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(54) **POLYMERISATION DE CYCLOALCENES**

(54) **POLYMERISATION OF CYCLOALKENES**

(57) On produit des polyalcénamères en polymérisant des cycloalcènes par ouverture de noyau. La polymérisation s'effectue en présence d'un système catalyseur homogène comprenant (a) un sel d'un métal de transition choisi dans le groupe VIb du tableau périodique; (b) un composé organométallique de métal choisi dans les groupes III et IVa du tableau périodique; et (c) un polymère organo-silique représenté par la formule (Ia), (Ib) ou (Ic), R étant identique ou différent et représentant un atome d'hydrogène, un groupe phényle ou alkyle C₁-C₄; R' représentant un groupe phényle ou alkyle C₁-C₄; 0 < x < 3; 0 < y < 4; z = 0 ou 1; y + z > 1; x + y + z = 4; le rapport molaire entre le composant (c) et le composant (a) étant d'au moins 0,5:1. Le système catalyseur selon l'invention permet d'augmenter sensiblement la vitesse de polymérisation.

(57) Polyalkenamers are produced by the ring-opening polymerization of cycloalkenes. The polymerization is conducted in the presence of a homogeneous catalyst system comprising (a) a salt of a transition metal selected from the Group VIb of the periodic table; (b) an organometallic compound of a metal selected from Groups IIIa and IVa of the periodic table; and (c) an organo-silicon compound having the formula: R_xSi(OR')_y(OSiR₃)_z (Ia), (R₂SiO)₃ (Ib) or (R₃Si)₂NH (Ic), wherein R is identical or different and represents a hydrogen atom, a C₁-C₄ alkyl or phenyl group; R' represents a C₁-C₄ alkyl or phenyl group; 0 < x < 3; 0 < y < 4; z = 0 or 1; y + z > 1; x + y + z = 4; and wherein the molar ratio of component (c) to component (a) is at least about 0.5:1. The catalyst system of the invention enables one to significantly increase the rate of polymerization.





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(54) Title: POLYMERISATION OF CYCLOALKENES

(57) Abstract

Polyalkenamers are produced by the ring-opening polymerization of cycloalkenes. The polymerization is conducted in the presence of a homogeneous catalyst system comprising (a) a salt of a transition metal selected from the Group VIb of the periodic table; (b) an organometallic compound of a metal selected from Groups IIIa and IVa of the periodic table; and (c) an organo-silicon compound having the formula: $R_xSi(OR')_y(OSiR_3)_z$ (Ia), $(R_2SiO)_3$ (Ib) or $(R_3Si)_2NH$ (Ic), wherein R is identical or different and represents a hydrogen atom, a C₁-C₄ alkyl or phenyl group; R' represents a C₁-C₄ alkyl or phenyl group; $0 < x \leq 3$; $0 \leq y < 4$; $z = 0$ or 1 ; $y + z \geq 1$; $x + y + z = 4$; and wherein the molar ratio of component (c) to component (a) is at least about 0.5:1. The catalyst system of the invention enables one to significantly increase the rate of polymerization.

"POLYMERIZATION OF CYCLOALKENES"

TECHNICAL FIELD

5 The present invention pertains to improvements in the field of polymerization catalysts. More particularly, the invention relates to an improved catalyst system for the ring-opening polymerization of cycloalkenes to produce polyalkenamers as well as to a process for the production of polyalkenamers utilizing such a catalyst system.

BACKGROUND ART

15 Polyalkenamers play an important role in the rubber industry as they enter in the manufacture of synthetic rubbers. Some of these polymers are produced by a catalyzed ring-opening polymerization of cycloalkenes. The catalysts employed are generally two-component catalytic systems comprising a salt of a transition metal from Group VIb or VIIb of the Periodic Table and an organometallic compound of a metal from Groups Ia to IVa of the Periodic Table. The most common catalyst systems are based on tungsten or molybdenum salts, such as the well known system 20 $WCl_6/Sn(CH_3)_4$. However, the ring-opening polymerization of cycloalkenes such as cyclooctene with this system may take from several days to several weeks to reach completion.

30 In order to accelerate the polymerization, it is known to add a third catalyst component containing oxygen, such as water or an alcohol. For instance, the use of ethanol as an accelerator with the catalyst system $WCl_6/C_2H_5AlCl_2$ for polymerizing cyclopentene in

benzene has been reported by E. A. Ostead et al in Die Makromolekulare Chemie, Vol. 154 (1972), pages 21-34. The amount of ethanol added must be strictly controlled as too little ethanol does not provide the desired accelerating effect and the reaction is extremely slow. Too much ethanol, on the other hand, has been found to terminate the reaction and inhibit same so that the polymerization cannot be re-initiated.

10 DISCLOSURE OF THE INVENTION

It is therefore an object of the present invention to overcome the above drawbacks and to provide an improved catalyst system for the ring-opening polymerization of cycloalkenes, whereby to increase the rate of polymerization.

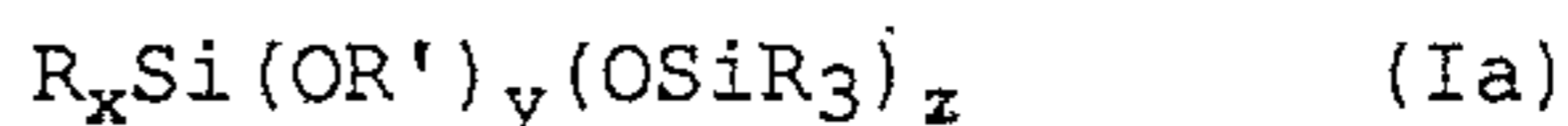
It is another object of the invention to provide a catalyst system which does not adversely affect the polymerization if an excess of the catalyst system is used.

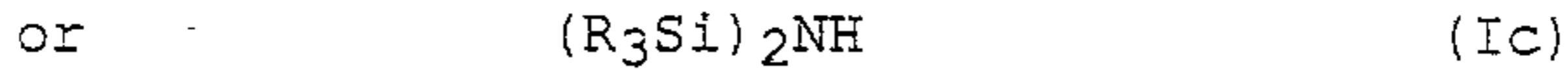
20 In accordance with the present invention, there is thus provided a homogeneous catalyst system for the ring-opening polymerization of cycloalkenes to produce polyalkenamers, comprising:

25 a) a salt of a transition metal selected from the Group VIb of the Periodic Table;

b) an organometallic compound of a metal selected from Groups IIIa and IVa of the Periodic Table; and

30 c) an organosilicon compound having the formula:





5

wherein:

R is identical or different and represents a hydrogen atom, a C₁-C₄ alkyl or phenyl group;

10

R' represents a C₁-C₄ alkyl or phenyl group;

$$0 < x \leq 3;$$

$$0 \leq y < 4;$$

$$z=0 \text{ or } 1;$$

15

$$y+z \geq 1;$$

$$x+y+z=4;$$

with the proviso that $x=1$ and $y=3$ when $z=0$;

and wherein the molar ratio of component (c) to component (a) is at least about 0.5:1.

20

Applicant has found quite unexpectedly that organosilicon compounds of formula (Ia), (Ib) or (Ic) as defined above can be used to activate known two-component catalytic systems, such as the system $WCl_6/Sn(CH_3)_4$, thereby increasing the rate of polymerization of cycloalkenes. These organosilicon compounds may be used in excess relative to the other catalyst components without adversely affecting the polymerization.

25

30

The catalyst system of the invention is prepared by first reacting component (a) with component (b), and thereafter adding component (c). The reaction of the transition metal salt with the organometallic compound is known to generate a carbene ligand.

Representative examples of the transition metal salts which may be used as the first catalyst component (a) are tungsten and molybdenum halides, such as molybdenum trichloride, molybdenum tetrachloride, molybdenum pentachloride, tungsten dichloride, tungsten tetrachloride, tungsten pentachloride, tungsten hexachloride, molybdenum dibromide, molybdenum tribromide, molybdenum tetrabromide, tungsten dibromide, tungsten pentabromide, tungsten hexabromide, molybdenum hexafluoride, tungsten hexafluoride, tungsten diiodide and tungsten tetraiodide. It is usually preferred to employ tungsten hexachloride.

Representative examples of organometallic compounds useful as the second catalyst component (b) include aluminum and tin compounds. Representative of such compounds are trialkylaluminums such as trimethylaluminum, triethylaluminum, tri-n-propylaluminum, tri-n-butylaluminum, triisopropylaluminum, triisobutylaluminum, trihexylaluminum, trioctylaluminum and the like; dialkylaluminum halides such as diethylaluminum chloride, di-n-propylaluminum chloride, diisobutylaluminum chloride, diethylaluminum bromide, diethylaluminum iodide, diethylaluminum fluoride and the like; alkylaluminum dihalides such as ethylaluminum dichloride, ethylaluminum dibromide, propylaluminum dichloride, isobutylaluminum dichloride, ethylaluminum diiodide and the like; and tetraalkyltins such as tetramethyltin, tetraethyltins, tetra-n-propyltin, tetra-n-butyltin, tetraisopropyltin, tetraisobutyltin, tetrahexyltin, tetraoctyltin and the like. It is usually preferred to employ tetramethyltin.

Examples of suitable organosiloxanes of formula (Ia) or (Ib) which may be used as the third catalyst component (c) include phenyl-triethoxysiloxane, 1,1,3,3-tetramethyldisiloxane, 5 hexamethyldisiloxane and hexamethylcyclotri-siloxane. It is also possible to use an organo-silazane of formula (Ic), such as 1,1,3,3-tetramethyldisilazane, 1,3-diphenyl-1,1,3,3-tetra-methyldisilazane or hexamethyldisilazane.

10 The molar ratio of the organosilicon compound to the transition metal salt must be at least $0.5(\pm 0.1):1$, since at a lesser molar ratio the organosilicon compound does not have any substantial activating effect. Preferred molar ratios 15 of components (a):(b):(c) are 1:2:2.3 and 1:2:4.7.

The ring-opening polymerization of cycloalkenes with the catalyst system of the invention is generally carried out at a tempera- 20 ture ranging from about 0° to about 160°C . At a temperature above 160°C , there is a tendency for the catalyst system to decompose. The polymeriza- tion can be carried out in bulk or in solution since the catalyst system is homogeneous and thus soluble in the monomer or solvent.

25 Applicant has also found quite unexpected- edly that saturated polycyclic oligomers can be produced by conducting the polymerization of cycloalkenes in the presence of the above catalyst system, but containing no organometallic compound. 30 It is believed that the polymerization proceeds through a mechanism different than ring-opening, such as a cationic mechanism. The saturated poly- cyclic oligomers produced are stable at high

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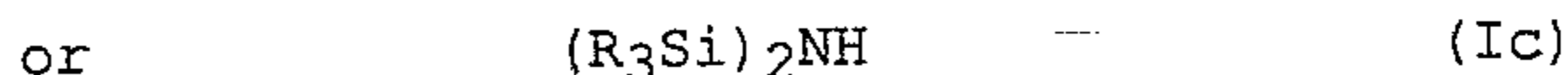
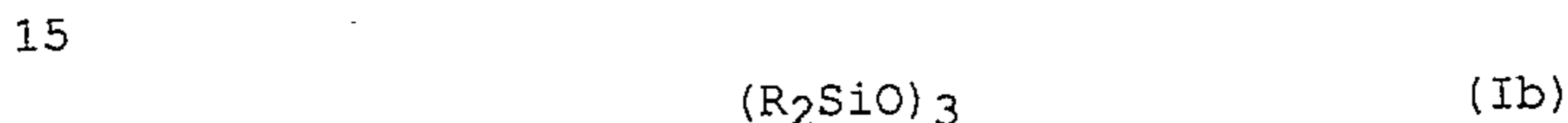
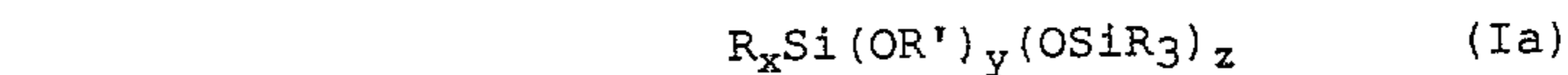
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temperature and can thus be used as lubricants or release agents.

The present invention therefore provides, in another aspect thereof, a homogeneous catalyst system for the polymerization of cycloalkenes to produce saturated polycyclic oligomers, comprising:

a) a salt of a transition metal selected from the Group VIb of the Periodic Table; and

b) an organosilicon compound having the formula:



wherein:

R is identical or different and represents a hydrogen atom, a C₁-C₄ alkyl or phenyl group;

R' represents a C₁-C₄ alkyl or phenyl group;

$$0 < x \leq 3;$$

$$0 \leq y < 4;$$

$$z=0 \text{ or } 1;$$

$$y+z \geq 1;$$

$$x+y+z=4;$$

and wherein the molar ratio of component (b) to component (a) is at least about 0.5:1.

BRIEF DESCRIPTION OF DRAWINGS

Further features and advantages of the invention will become more readily apparent from the following non-limiting examples and the accompanying drawings, in which:

Figure 1 graphically illustrates the degree of conversion with time for the bulk polymerization of cyclooctene at 45.0°C catalyzed with $WCl_6/Sn(CH_3)_4/HMDS$ having a molar ratio of 1:2:4.7, where HMDS represents hexamethyldisiloxane;

Figure 2 graphically illustrates the degree of conversion with time for the bulk polymerization of cyclooctene at 103.3°C catalyzed with $WCl_6/Sn(CH_3)_4/HMDS$ having a molar ratio of 1:2:4.7;

Figure 3 graphically illustrates the degree of conversion with time for the bulk polymerization of cyclooctene at 157.3°C catalyzed with $WCl_6/Sn(CH_3)_4/HMDS$ having a molar ratio of 1:2:4.7;

Figure 4 graphically illustrates the relation between the molar ratio of $HMDS:WCl_6$ and $\ln t$, where t is the time required to reach equilibrium;

Figure 5 graphically illustrates the relation between $\ln (HMDS/WCl_6)$ and $\ln t$; and

Figure 6 which is on the same sheet of drawings as Fig. 3, graphically illustrates the degree of conversion with time for the bulk polymerization of cyclooctene at 100°C catalyzed with $WCl_6/Sn(CH_3)_4/HMDS$ having a molar ratio of 1:2:0.01.

EXAMPLE 1

The bulk polymerization of cyclooctene was carried out under dry argon in order to eliminate water and oxygen which react with WCl_6 and destroys its activity. The monomer (99% pure) was distilled and dried over calcium hydride for at least one hour prior to its use. WCl_6 99.9% pure was kept under dry argon. $Sn(CH_3)_4$ 99% pure was used without further purification and kept under dry argon. Hexamethyldisiloxane (HMDS) 99% pure was also kept under dry argon.

For the polymerization, WCl_6 was transferred into a dry ampoule through a side arm and then closed with a septum. $Sn(CH_3)_4$ was added with a syringe through the septum. The mixture was cooled, the side arm sealed and removed. The mixture was warmed up to room temperature and allowed to react for about 20 seconds. Then the monomer containing 4.7 moles of HMDS was transferred into the ampoule, cooled down and the ampoule sealed off. The ampoule was plunged into water, five minutes being necessary for melting of the mixture. The ampoule was then shaken in order to ensure homogeneity of the mixture and placed in a thermoregulated bath set at a temperature of 45°C.

The polymerization was stopped by the addition of methanol. The polymer samples were analyzed through nuclear magnetic resonance, gel permeation chromatography and mass spectrometry. The samples comprised high-molecular-weight unsaturated linear polymer and unsaturated oligomers. The proportion of oligomers varied with polymerization time. In this case, the proportion

was very high (as high as 80%) in the first few minutes and decreased with time.

5 In the experiment, 3 g of cyclooctene were polymerized using 10 mg of WCl_6 . The molar composition of the catalyst system $WCl_6/Sn(CH_3)_3/HMDS$ was 1:2:4.7.

10 As shown in Figure 1, the degree of conversion is plotted against time for the polymerization at 45°C. After approximately 5 minutes (0.1 h), the % conversion reached 50%. The % conversion levelled off at about 80%. The conversion did not go higher due to the formation of a gel, the monomer being trapped in the gel, preventing higher conversion.

15 **EXAMPLE 2**

The same polymerization as described in Example 1 was carried out at 103°C and 157°C. Figures 2 and 3 show the conversion against time. At these temperatures, the % conversion reached almost 100%. At 103°C, the conversion was approximately 65% after 3 min. and about 80% at 157°C after the same time.

20 **EXAMPLE 3**

25 The bulk polymerization of cyclooctene was carried out between 100° and 115°C using the following organosilicon compounds as accelerators:

- 30 - phenyltriethoxysiloxane (PTEOS)
- 1,1,3,3-tetramethyldisiloxane (TMDS)
- hexamethyldisiloxane (HMDS)
- hexamethylcyclotrisiloxane (HMCTS)
- hexamethyldisilane (HMDSi)
- 1,1,3,3-tetramethyldisilazane (TMDZ)
- hexamethyldisilazane (HMDZ)
- heptamethyldisilazane (HPMDZ)

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The molar composition of the catalyst system $WCl_6/Sn(CH_3)_4$ /accelerator was 1:2:5.

5 The efficiency of the accelerators in activating the system $WCl_6/Sn(CH_3)_4$ and accelerating the rate of polymerization was obtained through comparison of the yield obtained after 8 hours. The following data were obtained:

<u>Accelerator</u>	<u>Yield (%)</u>
TMDS	100
HMCTS	88
PTEOS	67
HMDS	65
HMDZ	35
TMDZ	35
HPMDZ	inactive
HMDSi	inactive

10 Based on the above results, the following qualitative efficiency scale was obtained:

TMDS > HMCTS > PTEOS, HMDS >
HMDZ, TMDZ >> HPMDZ, HMDSi = 0

15 As it is apparent, the presence of a methyl group on the nitrogen atom in heptamethyl-disilazane inactivates the compound. The absence of a Si-O bond in hexamethyldisilane has also the same effect. Thus, a Si-O or a Si-NH-Si bond must be present in order to form an accelerator.

20 EXAMPLE 4

Using the same technique as described in Example 1, the polymerization of norbornene was carried out in cyclohexane. The catalyst system

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5 used was $WCl_6/Sn(CH_3)_4/HMDS$ with a molar ratio of 1:2:4.7. The monomer concentration was 1.88 g in 10.0 ml of cyclohexane. The amount of WCl_6 was 10 mg. At room temperature, the % conversion reached 95% in approximately two minutes. No oligomers were found and the molecular weight of the polymer produced was about 10^6 .

EXAMPLE 5

10 The polymerization of cyclopentene was carried out in benzene, in the same manner as described in Example 1. The polymerization temperature was $0^\circ C$. The catalyst system used was $WCl_6/Sn(CH_3)_4/HMDS$ with a molar ratio of 1:2:2.3. The monomer concentration was 33% and the WCl_6 concentration was 10 mg in 6 ml of monomer-solvent mixture. Because of a monomer-polymer equilibrium in this system, the polymerization never reached completion. It is known that for the polymerization of cyclopentene at $0^\circ C$, the maximum yield is 70% conversion. In the present case, this was easily achieved within 24 hours. No oligomers are found and the molecular weight of the polymer produced was about 10^6 .

EXAMPLE 6

25 The bulk polymerization of cyclooctene at $160^\circ C$ using the catalyst system $WCl_6/HMDS$ with a molar ratio of 1:4.7 led to the formation of low-molecular-weight polymer ($< 10^3$). The polymer produced comprised a mixture of dimers, trimers and higher oligomers. The polymer yield was 50% and was made of saturated and unsaturated oligomers. The proportion of saturated polycyclic oligomer was about 80% and reached 90% upon repeating the experiment at $100^\circ C$.

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EXAMPLE 7

The bulk polymerization of cyclooctene was carried out at 103°C in the same manner as described in Example 1, using the catalyst system $WCl_6/Sn(CH_3)_4/HMDS$. The $WCl_6/Sn(CH_3)_4$ molar ratio was 1:2. The $HMDS/WCl_6$ ratio was allowed to vary and the time required to obtain equilibrium, measured accordingly. The $HMDS/WCl_6$ ratio is plotted against $\ln t$ (time in hours) in Figure 4. As shown in Figure 5, when $\ln (HMDS/WCl_6)$ is plotted against $\ln t$, using the same data and interpolated values between 5.0 and 7.0 for $\ln t$, then two straight lines are obtained with a break-up point corresponding to a molar ratio of $HMDS/WCl_6$ equal to 0.5 (± 0.1). This molar ratio is the minimum ratio at which the organosilicon compound provides a substantial activating effect on the polymerization.

COMPARATIVE EXAMPLE

The polymerization described in Example 1 was repeated, but the proportion of $HMDS$ was reduced to a molar ratio of $HMDS/WCl_6$ equal to 0.01. Figure 6 shows the % conversion with time in terms of days. This can be compared with Figure 2 which shows that for nearly the same temperature, the % conversion is about 65% within 5 minutes, using a $HMDS/WCl_6$ molar ratio equal to 4.7.

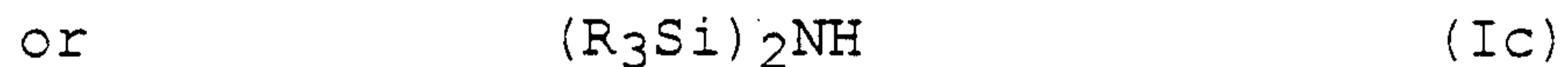
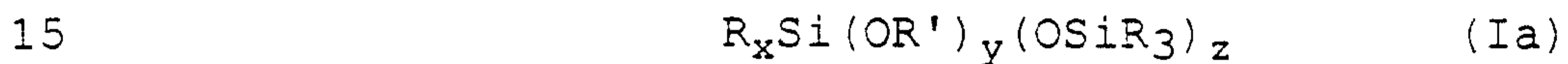
CLAIMS:

1. A homogeneous catalyst system for the ring-opening polymerization of cycloalkenes to produce polyalkenamers, comprising:

a) a salt of a transition metal selected from the Group VIb of the Periodic Table;

b) an organometallic compound of a metal selected from Groups IIIa and IVa of the Periodic Table; and

c) an organosilicon compound having the formula:



wherein:

R is identical or different and represents a hydrogen atom, a C₁-C₄ alkyl or phenyl group;

R' represents a C₁-C₄ alkyl or phenyl group;

$$0 < x \leq 3;$$

$$0 \leq y < 4;$$

$$z=0 \text{ or } 1;$$

$$y+z \geq 1;$$

$$x+y+z=4;$$

with the proviso that x=1 and y=3 when z=0; and wherein the molar ratio of component (c) to component (a) is at least about 0.5:1.

2. A catalyst system according to claim 1, wherein component (a) is a tungsten or molybdenum halide.
- 5 3. A catalyst system according to claim 2, wherein component (a) is tungsten hexachloride.
4. A catalyst system according to claim 1, wherein component (b) is an organoaluminum or
10 organotin compound.
5. A catalyst system according to claim 4, wherein component (b) is tetramethyltin.
- 15 6. A catalyst system according to claim 3, wherein component (b) is tetramethyltin.
7. A catalyst system according to claim 1, wherein component (c) is an organosiloxane
20 selected from the group consisting of phenyl-triethoxysiloxane, 1,1,3,3-tetramethyldisiloxane, hexamethyldisiloxane and hexamethylcyclotri-siloxane.
- 25 8. A catalyst system according to claim 7, wherein component (c) is hexamethyldisiloxane.
9. A catalyst system according to claim 6, wherein component (c) is hexamethyldisiloxane.
30
10. A catalyst system according to claim 9, wherein the molar ratio of components (a):(b):(c) is 1:2:2.3 or 1:2:4.7.

11. A catalyst system according to claim 1,
 wherein component (c) is an organosilazane
 selected from the group consisting of 1,1,3,3-
 tetramethyldisilazane, 1,3-phenyl-1,1,3,3-tetra-
 5 methyldisilazane and hexamethyldisilazane.

12. A process for the production of
 polyalkenamers by the ring-opening polymerization
 of cycloalkenes, which comprises conducting the
 10 polymerization at a temperature ranging from
 about 0° to about 160°C in the presence of a
 homogeneous catalyst system comprising:

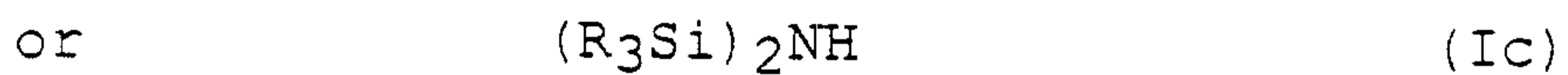
a) a salt of a transition metal
 selected from the Group VIb of the Periodic
 15 Table;

b) an organometallic compound of a
 metal selected from Groups IIIa and IVa of the
 Periodic Table; and

c) an organosilicon compound having the
 20 formula:



25



wherein:

R is identical or different and repre-
 30 sents a hydrogen atom, a C₁-C₄ alkyl or phenyl
 group;

R' represents a C₁-C₄ alkyl or phenyl
 group;

$$0 < x \leq 3;$$

$$\begin{aligned} 0 &\leq y < 4; \\ z &= 0 \text{ or } 1; \\ y+z &\geq 1; \\ x+y+z &= 4; \end{aligned}$$

5 with the proviso that $x=1$ and $y=3$ when $z=0$;
and wherein the molar ratio of component (c) to
component (a) is at least about 0.5:1.

10 13. A process according to claim 12,
wherein component (a) of said catalyst system is
a tungsten or molybdenum halide.

15 14. A process according to claim 13,
wherein component (a) of said catalyst system is
tungsten hexachloride.

20 15. A process according to claim 12,
wherein component (b) of said catalyst system is
an organoaluminum or organotin compound.

16. A process according to claim 15,
wherein component (b) of said catalyst system is
tetramethyltin.

25 17. A process according to claim 14,
wherein component (b) of said catalyst system is
tetramethyltin.

30 18. A process according to claim 12,
wherein component (c) of said catalyst system is
an organosiloxane selected from the group
consisting of phenyltriethoxysiloxane, 1,1,3,3-
tetramethyldisiloxane, hexamethyldisiloxane and
hexamethylcyclotrisiloxane.

19. A process according to claim 18, wherein component (c) of said catalyst system is hexamethyldisiloxane.
- 5 20. A process according to claim 17, wherein component (c) of said catalyst system is hexamethyldisiloxane.
- 10 21. A process according to claim 20, wherein the molar ratio of components (a):(b):(c) of said catalyst system is 1:2:2.3 or 1:2:4.7.
- 15 22. A process according to claim 12, wherein component (c) of said catalyst system is an organosilazane selected from the group consisting of 1,1,3,3-tetramethyldisilazane, 1,3-diphenyl-1,1,3,3-tetramethyldisilazane and hexamethyldisilazane.
- 20 23. A process according to claim 12, wherein the cycloalkene is selected from the group consisting of cyclopentene, cyclooctene and norbornene.
- 25 24. A process according to claim 12, wherein the cycloalkene is cyclooctene.

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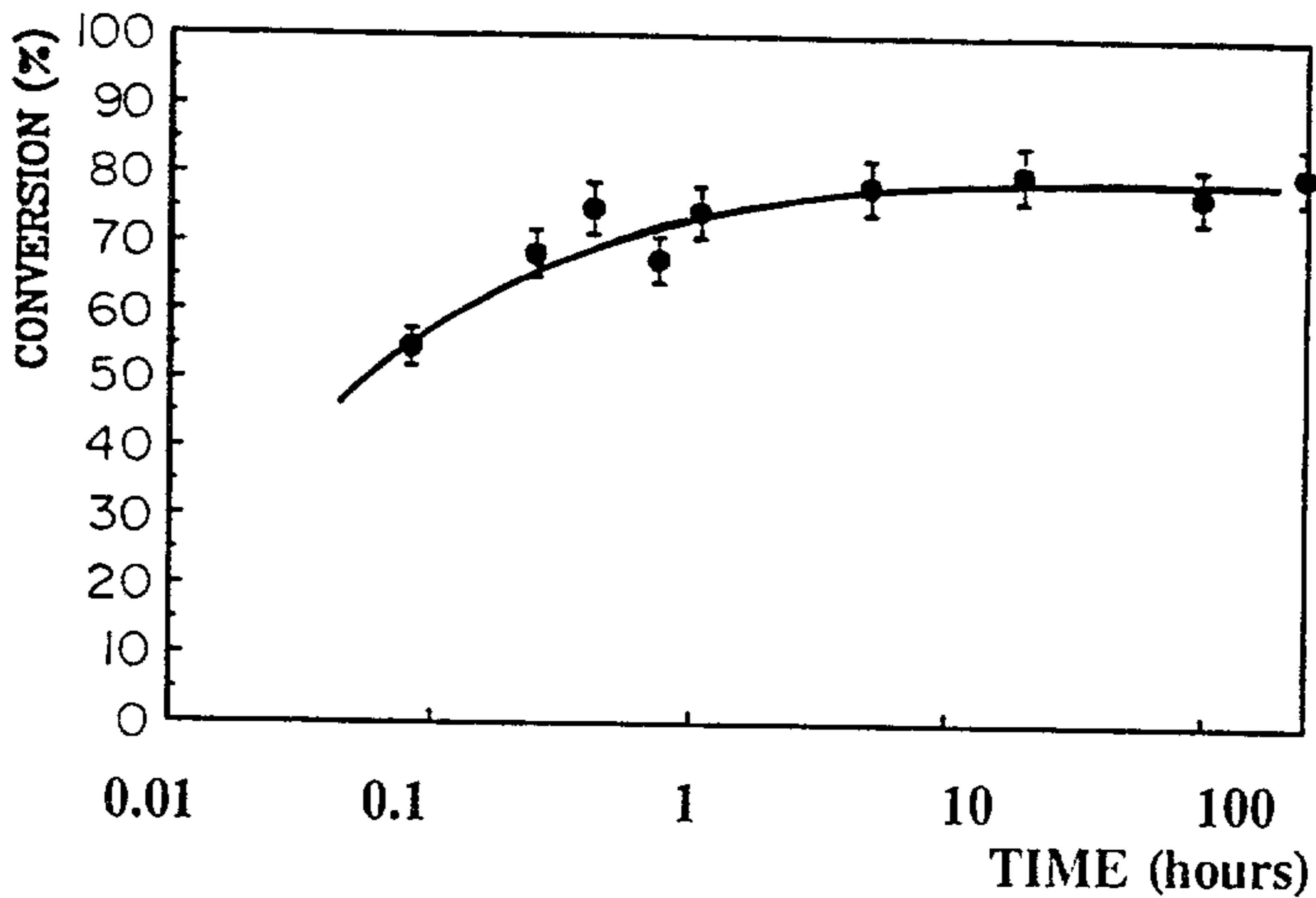


FIG. 1

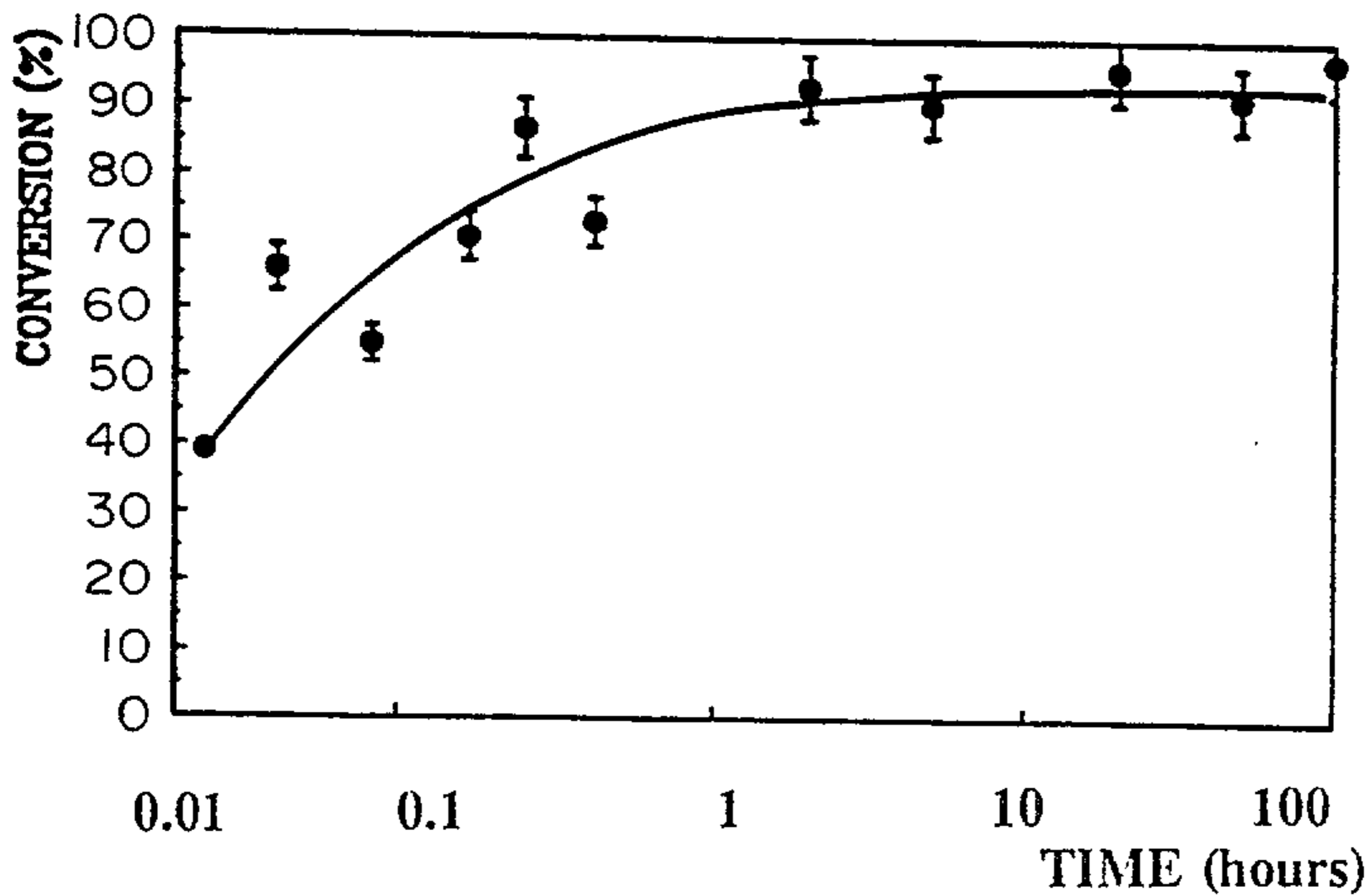


FIG. 2

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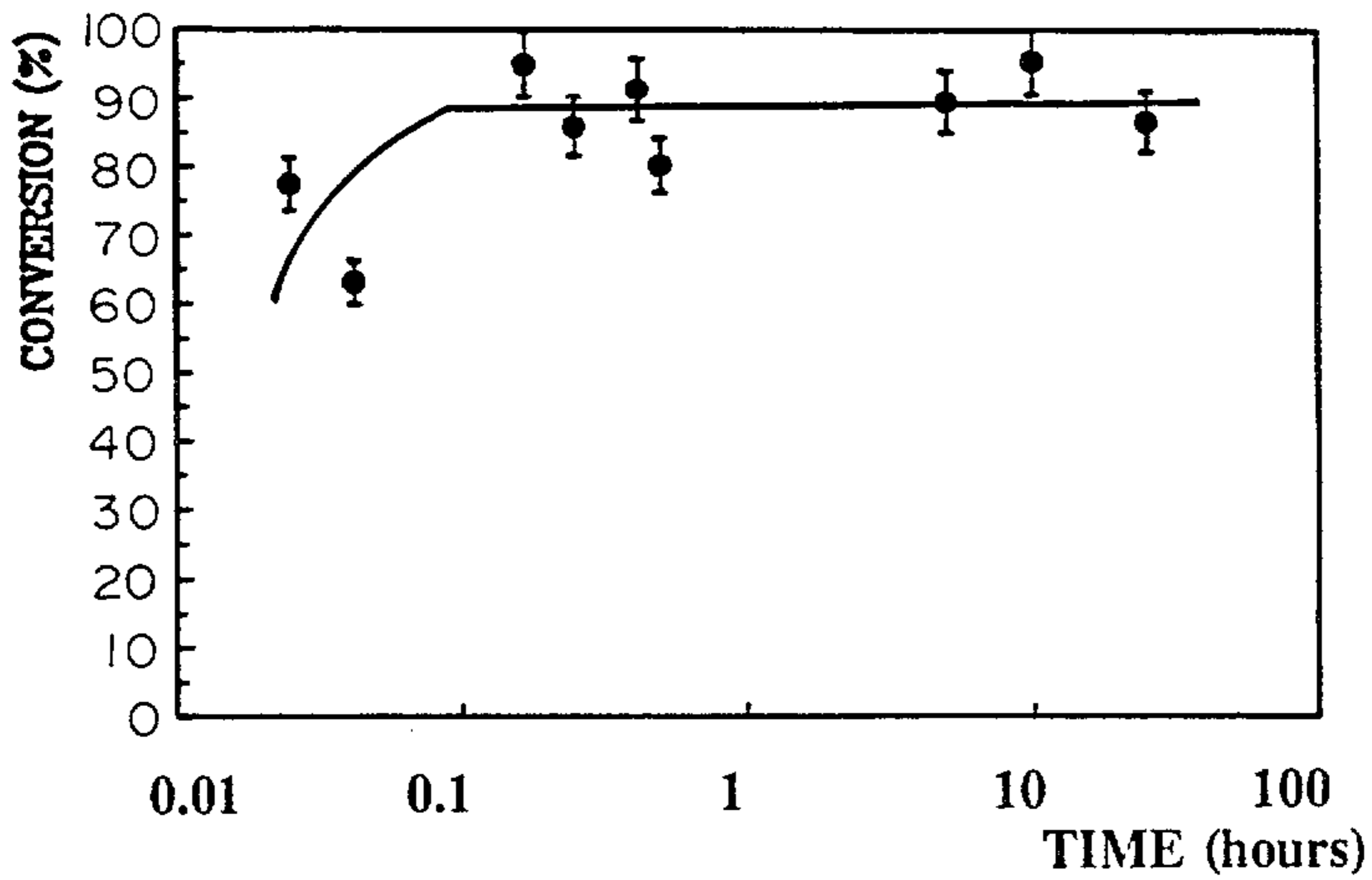


FIG. 3

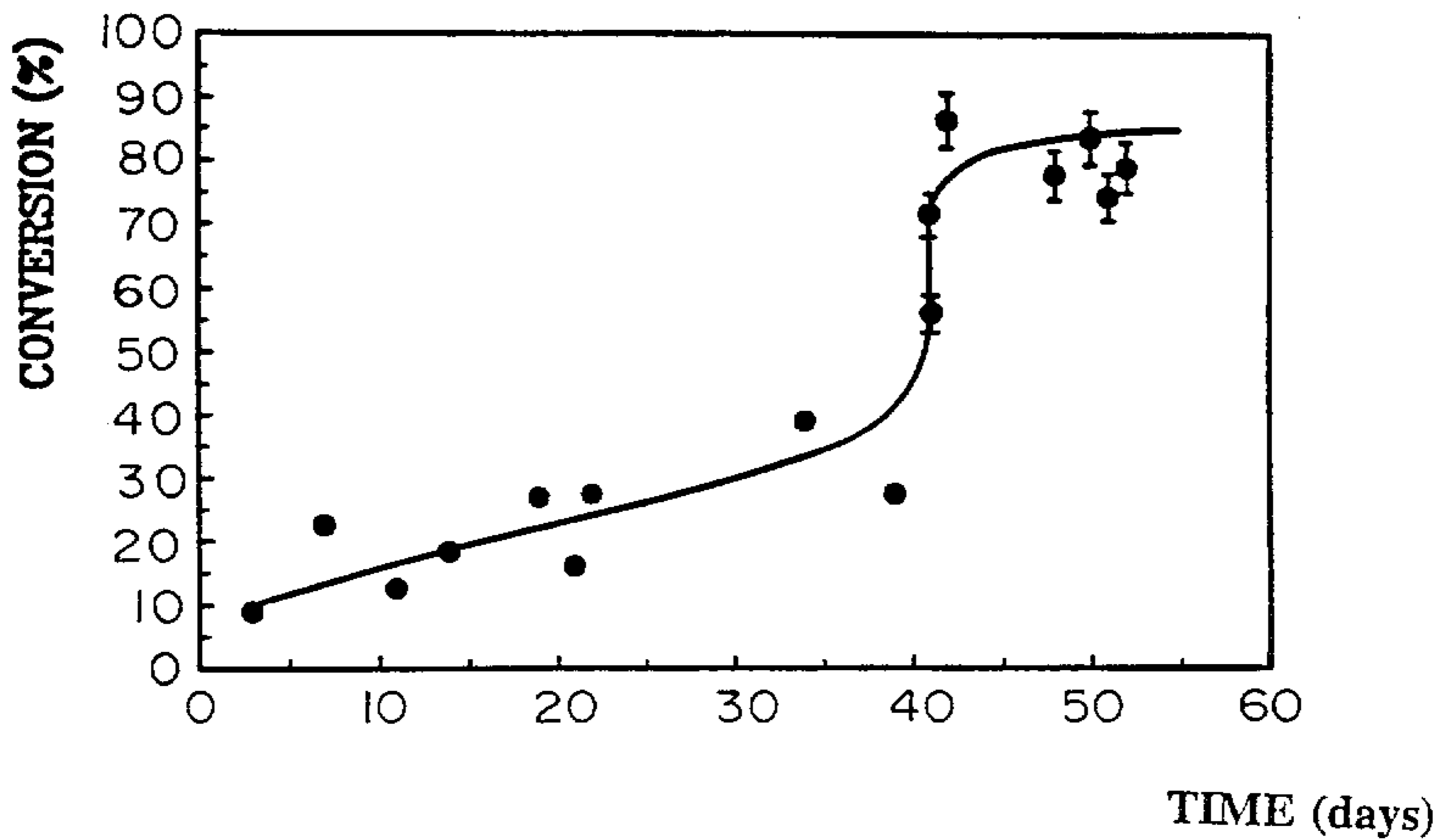


FIG. 6

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