Dispiro [5.1.5.1.] tetradecane-7,14-dione (I). Its synthesis and hydrogen acceptor properties

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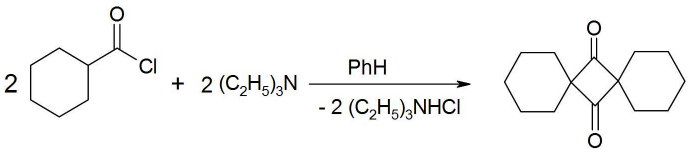
Goals and ways

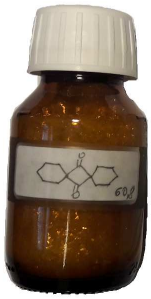
The goal was to synthesize dispiro[5.1.5.1]tetradecan-7,14-dione (I) from cyclohexanecarbonyl chloride and then pure (**I**) used as hydrogen acceptor in the transfer hydrogenation with 2-propanol as hydrogen donor in the presence of magnesium oxide as a catalyst.

Synthesis

Dispiro[5.1.5.1]tetradecan-7,14-dione (I) was prepared according to the procedure described in Org. Synth. 47 (1967). Its synthesis is based on the reaction of cyclohexanecarbonyl chloride with triethylamine in nitrogen atmosphere which proceeds according to the following equation:



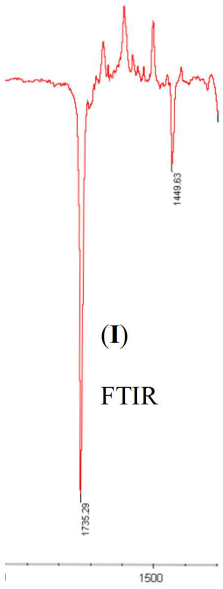
Crystals of (**I**)



The crude product was crystallized from n-hexane-ethanol mixture leaving pure (**I**) as colorless lamellas with m.p. 167-8°C, 165-6°C (lit). Yield 54.0%, purity 99.6% (GC).

Tests

The reaction was performed in liquid-phase at 82°C (reflux) with a molar ratio of 2-propanol to (**I**) equal to 64. It was expected that under such mild reaction conditions (**I**) would be reduced to the appropriate ketoalcohol and/or diol. It was shown that after 30 minutes of reaction (**I**) is transformed quantitatively into product (**II**) which does not undergo further transformations. Pure (**II**), colorless cubes m.p. 55-57°C, was isolated from a post-reaction mixture. Its structure was confirmed by analysis of ^{1}HNMR, ^{13}CNMR, IR and GC-MS spectra.

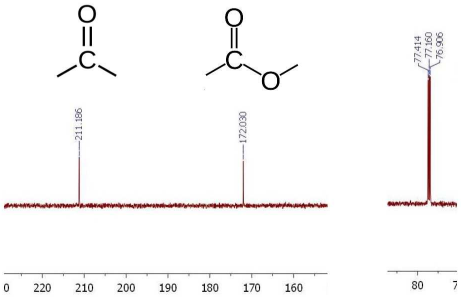


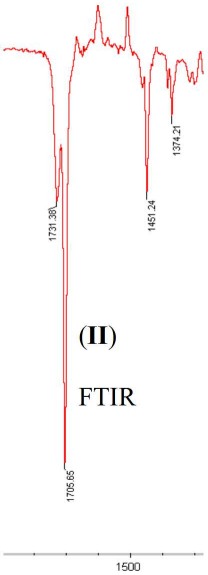
(**I**)

FTIR

^{13}C NMR

(**II**)

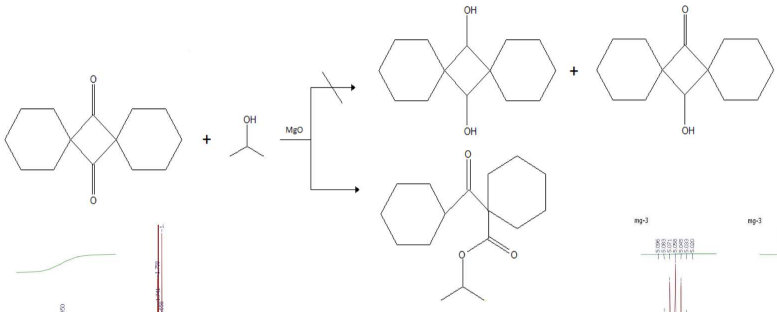




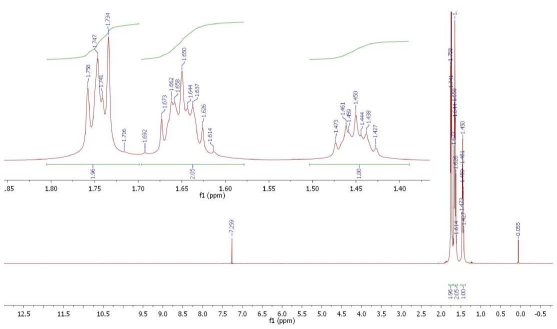
(**II**)

FTIR

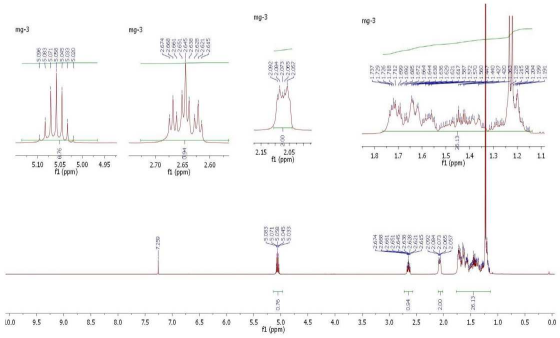
It was shown that in the presence of MgO 2-propanol reacts with (**I**) with the formation of isopropyl 1-(cyclohexylcarbonyl)-cyclohexanecarboxylate (**II**) according to the following equation:



^{1}H NMR (**I**)



^{1}H NMR (**II**)



[Projekt został zrealizowany w ramach Wolontariatu Naukowego na Wydziale Chemicznym PW w 2017 roku]