

Internal distribution code:

- (A) Publication in OJ
(B) To Chairmen and Members
(C) To Chairmen
(D) No distribution

D E C I S I O N
of 24 September 2002

Case Number: T 0973/98 - 3.3.7

Application Number: 92307260.7

Publication Number: 0529853

IPC: C07C 253/24

Language of the proceedings: EN

Title of invention:
Catalyst and process for producing nitriles

Patentee:
MITSUBISHI CHEMICAL CORPORATION

Opponent:
BASF Aktiengesellschaft

Headword:
-

Relevant legal provisions:
EPC Art. 83, 84, 123(2), 123(3)
EPC R. 57a

Keyword:
"Amendments - added subject-matter (no) - broadening of claim
(no) - clarity (yes)"
"Disclosure - sufficiency (yes)"

Decisions cited:
T 0133/85, T 0367/96, T 0939/92

Catchword:
-



Case Number: T 0973/98 - 3.3.7

D E C I S I O N
of the Technical Board of Appeal 3.3.7
of 24 September 2002

Appellant: MITSUBISHI CHEMICAL CORPORATION
(Proprietor of the patent) 5-2, Marunouchi 2-chome
Chiyoda-ku
Tokyo (JP)

Representative: Keen, Celia Mary
J. A. Kemp & Co.
14 South Square
Gray's Inn
London WC1R 5JJ (GB)

Respondent: BASF Aktiengesellschaft
(Opponent) Patentabteilung C6
Carl-Bosch-Strasse 38
D-67056 Ludwigshafen (DE)

Representative: -

Decision under appeal: Decision of the Opposition Division of the
European Patent Office posted 5 August 1998
revoking European patent No. 0 529 853 pursuant
to Article 102(1) EPC.

Composition of the Board:

Chairman: R. E. Teschemacher
Members: G. Santavicca
B. L. ter Laan

Summary of Facts and Submissions

I. The mention of the grant of European patent 0 529 853, in respect of European patent application 92 307 260.7, filed on 7 August 1992 and claiming a right of priority in Japan of 8 August 1991 (JP 199573/91) and 4 February 1992 (JP 18962/92), was published on 28 February 1996. The patent as granted had 15 claims, independent claims 1, 5 and 9 reading as follows:

"1. A catalyst which is suitable for the production of a nitrile from an alkane, wherein:

① the catalyst has the empirical formula:



X is at least one of Nb, Ta, W, Ti, Al, Zr, Cr, Mn, Fe, Ru, Co, Rh, Ni, Pd, Pt, Sb, Bi, B and Ce;

b is from 0.01 to 1.0;

c is from 0.01 to 1.0;

x is from 0.01 to 1.0; and

n is a number such that the total valency of the metal elements is satisfied; and

② the catalyst has X-ray diffraction peaks at the following angles at 2θ in its X-ray diffraction pattern:

Diffraction angles at 2θ ($^\circ$)

22.1 \pm 0.3

28.2 \pm 0.3

36.2 \pm 0.3

45.2 \pm 0.3

50.0 \pm 0.3."

"5. A process for producing a catalyst as defined in any one of the preceding claims which comprises drying an aqueous solution containing compounds of molybdenum, vanadium, tellurium, and at least one of niobium, tantalum, tungsten, titanium, aluminium, zirconium,

chromium, manganese, iron, ruthenium, cobalt, rhodium, nickel, palladium, platinum, antimony, bismuth, boron and cerium, and calcining the dried product in the absence of oxygen."

"9. A process for producing a nitrile, which comprises subjecting an alkane and ammonia in the gaseous state to catalytic oxidation in the presence of a catalyst as defined in any one of claims 1 to 4."

Dependent claims 2 to 4, 6 to 8 and 10 to 15 were directed to preferred embodiments of the catalyst, its process of manufacture and the process for producing a nitrile, respectively.

II. A notice of opposition was received on 20 November 1996. The opponent requested the revocation of the patent as a whole on the grounds of Articles 100(b) and 100(a) EPC (lack of novelty and an inventive step), having regard *inter alia* to EP-A-0 318 295 (D1).

During the opposition proceedings, the opponent also filed two declarations by Messrs Maruno and Aoki, along with experiments in which Example 1 of the patent in suit and Reference Example 1 of D1 were reproduced.

III. The proprietor amended the claims and *inter alia* referred to a commercial pamphlet for a nitrogen/oxygen determinator called "TC-46", manufactured by LECO Corporation, used in the examples to determine the oxygen content of the catalysts.

IV. By a decision of the Opposition Division, posted on 5 August 1998, the patent was revoked. The decision was based on amended claims submitted as the main and first to third auxiliary requests (letters dated 8 August 1997, 30 April 1998 and 23 June 1998, respectively).

In its decision, the Opposition Division held that:

- (a) The amendment to claim 1 of the main request was in compliance with Article 123(2) EPC.
- (b) The experiments carried out by the opponent were sufficiently close to the examples of the patent in suit and showed that the claimed catalysts did not achieve the exemplified performance. Hence, the invention could not be successfully reproduced on the basis of the disclosure in the description. This applied to all of the requests on file, since the catalyst, which was not reproducible, was part of all of these requests.
- (c) Therefore, the patent was revoked.

V. On 2 October 1998, the proprietor lodged an appeal, the prescribed fee being paid on the same day. With the statement of grounds of appeal, received on 15 December 1998, the appellant submitted:

- New sets of amended claims as the main and first to third auxiliary requests;
- Two declarations by Professors Iwasawa and Moro-Oka;
- Powder X-ray diffraction charts for the catalysts of Examples 15 to 20 of the patent in suit; and

- Further items of evidence in the form of calculations, diagrams and publications.

Furthermore, the appellant quoted from a number of decisions of the Boards of Appeal in relation to the legal principles applicable to the issue of sufficiency of disclosure.

- VI. In reply, the opponent (respondent) maintained that the invention was insufficiently disclosed; requested a sample of the active catalyst according to Example 1 of the patent in suit; submitted new evidence in the form of Declarations with tests reports by Messrs Lintz, Ueda, Aoki and Maruno as well as reports of experiments carried out by the respondent itself; referred to US-A-5 049 692 (D4) (equivalent to D1), as well as to two late-published documents (GEH1 = EP-A-0 603 836 and GEH2 = EP-A-0 608 838) (letters of 10 September 1999 and of 15 November 2001, respectively).
- VII. The points to be dealt with at the oral proceedings were detailed in a communication of the Board.
- VIII. The opponent submitted a further experimental report, to prove that the catalysts could not be obtained by vacuum drying the slurry (letter dated 4 September 2002).
- IX. With letter of 6 September 2002, the appellant submitted further sets of amended claims as the main and first to third auxiliary requests, respectively, to replace the requests then on file.
- X. The oral proceedings before the Board were held on 24 September 2002, during which, after a discussion of the amendments, the appellant submitted a further set of amended claims as the main request, to replace the main request then on file, and, as an auxiliary

request, the third auxiliary request then on file but with amendments yet to be made and corresponding to those made to the main request. Independent claims 1 to 3 of the main request read as follows:

"1. A catalyst which is suitable for the production of a nitrile from an alkane, wherein:

(i) the catalyst is a complex oxide which has the empirical formula:



a is 1

b is from 0.1 to 0.6;

c is from 0.05 to 0.4;

x is from 0.01 to 0.6; and

n is from 80% to 97% of $(3a + 2.5b + 3c + 2.5x)$;

(ii) the catalyst has X-ray diffraction peaks at the following angles at 2θ in its X-ray diffraction pattern:

Diffraction angles of $2\theta(^{\circ})$	Relative intensity
22.1±0.3	100
28.2±0.3	20 to 150
36.2±0.3	5 to 60
45.2±0.3	2 to 40
50 ± 0.3	2 to 40

; and

(iii) the catalyst is obtainable by a process which comprises drying an aqueous solution containing compounds of molybdenum, vanadium, tellurium, and niobium, and calcining the dried product in the absence of oxygen at a temperature of from 400°C to 650°C."

"2. A process for producing a catalyst as defined in claim 1 which comprises drying an aqueous solution containing compounds of molybdenum, vanadium, tellurium, and niobium, and calcining the dried product in the absence of oxygen at a temperature of from 400 to 650°C."

"3. A process for producing a nitrile, which comprises subjecting an alkane and ammonia in the gaseous state to catalytic oxidation in the presence of a catalyst as defined in claim 1."

Dependent claims 4 to 8 concerned preferred embodiments of the process defined in claim 3.

XI. The arguments of the appellant can be summarised as follows:

- (a) The amendments were based on the original description.
- (b) Claim 1 concerned a catalyst suitable for the conversion of alkanes to nitriles, being an oxygen deficient complex oxide having the specific crystal structure as defined. Since claim 1 did not require any specified minimum performance, a catalyst with low activity was not outside the scope of claim 1.
- (c) The patent in suit contained adequate teaching and ample exemplification to produce a catalyst as claimed. A catalyst under the terms of claim 1 was obtainable without undue burden.
- (d) For the production of the claimed catalysts, the patent required that:

- (i) an aqueous solution containing the components be dried;
- (ii) that dried product be calcined in the absence of oxygen to produce a complex oxide.

The term "complex oxide" was generally recognised as a "multi-metal-oxide", an oxide of more than one metal. Complex oxides were sensitive and their manufacture required care, which was well-known to a skilled person.

The patent in suit disclosed three methods of drying: evaporation to dryness, spray-drying and vacuum-drying, as suitable alternatives. All of them were common and the skilled person would have no difficulty in selecting the most appropriate conditions to manufacture the catalysts. In particular, the explicit indication of spray-drying, in which the drying was timed in seconds, implied that the drying time need not be long. Since a short drying time was also economical, the skilled person would not have selected a long drying time. Hence, the care and attention necessary to carry out the drying step were part of the routine capabilities of the person skilled in the art. So were the care and attention necessary to carry out the calcination step under an inert atmosphere.

- (e) The evidence submitted by the respondent did not show that the catalysts so prepared could not be produced, but only that they did not always perform well. However, that could be due to an incorrect reproduction of the examples, eg too

long a drying time. If good catalysts had not been obtained, the respondent should have tried other possibilities within the scope of claim 1, along the teaching of the patent specification.

As to vacuum drying, the single late-filed test submitted by the respondent did not mention whether or not any modification of temperature and drying time had been undertaken to obtain a better product. Anyhow, a single test could not show that vacuum drying in general did not work.

Therefore, the respondent had not shown that it was impossible to produce a catalyst according to claim 1, but rather that a manufacture under particular conditions could lead to a less effective performance. Since a number of experiments led to catalysts under the terms of claim 1, there was no proof that the conditions of Article 83 EPC had not been met by the patent specification. The opposition on the ground of insufficiency was not justified.

(f) Since late-cited documents EP-A-0 603 836 (GEH1) and EP-A-0 608 838 (GEH2) were available to the respondent well before 2002 and could have been produced well in advance, their late filing was not justifiable and the documents had to be disregarded.

(g) In so far as the evidence submitted by appellant and respondent were in conflict, the benefit of the doubt should be given to the appellant.

- (h) Since the main request represented a new case and the appellant had the right that the case be heard by two instances, the appellant expressed the desire that the case be sent back to the first instance for further prosecution.

XII. The respondent argued, in essence, as follows:

- (a) The amendments to the claims were not allowable under Article 123(2) EPC since the term "complex oxide" had no basis in the product claims; it had only been defined in granted process claims 7 and 8. Also, as established in T 133/85 (OJ EPO 1988, 441), the deletion of the feature "specific crystal structure" from the claims and the amended description contravened Article 123(2) EPC.
- (b) The amendments to the claims were not allowable either under Article 84 EPC:

The term "complex oxide" had been used in D1 and D4, but the appellant had brought the argument that the product of D1 was not a complex oxide as defined in the patent in suit. Hence, that term was not clear, nor was its meaning generally recognised in the field;

Since the feature "specific crystal structure" was presented as essential in the description, it should be included in the claims;

The indication of the peaks with their relative intensities was not sufficient for establishing with clarity any specific crystal structure. Also, no method of measure had been indicated.

- (c) The patent in suit did not contain any clear information regarding the conditions and effects of the drying methods mentioned. In particular, the importance of using spray-drying in the preparation of the catalysts was not indicated, nor that a short drying time should be applied. Since the patent in suit taught that the drying step could be followed by a decomposition step in air or inert atmosphere at a temperature of from 150° to 350°C, for the skilled person a long drying time was in accordance with the patent in suit. Also, the indication in Example 1 of the patent in suit that "the obtained slurry was evaporated to dryness at about 150°C to obtain a dried product" could only mean that the slurry had to be preliminarily concentrated at a temperature less than 100°C before being dried at 150°C.
- (d) The respondent had brought ample evidence that the reproduction of the invention failed despite following the conditions given in the examples and the patent specification.

In his tests, Professor Lintz had followed the instructions in the patent in suit. The results, however, despite the many variations made, showed that evaporation to dryness was not suitable for preparing catalysts fulfilling the conditions as claimed.

Even the catalysts obtained in the tests carried out by Professor Aoki, which fulfilled the conditions of claim 1, showed much lower performance than those exemplified in the patent in suit or no performance at all. Therefore, the X-ray diffraction peaks and intensities defined in

claim 1 could not be responsible for suitable catalysts. Vacuum drying was not suitable either, as shown in the latest test of the respondent in reply to the communication of the Board.

As could be deduced from EP-A-0 603 836, only the use of spray-drying resulted in a product fulfilling the requirements defined in claim 1 and approaching the performance exemplified in the patent in suit.

Indeed, the experiments carried out by the respondent in which spray-drying was applied gave catalysts fulfilling all the features of claim 1 in suit and approaching the catalytic performance exemplified in the patent in suit. Spray-drying also contributed to reduce the tellurium loss, the complete elimination of which could be obtained by the step of decomposition in air.

Since apart from spray-drying, the other drying methods did not allow for quick drying, the essentiality of spray-drying had not been indicated in the patent.

Therefore, the skilled person could not understand from the patent how to operate in order to obtain a well performing catalyst.

- (e) The X-ray diffraction patterns of the catalysts obtained according to the further examples of the patent in suit were not included in the patent specification. From these diffraction patterns the skilled person might have understood that the presence of two particular crystalline phases in the catalysts played an essential role for achieving the desired performance.

(f) Given the circumstances, the appellant had the burden of proof that evaporation to dryness and vacuum drying were suitable methods for the preparation of the catalysts defined in claim 1. This burden had not been discharged however, since no sample of the catalyst obtained by the procedure of Example 1 had ever been submitted.

In this regard, the declarations of Professors Iwasawa and Moro-Oka dealt with separate inventions, which could not be deduced from the patent in suit, or with arbitrary variations of the examples not indicated in the patent in suit. Thus, they were not suitable to establish sufficiency of disclosure.

XIII. The appellant (proprietor) requested that the decision under appeal be set aside and that the patent be maintained on the basis of the main request as submitted during the oral proceedings, or, alternatively, on the basis of an auxiliary request corresponding to the third auxiliary request as submitted in the letter dated 6 September 2002 with amendments yet to be made corresponding to those made to the main request during the oral proceedings.

XIV. The respondent (opponent) requested that the appeal be dismissed.

Reasons for the Decision

1. The appeal is admissible.
2. *Amendments*

2.1 Article 123(2) EPC

2.1.1 Claim 1 arises from the combination of claims 1 to 6 as granted and the addition of the following features:

"complex oxide", "a is 1", "n is from 80% to 97% of $(3a + 2.5b + 3c + 2.5x)$ " and "at a temperature of from 400°C to 650°C".

2.1.2 Claims 1 to 6 as granted are identical to claims 1 to 6 as originally filed. Claims 2 to 6 contain a reference to claim 1.

2.1.3 The term "complex oxide" is present in granted claims 7 and 8, which correspond to original claims 7 and 8, all of them referring to claims 1 to 4. Thus, in this respect, claim 1 concerns a specific object which as such had already been claimed both in the patent as granted as well as in the original application.

2.1.4 As regards the feature "a is 1", which refers to the coefficient of molybdenum (Mo), the amendment, which in fact amounts to a different way of indicating the same formula, has a basis on original page 6, line 20 (wherein "a" is the coefficient of Mo) as well as on page 7, line 9 (wherein "a is 1" is disclosed for formula (1)) of the application as originally filed.

2.1.5 The formula "n is from 80% to 97% of $(3a + 2.5b + 3c + 2.5x)$ " was disclosed in the original application, on page 6, lines 20 to 21.

2.1.6 The amendment "at a temperature of from 400°C to 650°C", to indicate the temperature range at which the calcination mentioned in claim 1 is carried out,

has a basis in original claim 6, as far as the combination of calcination and temperature range is concerned, and on original page 6, line 10, as far as the preferred temperature range is concerned.

2.1.7 The absence of the feature "specific crystal structure" in claim 1 according to the main request submitted during the oral proceedings does not contravene the requirements of Article 123(2) EPC, since that feature was not present in the original claims.

2.1.8 Therefore, the patent has not been amended in such a way that it contains subject-matter which extends beyond the content of the application as filed (Article 123(2) EPC).

2.2 Article 123(3) EPC

2.2.1 The absence of the feature "specific crystal structure" in claim 1 according to the main request does not contravene the requirements of Article 123(3) EPC, since that feature was not present in the granted claims.

2.2.2 The further amendments to the claims in fact restrict their scope.

2.3 Article 84 EPC

2.3.1 The respondent argued that the term "complex oxide" arose from a modification of the claims by a term taken from the description, so that its clarity should be examined. However, present claim 1, in this respect, results from the combination of granted claims 1 to 6, with the further restriction "complex oxide" taken from claim 7 or 8 (see point 2.1.3, *supra*). Therefore, an objection of lack of clarity in view of the term "complex oxide" is not allowable, as established eg in

T 367/96 of 3 December 1997, point 2 of the catchwords (cited in Case Law of the Boards of Appeal of the EPO, 4th edition 2001, VII.C.10.2).

2.3.2 In view of the absence of the term "specific crystal structure" in the claims as originally filed as well as in the granted claims, no objection under Article 84 EPC against its absence in the present claims can be allowed. Moreover, in the patent specification, "such a specific crystal structure" (page 3, lines 43 to 46) refers to the structures described before, which are defined by the powder X-ray diffraction pattern of present claim 1 as illustrated in the figures. Therefore, there is no reason to suppose that some essential crystal structure other than the one specified could have been meant in the description.

2.3.3 Therefore, the amendments do not give rise to objections under Article 84 EPC.

2.4 The amendments aim at overcoming the grounds of opposition, eg the ground of lack of novelty over D1 (Rule 57a EPC).

3. *Sufficiency of disclosure*

An invention is sufficiently disclosed within the meaning of Article 83 EPC if a person skilled in the art can carry it out on the basis of the information provided in the patent specification as filed in the light of the common general knowledge.

The patent specification is a written text addressed to a skilled person, who would consider the following elements when carrying out the invention:

- (a) The explicit items of information as given in the specification;

- (b) The relevant context thereof, ie the implicit items of information, which the skilled person understands when reading the specification, such as the direct and inevitable consequence of a described activity, or the content of a document explicitly referred to; and
- (c) The common general knowledge, ie those items of fundamental knowledge that need not be addressed in the specification because they are available to the ordinary practitioner in the field who knows them from or is expected to find them in generally known information sources, such as textbooks and general technical literature. On the basis of such common general knowledge, the skilled person can supplement the text with the commonly known items of information (Case Law, *supra*, II.A.2(a)).

3.1 The respondent's objections regarding a lack of sufficiently clear and complete disclosure were based on two lines of argument: the catalyst performance and the method of manufacture, in particular the method of drying the catalyst before its calcination.

3.1.1 Regarding the former, the respondent argued that the patent in suit did not contain sufficiently clear and complete information in order to arrive at a catalyst having a conversion, selectivity and yield in the production of nitriles from alkanes as exemplified in the patent in suit.

3.1.2 Claim 1 concerns a catalyst for the production of a nitrile from an alkane, which is defined in structural terms and process features relating to the calcination conditions. It contains no requirements regarding

performance apart from the suitability for the production of a nitrile from an alkane. Nor is a performance better than that of known catalysts required.

3.1.3 Whether or not a certain performance is achieved by the claimed catalysts may play a role in the assessment of inventive step under Article 56 EPC, if this technical result turns out to be the sole reason for the alleged inventiveness of the catalysts. However, if such an effect is not claimed it is irrelevant for the issue of sufficiency of disclosure (T 939/92, OJ EPO 1996, 309, Headnote point 2 and Reasons Nos. 2.4 to 2.6).

3.1.4 Therefore, the first line of argument of the respondent cannot be followed.

3.2.1 Regarding the second line of argument, concerning the drying of the catalyst, claim 1 is not restricted to any particular method of drying either.

3.2.2 According to the patent specification, three methods of drying may be used to manufacture the claimed catalysts: evaporation to dryness, spray-drying, and vacuum drying (page 3, lines 20 to 21).

3.2.3 In all of the 158 examples and 7 comparative examples of the patent in suit, the slurry had been evaporated to dryness to obtain a solid, before calcination. No details of drying are specified apart from a temperature which, if indicated, is 150°C.

3.2.4 The respondent argued that the desired catalyst could only be obtained when spray-drying was used as the drying method. The other two methods did not yield sufficiently active catalysts, even if they met the powder X-ray diffraction pattern as defined in claim 1, which was not always the case. Hence, the powder X-ray-

diffraction pattern as defined in claim 1 could not be responsible for improved catalyst performance. The patent in suit did not elucidate that spray-drying was an essential feature of the method of manufacture of the claimed catalysts.

3.2.5 In support of its arguments, the respondent relied upon a number of experiments, which show the following picture (only the experiments which concern the use of Nb have been considered, since the present claims are restricted to said element):

(1) The first experiments were carried out by the respondent as detailed in the written statement setting out the grounds of opposition, dated 19 November 1996, points IIa to IIe and I:

(a) In order to demonstrate insufficiency of disclosure, Examples 1, 2, 24 and 33 of the patent in suit were carried out by evaporating a slurry at 100°C until a paste was obtained, which was then dried at 150°C for 16h and calcined under nitrogen at 620°C.

According to the respondent, these experiments resulted in catalysts whose powder X-ray diffraction patterns were in discrepancy from the patterns in Figures 1 and 2 of the patent in suit. Also, their performance was much lower than that of the catalysts exemplified in the patent in suit (propane conversion of 5.1, 9.9, 3.8 and 7.3 mol%, nitrile selectivity of 55.7, 56.1, 63.2 and 50.9 mol%).

The appellant, however, pointed out that these catalysts still met the requirements of claim 1, including the powder X-ray diffraction pattern (Statement of grounds of appeal, point 4.2).

(b) Reference Example 1 of D1 was carried out with a view to demonstrating lack of novelty. It resulted in a catalyst having a powder X-ray diffraction pattern within the definition of claim 1, which catalyst was suitable for the conversion of propane to acrylonitrile.

(2) Further experiments were submitted during the opposition proceedings with a letter dated 27 April 1998:

(a) For the issue of sufficiency, Examples 1, 2, 24 and 33 of the patent in suit were carried out by the respondent in a reactor different to that used the first time (point 3.2.5(1)(a), *supra*). According to the respondent, these experiments resulted in catalysts whose performance was much lower than that of the catalysts exemplified in the patent in suit (Propane conversion of 4.3, 8.5, 2.5 and 4 mol%, nitrile selectivity of 28, 29, 29 and 28 mol%).

(b) Example 1 of the patent in suit was also carried out by Mr Aoki (Declaration dated 23 April 1998, annexed Exhibit) under the following conditions: use of niobium hydrogenoxalate in aqueous ammonia instead of "ammonium niobium oxalate"; evaporation conducted at 100°C for 2h to obtain a paste which was subsequently dried overnight at 150°C and calcination under a nitrogen

atmosphere. The obtained catalyst satisfied the powder X-ray diffraction pattern requirements of claim 1 and achieved a propane conversion of 10.4%, an acrylonitrile selectivity of 3.8% and an acrylonitrile yield of 0.4%.

The respondent drew the conclusion that a catalyst which satisfied the X-ray diffraction pattern of claim 1 did not achieve the high performance exemplified in the patent in suit.

- (c) In relation to the issue of novelty, a further rerun of Reference Example 1 of D1 had been carried out by Asahi Chemical Industry Co., Ltd., under the supervision of Professor Maruno (Declaration dated 20 April 1998, annexed Exhibit) under the following conditions: use of niobium hydrogenoxalate in aqueous ammonia instead of "ammonium niobium oxalate"; evaporation conducted at 70°C (as specified in the description of D1) for 3h to obtain a paste which was subsequently dried overnight at 130°C and calcination under two different air flowing conditions. The catalyst fulfilled the requirements of claim 1.
- (d) Since that result differed from the previous one of the respondent, in which the calcination had been interrupted prematurely, Reference Example 1 of D1 was carried out yet again by the respondent under the following conditions: evaporation of the slurry at 100°C until a paste was obtained, drying of that paste at 130°C for 16h and calcination at 350°C in air for 3h. The experiment resulted in a catalyst having a powder X-ray diffraction pattern within the definition of

claim 1, which was suitable for conversion of propane to acrylonitrile. According to the respondent (then opponent), this demonstrated a lack of novelty of the claimed catalysts.

- (e) In relation to the issue of inventive step, the catalyst obtained from the reproduction of Example 1 of the patent in suit was subjected to ammoxidation in the same reactor but under the conditions set out in D1, by the respondent. It resulted in a catalyst with a propane conversion of 3.8 mol% and a nitrile selectivity of 26 mol%. According to the respondent (then opponent), this demonstrated lack of inventive step, in particular because calcination under an inert atmosphere was known from (D3) DE-A-3 119 586.

- (3) A third series of experiments was submitted in appeal proceedings, with a letter dated 10 September 1999:

- (a) Regarding the issue of sufficiency of disclosure, Example 1, in which the Nb compound and the calcination conditions were varied, and Examples 2, 3 and 15 to 20 of the patent in suit were carried out by Professor Lintz ("Anlage 2": Declaration dated 30 July 1999) under drying conditions of 150°C for 3.5, 4, 5, 15, 17, 18, 66 or 68 hours and calcination conditions of 400, 500, 600 or 620°C for 2, 4, 5 or 10 hours.

None of the catalysts complied with the powder X-ray diffraction pattern of claim 1 in suit.

- (b) Since Professor Lintz did not possess any equipment for the ammoxidation of propane to acrylonitrile, the respondent undertook further experiments ("Anlage 3") to show the performance of two of the catalysts obtained by Professor Lintz on the basis of Example 1 ("Probe 1.1" and "Probe 1.4", "Anlage 2") as well as that of further catalysts obtained under drying conditions of 150°C for 1.5h and calcination conditions of 400°C for 2h, 4h or 8h.

In the ammoxidation of propane to acrylonitrile, conversions of 27, 36, 42 and 39 mol%, selectivities of 30, 12, 16 and 12 mol%, and yields of 8, 4, 7 and 5 mol% were achieved.

- (c) A further rerun of Example 1 of the patent in suit was carried out by Mr Aoki ("Anlage 4": Exhibit A to the Declaration dated 10 September 1999).

A catalyst obtained by drying at 150°C for 3.3h and calcining at 620°C for 2h fulfilled the powder X-ray diffraction pattern as well as the further conditions of claim 1 in suit and achieved a conversion of 15.7 mol%, a selectivity of 44.2 mol% and a yield of 6.9 mol% in the ammoxidation of propane.

- (d) In relation to the issue of novelty, Reference Example 1 of D1 was again carried out by Asahi Chemical Industry Co., Ltd., under the supervision of Professor Maruno ("Anlage 5": Declaration dated 27 August 1999, annexed Exhibit) under the following conditions: use of ammonium niobium oxalate; evaporation of

the slurry at 70°C until a paste was obtained, drying of that paste overnight at 130°C to obtain a dried precursor, calcination of that precursor under an air stream at 350°C for 2h, pulverization of the calcined catalyst to obtain a catalyst identified as "fresh catalyst", rinsing with oxalic acid, ammoxidation of propane under the conditions of Example 4 of D1 for 1h to obtain a catalyst identified as "used catalyst" and testing of both "fresh" and "used" catalysts.

According to the respondent, the catalysts satisfied the composition and X-ray requirements of claim 1 in suit, so that they were not distinct from the catalysts as claimed.

(4) A fourth series of experiments was submitted with a letter dated 15 November 2001:

(a) For the issue of sufficiency of disclosure, the respondent carried out 8 further experiments on the basis of Example 1 of the patent in suit, with variations in the drying methods and compositions.

(a1) In the first experiment ("Versuch 1") the catalyst solution was evaporated to dryness at 150°C for 30 min and calcined at 620°C for 2h. The X-ray diffraction pattern of the catalyst did not comply with that of claim 1 in suit. In two different tests, that catalyst had a conversion of 7 and 2 mol%, a selectivity of 27 and 28 mol% and a yield of 1.9 and 0.6 mol%.

- (a2) Two further experiments ("Versuch 2" and "Versuch 3"), in which the slurry was spray-dried, resulted in catalysts fulfilling the requirements of claim 1 in suit with a conversion of 51 and 58.4 mol%, a selectivity of 60 and 44.8 mol% and a yield of 30.6 and 26.2 mol%, respectively, in the ammoxidation of propane.
- (a3) In the next experiment ("Versuch 4"), the catalyst underwent an extra treatment, not indicated in the patent in suit, for the purpose of obtaining particular phases and crystal structures. That analysis was worked out in the following experiments ("Versuche 5, 6, 7"), in which it was shown that a particular phase identified with the letter "k" was inactive.
- (a4) The last experiment ("Versuch 8") concerned a rerun of the third experiment ("Versuch 3") while applying an additional step of decomposition in air (275°C, 1h) of the dried precursor before calcination, described as an option in the patent in suit. It resulted in a catalyst fulfilling the conditions of claim 1 in suit and achieving a conversion of 62.8 mol%, a selectivity of 48.8 mol% and a yield of 30.4 mol%. Also, in comparison to "Versuch 3", a smaller loss of Te was observed. That catalyst was also further analysed as regards the crystal structures and their effects. The respondent's conclusion was that two

particular crystal structures should be present for the catalyst to fulfil the conditions of claim 1 and be active, the presence of which was only achieved by spray-drying.

- (b) Six catalysts were prepared on the basis of Example 37 of the patent in suit by Mr Aoki under the varying compositions and drying conditions detailed in the exhibits to the declaration dated 13 November 2001.
 - (b1) The first catalyst (Y-1) solution or slurry was evaporated to dryness at 150°C for 10h and then calcined at 600°C, 2h. The X-ray diffraction pattern of the catalyst complied with that defined in claim 1 in suit and the catalyst achieved a conversion of 4.5 mol%, a selectivity of 26.7 mol% and a yield of 1.2 mol% in the ammoxidation of propane.
 - (b2) The second catalyst (Y-2) solution was spray-dried and calcined at 600°C for 2h. The X-ray diffraction pattern complied with that defined in claim 1 in suit and the catalyst achieved a conversion of 65.9 mol%, a selectivity of 60.2 mol% and a yield of 39.7 mol% in the ammoxidation of propane.
 - (b3) Catalysts Y-3 to Y-6 were prepared with substantially the same procedure as in the production of catalyst Y-2, but different starting solutions, to show the effect of a particular phase on the catalyst performance. The last two

(Y-5, Y-6) had a composition outside the claimed scope. The catalysts that fulfilled the requirements of claim 1 in suit exhibited a conversion of 1.2 and 0.8 mol%, a selectivity of 0 and 0 mol% and a yield of 0 and 0 mol%, respectively.

From these results the conclusion was drawn that the X-ray characteristics requirement defined in claim 1 in suit could not be responsible for suitable catalysts.

- (c) For the purpose of demonstrating lack of novelty, a further rerun of Reference Example 1 of D1 was again carried out under the supervision of Professor Maruno (Declaration dated 24 October 2001, annexed Exhibit).

Three catalyst samples were prepared by carrying out three times Reference Example 1 of D1 and thereafter mixing the three catalysts thus obtained into a mixture.

The slurry was evaporated to dryness at 70°C until a paste was obtained, which paste was dried overnight at 130°C to obtain a dried precursor. Calcination of that precursor was carried out under an air stream at 350°C for 3h. The so calcined catalyst was pulverized and that procedure was repeated twice to obtain two additional samples. The three samples were mixed into a mixture identified

as "fresh catalyst". That catalyst was rinsed with oxalic acid. An ammoxidation of propane was carried out under the conditions of Example 4 of D1 for 1h to obtain a catalyst identified as "used catalyst".

According to the respondent, both "fresh" and "used" catalysts satisfied the composition and X-ray requirements of claim 1 in suit. Hence, these catalysts were not distinct from the catalysts as claimed.

(d) The experiments carried out under the supervision of Professor Ueda concerned Example 1 of EP-B-0 608 838 and have no bearing to the question whether the patent in suit fulfils the requirements of Article 83 EPC.

(e) The two further declarations by Professor Maruno and Professor Lintz, respectively, which were also added to this series of experiments, *inter alia* dealt with the information which was necessary for a clear definition of a crystalline structure of a multi-metal-oxide.

(5) A fifth series of experiments was submitted with letter dated 4 September 2002:

The respondent carried out a further rerun of Example 1 of the patent in suit, but this time vacuum drying was applied to the slurry for 45 min at 55°C and 30 mbar, after which calcination took place at 620°C during 2 or 6h.

The resulting catalysts exhibit an X-ray diffraction pattern with peaks as defined in claim 1 in suit. However, the relative intensity of the peak at 28.2 ± 0.3 fell outside the required range. The respondent drew the conclusion that vacuum drying was not suitable for obtaining the claimed catalysts.

The appellant objected that these experiments had been conducted too late, so that a proper analysis and the submission of counter-evidence had not been possible.

3.2.6 The appellant maintained that the respondent had not (correctly) repeated the examples of the patent in suit and, in their turn, relied upon a number of experiments annexed to the statement of the grounds for appeal dated 15 December 1998:

- (1) Example 1 of the patent in suit had been carried out by Professor Iwasawa (Declaration dated 26 November 1998).

Catalysts were prepared using two different drying procedures, A and B.

In process A the slurry was sprayed on a commercially available Teflon-treated hot plate at 150°C and a powder was collected after 4 min. In Process B, the slurry was sprayed on a stainless steel plate and dried at 150°C for 15 min, after which a powder was collected.

The powders were again sprayed, dried and collected, and then calcined at 620°C for 2h.

The resulting catalysts exhibited an X-ray diffraction pattern as claimed and possessed a catalytic performance, as follows:

Catalyst of Process A: propane conversion of 83.5% and acrylonitrile yield of 43.8%.

Catalyst of Process B: propane conversion of 78% and acrylonitrile yield of 35.1%.

The appellant concluded that Professor Iwasawa, an independent expert who had not been asked to repeat the example but had arranged for it in the light of the common general knowledge in the field, was able to carry out the invention within the scope of claim 1 in suit, with an activity at a level comparable to that of Example 1 of the patent in suit.

- (2) Professor Moro-Oka was asked by the appellant to rerun Example 37 of the patent in suit (Declaration dated 25 November 1998). The slurry was evaporated to a paste under stirring until the paste began to foam, which paste was dried to a solid at 150°C for 3 min. After pulverization of that solid, calcination was carried out at 600°C for 2h.

The resulting catalyst exhibited an X-ray diffraction pattern as claimed and had a catalytic performance in the ammoxidation of propane to acrylonitrile, as follows: propane conversion of 73.3% and acrylonitrile yield of 44.5%.

The appellant concluded that care should be taken during the preparation of the catalyst, but no more than that which a skilled person would normally apply. Therefore, no information beyond that in the patent was required in order to carry out the examples of the patent in suit.

4. From the above described experiments submitted by the respondent and the appellant, the following picture appears:
 - 4.1 As regards the method of evaporation to dryness, the majority of the catalysts so prepared fulfil the requirements of claim 1 in suit in that they have the required X-ray diffraction pattern as well as properties that render them suitable for the production of a nitrile from an alkane.
 - 4.1.1 In particular, in all of the respondent's experiments in which examples of the patent were carried out using evaporation to dryness, with the exception of those of Professor Lintz (point 3.2.5(3)(a) *supra*), the catalysts have the required features and, even though having a lower performance than described in the patent examples, are suitable for their purpose.
 - 4.1.2 Regarding the catalyst performance, as already pointed out above (point 3.1.3), no specific performance, such as conversion, selectivity or yield, is required by the wording of claim 1 in suit, so that it is irrelevant for the issue of sufficiency of disclosure whether or not the performance is low. Moreover, it is a well-known fact that each catalyst needs its own optimal conditions for optimal performance and it has not been

shown that the conditions actually used in the experiments were the optimal ones for the particular catalysts tested. The skilled person can however be expected to look for the optimal conditions for a given catalyst.

- 4.1.3 The catalysts resulting from the experiments carried out by Professor Lintz did not meet the X-ray diffraction pattern requirements of claim 1 in suit (point 3.2.5(3)(a) *supra*).

In these experiments, apart from the absence of any concentration (evaporation) of the slurry to a paste, a drying time ranging from 3.5 to 68h had been used, which according to the appellant was too long but according to the respondent was within the teaching of the patent.

The fact that the feature "drying an aqueous solution of ..." is defined in claim 1 without specifying the exact operating conditions thereof does not imply that any drying condition should lead to the claimed product, in order that the disclosure be sufficiently clear and complete. Rather, these conditions should follow the teaching of the patent in suit in its context as understood by the person skilled in the art.

In this respect, D1 represents background art for the patent in suit and discloses a suitable protocol for evaporation to dryness.

The mention of spray-drying in the patent in suit (page 3, lines 20 to 21) indicates that the catalysts are obtainable by a quick and uniform drying of the slurry, ie a long drying time was not a teaching of the patent.

In fact, it has not been shown why the skilled person on the basis of common general knowledge in the handling of a catalyst such as the one claimed, the delicacy of which has not been disputed, would not have preferred a short drying time, which moreover would be of economic advantage.

It has not been shown either that tests routinely carried out to establish the appropriate drying conditions would in the present case constitute an undue burden for the skilled person.

The fact that experiments may be devised with the help of which particular characteristics or lower performance can be caused to occur under specially controlled conditions is not appropriate proof of insufficiency.

Therefore, it cannot be excluded that the prolonged drying time used by Professor Lintz may have damaged the catalysts and influenced the relevant properties thereof. On the basis of the teaching of the patent as a whole it is to be assumed that the skilled person would have tried to avoid such a failure by shortening the drying time. Hence, the Board is not convinced that the failure of these examples can convincingly prove insufficiency.

- 4.1.4 The appellant's results confirm the above conclusions. The catalysts obtained from the experiments carried out by Professors Iwasawa and Moro-Oka for the appellant (points 3.2.6(1) and (2), *supra*) fulfil the requirements of claim 1 in suit with a good performance in the production of a nitrile from an alkane.

The respondent argued that the drying methods used in these experiments were exotic and the skilled person would never have arrived at them on the basis of the

patent specification. However, the drying methods used in these experiments fall under the terms of "evaporation to dryness" and lead to catalysts fulfilling the requirements of claim 1 in suit.

Even if they were exotic and not immediately apparent, this would demonstrate that the claimed catalysts are obtainable by a method including a drying step which proceeds as quickly as possible, in line with the patent, and that the patentee had a legitimate interest also at protecting these products and methods. As will also become clear from the following, it has not been established that only exotic drying conditions result in products within the scope of claim 1.

4.1.5 In order to support the argument of lack of novelty, Reference Example 1 of D1 has been carried out five times, each time with the result that a catalyst was obtained that allegedly fulfilled the requirements of claim 1 in suit. In these experiments, the slurry was concentrated to a paste, eg for 3 hours at 70°C, and then dried, eg overnight at 130°C.

Since D1 belongs to the background of the invention as acknowledged in the patent in suit, see page 2, line 17 ("... we have described a Mo-V-Te-Nb-O catalyst ..."), and the only difference from the present catalyst preparation is the calcination under oxygen, instead of in its absence as now claimed, there can be no doubt that a skilled person was able to prepare a catalyst fulfilling the requirements of claim 1 in suit using evaporation to dryness.

4.1.6 In this light, the one catalyst prepared by the respondent (obtained by drying the slurry at 150°C for 30 min, without concentration (evaporation) of the slurry to a paste) which did not fulfil the requirements of claim 1 in suit (point 3.2.5(4) (a1), *supra*) cannot prove insufficiency of disclosure.

4.2 Regarding spray-drying, the respondent argued that although spray-drying was essential to achieve the claimed catalysts with the exemplified performance, such was not evident from the patent in suit and the skilled person became aware of its essentiality after reading EP-A-0 603 836 (GEH1), a later application of the appellant, in particular Comparative Example 1 thereof.

However, spray-drying was mentioned *ab initio* in the patent in suit as one of the possible ways for drying the slurry and it has not been argued that the determination of the relevant conditions required undue burden.

Furthermore, the argument that the catalyst of Comparative Example 1 of GEH1 presented further peaks in the diffraction pattern, which difference allegedly showed that spray-drying was necessary (letter dated 15 November 2001, pages 2 and 3) cannot be followed. Comparative Example 1 of GEH 1 does not disclose all the diffraction data, nor are the same conditions used as in the patent in suit, since the slurry was boiled to remove the water and no temperature is specified, contrary to Example 3 of the patent in suit. The catalyst obtained thereby was nevertheless suitable for the ammoxidation of propane to acrylonitrile. Therefore, at most the conclusion can be drawn that spray-drying resulted in an improvement, but not that it would be indispensable.

The respondent maintained that an active catalyst was obtainable if spray-drying was used and, since that was not the case with evaporation to dryness, spray-drying was an essential feature of the invention. No matter whether the argument of the essentiality of spray-drying refers to clarity or not, it cannot be followed in the light of the fact that: not all the catalysts obtained by spray-drying were active (point 3.2.5(4)(b3), *supra*) and, moreover, evaporation to dryness resulted in active catalysts.

4.3 As regards vacuum drying, a single experiment (point 3.2.5(5)(a), *supra*), which does not cover all of the possible ways of vacuum drying, resulted in a catalyst which did not fulfil the X-ray diffraction peaks of present claim 1. However, because of its filing shortly before the oral proceedings, that test could not be challenged by the appellant. Anyhow, one failed experiment is not sufficient proof that vacuum drying in general, as disclosed in the patent in suit, does not lead to the claimed catalysts. Therefore, the test is not relevant to this decision and the Board need not decide on its admission into the proceedings.

4.4 In respect of the experiments which did not achieve the desired result, the Board points out that although it may be possible to find conditions within a claim that do not result in the desired effect, eg by using extreme circumstances or correlations, the question to be answered is rather whether or not the skilled person using the best of his abilities and trying to succeed would, in reality, consider them. This applies particularly to features of a claim that are part of the operating conditions but do not form the core of the invention, such as, in the present case, the method of drying the catalyst slurry whereas the gist of the invention is formed by calcination in the absence of oxygen.

4.5 Regarding the respondent's argument that the patent lacked essential information about the exact crystal phase composition of the claimed catalysts, it is pointed out that knowledge of the exact composition of the catalysts is not a prerequisite as long as the guidance provided by the patent in suit is sufficient for the skilled person to obtain a catalyst having the required properties. Moreover, the respondent's argument is based on analytical experiments carried out with knowledge of the patent (point 3.2.5(4)(b3), *supra*).

4.6 In view of the above, the Board comes to the conclusion that the evidence submitted fails to establish that the skilled person would not have been able to carry out the invention without undue burden.

5. Although the Board has come to the conclusion that lack of sufficient disclosure has not been shown, the appellant's request that the patent be maintained on the basis of the claims submitted as the main request during the oral proceedings cannot be allowed at this stage of the proceedings because the grounds of opposition pursuant to Article 100(a) EPC have not yet been considered.

Therefore, in order to enable full consideration of the case by the Opposition Division, without depriving the parties of the possibility to be heard by two instances, the Board decides to remit the case to the first instance for further prosecution pursuant to Article 111(1) EPC.

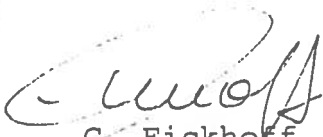
Order

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 for further prosecution.


The Registrar:

The Chairman:


C. Eickhoff




R. Teschemacher

 3/4/2003
H 080403

