

Internal distribution code:

- (A) [ ] Publication in OJ  
(B) [ ] To Chairmen and Members  
(C) [X] To Chairmen

D E C I S I O N  
of 17 October 1994

Case Number: T 0813/93 - 3.3.1

Application Number: 87202613.3

Publication Number: 0277386

IPC: C07C 79/10

Language of the proceedings: EN

Title of invention:  
A method for the production of HNS II

Applicant:  
Nobel Kemi AB

Opponent:  
-

Headword:  
Hexanitrostilbene/NOBEL

Relevant legal norms:  
EPC Art. 56

Keyword:  
"Technical problem - proper definition"  
"Inventive step (yes) - non-obvious selection of a solvent"

Decisions cited:  
T 0020/81, T 0459/91, T 0741/91, T 0439/92, T 0380/93

Catchword:  
-



Case Number: T 0813/93 - 3.3.1

**D E C I S I O N**  
of the Technical Board of Appeal 3.3.1  
of 17 October 1994

**Appellant:** Nobel Kemi AB  
Box 800  
691 85 Karlskoga (SE)

**Representative:** Falk, Bengt  
Bofors AB  
Patents and Trademarks  
S-691 80 Karlskoga (SE)

**Decision under appeal:** Decision of the Examining Division of the European Patent Office dated 15 June 1993 refusing European patent application No. 87 202 613.3 pursuant to Article 97(1) EPC.

**Composition of the Board:**

**Chairman:** A. Jahn  
**Members:** P. Krasa  
R. E. Teschemacher

## Summary of Facts and Submissions

- I. European patent application No. 87 202 613.3 (publication No. 0 277 386) was filed on 23 December 1987 with the following independent Claim 1:

"A method for the production of a 2,2',4,4',6,6'-hexanitrostilbene (HNS) with specified purity from a less pure raw-product, generally referred to as HNS I, comprising recrystallisation of the above raw-product from N-methylpyrrolidone (1-methyl-2-pyrrolidone)."

- II. By a decision dated 15 June 1993, the Examining Division refused the application on the ground that the subject-matter of the application in suit as defined in the then pending only claim did not result from an inventive step having regard to the documents

- (2) US-A-4 604 489,
- (3) US-A-3 505 413, and
- (4) F.A. Carey and R.J. Sundberg, "Advanced Organic Chemistry", second edition (1984), p. 268.

The Examining Division argued in particular that it was known from citation (3) that a purified HNS could be obtained by recrystallisation of impure HNS (HNS I) in solvents such as N,N-dimethylformamide (DMF). In view of the statement in the application in suit, that this process was not satisfactory, the problem to be solved was defined as to provide "... an improved process for the purification of HNS I that would yield a more satisfactory end product". The use of N-methylpyrrolidone (NMP) as the recrystallisation solvent, optionally in the presence of a co-solvent such as chlorobenzene or toluene, suggested in the application in suit as solution of the said technical

problem, was deemed to have been obvious from the combined teachings of document (2) and (4). According to the Examining Division, citation (2) discloses "... a recrystallisation process for the purification of HNS I using dimethylsulfoxide (DMSO) ..." and document (4) lists up DMF and DMSO as part of a group of organic solvents generally named 'aprotic polar solvents', this list further comprising only hexamethylphosphoric triamide (HMPT), NMP and sulfolane. The Examining Division held that the true teaching of the citations (2), (3) and (4) was that aprotic polar solvents were generally suitable for the recrystallisation of HNS and that the selection of the best solvent was a matter of routine trials.

III. An appeal was lodged against this decision on 10 August 1993 and the appropriate fee was paid on 17 August 1993. In his Statements of Grounds of Appeal, filed on 31 August 1993, the Appellant argued, inter alia, that the Examining Division did not pay sufficient attention to the drawbacks of DMF as a solvent for the purification of HNS. Having regard to the fact that the results of recrystallising HNS with DMF are unsatisfactory, while DMSO is apt for this purpose, it cannot be argued that a skilled person would have directed his attention to other aprotic polar solvents such as NMP when aiming at better solvents.

IV. In a reply to a communication of the Board pursuant to Article 110(2) EPC, the Appellant submitted on 26 September 1994 an amended claim and amended pages 1 to 10 of the description and requested that the decision under appeal be set aside and a patent be granted on that basis. The new claim differs from original Claim 1 by replacing --with specified purity from-- by "with the specified purity of HNS II from". Following a phone call

from the Rapporteur, the Appellant submitted on 6 October 1994 new pages 1, 3 and 6 and replaced page 1 on 7 October 1994 by a further version.

## Reasons for the Decision

1. The appeal is admissible.

2. *Amendments*

The amendments in the claim are supported by the specification as originally filed (see Claims 1 and 3, in combination with the 3<sup>rd</sup> column in table 1 on page 4). Therefore, no objections arise against the only claim under Article 123(2) EPC.

3. *The Technical Problem and the Solution*

3.1 The Examining Division, starting from document (3), which relates, inter alia, to the recrystallisation of HNS from DMF (see below no. 4.3) defined the technical problem as "... the provision of an improved process for the purification of HNS I that would yield a more satisfactory end product". However, such a technical problem is too vague to be resolved by specific technical means (see T 0020/81, no. 3 of the Reasons for the Decision; OJ EPO 1982, 217, 221) and, therefore, cannot form the basis for the evaluation of inventive step.

3.2 In a number of decisions the Boards of Appeal have made clear that a re-definition of the technical problem is only appropriate if the technical problem as presented in the specification of the patent or patent application was not solved or was based on an objectively wrong

assessment of the prior art (T 0741/91, no. 3.3 of the Reasons for the Decision [not published in the OJ EPO]; T 0380/93, no. 3.3 of the Reasons for the Decision [not published in the OJ EPO] - both decisions following T 0459/91, no. 4.2 of the Reasons for the Decision [not published in the OJ EPO]; and T 0439/92, no. 6.2.2 [not published in the OJ EPO]). Therefore, it has first to be established whether or not the technical problem as disclosed in the present application can serve as a proper basis for evaluating the inventive step of the respective subject-matter.

3.3 It is set out in the description that there were several processes available in the state of the art for the purification of HNS I - which is an impure raw-product - to obtain HNS II - which is a purified product meeting particular product specifications (page 2, lines 14 to 48; here and in the following, reference to the application in suit is made to its published text). All these processes involve recrystallisation or digestion of the HNS I using either an organic solvent system or nitric acid (page 2, lines 16 to 17) and suffer, according to the application in suit, from various drawbacks:

3.3.1 HNS obtained by recrystallisation from 90% HNO<sub>3</sub> is almost always contaminated by the solvent resulting in a decrease of detonation velocity after storage or after thermal treatment (page 2, lines 28 to 30 and lines 36 to 39).

3.3.2 While the recrystallisation of HNS from DMF yields a product complying with most of the specification requirements for HNS II, the vacuum stability of this product is insufficient according to an, at present, irrefutable statement in the application in suit that it

does not meet, almost invariably, the required value of <0.6 mlg' for the first 20 minutes at 260 °C (page 2, lines 23 to 25).

3.3.3 It is further set out that, on the contrary, a process resulting in HNS with satisfying vacuum stability is disclosed in document

(1) US-A-3 699 176,

according to which HNS I is extracted with hot acetonitrile and HNS II is precipitated from the solution by a non-solvent for HNS. However, this process is rather slow due to the low solubility of HNS in acetonitrile (page 2, lines 41 to 48). In document (1) process times of from 12 to 30 hours are given (column 3, lines 32 to 33).

3.4 According to the application in suit, the deficiencies of the above methods should be overcome (page 2, line 49). Thus, taking citation (1) as the starting point for the evaluation of inventive step - which is appropriate as the process disclosed there leads to a product which has the desired vacuum stability and, moreover, as this document was already correctly discussed in the application in suit - the technical problem underlying the present invention, which can be directly deduced from the application in suit, was to find a simpler process with reduced processing times (see above No. 3.3.3).

3.5 The Board is satisfied that the process of the only claim, which essentially consists in recrystallising HNS I from NMP, solves this existing technical problem, having regard to the examples of the application in suit. It is clear that a simple recrystallisation from a solvent is less time consuming than an extraction

process of document (1), which became necessary in view of the acetonitrile's reduced solvent capacity for HNS (see above no. 3.3.3).

4. *Inventive Step*

It remains to be decided whether or not the solution suggested in the claim of the application in suit results from an inventive step.

- 4.1 Document (1) is concerned with the problem of the low efficiency of purifying HNS I via an extraction method using a mixture of acetonitrile with toluene (column 1, line 54 to column 2, line 30) and suggests to replace the toluene (which acts as a non-solvent for the precipitation of the purified HNS) by xylene and to use a particular apparatus (column 2, lines 31 to 34, and column 3, lines 1 to 13). No clue emanates therefrom to a purification process applying mere recrystallisation.
- 4.2 The Examining Division wrongfully stated that document (2) relates to the purification of HNS I. In fact, this document discloses the use of DMSO for recrystallising HNS II (column 1, lines 39 to 41). The underlying technical problem was to reduce the particle size of this product and to free it thereby from HNO<sub>2</sub> (used in a prior purification step; column 2, lines 4 to 16). Thus, this document is concerned with a completely different technical problem than the application in suit. It is silent on the usefulness of DMSO in the purification of HNS I maintaining a sufficient vacuum stability of the purified product. Thus, this citation contributes nothing to the solution of the present technical problem and, therefore, would have been disregarded by the skilled person.



4.3 Document (3) relates, inter alia, to a process for the manufacture of HNS e. g. by treating 2,4,6-trinitrotoluene with alkali metal hypochlorite or alkaline earth metal hypochlorite (column 1, lines 39 to 45). In example 1, DMF or nitrobenzene are used as solvent for the recrystallisation of the obtained HNS (column 4, lines 32 to 34). No difference is indicated for the results obtained either with DMF or with nitrobenzene. While a value for the thermal stability is given for the recrystallised product (0.5 cm<sup>3</sup>g<sup>-1</sup>h<sup>-1</sup> at 260 °C; table 2 in column 4), no figures are disclosed for the vacuum stability at 260 °C for 20 minutes which, according to the application in suit is insufficient when DMF is used as the recrystallisation solvent (see above no. 3.3.2). It is also important to note that the recrystallised HNS in document (3) is not designated as HNS II, which - in the absence of further information in this respect - leaves it open whether or not this product met the purity specifications of HNS II. Therefore, in the Board's judgement, document (3) discloses nothing which would have suggested to the skilled person that acetonitrile known from document (1) should be replaced by another aprotic polar solvent to solve the existing problem, let alone that NMP should be selected to such purpose.

4.4 The Examining Division's argument that a skilled person would have realised that DMF and DMSO, which both were known to dissolve HNS, are aprotic polar solvents and that he simply would have had to select another 'aprotic polar' solvent from the short list of document (4) enumerating DMF, DMSO, HMPT, NMP and sulfolane (page 268, lines 25 to 26) is not conclusive. It seems to result from a misconception of the term 'aprotic polar' (or, more accurately, 'aprotic dipolar'). This group of solvents comprises, according to common general knowledge, a host of solvents belonging to the groups of

e. g. ketones, N,N-disubstituted amides, nitroalkanes, nitriles, sulfoxides, and sulfones (see e. g. RÖMPP, Chemielexikon, 9<sup>th</sup> ed., vol. 1, p. 232 [1989]) and is definitely not limited to the above list of five solvents which, as a matter of fact, were not intended to define but only to exemplify this group of solvents ("... highly polar aprotic solvents such as ..."; document (4), page 268, lines 24 to 25). Therefore, even if the technical problem would have been to find the best solvent for recrystallising HNS (the decision under appeal, page 3, last but one paragraph), which is not the case as explained above, the skilled person would have been confronted with a vast number of possibilities without any guidance which one to select. Furthermore, in the present situation, he would have learned that at least two 'aprotic polar' solvents (DMF and acetonitrile, possibly also nitrobenzene) were not apt to solve the existing technical problem and, thus, would have had no reason to try NMP to that end with a reasonable expectation of success.

4.5 Therefore, the Board concludes that none of the citations (1), (2), (3) or (4), either alone or in combination, renders obvious the subject matter of the application in suit.

**Order**

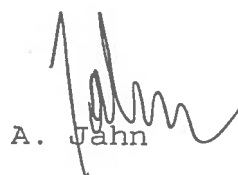
**For these reasons it is decided that:**

1. The Examining Division's decision is set aside.
2. The case is remitted to the Examining Division with the order to grant a patent with the following documents:
  - The claim and pages 2, 4, 5, 7 and 8 of the description as submitted on 26 September 1994;
  - pages 3 and 6 of the description as submitted on 6 October 1994; and
  - page 1 of the description as submitted on 7 October 1994.

The Registrar:

  
E. Görgmaier

The Chairman:

  
A. Jahn

