



ISO 1897 PT\*II 77 4851903 0091745 1

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 II : Determination of water — Dean and Stark method

*Phénol, o-crésol, m-crésol, p-crésol, acide crésylique et xylénols à usage industriel — Méthodes d'essai —  
Partie II : Dosage de l'eau — Méthode de Dean et Stark*

First edition — 1977-04-15

UDC 661 : 547.562/563 : 543.812

Ref. No. ISO 1897/II-1977 (E)

**Descriptors :** phenols, phenol, cresols, xyleneol, 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 1898-1971 and found it technically suitable for transformation. Number 1898, however, has been changed to 1897/II. International Standard ISO 1897/II therefore replaces ISO Recommendation R 1898-1971, to which it is technically identical.

ISO Recommendation R 1898 had been approved by the member bodies of the following countries :

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

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

# Phenol, *o*-cresol, *m*-cresol, *p*-cresol, cresylic acid and xylenols for industrial use — Methods of test —

## Part II : Determination of water — Dean and Stark method

### 1 SCOPE AND FIELD OF APPLICATION

This part of ISO 1897 specifies 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.

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

### 2 PRINCIPLE

Distillation of a test portion, in the presence of an appropriate solvent, in a Dean and Stark apparatus.

Determination of the volume of condensate collected in the receiver tube, brought to ambient temperature, assuming this liquid to be water.

### 3 REAGENTS

During the analysis, use only a reagent of recognized analytical grade, *free from water*.

#### 3.1 Xylene.

The difference between distillation temperatures at which 5 % and 95 % of the volume taken have been collected shall not exceed 5 °C and this range shall lie between 137,0 and 145,5 °C.

Or, alternatively,

#### 3.2 Solvent naphtha.

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

Or, alternatively,

#### 3.3 Toluene.

### 4 APPARATUS

Ordinary laboratory apparatus and

**4.1 Dean and Stark apparatus**, comprising a 500 ml glass distillation flask fitted to a condenser with a 2 ml receiver having a maximum error of  $\pm 0,02$  ml, or a 10 ml receiver having a maximum error of  $\pm 0,06$  ml, or a 25 ml receiver having a maximum error of  $\pm 0,1$  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.

### 5 PROCEDURE

#### 5.1 Test portion

Weigh, to the nearest 0,1 g, at ambient temperature, 200 g of the test sample.

In the case of liquefied phenol, weigh, to the nearest 0,1 g, 100 g of the test sample.

#### 5.2 Determination

Transfer the test portion (5.1) to the flask of the Dean and Stark apparatus (4.1) and add 100 ml of the solvent (3.1, 3.2 or 3.3).

Attach the flask to the apparatus. 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 temperature of the water collected to ambient level and note its volume.

### 6 EXPRESSION OF RESULTS

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

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

where

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

$m$  is the mass, in grams, of the test portion (5.1).

NOTE — It is assumed that the density of the water collected in the graduated tube, at ambient temperature, is 1,00 g/ml.

Dimensions in millimetres

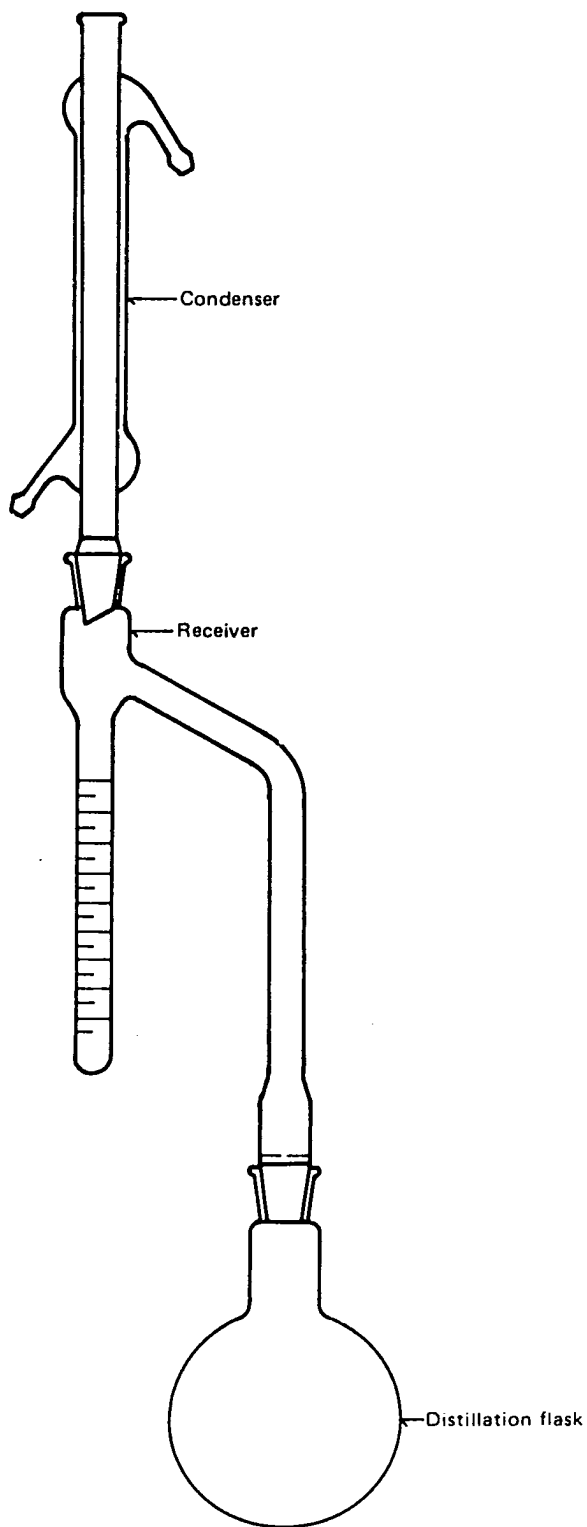


FIGURE 1 – Typical assembly of Dean and Stark apparatus (4.1)

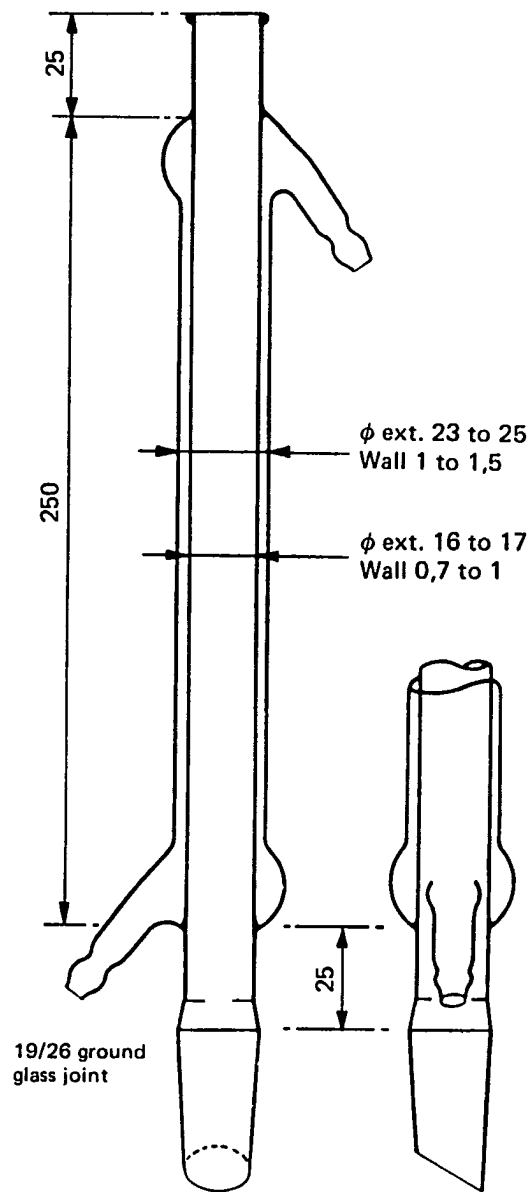


FIGURE 2 – Suitable form of condenser

Dimensions in millimetres

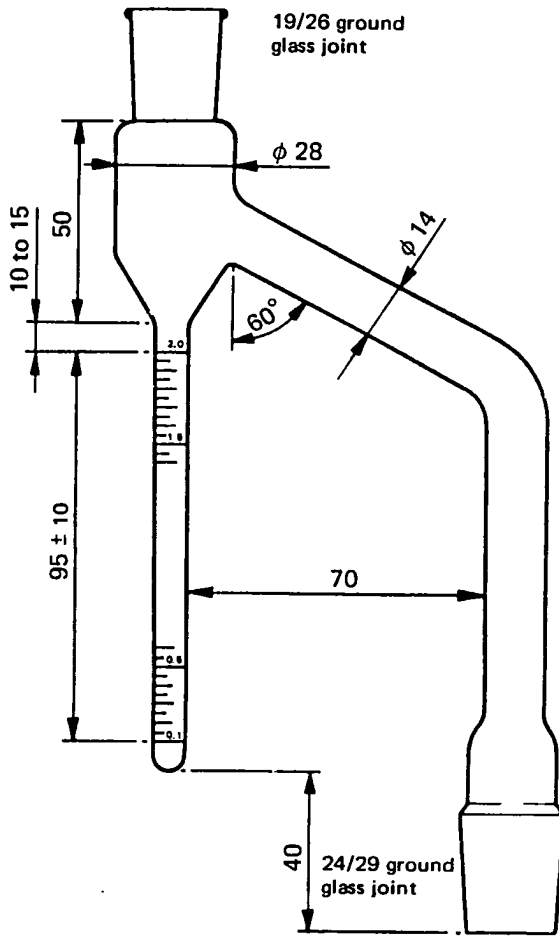


FIGURE 3 - 2 ml receiver

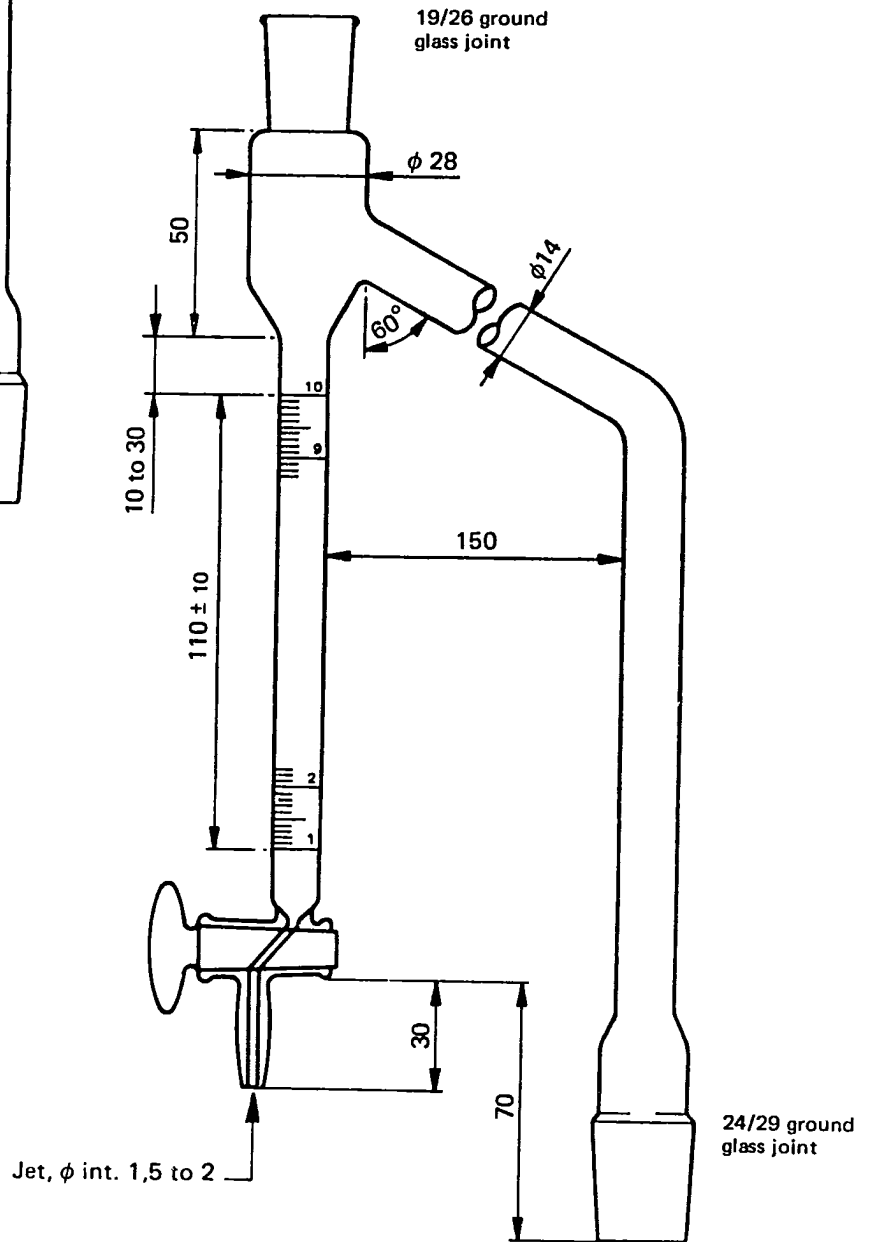


FIGURE 4 - 10 ml receiver



Dimensions in millimetres

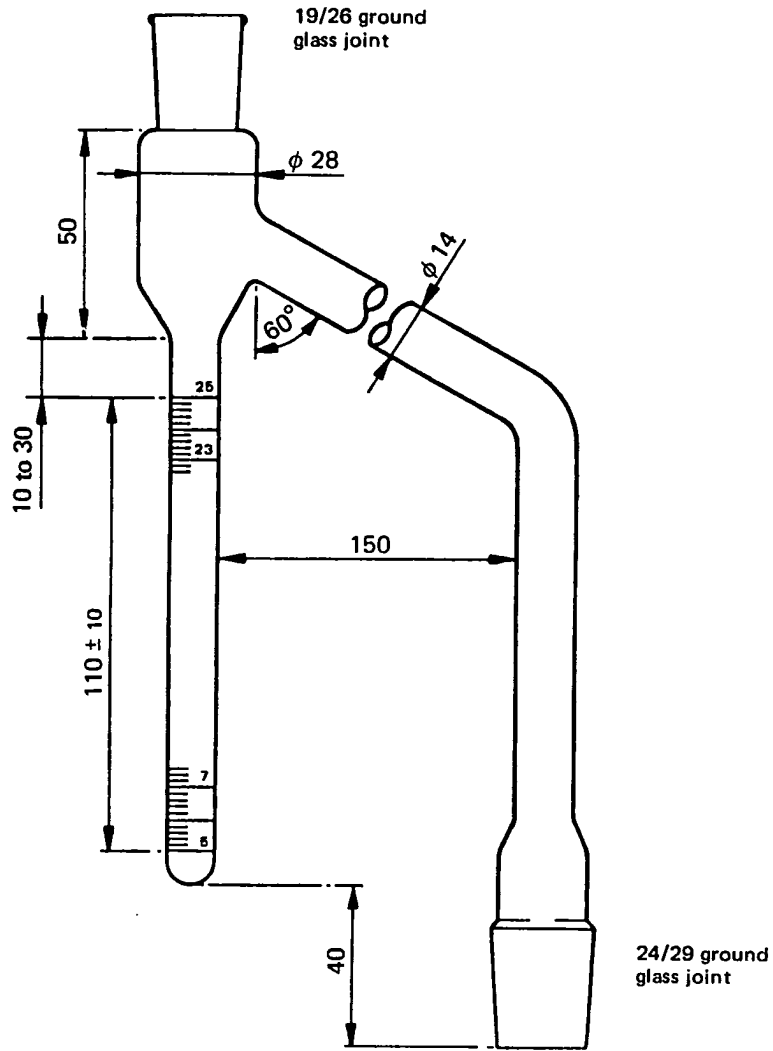


FIGURE 5 - 25 ml receiver

## 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 <sup>1)</sup>	B <sup>2)</sup>	C	D <sup>2)</sup>	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 <i>o</i> -cresol content.
			E			ISO 1897/IX – Determination of <i>m</i> -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 <sup>3)</sup>						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.