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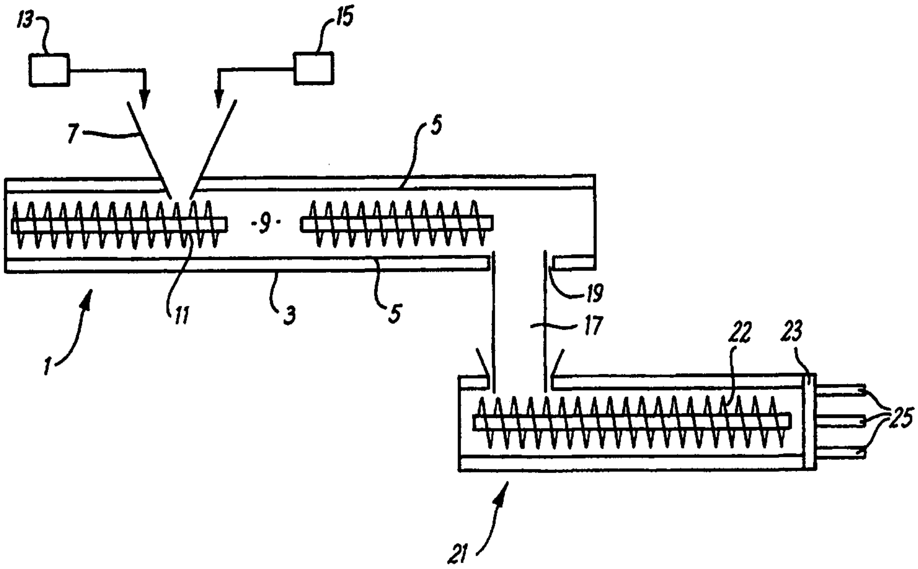
(54) Title: COMPOUNDING POLYMERIC AND FILLER MATERIALS TO PRODUCE HIGH FILLER CONCENTRATES



(57) Abstract

A method of compounding a polymeric matrix material with a non–thermoplastic filler material which includes delivering to a compounding machine a first feed comprising the polymeric matrix material and a second feed comprising particles of the non–thermoplastic filler material, mixing the feeds in the compounding machine to form a mixed material and heating and compounding the mixed material in the compounding machine whereby the polymeric material therein is softened to facilitate intimate coating of the particles of the filler material thereby, and wherein the improvement comprises delivering the material of at least one of the feeds in a heated state to the compounding machine whereby heat input to the mixed material is provided by heating of the material of the feed externally to the compounding machine as well as by heating in the compounding machine.

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TITLE OF THE INVENTION

COMPOUNDING POLYMERIC AND FILLER MATERIALS TO PRODUCE HIGH FILLER CONCENTRATES

BACKGROUND OF THE INVENTION

The present invention relates to compounding polymeric and filler materials, especially melt compounding of such materials, especially to produce high filler concentrates.

Melt compounding of thermoplastic polymeric material with filler material is well known. The filler material which may for example comprise inorganic particulate material may act as an extender or property modifier of the polymeric material and may thereby allow properties, eg physical and/or mechanical properties of the material, to be improved or may allow the unit production cost of articles produced from the material to be reduced.

Melt compounding may be carried out in various kinds of known compounding machine depending on the materials involved and the uses to which the product is to be put. Generally, the materials to be compounded are fed to the compounding machine as dry materials, eg powders, granules or pellets and heat and a mechanical compounding action are applied inside the machine. The applied heat, together with that generated frictionally by the mechanical compounding action in this conventional method does not always provide efficient compounding. We have found that where the filler material comprises an inorganic particulate material which is required to be present in a high concentration in the mixed material it can absorb a substantial amount of the heat applied in the compounding machine. Also, because the thermoplastic material is present in a lower concentration it may not be possible to generate substantial frictional heating in the thermoplastic material. The result can be inefficient compounding of the thermoplastic and filler materials.

There is a need therefore for a new method of compounding a thermoplastic polymeric material and a filler material which does not suffer from these problems. It is an object of the present invention to provide such a new method.

SUMMARY OF THE INVENTION

According to the present invention there is provided a method of compounding a polymeric matrix material with a non-thermoplastic filler material which includes delivering to a compounding machine a first feed comprising the polymeric matrix material and a second feed comprising particles of the non-thermoplastic filler material, mixing the feeds in the compounding machine to form a mixed material and heating and compounding the mixed material in the compounding machine whereby the polymeric material therein is softened to facilitate intimate coating of the particles of the filler material thereby, and wherein the improvement comprises delivering the material of at least one of the feeds in a heated state to the compounding machine whereby heat input to the mixed material is provided by heating of the material of the feed externally to the compounding machine as well as by heating in the compounding machine.

Desirably, the material of the first feed is heated by external heating prior to delivery to the compounding machine. The material of the second feed may also be pre-heated prior to delivery to the compounding machine if required.

The method according to the present invention is especially advantageous where the filler material comprises an inorganic particulate filler material of the kind known for use as fillers, pigments, extenders, property modifiers and the like in thermoplastic polymeric matrix materials and the mixed material is inorganic filler rich composition, ie incorporates more than 50 per cent by weight of the inorganic filler material. The mixed material may incorporate more than 70 per cent by weight, especially more than 80 per cent by weight, eg from 85 per cent to 95 per cent by weight of the inorganic filler material.

DESCRIPTION OF THE INVENTION

Desirably, the thermoplastic matrix material of the first feed in the method according to the invention is delivered to the compounding machine in the form of a hot melt. Hot melts of thermoplastics are known per se and are widely used for example as adhesives but have not hitherto been known as delivery feeds for compounding with non-thermoplastic particles, especially inorganic filler particles, wherein the non-thermoplastic particles forms a substantial component of the mixed material formed, eg at least 50 per cent by weight of the mixed material.

The compounding machine employed in the method according to the present invention may comprise a machine having one or more screw conveyors. For example, the materials of the first and second feeds may be introduced in an inlet to the machine to be deposited on a first screw conveyor in the machine which conveys the materials to a compounding zone wherein heating is applied and the materials are compounded to form an intimate mix.

A second screw conveyor may convey the mixed material from the compounding zone to an outlet in the machine distant from the inlet. The mixed material may emerge from the outlet in the form of a soft, eg dough-like, continuous elongate piece of mixed material, eg in the form of an extrudate such as one or more strips, ribbons, noodles, films or the like. The emerging mixed material may be further processed, eg in a pelletizer to form pellets thereof. In the form of compounding machine which has been described above, the inlet may comprise a feed hopper which allows the materials of the first feed and the second feed to be dropped together into the compounding machine eg onto or into the path of a screw conveyor, eg having a substantially horizontal axis.

The method according to the present invention may be operated as a batch or continuous process for the production of filled thermoplastic material. Most conveniently, it is operated as a continuous process. The rate of delivery of materials in the first and second feeds may be metered so that the required composition is continuously formed in the mixed material. The delivery and metering of the feed materials may be carried out in a known way. For example, the materials of the first and second feeds may be separately conveyed via conveyor belts and dropped from the belts via a hopper into the compounding machine. The amounts of materials deposited on the belts may be controlled by suitable known deposition means.

We have found that by delivering the material of at least one of the feeds, preferably at least the first feed, in a pre-heated form as well as heating the mixed material in the compounding machine, sufficient heat can be provided to the mixed material whereby heat absorbed by the filler material, which where an inorganic material present in a high concentration can cause absorption of a substantial amount of the input heat, is not so great as to cause inefficient compounding of the mixed material. In other words, the heat provided by the two sources, ie supplied externally via the feed and internally to the mixed material in the compounding machine, can be sufficient to maintain the thermoplastic matrix material of the mixed material in a soft, flowable state whereby the polymeric matrix material and the filler material are intimately and efficiently compounded together.

In the method according to the present invention the heating applied inside the compounding machine may be applied by a conventional heater, eg a heating jacket, eg comprising electrically energised resistance heating coil or other known heater.

The heating applied to the polymeric matrix material and/or to the filler material externally to the compounding machine may be applied in any suitable way known for heating such materials. The external heater may comprise an extrusion machine or a simple heating means.

The temperature to which the mixed material is heated inside the compounding machine will depend upon the specific thermoplastic polymeric materials and filler material employed in the mixed material and their relative concentrations and the work input to the compounder. In general, the heating applied internally in the compounding machine and externally to one or both of the feed materials should together provide an overall heat capacity which maintains the thermoplastic material in the mixed material in the compounding machine in a suitable melted and flowing form. The required heat input can be determined experimentally in a manner as will be evident to those skilled in the compounding art.

The filler material employed in the method according to the present invention may be selected from calcium carbonate, kaolinitic clay, calcined kaolinitic clay, mica, talc, aluminium silicate, including natural aluminium silicates such as feldspar and nepheline syenite, calcium silicate, including natural calcium silicates such as wollastonite, bauxite, alumina trihydrate, dolomite, a carbonate or hydroxide of magnesium, calcium sulphate, titanium dioxide, or a mixture of any two or more of these. Most conveniently the particulate material comprises calcium carbonate, which may be prepared either by chemical precipitation or by grinding a natural calcium carbonate mineral, eg marble or chalk, or may be a blend of precipitated and ground materials.

In general, the particle size distribution of the inorganic particulate material will depend upon application of the material filled with the inorganic material. Desirably, for most applications, the inorganic particulate material comprises inorganic particles at least 80%, preferably at least 90%, of which have an equivalent spherical diameter (esd) (diameter of a sphere which falls at the same rate as measured by sedimentation) of not greater than 10 microns (micrometers).

For high gloss, high impact, filled thermoplastic materials applications, eg for household uses, the inorganic particulate material may comprise predominantly calcium carbonate particles at least 20% of which by weight have an esd less than 0.5 microns.

For more general purpose applications, eg filling of adhesives, sealants and the like, the particulate material desirably comprises particles at least 50% of which by weight have an esd less than 1 micron.

As is well known in the art, the filler material may be surface treated with a hydrophobising surface treatment agent. The hydrophobising surface treatment agent may be one of the hydrophobising surface treatment agents known in the prior art. For example, the surface treatment agent may be selected from one or more of carboxylic acids, or their salts or esters, having from 10 to 25 carbon atoms in their hydrocarbon chain such as lauric, oleic, stearic or behenic acid or salts thereof, organosilane coupling agents, organotitanates and zircoalumninates. The preferred surface treatment agents are saturated fatty acids having from 15 to 25 chain carbon atoms, eg stearic acid, and its Group II metal salts.

The surface treatment agent may be present, together with the inorganic particulate material, in an amount of from 0.5% to 5%, especially 0.5% to 2%, by weight based on the dry weight of the inorganic particulate material present.

The polymeric matrix material employed in the method according to the present invention may comprise any of the thermoplastic materials known for use as binders of filler materials wherein the filler concentration is more than 50% by weight, eg in high concentrations preferably of 85% or more of the mixed material. Preferred polymeric matrix materials include polyolefins especially amorphous polyolefins or branched polyolefin waxes, eg derived from polyethylenes, polypropylenes or poly(ethylenepropylenes). In principle, however, the polymeric matrix material may be selected from single materials, blends or copolymers obtained from any of the various known monomer species providing polymeric thermoplastic binders such as olefins, halogenated olefins, vinyl acetate, vinyl chloride, acrylic acid, styrene, esters eg terephthalates, phenylenes, phenylene oxides, ketones.

The mixed material produced by the compounding process in accordance with the present invention may be further processed to form a suitable product form. For example, continuous elongated form of the mixed material, eg ribbon, strips, noodles and the like, produced by the compounding process may be converted into a granule or pellet form, eg by feeding the material through a pelletising extruder. The granules or pellets formed in this way are a highly concentrated form of the filler material which can easily be further dispersed in a polymeric material to form a variety of end products.

Examples of methods of use of granules or pellets produced in this way include melt compounding with further (unfilled) thermoplastic material followed by extrusion of films, tubes, shapes, strips and coatings onto other materials, eg paper, metal sheet, foil, injection moulding, blow moulding, casting, thermoforming or other product shaping processes known for filled thermoplastic materials. The melt compounding with the further thermoplastic material may for example be carried out in a suitable known compounder or screw extruder or moulding apparatus, eg blow moulder, and may be carried out in a known way. The further thermoplastic material to be compounded may suitably be in a granular or pelletised form. The temperature of the compounding and moulding, shaping or extrusion processes will depend upon the further thermoplastic material being processed and materials incorporated therein. The temperature will be above the softening point of the thermoplastic material. Pellets or granules produced in accordance with the present invention may be employed by dispersion in a further polymeric material which comprises a non-thermoplastic material, eg thermosetting or cold setting resin, which may be processed with incorporation of the added pellets or granules in a known way. The product formed in this way may include up to 80% by weight, in particular from 1% up to 50% by weight, eg from 10% to 40% by weight, of the mineral rich granules or pellets obtained from the compounding process according to the invention, the amount depending upon the materials involved and the application of the product.

The final polymeric blend produced by blending of pellets or granules with a thermoplastic or other polymeric material in one or the ways described above may include other additives well known to those familiar in the art, eg processing agents, such as lubricants, thermal or photochemical stabilising agents, colouring agents, plasticisers, antistatic agents, fire retardants, anti-oxidants, metal passivating agents or other reinforcing or filling agents such as natural or artificial fibres, metal particles, strands or foils, glass beads or microspheres and the like or other mineral (inorganic) fillers.

It may be formed into products either alone or together with other materials such as plastics, metals, refractories, wood, paper etc. in the form of laminates, coatings and the like.

The mineral concentrate (eg in pellet form) produced by compounding in the manner of the present invention may be one of the products described in Assignee's WO 95/17441, the contents of which are incorporated herein by reference, which are commercially available under the trade name ZYTOCAL™.

DESCRIPTION OF THE ACCOMPANYING DRAWING

Figure 1 is a diagrammatic cross-sectional side view of an apparatus for carrying out a method embodying the invention.

DESCRIPTION OF AN ILLUSTRATIVE EMBODIMENT OF THE INVENTION

An illustrative embodiment of the invention will now be described by way of example with reference to Figure 1.

As shown in Figure 1, a continuous mixer device 1 comprises a barrel 3 heated by a heating jacket 5. Input feed material is supplied into the barrel 3 via a feed hopper 7. The feed material is delivered to a mixing zone 9 in the barrel 3 by a first delivery screw 11. The ingredients of the feed material are melt compounded in the mixing zone 9. The ingredients comprise a particulate mineral filler and a thermoplastic polymeric binder. The ingredients of the feed material are delivered into the hopper 7 from two different feed supplies 13 and 15 respectively. The mineral filler is provided by the supply 13 which comprises a known powder metering delivery system, eg a conventional loss in weight feeder. The mineral filler delivered may optionally be heated. The polymer is provided by the supply 15 which comprises a pumped melt feed which delivers molten polymer obtained by heating solid pellets of the polymer.

The heat supplied to the feed material prior to delivery to the barrel 3 allows the additional heat required in the mixing zone 9 to be sufficient to give efficient compounding of the ingredients of the feed material without relying on the generation of frictional heat within the barrel 3. The compounded material is extracted from the mixing zone 3 and is extruded as a dough in the form of a continuous ribbon 17 via an outlet 19. The ribbon 17 is fed as an input to a pelletizing extruder 21 comprising a counter rotating twin screw extruder having a heated internal barrel 22. The material of the ribbon 17 is further compounded and compacted in the extruder 21 and is thereby extruded through a die face 23 to form pellets 25 which (when optionally cut) when subsequently cooled provide the required product form, ie high mineral content loaded polymer composition.

In a specific example of use of the apparatus shown in Figure 1, the polymer used is an amorphous polypropylene supplied under the trade name Eastoflex P1023. The mineral comprises fine calcium carbonate having a mean particle size of from 1μm to 5μm and coated with from 0.5% to 1.5% by weight of stearic acid. The polymer is pre-heated to a temperature of about 150°C prior to delivery to the mixer device 1. The heaters on the mixer device are set to give a heating temperature of 200°C to 235°C giving a resultant melt temperature of 160°C to 170°C. The temperature through the extruder 21 ranges from about 135°C to 150°C at the feed end to about 230°C to 260°C at the extrusion head end. The pelletized product obtained had a mineral content by weight of 85% to 90%.

CLAIMS

1. A method of compounding a polymeric matrix material with a non-thermoplastic filler material which includes delivering to a compounding machine a first feed comprising the polymeric matrix material and a second feed comprising particles of the non-thermoplastic filler material, mixing the feeds in the compounding machine to form a mixed material and heating and compounding the mixed material in the compounding machine whereby the polymeric material therein is softened to facilitate intimate coating of the particles of the filler material thereby, and wherein the improvement comprises delivering the material of at least one of the feeds in a heated state to the compounding machine whereby heat input to the mixed material is provided by heating of the material of the feed externally to the compounding machine as well as by heating in the compounding machine.

2. A method as claimed in claim 1 and wherein the material of the first feed is heated by external heating prior to delivery to the compounding machine.

3. A method as claimed in claim 2 and wherein the material of the second feed is also pre-heated prior to delivery to the compounding machine if required.

4. A method as claimed in claim 1 and wherein the method comprises a continuous or semi-continuous process.

5. A method as claimed in claim 1 and wherein the polymeric matrix material comprises a thermoplastic

material and the non-thermoplastic material comprises an inorganic particulate filler material.

6. A method as claimed in claim 5 and wherein the inorganic particulate material comprises calcium carbonate.

7. A method as claimed in claim 5 and wherein the polymeric matrix material comprises one or more thermoplastic polyolefins.

8. A method as claimed in claim 1 and wherein the mixed material includes at least 70 per cent by weight of the non-thermoplastic filler material.

9. A method as claimed in claim 1 and wherein the polymeric matrix material is delivered to the compounding machine in the form of a hot melt of a thermoplastic material.

10. A method as claimed in claim 1 and wherein the compounding machine has one or more screw conveyers.

11. A method as claimed in claim 10 and wherein the materials of the first and second feeds may be introduced in an inlet to the machine to be deposited on a screw conveyor in the machine which conveys the materials to a compounding zone wherein heating is applied and the materials are compounded to form an intimate mix.

12. A method as claimed in claim 11 and wherein a second screw conveyor conveys the mixed material from the compounding zone to an outlet in the machine distant from the inlet, the mixed and compounded material emerging from the outlet in the form of a soft, continuous elongate piece of mixed material.

13. A method as claimed in claim 11 and wherein the screw conveyer has a substantially horizontal axis and the inlet comprises feed hopper which allows the materials of the first and second feeds to be dropped together into the compounding machine into the region of the screw conveyer.

14. A method as claimed in claim 12 and wherein the mixed and compounded material which emerges from the outlet is delivered to a pelletizer which produces pellets of the mixed and compounded material.

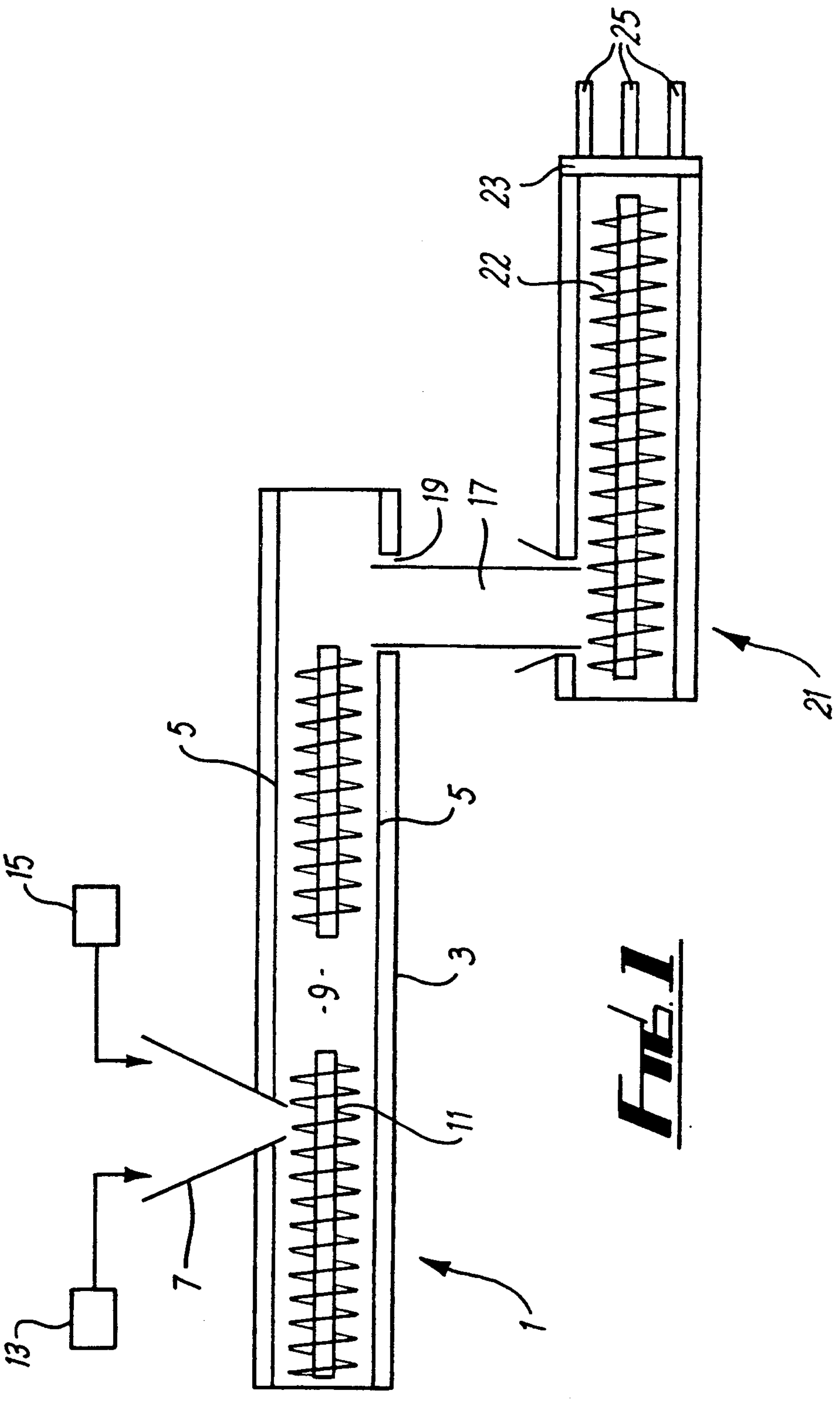

FIG. 1

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US99/00509

A. CLASSIFICATION OF SUBJECT MATTER

IPC(6) :C08J 3/20, 3/22; B29B 9/12; C08K 3/26
US CL : 523/346, 348, 351, 353, 200; 524/425; 264/7
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 523/346, 348, 351, 353, 200; 524/425; 264/7

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

APS; extru####, polyolefin, calcium carbonate, melt###, screw#, hopper# and feed###

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 5,585,054 A (EVANS) 17 December 1996, col. 3, line 14 to col. 4, line 15 and Figs. 1 and 2.	1-4, 8-14
X	US 5,256,718 A (YAMAMOTO et al) 26 October 1993, col. 7, lines 7-68, col. 26, lines 39-46 and example 26.	1-7, 11-14
X	US 2,470,001 A (STOBER) 10 May 1949, Fig. 2, col. 1, lines 26-41, col. 2, line 29 and col. 3, lines 53-70.	1-5, 9-13
X	US 3,413,249 A (LUFTGLASS et al) 26 November 1968, col. 7, lines 6-26 and Fig. 1.	1-5, 9-11
X	US 4,868,226 A (MITSUNO et al) 19 September 1989, col. 5, line 37 to col. 6, line 2.	1, 4-7, 10-14

[X] Further documents are listed in the continuation of Box C. [ ] See patent family annex.

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Date of the actual completion of the international search	Date of mailing of the international search report
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INTERNATIONAL SEARCH REPORT

International application No.
PCT/US99/00509

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 5,017,015 A (SCHLUMPF et al) 21 May 1991, col. 8, lines 6-13 and 60-64.	6-8
Y	US 5,225,488 A (BAIRD et al) 06 July 1993, Fig. 1 and example 2.	12, 14