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(12) United States Patent
Kobayashi**(10) Patent No.: US 6,297,186 B1**
(45) Date of Patent: Oct. 2, 2001**(54) OSMIUM OXIDE COMPOSITION****(75) Inventor: Shu Kobayashi**, 6-6-702, Sarugakucho 1-chome, Chiyoda-ku, Tokyo 101-0064 (JP)**(73) Assignees: Shu Kobayashi**, Tokyo; **Wako Pure Chemical Industries, Ltd.**, Osaka, both of (JP)**(*) Notice:** Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 0 days.**(21) Appl. No.: 09/258,268****(22) Filed: Feb. 26, 1999****(30) Foreign Application Priority Data**

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(51) Int. Cl.⁷ **B01J 31/00; B01J 23/00; C08K 3/18; B32B 27/00; A61K 51/00****(52) U.S. Cl.** **502/159; 502/325; 502/402; 502/406; 502/507; 524/431; 428/328; 428/319.3; 428/319.7; 428/319.9; 428/357; 428/402.2; 428/402.24; 428/403; 428/407; 424/1.29; 424/489; 987/18; 252/514****(58) Field of Search** **502/159, 325, 502/402, 406, 507; 524/431; 428/328, 403, 407, 357, 402.2, 319.3, 319.7, 319.9, 402.24; 424/1.29, 489; 987/18; 252/514****(56) References Cited**

U.S. PATENT DOCUMENTS

Re. 29,419 * 9/1977 DeCraene 204/290 F
 Re. 35,692 * 12/1997 Debe 427/154
 3,947,277 * 3/1976 Carnahan et al. 106/26
 4,236,023 * 11/1980 Kesling, Jr. et al. 560/207
 4,824,969 * 4/1989 Austin et al. 549/230
 4,918,238 * 4/1990 Costantini et al. 568/342
 5,260,461 11/1993 Hartung et al. 549/447
 5,302,257 * 4/1994 Gao et al. 204/86
 5,314,727 * 5/1994 McCormick et al. 427/584
 5,380,179 * 1/1995 Nishimura et al. 419/36
 5,391,533 * 2/1995 Peterson et al. 502/262
 5,419,817 * 5/1995 Gao et al. 204/86
 5,707,738 * 1/1998 Hou 428/402
 5,712,072 * 1/1998 Inaba et al. 430/110
 5,853,675 * 12/1998 Howorth 422/179
 5,882,723 * 3/1999 Tsou 427/125
 6,040,077 * 3/2000 Debe et al. 429/40

FOREIGN PATENT DOCUMENTS

2835 943 3/1980 (DE) .
 3-081337 5/1991 (JP) .
 4-505884 10/1992 (JP) .
 WO 91/00143 1/1991 (WO) .

OTHER PUBLICATIONS

Japanese Patent Abstract, Publication No. 03081337, Publication Date May 4, 1991.
 Chem. Rev., 1994, "Catalytic Asymmetric Dihydroxylation", pp. 2483-2547.

Communications, 1989, "Catalytic Hydroxylation of Olefins by Polymer-Bound Osmium Tetroxide", pp. 45-47.

J. Org. Chem., 1998, "Microencapsulated Osmium Tetraoxide. A New Recoverable and Reusable Polymer-Supported Osmium Catalyst for Dihydroxylation of Olefins", pp. 6094-6095.

Tetrahedron Asymmetry, vol. 6, No. 11, "Polymeric Cinchona Alkaloids for the . . .", pp. 2687-2694.

Tetrahedron Asymmetry, "Heterogeneous Catalytic Asymmetric Dihydroxylation of Olefins: a New Polymeric Support and a process Improvement", pp. 11321-11328.

Wolfgang A Herrmann et al; Journal of Molecular Catalysis A: Chemical 120 pp. 197-205, Oct. 1996.

Roy A. Johnson et al; Catalytic Asymmetric synthesis, VHC Publisher, New York, pp. 227-272, New York, 1993.

Carsten Bolm et al; Eur. J. Org. Chem., pp. 21-27, Aug. 1997.

Cainelli et al, Catalytic Hydroxylation of Olefins by Polymer-Bound Osmium Tetroxide, SYNTHESIS 1; No. 1, 1989, pp. 45-47. No month.

Choong et al, Polymeric Cinchona Alkaloids for the Heterogeneous Catalytic Asymmetric Dihydroxylation of Olefins: The Influence of the Polymer Backbone Polarity on the Compatibility between Polymer Support and Reaction Medium; Tetrahedron: Asymmetry: vol. 6, No. 11, pp. 2687-2694, 1995. No month.

Pini et al., Heterogeneous Catalytic Asymmetric Dihydroxylation of Olefins: a new Polymeric Support and a Process Improvement; Tetrahedron; vol. 50, No. 38, 1994, pp.11321-11328. No month.

Kolb et al., Catalytic Asymmetric Dihydroxylation; Chemical Rev.1994; vol. 94, No. 8; pp. 2483-2547, Jul. 1994.

Nagayama et al., Microencapsulated Osmium Tetraoxide. A New Recoverable and Reusable Polymer-Supported Osmium Catalyst for Dihydroxylation of Olefins; J. Org. Chem., 1998, vol. 63, pp. 1998.

* cited by examiner

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The present invention provides an osmium oxide composition comprising an osmium oxide microencapsulated in an aromatic polyolefin (hereinafter abbreviated as MCOsO_x), a method for preparation of MCOsO_x, which comprises allowing an osmium oxide to contact with an aromatic polyolefin in an organic solvent, and precipitating MCOsO_x, an oxidizing agent comprising MCOsO_x, a method for preparing a chiral diol compound, which comprises reacting MCOsO_x, a chiral ligand and an olefin compound with each other, and a method for preparing a chiral diol compound, which comprises oxidizing an olefin compound with MCOsO_x wherein a chiral ligand further coordinates to an osmium oxide.

9 Claims, No Drawings