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Datasheet for the decision of 4 September 2014

Case Number: T 2340/09 - 3.4.01

Application Number: 01982459.8

Publication Number: 1330182

IPC: A61B5/055, A61K49/06, G01R33/28

Language of the proceedings: EN

Title of invention:
METHODS AND DEVICES FOR POLARISED NMR SAMPLES

Applicant:
GE HEALTHCARE AS

Headword:

Relevant legal provisions:
EPC 1973 Art. 84, 54(1), 54(2), 56
EPC Art. 123(2)

Keyword:
Main request: amendments allowable (no), clarity (no)
Auxiliary request I: amendments allowable (yes), clarity (yes), novelty (yes), inventive step (yes)

Decisions cited:

Catchword:

Europäisches Patentamt
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Case Number: T 2340/09 - 3.4.01

D E C I S I O N
of Technical Board of Appeal 3.4.01
of 4 September 2014

Appellant: (Applicant)	GE HEALTHCARE AS Nycoveien 2 0485 Oslo (NO)
Representative:	Livgard, Kim Are Birkeli Zacco Norway AS Haakon VII's gt. 2 P.O. Box 2003 Vika 0125 Oslo (NO)
Decision under appeal:	**Decision of the Examining Division of the European Patent Office posted on 27 July 2009 refusing European patent application No. 01982459.8 pursuant to Article 97(2) EPC.**

Composition of the Board:

Chairman	G. Assi
Members:	T. Zinke
	J. Geschwind

Summary of Facts and Submissions

I. The appeal filed on 25 September 2009 lies from the decision of the examining division, posted on 27 July 2009, refusing European patent application No. 01 982 459.8 published with publication No. 1 330 182 (WO-A-02/036005). The appeal fee was paid on the same date. The statement setting out the grounds of appeal was filed on 25 November 2009.

II. In the decision under appeal, the examining division, with regard to a main request then on file, held that the broad wording of claim 1 would encompass microwave arrangements for ESR (electron spin resonance) excitation and variable temperature cryostats as *"means for melting"*. With this understanding, the examining division held that the subject-matter of claim 1 was not novel (Article 54(1),(2) EPC 1973) having regard to document D3 (Almanac 1996 of Bruker Corporation, November 1995, pages 14, 15, 26, 27).
Further, the examination division held that the subject-matter of claim 1 did not involve an inventive step (Article 56 EPC 1973) starting from document D4 (WO-A-00/23797) as closest prior art and considering the distinguishing feature of the temperature being of the order of 0.1 to 10 K to be an obvious choice.
For similar reasons, the examining division also considered independent claim 5 to be obvious starting from document D4. Moreover, the examining division raised an objection against claim 5 based on Article 84 EPC 1973.

With regard to the first to seventh auxiliary requests then on file, the examining division raised similar objections under Articles 54(1),(2), 56 and 84 EPC 1973.

III. With the notice of appeal the appellant (applicant) requested that the decision under appeal be set aside and a patent be granted.

IV. With the statement setting out the grounds of appeal the appellant specified that a patent be granted on the basis of a set of amended claims according to a main request filed with the grounds of appeal.
In the appellant's view, the amended claims met the requirements of Articles 54(1),(2) and 56 EPC 1973, with regard to the disclosures of documents D3 and D4, as well as Article 84 EPC 1973 and Article 123(2) EPC.

Moreover, as an auxiliary request, the appellant requested oral proceedings.

V. On 17 April 2014 the Board issued a summons to oral proceedings scheduled to take place on 4 September 2014. A communication under Article 15(1) RPBA was issued on 22 April 2014 drawing attention to the issues to be discussed during the oral proceedings.

In particular, the Board, with regard to claim 1, raised an objection under Article 123(2) EPC against the feature *"100 watts or more"* of claim 1. Further, the Board provided its preliminary opinion that the subject-matter of independent claims 1 and 4 would not involve an inventive step taking into account the teachings of documents D3 and D4.

VI. With a letter dated 1 August 2014 the appellant filed new requests. In particular, the appellant requested that a patent be granted on the basis of sets of claims according to a main request corresponding to the main

request underlying the decision under appeal or one of auxiliary requests I to V.

Moreover, the appellant, with regard to all the requests, provided arguments for support of the amendments made, novelty and inventive step. In particular, the appellant provided new documents D8 (press release of Bruker Corporation of 9 March 2009) and D9 (extract from an Almanac 2010 of Bruker Corporation), which allegedly would show that a DNP-NMR spectrometer, i.e. a device with dynamic nuclear polarisation and nuclear magnetic resonance capabilities, became commercially available in 2009, thus refuting the examining division's understanding that such a device was disclosed by document D3 dated 1996.

VII. On 14 August 2014 the Board forwarded by fax a copy of documents D10 (Becerra et al. *"A Spectrometer for Dynamic Nuclear Polarization and Electron Parametric Resonance at High Frequencies"*, Journal of Magnetic Resonance, Series A, Volume 117, 1995, pages 28-40) and D11 (Ferguson et al. *"Temperature-Jump MAS NMR with a Laser Heater"*, Journal of Magnetic Resonance, Series A, Volume 109, 1994, pages 273-275) and introduced them into the proceedings.

VIII. The oral proceedings were held on 4 September 2014 as scheduled. During the oral proceedings the appellant finally requested that the decision under appeal be set aside and that a patent be granted on the basis of sets of claims according to:
a main request filed by letter of 1 August 2014, or
an auxiliary request I filed during the oral proceedings, or

an auxiliary request II filed during the oral proceedings, or
one of auxiliary requests III to VII corresponding to auxiliary requests I to V filed by letter of 1 August 2014, which have been renumbered.

IX. Claim 1 of the appellant's main request reads as follows:
"Device for melting a solid hyperpolarised sample, said device comprising:
(a) a cryostat able to receive a sample-retaining container for retaining a polarisable solid sample;
(b) dynamic nuclear polarisation means for hyperpolarising said solid sample at a temperature of the order of 0.1 to 10 K said dynamic nuclear polarisation means comprising magnetic field producing means for generating a magnetic field inside said device, and a microwave arrangement for irradiation of said sample;
(c) means for melting said hyperpolarised solid sample (4,8), while said sample-retaining container is inside said cryostat and within said magnetic field and;
(d) coils (31-31'') for the nuclear magnetic resonance spectroscopic analysis of said melted sample."

X. Claim 1 of the appellant's auxiliary request I reads as follows:
"Device for producing a melted hyperpolarised sample, said device comprising:
(a) a cryostat able to receive a sample-retaining container for retaining a polarisable solid sample;
(b) dynamic nuclear polarisation system for hyperpolarising said solid sample at a temperature of the order of 0.1 to 10 K, said dynamic nuclear polarisation system comprising a magnet for generating a magnetic field with a field strength of 1-25 T or

more inside said device, and a microwave arrangement for irradiation of said sample;
(c) means for melting said hyperpolarised solid sample (4,8) on a time scale of T_1 or less for the nuclear spin, while said sample-retaining container is inside said cryostat and within said magnetic field and;
(d) an NMR analysis device comprising coils (31-31'') for the nuclear magnetic resonance spectroscopic analysis of said melted sample."

XI. The claims of the appellant's auxiliary requests II to VII are not relevant for this decision.

Reasons for the Decision

1. The appeal is admissible.

2. Article 13(1) RPBA

Documents D8 and D9 as filed with the letter of 1 August 2014 raise doubts about the way the examining division understood the disclosure of document D3. For this reason, the Board admitted these documents into the proceedings.
The Board also admitted the sets of claims according to the requests submitted with the same letter into the proceedings because they represent serious attempts to overcome the Board's objections raised with the communication of 22 April 2014.

Moreover, the Board also considered the auxiliary requests I and II filed during oral proceedings as serious attempts to overcome the Board's objections under Article 84 EPC 1973 and Article 123(2) EPC raised during the oral proceedings against the main request as filed with the letter of 1 August 2014.

Therefore, the Board admitted the auxiliary requests I and II requests into the proceedings.

3. Main request

3.1 Article 123(2) EPC

Claim 1 includes the feature that the DNP temperature is "*of the order of 0.1 to 10 K*". This temperature range is not mentioned in any of the claims of the application as originally filed. As a basis for this amendment the appellant referred to page 5, line 29 of the originally filed specification. The paragraph bridging pages 5 and 6 and including the mentioned citation concerns the embodiment of Figure 1. In this respect, it is stated that the dynamic nuclear polarisation is a function of the field strength of the magnetic field and the temperature. In particular, according to page 5, lines 22-23 the magnetic field strength must be *"sufficiently high, e.g. between 1-25 T or more,"* for polarisation of the sample to take place.

Hence, the amendment which only introduces the temperature range but not the required magnetic field strength range is considered to represent a generalization, for which there is no basis in the application as originally filed.

3.2 Article 84 EPC 1973

Throughout the application it is emphasized that in order to overcome the problems of the prior art it is essential that the melting occurs in the same region as the hyperpolarisation and also that the melting occurs rapidly.

With regard to the second condition, the originally filed dependent claims stress the need for a low loss of polarization during melting.
On page 9, line 33 to page 10, line 1 it is stated that *"It is important that the melting happens on a time scale of T1 (or preferably less) for the nuclear spin."*
In the communications of the applicant/appellant during the examination and the appeal proceedings, it is emphasized as well that *"rapidly melting"* was a major aspect of the invention (cf. e.g. letter dated 19 December 2008, page 4, 3rd paragraph (*"the hyperpolarised solid sample can be quickly transferred to the liquid state"*); letter dated 6 July 2009, page 5, 4th paragraph (*"The faster the melting of the sample, the higher is the retained polarization."*); statement setting out the grounds of appeal, passage bridging pages 2 and 3 (*"However, the variable temperature operation of the ESP 360 device would be much too slow to retain any polarization following melting"*)).

Since the melting speed is not claimed, an essential feature is missing.

3.3 It follows from the foregoing that the independent claims of the main request do not meet the requirements of Article 123(2) EPC and Article 84 EPC 1973.

The main request is therefore not allowable.

4. Auxiliary request I

4.1 Article 123(2) EPC

The Board is satisfied that the amended independent claims 1 and 5 meet the requirements of Article 123(2) EPC.
In particular, the wording *"dynamic nuclear polarisation system"* is disclosed on page 4, line 5.
The feature concerning the magnetic field strength of "*1-25 T or more*" is originally disclosed on page 5, line 23.
The feature concerning an *"NMR analysis device comprising coils"* can be derived from page 12, line 11 to page 14, line 5.
The feature concerning melting *"on a time scale of T_1 or less for the nuclear spin"* is originally disclosed on page 9, line 33 to page 10, line 1.

4.2 Article 84 EPC 1973

The Board is satisfied that with the addition of the essential feature referring to the *"time scale of T_1 or less for the nuclear spin"* the requirement of clarity under Article 84 EPC 1973 is met with regard to the independent claims 1 and 5.

4.3 Novelty

4.3.1 Document D10

Document D10 discloses in a first example under the headline *"DNP SPECTROMETER"* a DNP spectrometer with a static-DNP assembly. In Fig. 4 the static-DNP assembly is shown with a probe housed in a low temperature cryostat, whereby the "*probe functions in the regime 5-300 K*" (page 30, right-hand column). Leaving open whether the disclosure of said temperature range directly and unambiguously discloses "*means for melting*" within the meaning of claim 1, the claimed

melting speed "*on a time scale of T1 or less for the nuclear spin*" cannot be inferred from document D10.

The subject-matter of claim 1 is therefore considered to be novel with regard to the mentioned first example of document D10 (*"DNP SPECTROMETER"), t*he same conclusion applying with regard to independent claim 5 mutatis mutandis.

Document D10 also discloses in a second example under the headline *"EPR SPECTROMETER"* an EPR spectrometer in which "*variable temperature operation (4-300 K)*" is achieved (cf. page 33, left-hand column). As for the first example mentioned above, leaving open whether the disclosure of said variable temperature operation in said range directly and unambiguously discloses "*means for melting*" within the meaning of claim 1, the claimed melting speed "*on a time scale of T1 or less for the nuclear spin*" cannot be inferred from document D10.

The subject-matter of claim 1 is therefore considered to be novel with regard to the mentioned second example of document D10 (*"EPR SPECTROMETER"), t*he same conclusion applying with regard to independent claim 5 mutatis mutandis.

4.3.2 Document D4

Document D4 deals with the production of hyperpolarised xenon. In section *"H. Electron-Nuclear Dynamic Polarization with Removable Radicals"* (page 10), which represents the disclosure most pertinent with regard to the present main request, hyperpolarisation based on DNP is described.

As the examining division in the decision under appeal (cf. page 9) also found, temperature values "*of the order of 0.1 to 10 K*" are not disclosed in this section, either explicitly or implicitly.

In view of this finding, the subject-matter of claim 1 is considered to be novel with regard to document D4, the same conclusion applying with regard to independent claim 5 mutatis mutandis.

4.3.3 Document D3

In the decision under appeal the examining division found that the subject-matter of claim 1 of the then pending main request was not novel with regard to document D3 due to the broad formulation of the claim features. Due to the amended wording of the claim 1 of the auxiliary request I on file, this objection no longer applies.

In particular, the EPR and ENDOR equipment disclosed in document D3 (cf. "*ESP 360 Series*") can not be regarded as a DNP system in view of the knowledge of a skilled person as evidenced by the disclosures of documents D8, D9 and D10.

Further, document D3 discloses a "*variable temperature operation from 3.8K to 500K*" but does not provide any information about the speed of temperature changes. Hence, document D3 does not disclose the claimed "*means for melting said hyperpolarised sample ... on a time scale of T_1 or less for the nuclear spin*".

Consequently, the subject-matter of claim 1 is considered to be novel with regard to document D3, the

same conclusion applying with regard to independent claim 5 mutatis mutandis.

4.4 Inventive step

4.4.1 Document D10 deals with the technique of DNP. In particular, it discloses in the example under the headline *"DNP SPECTROMETER"* a DNP spectrometer with a static-DNP assembly that has the most features in common with the subject-matter of claim 1. For these reasons, the Board considers document D10 as representing the closest prior art.

4.4.2 The distinguishing features of the subject-matter of claim 1 consists in the provision of *"means for melting said hyperpolarised solid sample ... on a time scale of T1 or less for the nuclear spin, while said sample-retaining container is inside said cryostat and within said magnetic field".* These features have the technical effect that the solid polarised sample is melted inside the device in which it was polarised, so that a low loss of polarisation is achieved in a repeatable manner. In addition, the melted polarised sample may be analysed in the same device in which it was melted.

4.4.3 Hence, starting from document D10 the technical problem to be solved can be formulated as improving the known device for producing a hyperpolarised melted sample so that before NMR analysis of the melted sample a minimal loss of polarisation is achieved in a repeatable manner.

4.4.4 Neither document D10 per se nor the combination of this document with anyone of the other prior art documents on file gives the person skilled in the art a hint to

modify the disclosure of document D10 in accordance with the claimed invention.

With regard to document D10, it discloses an EPR-spectrometer having a "*variable temperature operation (4-300 K)*" (cf. page 33, left-hand column). However, no hint is given as to whether this variable temperature operation could be used for melting a sample. In addition, no information is provided about the time scale to be used for the variable temperature operation. Further, the fact, that the variable temperature operation is only mentioned for the EPR equipment and not for the DNP equipment is an indication that document D10 does not consider a variable temperature operation, including melting of a sample, to be valuable for DNP hyperpolarising a sample. Hence, a person skilled in the art would not have any incentive to provide the static-DNP assembly with means for rapidly melting a solid hyperpolarised sample.

With regard to document D4, there is also no direct and unambiguous disclosure of "*means for melting said hyperpolarised solid sample ... while said sample-retaining container is inside said cryostat and within said magnetic field*" in combination with DNP. Document D4 discloses a dilution refrigerator having a "*Vari-Temp feature*" (cf. page 11, lines 3 to 6) as one of several arrangements for removing radicals after polarising solid xenon. However, it is not unambiguously and directly derivable whether document D4 considers this particular arrangement to be useful for removing the radicals with electronic paramagnetic properties used for the DNP as described on page 10 under section *"H."*. The only reference to removal of electronic paramagnets for DNP can be found in the

section *"Example"* on page 12, line 20 to page 13, line 2. This paragraph is, however, ambiguous with regard to the removal of the radicals or electron paramagnets used as relaxants. On page 12, lines 27 to 28 it is stated that *"the radicals are more difficult to remove."* On the other hand it is mentioned that *"After polarization, the microwave frequency employed is turned off and the electronic paramagnets removed as before."* (page 12, lines 28 to 30). As several alternatives for removing radicals and electronic paramagnets are mentioned *"before"* (e.g. warming of solid hyperpolarised xenon after transferral to a *separate* storage cryostat (see page 10, line 27 to page 11, line 2)), it is unclear, which alternative is suited for the *"more difficult"* removal of the electronic paramagnets in the case of DNP. Furthermore, the passage on page 11, lines 3 to 6 only mentions that after having quickly warmed the sample, "*the gas can be cryopumped directly into another cryostat*". Hence, no information is given about an analysis of the sample in the melted phase.

On page 9, paragraph 6 of the appealed decision the examining division stated that *"When gases are polarized, it is common to use small pickup coils to monitor build-up of the polarisation in order to optimize the velocity of the production process. Such pick-up coils had already been foreseen in the very first experiments describing possibilities of polarizing Xenon-129 via spin-exchange with Alkali atoms."* There is, however, no evidence provided for this assumption. Even assuming, arguendo, that such pick-up coils were known, it is still not evident that the pick-up coils and the corresponding NMR apparatus are adapted to analyse the sample in a melted stage in the Vari-Temp dilution refrigerator disclosed in document D4. Hence, the person skilled in the art gets

no hint from document D4 to adapt the static-DNP assembly of D10 with the Vari-Temp dilution refrigerator of D4 as *"melting means"*.

With regard to document D11, it discloses melting of a sample with a laser and afterwards performing NMR analysis of the melted sample (cf. Fig. 4 and page 274, left column, last paragraph). The temperatures used, however, are above room-temperature and no cryostat is used. Hence, the person skilled in the art would not consider that a laser as melting means can be used in a cryostat at temperatures of the order of 0.1 to 10 K as well. Furthermore, document D11 does not deal with DNP at all.

With regard to document D3, it is a catalogue describing equipment for NMR devices. The "*variable temperature operation from 3.8K to 500K*" disclosed on page 27 does not provide any indication that rapidly melting of a sample "*on a time scale of T1 or less of the nuclear spin*" is feasible.

With regard to documents D1, D2, D5, D6 and D7, none of them discloses or hints at the distinguishing feature mentioned above.

Hence, the subject-matter of claim 1 involves an inventive step starting from document D10 as closest prior art and taking into account the disclosure of any of the other prior art documents on file, the same conclusion applying with regard to independent claim 5 mutatis mutandis.

5. Auxiliary request II

In view of the foregoing, it is not necessary to consider the Auxiliary request II.

Order

For these reasons it is decided that:

1. The decision under appeal is set aside.

2. The case is remitted to the examining division with the order to grant a patent on the basis of the auxiliary request I filed during oral proceedings on 4 September 2014 and a description to be adapted thereto.

The Registrar: The Chairman:

R. Schumacher G. Assi

Decision electronically authenticated