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(71) Applicant: **Akademia Gorniczo-Hutnicza im. Stanislawa Staszica w Krakowie**
30-059 Krakow (PL)

(72) Inventors:
- **JANUS, Rafa**
  32-005 Niepo omice (PL)
- **W DRZYK, Mariusz**
  31-425 Kraków (PL)
- **LEWANDOWSKI, Marek**
  40-881 Katowice (PL)

(74) Representative: **Balinska, Ewa et al**
Polservice
Kancelaria Rzecznikow
Patentowych sp. z o.o.
Bluszczanska 73
00-712 Warszawa (PL)

(54) CAPILLARY STABILIZER FOR A LIQUID SAMPLE

(57) A capillary stabilizer for a liquid sample, consisting of a capillary and a supporting element (a holder) being characteristic in that the capillary (1) is made of borosilicate glass and has a length in the range from 35.0 to 60.0 mm, an outer diameter in the range from 6.5 to 10.0 mm, and an inner diameter in the range from 1.2 to 1.8 mm. In the upper portion of the capillary there is a widening (2), which in its inlet portion has a diameter in the range of from 4.0 to 8.0 mm, and a height in the range of from 5.0 to 10.0 mm. In the lower portion of the capillary there is a widening (3) with a diameter of its base in the range of from 3.0 to 3.5 mm and a height in the range of from 0.7 to 1.0 mm. On the outer perimeter of the upper outlet of the capillary a chamfer (4) is made having a depth in the range of from 0.3 to 0.5 mm, simultaneously the capillary is placed in a supporting element (a holder) (5). Preferably, the capillary (1) has a funnel-shaped widening (4) in its upper portion. Preferably, the capillary (1) has a conical widening (3) in its lower portion.

Preferably, the holder (5) that is designed for operation at a room temperature is made of plastic. Preferably, the plastic from which the holder (5) is made is organic glass. Preferably, the holder (5) in the high-temperature variant is made of metal. Preferably, the metal from which the holder (5) is made is aluminum. Preferably, the holder (5) has a cylindrical hollow recess (6) at the bottom. Preferably, the holder (5) has a conical hollow recess (7) on the bottom located around the opening through which the capillary (1) passes.

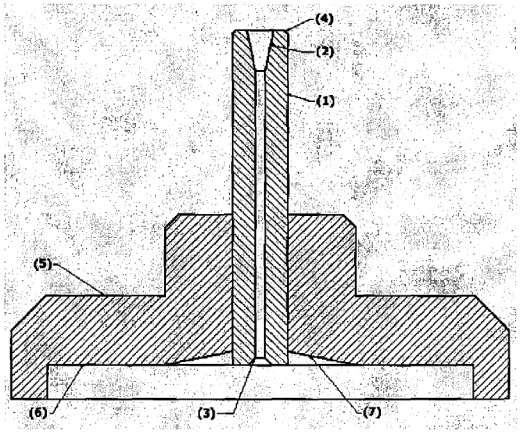

Fig. 1

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Description

[0001] The invention relates to a capillary stabilizer for a liquid sample designed for recording of infrared spectra of volatile liquids by the Attenuated Total Reflectance Infrared (ATR-FTIR) technique.

[0002] From U.S. patent specification of US8223429B2 an auxiliary tool for testing samples is known that consists of a platform mounted on a stage of a microscope that is designed for ATR mode measurements, which platform includes a mounting element located on the platform and a sample holding element that is also located on the platform, wherein the holding element is movable to a limited extend relative to the stage and it is located below an ATR crystal.

[0003] A solution known from the manufacturer's PerkinElmer brochure (available at the address https://www.s4science.at/wordpress/wp-content/uploads/2019/04/ L105023.pdf) is based on use of a stainless steel tube with an outer diameter of 7 mm and an inner diameter of 3 mm and a height of a few millimeters, which tube is centered coaxially with respect to an ATR crystal by means of a high-pressure arm. Once the tube is positioned, a sample is introduced into the tube (on a surface of the crystal). When measuring spectra of particularly volatile liquids the manufacturer recommends positioning the high-pressure arm over the tube and pressing down on a tip, to reduce evaporation.

[0004] A solution known from the manufacturer's Pike Technologies brochure (available online at https://www.piketech.com/wp-content/uploads/PIKE-Technologies_GladiATR.pdf) consists of a thin disk with a funnel-shaped cavity on the bottom of which an opening having a dimeter of 3 mm is located (*Liquids Retainer* - an element that prevents a sample from spilling over a surface of an ATR stage), a cover to inhibit evaporation of a sample (*Volatiles Cover*) and a U-shaped bridge that serves to centering an accessory and to tighten it when pressed with the high-pressure arm of the apparatus.

[0005] The solutions presented above do not protect a sample from contact with air, which can affect its composition, require the use of a high-pressure arm, and make it difficult - or impossible - to observe the sample as it comes into contact with the crystal.

[0006] The essence of an inventive capillary stabilizer for a liquid sample, that stabilizer consists of a capillary and a supporting element (a holder), is that the capillary is made of borosilicate glass and it has a length in the range of from 35.0 to 60.0 mm, an outer diameter in the range of from 6.5 to 10.0 mm, and an inner diameter in the range of from 1.2 to 1.8 mm. In an upper portion of the capillary there is a widening (extension), which in its inlet portion has a diameter in the range of from 4.0 to 8.0 mm and a height in the range of from 5.0 to 10.0 mm. In a lower portion of the capillary there is located a widening having a diameter of its base in the range of from 3.0 to 3.5 mm and a height in the range of from 0.7 to 1.0 mm, whereas a chamfer is provided on an outer perimeter of an upper outlet of the capillary having a depth in the range of from 0.3 to 0.5 mm. Simultaneously, the capillary is placed in a supporting element (a holder).

[0007] Preferably, the capillary at its upper portion has a widening of a funnel-shape.

[0008] Preferably, the capillary in its lower portion has a widening of a conical-shape.

[0009] Preferably, the holder is made of plastic.

[0010] Preferably, the plastic of which the holder is made is organic glass.

[0011] Preferably, the holder is made of metal.

[0012] Preferably, the metal of which the holder is made is aluminum.

[0013] Preferably, the holder has a cylindrical hollow recess at the bottom.

[0014] Preferably, the holder has a conical hollow recess located at the bottom thereof around the opening through which the capillary passes.

[0015] The inventive solution ensures that the effect of changes in concentrations of analyzed samples caused by evaporation of their volatile components from the ATR crystal attachment during the measurement and by chemical transformations associated with a contact of the sample with atmospheric oxygen is eliminated. This makes it possible to record a large number of scans (increasing in the measurement time), which significantly improves a quality of IR spectra as a result of maximizing the signal-to-noise ratio (spectrum *smoothing* effect). This, in turn, enables observation of absorption bands having low intensity, and thus allows for an in-depth analysis of spectra.

[0016] The solution relates to the field of molecular spectroscopy as one of the branches of instrumental analysis and can be used in recording of IR spectra of a high quality of problematic analytical samples in the form of, among others, volatile compounds/mixtures or those containing volatile components, such as crude oil, low-boiling petroleum fractions and their mixtures (e.g. motor gasoline, aviation fuels), volatile solvent-based solutions (mixtures), extracts containing low-boiling components, paint and varnish solvents, essential oils and fragrance compositions, volatile pharmaceutical and cosmetic products, condensates of volatile organic compounds and other substances characterized by high vapor pressure.

[0017] Thanks to possibility of stabilizing of the composition of the aforementioned problematic samples during IR analysis, there is an opportunity to expand the use of the IR technique for studying of such samples' composition or physicochemical properties. In the high-temperature variant of the solution (the capillary made of borosilicate glass and a centering holder made of aluminum), the subject stabilizer according to the application is compatible with high-temperature ATR devices designed for operation at temperatures generally not exceeding 300 °C. Depending on the operating conditions, the holder can be made of metal (to work in a high-temperature) or of plastic (to work in a room temperature).

The conical-shaped widening 3 (extension) prevents to contact of the glass with the ATR crystal and facilitates application of the sample to its surface.

[0018] The inventive solution in the embodiment is shown in the drawing which presents a cross-section of the capillary stabilizer.

[0019] The capillary stabilizer according to the invention consists of a capillary 1 and a supporting element (a holder) 5. The capillary 1 is in the form of a segment of a thick-walled tube made of borosilicate glass, which segment has a length of 49.0 mm, an outer diameter of 8.0 mm and an inner diameter of 1.5 mm. A conical-shaped widening 2 in the upper portion of the capillary 1 is realized by means of hollow-out method, and having a diameter of its inlet portion in the range of from 4.0 to 8.0 mm, and a height in the range of from 5.0 to 10.0 mm. A conical-shaped widening 3 (extension) is made in the lower portion of the capillary 1 and having a diameter of its base of 3.5 mm and a height of 1.0 mm, whereas a chamfer 4 having a depth of 0.5 mm is made on an outer perimeter of the upper outlet of the capillary 1. Simultaneously, the capillary 1 is placed in a supporting element - a holder 5, that is made of plastic, such as organic glass. The holder 5 has a cylindrical hollow recess 6 at its bottom having a diameter of 62.0 mm and a depth of 5.0 mm. The holder 5 at its bottom underside and located around the opening through which the capillary 1 passes has a conical hollow recess 7 with a diameter of from 25.0 to 30.0 mm and a height of from 2.0 to 3.0 mm at the point of contact to the capillary, that recess is made coaxially with the axis of the holder 5.

[0020] The capillary 1 having an inner diameter of 1.5 mm is designed to provide the height of a liquid column over the ATR crystal in the range of from a few to several (between ten and twenty) millimeters.

[0021] Cylindrical hollow recess 6 is provided to ensure that the capillary 1 and the ATR crystal are positioned coaxially to each other.

[0022] The conical hollow recess 7 is provided to prevent a contact between the sample and the material of the holder 5.

Claims

1. Capillary stabilizer for a liquid sample, that consists of a capillary and a supporting element (a holder), **characterized in that** the capillary (1) is made of borosilicate glass and it has a length in the range of from 35.0 to 60.0 mm, an outer diameter in the range of from 6.5 to 10.0 mm and an inner diameter in the range of from 1.2 to 1.8 mm, wherein a widening (2) is located in the upper portion of the capillary (1), which the widening in its inlet portion has a diameter in the range of from 4.0 to 8.0 mm, and a height in the range of from 5.0 to 10.0 mm, while in the lower portion of the capillary (1) there is a widening (3) having with a diameter of its base in the range of from 3.0 to 3.5 mm and a height in the range of from 0.7 to 1.0 mm, and whereas a chamfer (4) is made on the outer perimeter of the upper outlet of the capillary (1) which chamfer (4) having a depth in the range of from 0.3 to 0.5 mm, simultaneously the capillary (1) is placed in a supporting element - holder (5).

2. The capillary stabilizer for a liquid sample according to claim 1, **characterized in that** the capillary (1) at its upper portion has a funnel-shaped widening (2).

3. The capillary stabilizer for a liquid sample according to claim 1, **characterized in that** the capillary (1) in its lower portion has a conical-shaped widening (3).

4. The capillary stabilizer for a liquid sample according to claim 1, **characterized in that** the holder (5) is made of plastic.

5. The capillary stabilizer for a liquid sample according to claim 4, **characterized in that** the plastic of which the holder (5) is made is organic glass.

6. The capillary stabilizer for a liquid sample according to claim 1, **characterized in that** the holder (5) is made of metal.

7. The capillary stabilizer for a liquid sample according to claim 6, **characterized in that** the metal of which the holder (5) is made is aluminum.

8. The capillary stabilizer for a liquid sample according to claim 1, **characterized in that** the holder (5) has a cylindrical hollow recess (6) at its bottom.

9. The capillary stabilizer for a liquid sample according to claim 1, **characterized in that** the holder (5) has a conical hollow recess (7) located at its bottom and around the opening through which the capillary (1) passes.

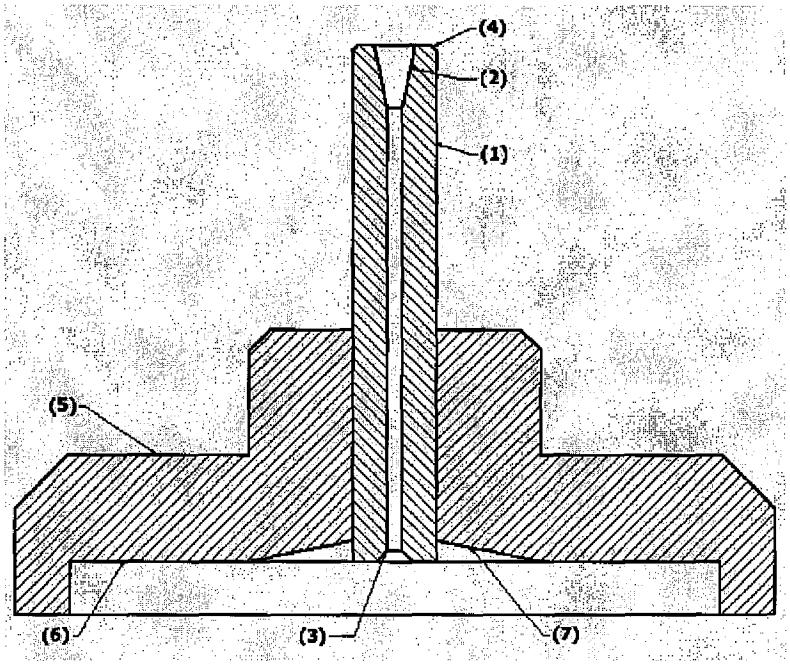

Fig. 1

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EUROPEAN SEARCH REPORT

Application Number
EP 22 21 3041

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (IPC)
X,P	JANUS RAFAL ET AL: "A novel capillary forces-founded accessory for reliable measurements of ATR-FTIR spectra of volatile liquids", MICROCHEMICAL JOURNAL, NEW YORK, NY, US, vol. 185, 24 November 2022 (2022-11-24), XP087235381, ISSN: 0026-265X, DOI: 10.1016/J.MICROC.2022.108219 [retrieved on 2022-11-24] * the whole document *	1-9	INV. G01N21/01
X	JP 2001 318270 A (NIPPON ELECTRIC GLASS CO) 16 November 2001 (2001-11-16) * the whole document *	1-9	
X	US 7 391 953 B2 (NIPPON ELECTRIC GLASS CO [JP]) 24 June 2008 (2008-06-24) * column 7, line 32 - column 11, line 24; figures *	1	
			TECHNICAL FIELDS SEARCHED (IPC)
			G01N

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
Munich	10 August 2023	Politsch, Erich

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For more details about this annex : see Official Journal of the European Patent Office, No. 12/82

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