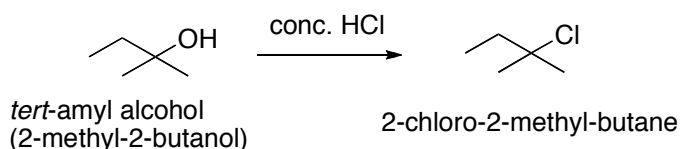


Lab 3: 2-Chloro-2-Methylbutane And Lucas Test

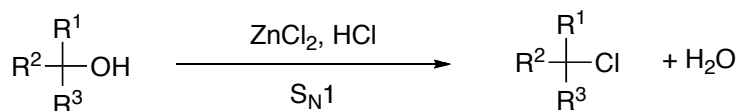


WARNING: wear gloves because of the strongly acidic solutions.

I. Synthesis of 2-Chloro-2-Methylbutane. Add 8 mL of *tert*-amyl alcohol and 20 mL of concentrated (~12 M) HCl to the separatory funnel. Swirl the contents gently without the stopper for about 1 min. Invert and vent to let the pressure equalize before shaking again. Repeat the shaking and venting for several minutes. Allow the mixture to separate into two distinct layers. (Which one is the organic layer?) The organic layer is washed with 10 mL of saturated aqueous NaCl. ["Wash" implies that the aqueous layer is removed before adding the next solution to the organic layer! Even though it is not written, the draining step still must be done.] Add a cold saturated aqueous solution of NaHCO₃ (10 mL) and swirl gently **without the stopper**. Once the effervescence ceases, stopper the separatory funnel, invert, vent. Wash with 10 mL of water, then with 10 mL of saturated aqueous NaCl. Dry the organic layer over anhydrous sodium or magnesium sulfate. If there is any visible drop of aqueous layer, this must be removed before the drying step (either in the separatory funnel, or with a pipet). Transfer to a 25 mL RBF and perform simple distillation (power controller on about 6, tare the receiver, cool the receiver in ice). The product is collected in the range of 82-85°C. Determine the percent yield. Purified 2-chloro-2-methylbutane will be turned in in an appropriately labeled test tube, corked and para-filmed.

II. Lucas Test. You will run the test in 4 test tubes (3 known alcohols (1°, 2°, 3°), and one unknown alcohol you will need to classify). Use 5-10 drops of compound to be tested. Fill about 2/3 of a long pasteur pipette with the Lucas test solution (approx. 1.5 mL) and add to the test tubes.

The Lucas test proceeds via



Notes on the Lucas Test: **the reagent is made by dissolving 16 g of anhydrous ZnCl₂ in 10 mL of concentrated (12N) hydrochloric acid and cooling to avoid HCl loss (you will not need to prepare the reagent).**

The Lucas test reagent is highly acidic: WEAR GLOVES. If the reagent comes into contact with the skin, wash immediately and thoroughly with water, and 5% aqueous bicarbonate.

Tertiary alcohols form an emulsion that appears as two layers (due to the water-insoluble alkyl halide) almost immediately. Secondary alcohols form this emulsion after several minutes, while primary alcohols react after a very long time (if at all). Some secondary alcohols (e.g. isopropyl) may not *visually* form the layers because of the low-boiling alkyl halide, which may evaporate.

Positive Test (Alcohols (Secondary and Tertiary):

Appearance of a cloudy second layer or emulsion; 3° alcohols: immediate to ~2 minutes; 2° alcohols: 3 - 10 minutes; 1° alcohols: no reaction (or very slow > 10 min)

Complications

The test applies *only to those alcohols soluble in the reagent* (monofunctional alcohols

lower than hexyl and some polyfunctional alcohols).