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File Number: T 148/91 - 3.3.1
Application No.: 83 303 634.6
Publication No.: 0 098 108
Title of invention: Detergent composition

Classification: C11D 3/39

D E C I S I O N
of 1 September 1992

Proprietor of the patent: Unilever PLC
Unilever N.V.

Opponent: 01) Henkel Kommanditgesellschaft auf Aktien
02) The Procter & Gamble Company
03) Interlox Chemicals Limited
04) Colgate-Palmolive Company

Headword: Detergent composition/UNILEVER

EPC Articles 83, 54 and 111(1)

Keyword: "Sufficiency (yes) - well known parameter"
"Novelty - remittal to Opposition Division"



Case Number : T 148/91 - 3.3.1

D E C I S I O N
of the Technical Board of Appeal 3.3.1
of 1 September 1992

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(Proprietors of the patent)

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Decision under appeal : Decision of Opposition Division of the European
Patent Office of 15 October 1990, with written
reasons issued on 13 December 1990, revoking
European patent No. 0 098 108 pursuant to
Article 102(1) EPC.

Composition of the Board :

Chairman : K.J.A. Jahn
Members : R.W. Andrews
J.-C. Saisset

Summary of Facts and Submissions

- I. European patent No. 0 098 108 in respect of European patent application No. 83 303 634.6, which was filed on 23 June 1983, was granted on 29 April 1987 (cf. Bulletin 87/18).
- II. Notices of opposition, which were filed on 20, 22, 23 and 29 January 1988, requested the revocation of the patent on the grounds of insufficiency and lack of novelty and inventive step.
- III. By a decision delivered orally on 15 October 1990, with written reasons issued on 13 December 1990, the Opposition Division revoked the patent on the ground that, in the absence of any indication of the gas used for the measurement of the specific surface area of the sodium perborate monohydrate by gas adsorption, the disclosure of the disputed patent was insufficient.
- IV. An appeal was lodged against this decision on 12 February 1991 with payment of the prescribed fee. A statement of grounds of appeal was filed on 6 April 1991. At the commencement of the oral proceedings held on 1 September 1992, the Appellants requested that the Board decide not only the issue of insufficiency but also those of novelty and inventive step. Respondents 01, 02 and 04 had no objections to the Board deciding all the outstanding issues. Respondent 03, who did not file any written submissions, was duly invited to the oral proceedings. However, this Respondent chose not to be represented at them.

The following documents are relevant to the present decision:

- (1) EP-A-0 038 591
- (2) EP-A-0 053 859
- (4) Zeitschrift für Anorganische und Allgemeine Chemie, Vol. 374, pp. 125-146, 1970
- (5) GB-A-1 520 127
- (18) Journal of the American Chemical Society, Vol. 60(2), pp. 309-319, 1938
- (19) DIN 66131 (Determination of specific surface area of solids by gas adsorption using the B.E.T. method)
- (23) Declaration of P.F. Garner-Gray of 13 September 1990
- (25) The brochures entitled Quantochrome, Monosorb, Degassers, Quantosorb and Flowsorb II 2300
- (28) Methods for the Determination of Specific Surface of Powders Part I. Nitrogen adsorption (B.E.T. method) BS 4359, Part I, 1969.

Document (28) was submitted by the Appellant during the course of oral proceedings.

- V. The Appellants contended that the concept of specific surface area of solid particulate material in the context of detergent manufacture was well known and that there were several automatic and fully automated surface area analysers commercially available. The Appellants have also argued that documents (25) and (28) clearly demonstrated that the adsorbate employed for surface area studies is almost universally nitrogen. Therefore, since the method of accurately measuring the specific surface area of a particulate material is so apparent that it can be carried out by the skilled person using his common general knowledge, the disputed patent discloses the invention in a sufficiently clear and complete manner.

- VI. The Respondents maintained that the disclosure of the disputed patent is insufficient insofar as it contains no indication of how the sample is prepared for measurement,

the partial pressures used during the measurement, the adsorbate used, and the mathematical model applied to calculate the results. Thus, document (4) which is an important article dealing with sodium perborate, clearly shows that different values of specific surface area were obtained when n-butane and nitrogen were used as the adsorbate. The disclosure of document (25), which indicates that gases other than nitrogen may be used as adsorbates, is of a very general nature and gives no special guidance regarding which gases should be used for which substances or the advantages or disadvantages of any particular combination of adsorbate and adsorbent. In the absence of such guidance the skilled person would turn to document (4).

Respondent 02 also contended that the disclosure was insufficient in the absence of any indication of the stage of manufacturing process the surface area of the sodium perborate monohydrate (PBM) should be measured.

The Respondents alleged that in the absence of such detailed information the expression "more than $7\text{m}^2/\text{g}$ " was meaningless.

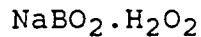
With respect to novelty, the Respondents argued that the disclosure of document (1) combined with common general knowledge as represented by documents (4) and (5) destroyed the novelty of the claimed subject-matter.

- VII. With respect to novelty, the Appellants emphasised that the PBM should have a specific surface area greater than $7\text{m}^2/\text{g}$ at the point of dosing. Therefore, it is not the specific surface area of the PBM as received, but its specific surface area after it has passed through the normal factory fluid lift transport system used in the manufacture of detergent compositions in bulk.

VIII. The Appellants requested that the decision under appeal be set aside and that the patent be maintained on the basis of the set of Claims 1 to 9 filed during oral proceedings. The only independent claim of this set of claims reads as follows:

"A solid detergent composition containing

- (a) from 6 to 40% by weight of a detergent active material; wherein the content of soap, if present, is from 0.5 to 25% by weight;
- (b) from 15 to 50% by weight of an alkalimetal aluminosilicate material as a detergency builder;
- (c) from 2 to 50% by weight of sodium perborate monohydrate having the empirical formula



- (d) 0.5 to 10% by weight of an activator for said perborate characterised in that the sodium perborate monohydrate is in particulate form having a specific surface area of more than $7\text{m}^2/\text{g}$."

The Respondents requested that the appeal be dismissed.

IV. At the conclusion of the oral proceedings the Board's decision to remit the case to the first instance for further prosecution on the basis of these claims was announced.

Reasons for the Decision

1. The appeal is admissible.
2. There are no objections under Article 123 EPC to the present version of the claims. In particular, Claim 1 is based on Claims 1, 3 and 5 as filed and granted in combination with page 2, lines 15 and 16, page 3, lines 24 to 26, page 5, lines 30 and 31, page 7, lines 10 to 12 and page 10, lines 19 to 21 of the originally filed application (cf. page 2, lines 32 and 33 and 57 and 58, page 3, lines 33 and 34 and page 4, line 1 and lines 59 to 62 of the printed patent specification). Claims 2 to 9 correspond to Claims 2, 4, 6 to 10 and 12 as filed and granted.
3. The first issue to be decided is whether the disclosure in the disputed patent that the specific surface area of the PBM is measured by gas adsorption is sufficient to enable the invention to be carried out by the skilled person as required by Article 83 EPC (cf. page 6, lines 27 and 28 of the printed patent specification).
 - 3.1 In the Board's judgment, the person skilled in the art of determining the specific surface area of particulate material reading that a certain specific surface area had been measured by gas adsorption would mentally add the phrase "according to the B.E.T. method". This is confirmed, for example, by the heading on page 5 of document (28) which reads Part I. Nitrogen adsorption (B.E.T. method). Additionally, document (25) discloses that the application of the Langmuir model for determining surface areas of solid materials has been superceded by the BET model (cf. page 3, second paragraph). Therefore, the omission of any reference to the B.E.T. method does not in itself render the disclosure of the patent in suit insufficient.

3.2 Furthermore, in the Respondents' opinion, the failure to indicate the gas used as the adsorbate in the determination of the specific surface area renders it impossible to obtain meaningful and reproducible results.

It is true that gases other than nitrogen may be used as the adsorbate provided their behaviour in contact with the solid under investigation conforms with the mathematical model used to analyse the data. Thus, documents (19) and (25) mention the possible use of argon, krypton, xenon, carbon dioxide, carbon monoxide, ethane and n-butane (cf. document (19), page 5, paragraph 6.1; document (25), pages 9 and 18, Appellant's numbering). Document (4), which is an important article in the sodium perborate field, discloses the measurement of the specific surface area of PBM I and PBM II using nitrogen and n-butane as the adsorbate (cf. page 134, paragraph 5). From the results reported in this paragraph it is clear that the specific surface varies according to the adsorbate used. Thus the cited values for PBM I and PBM II using nitrogen are $10\text{m}^2/\text{g}$ and $5\text{m}^2/\text{g}$ respectively, the corresponding values obtained using n-butane as the adsorbate are $12\text{m}^2/\text{g}$ and $8\text{m}^2/\text{g}$.

Nevertheless, it is clear from documents (19) and (25) that nitrogen is the preferred and most frequently adsorbate used in the determination of specific surface area (cf. document (19), page 5, paragraph 6.1; document (25), page 4, fourth to second line above equation (7); page 14, first line of the paragraph headed "Operation of the Quantasorb"; and page 18, right-hand column under "Gas"). Moreover, the British Standard (document (28)) is based on the use of nitrogen as the adsorbate.

Although n-butane was used as an adsorbate in document (4) to determine the specific surface area of PBM, in the

Board's judgment, this would not lead the skilled person to consider using it instead of nitrogen, if, as in the present case, no specific adsorbate is mentioned. This finding is based on the fact that the skilled person is aware that the amount of butane adsorbate on charcoal is decidedly smaller than would be expected on the basis of the estimated cross-sectional area of the n-butane molecule (cf. document (18), left-hand column of page 318, 8th to 12th lines below Figure 5) and that the cross-sectional area of n-butane relative to nitrogen as a function of B.E.T. C constant apparently changes from one adsorbent to another because at low C values the cross-sectional area is a function of the area covered by the adsorbate plus the distance swept out during its short residence time on the surface (cf. document (25), first complete paragraph on page 5).

Further indications for not using n-butane as the adsorbate are that it is considered to behave as a two-dimensional non-ideal gas on the surface of the adsorbent (cf. the above-mentioned paragraph in document (25)) and that the adsorbate should be relatively spherical in shape (cf. document (19), paragraph 6.1)).

Respondent 01 argued that according to page 2198 O of Gmelins Handbuch der Anorganischen Chemie, 8th Edition, Sauerstoff, Lieferung 7, 1966, a mixture of 90% hydrogen peroxide with n-pentane, 1-pentene, cyclohexane or toluene does not react, even when boiled under reflex (cf. paragraph headed "Kohlenwasserstoffe"). Therefore, the skilled person would consider that n-butane is inert to PEM. However, the skilled person is aware that even if only a few parts per million of n-butane were oxidised by PEM, the results of the determination of the specific surface area would be affected, therefore, he would be

very hesitant to consider using n-butane as adsorbate even in the light of the disclosure of document (4).

In the light of the above, the Board is satisfied that, unless there were reasons for not using nitrogen as the adsorbate, for example, with samples having specific surface areas below $0.5\text{m}^2/\text{g}$ the skilled person would consider using an inert gas such as krypton (cf. document (28), paragraph bridging pages 5 and 6), the skilled person would automatically use nitrogen as the adsorbate in the determination of specific surface area by gas adsorption.

Therefore, the failure to indicate that nitrogen was the gas used in the determination of the specific surface areas referred to in the disputed patent is not detrimental to the sufficiency of the disclosure.

3.3 Before the specific surface area of a sample can be measured the sample must be conditioned. This conditioning can be accomplished by vacuum pumping or purging with an inert gas. Naturally, care must be taken during the sample conditioning so as to avoid any change in the surface morphology of the sample. It is known for example, that such processes as melting, sintering, dehydration and decomposition can markedly change the surface properties (cf. document (25), the sentence bridging the left-hand and right-hand columns on page 2).

3.4 The skilled person is aware that PBM is a desiccant and that its surface area decreases with pick-up of moisture. Therefore, the skilled person wishing to measure the specific surface area of PBM would take steps to minimise its exposure of PBM to moist air and would ensure that the conditions under which the vacuum degassing or purging are carried out are such as to avoid any change in the water

content of the sample. It is well within the competence of the skilled person to select those conditions which give reproducible results within the limits of the experimental error for the particular instrument used. Therefore, it is not necessary to give details of these conditions to comply with the requirements of Article 83 EPC.

- 3.5 In the interests of accuracy, the skilled person would employ the multipoint rather than the single point determination using a modified B.E.T. equation. The multipoint method requires that a linear plot of adsorption data be made, usually from three adsorbate weight determination. Since a linear plot is generally only obtained in the range of relative pressures P/P_0 of from about 0.5 to 0.35 (P is the adsorbate pressure and P_0 is the saturated equilibrium vapour pressure of the adsorbate at the temperature at which the measurement is made), it is unnecessary to specify the partial pressures under which the measurement is made.
- 3.6 Having regard to the fact that the measurement of specific surface area using the multipoint B.E.T. nitrogen adsorption method is well known and widely used, it is not necessary to specify the other conditions such as the temperature at which it is measured or that special guidance should be given for the measurement of the specific surface area of a particular substance.
- 3.7 Since the specific surface area of PBM changes due to the uptake of water it was alleged that the disclosure was insufficient insofar as there was no indication in the disputed patent when the specific surface area was to be measured. It was also alleged that the wording of the claim could imply that the specific surface area had to be more than $7\text{m}^2/\text{g}$ after it had been added to the other ingredients of the composition.

However, in the Board's judgment, the skilled person would realise in the absence of any indication to the contrary and in view of page 5, lines 50 to 53 and Examples 1, 2 and 4, that, in order to obtain the claimed benefits, the specific surface area of PBM must be more than $7\text{m}^2/\text{g}$ immediately before it is added to the detergent base powder.

3.8 The present case is distinguished from that decided in the appeal T 241/89 of 14 August 1990 (unpublished) of this Board since the parameter (not more than $3\text{mg}/\text{kg}$ of reactive titanium (IV)) relied on to differentiate the claimed compositions from the prior art ones was unknown, whereas specific surface area is an extremely well known concept in powder technology.

3.9 Therefore, in the Board's judgment, the disclosure of the disputed patent is sufficient to enable the skilled person to put its teaching into practice.

4. The next issue to be considered is whether the subject-matter of the present Claim 1 is novel.

4.1 Since it was concluded by the Respondents during the oral proceedings that the claimed subject-matter was novel with respect to the disclosure of document (2) and the Board agrees with this conclusion, it is not necessary to discuss this document in detail.

4.2 Example VI of document (1) describes a detergent composition containing 35% by weight of an alkali metal aluminosilicate as a detergency builder, 30% by weight of sodium tallowate, 10% by weight of sodium carbonate, 6% by weight of coconut alkyl dimethylamine oxide, 15% by weight of PBM, the balance being water and miscellaneous.

Thus, this composition differs from those of the present Claim 1 insofar as the specific surface area of the PBM is not indicated, it contains 30% by weight of soap and it does not contain an activator for the PBM.

- 4.3 However, if it can be established that it was common general knowledge at the claimed priority date of the disputed patent that PBM had a specific surface area greater than $7\text{m}^2/\text{g}$, it is the established jurisprudence of the Boards of Appeal that this common general knowledge can be combined with the disclosure of document (2), i.e. the feature PBM having a specific surface area of more than $7\text{m}^2/\text{g}$ would be considered to be disclosed in this document.

In order to establish that this was in fact common general knowledge Respondents 01 and 02 referred to documents (4) and (5). As previously mentioned, document (4) discloses a PBM (Sample PBI) with a specific surface area of $10\text{m}^2/\text{g}$ as measured by nitrogen adsorption (cf. page 134). However, this article represents the results of a scientific investigation into the structure and properties of sodium peroxyborate hydrates and this specialist literature cannot be equated with textbook knowledge and, therefore, does not represent common general knowledge.

Document (5) is a patent specification and in accordance with the established case law of the Boards of Appeal, a patent specification may only be considered to represent common general knowledge in exceptional circumstances. The decision T 51/87 "Starting compounds/MERCK" (cf. OJ EPO 1991, 177) considered that in a field of research which is so new that technical knowledge is not yet available from textbooks, special consideration may prevail on whether or not the common general knowledge of the skilled person may include a patent specification. However, this is in

contrast to the situation in classical technological fields such as those underlying the subject-matter of the appeals T 171/84 "Redox Catalyst", OJ EPO 1986, 95 and T 206/83 "Herbicides" OJ EPO 1987, 5. Therefore this document, which discloses a specific surface area of the PBM obtained by the process claimed therein of $7.8\text{m}^2/\text{g}$ as measured according to the B.E.T. method (cf. page 2, lines 19 and 20 and the Example), clearly cannot be considered to be common general knowledge and cannot be combined with the disclosure of document (1).

Furthermore, Respondent 01 alleged that a passage in document (5) provided evidence that PBM having a specific surface area of more than $7\text{m}^2/\text{g}$ is part of the skilled person's common general knowledge. This passage reads "Monohydrate granulate prepared by dehydrating sodium perborate tetrahydrate with an inert gas, often at a supply temperature in the range 120 to 140°C , the bed of hydrated sodium perborate being maintained always at a temperature below its melting point, and exhaust gas temperatures of often below 55°C , normally has a specific surface area of 10 to $14\text{m}^2/\text{g}$...". However, since this range of specific surface area results from a particular process, the Board does not consider that this passage supports the allegation that it was common general knowledge that PBM had a specific surface area greater than $7\text{m}^2/\text{g}$ at the claimed priority date.

- 4.4 According to document (23) two Degussa "UK Standard Grades" of PBM which were received by the Appellants in 1982, were found to have specific surface areas of $7.6\text{m}^2/\text{g}$ and $7.82\text{m}^2/\text{g}$ (cf. page 3, first paragraph). The Appellants agreed during the oral proceedings that 1982 in this instance had to be interpreted in the light of paragraph 6 on page 2, where it is stated that samples of PBM were received unprompted from commercial companies in

the period 1980 to mid 1982. From this the Board concludes that the above-mentioned samples were capable of being used before the claimed priority date of the disputed patent and, therefore, that a composition according to Example VI of document (1) containing PBM having a specific surface area greater than $7\text{m}^2/\text{g}$ was made available to the public as a technical teaching and thus part of the state of the art for the purpose of Article 54 EPC.

The Appellants argued that if the above-mentioned specific surface areas are those of the samples as received, and if these samples were passed through the normal factory fluid transport system, their specific surface areas at the point of incorporation in the detergent base powder would be less than $7\text{m}^2/\text{g}$. However, the present Claim 1 is to a composition per se and is in now way restricted to compositions manufactured on an industrial scale. In the Board's opinion, there is nothing to prevent the skilled person using a sample of PBM as received or after storage in a sealed container in the compositions of document (1) and thereby obtaining a detergent composition containing PBM having a specific surface area of more than $7\text{m}^2/\text{g}$.

The Appellant stressed that only two of the seven samples referred to in paragraph 6 of document (23) had a specific surface area of more than $7\text{m}^2/\text{g}$. However, it is sufficient for the purposes of Article 54 EPC if only one batch of PBM having a specific area of more than $7\text{m}^2/\text{g}$ was in fact available to the public before the claimed priority date and it is not necessary as a matter of law that the public be aware of PBM's specific surface area (cf. T 381/87 "Publication/RESEARCH ASSOCIATION", OJ EPO 1990, 213, in particular paragraph 4(4)b).

4.5 The Appellants considered that the fact that the maximum amount of soap which may be present in the compositions according to the present Claim 1 is 25% by weight rendered them novel with respect to document (1) since the composition of Example VI of this document contains 30% by weight of soap.

However, in accordance with the established jurisprudence of the Boards of Appeal (cf. for example, T 4/82 "Purification of sulphonic acids/EXXON", OJ EPO 1983, 498, paragraph 4; T 198/84 "Thiochloroformates", OJ EPO 1985, 209, paragraph 4; T 124/87 "Copolymers/DUPONT", OJ EPO 1989, 491, paragraph 3.2 and T 666/89 Headnote published OJ EPO 6/1992, paragraph 5) it is necessary to consider the whole content of a citation when deciding the question of novelty. Therefore, in applying this principle, the evaluation must not be confined solely to a comparison of the claimed subject-matter with the examples of the citation but must extend to all the information contained therein.

In the present case this means that the amount of soap that may be present in these prior art compositions may be as low as 5% by weight (cf. Claim 1, paragraph b)).

4.6 The compositions in accordance with the present Claim 1 must also contain from 0.5% to 10% by weight of an activator for the PBM. According to page 10, lines 13 to 17 of document (1), activators for PBM may also be present in these prior art compositions. However, document (1) provides no indication of the amount of the activator to be incorporated into the compositions.

During the course of oral proceedings, the representative of Respondent 01, who said he had worked for ten years in the detergent field and, therefore, should be considered

as an expert, maintained that it was common general knowledge that the amount of activator in a detergent composition was dependent on the amount of PBM present. Therefore, levels of activator between 0.5% and 10% by weight must be considered as being part of the common general knowledge of the person skilled in the art of formulating detergent compositions.

Since the Board itself is not in a position to decide whether such levels may be considered to be common general knowledge which could be combined with the disclosure of document (1), it considers it necessary to remit the case to the Opposition Division in order that the parties may provide evidence in this respect.

Order

For these reasons, it is decided that:

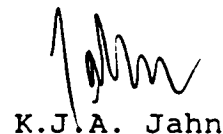
1. The decision under appeal is set aside.
2. The case is remitted to the first instance for further prosecution on the basis of the set of Claims 1 to 9 filed during oral proceedings.

The Registrar:



E. Gorgmaier

The Chairman:



K.J.A. Jahn