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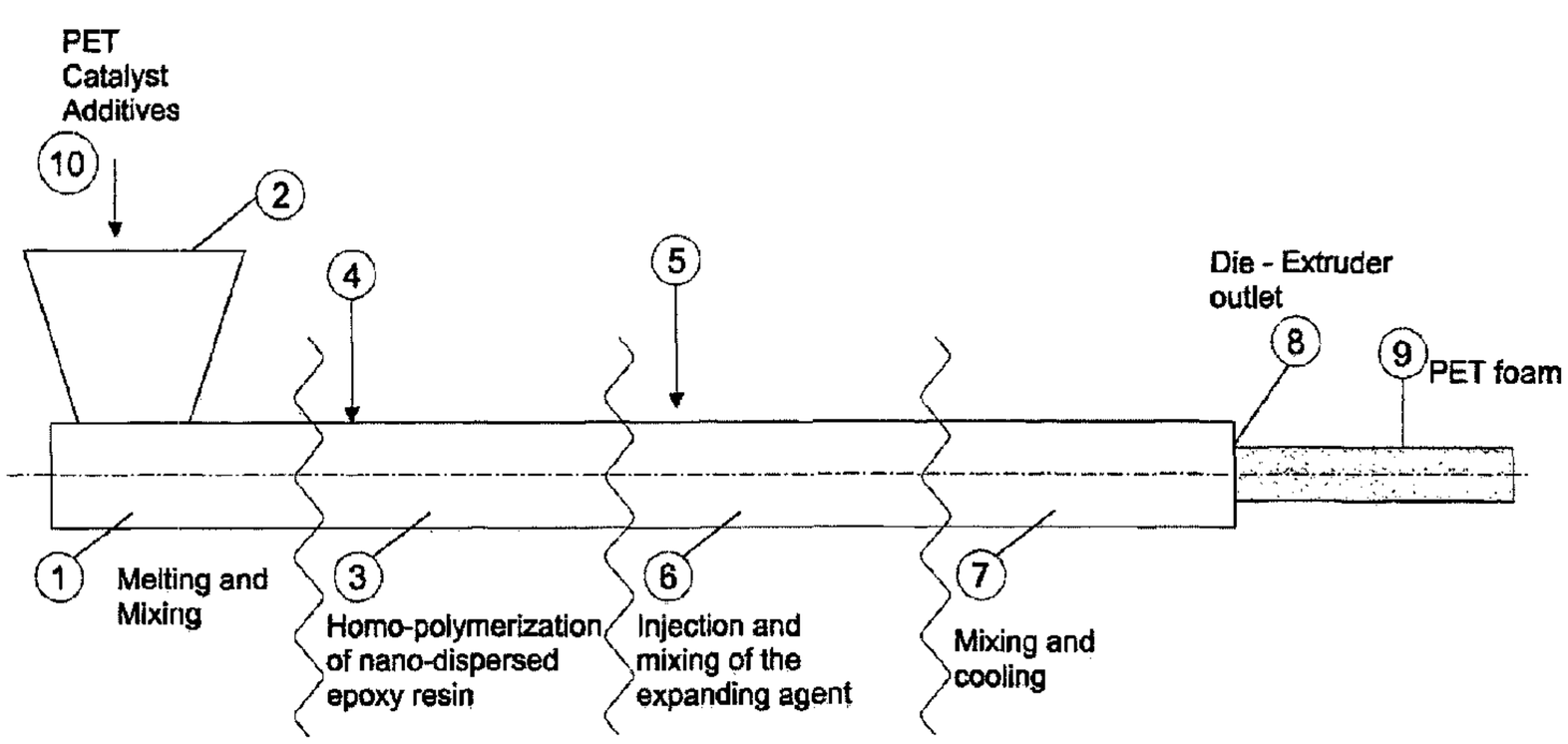
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(54) Title: PROCESS FOR THE PRODUCTION OF PET FOAMS AND PET FOAMS OBTAINED WITH SAID PROCESS



(57) Abrégé/Abstract:
A process for the production of PET foams which comprises the extrusion of a mixture of PET, epoxy resin, catalyst consisting of imidazole, derivatives of imidazole or mixtures thereof plus a blowing agent, wherein the homopolymerization reaction of the epoxy phase is effected in the extruder, upstream of the introduction of the blowing agent. Compared to the methods of the known art, the process of the invention offers the advantage of providing a stable PET foam, in which the structure of the polymer chains is kept unaltered. The system therefore has viscoelasticity characteristics which allow the PET/epoxy homopolymer to receive the expanding action of the blowing agent, as far as the innermost and deepest layers of foam also having a significant thickness.

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(54) **Title**: PROCESS FOR THE PRODUCTION OF PET FOAMS AND PET FOAMS OBTAINED WITH SAID PROCESS

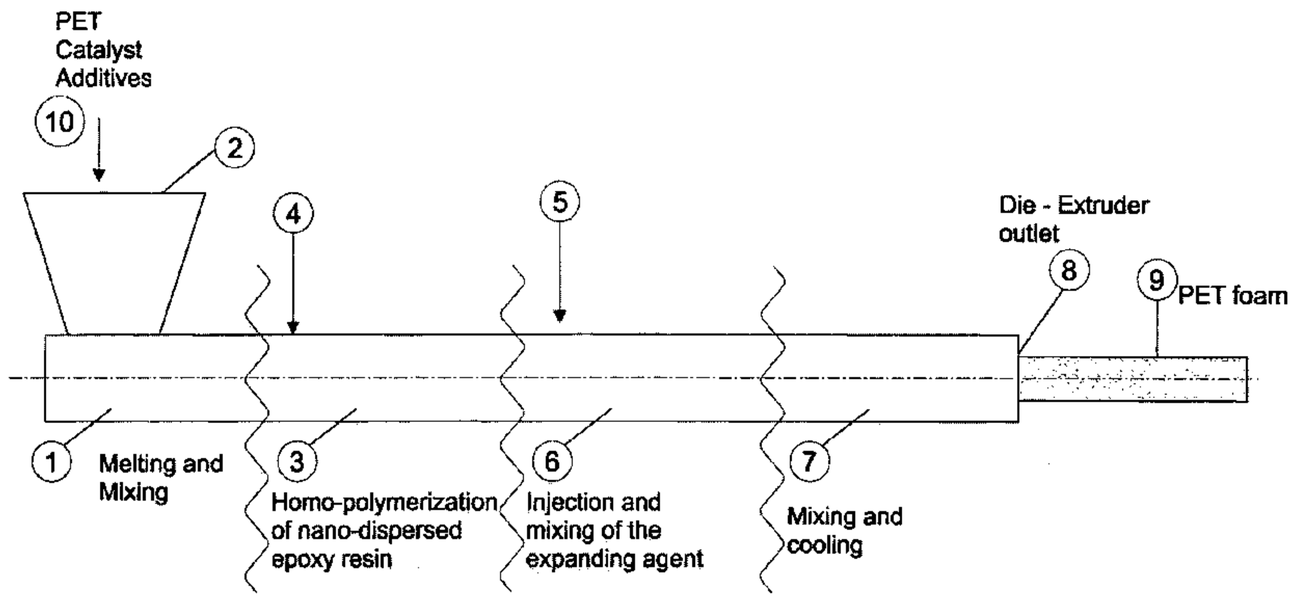


Fig. 1

(57) **Abstract**: A process for the production of PET foams which comprises the extrusion of a mixture of PET, epoxy resin, catalyst consisting of imidazole, derivatives of imidazole or mixtures thereof plus a blowing agent, wherein the homopolymerization reaction of the epoxy phase is effected in the extruder, upstream of the introduction of the blowing agent. Compared to the methods of the known art, the process of the invention offers the advantage of providing a stable PET foam, in which the structure of the polymer chains is kept unaltered. The system therefore has viscoelasticity characteristics which allow the PET/epoxy homopolymer to receive the expanding action of the blowing agent, as far as the innermost and deepest layers of foam also having a significant thickness.

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PROCESS FOR THE PRODUCTION OF PET FOAMS AND PET FOAMS OBTAINED WITH SAID PROCESS

The invention relates to an enhanced process for the production of PET foams.

The invention is also extended to PET foams obtained with this process and to the bodies of expanded material obtained from these foams.

The field of the invention is the production of PET (polyethylene terephthalate) foams, normally used in the production of panels with numerous types of processing to make them suitable as a "core material" for various types of sandwich structures and also with a thermal insulation function.

Extending PET chains with pyromellitic anhydride and other crosslinking agents is known, for giving PET the necessary consistency for receiving the blowing agent, thus obtaining an expanded and stable foam. As a result, during the following extrusion phases of the foam, more or less significant phenomena of polymer degradation may arise, which in any case represent problems with respect to the quality of the final product.

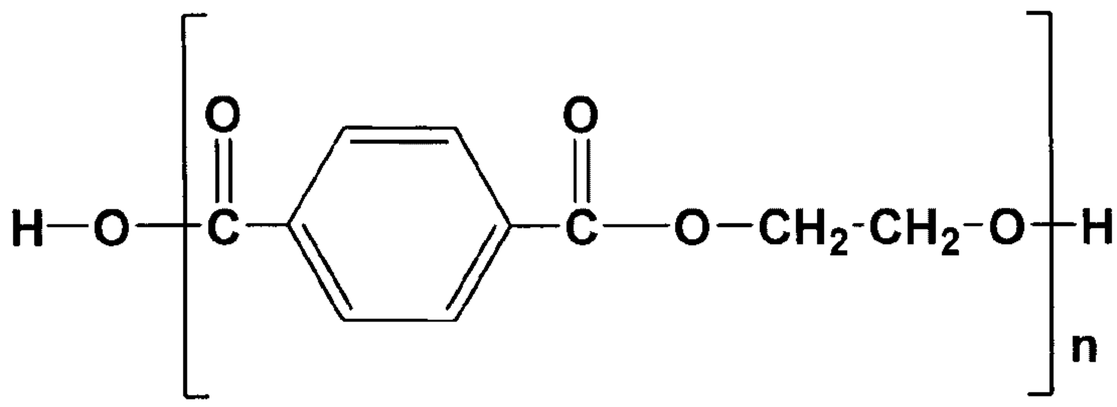
Preparing a thermoplastic polymer foam in the presence of epoxides, crosslinked by means of photosensitive catalysts is also known (US 6,323,251),

wherein the crosslinking reaction of the epoxide is effected downstream of the injection of the blowing agent, in particular outside the extrusion. The known process is consequently not suitable for providing a foam having the necessary consistency for the formation of the final product. In addition, photosensitive catalysts only act on the surface layer of the foam, which does not allow the crosslinking reaction to also take place inside the foamy mass.

The main objective of the present invention is to provide a method suitable for obtaining a PET foam which, unlike the known methods, has characteristics of stability at the outlet of the extruder, without altering, however, the structure of the PET chains.

One embodiment of the invention relates to a process for the production of PET foams, characterized in that said process includes an extrusion of a mixture of PET, epoxy resin, a catalyst consisting of imidazole, derivatives of imidazole or mixtures thereof and a blowing agent in an extruder, wherein an homopolymerization reaction of the epoxy resin is effected in the extruder, upstream of an introduction of the blowing agent.

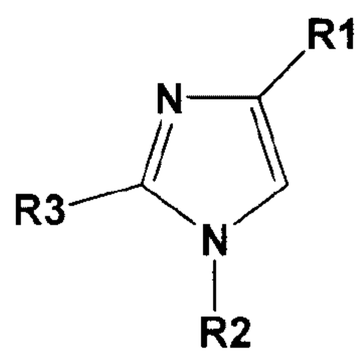
Another embodiment of the invention relates to the process defined hereinabove, characterized in that said PET is of the type to be processed by extrusion and having the chemical formula **(I)**:



(I)

wherein n is an integer representing a number of repetitive units.

Another embodiment of the invention relates to the process defined hereinabove, characterized in that said catalyst consists of imidazole, derivatives of imidazole or mixtures thereof, having the formula:

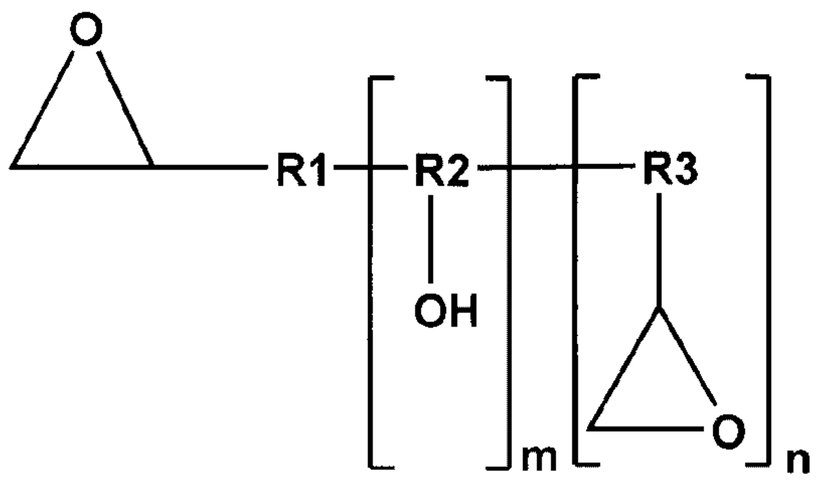


(II)

wherein R1, R2 and R3 are inorganic or organic groups of the aromatic or aliphatic type.

Another embodiment of the invention relates to the process defined hereinabove, characterized in that said catalyst is mixed with PET in a melting and mixing step (1), with the catalyst in a liquid state.

Another embodiment of the invention relates to the process defined hereinabove, characterized in that said epoxy resin is in a liquid state and has the following formula:



(III)

wherein:

m is an integer representing a number of repetitive units,

n is an integer representing a number of repetitive units, and

R1, R2 and R3 are organic, aliphatic or aromatic groups, or an inorganic chain.

Another embodiment of the invention relates to the process defined hereinabove, characterized in that said process comprises a step (3) in which the homopolymerization reaction of the epoxy resin is effected in the extruder.

Another embodiment of the invention relates to the process defined hereinabove, characterized in that in said step (3) particles of the epoxy homopolymer are nanodispersed inside a continuous mass of the PET, the PET having a chemical structure where chains are kept unaltered in the reaction environment.

Another embodiment of the invention relates to the process defined hereinabove, characterized in that said process also comprises a step (6) for the introduction of the blowing agent (5) into a continuous mass of the PET having particles of the epoxy homopolymer dispersed therein, a cooling step (7) and a foaming step (8).

Another embodiment of the invention relates to the process defined hereinabove, characterized in that said mixture also includes, alone or combined with each other, at least one of plasticizers, pigments, flame-retardants, fillers, stabilizers, surfactants, nucleating agents and nano-additives.

Another embodiment of the invention relates to a PET foam characterized in that it is obtained by means of the process defined hereinabove.

Another embodiment of the invention relates to a body of expanded material, characterized in that said body consists of the PET foam defined hereinabove.

Compared to the methods of the known art, the invention offers the advantage of providing a stable PET foam, in which the structure of the polymer chains is kept unchanged. The system also has viscoelasticity characteristics which allow the mixture of PET/epoxy homopolymer to receive the expanding action of the

blowing agent, as far as the innermost and deepest layers of foam also having a significant thickness.

These and other objectives, advantages and characteristics appear evident from the following description of a preferred embodiment of the invention illustrated, as a non-limiting example, in the figures of the enclosed drawings.

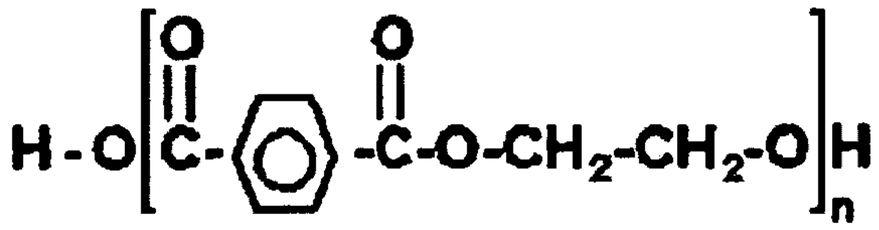
In these:

- figure 1 illustrates a scheme of the extruder used for effecting the process of the invention;
- figures 2 and 3 illustrate the exothermic homopolymerization reaction of the epoxy resin, when activated in the presence of imidazole and its derivatives, compared to those in which different catalysts or no catalyst is used;
- figure 4 illustrates the graph of a DSC (Differential Scanning Calorimetry) analysis for evaluating the crosslinking of the epoxy resin, of the samples processed as in figure 2; and
- figure 5 represents an image obtained with an Atomic Force Microscope (AFM) of the nanodispersion of an epoxy homopolymer in the PET thermoplastic matrix.

The extruder, as schematically represented in figure 1 and used for effecting the process of the

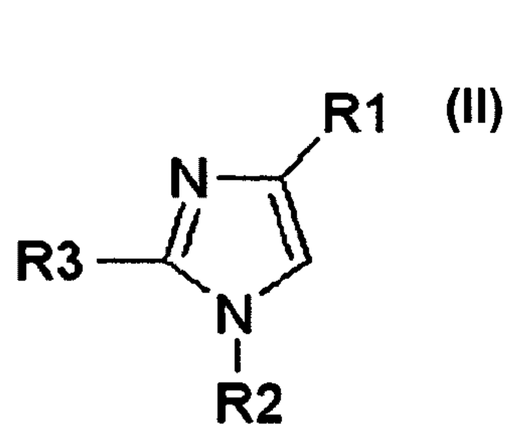
invention, is substantially divided into a step 1 for melting and mixing the PET mass 10, catalyst and other possible additives fed through the hopper 2, a reactive extrusion step 3 (homopolymerization of the epoxy phase) an injection step 6 of the blowing agent and a cooling step 7.

The PET used is in particular a PET suitable for being processed by extrusion, having the following chemical formula (I):



(I)

The catalyst is then added to the PET, which, according to the invention, consists of imidazole, derivatives of imidazole and mixtures thereof, represented by the following formula (II):



(II)

wherein R1, R2, R3 are inorganic or organic groups of the aromatic or aliphatic type.

Some non-limiting examples of catalysts used in the process of the invention are: 2-methylimidazole, 2-

ethyl-4-methylimidazole, 1-Benzyl-2-methylimidazole, 1-Methylimidazole, 1,2-Dimethylimidazole and mixtures thereof.

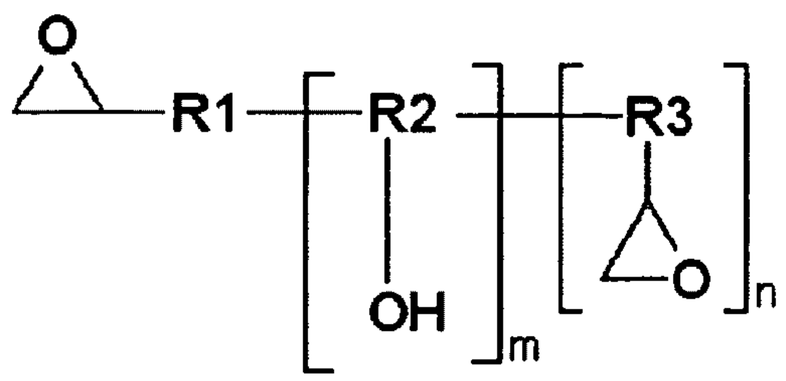
Among the additives that can be used in the process of the invention, suitable for improving the processability, appearance, and properties of the PET foam, the following can be mentioned, alone or combined with each other:

- plasticizers
- pigments
- flame-retardants
- fillers
- stabilizers
- surfactants
- nucleating agents
- nano-additives

The catalyst used in the invention is in liquid phase and, in step 1 of the extruder, it is closely mixed inside the PET mass. The mixture thus obtained is then sent, from the screw of the extruder (not represented), towards a subsequent step 3 for putting the PET mass in contact with the catalyst and mixing them with an injection 4 of epoxy resin, also in liquid state.

Preferred epoxy resins for the invention are those

having the following formula (III):



(III)

wherein:

m and n are integers(0, 1, 2, 3, ..)

R1, R2 and R3 represent an aliphatic or aromatic group, or an inorganic chain.

Among the epoxy resins that can be used in the process of the invention, the following can be cited:

- epoxidized novolacquers from phenol or cresol (polyphenyl-glycidyl ether - co - formaldehyde, poly-o-cresyl-glycidyl ether - co - formaldehyde);
- mono- or poly- glycidyl ether or ester, aliphatic or aromatic (polibisphenol A-co-epichlorohydrin-glycidyl terminated; 2-ethylhexyl glycidyl ether; 1-4-butanediol of glycidyl ether; tertaglycidyl-methylbisbenzeneamine; butylphenyl- glycidyl ether),
- organic or inorganic epoxidized chains (glycidoxypropyltrimethoxysilane; glycidoxypropyltriethoxysilane; butyldimethylsilane-glycidyl ether; bis-glycidyloxypropyltetra-methyldisiloxane);

polyhedral glycidyl oligomer based on silicon-

silicone (POSS) (example: CAS: 445379-56-6; CAS: 307496-38-4).

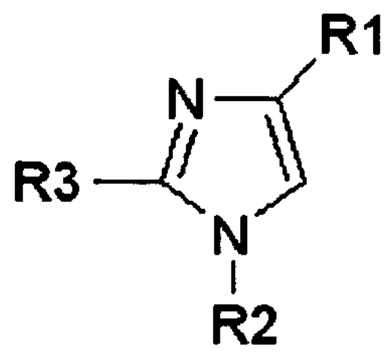
In this step of the process of the invention, the epoxy resin undergoes a homopolymerization process, which develops inside the extruder itself (so-called reactive extrusion). Furthermore, due to the use of epoxy resins in the liquid state (or previously not crosslinked), a nanodispersion of homopolymer particles can be obtained inside the continuous PET mass (see figure 5).

As a result of the presence of nanodispersed epoxy homopolymer in the PET mass, the latter is provided with the viscoelasticity necessary for the introduction of the blowing agent, to allow the subsequent foaming phase to take place.

The high catalytic activity of imidazole and its derivatives towards the homopolymerization reaction of the epoxy resin also allows this homopolymerization reaction to be completed within the above-mentioned step 3, therefore without involving the PET chains present in the reaction environment, thus leaving their chemical structure unaltered.

The elective behaviour towards the epoxy resin of the catalyst used in the process of the invention, without involving the molecules of the PET polymer, is

shown by the graphs in figures 2 to 4. In particular, figure 2 compares the elective action of 1,2-dimethylimidazole having formula



(IV)

wherein R1=H; R2= CH_3; R3=CH_3

with that offered by the catalysts of the known art, in particular triphenyl sulfonium hexafluoroantimonate, dimethylbenzylamine, 2-heptylpiperazine, or in the absence of catalyst.

From the graph of figure 2, which simulates upon DSC analysis, the operative conditions of the extrusion process of a mixture of epoxy resin alone (100 parts) with catalyst (2 parts), it can be observed that the catalyst according to the invention has an exothermic section (a) whose peak, compared with the flat trend of the corresponding sections (b) of the curves relating to the catalysts used according to the known art, or with the trend of the curve in the absence of catalyst, indicates the completion of the homopolymerization reaction of the epoxy phase, without involving the PET molecules, thus leaving them unaltered.

The same conclusions can be drawn from examining

the exothermic sections represented in figure 3, this time relating to the family of imidazoles that can be used in the process of the invention.

Figure 4 shows the DSC analysis of the samples tested as in figure 2, after simulation of the extrusion process. From the graph, it can be observed that the only sample that has undergone the homopolymerization reaction is that corresponding to the catalyst 2-ethyl-4-methylimidazole (curve c) of figure 4), which in fact has a glass transition temperature value (Tg) of 99.3°C. The flat trend of the remaining curves, relating to the samples of epoxy resin treated with catalysts different from that according to the process of the invention or samples without catalyst, indicates that the homopolymerization reaction has not taken place.

As a result of the formation of nanodispersed epoxy homopolymer, in step 3 of the extruder, within the PET mass, the latter is provided with a viscoelasticity sufficient for introducing 5 the blowing agent in step 6 of the extrusion, thus making it suitable for the subsequent foaming phase at the outlet 8 of the extruder. Said foaming phase is preceded by a cooling step 7 of the extruded product 9, the latter consisting of a continuous flow of expanded

material, whose cooling process is completed at room temperature. End-products consisting of bodies of expanded material are subsequently obtained from this extruded product 9.

Some examples of formulations of the PET foam used in the process of the invention are provided hereunder, wherein the percentages refer to the weight of the mixture.

EXTRUDER

Foaming process by extrusion obtained using a Leistritz ZSE40MAXX-44D extruder, as schematized in figure 1 and with the following temperature profile:

SET °C	ZONE 1	ZONE 3	ZONE 6	ZONE 7	ZONE 8
B	295	280-260	255	255-230	260

EXAMPLE 1 - Absence of catalyst and epoxy resin

The following reagents were charged into the extruder through the hopper 2:

- PET: Polyclear 1011 of Invista (IV=0.83)
- Talc dispersed in PET (1.3-3% with respect to PET)

The blowing agent was added through the injection point (5):

- Cyclohexane in liquid phase

The pressure value (indication of the

viscoelasticity of the mixture) inside the extruder is not such as to keep the blowing agent in a homogeneously dispersed supercritical fluid phase. Attempts at foaming the PET therefore failed as the polymeric mass at the outlet of the extruder does not have a sufficient viscoelasticity for keeping the gas phase enclosed in cells.

EXAMPLE 2 - Absence of catalyst and presence of epoxy resin

The following reagents were charged into the extruder through the hopper 2:

- PET: Polyclear 1011 of Invista (IV=0.83)
- Talc dispersed in PET (1.3-3% with respect to PET)

The following epoxy resin in liquid phase was added through the injection point (4):

- Poly-epoxy: Lindoxy 290 (in liquid phase)

The blowing agent was added through the injection point (5):

- Cyclohexane in liquid phase

Also in this case, the pressure value (indication of the viscoelasticity of the mixture) inside the extruder is not such as to keep the blowing agent in a homogeneously dispersed supercritical fluid phase. Attempts at foaming the PET therefore failed as the polymeric mass with a low viscoelasticity at the outlet

of the extruder is not capable of keeping the gas phase enclosed in cells.

EXAMPLE 3 - Presence of epoxy resin and catalyst - dimethylbenzylamine

The following reagents were charged into the extruder through the hopper 2:

- PET: Polyclear 1011 of Invista (IV=0.83)
- Talc dispersed in PET (1.3-3% with respect to PET)
- dimethylbenzylamine (in a percentage of up to 2.3% with respect to the epoxy resin)

The following epoxy resin in liquid phase was added through the injection point (4):

- Poly-epoxy: Lindoxy 290 (in liquid phase) from 1.3 to 5%

The blowing agent was added through the injection point (5):

- Cyclohexane in liquid phase

Also in this case, the pressure value (indication of the viscoelasticity of the mixture) inside the extruder is not such as to keep the blowing agent in a homogeneously dispersed supercritical fluid phase. Attempts at foaming the PET therefore failed as the polymeric mass with a low viscoelasticity at the outlet of the extruder is not capable of keeping the gas phase enclosed in cells.

EXAMPLE 4 - Presence of epoxy resin and catalyst - 2-ethylpiperidine

The following reagents were charged into the extruder through the hopper 2:

- PET: Polyclear 1011 of Invista (IV=0.83)
- Talc dispersed in PET (1.3-3% with respect to PET)
- 2-ethylpiperidine (in a percentage of up to 2% with respect to the epoxy resin).

The following epoxy resin in liquid phase was added through the injection point (4):

- Poly-epoxy: Lindoxy 290 (in liquid phase) from 1.3 to 5%.

The blowing agent was added through the injection point (5):

- Cyclohexane in liquid phase

Also in this case, the pressure value (indication of the viscoelasticity of the mixture) inside the extruder is not such as to keep the blowing agent in a homogeneously dispersed supercritical fluid phase. Attempts at foaming the PET therefore failed as the polymeric mass with a low viscoelasticity at the outlet of the extruder is not capable of keeping the gas phase enclosed in cells.

EXAMPLE 5 - Presence of epoxy resin and catalyst based on imidazole

The following reagents were charged into the extruder through the hopper 2:

- PET: Polyclear 1011 of Invista (IV=0.83) or CLEARTUF P76 by M&G (IV=0.74)
- Talc dispersed in PET (1.3-3% with respect to PET)
- Catalyst based on imidazole (tests respectively effected with 2-methylimidazole, 2-ethyl-4-methylimidazole and 1,2-dimethylimidazole, in percentages of up to 2% with respect to the epoxy resin).

The following epoxy resins in liquid phase were added through the injection point (4):

- Poly-epoxy: Lindoxy 290 (in liquid phase, from 1.3 to 5% with respect to PET)
- Mono- or di-epoxy: 2-Ethylhexyl glycidyl ether or 1,6-hexanediol diglycidyl ether (from 0 to 0.5% with respect to PET)

The blowing agent was added through the injection point (5):

- Cyclohexane in liquid phase

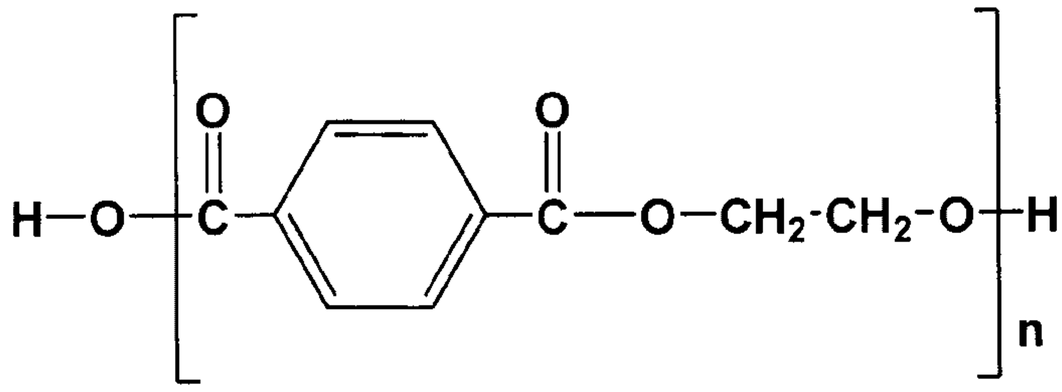
The pressure value (indication of the viscoelasticity of the mixture) inside the extruder is such as to keep the blowing agent in a homogeneously dispersed supercritical fluid phase. For the various

percentages of epoxy resin and catalyst, suitably dosed, attempts at foaming the PET were therefore successful, as the polymeric mass at the outlet of the extruder was sufficiently viscoelastic to keep the gas phase inside closed cells having a reduced dimension (less than 0.7 mm), producing a stable foam having a density ranging from 60-140 Kg/m^3.

CLAIMS

1) A process for the production of PET foams, characterized in that said process includes an extrusion of a mixture of PET, epoxy resin, a catalyst consisting of imidazole, derivatives of imidazole or mixtures thereof and a blowing agent in an extruder, wherein an homopolymerization reaction of the epoxy resin is effected in the extruder, upstream of an introduction of the blowing agent.

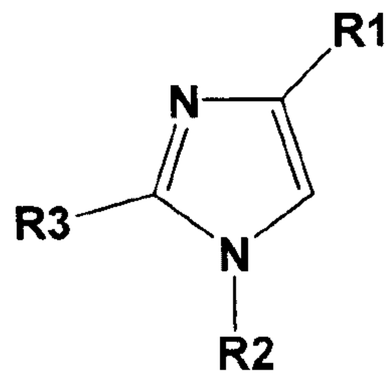
2) The process according to claim 1, characterized in that said PET is of the type to be processed by extrusion and having the chemical formula (I):



(I)

wherein n is an integer representing a number of repetitive units.

3) The process according to claim 1, characterized in that said catalyst consists of imidazole, derivatives of imidazole or mixtures thereof, having the formula:

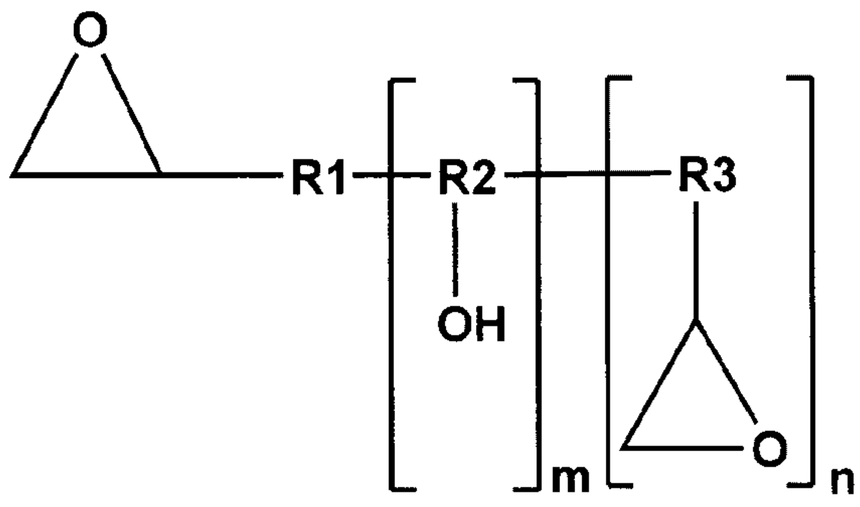


(II)

wherein R1, R2 and R3 are inorganic or organic groups of the aromatic or aliphatic type.

4) The process according to claim 1 or 3, characterized in that said catalyst is mixed with PET in a melting and mixing step (1), with the catalyst in a liquid state.

5) The process according to claim 1, characterized in that said epoxy resin is in a liquid state and has the following formula:



(III)

wherein:

m is an integer representing a number of repetitive units,

n is an integer representing a number of repetitive units, and

R1, R2 and R3 are organic, aliphatic or aromatic groups, or an inorganic chain.

6) The process according to claim 1, characterized in that said process comprises a step (3) in which the homopolymerization process of the epoxy resin is effected in the extruder.

7) The process according to claim 6, characterized in that in said step (3) particles of the epoxy homopolymer are nanodispersed inside a continuous mass of the PET, the PET having a chemical structure where chains are kept unaltered in the reaction environment.

8) The process according to claim 7, characterized in that said process also comprises a step (6) for the introduction of the blowing agent (5) into a continuous mass of the PET having particles of the epoxy homopolymer dispersed therein, a cooling step (7) and a foaming step (8).

9) The process according to any one of claims 1 to 7, characterized in that said mixture also includes, alone or combined with each other at least one of :

* plasticizers

* pigments

* flame-retardants

* fillers

* stabilizers

* surfactants

* nucleating agents and

* nano-additives.

10) A PET foam characterized in that it is obtained by means of the process defined in any one of claims 1 to 9.

11) A body of expanded material, characterized in that said body consists of the PET foam defined in claim 10.

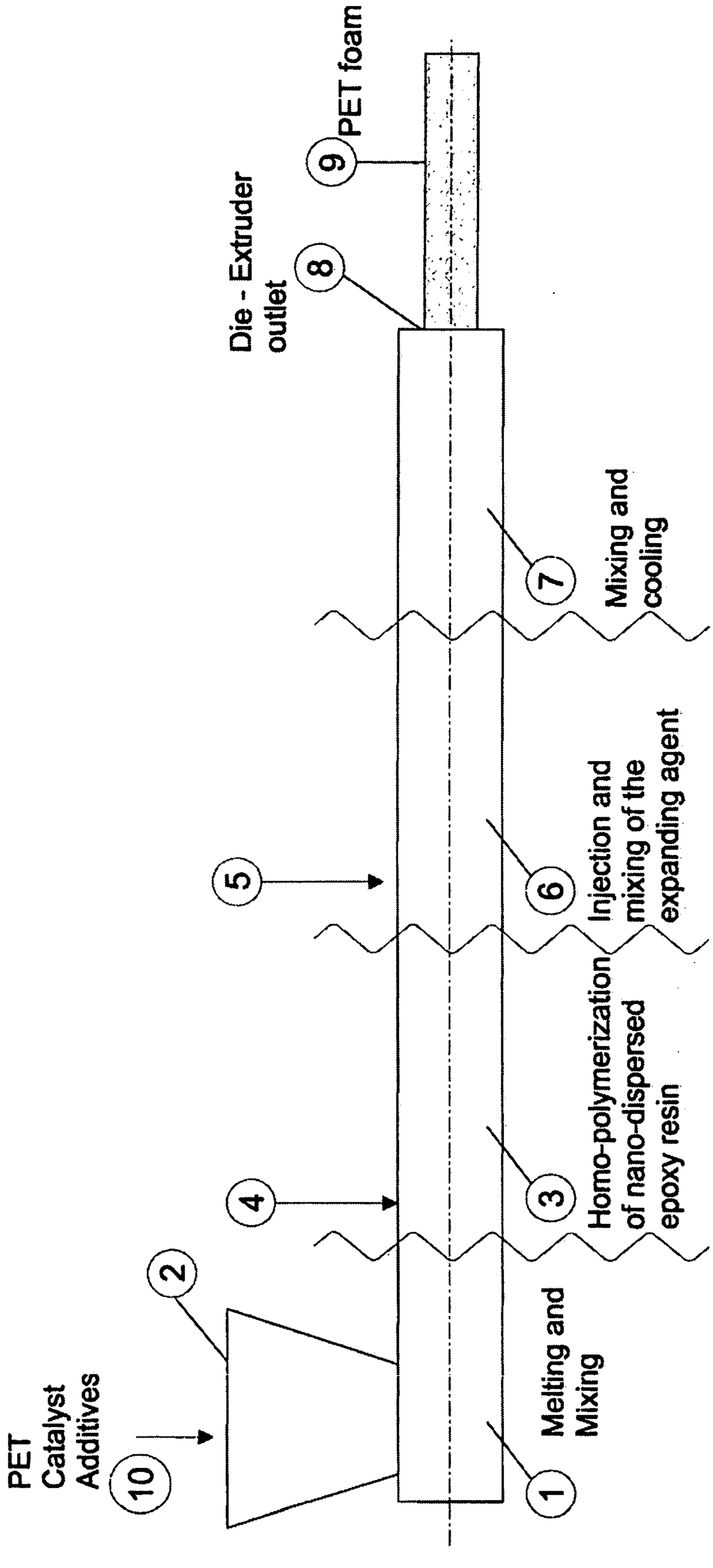

PET foam
9
Die - Extruder outlet
8
7
Mixing and cooling
5
6
Injection and mixing of the expanding agent
4
3
Homo-polymerization of nano-dispersed epoxy resin
2
PET
Catalyst
Additives
10
1
Melting and Mixing

Fig. 1

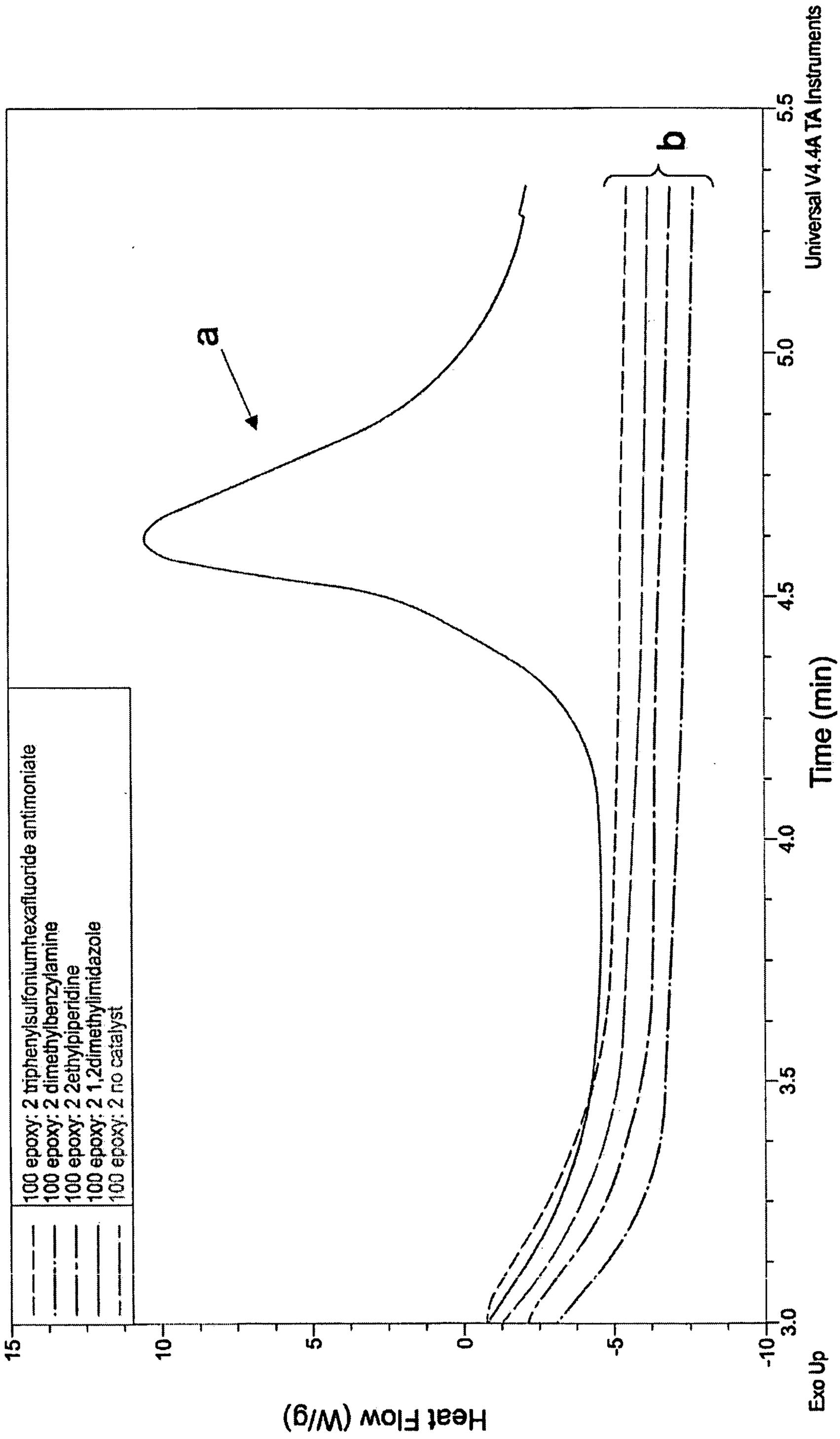

a
b
100 epoxy: 2 triphenylsulfoniumhexafluoride antimoniate
100 epoxy: 2 dimethylbenzylamine
100 epoxy: 2 2ethylpiperidine
100 epoxy: 2 1,2dimethylimidazole
100 epoxy: 2 no catalyst
Universal V4.4A TA Instruments
Heat Flow (W/g)
Time (min)
Exo Up
15
10
5
0
-5
-10
5.5
5.0
4.5
4.0
3.5
3.0

Fig. 2

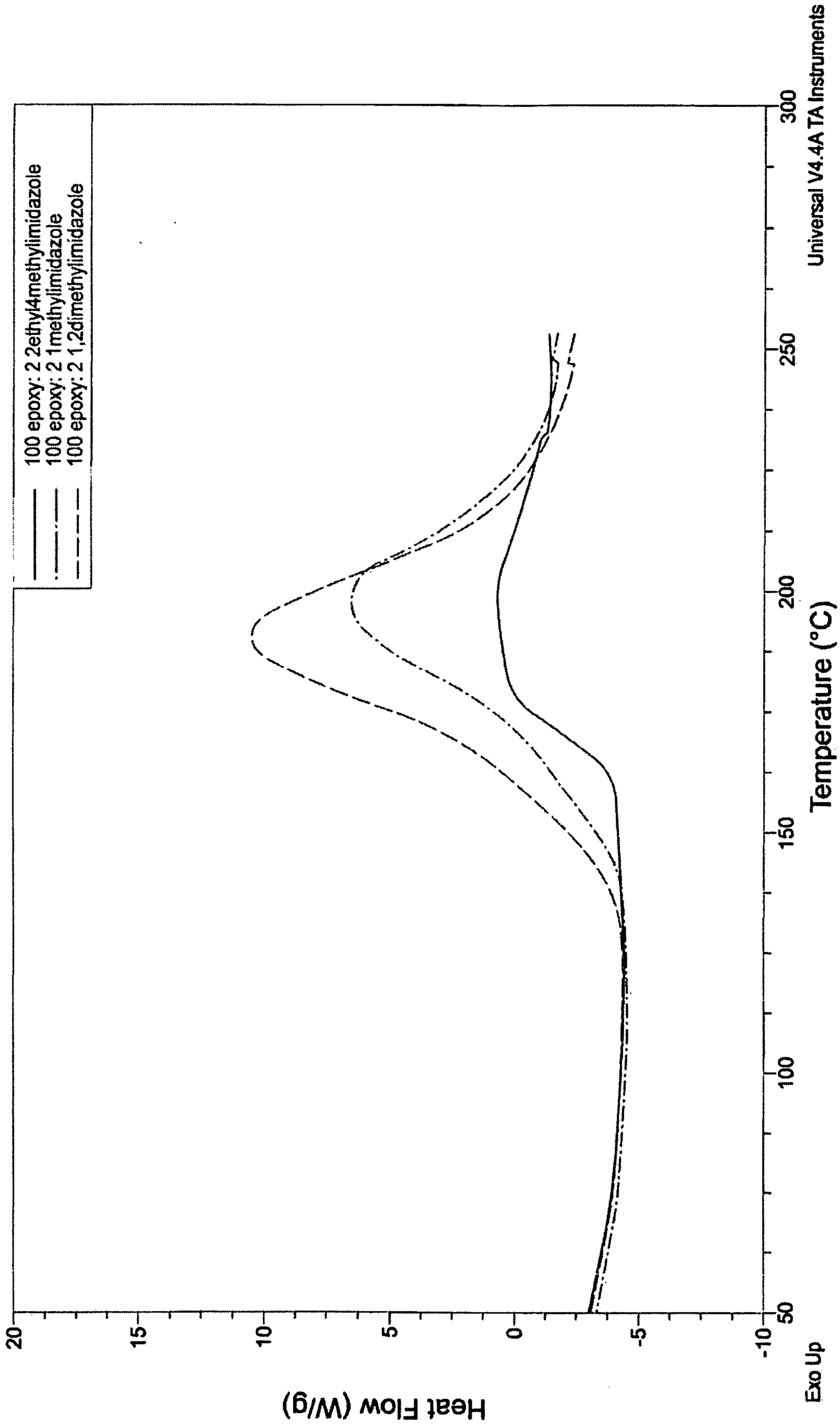

100 epoxy: 2 2ethyl4methylimidazole
100 epoxy: 2 1methylimidazole
100 epoxy: 2 1,2dimethylimidazole
Universal V4.4A TA Instruments
Temperature (°C)
Heat Flow (W/g)
Exo Up
300
250
200
150
100
50
20
15
10
5
0
-5
-10

Fig. 3

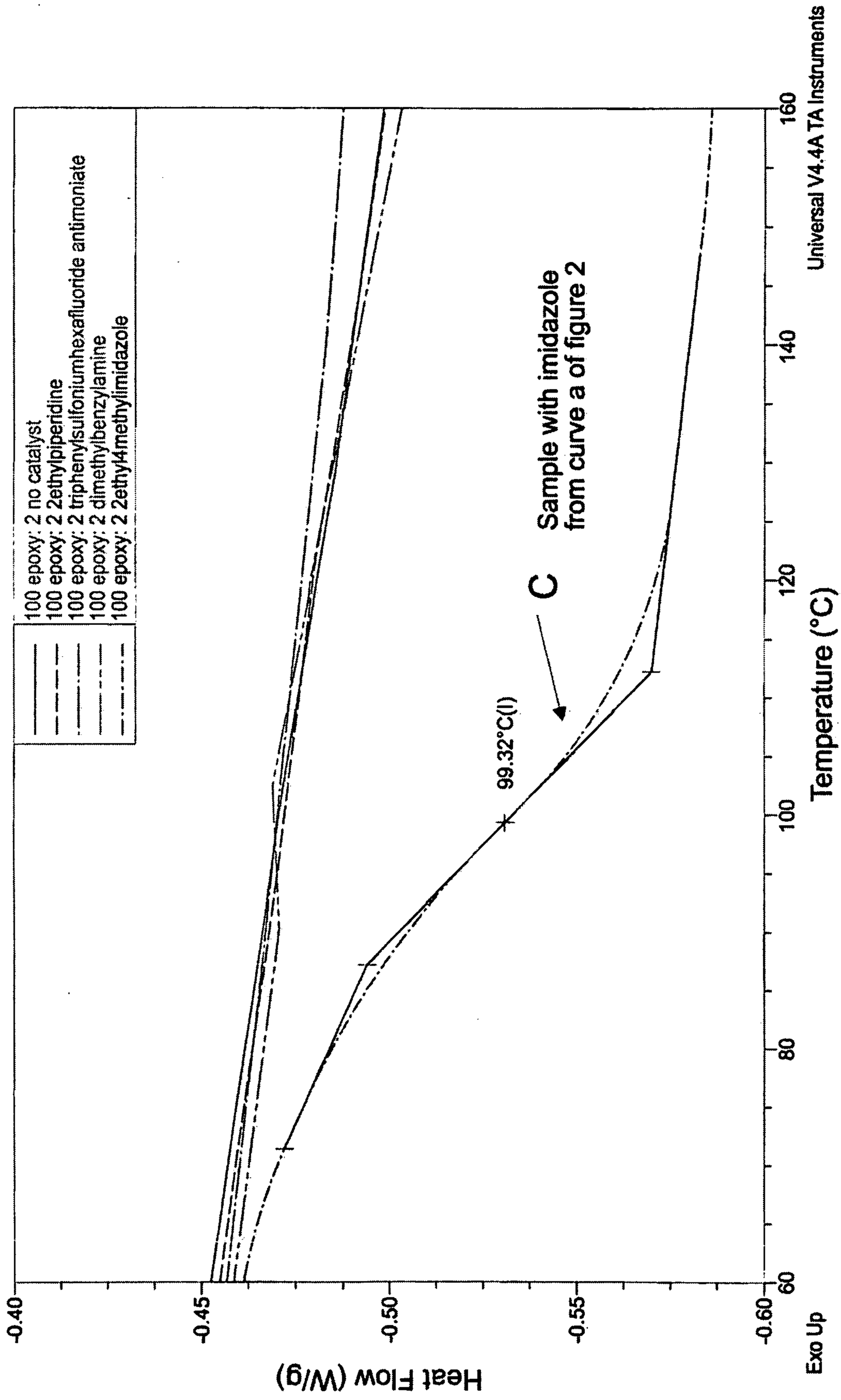

100 epoxy: 2 no catalyst
100 epoxy: 2 2ethylpiperidine
100 epoxy: 2 triphenylsulfoniumhexafluoride antimoniate
100 epoxy: 2 dimethylbenzylamine
100 epoxy: 2 2ethyl4methylimidazole
C
Sample with imidazole
from curve a of figure 2
99.32°C(l)
Temperature (°C)
Universal V4.4A TA Instruments
Heat Flow (W/g)
Exo Up
-0.40
-0.45
-0.50
-0.55
-0.60
60
80
100
120
140
160

Fig. 4

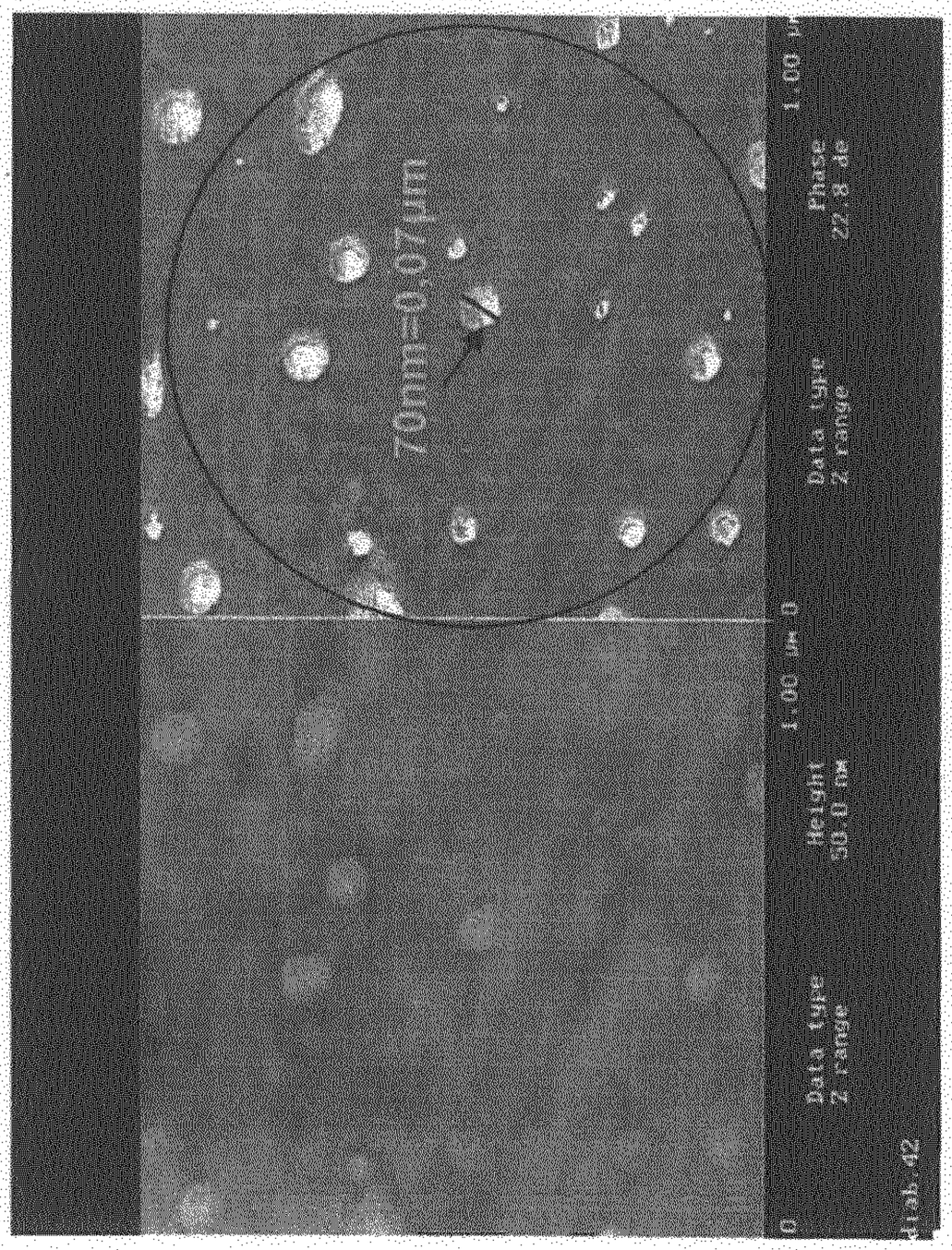
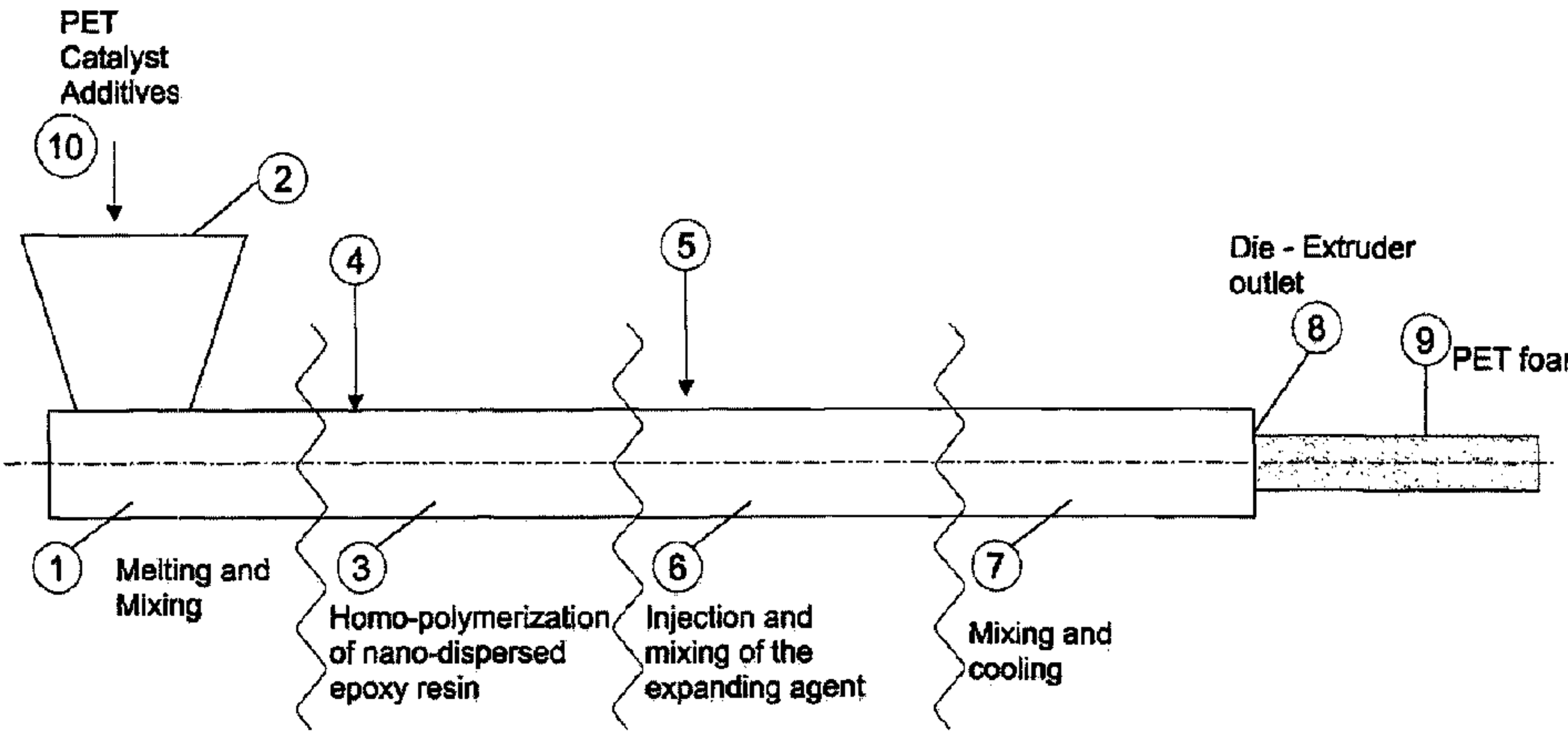

5% epoxy – catalyst (0.1% imidazole) in 94.9% PET
70mm=0.07µm
Data type
Z range
Height
50.0 nm
Data type
Z range
Phase
27.8 de

Fig.5


PET
Catalyst
Additives
10
2
4
5
Die - Extruder
outlet
8
9
PET foam
1
Melting and
Mixing
3
Homo-polymerization
of nano-dispersed
epoxy resin
6
Injection and
mixing of the
expanding agent
7
Mixing and
cooling