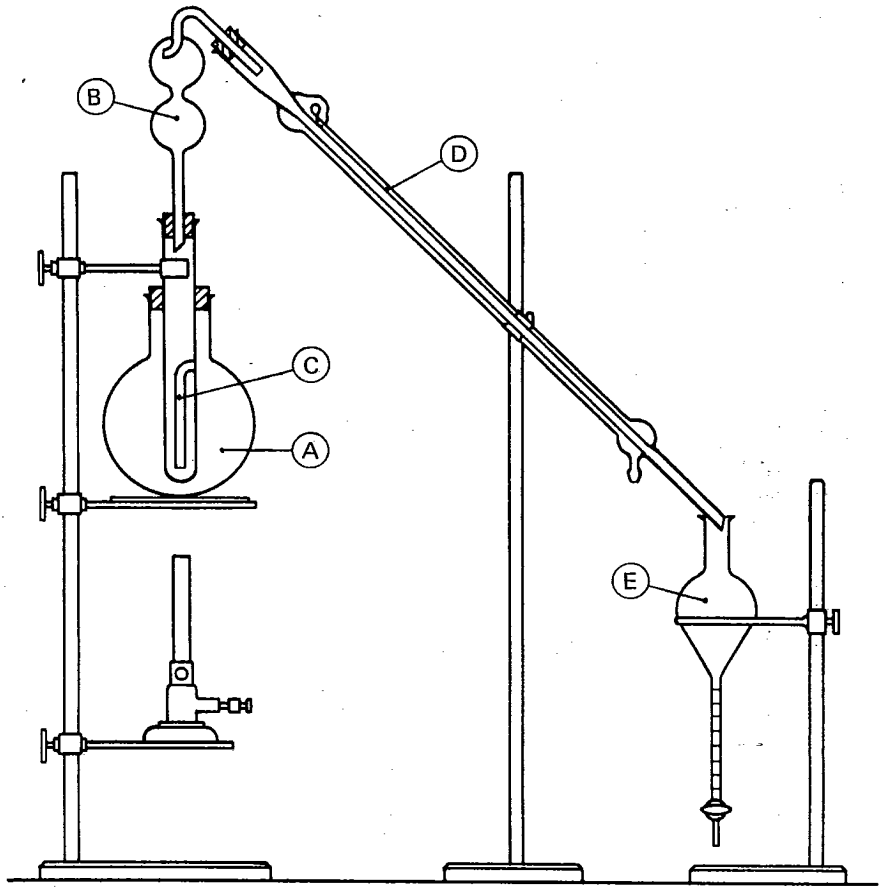


FIGURE 1 — **Assembled distillation apparatus**

Dimensions (approximate) in millimetres

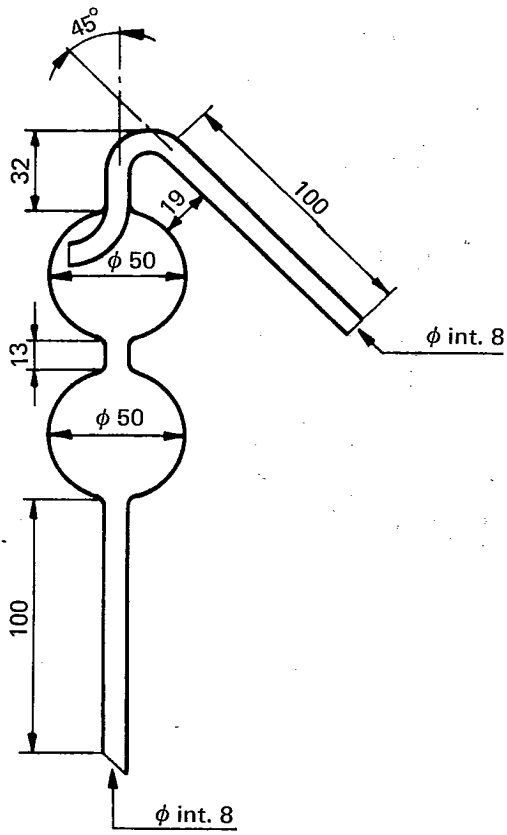


FIGURE 2 — **Splash head (B)**

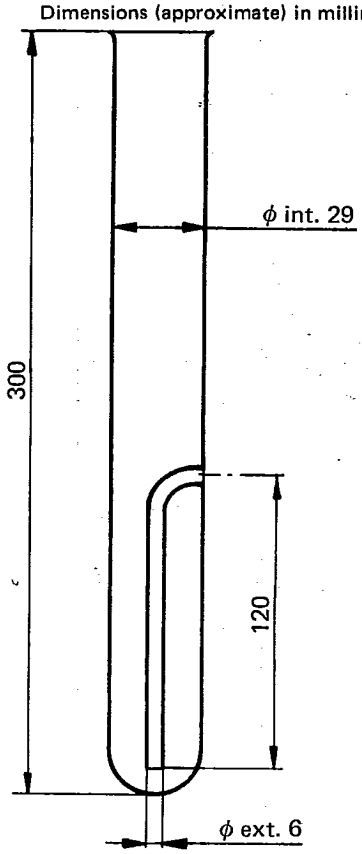


FIGURE 3 — **Bubbler tube (C)**

Dimensions in millimetres

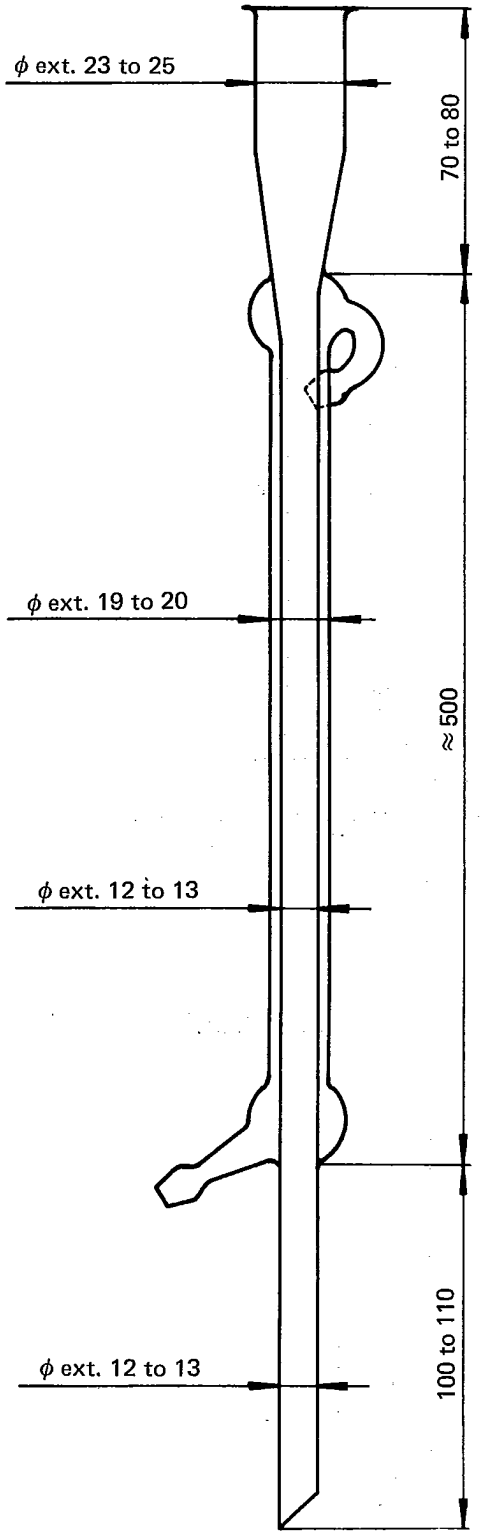
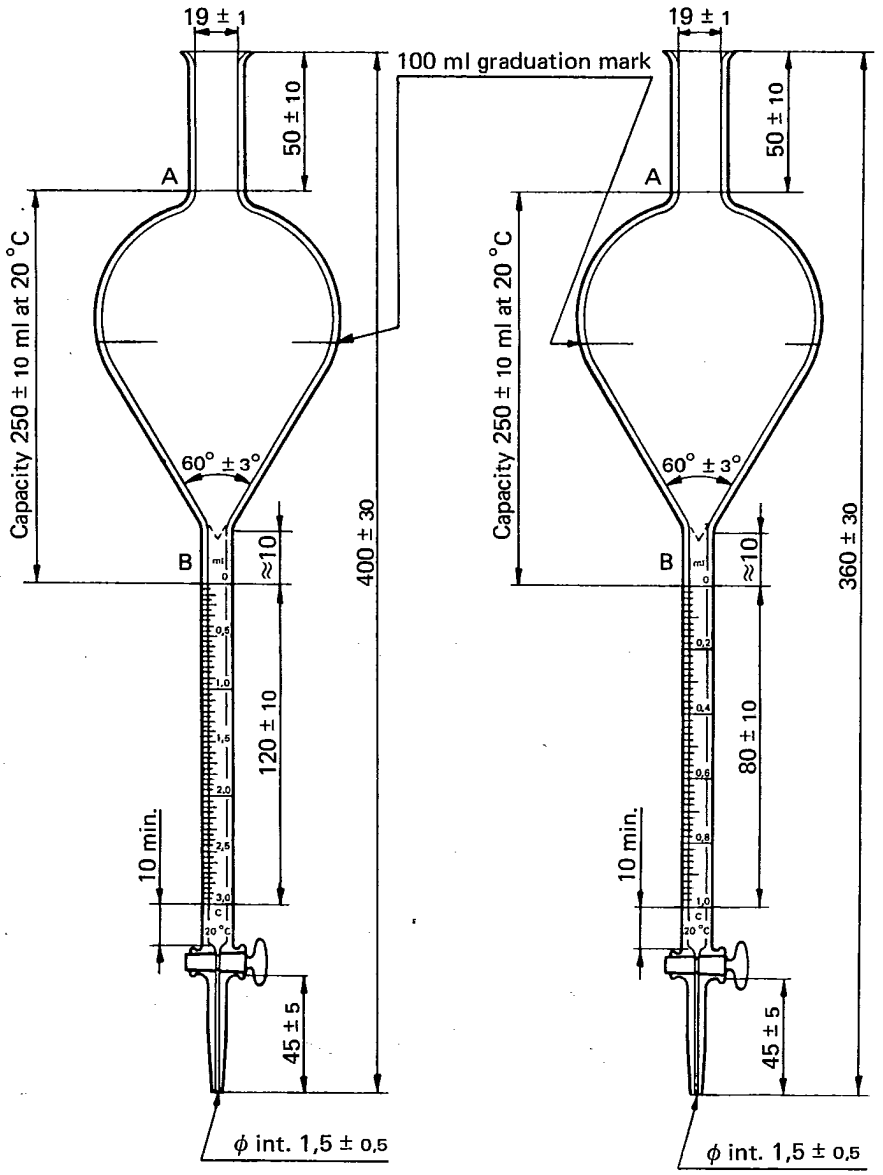


FIGURE 4 — **Liebig condenser, all borosilicate glass (D)**



a) For samples expected to contain more than 1 % (*m/m*) of neutral oil

b) For samples expected to contain less than 1 % (*m/m*) of neutral oil

FIGURE 5 — **Separating funnel receiver (E)**

ANNEX

ISO PUBLICATIONS RELATING TO (A) PHENOL, (B) *o*-CRESOL, (C) *m*-CRESOL, (D) *p*-CRESOL, (E) CRESYLIC ACID, AND (F) XYLENOLS, FOR INDUSTRIAL USE

Applicability						
A[1]	B[2]	C	D[2]	E	F	ISO 1897/I — General.
A	B	C	D	E	F	ISO 1897/II — Determination of water — Dean and Stark method.
A	B	C	D	E	F	ISO 1897/III — Determination of neutral oils and pyridine bases.
A	B	C	D			ISO 1897/IV — Visual test for impurities insoluble in sodium hydroxide solution.
A						ISO 1897/V — Visual test for impurities insoluble in water.
				E	F	ISO 1897/VI — Test for absence of hydrogen sulphide.
				E	F	ISO 1897/VII — Measurement of colour.
				E	F	ISO 1897/VIII — Determination of *o*-cresol content.
				E		ISO 1897/IX — Determination of *m*-cresol content.
A	B	C	D			ISO/R 1900 — Determination of residue on evaporation.
A	B	C	D			ISO/R 1901 — Determination of crystallizing point.
A[3]						ISO 1904 — Determination of phenols content — Bromination method.
				E	F	ISO/R 1906 — Determination of distillation range.
				E	F	ISO/R 1907 — Determination of residue on distillation.
A	B	C	D			ISO 2208 — Determination of crystallizing point after drying with a molecular sieve.

1) In the case of phenol, the determination of density at 20 °C specified in ISO 1897/I is applicable only to liquefied phenol.

2) The determination of density at 20 °C specified in ISO 1897/I is not applicable to these products.

3) Applicable only to liquefied phenol.