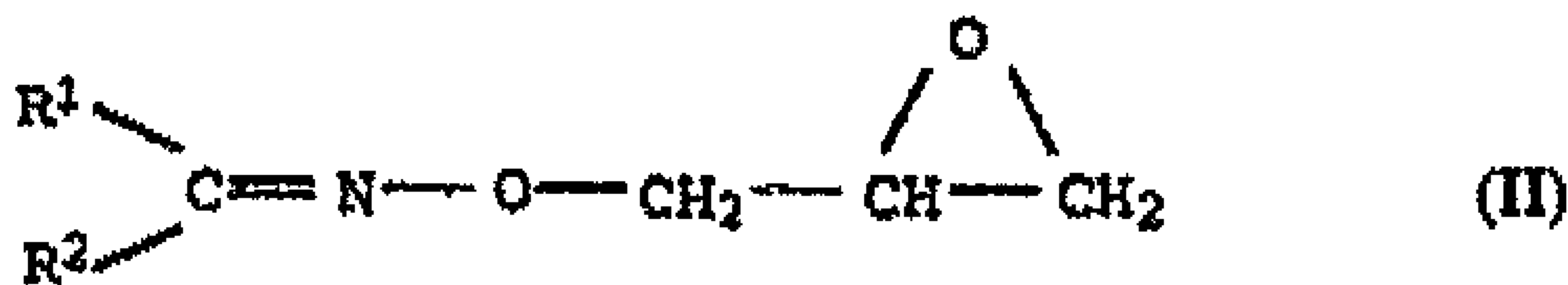
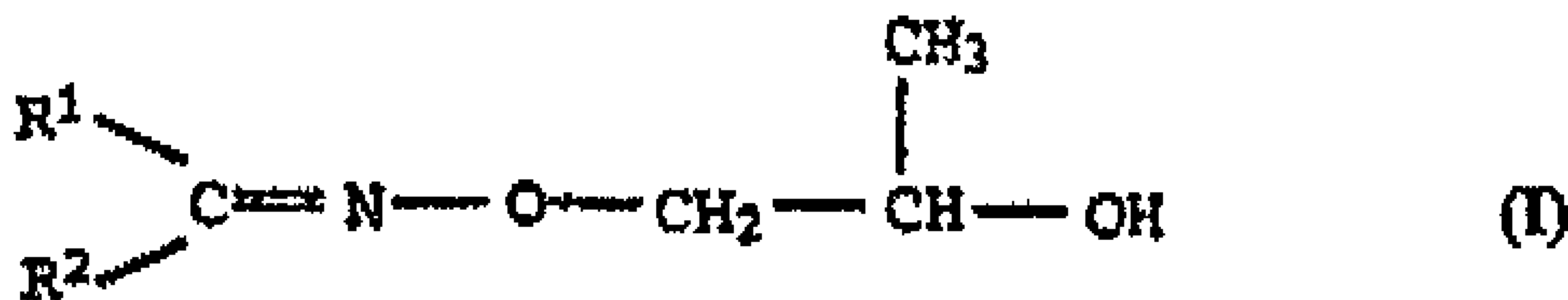




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(54) Titre : PROCEDE DE PRODUCTION DE O-(2-HYDROXYALKYL)-OXIMES  
 (54) Title: PREPARATION OF O-(2-HYDROXYALKYL)OXIMES



(57) Abrégé/Abstract:

A method is disclosed of producing O-(2-hydroxyalkyl)-oximes of formula (1), wherein R<sup>1</sup> and R<sup>2</sup> represent an alkyl, or R<sup>1</sup> and R<sup>2</sup> and the carbon atom which carries them represent a 5- to 7-membered cycloalkyl, by the catalytic hydration of O-(2,3-epoxyalkyl)-oxime of general formula (II) with hydrogen. The products of formats (I) are suitable for use as intermediate products in the production of herbicides.

PCT

WELTORGANISATION FÜR GEISTIGES EIGENTUM  
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INTERNATIONALE ANMELDUNG VERÖFFENTLICHT NACH DEM VERTRAG ÜBER DIE  
INTERNATIONALE ZUSAMMENARBEIT AUF DEM GEBIET DES PATENTWESENS (PCT)

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<p>(21) Internationales Aktenzeichen: PCT/EP95/02932</p> <p>(22) Internationales Anmeldedatum: 25. Juli 1995 (25.07.95)</p> <p>(30) Prioritätsdaten: P 44 27 290.1      2. August 1994 (02.08.94)      DE</p> <p>(71) Anmelder (für alle Bestimmungsstaaten ausser US): BASF AKTIENGESELLSCHAFT [DE/DE]; D-67056 Ludwigshafen (DE).</p> <p>(72) Erfinder; und (75) Erfinder/Anmelder (nur für US): HARREUS, Albrecht [DE/DE]; Teichgasse 13, D-67063 Ludwigshafen (DE). GÖTZ, Norbert [DE/DE]; Schöfferstrasse 25, D-67547 Worms (DE). RANG, Harald [DE/DE]; Ziegeleistrasse 76, D-67122 Altrip (DE).</p> <p>(74) Gemeinsamer Vertreter: BASF AKTIENGESELLSCHAFT; D-67056 Ludwigshafen (DE).</p>	<p>(81) Bestimmungsstaaten: AU, BG, BR, BY, CA, CN, CZ, FI, HU, JP, KR, KZ, MX, NO, NZ, PL, RU, SG, SK, UA, US, UZ, europäisches Patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).</p> <p>Veröffentlicht Mit internationalem Recherchenbericht.</p> <p style="font-size: 2em; text-align: right;">45074 ✓ 2196451</p>	

(54) Title: METHOD OF PRODUCING O-(2-HYDROXYALKYL)-OXIMES

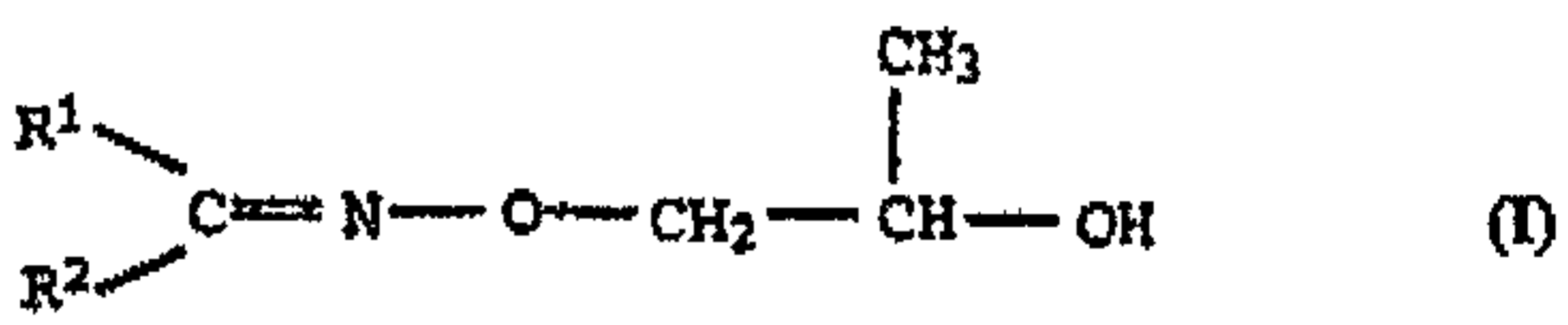
(54) Bezeichnung: VERFAHREN ZUR HERSTELLUNG VON O-(2-HYDROXYALKYL)-OXIMEN

(57) Abstract

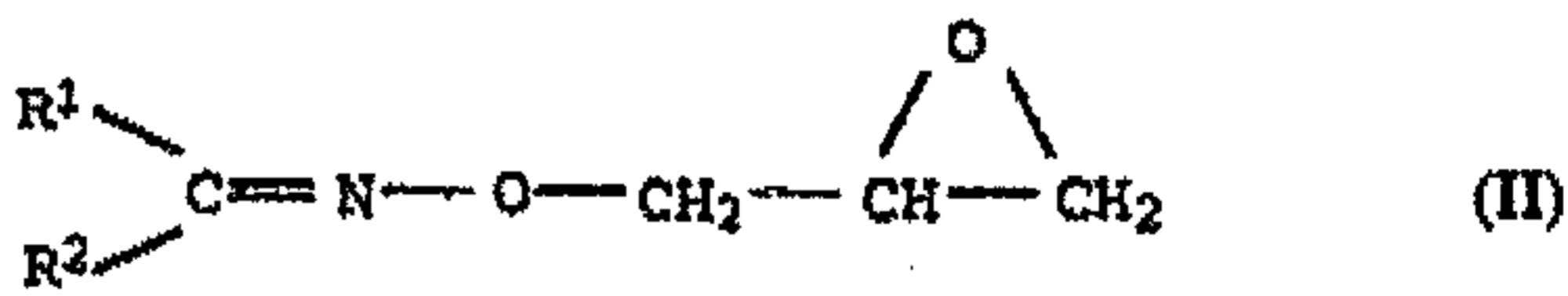
A method is disclosed of producing O-(2-hydroxyalkyl)-oximes of formula (I), wherein R<sup>1</sup> and R<sup>2</sup> represent an alkyl, or R<sup>1</sup> and R<sup>2</sup> and the carbon atom which carries them represent a 5- to 7-membered cycloalkyl, by the catalytic hydration of O-(2,3-epoxyalkyl)-oxime of general formula (II) with hydrogen. The products of formula (I) are suitable for use as intermediate products in the production of herbicides.

(57) Zusammenfassung

Herstellung von O-(2-Hydroxyalkyl)-oximen (I) (R<sup>1</sup>, R<sup>2</sup> = Alkyl oder R<sup>1</sup> und R<sup>2</sup> und das C-Atom das sie trägt = 5- bis 7-gliedriges Cycloalkyl), indem man ein O-(2,3-Epoxyalkyl)-oxim der allgemeinen Formel (II) mit Wasserstoff katalytisch hydriert. Die Verfahrensprodukte (I) eignen sich als Zwischenprodukte für Herbizide.



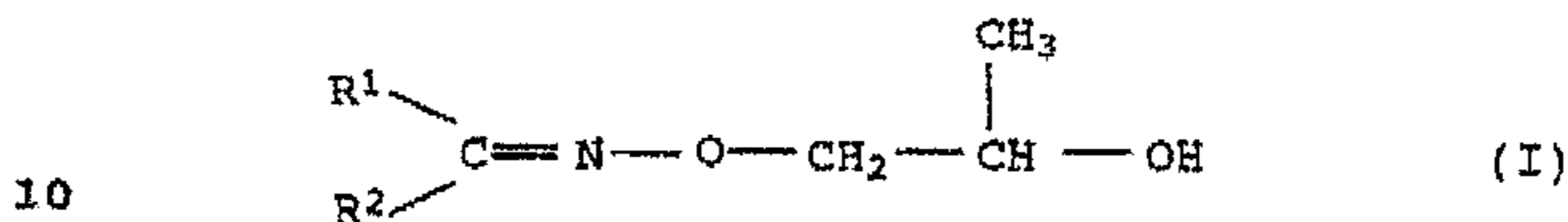
(I)



(II)

## Preparation of O-(2-hydroxyalkyl)oximes

The present invention relates to a process for preparing O-(2-hydroxyalkyl)oximes of the general formula I



where R<sup>1</sup> and R<sup>2</sup> are each alkyl groups having 1 to 10 carbon atoms or, together with the carbon atom which carries them, are bonded to give a 5- to 7-membered cycloalkyl radical.

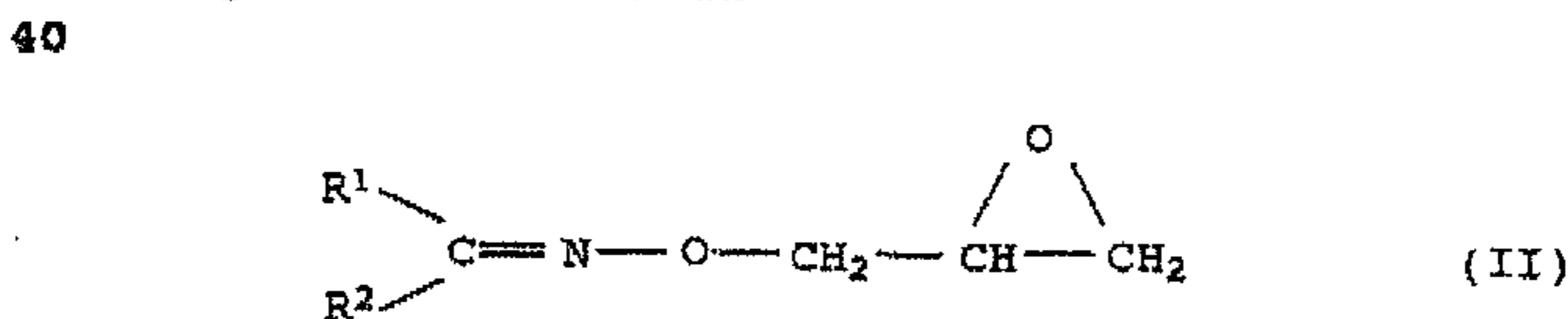
O-(2-Hydroxyalkyl)oximes are of great importance as intermediates for crop protection agents (cf. eg. the earlier German Application DE-A 44 15 887).

20 In the preparation of compounds of the type I by O-alkylation of hydroxylamines, to a certain extent the compound isomeric to I which carries the CH<sub>3</sub> group on the C atom which is adjacent to the oxime oxygen is also always formed.

25 The previously described hydrogenations of oxiranes lead either to the primary alcohol or to a mixture of secondary and primary alcohol (cf. eg. US 4,064,186 and Houben-Weyl, Methoden der Organischen Chemie [Methods of Organic Chemistry], Georg Thieme Verlag, Stuttgart, Vol. 6/3, 4th Edition, 1965, pages 442-446).

35 It is an object of the present invention to provide a process which leads with higher regioselectivity and substantially without formation of by-products to the O-(2-hydroxyalkyl)oximes I.

We have found that this object is achieved by a process for preparing the O-(2-hydroxyalkyl)oximes of the formula I, which comprises catalytically hydrogenating an O-(2,3-epoxyalkyl)oxime of the general formula II



using hydrogen.

In the following, the Houben-Weyl literature references relate to: Houben-Weyl, Methoden der Organischen Chemie (Methods of Organic Chemistry), 4th Edition, Thieme Verlag, Stuttgart.

Among the compounds I which can be prepared by the process according to the invention, those are preferred in which  $R^1$  and  $R^2$  each are a  $C_1-C_4$ -alkyl group and especially a  $C_1-C_3$ -alkyl group, or, together with the carbon atom which carries them, form a cyclopentyl or cyclohexyl ring. Particularly preferably,  $R^1$  and  $R^2$  are each methyl or ethyl, in particular both methyl.

The O-(2,3-epoxyalkyl)oximes II are generally known or are obtainable by known methods, especially by base-catalyzed reaction of the corresponding epihalohydrins with the free oximes in a dipolar aprotic solvent (cf. eg. Zh. Org. Khim. 5 (1969), page 1353 to page 1355).

Suitable catalysts for the hydrogenation are catalysts generally customary for this purpose, as are described e.g. in Houben-Weyl, Volume 4/1c.

Preferred hydrogenation catalysts are those which comprise a metal from groups 8 to 10 according to the IUPAC classification of the Periodic Table, preferably from the group consisting of the elements cobalt, ruthenium and rhodium, especially from the group consisting of the elements platinum and nickel, and in particular palladium.

The catalysts can be used as such or preferably on a support. Customary support materials such as silica, alumina, titanium dioxide, silicates and zeolites and especially activated carbon are suitable.

For preparation of the supported catalysts binders or shaping aids can be additionally used. The catalysts can be employed in the form of chips, extrudates, tablets or balls.

In general, 0.1 to 2, preferably 0.1 to 1%, by weight of catalyst are employed per mole of the compound II to be hydrogenated, these quantitative data relating to the active mass of the catalyst without support materials.

The hydrogenation can be carried out continuously or, preferably, batchwise.

In batchwise procedure in the liquid phase, the hydrogenation can be carried out in the presence of a solvent. Suitable solvents are polar solvents such as ethers and alcohols as well as mixtures thereof. Preferred solvents are ethers or alcohols having up to 6 carbon atoms such as 1,2-dimethoxyethane, ethanol, n-propanol, isopropanol and n-butanol.

The pressure can be selected within wide limits which extend from 1 to 400 bar, but the reaction is preferably carried out in a pressure range from 1 to 100, especially from 10 to 70 and in particular from 30 to 50 bar.

The temperature during the hydrogenation is preferably 0 to 100, especially 10 to 50 and in particular 25 to 30°C.

15

The reaction products I are isolated by methods known per se, preferably by distillation. The yield of the compounds I is normally from 70 to 90%, with a selectivity of, as a rule, over 98%.

20 The O-(2-hydroxyalkyl)oximes I obtainable by the process according to the invention are suitable as precursors for herbicides, in particular of the cyclohexenone type (cf. eg. DE-A 44 15 887).

25 Example

Preparation of 2-propanone O-(2-hydroxypropyl)oxime

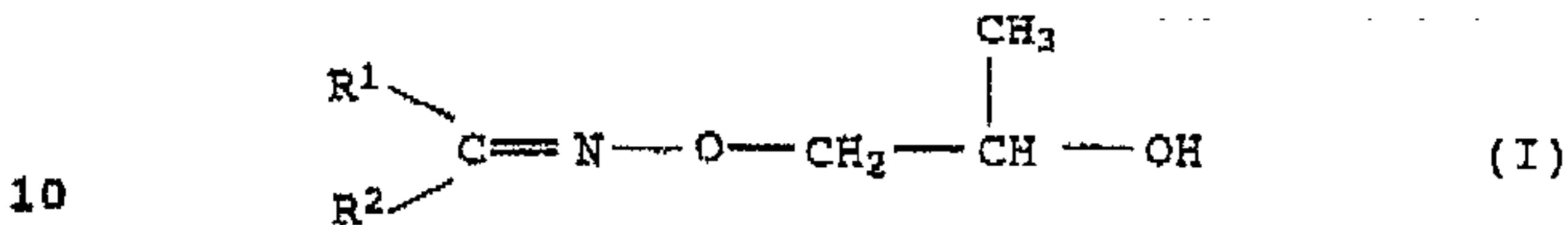
130 g (0.94 mol) of 2-propanone O-(2-epoxypropyl)oxime (cf. EP-A 23 560; Zh. Org. Khim. 5 (1969), pages 1353 to 1355) and 600 ml of ethanol were treated with 4 g of palladium/carbon (10% by weight palladium) and the mixture was hydrogenated at 25°C and a hydrogen pressure of 50 bar. After 17 hours the reaction was complete. The catalyst was filtered off. The solvent was then removed and the residue was distilled through a packed column of length 50 cm and diameter 5 cm, which was packed with wire mesh rings of diameter 3 mm (b.p.: 74°C/20 mbar).

The yield of 2-propanone O-(2-hydroxypropyl)oxime was 84%, with a purity of 99.8%. Isomeric 2-propanone O-(2-hydroxy-1-methyl-ethyl)oxime was not formed in this case.

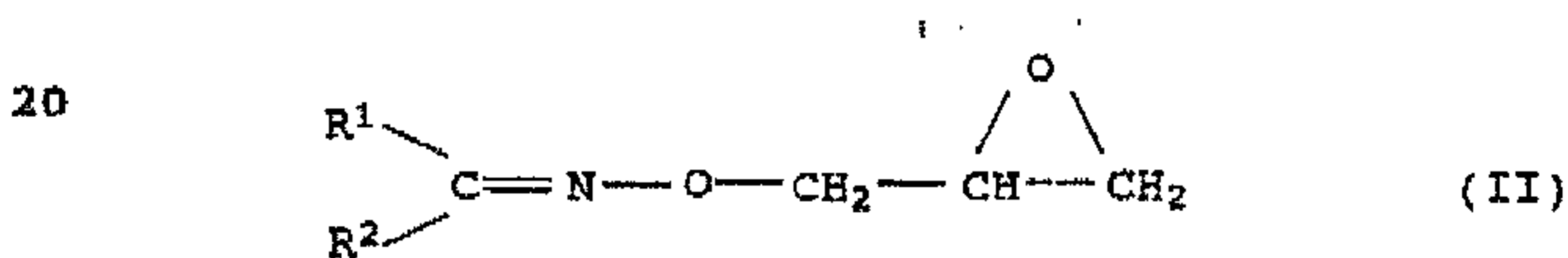
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We claim:

1. A process for preparing O-(2-hydroxyalkyl)oximes of the  
5 general formula I



where R<sup>1</sup> and R<sup>2</sup> are each alkyl groups having 1 to 10 carbon atoms  
or, together with the carbon atom which carries them, are bonded  
15 to give a 5- to 7-membered cycloalkyl radical, which comprises  
catalytically hydrogenating an O-(2,3-epoxyalkyl)oxime of the  
general formula II



25 using hydrogen.

2. A process as claimed in claim 1, wherein the starting  
material used is an O-(2,3-epoxyalkyl)oxime where R<sup>1</sup> and R<sup>2</sup>  
are methyl.
- 30
3. A process as claimed in claim 1 or 2, wherein the catalyst  
used is an element of groups 8 to 10 of the Periodic Table.
4. A process as claimed in claims 1 to 3, wherein the catalyst  
35 used is palladium, if desired on a support.

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