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(47)

ISO RECOMMENDATION

R 1906

CRESYLIC ACID AND XYLENOLS FOR INDUSTRIAL USE

DETERMINATION OF DISTILLATION RANGE

1st EDITION

May 1971

Printed in Switzerland

Also issued in French and Russian. Copies to be obtained through the national standards organizations.

BRIEF HISTORY

The ISO Recommendation R 1906, *Cresylic acid and xylenols for industrial use – Determination of distillation range*, 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. 1906, 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.

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CRESYLIC ACID AND XYLENOLS

FOR INDUSTRIAL USE

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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.

1. SCOPE AND FIELD OF APPLICATION

This ISO Recommendation describes a method for the determination of distillation range of cresylic acid of high *m*-cresol content, cresylic acid of high *o*-cresol content and xylenols for industrial use.

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.

3. DEFINITIONS

3.1 *Pitching point*. The point at which the temperature ceases to rise and begins to fall, while there is residue in the flask.

3.2 *Dry point*. The temperature at which the liquid just disappears from the bottom of the flask.

NOTE. – The flame of the burner should be removed or extinguished immediately the "dry point" is reached, otherwise the temperature recorded will be too high. The "dry point" is affected by any superheating of the vapours which may occur, and cannot therefore be recommended as an indication of the presence of high boiling constituents.

4. PRINCIPLE

Distillation of a given volume of the sample, under carefully defined conditions, and determination of either the volume of distillate collected as a function of temperature, or vice versa.

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

5. PROCEDURE

Use the method described in ISO Recommendation R 918, *Test method for distillation* (*distillation yield and distillation range*). The following particulars and modifications, specific to cresylic acid and xylenols, should be introduced in the above-mentioned ISO Recommendation.

5.1 Thermometer (see clause 3.2 in ISO/R 918), of the mercury-in-glass type, certified for accuracy and conforming to the following requirements :

Scale range	175 to 275 °C, 180 to 215 °C or 205 to 235 °C as appropriate to the material under test
Immersion	100 mm
Graduation interval	0.1 °C
Maximum error	± 0.6 °C
Minimum scale length	240 mm
Maximum overall length	430 mm

5.2 Condenser (see clause 3.4 in ISO/R 918)

Air cooled

5.3 Asbestos gauze (see clause 3.6 in ISO/R 918)

In place of the asbestos board

5.4 Temperature correction (see clause 7.2 in ISO/R 918)

A correction of $0.082\,(1.013 - p)\,(273 + t)$ where p is the barometric pressure in bars, and t is the mid-boiling temperature of the cresylic acid or xylenol in degrees Celsius, is added to the specified distillation temperatures.

5.5 Distillation (see section 6 of ISO/R 918)

Proceed as described in clause 6.1 of ISO/R 918. Distil slowly until all the water has come over (as shown by the distillate becoming clear at the end of the condenser) before increasing the distillation rate to 3 to 4 ml per minute (see clause 6.2 of ISO/R 918). Proceed as described in clause 6.3 of ISO/R 918.

Extinguish the flame of the burner as soon as 95 % (V/V) of the distillate has been obtained. Record this temperature. If the total distillate is required, continue the distillation until either the "dry point" or the "pitching point" (see Note in section 3) is reached and then extinguish the flame. The total distillate should include that which drains from the condenser within 5 minutes of extinguishing the flame.

6. 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.

ANNEX

This document forms one of a series of ISO Recommendations on methods of test for phenol, cresols, cresylic acid and xylenols for industrial use.

The complete list of the Recommendations already prepared or in course of preparation is as follows :

PHENOL, *o*-CRESOL, *m*-CRESOL, *p*-CRESOL, CRESYLIC ACID, XYLENOLS

ISO/R 1897, *Determination of water by the Karl Fischer method.*
ISO/R 1898, *Determination of water by the Dean and Stark method.*
ISO/R 1899, *Determination of neutral oils and pyridine bases.*

PHENOL, *o*-CRESOL, *m*-CRESOL, *p*-CRESOL

ISO/R 1900, *Determination of residue on evaporation.*
ISO/R 1901, *Determination of crystallizing point.*
ISO/R 2208, *Determination of crystallizing point after drying with a molecular sieve.**
ISO/R 1902, *Test for impurities insoluble in sodium hydroxide solution – Visual test.*
ISO/R 2273, *Determination, after combustion, of total sulphur (conductimetric method) and chlorine content (potentiometric or spectrophotometric method).**

LIQUEFIED PHENOL, *m*-CRESOL, CRESYLIC ACID, XYLENOLS

ISO/R 1903, *Determination of density at 20 °C.*

PHENOL

ISO/R 1904, *Determination of phenol content – Bromination method.**

LIQUEFIED PHENOL

ISO/R 1905, *Test for impurities insoluble in water – Visual test.*

CRESYLIC ACID AND XYLENOLS

ISO/R 1906, *Determination of distillation range.*
ISO/R 1907, *Determination of residue on distillation.*
ISO/R 1908, *Test for absence of hydrogen sulphide.*
ISO/R 1909, *Measurement of colour.*
ISO/R 1910, *Determination of o-cresol content.*

CRESYLIC ACID

ISO/R 1911, *Determination of m-cresol content.*

NOTE. – A laboratory sample of not less than 500 ml (for phenol and cresols) or 1000 ml (for cresylic acid and xylenols) is necessary to carry out the whole series of tests described in these documents.

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* At present at the stage of Draft ISO Recommendation.

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