International Standard 5274

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Aromatic hydrocarbons — Acid-wash test

Hydrocarbures aromatiques — Détermination de l'indice de coloration sulfurique

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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 5274 was developed by Technical Committee ISO/TC 78, *Aromatic hydrocarbons*, and was circulated to the member bodies in October 1977.

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

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No member body expressed disapproval of the document.

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Aromatic hydrocarbons — Acid-wash test

WARNING — Aromatic hydrocarbons are generally toxic by inhalation, ingestion or skin absorption. Volatile aromatic hydrocarbons are also highly flammable.

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies an acid-wash test for aromatic hydrocarbons.

The method gives some indication of the degree of refining, but it does not give a precise measure of the amount of unsaturated compounds present, as different unsaturated compounds produce different colorations with sulphuric acid.

2 PRINCIPLE

Shaking together of equal volumes of the sample and 95 % (*m/m*) sulphuric acid. Comparison of the colour of the acid layer with that of standard solutions.

3 REAGENTS

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

3.1 Potassium dichromate, anhydrous.

3.2 Sulphuric acid, concentrated, about 98 % (*m/m*) or approximately 18 M.

3.3 Sulphuric acid, 95 ± 0,5 % (*m/m*), checked by titration.

3.4 Potassium permanganate, 3,2 g/l solution.

4 APPARATUS

4.1 Three stoppered cylinders, made of thin-walled, "colourless" glass tubing, having an internal diameter of 13 ± 0,5 mm. Each cylinder shall be about 100 mm in height to the shoulder and encircled with two marks. The upper mark shall be 70 ± 1 mm from the outside of the base of the cylinder and the lower mark shall be so placed that the capacity of the cylinder to that mark differs from that between the upper and the lower mark by not more than 0,1 ml.

It is essential that the three cylinders be similar in all respects, including colour.

4.2 Water bath, capable of being controlled at 20 ± 1 °C.

5 SAMPLING[1)]

Take a representative sample of not less than 1 000 ml from the bulk of the material.

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6 PROCEDURE

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WARNING — This test is potentially dangerous to the operator and adequate precautions should be taken to avoid contact of the highly corrosive and oxidizing solution with the skin or eyes. Goggles should be worn.

6.1 Preparation of colour standards

The colour standards shall be prepared by dissolving the potassium dichromate (3.1) in sulphuric acid and water. In preparing them, careful attention shall be paid to the following details.

6.1.1 The standards have oxidizing properties and shall therefore be made and stored out of contact with organic matter of any kind. Use glass-stoppered apparatus, entirely free from grease and dust, for preparing and storing the solutions.

6.1.2 Prepare a stock dilute acid solution, containing equal volumes of water and the 98 % (*m/m*) sulphuric acid (3.2), by cautiously adding the acid to water with constant stirring, and bring to room temperature before use.

6.1.3 Check the water and the sulphuric acid for freedom from oxidizable matter by adding 2 drops (0,1 ml) of the potassium permanganate solution (3.4) to 100 ml of the stock dilute acid solution. The permanganate should not be decolorized within 5 min.

1) The sampling of aromatic hydrocarbons will form the subject of ISO 1995.

6.1.4 From a part of the stock dilute acid solution, prepare a stock dichromate solution containing a known quantity of potassium dichromate in excess of the highest standard required. Make the standard solutions for comparison by dilution of portions of the stock dichromate solution with appropriate quantities of stock acid solution. The highest concentration of potassium dichromate that can be prepared is 10 g per 1 000 ml of stock acid solution, and this quantity dissolves only very slowly.

6.1.5 The standard solutions for comparison shall be prepared and used on the same day.

6.1.6 The stock acid solution, the stock dichromate solution and the standard solutions for comparison shall be kept in the dark when not in use.

6.2 Measurement

Clean the three cylinders (4.1) with chromic acid mixture, rinse with water and dry.

Do not carry out the test in direct sunlight.

Fill one of the cylinders (4.1) to the upper mark with a dichromate solution of the concentration represented by the specified standard. Similarly fill a second cylinder with a solution of the concentration higher than the specified standard by the equivalent of the precision figure, *r* (see 8.1). Prepare these dichromate solutions, as described in 6.1, so that they contain the specified amount of dichromate, to the nearest 0,01 g per 1 000 ml.

Filter the sample and reject the first 10 ml of the filtrate. Fill the third cylinder to the lower mark, with the filtered sample, stopper, and place it in the water bath (4.2), controlled at 20 ± 1 °C. Place a quantity of the 95 % (*m/m*) sulphuric acid (3.3) in a suitable container and leave it in the water bath until it reaches the temperature of the bath, then pour the acid into the cylinder until the contents are level with the upper mark.

Stopper the cylinder, shake it vigorously for 2 min ± 5 s return it to the water bath and leave it undisturbed for 10 min ± 5 s. Immediately compare the colour of the acid layer with that of the contents of the other cylinders by the illumination of a good north light, or by a means of a white-light cabinet.

7 EXPRESSION OF RESULTS

Report the result as paler than the standard, equal to the standard, darker than the standard but within the precision of the test, or as darker than the standard.

8 PRECISION

The precision of the test method, as obtained by statistical examination of interlaboratory results, is as follows.

8.1 Repeatability (*r*)

The value below which the absolute difference between two single test results, on identical test material, obtained by one operator in one laboratory, using the same equipment within a short interval of time, and applying the standardized test method, may be expected to lie with a 95 % probability, is given in the table.

8.2 Reproducibility (*R*)

The value below which the absolute difference between two single test results, on identical test material, obtained by operators in different laboratories, applying the standardized test method, may be expected to lie with a 95 % probability, is given in the table.

TABLE – **Precision of the acid-wash test**

Potassium dichromate content of the colour standard	*r*	*R*
g/l of dilute acid solution		
0,1 to 0,4	0,05	0,1
0,5	0,1	0,2
1,0	0,1	0,5
1,5	0,2	0,6
2,0	0,4	1,0
3,0	0,8	1,8
5,0	2,0	3,5

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NOTES

1 The test is less precise for higher levels.

2 Some samples are liable to give higher results as the age of the sample increases.

9 TEST REPORT

The test report shall include at least the following information :

a) the type and identification of the product tested;

b) a reference to this International Standard;

c) any deviation, by agreement or otherwise, from the procedure specified;

d) the result of the test;

e) the date of the test.