SYNTHESIS OF DIMETHYLACETAL OF 5-METHOXY-TETRAHYDROFURAN-3-CARBOXALDEHYDE

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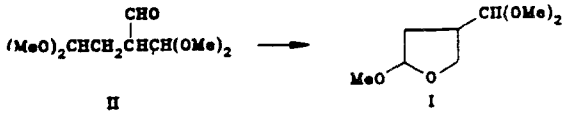
Previously [1], we obtained 5-methoxytetrahydrofuran-3-carboxyaldehydes by the acid-catalyzed reduction of 3-hydroxymethyl-2,5-dimethoxytetrahydrofurans.

We have now developed a synthesis of the dimethylacetal of 5-methoxytetrahydrofuran-3-carboxyaldehyde (I) from 2-dimethoxymethyl-4,4-dimethoxybutanal (II) by hydrogenation over Raney Ni (methanol, 80°C).



Aldehyde (II) was synthesized for the first time by the hydroformylation of 1,1,4,4-tetramethoxybut-2-ene over $RhHCO(PPh_3)_3$ in toluene at 90°C.

Dimethylacetal of 5-Methoxytetrahydrofuran-3-carboxaldehyde (I). Bp 86-88°C (5 mm Hg), n_D^{20} 1.4308, 73% yield. PMR spectrum ($CDCl_3$): 1.90 (2H, m, 4-H), 2.66 (1H, m, 3-H), 3.35 (9H, set of singlets, 3-OCH_3), 3.85 (2H, m, 2-H), 4.21 (0.6H, d, J = 8.7 Hz, 3-CH-trans), 4.38 (0.4H, d, J = 8.9 Hz, 3-CH-cis), 5.00 ppm (1H, m, 5-H). Ratio of cis:trans isomers = 40:60.

2-Dimethoxymethyl-4,4-dimethoxybutanal (II). Bp 107-108°C (3 mm Hg); n_D^{20} 1.4391, 75% yield. PMR spectrum ($CDCl_3$): 1.95 (2H, m, 3-H), 2.80 (1H, m, 2-H), 3.30 (6H, s, 4-OCH_3), 3.40 (6H, s, 2'-OCH_3), 4.40 (1H, t, J = 5.8 Hz, 4-H), 4.50 (1H, d, J = 5.8 Hz, 1-H), 9.70 ppm (1H, s, CHO).

LITERATURE CITED

1. A. V. Khandin, M. M. Vartanyan, and L. Yu. Brezhnev, *Izv. Akad. Nauk SSSR, Ser. Khim.*, No. 10, 2393 (1989).

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