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**Datasheet for the decision
of 25 February 2008**

Case Number: T 0746/05 - 3.4.02

Application Number: 01100364.7

Publication Number: 1106988

IPC: G01N 21/05

Language of the proceedings: EN

Title of invention:

Flow cell with an inner layer of an amorphous fluoropolymer having a refractive index less than the refractive index of water

Applicant:

Waters Investments Limited

Headword:

-

Relevant legal provisions:

EPC Art. 56

Keyword:

"Inventive step (yes)"

Decisions cited:

-

Catchword:

-



Case Number: T 0746/05 - 3.4.02

D E C I S I O N
of the Technical Board of Appeal 3.4.02
of 25 February 2008

Appellant: Waters Investments Limited
109 Lukens Drive
New Castle
Delaware 19720 (US)

Representative: Vossius, Corinna
Dr. Volker Vossius
Patent- und Rechtsanwaltskanzlei
Geibelstrasse 6
D-81679 München (DE)

Decision under appeal: Decision of the Examining Division of the
European Patent Office posted 18 March 2005
refusing European application No. 01100364.7
pursuant to Article 97(1) EPC.

Composition of the Board:

Chairman: A. G. Klein
Members: F. J. Narganes-Quijano
C. Rennie-Smith

Summary of Facts and Submissions

- I. The appellant (applicant) lodged an appeal against the decision of the examining division to reject European divisional patent application No. 01100364.7 published with the publication No. 1106988 and deemed to have been filed on the date of filing (15.07.1992) of the earlier European patent application No. 92112114.1 (publication No. 0523680) pursuant to Article 76(1) EPC.

In its decision the examining division held that the subject-matter of claim 1 then on file did not involve an inventive step (Articles 52(1) and 56 EPC). In particular, the examining division referred to the disclosure of documents

Y1 : US-A-4754009

Y5 : "The use of poly(tetrafluoroethylene-co-hexafluoropropylene) tubing as a waveguide capillary cell for liquid absorption spectrometry", K.-I. Tsunoda *et al.*, Applied Spectroscopy (US), Vol. 44 (1990); pages 163 to 165

A3 : "The preparation and properties of a new family of amorphous fluoropolymers: Teflon® AF", P. R. Resnick *et al.*, Polymer Preprints, Division of Polymer Chemistry, American Chemical Society, Vol. 31 (1990); pages 312 and 313

A6 : "Axial-beam on-column absorption detection for open tubular capillary liquid chromatography", X. Xi *et al.*, Analytical Chemistry (US), Vol. 62 (1990); pages 1580 to 1585

A7 : "Liquid core optical fiber total reflection cell as a colorimetric detector for flow injection

analysis", K. Fujiwara *et al.*, Analytical Chemistry (US), Vol. 57 (1985); pages 1012 to 1016
T2 : "Properties of amorphous fluoropolymers based on 2,3-bistrifluoromethyl-4,5-difluoro-1,3-dioxole", W. H. Buck *et al.*, Teflon® AF - Amorphous Fluoropolymers - Technical Information (1993) - 183rd Meeting of the Electrochemical Society, Honolulu (US), 1993; pages 1 to 11

and found that starting with document Y5 or alternatively with any of documents A6 and A7 as the closest state of the art, the subject-matter of claim 1 was obvious in view of the teaching of document Y1 and/or document A3 and of the technical information disclosed in the post-published document T2.

- II. With the statement setting out the grounds of appeal the appellant requested that the decision under appeal be set aside and that a patent be granted on the basis of the application documents upon which the contested decision was based and including a set of amended claims and amended pages 7 and 8 of the description filed with the letter dated 30.05.2003.
- III. In response to a telephone consultation with the rapporteur, the appellant submitted with its letter dated 19.12.2007 an amended set of claims 1 to 6 and amended pages 4 and 4a of the description replacing the corresponding application documents on file.
- IV. Claim 1 of the present request of the appellant reads as follows:

"An absorbance flow cell (10) for a separation column such as HPLC, capillary LC or capillary electrophoresis for containing a liquid sample and for exposing said liquid sample to light, said cell comprising

a conduit (32) having a smooth inner layer (12) provided by an amorphous fluoropolymer bonded to the conduit substrate,

said inner layer (12) having a refractive index less than the refractive index of water, and a thickness at least as great as the wavelength of light such that when said conduit is filled with water, visible light and ultra-violet light can be transmitted along the axis of said conduit by total internal reflection, substantially without loss."

Claim 4 defines a photometric analysis system including, *inter alia*, a flow cell as defined in claim 1, and claims 2-3 and 5-6 are dependent claims referring back to claims 1 and 4, respectively.

V. The arguments submitted by the appellant in support of its requests can be summarised as follows:

The invention is directed to an absorbance flow cell of the liquid-core waveguide type. This type of cell confines the light to the liquid sample by total internal reflection at the boundary between the liquid and the capillary inner wall. This requires that the refractive index of the liquid is above that of the tube material. Tube materials are typically fused silica and conventional fluoropolymer (Teflon) materials having a refractive index higher than that of water, so that the liquids are limited to high-index

organic liquids or to solutions containing added solutes that elevate the refractive index. In addition, at shorter UV wavelengths the transparency is poor and UV measurements down to 200 nm and below are not possible. The claimed flow cell solves all these problems.

Document Y5 discloses waveguide capillary cells comprising a FEP tubing and operating by total internal reflection at the inner capillary wall. The document teaches that the use of FEP instead of borosilicate and quartz glasses and having a lower refractive index renders possible the application to more solvents systems and to measurements in the UV region. However, the efficiency of light transmission decreases with wavelengths below 350 nm down to 285 nm (Figures 2 and 3), and - contrary to the case of the claimed flow cells - measurements below 200 nm will not be possible. In addition, light would not be efficiently confined if aqueous solvents or solutes in water are used. The flow cells described in document Y5 are solid hollow tubes of FEP and there is not the slightest hint in the document that a flow cell could be provided by a cladding as claimed.

The disclosure of document Y1 only refers to unusually low refractive indices (abstract and column 3, lines 48 to 64), and the post-published document T2 merely states that "it appears possible to make PDD copolymers with refractive indices between 1.37 and 1.29" (page 9, third paragraph together with Figure 15). In addition, a comparison of the glass transition temperatures given in documents Y1 (abstract and claim 1) and T2 (Figure 15) shows that the refractive index of the PDD-

CTFE copolymers having a glass transition temperature of 140° C is significantly higher than that of water. Thus, the examining division's conclusion that document Y1 implicitly discloses refractive indices lower than that of water is wrong. The same applies to document A3 the disclosure of which is not limited to copolymers of PDD with TFE but rather refers to Teflon® AF *per se* (page 312, first and second paragraphs) which also includes PDD-CTFE copolymers with glass transition temperatures of 140° C and below and refractive indices significantly above that of water. In addition, none of these documents teach the behaviour of the refractive index of the copolymers in the UV region or that the copolymers would exhibit the good sample compatibility required for the application under consideration.

The fact that tubes of Teflon® AF or other suitable copolymers of PDD apparently were not freely available on the market does not create an obstacle to fabricating such tubes in view of the fabrication methods referred to in document A3 (page 312, first column, fifth paragraph). In addition, it was unknown at the priority date to construct a liquid-core waveguide in such a way that the material giving the waveguide its optical function was provided by a coating.

In addition, providing the amorphous fluoropolymer in the form of an inner layer on a supporting conduit as claimed and not in the form of a conduit leads to technical advantages over the flow cells of the prior art. Firstly, amorphous fluoropolymers, and in particular Teflon AF, are very brittle materials, and according to the invention the mechanical rigidity of

the flow cell is not provided by a cylindrical solid tube made of amorphous fluoropolymer having a certain minimum outer diameter guaranteeing the necessary mechanical rigidity, but by a supporting conduit, the desired optical properties being then provided by the inner layer of amorphous fluoropolymer. This approach also leads to substantial cost savings because most amorphous fluoropolymers are expensive materials and the claimed flow cell only requires a thin layer of the fluoropolymer. Secondly, the provision of a layer of the amorphous fluoropolymer on the conduit provides for a large design flexibility because the design restrictions imposed by the mechanical properties of the fluoropolymer and required in the case of a solid tubing made of the fluoropolymer are not given any longer.

Therefore, the approach followed by the examining division goes beyond that which one would have expected of the skilled person, and the examining division's view that the claimed invention is obvious from a combination of document Y5 and document Y1 or A3 is nothing but an *ex-post-facto* analysis and a combination of these documents could not have prompted the skilled person to the claimed invention. Similar considerations apply when starting with any of documents A6 or A7 as closest state of the art.

Reasons for the Decision

1. The appeal is admissible.

2. *Amendments*

The Board is satisfied that the application documents amended according to the present request of the appellant satisfy the formal requirements of the EPC. In particular,

- claim 1 is based on claim 1 and on the passage on page 6, lines 10 to 12 of the description as originally filed (Article 123(2) EPC), and also based on claim 1 and the passages on page 5, lines 2 to 6 and page 6, lines 10 to 12 of the earlier European patent application (92112114.1) as originally filed (Article 76(1) EPC), and claims 2 to 6 correspond with claims 2 to 6 as originally filed (Article 123(2) EPC) and with claims 2 to 6 of the earlier application as originally filed (Article 76(1) EPC), and
- the description has been brought into conformity with the invention as defined in present claim 1 (Article 84, second sentence and Rule 42 (1) (c) EPC).

3. *The prior art*

- 3.1 Document Y5 discloses a capillary cell for liquid absorption spectrometry constituted by a hollow capillary tubing filled with a solvent (Figure 1 and page 163, first paragraph). According to the document, the operation of the capillary cell requires that the refractive index of the solvent is higher than that of the tubing material so that the cell operates as a liquid-core optical waveguide along which light can be transmitted by total internal reflection at the boundary between the liquid and the capillary inner

wall. When the tubing is made of borosilicate glass or quartz, however, only few organic solvents can be used, thus limiting the applicability of the method (page 163, first paragraph and Table I), and the document proposes extending the applicability of the method to more solvents by using a tubing made of poly(tetrafluoroethylene-co-hexafluoropropylene) (FEP) having a refractive index of 1.338 which is lower than that of borosilicate and quartz glass. According to the document, the FEP tubing renders possible the use of mixed solutions of water and ethanol and of aqueous solutions with high salt concentration and also improves the operation of the cell in the UV region (page 163, second paragraph, and page 165, last paragraph together with Figures 2 and 3).

3.2 Document Y1 discloses amorphous perfluoropolymers having a low refractive index and suitable for cladding optical fibres (abstract together with column 1, lines 15 to 40 and column 3, lines 13 to 62). The document refers to pipe, tubing and fittings made from or lined with the fluoropolymers, to films and coatings deposited by solvent casting, and to optical fibre claddings (column 3, lines 59 and 60, column 4, line 60 *et seq.*, and column 7, lines 26 to 37).

3.3 Document T2 is a document published in 1993 and therefore published after the date (15.07.1992) on which the present application is deemed to have been filed pursuant to Article 76(1) EPC. The document reports on the properties of amorphous fluoropolymers. According to the document, the refractive index of copolymers of 2,2-bistrifluoromethyl-4,5-difluoro-1,3-dioxole (PDD) and tetrafluoroethylene (TFE) sold under

the trademark Teflon® AF (first page, penultimate paragraph) can be adjusted between 1.325 and 1.291 depending on the molar ratio of PDD to TFE in the copolymer (Figure 14 on page 9 and the second and third paragraphs on the same page); more particularly, Teflon® AF 1600 and AF 2400 would have a refractive index of about 1.305 and 1.291, respectively (page 9, second paragraph).

3.4 Document A3 reports on the preparation and properties of the family of amorphous fluoropolymers Teflon® AF. According to the document, these fluoropolymers have high optical clarity (first paragraph) and a refractive index between about 1.29 and 1.31 (table on page 312 and the second of the graphs in the first column on page 313), and Teflon® AF 1600 has a high light transmission in the visible and UV spectrum above about 200 nm (first graph in the second column of page 313). The document also refers to processing techniques that are applicable to the polymers such as solution casting into micron-thin films, melt processing into a variety of forms, extrusion and injection or compression moulding, and spin casting and solution or spray coating into clear pin hole-free films less than one micron thick (first column, first and penultimate paragraphs).

3.5 Document A6 discloses optical waveguide capillary columns for absorption detection in tubular capillary liquid chromatography (abstract and Figure 1). The capillary columns are constituted by a hollow glass fibre of fused silica having a refractive index of 1.458 filled with a liquid containing pyridine and dimethyl sulfoxide having a higher refractive index

(1.477), light being transmitted along the column by total internal reflection (page 1583, first column, last two paragraphs). The document also refers explicitly to document Y5 and to the FEP tubing disclosed in the document (page 1584, second column, third paragraph).

- 3.6 Document A7 discloses an absorption colorimetric capillary cell constituted by a hollow fibre filled with a liquid and operating by total internal reflection of light inside the capillary (abstract and Figure 2). More particularly, the document discloses a capillary cell used for capillary gas chromatography and constituted by a hollow fibre made of Pyrex having a refractive index of 1.474 and filled with carbon disulfide (page 1013, first column, section "Experimental section").

4. *Inventive step*

- 4.1 The Board concurs with the examining division in considering the absorbance flow cell disclosed in document Y5 and referred to in point 3.1 above as the closest prior art.

- 4.1.1 The subject-matter of claim 1 differs from the absorbance flow cell disclosed in document Y5 (see point 3.1 above) in that the tubing is constituted by a conduit having a smooth inner layer provided by an amorphous fluoropolymer bonded to the conduit substrate, the inner layer having a refractive index less than the refractive index of water and a thickness at least as great as the wavelength of light such that, when the conduit is filled with water, visible light and ultra-

violet light can be transmitted along the conduit by total internal reflection substantially without loss.

4.1.2 According to the reasoning of the examining division in its decision, in view of the teaching of document Y5 that the lower the refractive index of the cell tubing, the greater the classes of solvents available for the capillary cell (point 3.1 above), the skilled person would have seen in the disclosure of document Y5 a clear teaching towards the use of tubing materials having a refractive index as low as possible, and in particular lower than that of FEP considered in the document, as such tubing materials would then further extend the applicability of the method to even more solvent systems having the required characteristics. In addition, in view of the developments in optical materials reported in document Y1 disclosing amorphous fluoropolymers (point 3.2 above) having an "unusually low refractive index" (column 3, line 62) which, according to the examining division, are below that of water (~ 1.33) as may be inferred from Figure 14 of the post-published document T2 (point 3.3 above), and also in view of document A3 (point 3.4 above) disclosing the family of amorphous fluoropolymers Teflon® AF having a refractive index between 1.29 and 1.31 (table and second paragraph on page 313) and referring more particularly to Teflon AF 1600 and 2400 (page 312, lines 24 and 25) also used in specific embodiments of the claimed invention (page 6 of the application, last paragraph), the examining division concluded that at the time of the priority date of the application it would have been obvious for the skilled person to use in the capillary cell of document Y5 these new materials having a refractive index lower

than the refractive index of water. Furthermore, according to the examining division, the latter characteristic would manifestly and straightforwardly have rendered possible the use of liquids within the tubing composed of aqueous solutions as aimed at by the claimed invention (page 1, first paragraph, page 2, second paragraph, and page 4, last paragraph of the application) and, in addition, the resulting cell would have exhibited, as an inevitable bonus effect, an improved transparency at shorter UV wavelengths as also aimed at by the claimed invention (page 3, second paragraph, and page 5, last paragraph), thus rendering possible the transmission of both visible and ultra-violet light substantially without loss as claimed.

- 4.1.3 Nonetheless, even if - contrary to the appellant's contention - the aforementioned line of argument of the examining division were to be considered persuasive, the approach relied upon by the examining division would then have led to a tubing made of an amorphous fluoropolymer having a refractive index lower than that of water, and not to a conduit having a smooth inner layer provided by the amorphous fluoropolymer bonded to the conduit substrate and having a thickness at least as great as the wavelength of light as required by the claimed subject-matter.

As regards this distinguishing feature of the claimed invention, the examining division further held in its decision that the provision of the amorphous fluoropolymer as an inner layer bonded to a conduit constitutes an obvious alternative to the provision of the amorphous fluoropolymer in the form of a tubing and in fact the only technically feasible possibility in

view of, first, the fact that at the priority date of the application the amorphous fluoropolymers were "both very expensive and unavailable in the form of tubing" as acknowledged in the application (page 5, lines 10 and 11) and, second, in view of the disclosures of documents Y1 and A3 relating to the use of the amorphous fluoropolymers as optical fibre claddings and as pipe and tubing linings and to the different methods referred to in the documents for forming coatings and micron-thin films made of the fluoropolymers (points 3.2 and 3.4 above).

The Board, however, cannot endorse the latter line of argument of the examining division for the following reasons.

According to the application (page 5, second paragraph) and as persuasively argued by the appellant (fourth and fifth paragraphs of point V above), the provision of the cell as a conduit having a smooth inner layer of the amorphous fluoropolymer improves the design flexibility and minimizes the material costs of the cell. However, none of the documents considered by the examining division addresses or even mentions these technical aspects. In addition, all the flow cell conduits disclosed in the documents considered by the examining division and relying on the confinement of light by total internal reflection at the inner wall of the conduit are constituted by a tubing made of the optical material having a refractive index lower than that of the fluid within the tubing. Thus, in the absence of any teaching or suggestion in the available prior art departing from this standard procedure, the skilled person - assuming that he would have followed

the approach indicated by the examining division and summarised in point 4.1.2 above - would then have considered the provision of the amorphous fluoropolymer in the form of a conduit or tubing.

As regards the disclosures of documents Y1 and A3 relating to tubing linings, coatings, claddings and micron-thin films made of the amorphous fluoropolymers and referred to by the examining division, these disclosures may, at the most, provide evidence that it was technically possible at the priority date of the application to manufacture tubings having a smooth inner layer of the fluoropolymers as claimed, but the documents contain no specific disclosure or teaching as to the actual manufacture of such innerly coated tubings, let alone as to the provision of coatings in the inner surface of conduits of flow cells of the type at issue. Therefore, the disclosure of these documents is in the Board's view insufficient to suggest to the skilled person the provision of conduits having an inner layer as claimed. On the contrary, document Y1 also refers to "pipe, tubing and fittings" and to "other useful fluid handling articles" such as "pump housings, [...], tanks, trays, pipettes, laboratory vessels" made from the fluoropolymers (column 4, line 60 *et seq.*), and document A3 also discloses that the fluoropolymers can be "melt processed into a variety of forms" and "injection or compression molded" (first and penultimate paragraphs on page 312); the skilled person would have therefore seen in these documents a clear disclosure of techniques that would enable the effective manufacture of tubings made of the fluoropolymers, and there is *a priori* no reason for the skilled person to consider alternative approaches. It

is also noted in this respect that according to the application as filed the manufacture of tubular conduits having an inner layer of the amorphous fluoropolymer as claimed requires specific manufacturing techniques such as solution tube filling followed by solvent-evaporation and baking coating (page 7, lines 1 to 8) or - as it is claimed in the patent granted on the earlier application - encapsulation coating on a soluble tube (paragraph bridging pages 7 and 8) that are not addressed in the aforementioned prior art.

Accordingly, in the absence in the prior art of any teaching or suggestion towards the provision of the low refractive optical material of a liquid-core optical waveguide of the type disclosed in document Y5 as an inner layer on a conduit and of the technical advantages associated with this approach when the optical material is of an amorphous fluoropolymer as claimed, the Board cannot accept the lengthy chain of reasoning steps followed by the examining division in support of its view that the skilled person would have arrived in an obvious way at a flow cell as claimed when starting with the flow cell disclosed in document Y5.

4.1.4 In view of the above, the Board concludes that the subject-matter of claim 1 is not rendered obvious by the disclosures of documents Y1 and A3 and the technical information shown in T2 when starting with document Y5 as the closest state of the art.

4.2 The examining division held in its decision that each of documents A6 and A7 can also be alternatively

considered as the closest state of the art and that the claimed invention would also result therefrom in an obvious way in view of documents Y1, A3 and T2. However, each of documents A6 and A7 is directed to a flow cell of the type disclosed in document Y5 (see points 3.5 and 3.6 above), and none of them goes beyond the disclosure of document Y5 (point 3.1 above). It follows that starting with any of documents A6 and A7 as the closest prior art would not lead to a conclusion different to that drawn in point 4.1.4 above and based on document Y5 as the closest state of the art.

The Board is also satisfied that none of the remaining documents on file, either taken alone or in combination with the documents already considered in the decision, would render obvious the claimed subject-matter.

- 4.3 In view of the above considerations and conclusions, the Board is of the opinion that the subject-matter of claim 1 involves an inventive step within the meaning of Article 56 EPC with regard to the prior art on file.
- 4.4 Claims 2 and 3 are directed to a flow cell comprising all the features of the flow cell defined in claim 1, and claims 4 to 6 are directed to a photometric analysis system comprising a flow cell as defined in claim 1. Consequently, the conclusion reached in point 4.3 above with regard to claim 1 also applies to claims 2 to 6 (Articles 52(1) and 56 EPC).
5. The Board is also satisfied that the application documents amended according to the appellant's request and the invention to which they relate meet the

remaining requirements of the EPC within the meaning of Article 97(2) EPC.

In these circumstances, the Board concludes that the decision under appeal is to be set aside and a patent be granted on the basis of the application documents amended according to the present request of the appellant (Articles 97(2) and 111(1) EPC).

Order

For these reasons it is decided that:

1. The decision under appeal is set aside.
2. The case is remitted to the department of first instance with the order to grant a patent in the following version:
 - description pages 1 to 3, 5, 6 and 9 as originally filed, pages 7 and 8 filed with the letter dated 30.05.2003, and pages 4 and 4a filed with the letter dated 19.12.2007,
 - claims 1 to 6 filed with the letter dated 19.12.2007, and
 - drawing sheet 1/1 as originally filed.

The Registrar:

The Chairman:

M. Kiehl

A. G. Klein