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**Datasheet for the decision  
of 20 December 2013**

**Case Number:** T 0130/11 - 3.3.06

**Application Number:** 01272112.2

**Publication Number:** 1363985

**IPC:** C10L3/10

**Language of the proceedings:** EN

**Title of invention:**

PROCESS FOR THE REDUCTION OR ELIMINATION OF HYDROGEN SULPHIDE

**Patent Proprietors:**

- 01) M-I L.L.C.  
02) Statoil ASA

**Opponent:**

Clariant Produkte (Deutschland) GmbH

**Headword:**

Reduction of hydrogen sulphide/M-I

**Relevant legal provisions:**

EPC Art. 52(1), 56, 54, 84, 114(2), 123(2)  
RPBA Art. 13(3), 12(4)

**Keyword:**

Late-filed document - admitted (no) -  
lack of prima facie relevance  
New main request - admissible (yes)  
Amendments - allowable (yes) (main request)  
Novelty - (yes) (main request)  
Inventive step - non-obvious solution (main request)

**Decisions cited:**

**Catchword:**



**Beschwerdekammern  
Boards of Appeal  
Chambres de recours**

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Case Number: T 0130/11 - 3.3.06

**D E C I S I O N**  
**of Technical Board of Appeal 3.3.06**  
**of 20 December 2013**

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**Decision under appeal:** **Interlocutory decision of the Opposition  
Division of the European Patent Office posted on  
23 December 2010 concerning maintenance of the  
European Patent No. 1363985 in amended form.**

**Composition of the Board:**

**Chairman:** B. Czech  
**Members:** G. Santavicca  
U. Lokys

## Summary of Facts and Submissions

- I. The appeal by the opponent lies from the interlocutory decision of the opposition division concerning maintenance in amended form of European patent n° 1 363 985.
- II. The patent in suit had been opposed in its entirety on the grounds of lack of novelty and inventive step (Article 100(a) EPC).

The evidence relied upon in the opposition proceedings includes the following documents:

D1: US 4,748,011 A;

D2: WO 98/19774 A1;

D4: J. F. Walker, Formaldehyde, Third edition, Reinhold Publishing Corp., 3rd revised edition, 1964, pages 264-273, 584, 585);

D7: W. Paulus, "Microbiocides for the protection of materials, A handbook", Chapman & Hall, 1993, pages 55 to 64; and,

D8: WO 90/07467 A1.

- III. Amended Claim 1 according to the main request held allowable by the opposition division (filed during the oral proceedings held on 4 September 2009) reads as follows (amendments to Claim 1 as granted made apparent by the board):

*"1. A process for reducing the level of hydrogen sulphide in a liquid or gaseous hydrocarbon or in sewage gas by treatment of the liquid or gas with an H<sub>2</sub>S-scavenger product comprising the reaction product of*  
*i) ~~a carbonyl group containing compound selected from aldehydes and ketones having up to 10 carbon atoms and containing no other functional group or containing a~~*

~~fatty acid group or ether group but no other functional group~~ formaldehyde, with  
ii) ~~an alcohol, thiol, amide, thioamide, urea or thiourea, containing no other functional group or containing a fatty acid group or ether group but no other functional group~~ ethylene glycol, propylene glycol, glycerol, diethylene glycol, triethylene glycol, or urea,  
~~said carbonyl group containing compound, said alcohol, thiol, amide, thioamide, or urea or thiourea, and said reaction product being free of basic groups."~~

- IV. In the decision under appeal, it was inter alia held that:
- a) The amended claims complied with the requirements of Articles 123(2), 123(3) and 84 EPC.
  - b) The claimed subject-matter was new having regard to documents D1 and D2
  - c) Starting from D1 as the closest prior art, and taking into account document D4, the claimed subject-matter also involved an inventive step.
- V. With its statement setting out the grounds of appeal, the appellant submitted a new item of evidence, namely: D15: DE 3 604 521 A (numbering according to a consolidated list filed by the respondents, *infra*).
- VI. With their reply, the respondents (patent proprietors) submitted a new set of claims as main request, two auxiliary claim requests and the explanatory document D16: "Desulphurization by use of H<sub>2</sub>S scavenger"
- VII. With a further letter, the appellant submitted more new items of evidence, namely documents  
D17: printout of web page [http://www.chemicalbook.com/ChemicalProductProperty\\_DE\\_CB2898544.htm](http://www.chemicalbook.com/ChemicalProductProperty_DE_CB2898544.htm),

"(Ethylendioxy)dimethanol Produkt Beschreibung";  
and

D18: Abstract of Römpps Chemie-Lexikon, 8th edition,  
1988, pages 4336 and 4337.

- VIII. In a further letter, the appellant raised objections under Article 123(2) EPC against the first auxiliary request, and called into question the admissibility of this request into the proceedings.
- IX. In reaction thereto, the respondents submitted new first and third auxiliary claim requests, a consolidated list of the documents/evidence filed until then (the documents numbering in this decision is in accordance with this list), as well as the following document, stated to be the US patent corresponding to the German patent application D15:  
D15b: US 4,708,720 A.
- X. In a communication issued in preparation for the oral proceedings, the Board *inter alia* expressed a positive opinion concerning the allowability of the amendments in the claims according to the pending main request under Article 123(2) EPC, but called into question the clarity of a feature of claim 1 (main request) which had not been amended together with the other features, thereby creating ambiguity (Article 84 EPC).
- XI. Thereupon, the respondents, with their letter of 6 December 2013, submitted eight sets of amended claims labelled main and first to seventh auxiliary requests.
- XII. In response to the comments made in the Board's communication, the appellant submitted the bibliographical data of D4 and (re)filed pages 266 and 267 thereof.

XIII. Claim 1 according to the respondents' new main request filed with letter of 6 December 2013 reads as follows:

*"1. A process for reducing the level of hydrogen sulphide in a liquid or gaseous hydrocarbon or in sewage gas by treatment of the liquid or gas with an H<sub>2</sub>S-scavenger product comprising the reaction product of*

- i) formaldehyde, with*
- ii) ethylene glycol, propylene glycol, glycerol, diethylene glycol, triethylene glycol, or urea,*

*said ethylene glycol, propylene glycol, glycerol, diethylene glycol, triethylene glycol or urea and said reaction product being free of basic groups."*

Claim 2 to 14 according to this request are directed to more specific embodiments of the process according to claim 1.

XIV. Oral proceedings were held on 20 December 2013. The debate focussed on the issues of admissibility of document D15 and inventive step over D1 as the closest prior art document, taking into account also D4, D7 and D8. At the end of the oral proceedings, the decision was announced.

XV. The appellant (opponent) requested that the decision under appeal be set aside and the patent be revoked.

XVI. The respondents (patent proprietors) requested that the patent be maintained on the basis of the claims of the main request or, in the alternative, one of the auxiliary requests 1 to 7, all requests submitted with the letter dated 6 December 2013.

XVII. The arguments of the appellant regarding the main request can be summarised as follows:

*New item of evidence*

- a) D15 was *prima facie* highly relevant against novelty, so that despite its late filing it should be admitted into the proceedings:
  
- b) As apparent from page 4, penultimate paragraph, D15 had to do with biocidal agents which inhibited the growth of microorganisms in liquid hydrocarbons, *inter alia* the sulphato-reducing bacteria which produced gases such as H<sub>2</sub>S. D15 thus taught how to inhibit their growth, whereby reduction of these bacteria implied that also the H<sub>2</sub>S content was reduced. The biocidal agents of D15 comprised hemi-acetals liberating formaldehyde such as ethylene glycol bis-semi-formal (Dascocide®). The process defined in Claim 1 of the main request was also applicable to liquid hydrocarbons and contained as the only step the definition of the reaction product which should reduce the hydrogen sulphide content, e.g. that between formaldehyde and ethylene glycol. Although there was no example in D15 showing what reduction of hydrogen sulphide was achieved by the biocidal treatment, the process of D15 inevitably led to a reduction of the hydrogen sulphide content, so that it was novelty destroying.

*Inventive step*

- c) The closest prior art was disclosed by D1, which concerned a method for sweetening natural gas.



- d) According to the patent in suit, the process according to the invention was suitable for:
- i) Reducing the level of hydrogen sulphide.
  - ii) Minimising precipitation of calcium carbonate as scale.
  - iii) Minimising the crystalline hydrate problem.
  - iv) Eliminating or minimising the formation of trithiane.
- e) D1 was not acknowledged in the patent in suit, which thus did not contain any comparative example over D1.
- f) It was apparent from page 4, lines 17-24, of the application as filed, that both mono- and polyalcohols were originally disclosed without any particular preference for polyalcohols. This fact was more particularly apparent from Figures 6A and 6B of the application as filed, showing that after 12 minutes no difference was attained by using polyalcohols instead of monoalcohols, i.e. they all showed the same efficiency at reducing hydrogen sulphide. Therefore, in this respect, no improvement whatsoever over D1 was apparent, let alone made plausible.
- g) As regards the formation of trithiane, or of crystalline hydrates, or of calcium carbonate precipitation, it did not arise in D1, as apparent from Figure 1 of D1, according to which Gas 16 was a gas to sale, i.e. could not contain precipitates. The appellant contested the new argument that imidazoline inhibitor was a dispersant present because there was a precipitate. It was not clear how a dispersant could work in a gas. In any case, the table in the

patent in suit showed that when ethanolamines, known dispersants, were used, better results were attained, and Claim 1 did not exclude the use of dispersants.

- h) Also, if the examples of the patent in suit were considered, in particular the test data, the following picture would result therefrom: the reference (triazine) chosen attained a 100% relative efficiency as H<sub>2</sub>S scavenger, without any comment on any precipitation; Product A (reaction product of 2 mols HCHO with 1.05 mols ethylene glycol) attained a worse result (76% relative efficiency) and some delayed precipitation; Products B (reaction product of 2 mols formaldehyde with 1 mol glycerol) and, more particularly, C (reaction product of 2 mols formaldehyde and 1 mol glucose) attained better results than Product A, although Product C no longer fell under Claim 1. Also, Claim 1 did not reflect the better results shown in the table arising from the use of NaOH or alkanolamine. Furthermore, as regards the comparative tests submitted during the examination phase, there was an effect due to the low pH of about 4 or 5, which reduced precipitation of calcium, as generally known. Thus, precipitation occurred, and it was not made clear what kind of precipitate was formed.
- i) No improvement over D1 had been convincingly shown. Therefore, starting from a process according to D1, the technical problem merely consisted in providing an alternative, not necessarily better process.

- j) Since the only distinction between the claimed subject-matter and the process of D1 was the use of di- and trialcohols instead of monoalcohols, such as methanol and isopropanol as in D1, in the formation of the reaction product, the question which arose was whether the skilled person would have obviously considered that the reaction products between formaldehyde and polyalcohols would have provided the same function as the known ones obtained from using monoalcohols, i.e. the scavenging of H<sub>2</sub>S.
- k) D4 taught that formals obtained from formaldehyde and mono-alcohols dissociated into formaldehyde and alcohols (under acidic conditions), as well as that the reactions of glycols with formaldehyde were similar to those with mono- alcohols, albeit cyclic or polymeric formals were thereby formed.
- l) Thus, the skilled person, knowing also that ethylene glycol formals were biocidal (this known fact was also acknowledged in the patent in suit), would obviously have considered using formals of glycols, firstly because they too provided a solution of formaldehyde in alcohol, secondly because they were biocidal, hence dual-use products. Moreover, there were also further considerations which would have motivated the skilled person, such as the lower boiling points of mono-alcohols and their flammability; hence formals of ethylene glycols were also safer.
- m) D8 did not dissuade the skilled person from using formals of polyalcohols in the method of D1, in which an alkaline solution was used, i.e. in which no formaldehyde was formed according to D4.

- n) Also, the mention in D1 that hydroxymethyl mercaptan was formed had to be considered in the context of Claim 1 at issue, which merely defined a reaction product of formaldehyde with glycols, without giving any structure of the thereby formed compounds.
- o) The argument of the respondents that some of the formals taught in D7 did not release formaldehyde was contested by the appellant

XVIII. The arguments of the respondents with regard to the main request can be summarised as follows:

*New item of evidence- Admissibility of document D15*

- a) D15 was filed without any explanation as to its lateness, and was not sufficiently relevant as regards novelty. It should, therefore, not be admitted into the proceedings:
- b) There was no evidence in document D15 that the composition disclosed was effective in killing hydrogen sulphide-producing bacteria, since in the examples thereof the presence of reductive sulphato bacteria could be calculated and was vanishingly small. Since the method did not discriminate among the bacteria present, the desired reduction in bacterial activity might have been attained even without the reductive sulphato bacteria being affected at all. Also, there was no disclosure in D15 of the treatment of a hydrocarbon that contained hydrogen sulphide. Furthermore, there was no disclosure in D15 of any reduction of the level of hydrogen sulphide. The disclosed killing of bacteria that could produce

hydrogen sulphide did not amount to a disclosure of a hydrogen sulphide level reduction. Thus, the process of D15 was not novelty destroying.

- c) If D15 were, however, admitted to the proceedings and considered to be sufficiently relevant to prejudice maintenance of the patent, the case should be remitted to the department of first instance.

*Inventive step*

- d) The closest prior art was disclosed by D1.
- e) The respondents acknowledged that the only difference over D1 consisted in the replacement of mono-alcohols, such as methanol or isopropanol, with poly-alcohols. They contested, however, that this difference did not provide any advantage.
- f) At oral proceedings reference was made to the patent in suit (page 2, lines 13-16, and page 11, lines 10-17, were referred to), arguing that the process of the invention was supposed to remove H<sub>2</sub>S while preventing trithiane formation and precipitation of calcium carbonate as scale.
- g) As acknowledged in D8 (page 3, last paragraph and page 4, first and second paragraphs were referred to) (D8 was published 6 months after the publication of D1), the process of D1 was suitable for reducing the level of hydrogen sulphide but led to the formation of trithiane. The formation of solids in the process of D1 was also apparent from the disclosed use of an imidazoline inhibitor (page 7, lines 22-25, was referred to), which was

a dispersant, and was obviously used because solids were formed. Thus, the process of D1 effectively reduced the level of H<sub>2</sub>S but also led to the formation of trithiane.

- h) The table on page 11 of the patent in suit made evident that no calcium carbonate precipitated as scale. Also the evidence presented before the Examining Division showed this lack of calcium carbonate precipitation. The term "some slight precipitation" concerned organic sulphur compounds of unidentified structure and meant that this precipitation did not affect the operation of the process. Also trithiane was not formed. Therefore, there was an improvement over D1.
- i) The mention of monoalcohols such as methanol and butanol in the application as filed occurred because when the application was written there was no awareness of D1.
- j) Therefore, the problem to be solved over D1 was still the provision of a process for scavenging H<sub>2</sub>S while avoiding formation of trithiane and precipitation of calcium carbonate.
- k) The skilled person starting from D1 and looking for a process for reducing hydrogen sulphide by using a reaction product which did not cause organic precipitation did not find any hint in the prior art invoked.
- l) In D1, the mechanisms of H<sub>2</sub>S removal was not made explicit by the mere mention that hydrogen sulphide was converted to hydroxymethyl mercaptan. D8 taught that aldehydes reacted with hydrogen

sulphide to *inter alia* form trithiane. D7 (page 55, lines 3-6, were referred to) warned about the reactivity of formaldehyde and disclosed formals (pages 62-63 were referred to) such as dioxolane, which neither was biocide nor released formaldehyde.

- m) D4 mentioned that the reactions of glycols with formaldehyde were similar to those of the mono alcohols with formaldehyde, but it disclosed nothing concerning the effects thereof on H<sub>2</sub>S removal.
- n) D7 merely addressed the microbiocidal properties of *inter alia* the formals obtained by reacting formaldehyde with glycols.
- o) D8 addressed the problem of avoiding formation of trithiane but proposed a different solution, namely the use of the products obtained by reacting a lower aldehyde such as formaldehyde with a lower alkanolamine.
- p) Hence, the cited prior art comprised no pointers towards the claimed process.
- q) This lack of pointers was likewise to be considered in case the problem solved were merely seen in the provision of an alternative process.

## Reasons for the Decision

1. The appeal is admissible.

### *Procedural issues - Non-admissibility of document D15*

2. Document D15 was only cited in substantiation of a novelty objection raised for the first time in the statement of grounds of appeal.
  - 2.1 As regards the relevance of this document, the following is observed:
    - 2.1.1 D15 (Claim 1) discloses a process for the inhibition of microorganisms in a petroleum distillate which comprises introducing into said distillate, a biocidal effective amount of a composition comprising *inter alia* a biocide for said microorganisms. In particular, the biocide (D15: Claim 5, page 8, second paragraph) can comprise ethylene glycol bis-semiformal. D15 (page 3, lines 8-20; page 4, penultimate paragraph) addresses the problems arising during storage of moist hydrocarbons, which undergo attack from certain microorganisms, for which hydrocarbons constitute a nutrients of choice. These may be, amongst others, sulphato-reductive bacteria and phototropic sulphobacteria. The metabolism of the microorganisms can lead to various problems, for instance the evolution of gas, in particular H<sub>2</sub>S, capable of causing the formation of a foam, which affect sealing, particularly in large capacity storage vessels.
  - 2.2 The novelty objection is based on the view that the biocide expressly mentioned in D15 (page 8, second paragraph), namely ethylene glycol bis-semi-formal (a



product of the reaction between formaldehyde and ethylene glycol) inhibits the growth of microorganisms which reduce the sulfates and produce H<sub>2</sub>S. Hence, because of the reduction of the amount of microorganisms, the level of H<sub>2</sub>S in the hydrocarbon treated will inevitably be reduced. Moreover, the level of H<sub>2</sub>S will also be inherently reduced by virtue of the injection of a formaldehyde-liberating product into the petroleum distillate.

2.3 For the board, these considerations are *prima facie* unconvincing the following reasons:

2.3.1 The biocide of D15 is used in particular for the the treatment of moisture-containing petroleum distillates, such as kerosene, petrol, gasoline (Claim 7; description, page 3, first paragraph, last sentence). D15 does not disclose that these products contain a level of hydrogen sulphide which should be reduced, nor that its method actually has any impact on the reduction of the level of H<sub>2</sub>S already present in the hydrocarbons, if any. Thus, it is not apparent that D15 directly and unambiguously discloses a treatment of H<sub>2</sub>S-containing hydrocarbons and leading to a reduction of its concentration.

2.3.2 The appellant alleged that the reduction of the population of the H<sub>2</sub>S-producing microorganisms inevitably led to a reduction of the level of H<sub>2</sub>S. However, Example 1 of D15 neither specifies the number of sulfate-reducing bacteria present, nor does it comprise an indication that the biocide used is actually effective in reducing also these microorganisms.

D15 thus at most discloses that the formation of further new H<sub>2</sub>S is reduced or prevented, i.e. that the level of H<sub>2</sub>S already present, if any, is merely prevented from increasing. Hence, not even Example 1 of D15 shows a reduction in number of the microorganisms producing H<sub>2</sub>S, let alone a reduction of the H<sub>2</sub>S level.

2.3.3 As concerns the alleged presence of formaldehyde inevitably liberated from the ethylene glycol bis-semi-formal biocide used, D15 does not mention any reaction possibly occurring between the ethylene glycol bis-semi-formal or of formaldehyde liberated therefrom and H<sub>2</sub>S already present in the hydrocarbon. The actual reduction of the H<sub>2</sub>S level already present in the stream, if any, is not a functional feature of the process as disclosed in D15.

2.4 Since D15 was filed late and is not *prima facie* novelty destroying, the Board decided not to admit it into the appeal proceedings (Articles 114(2) EPC and 12(4) RPBA).

*Procedural issues - Admissibility of the main request*

3. The present main request was filed after the issuance of the summons to oral proceedings. However, it merely addresses issues under Article 123(2) and 84 EPC raised in the communication of the Board issued in preparation for oral proceedings. This was not disputed by the appellant.

3.1 It essentially corresponds to the main request held allowable by the opposition division (see point III *supra*), the only additional amendment made being the replacement, in claim 1 according to the former, of "said alcohol" with "said ethylene glycol, propylene

*glycol, glycerol, diethylene glycol, triethylene glycol or urea".*

3.2 The amendment made is straightforward and does not raise any new, let alone complex issue. Moreover, the appellant did not object to the filing of this new request.

3.3 Accordingly, the Board decided to admit the main request at issue despite its very late filing (Articles 114(2) EPC and 13(3) RPBA).

*Main request - Amendments to the claims*

3.4 As regards Clarity (Article 84 EPC), the Board is satisfied that the objection raised in its communication in preparation for oral proceedings has been overcome by the amendment made to claim 1 previously on file.

3.5 The claims according to the main request held allowable by the opposition division were found to meet the requirements of Article 123(2) EPC. The Board is satisfied that also the amended claims according to the request at issue find a fair basis in the application as filed, as was already foreshadowed in detail in the Board's communication.

3.6 Since it was not disputed that the amendments in the claims at issue comply with the requirements of Articles 123(2) and 84 EPC, there is no need for giving more detailed reasons in this respect.

*Main request - Novelty*

4. In the present appeal proceedings, lack of novelty was

only invoked having regard to the disclosure of document D15. This document was, however, not admitted to the proceedings (see Points 2, *supra*).

Since the scope of Claim 1 at issue is narrower than that of Claim 1 held allowable in the decision under appeal, the Board has no reason to call novelty into question.

*Main request - Inventive step*

*The invention*

5. The invention concerns a process for the reduction or elimination of hydrogen sulphide from gaseous and liquid hydrocarbons and sewage gas, in particular from natural gas and liquid hydrocarbon streams (paragraph [0001] of the patent in suit).

*Closest prior art*

6. At the oral proceedings before the Board, it was common ground between the parties that D1 was the most appropriate starting point for the assessment of inventive step. Considering the similarities between the patent in suit and D1 in terms of process features and issues/problems addressed (see *infra*), the Board has no reason to take a different stance.
  - 6.1 More particularly, D1 (see Claim 1) concerns a method for collection and separation of natural gas, wherein a sour natural gas from a well head is passed through a knock out separator to remove free liquids, the treated gas being expanded through a choke into a low temperature separator to cool the gas sufficiently to condense water or hydrocarbon condensate therein and to

collect dry natural gas overhead therein. The method of D1 comprises the step of injecting into the flow line a sweetening solution consisting essentially of 10-50 % by weight of a low molecular weight aldehyde, or a low molecular weight ketone; 20-80% water; 10-50% methanol; 1-25% amine inhibitor; 0-5% sodium hydroxide or potassium hydroxide and 2-5% isopropanol, where the percentages total one hundred, and the pH is 6.0-14, said sweetening solution being continuously injected at a rate sufficient to react continuously with hydrogen sulfide to sweeten the natural gas.

6.2 In the method of D1 (see column 7, line 23; claims 3 and 4), the aldehyde is preferably formaldehyde and the sweetening solution can be introduced in an amount of 200-300 ppm thereof per 100 ppm of hydrogen sulfide in the flowing natural gas stream gas stream to reduce the hydrogen sulfide level to 4.0 ppm or less, whereby lower or higher concentrations of hydrogen sulfide can similarly be treated.

6.3 Thus, D1 (see column 3, lines 36-44) discloses a method for sweetening sour natural gas by atomizing a sweetening solution into the flowing stream of natural gas, the sweetening solution comprising an aqueous solution containing formaldehyde, methanol, isopropanol and sodium or potassium hydroxide. The amount of injected sweetening solution is sufficient to react with the hydrogen sulfide to convert it into a hydroxymethyl (or other lower molecular weight hydroxyalkyl) mercaptan and/or other sulfur compounds. The reaction is complete and effective, i.e. completely sweetens the sour gas.

6.4 The finding in the decision under appeal (point 4 of the reasons, page 5, third last paragraph) that the

additional presence of sodium or potassium hydroxide in the composition of D1 acts as a catalyst, so that a reaction product between formaldehyde and methanol or isopropanol is formed, is not in dispute. This finding is, moreover, confirmed by D4 (see page 265, second full paragraph, first sentence, *infra*).

- 6.5 Hence, D1 discloses a process with all of the features of Claim 1 as granted apart from the use of a reaction product of formaldehyde with polyalcohols as H<sub>2</sub>S scavenger. This was not in dispute either.

*The technical problem according to the respondents*

7. At the oral proceedings, the respondents maintained that, starting from the process disclosed in D1 taken as closest prior art, the technical problem was to provide an improved process for reducing the level of hydrogen sulphide with scavengers whilst avoiding or minimising the precipitation of calcium carbonate as scale and the formation of trithiane. This problem is mentioned in paragraphs [0008] and [0034] of the patent in suit.

*The solution*

8. The patent in suit as amended proposes to solve this problem by a process for the reduction or elimination of hydrogen sulphide as defined in Claim 1 at issue, which is characterised in that the hydrogen sulphide scavenger product used for treating the liquid or gaseous hydrocarbon or sewage gas comprises "*the reaction product of formaldehyde with ethylene glycol, propylene glycol, glycerol, diethylene glycol, triethylene glycol, or urea, and said reaction product being free of basic groups*".

*The alleged success of the solution*

9. Regarding the results achievable in comparison to previously known scavenging methods using triazine, the available evidence shows the following:

9.1 As regards scavenging efficiency

9.1.1 Example 2 of the patent in suit (Paragraph [0072] and Figure 6(B)) shows what advantages can be attained from the illustrated compositions A and B according to Claim 1 in comparison with monoethanolamine triazine, a standard scavenger, namely:

9.1.2 The results summarised in the table of paragraph [0072] of the patent in suit show that:

- (a) Product A (reaction product of 2 mols HCHO with 1.05 mols ethylene glycol) has a lower relative efficiency than triazine and shows "*some delayed precipitation*" of undefined nature. During the oral proceedings before the Board the respondent argued that the precipitation concerned unidentified organic sulphur compounds which did not affect the operation of the process.
- (b) Product A with 2% NaOH shows a better relative efficiency than triazine and "*slight delayed precipitation*".
- (c) Product A with 10% monoethanolamine shows an even better relative efficiency and "*insignificant precipitation*".
- (d) Product B (reaction product of 2 mols HCHO with 1 mol glycerol) shows a lower relative efficiency than triazine, whereby "*the reaction rate appears slow and the capacity is not reached*".
- (e) Product C (reaction product of 2 mols HCHO with 1 mol glucose) shows a better relative efficiency

than triazine and "*no precipitation at all*".  
However, product C is no longer encompassed by  
Claim 1 of the main request.

9.1.3 The diagram efficiency versus time of Figure 6B makes it apparent that after 8 minutes, monoethylene glycol hemiformal has a better H<sub>2</sub>S scavenging efficiency than triazine and all the other tested scavengers. After 12 minutes, the difference tends, however, to diminish.

9.1.4 Hence, as far as the achievable H<sub>2</sub>S scavenging efficiency is concerned, the claimed process is comparable but not necessarily better, at least not across the full breadth of claim 1 at issue, than previously known scavenging methods using triazines.

9.2 As regards precipitation of calcium carbonate scale

9.2.1 The patent in suit (paragraph [0006]) mentions that calcium carbonate precipitation as scale occurs when using triazines in the presence of sea/formation water. The results illustrated in the table of paragraph [0072] do not mention any calcium carbonate precipitation. The comparative examples provided during the examination phase (laboratory report filed with letter of 13 December 2004) show in particular, by means of a dynamic tube-blocking test P-MAC, that the reference triazine in contact with formation water resulted in scale formation after 5 minutes, whereas the use of Product A did not result in scaling for 60 minutes.

Thus, the Board accepts as plausible that the claimed scavenger leads to less calcium carbonate precipitation as scale than the use of triazine.



- 9.3 The same conclusion applies to the problem of crystalline hydrates formation, which is also acknowledged to arise when using triazine in the presence of water (paragraphs [0032] and [0033]).
- 9.4 As regards the invoked prevention or reduction of formation of trithiane, no specific example therefor is on file.
10. However, the method according to the closest prior art as disclosed by D1 was not taken into account in the application as filed, and on which the patent in suit was granted, at the time when the problem mentioned in the patent in suit was formulated. It is not in dispute that D1 too addresses and solves the problem of reducing the level of hydrogen sulphide in gaseous hydrocarbon streams. Hence, it has to be established whether or not the claimed method is actually more efficient in terms of H<sub>2</sub>S scavenging than the method of D1, and whether or not the precipitation of calcium carbonate as scale, the formation of crystalline hydrates and/or the formation of trithiane are reduced in comparison with the process of D1.
- 10.1 The examples contained in the patent in suit do not provide a comparison with the closest prior art process of D1. Hence, there is no evidence on file of a better scavenging efficiency of the claimed method over that of D1.
- 10.1.1 Also, it should be considered that in the application as filed, monoalcohols such as ethanol and n-butanol were also disclosed as possible reactants ii), i.e. within the same context of reducing hydrogen sulphide while minimising precipitation of carbonate and formation of hydrates and trithiane. As a case in

point, paragraph [0035] of the patent in suit mentions that butylformal has high efficiency and reduced liberation of free aldehyde. The diagram efficiency versus time of Figure 6A makes it apparent that after 12 minutes butylformal has the same efficiency as triazine.

10.1.2 Hence, no clear advantage is apparent that could be attributed to the use of reaction products of polyalcohols (in general) instead of monoalcohols. So, it must be reasonably assumed that the method of D1 also efficiently reduces the level of hydrogen sulphide.

10.2 D1 too addresses the problem of preventing crystalline hydrates formation (Figure 2, reference numeral 40; column 5, lines 57-64) and solves it by using dry ethylene glycol injection into the high-pressure, water-containing gas.

In this respect, no evidence for an improvement of the claimed method over the one of D1 is on file.

10.3 Regarding the allegedly decreased or delayed production of trithiane

10.3.1 The patent in suit (paragraph [0034]) stresses that trithiane, formed by the reaction of formaldehyde with hydrogen sulphide, is relatively insoluble in lower alcohols such as methanol and ethanol, whereas the product of the reaction of formaldehyde with ethylene glycol used in the inventive process reacts with hydrogen sulphide to produce a structure which is soluble in lower alcohols, thus leads to fewer problems. Claim 1 is, however, not restricted to the use of the specific reaction products mentioned in

subsequent paragraph [0035], which are stated to provide a reduced amount of free aldehyde. Claim 1 thus encompasses the use of reaction products for which no data are on file as regards their solubility in lower alcohols, thus their alleged suitability for lessening problems attributable to trithiane formation.

10.3.2 D1 is silent on the issue of trithiane formation. The argument of the respondent that the formation of solids in the method of D1 was apparent from the use of imidazoline inhibitor is not convincing, as according to D1 (e.g. Claim 7) the imidazoline inhibitor fulfils the function of water soluble oxidation and corrosion inhibitor.

10.3.3 However, D8 (paragraph bridging pages 3 and 4 and first full paragraph on page 4) expressly teaches that the method of D1 involves the use of aldehydes such as formaldehyde, which react rapidly with hydrogen sulphide to produce various types of addition products such as trithiane.

10.3.4 This has to be contrasted with the disclosure of D4 (*infra*) (page 264, penultimate sentence), according to which "the equilibrium in the hemiformal and polyoxymethylene hemiformals is far to the right so that the concentration of free monomeric formaldehyde in the liquid system is extremely low under neutral or alkaline conditions", which evidently applies to the liquid mixture injected in the method of D1, which contains formaldehyde and methanol at a pH of 6.8 to 14 (column 6, line 67). The formation of trithiane is, however, related to the amount of free formaldehyde, as acknowledged in both the patent in suit (paragraph [0034] and [0035]) and D8 (*idem, supra*).

10.3.5 Hence, considering also that the amount of free formaldehyde is not limited in Claim 1, there is no evidence of a improved inhibition of the formation of trithiane across the whole breadth of Claim 1.

10.4 As regards the formation of precipitates of incompletely defined identity

These precipitates might be sparingly soluble ringed sulphur compounds (as mentioned in paragraph [0041] and in the table of Example 2 of the patent in suit). Their avoidance, according to the patent in suit, requires the use of specific compounds, e.g. monoethanolamine, the presence of which is not, however, a requirement of Claim 1. Supposing that the precipitates mentioned in the table of paragraph [0072] of the patent in suit comprise trithianes, then the presence of monoethanolamine or another of said specific compounds would be necessary, but is not required by Claim 1 at issue.

10.5 As concerns calcium carbonate precipitation as scale

The patent in suit acknowledges that this problem arises when using triazines in the presence of formation water, which fact is not contested. The significance of the results of the comparative examples submitted with letter of 13 December 2004 were contested by the appellant in view of the low pH used, which had an inhibiting effect on calcium precipitation.

For the Board, it is plausible that the claimed scavenger is suitable for contributing to a reduction of the precipitation of calcium carbonate as scale. However, no evidence for an undisputable quantification

of any improvement thereof (compared to the process of D1) is on file.

- 10.6 Summing up, there is no evidence on file showing that, compared to the results achieved with the method of D1, H<sub>2</sub>S scavenging efficiency is improved or trithiane formation is more effectively reduced or delayed, let alone that some other effect obtained with the specific compositions A and B would amount to an improvement over the process of D1, and be achievable across the whole breadth of Claim 1.

*Reformulation of the technical problem*

- 10.7 Since the problem effectively solved cannot be formulated in terms of an improvement over the closest prior art D1, it has to be reformulated in a less ambitious way.
- 10.8 It can be seen in providing a further process for scavenging hydrogen sulphide contained in gaseous or liquid hydrocarbon or sewage gas whilst inhibiting or avoiding the formation of undesirable precipitates such as hydrates, trithiane and/or calcium carbonate scale.

*Success of the claimed solution*

Considering in particular the results over the known scavengers illustrated in the patent in suit, achieved using reaction products A and B as compared to the use of the monoethanolamine triazine scavenger disclosed in D8 (see test data in paragraph [0072] of the patent in suit), the Board accepts as plausible that this less ambitious problem is effectively solved by the processes according to Claim 1 at issue. This was also not in dispute between the parties.

*Obviousness*

11. It remains to be decided whether the claimed solution was obvious for the skilled person starting from the closest prior art D1 and aiming to solve the problem posed, in view of common general knowledge and the teachings of the prior art relied upon by the appellant.

11.1 D1 does not describe the function to be fulfilled by the reaction product between the mentioned mono alcohols and formaldehyde.

Since the method of D1 only teaches the injection of ethylene glycol as such into the gas for preventing formation of crystalline hydrates, i.e. not as a component of the mixture containing formaldehyde, D1 does not suggest the use of formals obtained from polyalcohols.

11.2 D4 generally discloses the reactions of formaldehyde with aliphatic hydroxy compounds and, more particularly, discloses the following elements of information:

(a) The equilibrium in the hemiformal and polyoxymethylene hemiformals is far to the right so that the concentration of free monomeric formaldehyde in the liquid system is extremely low under normal conditions (page 264, last paragraph, first sentence).

(b) Under neutral or alkaline conditions, hemiformals are substantially the sole product obtained when formaldehyde and alcohols are brought together (page 265, second full paragraph, first sentence).

(c) The reaction of glycols with formaldehyde are similar to those of the simple alcohols, with exception that cyclic and polymeric formals are obtained. In case of 1,2 or 1,3 glycols, cyclic formals are readily obtained on reaction in the presence of acidic catalysts (Paragraph bridging pages 267 and 268). Polymeric glycol formals can be prepared by reacting ethylene glycol and formaldehyde in the presence of an acidic catalyst (page 269, fifth paragraph).

It is immediately apparent that indications under points a) and b) above also apply to the composition used in the method of D1.

However, D4 does not contain any element suggesting the suitability of the reaction products of formaldehyde with polyalcohols for scavenging hydrogen sulphide in gaseous and liquid hydrocarbons or sewage gas.

11.3 Similar disclosure as in D4 can be found in D7 (see e.g. page 56, first paragraph), which relates to biocides, in particular formaldehyde releasing compounds as biocides. As regards ethylene glycolhemiformals and ethyleneformal (dioxolane), D7 (see page 63, lines 3-6) discloses that the formaldehyde content of ethyleneformal is not detectable with the Tannenbaum method and that dioxolane is not effective as antimicrobial.

Hence, D7 too does not contain any pointer towards the use, in a method as claimed, of the reaction products of formaldehyde with glycols as hydrogen sulphide scavengers.

11.4 D8 relates to methods for sweetening sour gas. It

generally discloses (page 3, lines 6-22) that for reducing the hydrogen sulfide content various chemicals may be added or injected "in line" to natural gas pipelines, or at the well head, separators, glycol units, coolers, compressors, etc., to provide contact with the natural gas. Materials used with such "in-line" injection systems include e.g., various aldehydes. Hydrogen sulfide reacts rapidly with the aldehyde compounds producing various types of addition products, such as polyethylene sulfide, polymethylene disulfide and trithiane.

11.4.1 In this respect, D8 (see paragraph bridging pages 3 and 4, and page 4, second and third full paragraphs) acknowledges the teachings of D4 and D1, the latter disclosing *"a method for the separation and collection of natural gas comprising the use of a sweetening solution ... consisting of an aldehyde, a ketone, methanol, an amine inhibitor, sodium or potassium hydroxides and isopropanol"*.

11.4.2 Still according to D8, the aldehydes (e.g. formaldehyde) are acknowledged as being effective in the reduction of the hydrogen sulfide level of natural gas and selective for sulfide compounds; they are however known to form trithiane compounds upon reaction with the sulfides. Trithianes are solids which do not easily dissolve and therefore clog gas lines. Also, aldehydes are unstable, temperature sensitive and tend to polymerize. Moreover, aldehydes are known carcinogens and environmental hazards. Accordingly, the use of aldehydes for sweetening natural gas has come under disfavor. Alkanolamines may also be used to sweeten sour gas streams, but alkanolamines are not selective in their reaction with hydrogen sulfide, which non-selectivity is not desirable in many



applications, so that also the usage of alkanolamines has also come under disfavor.

11.4.3 To overcome these problems, D8 proposes the use of the reaction product of a lower alkanolamine with a lower aldehyde, whereby the reaction product is a mixture of triazine and bisoxazolidine (see page 10, lines 13-16).

11.4.4 Therefore, D8 actually dissuades the skilled person from using the reaction products of aldehydes and alcohols, hence the method of D1, and instead points towards a method which differs from the claimed one in that it uses the reaction product between lower aldehydes and alkanolamines, i.e. of triazines, which is to be avoided according to the patent in suit.

11.5 It follows from the foregoing that the skilled person starting from D1 and trying to solve the technical problem posed is not induced by D1 itself or any of D4, D7 or D8 to use the reaction products of formaldehyde with the defined poly-alcohols or with urea, as a hydrogen sulphide scavenger in gaseous and liquid hydrocarbons or sewage gas.

11.6 The board concludes that the method according to claim 1 is not obvious in the light of the prior art invoked by the appellant.

11.7 In the Board's judgement, the subject-matters of claim 1 and, consequently, of claims 2 to 14 dependent thereon, thus involve an inventive step (Articles 52(1) and 56 EPC).

## Order

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

The case is remitted to the Opposition Division with the order to maintain the patent on the basis of the claims according to the main request submitted with the letter dated 6 December 2013, a description and figures to be adapted thereto where appropriate.

The Registrar:

The Chairman:



D. Magliano

B. Czech

Decision electronically authenticated