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D E C I S I O N
of 26 March 2004

Case Number: T 1288/01 - 3.3.3

Application Number: 92908695.7

Publication Number: 0527225

IPC: C08F 6/28

Language of the proceedings: EN

Title of invention:

Method of improving the oxidative thermal stability of ethylene polymers

Patentee:

THE DOW CHEMICAL COMPANY

Opponent:

E.I. Du Pont de Nemours & Company, Inc.

Headword:

-

Relevant legal provisions:

EPC Art. 83, 100(b)

Keyword:

"Sufficiency of disclosure (no)"

Decisions cited:

G 0004/95, T 0805/93, T 0172/99

Catchword:

-



Case Number: T 1288/01 - 3.3.3

D E C I S I O N
of the Technical Board of Appeal 3.3.3
of 26 March 2004

Appellant: THE DOW CHEMICAL COMPANY
(Proprietor of the patent) 2030 Dow Center
Midland, Michigan 48674 (US)

Representative: Smulders, Theodorus A.H.J., Ir.
Vereenigde
Postbus 87930
NL-2508 DH Den Haag (NL)

Respondent: E.I. Du Pont de Nemours & Company, Inc.
(Opponent) 1007 Market Street
Wilmington
Delaware 19898 (US)

Representative: Cockerton, Bruce Roger
Carpmaels & Ransford
43, Bloomsbury Square
London WC1A 2RA (GB)

Decision under appeal: Decision of the Opposition Division of the
European Patent Office dated 2 October 2001 and
posted 16 October 2001 revoking European patent
No. 0527225 pursuant to Article 102(1) EPC.

Composition of the Board:

Chairman: R. Young
Members: C. Idez
A. Pignatelli

Summary of Facts and Submissions

- I. The grant of the European patent No. 0 527 225 in the name of The Dow Chemical Company in respect of European patent application No. 92 908 695.7, filed on 4 March 1992 and claiming priority of the US patent application No. 663995 filed on 4 March 1991 was announced on 28 January 1998 (Bulletin 1998/05) on the basis of 20 claims.

Claims 1 to 20 read as follows:

"1. A method of improving the taste and odor properties and the oxidative thermal stability of a first thermoplastic ethylene polymer, characterized by removing residual unreacted monomer or monomers, solvent and thermally unstable species from first ethylene polymer, thereby forming a second more oxidatively thermally stable ethylene polymer having an oxidative exotherm of not more than 50 percent of the oxidative exotherm of the first polymer, as measured by differential scanning calorimetry (DSC) wherein the thermal activity at 10°C per minute from room temperature to 300°C was determined in the presence of oxygen using DSC reference chambers and calculated in joules per gram, which method comprises the melting of the polymer, mixing the molten polymer with at least one stripping agent and devolatilizing the mixture of the molten first polymer and the stripping agent.

2. The method of Claim 1 wherein the first ethylene polymer is low density polyethylene.

3. The method of Claim 1 wherein the first ethylene polymer is linear polyethylene.
4. The method of Claim 3 wherein the linear polyethylene is linear low density polyethylene.
5. The method of Claim 3 wherein the linear polyethylene is linear high density polyethylene.
6. The method of Claim 1 wherein the first ethylene polymer is an interpolymer of ethylene and at least one ethylenically unsaturated carboxylic acid.
7. The method of Claim 6 wherein the unsaturated carboxylic acid is acrylic acid.
8. The method of Claim 6 wherein the unsaturated carboxylic acid is methacrylic acid.
9. The oxidatively thermally stable polymer obtainable by the method of any of Claims 2-8.
10. The method of Claim 1 wherein the first ethylene polymer is extruded through a devolatilization extruder equipped with at least one devolatilization zone.
11. The method of Claim 10 further comprising the consecutive steps of:
 - (a) feeding the first ethylene polymer to an extruder comprising an optional stripping agent injection zone, an optional confined mixing zone, at least a first partially filled vacuum zone, at least a first stripping agent injection zone, at least one first

confined mixing zone, at least a second partially filled vacuum zone;

- (b) melting the first ethylene polymer;
- (c) applying vacuum to the first partially filled vacuum zone;
- (d) cocurrently injecting at least 0.1 percent by weight based on total ethylene polymer feed of at least one stripping agent to the first stripping agent injection zone;
- (e) mixing the stripping agent with the first ethylene polymer in the first confined mixing zone;
- (f) applying vacuum to the second partially filled vacuum zone; and
- (g) recovering the second more oxidatively thermally stable ethylene polymer.

12. The method of Claim 11 wherein step (a) immediately follows a homopolymerization reaction of ethylene.

13. The method of Claim 11 wherein step (a) immediately follows an interpolymerization reaction of ethylene with at least one other comonomer.

14. The method of Claim 13 wherein the comonomer is acrylic acid or methacrylic acid.

15. The method of Claim 11 wherein each vacuum zone of the extruder has a specific surface renewal ratio of 0.91 kg (two pounds) per square meter or less.

16. The method of Claim 14 wherein the stripping agent is at least one chosen from the group consisting of light hydrocarbons, water, aqueous solutions of metal hydroxides, nitrogenous bases, water-soluble strong

base organic amines, steam, alcohol, carbon dioxide and nitrogen.

17. The method of Claim 16 wherein the stripping agent is an aqueous solution of sodium hydroxide, thereby forming a more oxidatively thermally stable ionomer.

18. The ionomer obtainable by the method of Claim 17.

19. A multilayered film structure having an inner layer and an outer layer, at least one layer of which comprises the oxidatively thermally stable polymer of Claim 9.

20. A multilayered film structure having an inner layer and an outer layer, at least one layer of which comprises the ionomer of Claim 18."

II. On 28 October 1998 a Notice of Opposition was filed by E.I. Du Pont de Nemours and Company in which revocation of the patent in its entirety was requested on the grounds of lack of novelty and lack of inventive step (Article 100(a) EPC and insufficiency of disclosure (Article 100(b) EPC).

During the opposition proceedings, the Parties relied, *inter alia* on the following documents:

D16: Experimental Report I, "Determination of the Validity of the DSC test used to calculate the oxidative Exotherm in EP-B-0527225", submitted by the Opponent with letter of 2 August 2001;

- D19: W. W. Wendlandt, "Thermal Analysis", 3rd Edition, Vol.19 of Chemical Analysis, A Wiley-Interscience Publication, John Wiley & Sons, 1986, pages 114-115, 345-357;
- D20: J. C. Tou et al, "A Cradle-Glass Ampoule Sample Container for Differential Scanning Calorimetric Analysis", Thermochemica Acta, Elsevier Scientific Publishing Company, Amsterdam, Vol. 42, 1980, pages 21-34;
- D21: L. F. Whiting et al, "Evaluation of a Capillary Tube Sample Container for Differential Scanning Calorimetry", Thermochemica Acta, Elsevier Science Publishers B.V., Amsterdam, Vol. 136, 1988, pages 231-245;
- D22: W. W. Wendlandt, "Thermal Analysis", 3rd Edition, Vol.19 of Chemical Analysis, A Wiley-Interscience Publication, John Wiley & Sons, 1986, pages 320-325;
- D23: J. Chiu, "Calorimetric Studies of Chemical Reactions Using a Thermal Micro-Reactor", Thermochemica Acta, Elsevier Scientific Publishing Company, Amsterdam, Vol. 26 (1978), pages 57-65;
- D24: J. Chiu, "Thermal Analysis"; Chihara Ed., 1977, pages 228-229;
- D25: J. Chiu, "Dynamic Thermal Analysis of Polymers. An Overview", J. Macromol. Sci. Chem., 1974, Vol A8(1) pages 3-23;

- D26: H. W. Hoyer, "Thermal Analysis in Sealed Tubes: Inorganic, Organic and Biochemical Reactions", Thermal Analysis: Comparative studies on Materials, Kambe and Garn Ed. 1974, pages 65-73;
- D27: J. Chiu, "A Dynamic Differential Calorimetric Technique for Measuring Heats of Polymerization", Analytical Calorimetry, R. S. Porter and Julian F. Johnson Ed. 1970; pages 171-183;
- D28: G. R. Taylor et al., "A sealed glass ampoule for use with a commercial differential scanning calorimeter", Analytica Chimica Acta, Elsevier Publishing Company, Amsterdam, Vol. 53 (1971), pages 452-455;
- D29: E. J. Barrett et al, "Differential Thermal Analysis with Capillary Tubes", Mikrochimica Acta (Wien) Springer Verlag 1970; pages 1121-1134; and
- D30: ASTM D 3895-95 "Standard Test Method for Oxidative-Induction Time of Polyolefins by Differential Scanning Calorimetry."
- III. By a decision issued announced orally on 2 October 2001 and issued in writing on 16 October 2001, the Opposition Division revoked the patent. The decision was based on Claims 1 to 20 as granted as main request, on Claims 1 to 19 filed with letter of 2 August 2001, on Claims 1 to 18 filed with letter of 2 August 2001 and on Claims 1 to 18 filed with letter of 15 June 1999, representing respectively a first, a second and a third auxiliary request.

Claim 1 of the first auxiliary request differed from Claim 1 of the main request, in that the features of Claim 10 of the main request had been incorporated therein. Claims 2 to 9, and 10 (apart the change of the wording "cocurrently" into "concurrently" in step (d) in Claim 10) to 19 corresponded to Claims 2 to 9 and 11 to 20 of the main request, respectively.

Claim 1 of the second auxiliary request differed from Claim 1 of the first auxiliary request in that the features of Claim 10 thereof had been incorporated therein. Claims 2 to 9, and 10 to 18 corresponded to Claims 2 to 9, and 11 to 19 of the first auxiliary request, respectively.

Claim 1 of the third auxiliary read as follows:

"1. A method of improving the taste and odor properties and the oxidative thermal stability of a first thermoplastic ethylene polymer, characterized by removing residual unreacted monomer or monomers, solvent and thermally unstable species from first ethylene polymer, thereby forming a second more oxidatively thermally stable ethylene polymer having an oxidative exotherm of not more than 50 percent of the oxidative exotherm of the first polymer, as measured by differential scanning calorimetry (DSC) wherein the thermal activity at 10°C per minute from room temperature to 300°C was determined in the presence of oxygen using DSC reference chambers and calculated in joules per gram, which method comprises the consecutive steps of:

(a) feeding the first ethylene polymer to an extruder comprising an optional stripping agent injection zone,

an optional confined mixing zone, at least one first vacuum zone, at least one first stripping agent injection zone, at least one first confined mixing zone, at least one second vacuum zone; wherein the at least one confined mixing zone is situated at or immediately downstream of the stripping agent injection zone, the first vacuum zone is situated downstream of the at least one stripping agent injection zone and the confined mixing zone, and the second vacuum zone is situated downstream of the first vacuum zone;

(b) conveying the polymer through the extruder to partially fill the first vacuum zone;

(c) applying vacuum to the first vacuum zone;

(d) concurrently injecting at least 0.1 percent by weight based on total ethylene polymer feed of at least one inert stripping agent into the at least one stripping agent zone;

(e) conveying the polymer and the stripping agent through the extruder into the confined mixing zone and mixing the polymer and the stripping agent in the at least one confined mixing zone;

(f) conveying the polymer and the stripping agent mixture through the extruder into the second vacuum zone and applying vacuum to the second vacuum zone; and

(g) recovering the extruded ethylene polymer."

Claims 2 to 8 corresponded to Claims 2 to 18 of the second auxiliary request.

IV. In its decision, the Opposition Division held that the patent in suit did not disclose the invention in a manner sufficiently clear and complete for it to be carried out by the skilled person.

The decision stated that Claim 1 of all requests included the feature:

"by removing residual unreacted monomer or monomers, solvent and thermally unstable species from first ethylene polymer, thereby forming a second more oxidatively thermally stable ethylene polymer having an oxidative exotherm of not more than 50 percent of the oxidative exotherm of the first polymer, as measured by differential scanning calorimetry, wherein the thermal activity at 10°C per minute from room temperature to 300°C was determined in the presence of oxygen using DSC reference chambers and calculated in joules per gram, ...".

The decision held that the experimental report referred to as D16 submitted by the Opponent showed that the duration of the cooling step was critical for the determination of the oxidative exotherm. The argument of the Patent Proprietor that these tests were not pertinent since the cooling step had not been performed with a cold finger was not accepted. The decision held that the patent in suit did not disclose that the cooling step must be carried out using a cold finger and not by immersion in liquid nitrogen. Both methods were used in the art (cf. D27).

The decision further stated that none of the documents D19 to D26 and D28 to D29 disclosed a standard procedure for the measurement of oxidative exotherm. Thus, the Opposition Division came to the conclusion that, without the precise experimental details of the measurement by DSC such as the experimental apparatus and the duration of the cooling time, the person

skilled in the art would not be able to achieve and determine the oxidative exotherm.

- V. A Notice of Appeal was filed on 14 December 2001 by the Appellant (Patent Proprietor) with simultaneous payment of the requested fee. With the Statement of Grounds of Appeal filed on 25 February 2002, the Appellant submitted the following document:

D31: Bretherick's Handbook of Reactive Chemical Hazards; Butterworth Heinemann, Fourth Edition, 1990, pages 1346-1347.

It also argued essentially as follows:

- (i) The Opposition Division had applied an inappropriate standard for determining sufficiency of disclosure.
- (ii) The teaching in a patent was to be supplemented by the general knowledge of the person skilled in the art.
- (iii) Document D27 did not disclose that both immersion in liquid nitrogen and the use of a cold finger were known and used in the art.
- (iv) From the passage at page 7 of the patent (i.e. Section titled Differential Scanning Calorimetry), the skilled person could only understand that the cold finger technique was used.
- (v) As stated by the Opposition Division, the cold finger technique was known in the art.

(vi) Sealing the ampoule by direct immersion in liquid nitrogen would be dangerous and inappropriate. Furthermore, the cold finger technique reduced the oxygen condensation in the ampoule.

(vii) When using the cold finger technique, the effect of the immersion time was negligible. This was shown by the experimental data in Tables I and II (cf. pages 9 and 10 of the Statement of Grounds of Appeal) concerning the alleged influence of cooling time (e.g. cooling times of 20 seconds and of 5 minutes) on the oxidative exotherm.

VI. With its letter dated 8 November 2002, the Respondent submitted the following document:

D32: Affidavit of Dr M. Y. Keating, dated 28 October 2002.

It also argued essentially as follows:

- (i) The insufficiency was that there was no way of determining the meaning of reduction of oxidative exotherm since this feature depended on parameters not disclosed in the patent specification.
- (ii) Concerning the cooling method of the sealed ampoule, the specification of the patent did not exclude the immersion in liquid nitrogen.
- (iii) The sealed ampoule thermal analysis was not a routine method and was used only rarely. As

stated in the Affidavit of Dr Keating, the skilled person would not know how to conduct the measurement in the absence of detailed instruction.

- (iv) There was no standard method for the determination of oxidative exotherm.
- (v) Document D27 taught that cooling with a cold finger was an alternative to cooling by immersion.
- (vi) The danger of explosion was very minor. The analyses of the Opponent involving immersion cooling were conducted satisfactorily.
- (vii) Contrary to the submissions of the Appellant, the condensation of oxygen was inherently desirable in order to ensure the reliability of the data.
- (viii) The value of the oxidative exotherm would be independent of the cooling methods provided there was a stoichiometric amount or excess of oxygen in the ampoule.
- (ix) Concerning the duration of the cooling step, the results presented by the Appellant with the Statement of Grounds of Appeal showed a large standard deviation.
- (x) There was however no mention of whether the oxidative exotherms had been determined before or after treatment. If the standard deviations of the tests before and after treatment overlapped, this might imply that insufficient combustion had

taken place, and would explain the low variability between the 20 seconds and the 5 minutes data.

VII. With its letter dated 30 January 2004, the Appellant submitted the following documents:

D33: Affidavit of Dr J. C. Oxley, dated 29 January 2004;

D34: L. F. Whiting et al. "Evaluation of a Capillary Tube Sample Container for Differential Scanning Calorimetry"; Proceedings of the sixteenth North American Thermal Analysis Society Conference, September 27-30, 1987, Washington, D.C.; pages 394-399; and

D35: M. Y. Keating et al. "Low Temperature Specific Heats of Polystyrenes and Ethylene-Vinylacetate Copolymers", Proceedings of the sixteenth North American Thermal Analysis Society Conference, September 27-30, 1987, Washington, D.C.; pages 22-27.

It informed the Board that it would be accompanied by Dr J. C. Oxley as a technical expert at the oral proceedings scheduled on 26 March 2004.

The Appellant argued essentially that the Affidavit of Dr Oxley made clear that the person skilled in the art would realize that the patent pointed him to the cold finger method.

VIII. With its letter dated 28 January 2004, the Respondent informed the Board that it would be accompanied by

Dr M. Y. Keating as a technical expert at the oral proceedings.

IX. Oral proceedings were held on 26 March 2004.

At the oral proceedings the discussion focussed on the influence of the cooling method and of the cooling time on the value of the oxidative exotherm and, more generally, on the reliability of the test for determining the oxidative exotherm.

(a) The submissions of the Appellant may be summarized as follows:

- (i) The aim of the claimed process was to reduce not only the amount of volatile components such as solvents, diluents or residual monomers but, moreover, the amount of thermally unstable species (referred to as semi-volatiles) which could cause taste problems.
- (ii) While the volatile content might be easily determined by analytical techniques such as high performance liquid chromatography or gas chromatography, these techniques did not provide information on the content of semi-volatiles. Such information could however be provided by the determination of the oxidative exotherm as defined in the patent in suit.
- (iii) In that respect, Table 4 and Table 8 of the patent in suit showed that there was no

linear correlation between the amount of volatile component and the value of oxidative exotherm.

- (iv) The crucial point of the decision of the Opposition Division was based on the assumption, in view of the experimental data submitted by the Opponent (cf. D16) that the value of the oxidative exotherm was dependent on the duration of the cooling step.

- (v) In that respect, if one directly compared the average values of the exotherm obtained using different cooling time as disclosed in D16, one would get an average of value of 16 J/g (relative standard deviation 53%) at 2 minute cooling time and an average value of 16 J/g at 5 minute cooling time (relative standard deviation 85%) before purification treatment and, after purification, an average value of 12 J/g (relative standard deviation 40%) at 2 minute cooling time and an average value of 25 J/g (relative standard deviation 100%) with a 5 minute cooling time.

- (vi) However, in view of the obvious outlying data from the values reported in Table 1 of D16 (i.e. the value 48 J/g for the 5 minute cooling time before treatment, and the values 30, 39, 51, 58, and 73 for the 5 minute cooling time after treatment), one would have culled these values or have

increased the number of measurements so that the average value came closer to the true value. By discarding these values, one would come to an average value of 11 J/g (relative standard deviation 22%) at 5 minute cooling time before treatment and to an average value of 11 J/g (relative standard deviation 10%) after treatment. Relative standard deviation in the order of 22% was in line with the Patentee's own tests (relative standard deviation between 9% to 16%). Thus, the cooling time was not a factor influencing the exotherm, since the difference in the average values was not statistically significant.

(vii) This was not altered by the mention in document D27 that the precision of measurement by the sealed ampoule technique would be better than 3% relative, since this statement would appear to relate merely to the determination of the exotherm temperature.

(viii) Although the use of a cold finger would have been preferable for practical reasons (reducing the risks of explosion), the essential point was to ensure, independently of the cooling method used, that enough oxygen (i.e. at least a stoichiometric amount) should be present in the ampoule in order to oxidize the sample.

- (ix) In view of the volume of the ampoules generally used in the sealed ampoule technique (i.e. 25 :1) and of the quantity of the polymer sample (5 mg) specified in the patent in suit, it was evident that this criterion was fulfilled.

- (x) Thus, the skilled person was given sufficient guidance in the patent in suit (cf. page 7, lines 35 to 42) to carry out the determination of the oxidative exotherm.

- (b) The arguments submitted by the Respondent may be summarized as follows:
 - (i) There was no standard method for determining the oxidative exotherm.

 - (ii) As shown by the documents D21 and D27, different methods might be used for cooling the ampoule, i.e. by immersion in a suitable refrigerant (e.g. liquid nitrogen) or by using a cold finger.

 - (iii) As pointed out in the Affidavit of Dr Oxley the sealed ampoule technique disclosed in D27 allowed rapid and reliable measurement.

 - (iv) If, however, the use of a cold finger was essential for the determination of the oxidative exotherm, this would have to have been indicated in the patent in suit.

- (v) The tests conducted by the Respondent had been carried out in ampoules having a volume of 12.5 mm³. There was enough oxygen to oxidize the polymer sample.

- (vi) There was no reason, contrary to the submissions of the Appellant (cf. point a.(vi), above) to discard some data from Table I of D16, let alone to consider them as invalid. There was further no indication in the patent in suit concerning the number of tests which should be carried out.

- (vii) The data of Table 1 of D16 indeed showed the extreme variability of the determination of the oxidative exotherm. This variability might be related to parameters such as, *inter alia*, the particle size of the sample (powder), the position of the sample in the ampoule, the accessibility of the oxygen to the powder, the viscosity of the polymer when melted, the cooling time, and the cooling method, none of which were indicated in the patent in suit.

- (viii) Thus, the patent in suit did not disclose adequate instructions to a person skilled in the art for determining the oxidative exotherm in a reliable manner.

Following preliminary remarks under Article 123(2) EPC concerning the auxiliary requests 1 to 3 then on file the Appellant indicated that it withdrew its auxiliary requests.

- X. The Appellant requested that the decision of the Opposition Division be set aside and the patent be maintained as granted.

The Respondent requested that the appeal be dismissed.

Reasons for the Decision

1. The Appeal is admissible.

Procedural matters

2. As appears from the Summary of Facts and Submissions, the Board was faced with two procedural issues arising from written submissions of the Appellant and of the Respondent.
- 2.1 The first issue concerns the oral submissions by persons accompanying the representatives of both parties, i.e. Dr J. C. Oxley for the Appellant and Dr M. Y. Keating for the Respondent.
- 2.2 According to the principles set out in the decision G 4/95 (OJ EPO 1996, 412), if during oral proceedings before a board of appeal a party wishes that, in addition to the complete presentation of its case by its professional representative, oral submissions should be made on its behalf by an accompanying person, the professional representative should (i) request permission for such oral submissions to be made in advance to the oral proceedings, (ii) state the name and qualifications of the person for whom this

permission is requested, and (iii) specify the subject-matter on which this person wishes to speak; in any case, (iv) these oral submissions should be made under the control of the professional representative (cf. Reasons for the Decision, points 8 and 10).

- 2.3 There is no doubt in the Board's view, that the Appellant's letter of 30 January 2004 announcing the presence of Dr J. C. Oxley satisfied these principles. The same is true for the letter of 28 January 2004 of the Respondent, read in combination with the letter of 8 November 2003, announcing the presence of Dr M. Y. Keating.
- 2.4 Consequently, both were given the opportunity to provide additional information on the sealed ampoule technique used in the differential scanning calorimetry during the oral proceedings.
- 2.5 The second point concerns the late-filed documents D31, D32, D33, D34, and D35.
- 2.6 D31 was submitted by the Appellant with the Statement of Grounds of Appeal in order to show the risks associated with the use of liquid nitrogen. In that respect, it is noted by the Board, firstly, that the introduction of this document has never been contested by the Respondent, and, secondly, that the Respondent has further made counterstatements (cf. point VI(vi) above) in view of the alleged risks of the use of liquid nitrogen. Thus, the Board sees no reason to disregard document D31.
- 2.7 Document D34, which has a similar content to D21, and

document D35 were not relied upon by the Parties during the Oral proceedings, and there is therefore no need for the Board to consider them in the present decision. Thus, it was not necessary to decide on their admissibility into the proceedings (Article 114(2) EPC).

2.8 Document D32 is an Affidavit of Dr M. Y. Keating submitted by the Respondent, and document D33 is an Affidavit of Dr J. C. Oxley, submitted by the Appellant in response to D32. Both affidavits presented the views of two experts in the field of differential scanning calorimetry concerning the use of the sealed ampoule technique. Since the submissions presented therein were further supplemented by the contributions of Dr J. C. Oxley and Dr M. Y. Keating at the oral proceedings, the Board sees no reason not to admit them into the proceedings.

3. *Sufficiency of disclosure*

3.1 The patent in suit relates to a method of improving the taste and odour properties and the oxidative thermal stability of a first thermoplastic ethylene polymer, characterized by removing residual unreacted monomer or monomers, solvent and thermally unstable species from first ethylene polymer, thereby forming a second more oxidatively thermally stable ethylene polymer having an oxidative exotherm of not more than 50 percent of the oxidative exotherm of the first polymer.

3.2 It is thus clear that the process conditions should be chosen in such a manner that the second ethylene polymer has an oxidative exotherm of not more than 50

percent of the oxidative exotherm of the first polymer.

- 3.3 It thus follows that the implementation of the process according to the patent in suit presupposes that the values of the oxidative exotherm of the first and the second ethylene polymer could be determined in a reliable and reproducible manner.
- 3.4 This inevitably implies that the person skilled in the art knows the method and the essential operating conditions in order to determine this parameter, since he would otherwise be left in considerable doubt when choosing the process conditions in order to obtain a second ethylene polymer falling within the terms of the claimed process (cf. decision T 805/93 of 20 February 1993, not published in OJ EPO, Reasons point 5).
- 3.5 In that respect, the Board notes that it has been submitted by the Respondent that there was no standard method in the art for the determination of the oxidative exotherm of ethylene polymers as defined in the patent in suit. This has not been challenged by the Appellant and none of the documents D19 to D30, although relating to measurements by differential scanning calorimetry, refers to a method for the determination of this parameter.
- 3.6 It is further evident, in view of the submissions of the Appellant (cf. points IX(a.i) to (a.iii), above), that the oxidative exotherm relied on in the patent in suit represents a newly formulated parameter. Thus, the Board can only come to the conclusion that the Patentee (Appellant) has developed its own method for the determination of this new parameter.

3.7 As stated in the decision T 172/99 of 7 March 2002 (not published in OJ EPO), in the case of claimed subject-matter relying on a newly formulated and, hence, unfamiliar parameter to define the solution of a technical problem by which a relevant effect is achieved, the patentee, who has the duty of making a full and fair disclosure of his invention to the public (Article 83 EPC), is under a particular obligation to disclose all the information necessary reliably to define the new parameter not only (i) in a formally correct and complete manner such that its values can be obtained by a person skilled in the art without undue burden, but also (ii) in a manner which reliably retains the validity of the parameter for the solution of the technical problem for the application or patent in suit as a whole in the sense that the values routinely obtained will not be such that the claimed subject-matter covers variants incapable of providing the relevant effect or, therefore, of solving the associated technical problem.

3.8 According to the patent in suit differential scanning calorimetry (DSC) analysis is used to determine the oxidative exotherm of resin samples in the presence of oxygen. For each sample, a known amount of resin (approximately 5 milligram) was placed in a very small glass ampoule. The bottom of the glass ampoule was cooled with liquid nitrogen and the top of the ampoule was sealed. The sealed ampoule containing the resin sample is placed in the DSC sample chamber at room temperature. The DSC sample and reference chambers are then heated at a constant rate of 10°C per minute to a final temperature of 300°C. The thermal activity of the

sample resin was continuously monitored versus the reference. The reference was an empty glass ampoule. The exotherm of the resin sample up to 300°C was calculated in terms of joules per gram (page 7, lines 35 to 42).

- 3.8.1 It follows from the paragraph 3.8 above, that the method disclosed in the patent in suit merely requires that the ampoule be cooled with liquid nitrogen without, however, either specifying a particular cooling method (e.g. immersion in liquid nitrogen or use of a cold finger) or a specific cooling time. Even if emphasis has been put by the Appellant on the risks of direct immersion in liquid nitrogen in view of document D31, it is however clear in view of documents D27 (cf. page 175, lines 1 to 10) and D23 (Figure 2) and of the tests carried out by the Respondent (cf. point VI(vi), above) that both methods were at the disposition of the skilled person.
- 3.8.2 It is further evident that the method disclosed in the patent in suit did not define the volume of the sealed ampoule to be used. In that respect, both parties, however, agreed that enough oxygen (i.e. at least a stoichiometric amount) should be present in the ampoule and it has been shown that the ampoules selected by the Appellant as well those used by the Respondent fulfilled this criterion.
- 3.8.3 This leads the Board to the conclusion that the determination of the oxidative exotherm carried out by the Respondent in document D16, using a direct immersion in liquid nitrogen for the sealing of the ampoule, has been carried out in the framework of the

instructions given in the patent in suit.

- 3.9 In this connection, the analysis of the data submitted by the Respondent in document D16, shows, as calculated by the Appellant (cf. paragraph IX(a.v), above), a relative standard deviation of the oxidative exotherm of respectively 53% (cooling time 2 minutes) and 85% (cooling time 5 minutes) before treatment and of respectively 40% (cooling time 2 minutes) and 100% (cooling time 5 minutes) after treatment.
- 3.10 It thus follows that the extreme inaccuracy reflected by the high relative standard deviation of the measurements of the oxidative exotherm drastically questions the reliability of the parameter itself, and by way of consequence the reliability of the assessment of its required reduction of at least 50% which is essential to the solution of the technical problem underlying the patent in suit (i.e. improving the taste and the odor of ethylene polymer), since the standard deviation of the oxidative exotherm before and after treatment would inevitably overlap.
- 3.11 In that respect, the argument of the Appellant that the skilled person would have discarded, in its view, obviously outlying data (cf. point IX(a.vi) above), is not convincing, firstly since there is no evidence as to whether the alleged outlying values are in fact the invalid ones and secondly, even if they were, there is no justification as to why all these particular values should be disregarded. On the contrary, they contribute to a relative standard deviation associated with the experimental results.

3.12 Even if, for sake of argument, one would disregard the values deemed to be outlying by the Appellant, the relative standard deviation would nevertheless remain of the order of 53% and 40% before and after treatment for a cooling time of 2 minutes (cf. point 3.9, above) and it would be changed to a relative standard deviation of 22% before treatment for a cooling time of 5 minutes.

3.13 In this connection, the argument of the Appellant, that a relative standard deviation of 22% in the measurements would have been sufficiently small to reveal a "trend" in the figures which would have been sufficiently pronounced to enable the skilled person reliably to discern which compositions would fall within the scope of the claims and, hence, exhibit the relevant desirable qualities cannot not be accepted for the following reasons:

3.13.1 It has not been established that the highest relative standard deviation for the test would in fact lie at 22% (cf. point 3.12 above).

3.13.2 Even if it had, it must be borne in mind that the relative standard deviation of 22% would apply to the measurement both before and after purification, thus amounting to a total standard error of 44%. This would be a major proportion of the 50% minimum difference required by Claim 1, which cannot be regarded as corresponding to a reliable discernment of the relevant values.

3.14 Furthermore, whilst it might be true that the value of 22% for the relative standard deviation would come

closer to the values reported for the relative standard deviation by the Appellant in its Statement of Grounds of Appeal (i.e. relative standard deviation between 16% and 9% before treatment), this level of uncertainty in the determination of the oxidative exotherm is in any case much more than what the skilled person would normally have expected from a determination of a heat of reaction by differential scanning calorimetry by the sealed ampoule technique in view of the teaching of D27 which, contrary to the statement of the Appellant (cf. point IX(a.vii), above), clearly mentions a precision for the determination of such a heat of reaction of less than 3% (cf. D27, page 180; Conclusions).

- 3.15 Whilst a reasonable amount of experimental inaccuracy is permissible when it comes to sufficiency of disclosure, the level of uncertainty in the present case is, in the Board's view, such that there would have to have been available adequate instructions in the specification or on the basis of the general knowledge of the skilled person in order to reduce the level of uncertainty in the determination of the oxidative exotherm to a level which could be reasonably expected by the skilled person in measurements by differential scanning calorimetry (i.e. a precision in the order of 3%), and, hence, which would not jeopardize the validity of the measured parameter.
- 3.16 In that respect, however, the patent in suit neither contains indication on further factors such as, for instance, the cooling time, the particle size of the sample, or the viscosity of the ethylene polymer which, as submitted by the Respondent (cf. point IX(b.vii), above) might influence the reliability of the

determination and thus reduce the relative standard deviation of the results, nor gives guidance concerning the number of samples which should be tested in order to come closer to a "true" value (cf. point IX(a.vi), above). Nor could the lack of instructions in the patent in suit be overcome by the general knowledge of the skilled person, since the oxidative exotherm represents a newly formulated parameter for which, therefore, no common general knowledge on its determination was available in the art before the priority date of the patent in suit.

3.17 It thus follows that the patent in suit does not disclose the method for determining the oxidative exotherm in a manner which reliably retains the validity of the parameter for the solution of the technical problem, in the sense that the values routinely obtained would not be such that the claimed subject-matter covers variants incapable of providing the relevant effect (i.e. improving the taste of ethylene polymers).

3.18 For these reasons, the Board comes to the conclusion that the patent in suit does not comply with the requirements of Article 83 EPC, and therefore, in accordance with Article 100(b) and 102(1) EPC, the request of the Appellant must be refused.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar:

The Chairman:

E. Görgmaier

R. Young.