SYNTHESIS OF α-TETRAHYDROFURYLACETALDEHYDE

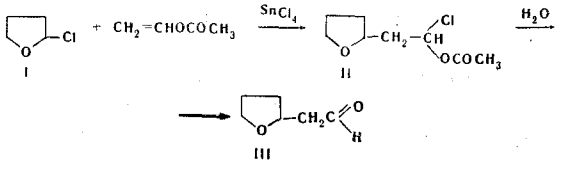
Yu. I. Tarnopol'skii, M. M. Tarnopol'skaya, and L. I. Kuzubova

Khimiya Geterotsiklicheskikh Soedinenii, Vol. 4, No. 2, p. 359, 1968

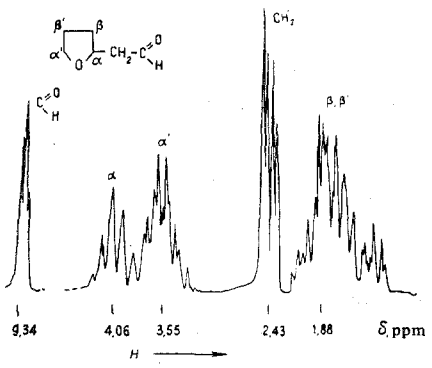
UDC 577.722.3.07

The synthesis of the previously unreported α-tetrahydrofurylacetaldehyde has been effected.

Aliphatic α-halo ethers add to vinyl acetate [1, 2]. This reaction is of great interest in the heterocyclic series. In the present work, we have synthesized the previously unreported α-tetrahydrofurylacetaldehyde by the following route:



The structure of the aldehyde was confirmed by its NMR spectrum (see figure). The reaction of cyclic α-halo ethers with other enol acetates is being studied.



NMR spectrum of α-tetrahydrofurylacetaldehyde.

2-(2'-Acetoxy-2'-chloroethyl)tetrahydrofuran (II). A mixture of 0.1 mole of 2-chlorotetrahydrofuran (I) [3], 30 ml of carbon tetrachloride, 0.12 mole of vinyl acetate, and 3 ml of a 10% solution of stannic chloride in carbon tetrachloride was kept for a day at 20° C and then 0.6 ml of pyridine was added; the mixture was filtered, the solvent was evaporated off, and the residue was distilled in vacuum. Yield 38%. Bp 70–71° C (1 mm); d_4^{20} 1.1722; n_D^{20} 1.4560. Found, %: Cl 18.35; MR_D 44.63. Calculated for $C_8H_{13}ClO_3$, %: Cl 18.45; MR_D 45.31.

α-Tetrahydrofurylacetaldehyde (III). A mixture of 0.38 mole of compound II and 100 ml of saturated $NaHCO_3$ was stirred with the temperature being gradually raised from room temperature to 45° C until the evolution of CO_2 ceased. As the reaction proceeded, solid $NaHCO_3$ was added. The product was extracted with ether and distilled in vacuum. Yield 29%. Bp 76° C (20 mm); d_4^{20} 1.0403; n_D^{20} 1.4420. Found, %: C 62.93; H 9.08; MR_D 29.03. Calculated for $C_6H_{10}O_2$, %: C 63.12; H 8.74; MR_D 29.60. **2,4-Dinitrophenylhydrazone,** mp 156° C (from ethanol). Found, %: N 19.16. Calculated for $C_{12}H_{14}N_4O_5$, %: N 19.04.

The NMR spectrum was recorded by M. L. Afanas'ev on a JNM 3H60 instrument (60 MHz).

REFERENCES

1. A. Rieche, H. Gross and E. Höft, J. pr. Chem., 28, 172, 1965.

2. S. A. Vartanyan, Sh. A. Gevorkyan, and F. V. Dangyan, Izv. AN ArmSSR, 18, 415, 1965.

3. M. Kratochvil and I. Hort, Coll., 27, 52, 1962.

24 October 1966

Siberian Technological Institute, Krasnoyarsk