The application of NMR spectroscopy for identification of the structures of 6-methyl-2-nitro-3-(trichloromethyl)-2,3-dihydro-4*H*-furo[3,2-c]pyran-4-one and its dehydrochlorination product

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1-Bromo-1-nitro-3,3,3-trichloropropene reacts with CH-acids via the formation of either dihydrofurans [1] or cyclopropanes [2]. The reaction with the heterocyclic CH-acid – 4-hydroxy-6-methylpyran-2-one – yields two types of products, depending on the reaction time. Investigation of the first product (shorter reaction time) by NMR methods allowed the assignment of a dihydrofuran structure characterized by doublets of the methine protons (4.95 and 6.55 ppm). The vicinal *J*-coupling constant (1.2 Hz) indicates their *trans* configuration.

The unexpected structure of the second compound (longer reaction time) was determined using one-dimensional (^{1}H; ^{13}C) and two-dimensional (HMQC, HMBC, COSY) NMR spectroscopy. It was found that a singlet at 6.89 ppm belongs to the proton of the nitromethine fragment, while the absence of the second dihydrofuran proton signal can be explained by the formation of a dichloromethylene derivative.

An interesting and important feature in the NMR spectra of both compounds is the correlation of the methyl protons with the singlet of the pyran-2-one ring in the ^{1}H-^{1}H COSY, caused by long-range *J*-coupling ($^{4}J_{HH}$).

The proposed structures were confirmed by single-crystal X-ray diffraction analysis performed for both compounds.

[1] S. V. Makarenko, E. V. Stukan', E. V. Trukhin, V. M. Berestovitskaya, Russ. J. Gen. Chem. 85 (2015) 2291;
[2] I. A. Pilipenko, M. V. Grigoriev, O. Y. Ozerova, I. A. Litvinov, D. V. Spiridonova, A. V. Vasilyev, S. V. Makarenko, Beilstein J. Org. Chem. 22 (2026) 123;