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ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 1898

PHENOL, *o*-CRESOL, *m*-CRESOL, *p*-CRESOL, CRESYLIC ACID AND XYLENOLS FOR INDUSTRIAL USE

DETERMINATION OF WATER BY THE DEAN AND STARK METHOD

1st EDITION

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BRIEF HISTORY

The ISO Recommendation R 1898, *Phenol, o-cresol, m-cresol, p-cresol, cresylic acid and xylenols for industrial use – Determination of water by the Dean and Stark method,* was drawn up by Technical Committee ISO/TC 47, *Chemistry,* the Secretariat of which is held by the Ente Nazionale Italiano di Unificazione (UNI).

Work on this question led to the adoption of Draft ISO Recommendation No. 1898, which was circulated to all the ISO Member Bodies for enquiry in November 1969. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

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

No Member Body opposed the approval of the Draft.

This Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 1898 October 1971

PHENOL, *o*-CRESOL, *m*-CRESOL, *p*-CRESOL, CRESYLIC ACID AND XYLENOLS FOR INDUSTRIAL USE

DETERMINATION OF WATER BY THE DEAN AND STARK METHOD

WARNING. These materials burn the skin and can be absorbed into the system through the skin. It is essential for the sampler to wear protective gloves, for example of polyvinyl chloride, and also a face shield. Inhalation of the vapours from hot material is to be avoided.

Phenols are extremely hygroscopic, and care should be taken to avoid contamination with atmospheric or other moisture.

1. SCOPE AND FIELD OF APPLICATION

This ISO Recommendation describes a procedure for the determination of water content by the Dean and Stark method and is applicable to phenol, *o*-cresol, *m*-cresol, *p*-cresol, cresylic acid and xylenols for industrial use.

NOTE. – Another procedure may be used as an alternative; it is based on the Karl Fischer method and is the subject of ISO Recommendation R 1897, *Phenol, o-cresol, m-cresol, p-cresol, cresylic acid and xylenols for industrial use – Determination of water by the Karl Fischer method.* More reproducible results are likely to be obtained by the Karl Fischer method; it is therefore preferable to use it, especially if the water content is less than 0.5 %.

2. SAMPLING

Apply the principles given in ISO Recommendation R . . . *. The following principles should also be observed :

Place the laboratory sample representative of the material taken from the bulk in a clean, dry, dark-coloured, glass-stoppered bottle of such a size that it is nearly filled by the sample. If it is necessary to seal this bottle, care should be taken to avoid contaminating the contents.

* Sampling of chemical products will form the subject of a future ISO Recommendation.

3. PRINCIPLE

Determination by means of the Dean and Stark apparatus of the volume of liquid separated, assuming it to be water.

4. REAGENTS

4.1 *Xylene*, free from water.

The difference between temperatures at which 5 % and 95 % of the volume taken have been collected should not exceed 5 °C and this range should lie between 137.0 and 145.5 °C.

Or, alternatively,

4.2 *Solvent naphtha*, free from water.

96 % of the product should distil below 160 °C.

Or, alternatively,

4.3 *Toluene*, free from water.

5. APPARATUS

Ordinary laboratory apparatus and

5.1 *Dean and Stark apparatus.*

A 500 ml glass distillation flask fitted to a Dean and Stark apparatus with a 2 ml receiver having a maximum error of ± 0.02 ml, or a 10 ml receiver having a maximum error of ± 0.06 ml, or a 25 ml receiver having a maximum error of ± 0.1 ml according to the expected water content. A typical assembly of the Dean and Stark apparatus, a suitable form of condenser and 2, 10 and 25 ml receivers are shown in Figures 1 to 5. The use of a 25 ml receiver with a stopcock is permitted as an alternative.

6. PROCEDURE

6.1 Test portion

Weigh, to the nearest 0.1 g, at laboratory temperature, 200 g of the laboratory sample.

In the case of liquefied phenol, weigh, to the nearest 0.1 g, 100 g of the laboratory sample.

6.2 Determination

Transfer the test portion (6.1) to the flask of the Dean and Stark apparatus (5.1) and add 100 ml of the solvent (4.1, 4.2 or 4.3).

Attach the flask to the apparatus (5.1). Heat the flask so that condensate falls from the end of the condenser at the rate of 2 to 5 drops per second.

Continue the distillation until condensed water is no longer visible in any part of the apparatus except the bottom of the graduated tube and until the volume of water collected remains constant. If a persistent ring of condensed water forms in the condenser tube, remove it by increasing the rate of distillation by a few drops per second.

Bring the water collected to the temperature of the laboratory and note its volume.

7. EXPRESSION OF RESULTS

The water content is given, as a percentage by mass, by the following formula :

$$\frac{V \times 100}{m}$$

where

V is the volume, in millilitres, of water collected at ambient temperature;

m is the mass, in grammes, of the test portion.

NOTE. – It is assumed that the density of the water collected in the graduated tube is 1.00 g/ml.

8. TEST REPORT

The test report should give 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 ISO Recommendation or regarded as optional.

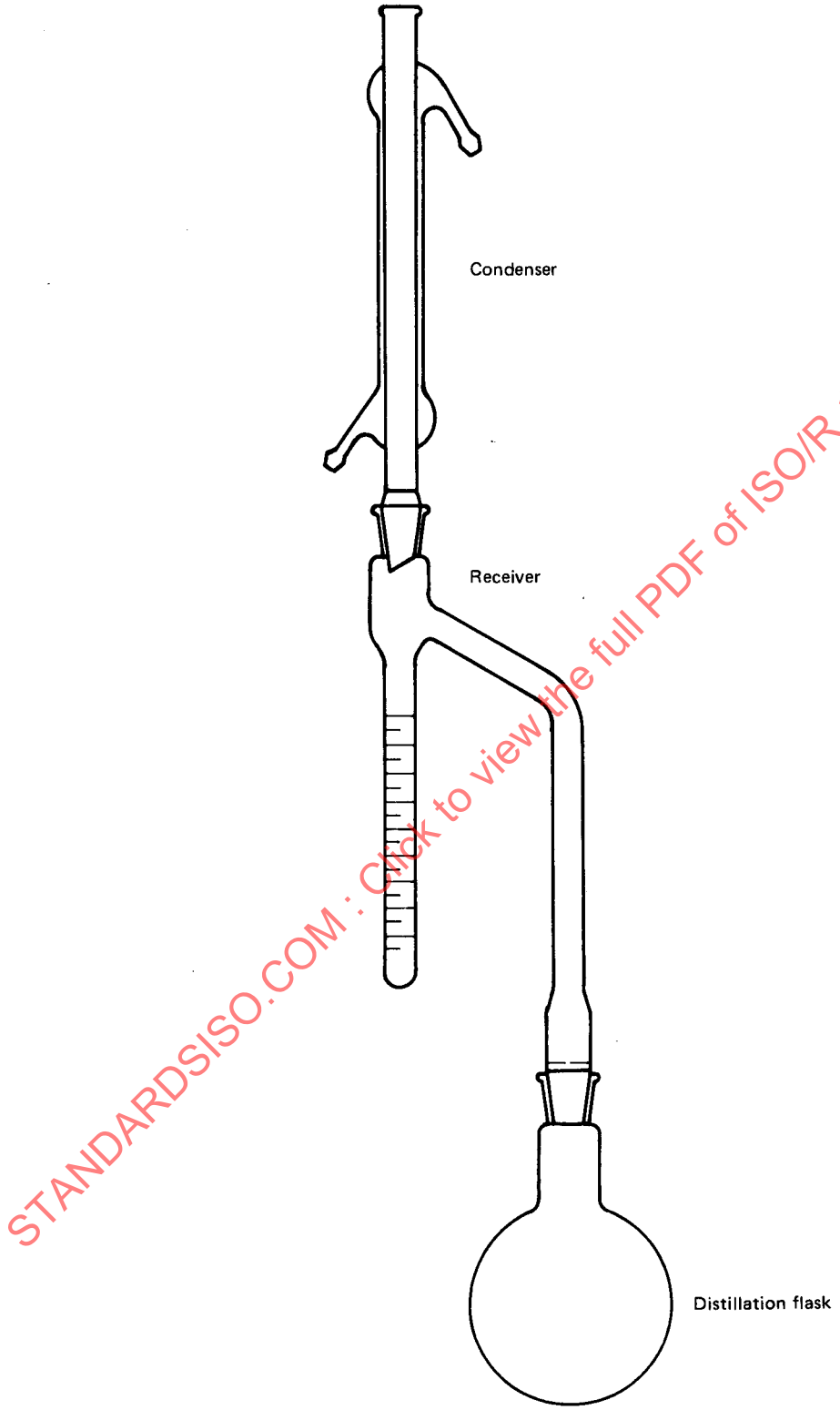

FIG. 1 – **Typical assembly of Dean and Stark apparatus (5.1)**

Dimensions in millimetres

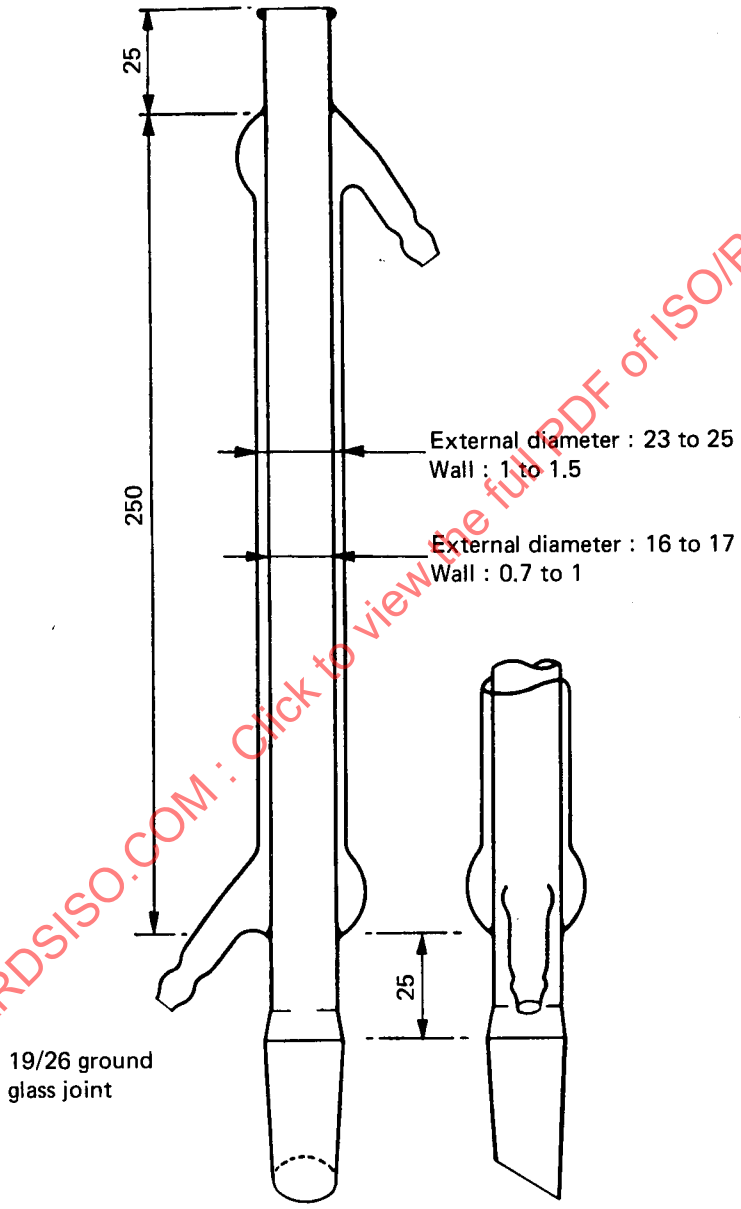

FIG. 2 – Suitable form of condenser

Dimensions in millimetres

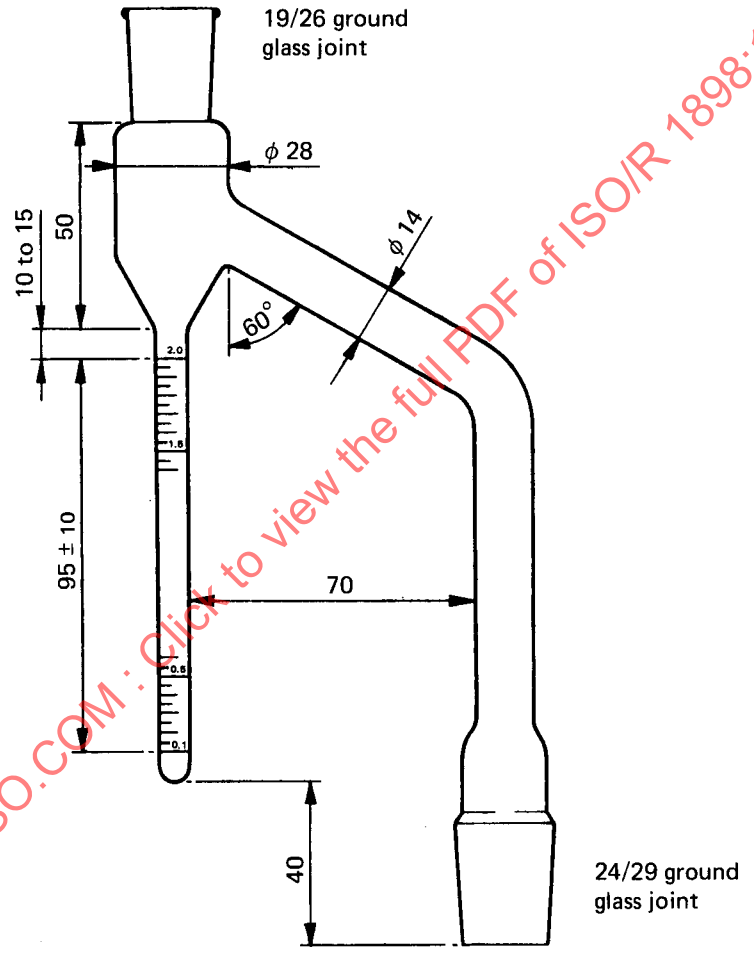

FIG. 3 – **2 ml receiver**

Dimensions in millimetres

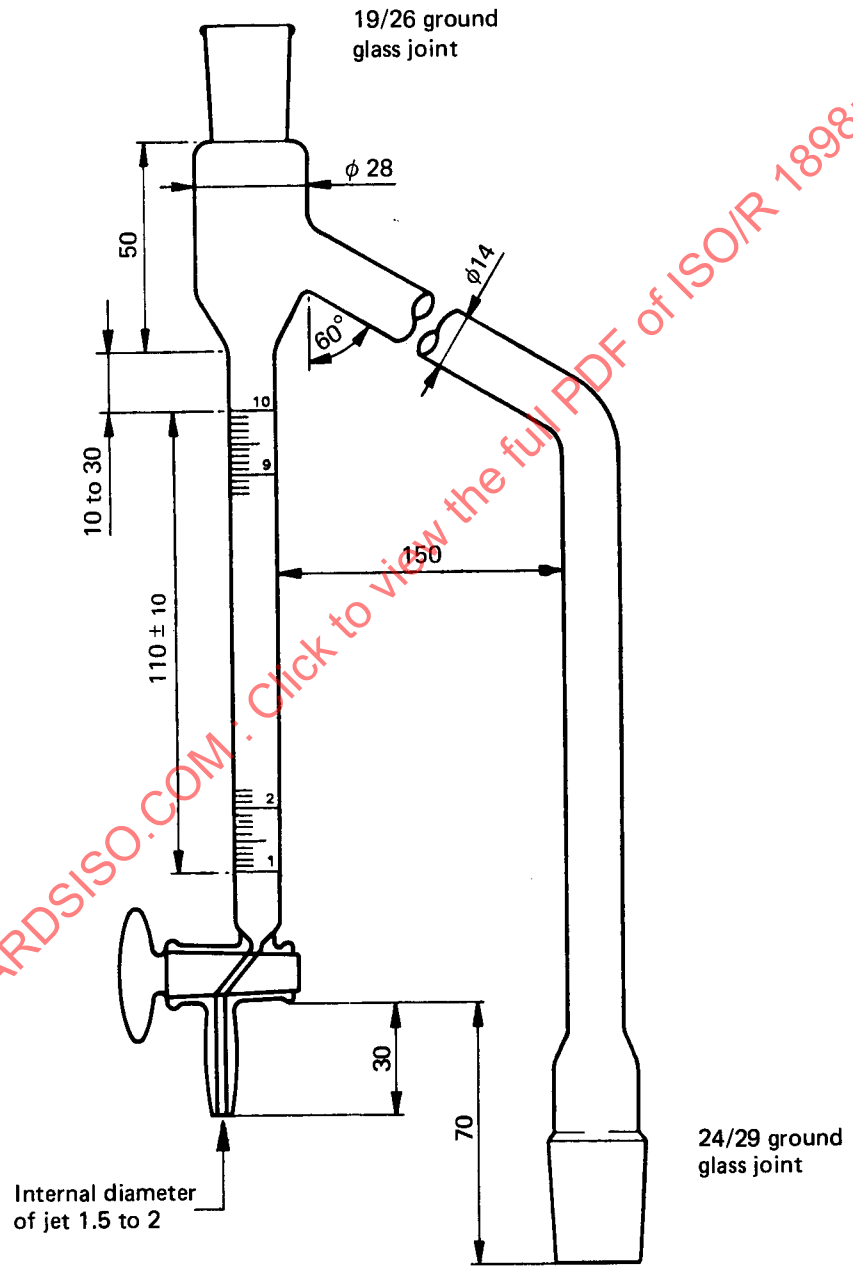

FIG. 4 – 10 ml receiver