BESCHWERDEKAMMERN DES EUROPÄISCHEN PATENTAMTS

BOARDS OF APPEAL OF THE EUROPEAN PATENT OFFICE

CHAMBRES DE RECOURS DE L'OFFICE EUROPEEN DES BREVETS

A		B	X	C	

File Number: T 677/91 - 3.4.1

Application No.: 83 307 458.6

Publication No.: 0 113 207

Title of invention: Method of mass analyzing a sample by use of a quadrupole ion trap

Classification: H01J 49/42

D E C I S I O N
of 3 November 1992

Proprietor of the patent: Finnigan Corporation

Opponent: Bruker-Franzen Analytik GmbH

Headword: Mass selective ejection/FINNIGAN

EPC Articles 54 and 56

Keyword: "Novelty (confirmed)" - "no clear and unmistakable disclosure of a claimed feature" - "Inventive step (confirmed)"

Catchwords

Not sufficient for a finding of lack of novelty that the claimed features could have been derived from a prior document - necessity for clear and unmistakable teaching (T 204/83 (OJ EPO 1985, 310) and T 56/87 (OJ EPO 1990, 18) followed) (paragraph 1.2).
In assessing inventive step, evidence of the practical impact of the claimed invention considered (paragraph 3.3).
No different standard of inventiveness between opposition proceedings and national courts - no benefit of doubt for the patentee (paragraph 3.4).

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Beschwerdekammern | Boards of Appeal | Chambres de recours

Case Number : T 677/91 - 3.4.1

D E C I S I O N
of the Technical Board of Appeal 3.4.1
of 3 November 1992

Appellant : (Opponent)
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Decision under appeal :
Decision of the Opposition Division of the European Patent Office dated 9 July 1991 rejecting the opposition filed against European patent No. 0 113 207 pursuant to Article 102(2) EPC.

Composition of the Board :

Chairman : G.D. Paterson
Members : Y. van Henden
R.K. Shukla

Summary of Facts and Submissions

I. The Respondent is proprietor of European patent No. 0 113 207 (application No. 83 307 458.6).

The claimed invention is concerned with a method of mass analysing a sample in which:

(a) ions over an entire mass range of interest are trapped in a three-dimensional quadrupole field;

(b) the three-dimensional quadrupole field is changed so that trapped ions of consecutive specific masses become sequentially unstable and leave the trapping field; and

(c) the ions leaving the trapping field are sensed to provide signals indicative of their masses.

This technique can be referred to as "successive mass-selective ejection", in contrast to the previously known methods of mass analysis using quadrupole ion traps. In this technique the trap is operated non-mass selectively, whereas in previous methods of mass analysis the trap has been operated mass selectively, that is, only ions within a specific narrow range of charge to mass ratios are trapped.

Claim 1 of the patent as granted reads:

"A method of mass analyzing a sample by use of a quadrupole ion trap, comprising defining a three-dimensional quadrupole field in the trap in which ions of interest can be trapped, introducing sample ions into or creating sample ions in the quadrupole field whereby ions of interest are trapped, and sensing the trapped ions to provide an output signal indicative of the trapped ion

mass, characterised by the steps of defining the three-dimensional quadrupole field such that ions over an entire mass range of interest can be simultaneously trapped, trapping ions within the entire mass range of interest, and changing the three-dimensional quadrupole field such that trapped ions of consecutive specific masses become sequentially unstable and leave the trapping field for sensing to provide output signals indicative of the ion masses."

Dependent Claims 2 to 10 are appended to Claim 1.

II. The patent was opposed by the Appellant on the grounds mentioned in Article 100(a) EPC, referring to the prior art which can be derived from documents

D1: US-A-2 939 952 (Paul)
D5: Physical Review Letters, Vol. 20, No. 2, (1968), pages 39 to 41
D6: J. Vac. Sci. Technol., Vol. 11, No. 2 (1974), pages 515 to 518
D7: P.H. Dawson, "Radiofrequency Quadrupole Mass Spectrometers", in "Applied Charged Particle Optics", Academic Press 1980, edited by A. Septier, Part B, pages 173 and 234 to 256.

Lack of novelty and lack of inventive step was alleged on the basis of each of the above documents.

III. The Opposition Division rejected the opposition.

IV. The Opponent lodged an appeal against the decision of the Opposition Division, requesting said decision to be set aside and the patent to be revoked.

In the Statement of grounds of appeal, reference was made to documents D1, D5 and D7, as well as to document

D3: US-A-3 829 689,

which had been cited in the European Search Report.

Each of these documents was said to destroy the novelty of Claim 1, or to make it obvious, so that the subject-matter of the attacked patent at least lacked inventive step.

V. With his letter of 13 March 1992 replying to the grounds of appeal, the Respondent requested that the appeal be rejected. Subsidiarily, he requested that the patent be maintained on the basis of an amended Claim 1, submitted as an auxiliary request before the Opposition Division. Both sides requested oral proceedings.

VI. In a communication dated 11 September 1992 pursuant to Article 11(2) RPBA annexed to the summons to oral proceedings, reference was made to documents D1, D5, D6, D7 and

D4: US-A-3 527 939 (Dawson)

the latter document being a further citation in the European Search Report. In this communication the provisional view was expressed that the claimed invention was novel but lacked inventive step, in view of documents D4 and D7.

VII. In a letter dated 1 October 1992 replying to this communication the Respondent contested the suggestion of lack of inventive step, primarily on the basis that the ion gauge described in D4 is operated by ejecting all

trapped ions simultaneously from the trap, and no attempt is made to perform mass analysis using successive mass selective ejection. The passage in D7 at page 240, final paragraph, cannot be regarded as suggesting performing mass analysis using successive mass selective ejection, because the author of D7 considers possible mass analysis methods on the pages preceding page 240.

With this letter the Respondent enclosed extracts from a recent textbook:

(B): R.E. March and R.J. Hughes: "Quadrupole Storage Mass Spectrometry" (1989), pages 30, 321, 323, 329, 332, 414,

in support of the inventiveness of the claimed invention.

VIII. In a letter dated 2 September 1992, the Appellant drew attention to the following additional documents

D8: A. Frigerio, "Essential Aspects of Mass Spectrometry", Spectrum Publications Inc., New York, Toronto, London, Sydney, page 1;

D9: M. von Ardenne et al., "Elektronenanlagerungs-Massenspektrographie organischer Substanzen", Springer-Verlag, Berlin, Heidelberg, New York, 1971, page 1;

D10: F. Aulinger et al., "Massenspektrometrie", Verlag Chemie, Weinheim, 1968, page 71.

IX. With telefax dated 20 October 1992, the Respondent filed document

D11: J.E. Fulford et al., "Radiofrequency mass selective excitation and resonant ejection of ions in a three-dimensional quadrupole ion trap", J. Vac. Sci.

Technol., Vol. 17, No. 4 (July/August 1980), pages 829-835

and submitted evidence by way of affidavits of Professor J.F.J. Todd, A.H. Smith, and C.S. Campbell.

With telefax of 23 October 1992, the Respondent filed a further affidavit by Professor R.E. March, the above identified pages from the textbook B being exhibited as Exhibit B.

X. Oral proceedings were held on 3 November 1992, during which statements were made by Professor Wanizek and Dr. Franzen on behalf of the Appellant, in reply to the evidence filed by the Respondent in October 1992, and by Professor Todd and Mr Stafford on behalf of the Respondent.

At the oral hearing, the Appellant relied in particular upon documents D1 and D5 as destroying the novelty of Claim 1 of both the main request and the auxiliary request, and upon combinations of documents D4 and D1, D4 and D7, D5 and D1, D5 and D7, or D1 and D7 as establishing lack of inventive step. A passage in document D7 at the bottom of page 240 was strongly relied upon as suggesting the selective ejection of trapped ions of consecutive masses.

XI. In support of his request, the Appellant substantially argued as follows, in writing and orally:

Novelty - D1

By performing, in the case of a three-dimensional field, a calculation similar to that disclosed in (D1) in relation to a two-dimensional field, it can be established that points representing the trapped ions in the Mathieu

diagram are distributed on the straight line ($a/q = 2U/V$), in accordance with the related values of the ratio (e/m). The skilled person knows that, in the absence of DC bias component, ion stability is achieved for all masses greater than a certain value and that, when the DC component increases, the mass range of stable ions becomes narrower. When ions are introduced into the trap known from (D1) and when the DC voltage has a given value (U_o), only ions comprised within a corresponding mass range of interest are trapped. Likewise, it is known from (D1) to detect successively or quantitatively measure individual components of an ionised gas mixture by correspondingly displacing the ranges of stability and instability. Furthermore, it is made clear there that the only way of achieving this purpose is to vary DC and AC voltage components simultaneously, and proportionally. If, as argued by the Respondent, the teachings in column 7 of (D1), lines 59 to 61, could equally well be taken to be an instruction to operate the trap in the usual successive mass selective fashion, this is indicative that the skilled person might also understand said passage of (D1) as the Appellant does. Finally, since (D1) relates to a mass analysis method, it is obvious that means for detecting ions must be provided in order to carry out the method and that said means output the signal representative of ion masses. Similar teachings can be derived from (D3), where it is explained that not only the position but also the width of the stable range can be influenced.

Novelty - D5

As regards the disclosure in (D5), the specific masses of two ion species define a mass range. Logically, the quadrupole field has to be progressively varied in order that the trapped ions become sequentially unstable and

leave the trap. This is actually confirmed by Figure 2, which shows that the trap drive voltage is controlled by the timer. Likewise, it is obvious that the output signal is indicative of ion masses. The measures proposed in D5 are suitable for mass analysis, so that (D5) destroys novelty of the claimed subject-matter.

Novelty - D7

Document (D7) does not explicitly state that the field should be progressively varied. Nevertheless, it teaches that selective ejection of chosen ions can be achieved by judiciously altering the operating conditions of the trap, i.e. not suddenly altering such conditions so as to cause simultaneous ejection. Furthermore, selective ejection means there sequential ejection, for any other solution would be economically unacceptable.

Inventive step

Additionally, the subject-matter of Claim 1 lacks an inventive step having regard to the combined teachings of the cited documents (as set out above). Any necessary step from the disclosures of the cited documents to the claimed invention was fully within the normal application of the general knowledge of the skilled person.

XII. The Respondent's argumentation may be summarised as follows:

Figures 11 and 12 of (D1) do not show any opening through which ejected ions could pass for detection externally of the trap. Further, means for detecting ejected ions are not provided: changing the quadrupole electric field such that trapped ions of consecutive specific masses become sequentially unstable and leave the trapping field is

disclosed in (D1) only in relation with the use of a trap as a gauge for measuring partial pressures in highly rarified gas mixtures.

Document (D3) is directed to an improvement over the two-dimensional quadrupole field arrangements disclosed in (D1), which arrangements can only operate as a filter and do not constitute ion traps, since a three-dimensional quadrupole field is required for trapping.

Document (D4) describes two applications of a quadrupole ion trap, namely as an ion gauge and as a mass spectrometer. In relation with this latter application, however, (D4) only contemplates the mass selective mode. The apparatus includes a pulse-out circuit (19) which ejects simultaneously all ions contained in the trap. Accordingly, it is necessary to go through repeated cycles of trapping ions of a selected (e/m) ratio and then ejecting the ions. It is only when operating the trap as an ion gauge that (D4) contemplates non-mass selective operation, but no attempt to use it for performing mass analysis is made.

Document (D5) is only concerned with trapping and extracting the two particular ions H_2 and H^+, of which the masses are already known and, anyway, do not constitute a mass range of interest within the meaning of the patent in dispute. No mass determination and analysis being performed, (D5) cannot be used as the basis for a challenge to the novelty of Claim 1. Further, nothing is said there on how the trapped ions are extracted from the trap. As a matter of fact, considering that (D5) was published in January 1968, one cannot even speculate how its author might have done this.

As explained in the affidavit of Professor J.F.J. Todd, an expert in the field of mass spectrometry, it is clear that the author of document (D7) only contemplated pulsed or sudden ejection of the contents of a trap. Moreover, the mention of chosen ions in document (D7) implies in fact that ion masses are already known. It is indeed explained in the passages of (D7) on which the Appellant relied that, in the non-mass selective mode of operation, the trap must be used as a part of systems comprising conventional mass analysis means, since all ions will be ejected at the same time. Nevertheless, while summing up the state of the art, the author of (D7) acknowledges that the aesthetic satisfaction given by such systems is dulled by the complexities of the interactions inherent therein. There is consequently no doubt that, if the author of (D7) had the Finnigan technique employed in the present invention in mind when he drafted his paper, he would have referred to it explicitly.

Therefore, the exercise of inventive ingenuity was required to envisage, at the priority date of the patent in suit, to change the three-dimensional quadrupole field of an ion trap such that trapped ions of consecutive specific masses become sequentially unstable and leave the trapping field.

XIII. At the end of the hearing, the decision was announced that the appeal is dismissed.

Reasons for the Decision

1. Novelty

1.1 Document (D1)

This relates to methods of separating or separately detecting ions of different specific charges and to arrangement therefor. Said arrangements comprise means for producing quadrupole fields, which fields are three-dimensional only in the device described in relation to Figures 11 and 12. Nevertheless, it is stated in the related part of the description that a gas to be tested is introduced into said device at a low pressure - see column 6, lines 7 to 9 - whereas low pressures are not mentioned in relation to the other arrangements disclosed in (D1)., which arrangements are mass spectrometers working with two-dimensional quadrupole fields. Having regard also to the wording of Claim 13, the Board agrees with the Respondent that the device according to Figures 11 and 12 of (D1) is a gauge for measuring partial pressures in highly rarified gas mixtures. The trap shown in these Figures is not provided with a hole for ion ejection. Therefore, despite the statement in (D1) that "ions of the stable e/m range remain stable between the electrodes (of the device according to Figures 11 and 12) when a potential U_o + V sin ωt is applied between the (ring) electrode A and the pair of (end cap) electrodes B" - cf. column 6, lines 17 to 19 - and that "separation of ions of different specific charges may be effected continuously with the arrangements of the invention" - cf. column 6, lines 47 to 52 - in the Board's view this document does not disclose to a skilled person working in the field of mass analysis the idea of using a trap in which a three-dimensional field is defined, and of

changing said field such that trapped ions of consecutive specific masses become sequentially unstable and leave the trapping field for sensing.

1.2 Document (D5)

Document (D5) deals with H_2 and H^+ ion production inside a trap under the action of electron bombardment and photo-dissociation. Figure 2 shows that the trap comprises a ring electrode and two end caps facing one another. The characteristics of the trap allow simultaneous trapping as well as sequential extraction of H^+ ions produced by photo-dissociation and remaining H_2 ions, whereupon ejected ions are sensed by a particle multiplier which provides an output signal - see page 40, second paragraph of the first column and Figure 2.

It was disputed between the parties whether (D5) could be said to disclose "a method of mass analysing a sample"; the Appellant relied on the passage on page 39 of (D5) at the bottom of the left-hand column. Furthermore, with reference to the feature of Claim 1 that the three-dimensional quadrupole field is changed such that trapped ions of consecutive specific masses become sequentially unstable and leave the trapping field for sensing to provide output signals indicative of the ion masses, the Appellant relied upon the passage in the left-hand column of page 40 of (D5), concerning "sequential extraction" of H^+ and H_2 ions, when interpreted at the priority date of the attached patent (1982). The Respondent, however, contended that (D5) had to be interpreted at its publication date of 1968 when considering novelty, and denied that (D5) disclosed either changing the quadrupole field to cause sequential ejection of ions, or sensing the ejected ions to provide output signals indicative of the ion masses.

In the Board's judgment, when considering whether a prior document discloses the technical features of a claim, the disclosure of a prior document must be determined at the date of publication of that document. The disclosure of a document does not change from time to time. The technical content of a document is what is disclosed to a skilled person at the time when it was written and published. In contrast, whether the technical features of a claim are obvious in view of a prior document of course has to be determined at the filing date of such claimed subject-matter.

With reference to (D5), it was put forward at the oral hearing by Professor Todd on behalf of the Respondent that in 1968, the publication date of (D5), a skilled person would have been likely to assume that the sequential ion extraction described in (D5) was achieved by a resonant ejection technique, in which a specific resonant frequency is applied to the end cap electrodes. The Board considers this to be plausible, and that there is in any event no clear and unequivocal disclosure in (D5) of the claimed feature of changing the quadrupole field to cause sequential ion ejection: such clear and unequivocal disclosure being a prerequisite for a finding of lack of novelty. It is not sufficient for a finding of lack of novelty of claimed features that such features could have been derived from a prior document. There must have been a clear and unmistakable teaching of the claimed features - see for example Decisions T 204/83 (OJ EPO 1985, 310) and T 56/87 (OJ EPO 1990, 188).

2.3 Document (D6)

Document (D6) teaches that, in a three-dimensional quadrupole mass spectrometer, mass-selected ions can be pulsed out of the trap into an electron multiplier by applying a short negative pulse to the cap electrode adjacent to said electron multiplier - see page 518, second paragraph of the first column. As an alternative drawout method, (D6) only proposes to suddenly reduce the magnitude of the AC voltage while maintaining the DC voltage on the end caps - see the same column, last paragraph.

Therefore, in the Board's judgment, (D6) also fails to disclose changing the three-dimensional quadrupole field of an ion trap such that trapped ions of consecutive specific masses become sequentially unstable, and therefore does not destroy the novelty of the claimed invention.

2.4 Document (D7)

In the section of document (D7) headed "Ion ejection", it is stated that ions trapped in a three-dimensional quadrupole field ion trap can be ejected "either by suddenly changing the operating point to a position outside the stability diagram, or by applying a mean voltage gradient in the Z (axial) direction" see the last but one paragraph of page 238. For changing the operating point, three methods are proposed, namely applying a DC voltage pulse to one of the end caps, reducing the RF voltage amplitude or changing simultaneously both the DC and RF voltages so as to change the parameters (a) and (q). As pointed out in Professor Todd's affidavit, however, a voltage gradient in the axial direction is not a quadrupole field, and does not form part of the trapping

field. This is also the case for the field variation produced when a voltage pulse is applied to one of the end caps. As regards a sudden decrease in RF voltage amplitude, the author refers to document (D6), where it is stated that the effect of such a change is to move out of the stable region a particle near the tip of said region. This, however, makes clear that the storage is mass selective and that a pulse-out technique is used. For the same reasons, this latter conclusion also applies to the third method.

Document (D7) furthermore discloses at the bottom of page 240 that, after ion accumulation, "there is a possibility of judiciously altering the operating conditions (of the trap) so as to selectively eject chosen ions, although practical application seems to be difficult". The Board agrees with Professor Todd that one cannot infer from (D7) how said conditions should be altered. Furthermore, the immediately following mention of the use of the ion trap as a specific ion reactor by adjusting the DC voltage is an indication that a mass selective storage is achieved there.

For the above reasons, in the Board's view document (D7) does not disclose the idea of changing the three-dimensional quadrupole field of an ion trap such that trapped ions of consecutive masses become sequentially unstable; and consequently this document does not destroy the novelty of the claimed invention.

2.5 In the Board's judgment, therefore, the subject-matter of Claim 1 as granted to the Respondent is novel in respect of the prior art on which the Appellant relied.

3. Inventive step

3.1 Document (D3) discloses a system comprising a quadrupole mass spectrometer working with a two-dimensional quadrupole field. As the Respondent pointed out, however, and as actually confirmed in (D3) - see column 2, lines 16 to 26, and from line 61 of column 2 to line 18 of column 4 - such a field can only operate as a filter. The Board thus cannot share the Appellant's view that teachings similar to those of (D1) - or any step of the method according to Claim 1 of the patent in suit - could be derived from (D3).

Document (D4) discloses the use, in mass spectrometers and low pressure gauges, of quadrupole ion traps with means for creating a three-dimensional quadrupole field in which ions over an entire mass range of interest can be simultaneously trapped. In relation to the operation of a mass spectrometer, however, said document teaches that stored ions can be swept out of the trapping region by "applying a pulse to the electric fields between the electrodes" - see column 4, lines 5 and 6. Furthermore, no other way of ejecting ions is mentioned in (D4).

3.2 As is clear from the above discussion of these documents with reference to "novelty", documents (D1) and (D5) do not disclose any method of ejecting ions from a trapping field, whereas documents (D6) and (D7) only refer to methods based on a rapid change of the field intensity and/or configuration. A combination of teachings given by these documents thus cannot lead to the present invention. As a matter of fact, to arrive at the latter, the skilled person has to propose a novel method, and this without knowing in advance whether said novel method works.

3.3 It was strongly and persuasively argued by the Appellant that, at the priority date, a skilled person who was aware of documents (D5) and (D7) (or (D1) and (D7)) would know from such documents and his own knowledge how to change the three-dimensional quadrupole field of an ion trap such that trapped ions of consecutive masses become sequentially unstable, and that he would know that such a method would work. Such a skilled person did not need any more than what such documents disclosed in order to carry out the claimed invention without any inventive activity.

However, the Respondent relied strongly on the fact that the claimed invention achieved wide-spread recognition and acclaim within interested circles following its publication in 1983, and that the claimed invention had provided the basis for the manufacture by the Respondent of commercially successful "ion trap products" since then.

The Appellant responded to this argument by contending that any commercial success was not due to the claimed invention, but resulted from other developments incorporated in the Respondent's products.

The Board is not in a position to determine the exact extent to which the evidence of commercial success can be directly attributed to the claimed invention. However, it is satisfied that, when assessing the patentability of the claimed invention in the present case, it would be wrong to ignore the practical impact that the invention has made in its own field since the priority date - see for example the passages relied upon by the Respondent in the textbook "Quadrupole Storage Mass Spectrometry" by R.E. March and R.J. Hughes, published in 1989 (document (B), identified in paragraph VII above), where reference is made to the various advantages of the "successive mass selective

ejection through instability" techniques of the claimed invention bringing in a new era in mass spectrometry. It is difficult to reconcile the contents of such passages with the idea that the claimed invention was in fact a matter of mere routine development for a skilled person at the priority date.

The relevant disclosures, especially in documents (D1), (D5) and (D7), clearly come technically close to the claimed invention. Nevertheless, the circumstances surrounding the claimed invention have also to be taken into account. Having regard to what is indicated above, in the Board's judgment the documents relied upon by the Appellant in the present case, taken together or individually, did not make the claimed invention of the opposed patent obvious at the priority date.

3.4 It was submitted by the Respondent that in opposition proceedings before the EPO, when assessing inventive step the benefit of any doubt should be given to the patentee, because if the patent is revoked, that is the end of it, whereas a maintained patent can always be subsequently attacked before national courts. However, in the Board's view, there is nothing in the EPC which indicates that there should be a different standard of inventiveness as between opposition proceedings and proceedings before national courts, or that any such benefit of doubt should be given.

3.5 The late filed documents (D8) to (D11) do not reveal anything of relevance that would not be known from the preceding ones. The Board consequently decides to disregard them in pursuance of Article 114(2) EPC.

3.6 In the Board's judgment, therefore, Claim 1 of the patent in suit involves an inventive step within the meaning of Article 56 EPC.

Order

For these reasons, it is decided that:

The appeal is dismissed.

The Registrar: The Chairman:

M. Beer G.D. Paterson