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D E C I S I O N
of 23 September 1994

Case Number: T 0347/92 - 3.3.2

Application Number: 84870093.6

Publication Number: 0130967

IPC: C01B 17/765

Language of the proceedings: EN

Title of invention:
Heat recovery from concentrated sulfuric acid

Patentee:
MONSANTO COMPANY

Opponent:
(01) Metallgesellschaft AG, Frankfurt/M
(02) Bayer AG, Leverkusen Konzernverwaltung RP Patente Konzern

Headword:
Heat recovery/MONSANTO

Relevant legal norms:
EPC Art. 54(1), 56

Keyword:
"Inventive step - prejudices in the art"

Decisions cited:
-

Catchword:
-

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Beschwerdekammern | Boards of Appeal | Chambres de recours

Case Number: T 0347/92 - 3.2.2

D E C I S I O N
of the Technical Board of Appeal 3.2.2
of 23 September 1994

Appellant: (Opponent 01)
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Representative:
-

Appellant: (Opponent 02)
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Respondent: (Proprietor of the patent)
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Decision under appeal: **Interlocutory decision of the Opposition Division of the European Patent Office dated 19 February 1992 concerning maintenance of European patent No. 0 130 967 in amended form.**

Composition of the Board:

Chairman: P. A. M. Lançon
Members: G. J. Wassenaar
R. L. J. Schulte

Summary of Facts and Submissions

I. European patent No. 0 130 967 was granted on 20 January 1988 in response to European patent application No. 84870093.6.

II. Notice of opposition was filed against the European patent by the Appellants. Revocation of the patent was requested on the grounds of lack of novelty and lack of inventive step.

During the procedure before the Opposition Division the following documents, remaining relevant for the present decision, were cited:

(1) Werkstoffe und Korrosion, 33 (1982), pages 254-26
(2) "Ullmanns Enzyklopädie der technischen Chemie", 4th edition 1982, vol. 1, pages 129-135
(7) Corrosion Engineering Bulletin 1983, INCO, pages 47-49 and 85-86
(8) Corrosion charts: Guide to Material Selection, R.K.Swandby, 1962, pages 186-201
(9) Proceedings of the British Sulphur Corporations Fifth International Conference, Part II, (1981), Paper 25, Sander ea.,pages 439-458
(10) Proceedings of the British Sulphur Corporations Fifth International Conference, Part II, (1981), Paper 26, Backhaus ea., pages 459-470.

III. The Opposition Division maintained the patent in amended form by a decision dated 19 February 1992, corrected by the decision dated 18 March 1992.

The amended main Claim numbered "D" reads as follows:

"D. A process for the manufacture of sulfuric acid, comprising the catalytic oxidation of sulfur dioxide to sulfur trioxide, contacting the gas comprising sulfur trioxide with sulfuric and acid having a concentration greater than 98% and less than 101% thereby absorbing sulfur trioxide in the sulfuric acid and generating heat of absorption, the absorption being carried out in a heat recovery tower to which said sulfuric acid is delivered at a temperature greater than 120°C, discharging sulfuric acid from the heat recovery tower and removing heat from said discharged sulfuric acid, characterised in that the sulfuric acid is discharged from the heat recovery tower at a concentration of between 99% and 101% at a temperature greater than 133.5°C, and in that heat is removed from said discharged sulfuric acid by transfer to water or steam in a heat exchanger, thus heating said water or steam to a temperature greater than 133.5°C, the discharged sulfuric acid being maintained at a concentration of between 99% and 101% and at a temperature greater than 133.5°C throughout the course of transfer of heat to said water or steam in the heat exchanger, the said heat exchanger being fabricated from an alloy selected from high nickel alloys and stainless steel alloys having an austenitic, ferritic or duplex structure, the composition of the alloy further corresponding to the following relationship:

$0.35\,(Fe + Mn) + 0.70(Cr) + 0.30(Ni) - 0.12(Mo) > 39$

where:

Fe = the weight percent of iron in the alloy,
Mn = the weight percent of manganese in the alloy,
Cr = the weight percent of chromium in the alloy,
Ni = the weight percent of nickel in the alloy,
Mo = the weight percent of molybdenum in the alloy.

IV. The Appellants lodged an appeal against this decision. Appellant O2 mentioned as ground lack of novelty over (1). Both Appellants mentioned as ground lack of inventive step. With respect to lack of inventive step, the Appellants argued in their written submissions and at the oral proceedings before the Board in essence that the only differences between the patent in suit and the closest prior art (1) are the new limits put upon the concentration of the acid and the temperature of the cooling water, that these new limits fall within the realm of (1) and that their choice was obvious in view of (2). In particular the Appellant O1 argued that it was known from (2) to use sulphuric acid with a concentration of at least 98% in the absorption tower and that by heat exchange with sulphuric acid of 160°C as indicated in (1), the cooling water is heated to a temperature above 133.5°C. It was further argued that document (8) discloses that corrosion resistant steels according to Claim 1 are resistant against 100% sulphuric acid up to 149°C and that document (7) discloses that some types of stainless steel can withstand 100,5 to 101,5% sulphuric acid at temperatures from 149 to 163°C so that neither the absorption conditions nor the choice of the steel involve an inventive step.

V. The Respondent disagreed with these submissions and expressed the view that the prior art would provide a clear prejudice against using acid temperatures high enough to heat cooling water in a heat exchanger above 133.5°C because it was believed that the available stainless steels and high nickel alloys, which were used in the art for the cooling of sulphuric acid, would not be suitable because of unacceptable corrosion rates. To make this prejudice credible, reference was made to (9) and (10). The Patentee had surprisingly found that by choosing the claimed high acid concentration and

selecting the claimed alloys, cooling water of a temperature above 133.5°C, which corresponds to a steam pressure of at least 300 kPa, could be produced with acceptable absorption performance and acceptable corrosion rates.

VI. The Appellants requested that the decision under appeal be set aside and that the patent be revoked.

VII. The Respondent requested that the appeal be dismissed and the patent be maintained.

Reasons for the Decision

1. The appeal is admissible

2. Allowability of the amendments

The main Claim contains a temperature value (greater than 133.5°C) which was not explicitly disclosed in the application as originally filed. This figure can however be unambiguously derived from the original disclosed steam pressure of 300 kPa mentioned on page 13, lines 4/5 of the description as originally filed. Since the steam originates from boiling hot water in the heat exchanger, the steam must be considered as saturated steam, in which case there exists a unique relationship between temperature and pressure. Thus the amendment is adequately supported by the original description. The amended temperature range is narrower than the range as granted, the amendment does not extend the protection conferred. The amendments therefore fulfil the requirements of Articles 123(2) and (3). This was not contested by the Appellants.

3. *Novelty*

None of the prior art documents considered in the present proceedings discloses a process for the manufacture of sulphuric acid with all the features of present Claim D. Although Appellant O2 did not deny that some of the figures of Claim D such as the minimum acid concentration of 98% and the minimum watertemperature of 133.5° were not disclosed in (1), he maintained that these figures were inherently disclosed because the skilled man would choose these conditions when applying the teaching of (1). In the opinion of the Board, such a reasoning applies to inventive step but not to novelty unless the performance of the process of (1) necessarily implies conditions within the claimed ranges. The latter is not the case here as will be explained in more detail in the following paragraphs. Thus Claim D and its more limited subclaims must be regarded as novel.

4. *Inventive step*

4.1 With respect to the invention as claimed, (1) is regarded as constituting the closest prior art. It relates to a process for the manufacture of sulphuric acid, whereby sulphur trioxide is contacted with sulphuric acid to increase the concentration of the acid by absorbing the gaseous sulphur trioxide, whereby heat is generated. The absorption is carried out in absorption towers (heat recovery towers) from which hot concentrated sulphuric acid is discharged. The heat is recovered from the discharged acid by heat exchange with water from a steam boiler to produce after further compression low-pressure steam (Figure 6). The concentration of the acid in the absorption and heat recovery system is generally indicated as being of 93 to 99% and a temperature range of 120 to 160°C is said to be suitable for heat recovery. It is further indicated

that under these operating conditions stainless austenitic steels, e.g. No. 1.4541 and 1.4591, can be used for the heat-exchanger provided that the steel is anodically protected (abstract and page 255, right column). In the only complete process design, Figure 6, an acid temperature of 160°C is indicated at the entrance of the cooler and a temperature of 130°C at the outlet. Under the conditions indicated in Figure 6 it would appear physically impossible to obtain a water temperature in the cooler above 130°C. In practise a temperature difference between the water/steam side and the acid stream of at least 10°C would be required to obtain a reasonable heat transfer efficiency so that the practical upper limit of the water/steam side according to said Figure 6 is 120°C.

4.2 As convincingly put forward by the Respondent, a disadvantage of the heat recovery system of (1) is that the steam pressure which can be developed with acid between 120 and 160°C is too low for practical use and must be upgraded by one or more compressor steps in order to obtain a suitable pressure of at least 300 kPa; this necessity is shown by the illustration of a vapour compressor in the plant of Figure 6 of (1).

Starting from (1), the technical problem underlying the invention is regarded as improving the heat recovery in the acid cooling system of the absorption tower of a sulphuric acid plant. In accordance with Claim D, the solution of this problem consists in feeding the acid into the absorption tower (heat recovery tower) at a concentration greater than 98%, maintaining the concentration of the discharged acid between 99 and 101% and heating the water or steam in the heat-exchanger to a temperature greater than 133.5°C. Example 2 of the patent in suit, which discloses a generated steam pressure of 450 kPa, which corresponds to 153°C,

confirms that the above-stated problem has actually been solved by the invention as claimed.

4.3 According to the Appellants the said problem was already solved by (1) because the acid temperature of up to 160°C mentioned in (1) would be sufficient to heat cooling water above 133.5°C. This allegation was however not supported and cannot be accepted by the Board for the following reasons. Although it is perhaps theoretically possible to heat water above 133.5°C with acid of 160°C, this is not in the context of (1) a realistic option to the skilled man. What can be obtained in a real acid plant is illustrated in Figure 6 of (1), according to which the water temperature must be below 120°C for the reasons given above under point 4.1. This view is confirmed by the available prior art such as (9) and (10) relied upon by the Respondent in relation to the technical problem. Although the skilled man was obviously trying to recover the heat at a temperature as high as possible to obtain the highest possible steam pressure and acid temperatures up to 160°C were taken into consideration, the best results mentioned with equipment similar to that of (1) is only 1.5 bar, corresponding to a water temperature of 112°C; cf. (9), pages 452, 454 and (10) pages 461, 466 and 470.

4.4 Since (1) clearly teaches a heat recovery at water/steam temperatures below 130°C it remains to be investigated whether the available prior art would give the skilled man an incentive to try to recover heat at higher temperatures with conventional steel or nickel alloys. Document (2), which is a part of a technical encyclopedia relating to the manufacture of sulphuric acid and illustrates the general knowledge of the skilled man with respect to the absorption of sulphur trioxide, mentiones as highest acid temperatures 130 to 140°C (page 135). The same range is mentioned in (9)

(page 452). A temperature of up to 160°C is mentioned in (10) on pages 461 and 466 if anodic protection is used. Both (9) and (10) are articles relating to the heat recovery in sulphuric acid plants, which were presented in an international conference of the British Sulphur Corporations and should be known to skilled persons in the relevant field. The remaining prior art documents cited in the opposition procedure do not relate to heat recovery by heat exchange to develop low pressure steam. Thus the prior art does not provide any incentive to increase the acid temperature above the limit of 160°C mentioned in (1), which is insufficient to heat cooling water above 133.5°C on an industrial scale.

4.5 In connection with the corrosion resistant steels, Appellant O1 relied on documents (7) and (8), both dealing with materials used in contact with sulphuric acid and known as such to persons skilled in the art. However, in view of the Board, the available corrosion tables as disclosed in (7) and (8) would also discourage the skilled man to develop a heat exchange system on the basis of nickel or steel alloys operating with acid temperatures substantially above 160°C in order to heat the cooling water above 133.5°C. The highest temperature at which steel can be used in contact with sulphuric acid is according to Figure 14 (page 194) of (8) below 149°C (300°F). The highest temperature mentioned in (7) for acceptable corrosion rates of stainless steel in contact with sulphuric acid with a concentration below 101% is 163°C (page 49, Table XLIV). The corrosion rates are however based on static tests. The skilled man would realize that under the conditions of an acid cooler with running acid the tolerable temperature will be substantially lower. But even an acid temperature of 163°C is not sufficient to heat cooling water above 133.5°C on an industrial scale.

4.6 The other citations not mentioned above are still farther removed from the subject matter of the patent in suit and are without relevance for the inventive step considerations.

Thus the available prior art, published immediately before the priority date of the patent in suit, provides in the Board's view a clear prejudice against the possibility of running a nickel or steel based acid cooler at acid temperatures sufficient high to heat cooling water above 133.5°C. The Patentee has surprisingly found that working under the conditions of Claim D, i.e. within a very limited acid concentration range and a specific choice of nickel and steel alloys, it is possible to overcome the prejudice and to use acid with temperatures substantially above 160°C. According to Example 2 of the specification an acid temperature of 201°C can be used.

The finding of a relatively small operating window in an area which, according to the teaching of the most recent publications was considered inaccessible, cannot be considered obvious to a person skilled in the art.

5. It follows from the foregoing considerations that Claim D is not only new but also involves an inventive step in the meaning of Article 56 EPC. Since Claims D1 to D6 are all subclaims dependent upon main Claim D, their allowability follows from that of Claim D without the need for any separate considerations for novelty and inventive step.

Order

For these reasons it is decided that:

The appeals are dismissed.

The Registrar: The Chairman:

P. Martorana P. A. M. Lançon