

Synthesis of maleic and fumaric acids from furfural in the presence of betaine hydrochloride and hydrogen peroxide

N. Araji,^a D. D. Madjinza,^a G. Chatel,^{a*} A. Moores,^b F. Jérôme^a and K. De Oliveira Vigier^{a*}

^a IC2MP UMR CNRS 7285, Université de Poitiers, ENSIP, B1, 1 rue Marcel Doré TSA 41105, 86073 Poitiers Cedex 9, France, E-mail: karine.vigier@univ-poitiers.fr and gregory.chatel@univ-poitiers.fr

^b Centre for Green Chemistry and Catalysis, Department of Chemistry, McGill University, 801 Sherbrooke St. W., Montreal, Canada.

General procedure for the oxidation of furfural to maleic and fumaric acids

Typically 0.5 g of furfural was added in a flask, in the presence of 5 g of water and 10 eq. of H₂O₂ (30%) and 2.5 eq. of BHC. The reaction was heated up to 90 °C. After the reaction, the medium was collected for analysis.

Analysis of furfural, maleic acid (MA), fumaric acid (FA) and 2-furanone

Furfural was analyzed using a GC equipment from BRUKER, equipped with a FID detector and a split/splitless injector. The column used was a CPWAX 52, 50 m. The calibration was performed by internal standard using DMSO.

MA, FA and 2-furanone were quantified by external calibration at 25 °C using a HPLC equipped with a ICE-Coragel 107H, a Varian Prostar UV-VIS detector, Varian Prostar pumps (model 210) and a mixture water/H₂SO₄ (90:10) as mobile phase (0.6 mL.min⁻¹). The wave length was 210 nm.

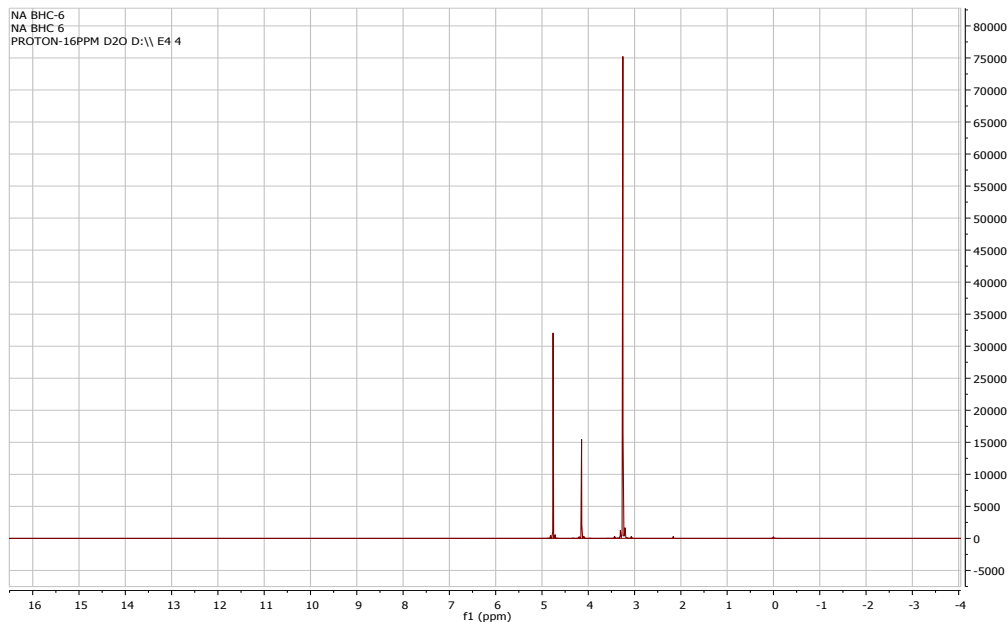
Recycling experiment

The reaction was carried out as mentioned before. After 30 min of reaction, the reaction medium was cooled down to room temperature. Acetone with a high purity was added to precipitate BHC. By filtration BHC was recovered and dried in an oven at 60 °C and was then used for another cycle.

Analysis of BHC after recycling

The BHC recovered after the 6th cycle was analyzed by ¹H NMR and it was shown that the NMR spectra was the same that the NMR spectra of commercial BHC.

a) BHC after recycling



b) Commercial BHC

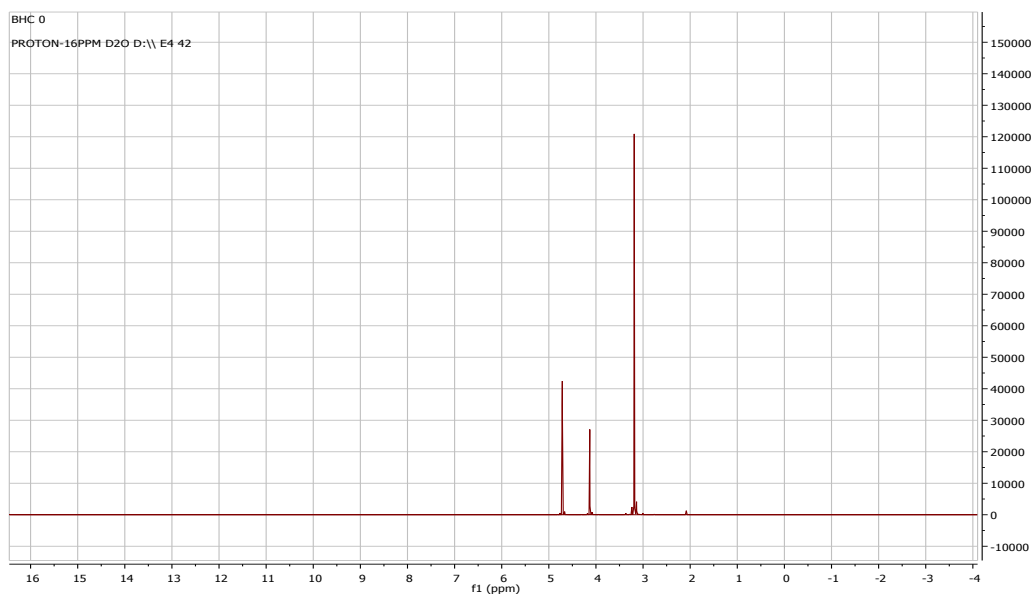


Figure S1: a) ¹H NMR spectra of BHC recovered after the 5th recycling ¹H NMR are recorded on a 400 MHz Bruker Advance DPX spectrometer using D₂O as solvent. Comparison with b) commercial BHC.