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D E C I S I O N
of 12 June 1996

Case Number: T 0036/93 - 3.3.1

Application Number: 84300762.6

Publication Number: 0119709

IPC: C10G 45/64

Language of the proceedings: EN

Title of invention:
Process for the dewaxing of hydrocarbon fractions

Patentee:
TORAY INDUSTRIES, INC., et al

Opponent:
Mobil Oil Corporation

Headword:
Dewaxing process/TORAY

Relevant legal provisions:
EPC Art. 83, 54, 56, 113

Keyword:
"Sufficiency of disclosure (confirmed) - experimental counter-
evidence insufficient"
"Novelty (confirmed)"
"Inventive step (confirmed) - non-obvious alternative"

Decisions cited:
T 0219/83

Catchword:
-



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Boards of Appeal

Chambres de recours

Case Number: T 0036/93 - 3.3.1

D E C I S I O N
of the Technical Board of Appeal 3.3.1
of 12 June 1996

Appellant:
(Opponent)

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Representative:

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Respondent:
(Proprietor of the patent)

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Representative:

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Decision under appeal:

Interlocutory decision of the Opposition Division
of the European Patent Office posted 5 November
1992 concerning maintenance of the European patent
No. 0 119 709 in amended form.

Composition of the Board:

Chairman: A. J. Nuss
Members: P. Krasa
W. Moser

Summary of Facts and Submissions

I. European patent No. 119 709, relating to a process for dewaxing of hydrocarbon fractions was granted on the basis of 15 claims.

II. An opposition was filed against the patent in its entirety, raising objections under Articles 100(a) and (b) EPC, and was based on a number of documents, in particular on

(1) US-E-28 398.

In the course of the opposition proceedings, the Appellant (Opponent) submitted an affidavit of Mr Sheppard, received by the EPO on 3 August 1990 (hereinafter 'SHEPPARD'), and the Respondent (Proprietor of the patent) submitted two affidavits of Mr Tada, the second one having been received by the EPO on 29 April 1992 (hereinafter 'TADA II').

III. In its decision delivered orally on 1 July 1992, with written reasons posted on 5 November 1992, the Opposition Division held that the grounds for opposition raised did not prejudice the maintenance of the patent as amended according to the Respondent's main request comprising fourteen claims, Claim 1 of which reads as follows:

"1. A process for dewaxing a hydrocarbon fraction comprising contacting a hydrocarbon fraction with hydrogen in the presence of a catalyst comprising a zeolite having in its alkaline form an X-ray diffraction pattern shown in the following table:

<u>d(nm)</u>	<u>100 I/I_{MAX}</u>
1.12±0.02	S
1.01±0.02	S
0.386±0.008	VS
0.372±0.008	S
0.366±0.005	M

being represented by a general formula



in which M represents hydrogen cation or a precursor thereof, n is a valence of M, X is a value of from 20 to 35, and Y is a value of from 0 to 25, and having a mesitylene adsorption of not less than 1.8 wt%."

IV. The Opposition Division held in essence that

- the catalyst of the claimed process having the required adsorption properties could be prepared according to the examples given in the specification of the patent in suit,
- the Appellant, when denying novelty, had not convincingly demonstrated that the adsorption of mesitylene was not merely an (outer) surface phenomenon of the catalyst and not linked to the zeolite structure, and

- document (1), disclosing a hydrocarbon dewaxing process requiring a zeolite having a mesitylene adsorption of less than 1.5 wt.%, was leading the skilled person away from the technical teaching of the patent in suit.

V. An appeal was lodged against this decision.

VI. The Appellant submitted in essence that

- the Opposition Division was wrong in finding that the subject-matter of the patent in suit was not sufficiently disclosed, since there was no teaching of how to achieve the mesitylene sorption as required by claim 1 for the zeolite catalyst,
- the catalyst's mesitylene sorption, if considered as consisting of or including external sorption, was not determined by the zeolite structure but rather by the crystallite size and, thus, was no distinguishing feature,
- neither the zeolite according to the patent in suit nor ZSM-5 could internally absorb mesitylene but only externally as proved by him in the course of the opposition proceedings (see SHEPPARD),
- the Respondent's adsorption testing method (i.e. JIS K-1412, referred to in the patent in suit) was incapable of distinguishing between internal and external sorption,
- the zeolite used according to example 1 of the patent in suit did not differ significantly from zeolite ZSM-5 used in document (1), as confirmed by an affidavit of Dr Chester (received by the EPO only on 27 December 1995, hereinafter 'CHESTER'),

- the Opposition Division erred in acknowledging an inventive step and, in particular, in stating that document (1) did not contain all the features of claim 1 as amended,
- the Opposition Division erred in stating that the hydrocarbon dewaxing process according to the patent in suit gave better results than the process of document (1) (see CHESTER, point 6).

VII. The Respondent did not comment in substance on the Appellant's arguments.

VIII. The Appellant requested that the decision under appeal be set aside and the patent be revoked.

The Respondent requested that the appeal be dismissed and the patent be maintained as amended according to the main request before the Opposition Division (main request) or, alternatively, that the patent be maintained on the basis of one of three auxiliary requests as submitted also before the Opposition Division. Furthermore, the Respondent requested oral proceedings in case the Board would intend refusing the main request.

Reasons for the Decision

Procedural Issues

1. The Appeal is admissible.

Respondent's main request

2. *Amendments*

The Board is satisfied that the amended claims, on which the Opposition Division's decision was rendered, meet the requirements of Article 123(2) and (3) EPC. This not being in dispute, no further comments are required.

3. *Sufficiency of Disclosure*

- 3.1 The Board deems it appropriate in the first instance to stress that **the subject-matter of claim 1 is a process** for dewaxing a hydrocarbon fraction in which **process a zeolite** as defined in claim 1 **is used as a catalyst**.

For a process to be sufficiently disclosed in a document, it is necessary in particular that the starting materials, including - where needed - catalysts, are available to those skilled in the art.

- 3.2 Examples 1, 2, and 4 of the patent in suit disclose that zeolites are obtained by heating aqueous reaction mixtures containing, inter alia, SiO₂ and Al₂O₃ in molar ratios of 30 and 25, respectively, in an autoclave to 160°C, in the presence of carboxylic acids as

specified. The zeolite obtained according to example 1 has in its hydrogen form a mesitylene adsorption of 2 wt.% (see TADA II, page 9, lines 1 to 3, in combination with page 3, line 1 to page 4, line 5).

The determination of the mesitylene adsorption is disclosed in detail in the patent in suit: After having been fully subjected to a dealkalisation treatment using an aqueous ammonium chloride solution, subsequently shaping of the product (in the absence of a binder) to have a size of 20 to 32 mesh and calcination at 550°C for 16 hours in air, the mesitylene adsorption of the thus obtained product is measured under the following conditions:

amount of zeolite	about 4g
adsorption temperature	25 °C
carrier gas	N ₂ ; 800 Nml/min
mesitylene partial pressure	0.5 mmHg
adsorption time	6 hours

(cf. page 4, lines 1 to 20 of the patent in suit).

3.3 However, the Appellant contended that the patent in suit did not sufficiently disclose how to manufacture a zeolite having the said mesitylene adsorption of not less than 1.8 wt.% as it was not possible to obtain such a zeolite according to example 1 of the patent in suit.

3.3.1 In support of this submission, the Appellant firstly relied on the statement in SHEPPARD that a zeolite produced "... according to Example 1 of the Patent ..." has a mesitylene adsorption of "... less than 1.6 wt.%" (page 1, penultimate paragraph and page 2, last paragraph). However, SHEPPARD contains, apart from the mere reference to the "Example 1 of the Patent", no

technical details concerning the preparation of the zeolite sample. In the Board's judgement, such a rudimentary piece of information is not sufficient evidence for the alleged insufficiency of disclosure, since both the Board and the Respondent are left without any chance to verify whether the said zeolite could be regarded to correspond to something produced according to example 1 of the patent in suit on the basis of their own independent assessment of the experimental details. SHEPPARD is, therefore, not accepted by the Board as convincing evidence supporting the Appellant's submission.

3.3.2 Secondly, the Appellant referred to CHESTER as supporting evidence for the alleged insufficiency of disclosure.

3.3.2.1 According to this affidavit, example 1 of the patent in suit and the preparation of zeolite ZSM-5, according to the first paragraph of example 5 of document (1), were repeated (CHESTER points 1 and 2, respectively, page 2, lines 8 to 9, and page 4, lines 15 to 16) and the mesitylene adsorption of the respective products obtained (in the ammonium form) was measured according to the **method disclosed in document (1)** (CHESTER, page 5, lines 11 to 14, in combination with page 6, lines 18 to 19, and 22 to 23).

3.3.2.2 On the basis of the compositional analysis, the X-ray diffraction data, and the mesitylene sorption tests, the conclusion was drawn in CHESTER that the product obtained by repeating example 1 of the patent in suit was zeolite ZSM-5 and could not be distinguished crystallographically and topologically from the product obtained by repeating example 5 of document (1) (CHESTER, page 7, lines 3 to 8).

3.3.2.3 However, in the Board's judgement, the experiment as reported in point 1 of CHESTER cannot be regarded as a proper repetition of example 1 of the patent in suit, in view of a number of deviations from the experimental parameters disclosed in this example:

- apart from differences concerning the concentrations or quantities in the starting solutions (cf. CHESTER, page 2, lines 10 to 17, and the patent in suit, page 6, lines 16 to 17), there is nothing in CHESTER indicating that the reaction product was washed with distilled water to **approximate neutrality** prior to further processing as prescribed by the patent in suit (cf. page 6, lines 28 to 29), and
- establishing of the X-ray diffraction pattern of the hydrogen form, instead of the alkaline form used in the patent in suit (cf. CHESTER, page 4, lines 17 to 20, in combination with lines 24 to 26, versus the patent in suit, page 6, lines 29 to 30).

3.3.2.4 For this reason, the identity of ZSM-5 and of the zeolite disclosed in example 1 of the patent in suit was **not** established by CHESTER and the reported values of the mesitylene adsorption of 1.1 wt.% and 0.8 wt.%, respectively, of the zeolites prepared according to CHESTER disqualify as evidence.

3.3.2.5 Consequently, the statement in CHESTER (page 6, line 26 to page 7, line 2):

"I am therefore satisfied that the zeolite produced according to Example 5 of D1 must possess a "mesitylene adsorption" (determined as prescribed in the Patent in Suit) of not less than 1.8 wt%."

must fail.

3.3.2.6 Finally, the Appellant did not challenge that a skilled person would have succeeded in preparing a zeolite as defined in claim 1 of the patent in suit by one of the other examples 2 or 4 of this patent.

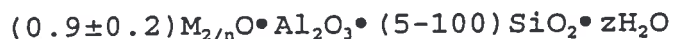
3.4 Therefore, in the absence of convincing evidence to the contrary, the Board, on the balance of probabilities, concludes that the patent in suit contains sufficient guidance for a skilled person, how to obtain a zeolite having in its hydrogen form a mesitylene adsorption of not less than 1.8% by weight. Consequently, the requirements of Article 83 EPC are met in the present case.

4. *Novelty*

4.1 Claim 1 of the patent in suit as amended relates to the hydrocarbon dewaxing of a hydrocarbon fraction whereby the hydrocarbon fraction is contacted with a zeolite as defined in the claim, in particular having in its hydrogen form a mesitylene adsorption of not less than 1.8 wt.%.

4.2 Document (1) discloses dewaxing processes carried out in the presence of crystalline zeolitic material whereby straight-chain and slightly branched-chain paraffins are removed from hydrocarbon feedstock

(column 1, lines 40 to 46). Examples of zeolites to be used according to document (1) are those either of the ZSM-5 type or the ZSM-8 type, all required to have in the acid form a mesitylene adsorption of **less than 1.5 wt.%** at a pressure of 0.5 torr, at 25 °C and a contact time of 6 hours (cf. the sentence bridging columns 3 and 4, in combination with column 3, lines 68 to 73; apart from the amount of zeolite used and the absence of a carrier gas, the conditions of the adsorption test are the same as those applied according to the patent in suit). Preferred zeolites are of the formula



wherein M is, inter alia, sodium, n is the valency of said cation, and z is from 0 to 40 (column 4, lines 14 to 22 in combination with line 11).

- 4.3 The Board cannot accept the Appellant's argument, that the higher mesitylene adsorption of the zeolites applied according to the patent in suit was not linked to the zeolites' structure and, therefore, in fact, was no distinguishing feature for the claimed process as compared with that of document (1). As stated above (point 4.2), document (1) clearly and unambiguously discloses that for the respective dewaxing process a zeolite **having a mesitylene adsorption of less than 1.5 wt.%** is required as the catalyst. This, in the Board's judgement, cannot be construed as reading on a process making use of a zeolite **having a mesitylene adsorption of not less than 1.8 wt.%** as established according to the method as disclosed in the patent in suit (cf. point 3.2 above), be that adsorption internal or external or a mixture thereof.

4.4 For the reasons already given (see point 3.3.2.3 and 3.3.2.4 above), the Board cannot accept the Appellant's submission that CHESTER proves the identity of zeolite ZSM-5 as obtained according to example 5 of document (1) on the one hand and of the zeolite obtained according to the process disclosed in example 1 of the patent in suit on the other hand. Therefore, the Appellant's argument must fail that the zeolite ZSM-5 of document (1) was a catalyst as defined in claim 1 of the patent in suit and that, consequently, the dewaxing process according to document (1) anticipated the subject-matter of that claim.

4.5 Neither can the Board accept the Appellant's argument that the Respondent's mesitylene adsorption testing procedure was inadequate. In the absence of any supporting evidence, this is a mere allegation which was contested by the Respondent in the course of the opposition proceedings and which must, thus, be disregarded by the Board (cf. T 219/83, OJ EPO, 1986, 211). Moreover, in TADA II the preparation of a zeolite in accordance with example 1 of the patent in suit is verifiably described (giving all the experimental conditions) and the adsorption performance of the thus obtained zeolite (Zeolite "R") is compared with that of a ZSM-5 zeolite (Zeolite "Z") having a $\text{Al}_2\text{O}_3/\text{SiO}_2$ molar ratio of 34.6. For three compounds of different molecular size, the following adsorbed amounts are given (TADA II, page 8, in combination with figures C, D, and E):

	Zeolite "R"	Zeolite "Z"
n-hexane (25 °C/45 mmHg)	11.9 wt.%	11.0 wt.%
2,2-dimethylbutane (93 °C/110 mmHg)	~6 wt.%	~3 wt.%
mesitylene (25 °C/0.5 mmHg)	1.9 wt.%	0.7 wt.%

4.6 These findings, which were not contested by the Appellant, indicate the existence of adsorbate-size dependent differences in the adsorption behaviour of the two zeolites and, thus, in the Board's judgement, on the balance of probabilities, render plausible that the differences found in respect to the adsorption of mesitylene also according to the JIS K-1412 method

- are significant,
- are dependent, at least in part, also on the zeolite's "internal" surface, and
- are likely to be responsible for the different dewaxing capacity of the two zeolites.

This confirms that the amount (expressed in weight-%) of mesitylene adsorbed by the catalyst is a distinguishing feature of the claimed process.

4.7 It follows that the process of claim 1 of the patent in suit is not disclosed in document (1) or any other document belonging to the state of the art. Thus, the subject matter of Claim 1 is novel.

5. *Inventive Step*

5.1 As already stated, document (1) - the only citation still relied upon by the Appellant in the appeal proceedings - is concerned with the dewaxing of a hydrocarbon feed whereby it is required for achieving this that the amount of mesitylene adsorbed by the zeolite catalyst used be less than 1.5 wt.% (cf. points 4.2 and 4.3 above).

The Appellant, relying on CHESTER, submitted that the dewaxing process claimed in the patent in suit did not produce an advantageous technical effect as compared with the dewaxing process disclosed in document (1).

5.2 The Board notes that this allegation is not adequately supported by experimental evidence, since the evidence relied upon by the Appellant fails to show that in the experiments carried out by CHESTER a zeolite was used as a catalyst having in its hydrogen form a mesitylene adsorption of not less than 1.8 wt.% measured according to the patent in suit (cf. points 3.3.2.3 to 3.3.2.5 above).

5.3 It is not necessary, however, to further investigate whether or not the process of claim 1 produces an advantageous effect as compared with the process disclosed in document (1), since, having regard to this citation, the technical problem underlying the patent in suit can be seen in any case in providing an alternative hydrocarbon dewaxing process.

In view of the examples 1, 2, and 4 of the patent in suit, the Board is satisfied that this existing technical problem is solved by the process of claim 1.

- 5.4 It still remains to be decided whether the subject-matter of claim 1 involves an inventive step.

In the Boards judgement, the clear technical teaching of document (1) that for a zeolite to be operable in the respective process it is required to have, in the acid form, a mesitylene adsorption of **less than 1.5 wt.%** under the specified conditions (see point 4.2 above) would have discouraged a skilled person to use a zeolite with a mesitylene adsorption of more than 1.5 wt.%, let alone **of 1.8 wt.% or more** as a catalyst in such a dewaxing process.

- 5.5 It follows from the above that the subject-matter of claim 1 is not rendered obvious by document (1). Dependent claims 2 to 14 relating to specific embodiments of this invention are based on the same inventive concept and derive their patentability from that of claim 1.

Right to be heard (Article 113 EPC)

6. In his letter dated 29 March 1996, received by the EPO on 2 April 1996, the Respondent requested that he be given at least a period of nine months to submit a response to CHESTER. Since the patent in suit is to be maintained in accordance with the Respondent's main request, such a response can be dispensed with without contravening Article 113(1) EPC.

The Respondent's auxiliary request

7. Under these circumstances, it was neither necessary to summon to oral proceedings, nor to consider the Respondent's auxiliary requests.

Order

For these reasons it is decided that:


The appeal is dismissed.

The Registrar:



E. Görgmaier

The Chairman:



A. Nuss

