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E-liquid — Determination of nicotine, propylene glycol and glycerol in liquids used in electronic nicotine delivery devices — Gas chromatographic method

E-liquide — Détermination de la teneur en nicotine, propylène glycol et glycérol dans les liquides utilisés avec les systèmes électroniques de délivrance de nicotine — Méthode par chromatographie en phase gazeuse

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Foreword

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This document was prepared by Technical Committee ISO/TC 126, *Tobacco and tobacco products*, Subcommittee SC 3, *Vape and vapour products*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

E-liquid — Determination of nicotine, propylene glycol and glycerol in liquids used in electronic nicotine delivery devices — Gas chromatographic method

1 Scope

This document specifies an analytical method to quantify the nicotine, propylene glycol and glycerol content in e-liquids by gas chromatography.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
e-liquid
liquid or gel consumable which may or may not contain nicotine intended for transformation into an aerosol and then inhaled with an *electronic nicotine delivery device* (3.2)

3.2
electronic nicotine delivery device
device used to transform an *e-liquid* (3.1) into an inhalable aerosol

4 Principle

The e-liquid sample is diluted with a solution of isopropanol containing internal standard(s). The nicotine, propylene glycol and glycerol content of the diluted sample is analysed by capillary gas chromatography with flame ionization detection (GC-FID) and quantified by using an internal standard.

5 Reagents

Use only reagents of recognized analytical grade.

5.1 Carrier gas: helium (CAS: 7440-59-7) or hydrogen (CAS: 1333-74-0) of high purity.

5.2 Auxiliary gases: hydrogen (CAS: 1333-74-0) of high purity and air for the flame ionization detector.

5.3 Isopropanol (CAS: 67-63-0), minimum purity 99 %, used with internal standard(s) to prepare the dilution solution.

5.4 Internal standards of high purity: quinaldine (CAS: 91-63-4), 1,3-butanediol (CAS: 107-88-0), n-heptadecane (CAS: 629-78-7), n-octadecane (CAS: 593-45-3). Anethol and other appropriate internal standards may be used after assessment of their purity and determination that the internal standard does not co-elute with other components in the dilution. The peak area of the internal standard on samples should be monitored for consistency. In cases where inconsistencies are found, analysis of a diluted sample without the internal standard should be performed to confirm the absence of a peak in the extract eluting at the same time as the internal standard.

5.5 Dilution solution: isopropanol (5.3) containing an appropriate concentration of the internal standard (5.4), e.g. 0,2 mg/ml of quinaldine, 1 mg/ml of 1,3-butanediol, 1 mg/ml of n-heptadecane or 1 mg/ml of n-octadecane (5.4).

5.6 Reference substances

5.6.1 Nicotine (CAS: 54-11-5), of known purity not less than 98 %. Nicotine salicylate (CAS: 29790-52-1) of known purity not less than 98 % may also be used.

NOTE The purity of the nicotine or nicotine salicylate can be verified using ISO 13276 or by any other validated method.

5.6.2 Propylene glycol (CAS: 57-55-6), analytical grade, minimum purity 99 %.

5.6.3 Glycerol (CAS: 56-81-5), analytical grade, minimum purity 99 %.

Store these reference substances at a temperature in accordance with the manufacturer's recommendation.

5.7 Calibration solutions

Prepare a series of at least five calibration solutions with concentrations that cover the range of expected levels to be found in the test portion by adding weighed amounts of nicotine (5.6.1), propylene glycol (5.6.2) and glycerol (5.6.3) to the dilution solution (5.5). The following linear ranges were used in the collaborative study: 0,005 mg/ml to 1,5 mg/ml for nicotine, 0,12 mg/ml to 10 mg/ml for propylene glycol and glycerol.

Store these solutions between 2 °C and 8 °C in a dark environment.

Solutions stored at low temperatures shall be allowed to equilibrate to room temperature before use.

6 Apparatus

Usual laboratory apparatus and, in particular, the following items.

6.1 Gas chromatograph, equipped with a flame ionization detector, recorder, integrator or data handling system.

6.2 Capillary column

DB-ALC1[1)] capillary column (30 m length × 0,32 mm ID, 1,8 µm film thickness) has been found to be satisfactory.

Alternative capillary columns (such as a WAX column) may be used provided that the peaks for solvent, internal standards, nicotine, propylene glycol, glycerol and other e-liquids components are well resolved which can require optimization of the instrument conditions.

1) ALC1 is an example of a suitable product commercially available. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.