Explanatory Notes to the Combined Nomenclature of the European Union

(2016/C 357/04)

Pursuant to Article 9(1)(a) of Council Regulation (EEC) No 2658/87 [1], the Explanatory Notes to the Combined Nomenclature of the European Union [2] are hereby amended as follows:

On page 138, Annex A of the Explanatory notes to Chapter 27 is replaced by the following text:

'ANNEX A

METHOD OF DETERMINING THE CONTENT OF AROMATIC CONSTITUENTS IN PRODUCTS WITH A DISTILLATION END POINT EXCEEDING 315 °C

1. Scope

This test method covers the determination of the content of aromatic and non-aromatic constituents in mineral oils.

2. Definition

2.1. Aromatic constituents: the portion of the sample dissolved in the solvent and adsorbed on silica gel. The aromatic constituents may contain: aromatic hydrocarbons, condensed naphthenic-aromatics, aromatic olefins, asphaltenes, aromatic compounds containing sulphur, nitrogen, oxygen and polar aromatics.

2.2. Non-aromatic constituents: the portion of the sample which is not adsorbed on silica gel and which is eluted by the solvent (such as non-aromatic hydrocarbons).

3. Principle of the method

The sample, dissolved in *n*-pentane, is allowed to percolate through a special chromatography column packed with silica gel. The nonaromatic constituents, eluted with solvent, are collected subsequently and assayed by weighing after the solvent has evaporated.

Samples not dissolving in *n*-pentane should be dissolved in cyclohexane.

4. Apparatus and reagents

Chromatography column: this is a glass tube with the dimensions and shape shown in the accompanying sketch. The top aperture must be capable of being sealed by a glass joint having its ground flat face pressed against the top of the column by two rubber-covered metal clamps. The seal must be completely leak tight against an applied pressure of nitrogen or air.

Silica gel: fineness of 200 mesh or more. It must be activated for seven hours in an oven kept at 170 °C before use and placed in a desiccator to cool. After activation, the silica gel must be used within a few days.

Solvent I *n*-pentane: minimum 95 % pure, aromatic-free.

Solvent II cyclohexane: minimum 98 % pure, aromatic-free.

5. Procedure 1 (chromatography column 1)

Preparation of the sample solution: dissolve approximately 3,6 g (exactly weighed) of the sample in 10 ml of *n*-pentane (I). If the sample is insoluble in *n*-pentane, dissolve it in cyclohexane and the determination is performed using cyclohexane (II) instead of *n*-pentane (I).

Pack the chromatography column (chromatography column 1) with the previously activated silica gel, up to about 10 cm from the upper glass bulb, by carefully tamping the contents of the column with a vibrator so as not to leave any channels. Then insert a plug of glass wool in the top of the silica gel column.

Pre-moisten the silica gel with 180 ml of solvent (I) or (II), and apply a pressure of air or nitrogen from above until the upper surface of the liquid reaches the top of the silica gel.

Carefully release the pressure inside the column and pour over it approximately 3,6 g (exactly weighed expression with 2 decimals) of sample dissolved in 10 ml of solvent (I) or (II), then rinse out the beaker with another 10 ml of solvent (I) or (II), and pour this also over the column.

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[1] Council Regulation (EEC) No 2658/87 of 23 July 1987 on the tariff and statistical nomenclature and on the Common Customs Tariff (OJ L 256, 7.9.1987, p. 1).

[2] OJ C 76, 4.3.2015, p. 1.

Apply the pressure progressively while allowing the liquid to flow in drops from the bottom capillary tube of the column at the approximate rate of 1 ml/min and collect this liquid in a 500 ml flask.

When the level of the liquid containing the substance to be separated falls to the surface of the silica gel, carefully remove the pressure once more and add 230 ml of solvent (I) or (II), re-apply the pressure immediately and bring down the level of the liquid to the surface of the silica gel while collecting the eluate in the same flask as before.

Before the level of the liquid containing the substance to be separated falls to the surface of the silica gel, check the eluate using FT-IR for the presence of aromatics. If the eluate contains only aliphatic hydrocarbons, add again 50 ml of solvent (I) or (II) after removing the pressure. Repeat this step if necessary.

Reduce the collected fraction to a small volume by evaporation in a vacuum oven at 35 °C or in a vacuum rotary evaporator or similar apparatus and then transfer it without loss into a tared beaker, using more solvent (I) or (II).

Evaporate the contents of the beaker in a vacuum oven at 35 °C to constant weight (W). The difference between the two last weights should not exceed 0,01 g. The time difference between the two weighings should be at least 30 minutes.

The percentage of non-aromatic constituents by weight (A) is given by the following formula:

$A = W/W_1*100$

where W_1 is the weight of the sample.

The difference from 100 is the percentage of aromatic constituents absorbed by the silica gel.

6. **Accuracy of the method**

Repeatability: 5 %.

Reproducibility: 10 %.

7. **Procedure 2 (chromatography column 2)**

Preparation of the sample solution: dissolve approximately 0,9 g (exactly weighed) of the sample in 2,5 ml of *n*-pentane (I). If the sample is insoluble in *n*-pentane, dissolve it in cyclohexane and the determination is performed using cyclohexane (II) instead of *n*-pentane (I).

Pack the chromatography column (chromatography column 2) with the previously activated silica gel, up to about 2,5 cm from the upper glass bulb, by carefully tamping the contents of the column with a vibrator so as not to leave any channels. Then insert a plug of glass wool in the top of the silica gel column.

Pre-moisten the silica gel with 45 ml of solvent (I) or (II), and apply a pressure of air or nitrogen from above until the upper surface of the liquid reaches the top of the silica gel.

Carefully release the pressure inside the column and pour over it approximately 0,9 g (exactly weighed expression with 2 decimals) of sample dissolved in 2,5 ml of solvent (I) or (II), then rinse out the beaker with another 2,5 ml of solvent (I) or (II), and pour this also over the column.

Apply the pressure progressively while allowing the liquid to flow in drops from the bottom capillary tube of the column at the approximate rate of 1 ml/min and collect this liquid in a 250 ml flask.

When the level of the liquid containing the substance to be separated falls to the surface of the silica gel, carefully remove the pressure once more and add 57,5 ml of solvent (I) or (II), re-apply the pressure immediately and bring down the level of the liquid to the surface of the silica gel while collecting the eluate in the same flask as before.

Before the level of the liquid containing the substance to be separated falls to the surface of the silica gel, check the eluate using FT-IR for the presence of aromatics. If the eluate contains only aliphatic hydrocarbons, add again 12,5 ml of solvent (I) or (II) after removing the pressure. Repeat this step if necessary.

Reduce the collected fraction to a small volume by evaporation in a vacuum oven at 35 °C or in a vacuum rotary evaporator or similar apparatus and then transfer it without loss into a tared beaker, using more solvent (I) or (II).

Evaporate the contents of the beaker in a vacuum oven at 35 °C to constant weight (W). The difference between the two last weights should not exceed 0,01 g. The time difference between the two weighings should be at least 30 minutes.

The percentage of non-aromatic constituents by weight (A) is given by the following formula:

$A = W/W_1*100$

where W_1 is the weight of the sample.

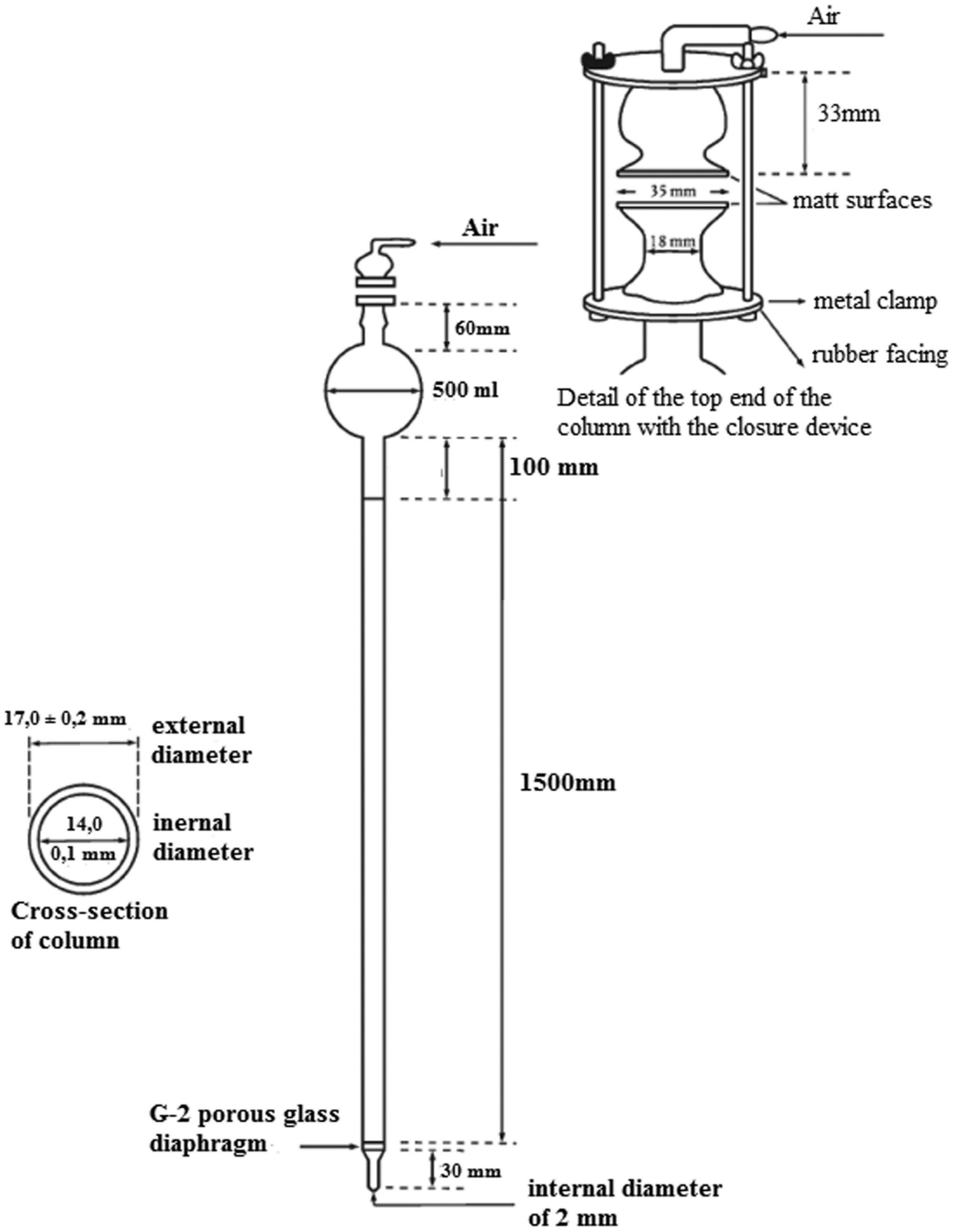
The difference from 100 is the percentage of aromatic constituents absorbed by the silica gel.

8. **Accuracy of the method**

Repeatability: 5 %.

Reproducibility: 10 %.

Chromatography column 1



Chromatography column 2