DECISION of 1 December 1999

Case Number: T 0360/95 - 3.3.5
Application Number: 84307767.8
Publication Number: 0145289
IPC: B01J 29/32
Language of the proceedings: EN

Title of invention:
Zeolite catalyst and process for using said catalyst

Patentee:
Exxon Research and Engineering Company

Opponent:
Chevron Research and Technology Company

Headword:
Pt dispersion/EXXON

Relevant legal provisions:
EPC Art. 100b, 54, 56

Keyword:
"Sufficiency of disclosure (yes)"
"Novelty (yes, after amendment)"
"Inventive step (yes)"

Decisions cited:

Catchword:
Case Number: T 0360/95 - 3.3.5

DECISION
of the Technical Board of Appeal 3.3.5
of 1 December 1999

Appellant:
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Decision under appeal: Decision of the Opposition Division of the European Patent Office posted 28 February 1995 revoking European patent No. 0 145 289 pursuant to Article 102(1) EPC.

Composition of the Board:
Chairman: R. K. Spangenberg
Members: M. M. Eberhard
          M. B. Günzel
Summary of Facts and Submissions

I. European patent No. 0 145 289 based on application No. 84 307 767.8 was granted on the basis of eight claims. Claim 1 thereof reads as follows:

"1. A catalyst comprising a type L zeolite containing exchangeable cations of which at least about 75% are selected from the group consisting of lithium, sodium, potassium, rubidium, cesium, calcium and barium cations and containing at least one Group VIII noble metal, characterized in that the particles of the noble metal being dispersed over the surface of the catalyst and more than 90% of the noble metal associated with the zeolite is dispersed in the form of particles having a diameter of less than 0.7 nm."

II. The respondent (previous opponent) filed a notice of opposition requesting revocation of the patent on the ground of lack of novelty and lack of inventive step as well as insufficiency of disclosure. During the opposition procedure the parties relied inter alia on the following documents in support of their arguments:

D1: GB-A-2 116 450

D2: EP-A-0 096 479


II. The opposition division revoked the patent on the ground of lack of novelty. According to the decision, the catalyst according to granted claim 1 lacked novelty over the disclosure of each of D1, D2 and D19. Numerous catalyst samples had been prepared by the respondent following the teaching of Examples 1 and 2 of D1, which all exhibited a Pt dispersion as defined in claim 1. Two catalyst samples, which had been prepared according to Example 17 of D2, also had a Pt dispersion falling within the definition of claim 1. The opposition division, relying on data in D52 and D53, took the view that D19 implicitly taught a dispersion of the noble metal in the form of particles having a diameter < 1 nm. Taking into account the measurement accuracy for the particle size determined by transmission electron microscopy (TEM), D19 also destroyed the novelty of the claimed catalyst.
IV. The appellant lodged an appeal against this decision. In the course of the appeal proceedings the appellant submitted two test reports, D60 and D61, as well as four further declarations, one of Dr Newsam, D62, and three of Dr Disko, D63 to D65. The appellant filed four amended sets of claims, as auxiliary requests, on 1 November 1999. Oral proceedings were held on 1 December 1999. At the oral proceedings the appellant withdrew all his previous requests except the request filed as third auxiliary request submitted on 1 November 1999. Claim 1 of this request differs from granted claim 1 by the incorporation of the words "hydrogen reduced reforming" before the word "catalyst" in the first line of claim 1. Claim 1 is thus directed to "a hydrogen reduced reforming catalyst".

V. The appellant contested the respondent's objection regarding the insufficiency of disclosure and relied on D63, D64 and D65 to support his arguments. Concerning the lack of novelty, the appellant argued that the test reports D60 and D61 demonstrated that the respondent's TEM measurements had been carried out on catalyst precursors but not on completely reduced catalysts and were meaningless. D61 further showed that if these precursors had completely been reduced, they would have exhibited a Pt dispersion clearly outside the claimed range. As claim 1 was directed to catalysts which had completely been reduced, D60 and D61 confirmed the novelty of the claimed catalyst over D1. In view of D60 and D61 and of the Pt dispersion of catalyst C after reduction, it could not be concluded that Example 17 of D2 inevitably led to a hydrogen reduced catalyst having the claimed dispersion. D19 did not provide any specific teaching as to how a very fine Pt dispersion could be achieved and contained no example with
zeolite L. The explanations in D62 and the experiments in D60 and D61 proved that following the teaching of D19 did not inevitably result in catalysts having the Pt dispersion defined in claim 1.

Concerning inventive step, the appellant argued that D10 was the closest prior art. The feed used in Example IV of D1 was very easily convertible into aromatics so that the reported results did not provide any reliable information on good activity maintenance. However, if D1 were considered to be the closest prior art, the problem solved by the invention would have been to obtain an improved activity maintenance under reforming conditions. The examples in the patent in suit showed that the activity maintenance was improved by increasing the fineness of the noble metal dispersion. D10 taught away from the present invention and none of the documents D1, D10, D12, D19 or D23a provided any suggestion that the claimed fine noble metal dispersion could result in an improved activity maintenance. The maximum degree of dispersion of 100% considered desirable by D4 was already achieved at a Pt particle size of about 8.5 Å. Accordingly, the claimed particle dispersion was not suggested by D4. Furthermore, the skilled person was aware of the fact that zeolite L was quite different from zeolite X and Y and constituted a unique catalyst base having extraordinary properties which did not allow simple transfer of findings obtained with other catalyst base systems.

VI. In a letter dated 11 December 1996, the respondent informed the board that the opposition against the patent was withdrawn. Before withdrawal of the opposition, the respondent put forward inter alia the following arguments:
The disclosure of the patent in suit was insufficient in its characterisation of the platinum dispersion of the catalyst. Firstly, it was not possible for the skilled person to detect and measure ALL of the Pt particles having a size of at least 0.7 nm by TEM. Secondly, the skilled person was not able to determine the VOLUME of zeolite attributable to the observed Pt particles and, thus, to calculate the total amount of Pt in the relevant portion of the catalyst. Thirdly, the patent in suit disclosed no rational criteria for selecting areas of the TEM micrographs which were representative of the whole catalyst. It was evident from the opinions of experts in electron microscopy techniques that the skilled person would not be able to establish whether a given Pt/zeolite L catalyst had the claimed Pt dispersion.

The claimed catalyst lacked novelty over the disclosure of D19, D1 or D2. D19 disclosed the finest possible dispersion for the noble metal and the location thereof in the innermost adsorption regions of the zeolite. The largest Pt cluster or Pt particle that would fit in the potassium L zeolite pores was about 8.5 Å in diameter. This value fell within the claimed particle size taking into account the size accuracy that could be detected by TEM. The multiple repeats of the D1 examples done by three different sets of research teams all showed anticipation of the claimed catalysts. The appellant's theory that pH 9 to 11.5 was required for obtaining a fine dispersion was incorrect as shown by the respondent's experiments in the letter dated 19 March 1996. The catalyst samples prepared in accordance with Example 17 of D2 were also shown to have a Pt dispersion falling within the definition of claim 1. D2 did not report the washing step because washing had been regular practice as shown by the numerous declarations of persons skilled in the art.
The claimed catalyst was clearly obvious in view of the teaching of D4, D19, D1 and D3. D4 taught the well-known concept that the finer the platinum dispersion in the catalyst, the better. It showed progressively better dehydrocyclisation activity as the Pt dispersion became progressively finer in a Pt/alumina reforming catalyst. D19, which was specifically directed to zeolite catalysts, confirmed the teaching of D4 in that it taught a Pt/L zeolite catalyst of finest possible Pt dispersion in the L zeolite pores. D1 also taught use of very finely dispersed Pt on zeolite L since it was known that using a very low calcination temperature, ie just above the decomposition temperature of the Pt salt, resulted in maximising the Pt dispersion in the zeolite L as stated in D3. D1 also showed excellent catalyst activity maintenance for over 2500 hours, ie as good or better than any activity maintenance shown in the patent in suit.

VII. The appellant requested that the decision of the opposition division be set aside, that the patent be maintained on the basis of the claims filed as the third auxiliary request on 1 November 1999 and that the case be remitted to the opposition division for adaptation of the description, if necessary.

Reasons for the Decision

1. The appeal is admissible.

2. The amended claims meet the requirements of Article 123(2) and (3) EPC. It can be directly and unambiguously derived from the application as filed that the platinum dispersion defined in claim 1 is that of the hydrogen reduced reforming catalyst at least in the embodiments where the catalyst is treated by the
"enhanced dispersion technique" described in the application (see original description page 4, line 29 to page 5, line 9; page 11, lines 9 to 10; page 29, lines 11 to 16; page 30, lines 21 to 22; page 34, lines 4 to 5; page 42, lines 17 to 31; page 46, lines 10 to 18, corresponding to the passages in the patent in suit, page 3, lines 10 to 16, page 5, lines 14 to 16; page 10, lines 52 to 54; page 11, lines 15 to 16; page 12, line 11; page 14, lines 35 to 43; page 16, lines 9 to 14). The scope of protection of amended claim 1 has clearly been restricted over that of granted claim 1.

3. Concerning the requirement of sufficiency of disclosure, the appellant has submitted further explanations and comments in his additional declarations D63, D64 and D65 as to how the noble metal dispersion was determined using high-resolution bright-field imaging in transmission electron microscopy. Furthermore, the appellant has confirmed at the oral proceedings that, at the priority date, the skilled person was aware of the fact that the visibility of the small metal particles is many times higher for zeolite crystals, which have received moderate electron beam doses and lost their crystallinity, than for a crystalline zeolite. When the zeolite lattice is not present, 0.7 nm platinum particle images can clearly be distinguished from the zeolite background in thin regions of the micrographs. Taking into account the appellant's confirmation and the further explanations in D63, D64, D65, the board is satisfied, in the absence of evidence to the contrary, that a skilled person would have been able to measure the noble metal dispersion defined in claim 1, using the methods referred to on page 7 of the patent in suit. Therefore, the requirement of sufficiency of disclosure is fulfilled.
Turning to the novelty issue, the board first observes that the noble metal dispersion stated in amended claim 1 does not relate any more to a catalyst which has only been calcined and may contain a certain amount of noble metal compounds but clearly concerns hydrogen reduced reforming catalysts, ie catalysts the noble metal of which has been completely reduced into the metallic form.

4.1 The Pt dispersion of the numerous catalyst samples prepared by the previous opponent on the basis of the instructions given in Examples 1 and 2 of D1 was measured on the calcined catalysts but not on the hydrogen reduced catalysts. Furthermore, the appellant has himself provided experimental evidence in which Pt/KL catalysts (ie potassium form of zeolite L loaded with Pt) and Pt/BaKL catalysts were prepared following the teaching of D1 with a washing step of the starting zeolite and a washing step after barium-exchange similar to those performed by the previous opponent: see D61. These experiments demonstrate that after calcination at 260°C for 2 hours, the calcined catalysts still contain a high amount of Pt in the form of the platinum amine salt (78% for sample D11-6-SS-D1-260, and 46% for sample D11-6-SS-D1-K-260). Therefore, the respondent's measurements of the Pt dispersion by TEM were carried out on incompletely reduced catalysts. These experiments further show that after reduction for one hour in hydrogen at 510°C, the Pt dispersion of the completely reduced catalysts lies outside the claimed range. The reduction step was carried out at 510°C instead of 480 to 500°C in Examples 1 and 2 of D1; however, the appellant argued that this slight difference in the reduction temperature had no material effect on the Pt dispersion. In the absence of evidence to the contrary, the board accepts these arguments. In view of the results in D61 and taking into account that
the previous opponent has not proved that the catalysts of Examples 1 and 2 of D1 exhibit a Pt dispersion falling within the claimed definition after complete reduction although the burden of proof lies with him in this respect, the board considers that the claimed catalysts are novel over the disclosure of D1.

4.2 Regarding novelty with respect to the disclosure of D2, the previous opponent has measured the Pt dispersion by TEM on catalysts which were calcined at 480°C under the conditions stated in Example 17 of D2. However, the Pt dispersion after reduction in hydrogen under the conditions disclosed on page 46 of D2 was not measured, and it was also not shown that the calcination step as disclosed in Example 17 of D2 leads to a complete reduction of the platinum compound to metallic Pt. Furthermore, in view of the experiments in D61 showing the amount of Pt remaining as salt after calcination at 480°C for 2 hours, the board is not convinced that the catalyst of Example 17 of D2 would completely be reduced after the calcination step at 480°C under the conditions stated in D2. As it was not shown by the respondent, who has the burden of proof, that the reproduction of Example 17 of D2 would inevitably lead to a completely reduced catalyst having a Pt dispersion within the claimed range, the board considers that the teaching of D2 does not destroy the novelty of the claimed catalyst.

4.3 D19 teaches that the noble metal is in the inner adsorption region of the zeolite L (see claim 8). However, this document does not disclose the particle size of the noble metal, nor does it contain examples concerning the preparation of a catalyst including zeolite L. As pointed out in the decision under appeal, D52 discloses that Pt clusters up to about 15 Pt atoms can be contained within a single channel lobe (see page 217, second complete paragraph) and D53 teaches
that in a barium exchanged form of K-L zeolite the maximum diameter of the Pt particles is 0.8 to 1 nm (see page L18, first paragraph). However, none of these documents, which were published after the priority date and about 33 and 30 years after the filing date of D19, makes reference to the Pt/KL catalyst of D19. Furthermore, D62 which is a declaration of one of the authors of D52, shows that the free dimensions between D sites of "the inner adsorption regions" of a K-zeolite L crystal, such as the zeolite L of D19, will vary from 9.1 Å to 13.0 Å, with 10.9 Å being the most frequently occurring free dimension between D sites. Exhibits B and C of D62 further demonstrate that a 35 atom Pt cluster can be accommodated in the "inner adsorption regions" of a potassium zeolite L crystal such as the zeolite of D19, and that the largest cross-sectional dimension of this Pt-cluster is 12.8Å. Thus, D62 shows that it is not directly and unambiguously derivable from the teaching of D19 that the noble metal dispersion falls within the definition of claim 1. Furthermore, the appellant has prepared a Pt/KL catalyst using one of the methods disclosed in D19 for the preparation of a Pt/zeolite X catalyst (there is no example in D19 with zeolite L): see D60 and D61. None of the obtained Pt/KL catalysts had a Pt dispersion falling within the definition of claim 1. For the preceding reasons, the board considers that the catalyst according to claim 1 meets the requirement of novelty with respect to D19. The claimed catalyst is also novel over the teaching of the other documents cited during the procedure. This was not in dispute at the appeal stage so that further considerations in this respect are not necessary.

5. Concerning the issue of inventive step, the board considers that D1 represents the closest prior art since this document, contrary to D10, deals with the problem of activity maintenance of the catalyst under
reforming conditions, proposes a solution to this problem and also discloses at least one example in which the activity maintenance of the catalyst was tested over a relatively long period of time.

5.1 D1 discloses a hydrogen reduced reforming catalyst containing a Ba-, Sr- or Ca-exchanged zeolite L and a Group VIII metal, preferably Pt. This catalyst is prepared for example by impregnating a calcined Ba-exchanged zeolite with a solution of tetrammineplatinum (II) nitrate, drying, calcining at about 260°C to 500°C, in particular 260°C, and then reducing in hydrogen at temperatures of preferably 480 to 620°C. The catalysts of D1 overcome the difficulties of the prior art catalysts, such as those of D12, which have an insufficient stability (ie an insufficient activity maintenance). They give superior selectivity for converting the acyclic hydrocarbons to aromatics and also satisfactory run length (see page 1, lines 3 to 5; page 2, lines 30 to 53; page 7, lines 7 to 9 and 44 to 46; page 8, lines 9 to 12; Examples I, II, IV; claims 1 to 4, 12, 15, 21 and 22).

The technical problem to be solved with respect to this prior art catalyst can be seen in the provision of a reforming catalyst which exhibits an improved activity maintenance under reforming conditions. It is proposed that this problem be solved by a reforming catalyst in which the noble metal particles are dispersed as defined in claim 1 after hydrogen reduction. This catalyst differs from that of D1 by a higher dispersion of the noble metal.

5.2 Before withdrawal of the opposition the respondent had contested that the claimed catalysts had an improved activity maintenance over those of D1 and had submitted comparative examples in support of his arguments (see D16). The respondent had also drawn the attention to
the relatively long run life of the catalyst according to Example IV of D1 which was prepared using a large crystal size zeolite (see Table IV on page 11). Therefore, the question arises whether the problem stated above was actually solved by the claimed catalysts. Table 2 of D16 reports the results of the ECAT test for a catalyst Lla which was prepared in a manner similar to that disclosed in Example II of D1 and for catalyst H of the patent in suit which contains >99% of Pt dispersed in the form of particles having a diameter < 0.7 nm. The board observes that catalyst Lla contains a Ba-exchanged zeolite L and 0.8% Pt while catalyst H contains a non-Ba-exchanged zeolite L and only 0.6 wt.% Pt. The zeolite L particle sizes were also different in catalysts Lla and H. Furthermore, the ECAT test was performed only with catalyst Lla and compared with the results indicated in the patent in suit for catalyst H. There is no evidence that the feed used for testing catalyst Lla contains the same amount of impurities, in particular the same amount of sulphur impurities, as the feed used in the patent in suit. In these circumstances and taking into account that the Pt content of the catalyst, the presence or not of barium, and the zeolite particle size all have an influence on the catalyst performance, the board is not convinced that a valid conclusion can be drawn from these tests as to whether or not the higher dispersion of the claimed catalyst results in an activity maintenance similar to, worse or better than that of the catalysts of D1.

Regarding the results of the tests reported in Table IV of D1, the appellant has argued at the oral proceedings that the run time of 2500 hours could not be compared with the results given in the patent in suit since the results in Table IV were obtained using a very easily convertible feed and a catalyst in which the zeolite
crystal size was optimised. The appellant pointed out in particular that the feed contained 24.6 vol.% naphthenes which are easily converted into aromatics and 5 vol.% aromatics in addition to the paraffins and that the crude oil usually contained only very small amounts of methylcyclopentane (MCP). Therefore, the naphthenes and aromatics present in the feed would already lead very easily to 29.6 vol.% of aromatics. In the absence of evidence to the contrary the board can accept these arguments taking into account that, on the one hand, D1 itself shows that the crystal size of the zeolite has an influence on the activity maintenance of the catalyst (see Tables IV and V of D1, and page 12, lines 3 to 4) and, on the other hand, the feed used for the SAT test in the patent in suit contains 30 wt.% methylpentane and 10 wt.% MCP in addition to the n-hexane. Furthermore, although Example IV gives no information as to whether or not the feed was hydrofined, a hydrofined feed was used in all the other examples. According to the appellant, for the SAT test of the patent in suit, the feed materials were used as received from commercial suppliers without additional treatment and, thus, contained sulphur impurities. Therefore, the feed used in Example IV might have contained less sulphur impurities than the feed of the SAT test. In these circumstances, the board considers that it cannot be concluded from the activity maintenance reported in Example IV of D1 that the problem stated above was not solved by the claimed catalysts.

The patent in suit itself contains comparative Examples in which catalysts containing the same zeolite L, the same amount of Pt and no barium, but differing by their Pt dispersion were tested for their activity maintenance using the same feed and the same reforming conditions. These comparative examples show that the activity maintenance of a hydrogen reduced catalyst
having a Pt dispersion falling outside the claimed Pt dispersion exhibits a lower activity maintenance under reforming conditions than a catalyst having the Pt dispersed in the form of finer particles as defined in claim 1: see page 15, Table IV, the results of ECAT for catalysts C and H; Table V the results obtained with catalysts I and J; Example 6 and Figure 12 the comparison of catalysts R and S with catalysts T and U. The appellant has confirmed at the oral proceedings that the same trend, i.e. the improvement of the activity maintenance with an increased fineness of the Pt dispersion, would also be observed with a zeolite L such as that of D11, whether Ba-exchanged or not, instead of the zeolite used for the preparation of catalyst C. In these circumstances, it is credible in the absence of evidence to the contrary that the problem stated above has actually been solved by the claimed catalyst.

5.3 It can be inferred from D1 that by using a catalyst comprising a zeolite L and an alkaline earth metal selected from Ba, Sr or Ca, preferably Ba, the catalyst selectivity for converting acyclic hydrocarbons to aromatics can be improved. Furthermore, the insufficient stability of the prior art catalysts can slightly be improved to give a satisfactory run length in particular when a large crystal size zeolite is used (see page 2, lines 30 to 53 and Example IV). However, D1 neither mentions the particle size of the Pt dispersed in the catalyst nor contains any information suggesting that the activity maintenance of the catalyst of D1 might be improved by increasing the fineness of the Pt dispersion.

5.4 D10 contains information about the particle size of the platinum dispersed in the zeolite and discusses the influence of four types of Pt particles on the activity of the catalyst for the n-hexane dehydrocyclisation
into benzene and for the benzene hydrogenation into cyclohexane (see pages 1602 to 1604). According to D10, the very small particles of 6 to 8 Å embedded in the inner sites of the zeolite L are probably not accessible enough to hydrocarbon molecules since the decrease of their number on heating does not lower the catalytic activity in both reactions studied. The accessible 10 to 25 Å Pt crystals and the small cylinders (15x 40 to 70 Å) in the channels are proposed as the seat of the catalytic activity for the selective dehydrocyclisation (see page 1603, second and third paragraphs). However, the problem of activity maintenance under reforming conditions is not addressed in D10, let alone the influence of the Pt particle size on the activity maintenance. Therefore, the teaching of this document could not give a hint towards the claimed solution.

5.5 It can be inferred from the teaching of D3 on page 164, second paragraph, that it is desirable to maximize the degree of metal dispersion of supported catalysts. However, no reference is made to a particular catalyst, a particular reaction or specific properties to be achieved. D4 discloses the effect of Pt crystallite size change on reforming reactions of n-heptane. It teaches that cyclisation reactions are catalyzed more selectively by highly dispersed Pt. The dehydrocyclisation rate decreases markedly, whereas the isomerisation rate increases when the average crystallite size of a Pt/Al₂O₃(Cl) catalyst is increased by sintering at high temperatures (see page 293, Table 3-14 and page 294, first paragraph). However, D4 is completely silent about the influence of the Pt dispersion on the activity maintenance of the catalyst. Furthermore, the studied catalyst is a Pt/Al₂O₃(Cl) and it was known to the skilled person before the priority date that Pt/alumina catalysts are bifunctional
catalysts contrary to the monofunctional Pt/KL catalysts and that these two kinds of catalysts behave very differently in reforming reactions so that the results obtained with the alumina catalysts cannot simply be transferred to the Pt/zeolite L catalysts (see D12, column 1, lines 17 to 27; col.3, lines 13 to 20; and D58). For these reasons, neither D3 nor D4 could give the skilled person an incentive to increase the fineness of the Pt dispersion of the catalysts according to D1 in order to improve their activity maintenance under reforming conditions.

5.6 D19, which is a relatively old document compared to D1, discloses that "the metals provide superior catalysts when contained in the inner adsorption area of molecular sieves because the metal is contained in the finest possible distribution in a highly active form" (see column 6, lines 34 to 37). The preferred compositions which have been found to be most satisfactory and useful for catalytic purposes are the metal-loaded zeolites X, Y and faujasite (see column 7, lines 46 to 49). D19 contains no examples of zeolite L loaded with a noble metal. The statements relating to the metal distribution do not differentiate between the different types of zeolite and are not supported by any data concerning the particle size. D19 does not teach that the noble metal must be dispersed in the form of particles having a diameter smaller than the dimension of the channels of the zeolite L. Furthermore, D19 does not address the problem of activity maintenance of the catalyst under reforming conditions. Therefore, the teaching of D19 even taken in combination with the disclosure of D3 and D4, could not suggest to the skilled person that the activity maintenance of catalysts such as those of D1 would be improved by increasing the fineness of the Pt particles to the claimed extent.
5.7 The remaining documents cited during the opposition and appeal proceedings and which were published before the priority date of the patent in suit, do not contain additional information which could hint at the claimed solution even when considered in combination with the teaching of D1, D3, D4 and D19.

5.8 It follows from the above that the subject-matter of claim 1 also meets the requirement of inventive step set out in Articles 52(1) and 56 EPC.

6. Claim 1 being allowable, the same applies to dependent claims 2 to 7 whose patentability is supported by that of claim 1. Claim 8, which is directed to a process for reforming naphta which employs the catalyst according to claim 1, derives its patentability from that of the claimed hydrogen reduced catalyst. Therefore, claim 8 is also allowable.

Order

For these reasons it is decided that:

1. The decision under appeal is set aside

2. The case is remitted to the opposition division with the order to maintain the patent with the claims filed as the third auxiliary request on 1 November 1999 and a description to be adapted.

The Registrar: The Chairman

S. Hue R. Spangenberg

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