Reference : Experimental Organic Chemistry
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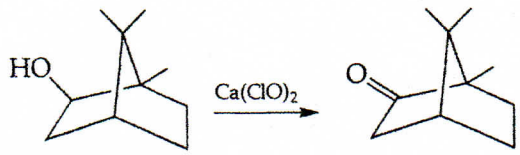
Safety First

- Calcium hypochlorite is an oxidizer.
- Acetic acid is corrosive and flammable. Acetonitrile is flammable and toxic.
- Camphor and borneol are flammable solids. Borneol is toxic.
- *tert*-Butyl methyl ether is flammable.
- $CDCl_3$ is a possible carcinogen. Handle it in a well-ventilated place.

Cleaning Up

- Dispose of the aqueous layers in the container labeled "Oxidation of Isoborneol Liquid Waste."
- Dispose of magnesium sulfate, Pasteur pipets, filter paper, and capillary tubes in the container labeled "Oxidation-Reduction Solid Waste."
- At the end of the lab period the instructor will empty the contents of the rota-vap traps into the container "Organic Solvents Waste."
- Dispose of $CDCl_3$ in the container labeled "$CDCl_3$-Waste."
- Turn in your products in labeled screw-cap vials.

E21.6 OXIDATION OF ISOBORNEOL



isoborneol camphor

In a 25-mL Erlenmeyer flask equipped with a stir bar, place 0.3 g of isoborneol and 2.0 mL of a mixture of acetonitrile and acetic acid (3 : 2). Secure the flask to a ringstand and place it in an ice-water bath on a magnetic stirrer. Start the stirring and add dropwise, with the help of a Pasteur pipet, 3.0 mL of a solution of calcium hypochlorite in water (5% w/v). Stir the mixture for about 30 minutes.

In the meantime inject standards of isoborneol and camphor in a GC with a polar column such as Carbowax 20 M (135°C).

Remove a small aliquot (a few drops) and place it in a small test tube with *tert*-butyl methyl ether (BME) (0.5 mL) and water (0.5 mL). Shake well, separate the layers with a Pasteur pipet (section 5.4), and transfer the organic layer to a scintillation vial. Evaporate the solvent by blowing a gentle stream of nitrogen until a few drops remain. Perform this operation in the fume hood. Inject the sample in a gas chromatograph. Compare the GC trace from the reaction mixture with those from the standards. If the reaction did not reach completion, and a peak for isoborneol is observed, continue the stirring of the reaction mixture for another 30 minutes and repeat the analysis at the end of this period.

When the reaction is complete, transfer the reaction mixture to a screw-cap test tube, and add 3.5 mL of water and 3.5 mL of BME; cap, shake, and vent. Separate the layers with a Pasteur pipet. Save the organic layer and extract the aqueous layer twice with a 2.5 mL portion (each time) of BME. Wash the combined BME layer with 3 mL of 3% sodium hydroxide aqueous solution, and finally with 3 mL of saturated sodium chloride solution. Dry the organic layer over anhydrous magnesium sulfate. Remove the drying agent by gravity filtration, collecting the liquid in a pre-tared round-bottom flask. Evaporate the solvent in a rota-vap. Weigh the product and calculate its percentage yield. Determine its melting point.

Obtain the IR of the product and the starting material using nujol (section 31.5). Obtain the ^{1}H-NMR of the product and the starting material using $CDCl_3$ as a solvent (section 33.20; adapted from Refs. 2 and 3).