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DES BREVETS

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File Number: T 489/88 - 3.3.1

Application No.: 82 301 557.3

Publication No.: 0 061 895

Title of invention: Flow improver additive for distillate fuels, and concentrate thereof

Classification: C10L 1/18

DECISION

of 9 January 1991

Applicant:

Proprietor of the patent: Exxon Research and Engineering Company

Opponent: BASF Aktiengesellschaft, Ludwigshafen

Headword: Flow improver/EXXON

EPC Articles 56, 114(2)

Keyword: "Inventive step (confirmed)" -
"New argument submitted for the first time in oral proceedings without supporting evidence (disregarded)"

Headnote

Europäisches Patentamt
Beschwerdekammern

European Patent Office
Boards of Appeal

Office européen des brevets
Chambres de recours

Case Number : T 489/88 - 3.3.1

DECISION
of the Technical Board of Appeal 3.3.1
of 9 January 1991

Appellant : (Proprietor of the patent)	Exxon Research and Engineering Company P.O. Box 390 180 Park Avenue Florham Park New Jersey 07932 (US)
Representative :	Franck Peter Dr. Uexküll & Stolberg Patentanwälte Beselerstraße 4 2000 Hamburg 52 (DE)
Respondent : (Opponent)	BASF Aktiengesellschaft, Ludwigshafen Patentabteilung C6 Carl-Bosch-Straße 38 6700 Ludwigshafen (DE)
Representative :	
Decision under appeal :	Decision of Opposition Division of the European Patent Office of 6 June 1988, posted on 18 August 1988 revoking European patent No. 0 061 895 pursuant to Article 102(1) EPC.

Composition of the Board :

Chairman : R.W. Andrews
Members : P. Krasa
J.-C. Saisset

Summary of Facts and Submissions

I. The mention of the grant of European patent No. 61 895 in respect of European patent application No. 82 301 557.3 filed on 24 March 1982, was announced on 5 March 1986 (cf. Bulletin 86/10).

II. A notice of opposition was filed on 3 December 1986 requesting the revocation of the patent on the grounds that its subject-matter was neither novel nor inventive in the light of the disclosure of four citations. Only the following citation was finally relied upon by the Respondents (Opponents) at the oral proceedings before the Board.

(3) GB-A-565 465.

In the course of the opposition proceedings additional documents were cited by the Respondents (Opponents) of which

(1a) GB-A-930 767

corresponding to FR-A-1 272 980 (1) cited in the grounds for opposition, gained particular importance.

The Appellants (Patentees) in turn also introduced documents into the proceedings in support of their case and, in particular, the following were submitted on 4 January 1991:

(7) EP-B-85 803
(8) S.R. Reddy and M.L. McMillan, "Understanding the Effectiveness of Diesel Fuel Flow Improvers", SAE-paper 811181, 1981

(9) H. Vogel "Entwicklung, Wirkung und praxisnahe Prüfung von Fließverbesserern für Mitteldestillate auf unterschiedlicher chemischer Basis", 26. DGMK-Conference, 4-6 October 1978 in Berlin, pages 740-779

(10) GB-A-1 263 151.

III. By a decision delivered orally on 6 June 1988, with written reasons posted on 18 August 1988, the Opposition Division revoked the European patent. The Opposition Division concluded that the claimed subject-matter was novel but did not involve an inventive step in view of document (1a). The Opposition Division considered that it was obvious to reduce the amount of additives acting as cold flow improvers in blends of fuel oil compositions comprising residual oils as disclosed in (1a) if these additives were to be used in fuel oil compositions free of residual oil.

IV. An appeal was lodged against this decision on 1 October 1988 and the prescribed fee duly paid. A Statement of Grounds of Appeal was filed on 15 December 1988.

In their statement and in submissions of 9 November 1990 and 4 January 1991 and during the oral proceedings held on 9 January 1991 the Appellants argued that the Opposition Division's decision was based on the incorrect technical assumption that the flow and filterability problems of fuel blends comprising residual fuel would be due to the presence of residual fuel. The Appellants also contended that (1a) was concerned with lowering of the pour point of fuel oil compositions, a problem different from that of the disputed patent which is concerned with the improvement of filterability, and that there was no correlation between the pour point lowering activity of fuel additives and their performance as filterability improvers. In support of their arguments Appellants referred to the experimental data disclosed in the patent in suit on pages 7 to 16, and the data submitted in the course of the opposition proceedings on 9 April 1988, and with the Statement of Grounds of Appeal.

The Appellants' statement was accompanied by two new sets of claims which were re-filed in amended form by the Appellants at the oral proceedings in view of observations made by the Board on that occasion.

The independent Claims 1, 4, 12 and 17 for all designated Contracting States except AT of the first set of claims in accordance with the Appellants' main request read:

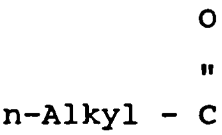
"1. The use as a cold flow improver additive for distillate fuel oil boiling in the range 120° to 500°C of from 0.0001 to 0.05 wt.% based on the weight of the fuel of polyoxyalkylene esters, ethers, ester/ethers or mixtures thereof containing at least two C_{10} to C_{30} linear saturated alkyl groups and a polyoxyalkylene glycol of molecular weight 200 to 2,000 the alkylene group of said polyoxyalkylene glycol containing from 1 to 4 carbon atoms."

"4.A middle distillate fuel oil having a boiling range from 120°C to 500°C containing 0.0001 to 0.05 wt.% of an ester, ether, or ester/ether or mixture thereof of the general formula

$$R - O - (A) - O - R^1$$

where R and R^1 are the same or different and may be

(i) n-Alkyl

(ii) 

$\text{n-Alkyl} - \overset{\overset{\displaystyle O}{\|}}{C}$

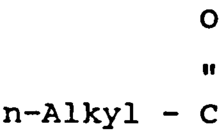
the alkyl group being linear and saturated and containing 10 to 30 carbon atoms and A is a polyoxyalkylene glycol of molecular weight 200 to 2,000 wherein the alkylene group contains 1 to 4 carbon atoms."

"12. A distillate fuel boiling in the range 120° to 500°C containing 0.0001 to 0.05 wt.% of a combination cold flow improver additive comprising (i) an ester, ether, ester/ether or mixtures thereof of the general formula

$$R - O - (A) - O - R^1$$

where R and R^1 are the same or different and may be

(i) n-Alkyl

(ii) 

$\text{n-Alkyl} - \overset{\overset{\displaystyle O}{\|}}{C}$

the alkyl group being linear and saturated and containing 10 to 30 carbon atoms and A is a polyoxyalkylene glycol of molecular weight 200 to 2,000 wherein the alkylene group contains 1 to 4 carbon atoms and (ii) an ethylene copolymer wax crystal growth inhibitor or a C_{30}-C_{300} oil soluble polar nitrogen compound wax crystal growth inhibitor being an amine and/or amide salt and/or ester/amide of a carboxylic acid having 1 to 4 carboxylic acid groups or an anhydride thereof."

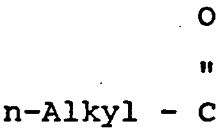
"17. An additive concentrate for incorporation into distillate fuel comprising a solution containing from 3 to

75 wt.% of an ester, ether or ester/ether or mixtures thereof of the general formula

$$R - O - (A) - O - R^1$$

where R and R^1 are the same or different and may be

(i) n-Alkyl

(ii) 

$\text{n-Alkyl} - \overset{\overset{\displaystyle O}{\|}}{C}$

the alkyl group being linear and saturated and containing 10 to 30 carbon atoms and A is a polyoxyalkylene glycol of molecular weight 200 to 2,000 wherein the alkylene group contains 1 to 4 carbon atoms, and also containing an ethylene copolymer wax crystal growth inhibitor or a C_{30}-C_{300} oil soluble polar nitrogen compound wax crystal growth inhibitor being an amine and/or amide salt and/or ester/amide of a carboxylic acid having 1 to 4 carboxylic acid groups or an anhydride thereof."

Claims 2, 3, 5 to 11, 13 to 16, 18 and 19 are dependent claims. The independent Claims 1, 7 and 11 for AT in accordance with the main request read:

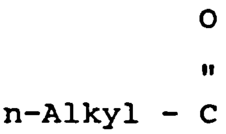
"1. A process for improving the cold flow of distillate fuel oil boiling in the range 120° to 500°C comprising incorporating therein 0.0001 to 0.05 wt.% of polyoxyalkylene ester, ether, ester/ether or mixtures thereof which contain at least two C_{10} to C_{30} linear saturated alkyl groups and a polyoxyalkylene glycol of molecular weight 200 to 2,000 the alkylene group of said polyoxyalkylene glycol containing from 1 to 4 carbon atoms."

"7. A process for improving the cold flow properties of distillate fuels boiling in the range 120° to 500°C comprising incorporating therein from 0.0001 to 0.05 wt.% of a combination of (i) an ester, ether, ester/ether or mixtures thereof of the general formula

$$R - O - (A) - O - R^1$$

where R and R^1 are the same or different and may be

(i) n-Alkyl



(ii) $\text{n-Alkyl} - \overset{\overset{\displaystyle O}{\|}}{C}$

the alkyl group being linear and saturated and containing 10 to 30 carbon atoms and A is a polyoxyalkylene glycol of molecular weight 200 to 2,000 wherein the alkylene group contains 1 to 4 carbon atoms and (ii) an ethylene copolymer wax crystal growth inhibitor or a C_{30}-C_{300} oil soluble polar nitrogen compound wax crystal growth inhibitor being an amine and/or amide salt and/or ester/amide of a carboxylic acid having 1 to 4 carboxylic acid groups or an anhydride thereof."

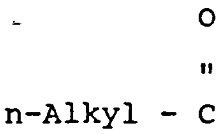
"11. A process according to any of the preceding claims wherein the polyoxyalkylene ester, ether or ester/ether is incorporated into the distillate fuel as an oil solution containing from 3 to 75 wt.% of an ester, ether or ester/ether or mixtures thereof of the general formula

$$R - O - (A) - O - R^1$$

where R and R^1 are the same or different and may be

.../...

(i) n-Alkyl

(ii) 

$n\text{-Alkyl}-\overset{\overset{O}{\|}}{C}$

the alkyl group being linear and saturated and containing 10 to 30 carbon atoms and A is a polyoxyalkylene glycol of molecular weight 200 to 2,000 wherein the alkylene group contains 1 to 4 carbon atoms.

Claims 2 to 6, 8 to 10 and 12 and 13 for AT are dependent claims.

The Appellants' first auxiliary request is identical to their main request apart from the deletion of Claims 17 to 19 from the claims for all designated States except for AT.

In accordance with the Appellants' second auxiliary request, all the claims for the Contracting designated States except for Austria are drafted as use claims. The claims for AT are identical to those of the main and first auxiliary request.

V. The Respondents argued in their submission and during the oral proceedings that the use according to the above Claim 1 (reference to claims means always reference to the claims for all the designated Contracting States except AT if not stated otherwise) differs from the use known from document (1a) only insofar as the distillate fuel is not blended with residual oil which is obtained from crude oil comprising waxy materials. According to the Respondents the additives used in present Claim 1 were already known from (1a). The Respondents submitted that document (1a) was already concerned with "easy pumping" of fuels at low temperatures and that this implied also the problem of filters being blocked by wax crystals as the respective fuels comprised waxy materials. Thus, the Respondents deduced that the additives known from and used according to (1a) as flow improvers also reduced the temperature of filter plugging by wax crystals. The Respondents further conclude that in view of (1a) it was to be expected that these additives could also be used advantageously as flow improvers with distillate fuel oils comprising wax but being free of residual oils.

According to the Respondents it could be deduced also from document (3) that dialkyl ethers of a tri- to penta ethylene glycol with alkyl groups of 10 or more carbon atoms are suitable to improve the low temperature performance of middle distillate fuels.

The Respondents also denied that the improvement of the filterability at low temperatures in terms of the cold filter plugging point (CFPP) or of the programmed cooling test (PCT) could support inventive step; according to the Respondents such data were only additional parameters characterising the flow performance of fuels at low temperature and an improvement of the low temperature flow performance of fuels in terms of lower pour points simultaneously means an improvement of the filterability of these fuels.

The Respondents further contested that the comparative tests submitted by the Appellant were suitable to support inventive step for the subject-matter claimed.

At the end of the oral proceedings the Respondents expressed their doubts in respect of the feasibility of additives resulting from polyoxyalkylene glycols where the alkylene moiety contains only 1 carbon atom.

The Respondents also raised a formal objection against Claim 1 questioning whether the upper concentration limit of 0.05 wt.% was properly disclosed in the application documents as originally filed.

VI. The Appellants requested that the decision under appeal be set aside and that the patent be maintained on the basis of the first set of claims (main request) or on the basis of two auxiliary requests. The Respondents requested that the appeal be dismissed.

VII. At the conclusion of the oral proceedings, the Board's decision to maintain the patent on the basis of the main request was announced.

Reasons for the Decision

1. The appeal is admissible.

2. There are no formal objections to the current claims in accordance with Article 123 EPC since they are supported by the original disclosure and do not extend the protection conferred when compared with the claims as granted.

2.1 Claim 1 as granted comprised neither a boiling range for the distillate fuel oils nor a range for the amount of the additives. The boiling range finds its support on page 5, line 10 of the patent (page 15, lines 11 to 12 of the application as originally filed). The concentration range (0.0001 to 0.05 wt.%) can be found on page 5, line 12 of the patent. However, in the application documents as originally filed the corresponding passage shows an upper limit of 0.5 wt.% (page 15, line 15). Nevertheless, the upper concentration limit of 0.05 wt.% finds support in the examples 2, 5, 12, 13 and 24 of both the patent as granted and the application as originally filed. In these examples the highest amount of the respective additives to be used according to Claim 1 is 500 ppm. Only Example 11 shows a higher concentration (0.1%) for these active ingredients.

All the other examples of the disputed patent show concentrations of the said additives of below 500 ppm. This holds also for Example 15 where the concentrations given relate to additive blends comprising additional active ingredients. Hence, in the Board's judgment the upper limit of 0.05 wt.-% was properly disclosed in the application documents as originally filed and is not an arbitrary one.

The molecular weight range for the polyoxyalkylene glycol of 200 to 2,000 results from the incorporation of Claim 3 as granted and as originally filed into Claim 1.

The insertion "cold" in the first line of Claim 1 before "flow" is supported by page 5, lines 11 and 12 of the patent (original application page 15, lines 14 and 15) and the examples as a whole.

2.2 The amendment of "alkyl group" to "alkylene group" is the correction of a clerical error which is admissible under Rule 88 EPC. All the other claims including those for AT are supported by the application documents as originally filed and do not extend the protection conferred when compared with the claims as granted. As these claims were not contested under Article 123 no further comments are necessary in this respect.

3. The Board considers document (1a) to be the closest state of the art and the starting point for defining the technical problem underlying the disputed patent.

3.1 Document (1a) discloses particular additives consisting of one or more diesters of a glycol or a polyglycol containing 2 to 10 carbon atoms and a saturated fatty acid having at least 10 carbon atoms. Monoethylene glycol, diethylene glycol, triethylene glycol and propylene glycol are mentioned as preferred glycols; the preferred and only exemplified additive is the diester of diethylene glycol and wool grease stearine (cf. (1a), page 1, lines 41 to 57). This citation also discloses the use of the respective additives for lowering the pour point of fuel blends comprising substantial amounts of residual fuel oil diluted with a gas oil. The pour point of such fuels is said to be often too high to permit storage and easy pumping at low temperatures (see (1a), page 1, lines 10 to 38).

However, it was found that the cold filterability of distillate fuel oils having the specified boiling range containing the exemplified additive of document (1a) was unsatisfactory.

3.2 Therefore in the light of this closest prior art the technical problem underlying the patent in suit may be seen in providing additives which show an improved effect, as compared with the known additives, on the filterability of these distillate fuel oils at low temperatures.

According to the disputed patent this technical problem is solved by the use of additives as defined in Claim 1.

In the light of the comparative tests submitted by the Appellant on 15 December 1988, especially Tables 6 and 8, the Board is satisfied that the above-defined technical problem is plausibly solved.

4. After examination of the cited documents the Board has reached the conclusion that the subject-matter of the disputed patent can be considered to be new. As the Respondent during oral proceedings only maintained that the additives per se are not novel which, however, are not claimed as such, it is not necessary to give further detailed reasons for this finding.

5. It still remains to be examined whether the requirement of inventive step is met by the claimed subject-matter.

5.1 As already indicated, document (1a) is concerned with the lowering of the pour point of fuel blends comprising residue oils to permit storage and to facilitate pumping at low temperature. Document (1a) is completely silent on the filterability of such blends at low temperature. According to the Appellants' uncontested submission in the oral proceedings these blends are dark liquids which become tarry at low temperature and their main use is in power stations and in sea-going ships where filterability at low temperature poses no problem in contrast to diesel engines in which distillate fuel oils are used.

Thus, (1a) as such gives no incentive for the skilled person to try the additives disclosed in (1a) or related compounds as cold filterability improvers in distillate fuel oil.

5.2 A different conclusion would only be possible if a relationship could be established between pour point depression and improved cold filterability - the latter characterised e.g. by means of the cold filter plugging point (CFPP) test.

Documents (7) to (10) were admitted by the Board in spite of the fact that they were received only 5 days before the oral proceedings as they were relevant to this case. They were submitted in rebuttal of Respondents' contention that the connection between pour point depression and CFPP is clear to the skilled person (see Respondents' submission of 19 December 1990, page 4, last but one paragraph). Moreover, the admission of these documents into the proceedings was not contested by the Respondents.

5.2.1 Document (7) was published on 30 December 1986 (the corresponding application on 17 August 1983) and, thus, can be taken as representing an expert opinion. It teaches that cold flow improvers for fuel oils which show excellent pour-point lowering activity may, in many cases, have substantially no effect in the CFPP test and that the pour-point test cannot forecast the plugging of a filter in a fuel supply system due to the formation of paraffin crystals at temperatures higher than the pour point (see (7), page 2, lines 29 to 36).

5.2.2 Document (8) confirms that many commercial flow improvers depress the pour point but have little or no effect on the CFPP (cf. (8), first page, right hand column, lines 12 to 14).

The paragraph bridging pages 1 and 2 of document (8) reads: "Flow improvers in a diesel fuel lower its pour point and the temperature at which fuel filter plug. Many commercially available flow improvers effectively depress the pour point...but have little or no effect on filter plugging temperatures... In the context of this report, a flow improver is an additive which both depresses the pour point and reduces the filter plugging temperature...".

While the first sentence indeed indicates two modes of action for flow improvers it cannot be interpreted in the light of the following two sentences as implying that a particular flow improver shows necessarily both effects. The third sentence makes it clear that the definition of a flow improver as a depressor of both the pour point and the CFPP holds only for the particular additives discussed in document (8) which all except one were identified as ethylene-vinyl acetate or ethylene-vinyl chloride copolymers (page 4, right column under "FLOW IMPROVERS").

5.2.3 Document (9) defines the effects which are exerted by flow or filterability improvers in middle distillate oils, i.e. pour point decrease, retardation of the paraffin crystal growth and CFPP decrease. Citation (9) further states that no flow improver exists which completely meets all requirements for cold performance (see (9), page 742, No. 1, introduction) and that optimised flow improvers on the basis of ethylene/vinyl ester are not always also optimised pour point improvers (page 744, lines 8 and 9).

Figure 4 of (9) also indicates that ethylene-vinyl ester copolymers with a particular vinyl ester content, e.g. 37 to 42 wt.%, show a pour point depression but practically no depression of the CFPP: The CFPP observed with such copolymers is e.g. about 0°C according to the first curve, which is the natural CFPP of the fuel investigated. Figure 2 of (9) is ambiguous in this respect as the CFPP of the untreated fuels concerned are not given.

5.2.4 From document (10) it can be concluded that a particular vinyl acetate ethylene copolymer (=polymer A) has a remarkable pour point decreasing activity while it has only a rather low CFPP decreasing effect when used as an additive. Other vinyl acetate-ethylene copolymers manufactured under particular reaction conditions showed both a pour point depression and a CFPP depression (pages 5 and 7, Tables II and IV in combination with page 1, lines 34 to 46).

5.2.5 In the Board's judgment, the documents (7) to (10) clearly demonstrate that there is no predictable relationship either quantitative or qualitative between the effects which can be achieved in respect to the pour point on the one hand and the CFPP on the other hand of a fuel by the addition of a specific additive.

At best it could be deduced from (8) to (10) that within particular groups of additives there may exist compounds which simultaneously depress both the pour point and the CFPP while there are also other compounds which lower only one of these parameters.

This makes it also clear that the CFPP is not merely a further parameter in addition to the pour point for the characterisation of the low temperature flow performance of fuels. These two parameters refer to two different properties which both contribute to this performance but, nevertheless, are independent from each other and address different technical problems. The same considerations apply to the PCT.

Thus, the Board finds that the skilled man in view of (1a) would have neither tested the additives disclosed in (1a) nor structurally related compounds in a CFPP test with the expectation that these compounds would improve the cold filterability of distillate fuels and solve the technical problem underlying the disputed patent.

5.3 Document (3) is concerned with additives for facilitating starting and warming up diesel engines at low temperatures and for increasing their power output. This is achieved by adding substantial amounts (i.e. at least 5% by volume) of selected dialkyl ethers of polyethylene glycols or dialkyl ethers of higher alkylene groups (page 1, lines 32 to 52). Preferred additives have the formula $R-(OR^1)_xOR$ with R being a normal alkyl of 1 to 5 carbon atoms, R^1 representing an ethylene or a higher alkylene group and x being an integer from 3 to 5. However, the alkyl groups R may be increased in length, e.g. to 10 or more carbon atoms (page 1, line 86 to page 2, line 7).

Document (3) also discloses that "...the indicated dialkyl ethers of polyethylene glycols when properly chosen and blended can be efficiently injected with the fuel into a diesel engine at a temperature as low as -40°F and lower..." without showing filterability problems (page 2, lines 65 to 70 in combination with page 2, lines 42 to 64).

This statement refers to fuel compositions where the polyethers concerned are substantial or even predominating constituents of the fuel blends and in fact act as co-fuels (page 2, lines 8 to 20 and lines 111 to 122). There is no indication in (3) that the additives to be used according to present Claim 1 would act as filterability improvers at low temperatures in concentrations of only 0.0001 to 0.05 wt.%.

Hence, the Board finds that the subject-matter of Claim 1 is not obvious to the skilled person in view of (3) either alone or in combination with (1a).

5.4 Claim 4 relates to fuel compositions which are complementary to the use defined in Claim 1 and derives its patentability from the unexpected improvement in cold filterability of such fuel compositions as discussed in relation to Claim 1. For the same reasons Claim 12 and 17, which relate to fuel compositions comprising additional additives and additive concentrates respectively, are also acceptable. Dependent Claims 2, 3, 5 to 11, 13 to 16, 18 and 19 claim preferred embodiments of the respective independent claims and are also allowable.

Claims 1 to 13 for the designated Contracting State Austria are acceptable due to the unexpected effect resulting from the processes claimed as discussed in connection with Claim 1 for the other designated Contracting States.

6. It was only at the end of the oral proceedings that the Respondents questioned, for the first time and without any material support, the feasibility of the additives of Claim 4 wherein the alkylene group of the polyoxyalkylene glycol is a methylene group. However, proper proceedings before the EPO require that the facts, evidence and arguments known to the Opponents which support the grounds of opposition to a patent are brought forward at the earliest possible date, i.e. in principle within the nine-month opposition period. One of the grounds for opposition is that it is not possible to achieve the claimed effect and, therefore, that the problem underlying the patent is not solved or, as in the present case, only solved for a part of the claimed area. The submission of this ground in the last stage of opposition appeal proceedings must be regarded as a procedural abuse.

The Board, in exercising its discretion under Article 114(2) has decided to disregard this ground of opposition introduced for the first time at the end of the oral proceedings and based solely on allegations without any supporting evidence.

7. The main request being allowable, there is no need to consider Appellants' auxiliary requests.

Order

For these reasons, it is decided that:

1. The decision under appeal is set aside.

2. The case is remitted to the first instance with the order to maintain the patent on the basis of the first set of claims submitted during oral proceedings (Claims 1 to 19 for all designated Contracting States except for Austria and Claims 1 to 13 for Austria) and a description to be brought into agreement with this set of claims.

The Registrar: The Chairman: