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[54] **PROCESS FOR SYNTHESIZING O-SUBSTITUTED OXIME COMPOUNDS AND CONVERSION TO THE CORRESPONDING O-SUBSTITUTED HYDROXYLAMINE**

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[58] **Field of Search** 562/512, 560, 405, 430, 562/440, 507, 556; 564/301, 256; 558/2, 3, 7

[56] **References Cited****U.S. PATENT DOCUMENTS**

| | | | |
|-----------|---------|-----------------|---------|
| 2,470,083 | 5/1949 | Hartung | 562/560 |
| 3,104,258 | 9/1963 | Ferris | |
| 3,207,787 | 9/1965 | Levy | 564/301 |
| 3,247,243 | 4/1966 | Villani | 564/301 |
| 3,839,449 | 10/1974 | Herold | |
| 4,038,317 | 7/1977 | Wermuth et al. | |
| 4,052,194 | 10/1977 | Wilcox | 562/560 |
| 4,425,360 | 1/1984 | Wolff | 562/560 |
| 4,584,014 | 4/1986 | Patterson | 562/560 |
| 4,739,118 | 4/1988 | Elbe | |
| 4,948,916 | 8/1990 | Uohama et al. | |
| 4,959,495 | 9/1990 | Currin | 562/560 |
| 4,973,753 | 11/1990 | Lantzsch et al. | |
| 4,981,996 | 1/1991 | Wyss et al. | 564/300 |
| 5,075,504 | 12/1991 | Schneider | 564/301 |
| 5,095,149 | 3/1992 | Tani | 562/560 |

FOREIGN PATENT DOCUMENTS

| | | | |
|-----------|---------|--------------------|---------|
| 1110642 | 10/1981 | Canada | |
| 1259629 | 9/1989 | Canada | |
| 0023560 | 2/1981 | European Pat. Off. | |
| 0158159 | 10/1985 | European Pat. Off. | |
| 259850 | 3/1988 | European Pat. Off. | 564/301 |
| 0121701 | 8/1988 | European Pat. Off. | |
| 62-215537 | 9/1987 | Japan | 562/560 |

347330 of 1972 U.S.S.R.

WO89/11473 11/1989 WIPO

OTHER PUBLICATIONS

Ault, "Techniques and Experiments for Organic Chemistry", 4th Ed., pp. 302-307 (1983).

"The Preparation of Aminoxyacetic Acid", Chemical World, vol. 9, No. 30, pp. 397-399, 1989. Dept. of Chemistry, Lanzhou University (Translation only).

"Carboxymethoxyamine Hemihydrochloride", Organic Syntheses Collective, vol. 3.

Dunstan and Goulding: "The Action of Alkyl"; J. Chem. Soc., 23, 628, 1901.

"Carboxymethoxyamine", J. Am. Chem. Soc., vol. LVIII, 58 2020 (1936).

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[57] **ABSTRACT**

A process for synthesizing O-substituted oxime compounds, which includes (a) reacting an alkali metal or alkaline earth metal hydroxide compound and a solution of an oxime compound to form a first mixture including the alkali metal or alkaline earth metal salt of the oxime compound and water; and (b) adding an organohalide compound while stirring to the first mixture to form a second mixture including an O-substituted oxime compound, the alkali metal or alkaline earth metal salt of the oxime compound, water, unreacted organohalide compound and excess oxime compound. The second mixture is heated in the presence of an amount of water sufficient to react hydroxide with the unreacted organohalide compound to obtain an O-substituted oxime compound substantially free of unreacted organohalide compound.

In a further embodiment, the O-substituted oxime compound can be hydrolyzed to the corresponding O-substituted hydroxylamine. The process is especially useful in the conversion of O-substituted oxime compounds to the corresponding aminoxy-compound.

14 Claims, No Drawings