SYNTHESIS OF AN ORGANIC COMPOUND

VOL 5

SUMMARY:

The synthesis of 2-butanone, a ketone, from 2-butanol, an alcohol, is shown as an example of a common type of organic synthesis. Three basic steps are developed: synthesis, purification, identification. In the synthesis, 2-butanol is oxidized by sodium dichromate and sulfuric acid to yield 2-butanone. Purification is accomplished by solvent extraction with ether followed by distillation of the 2-butanone. The identity of the product is established by infrared absorption spectroscopy and then more clearly by nuclear magnetic resonance spectroscopy.

PURPOSE:

To give students an opportunity to observe an outstanding organic chemist perform the synthesis of a simple compound and to indicate the general steps which are important in any synthesis.

OUTLINE:

1. *Oxidation of 2-butanol to 2-butanone using acidified sodium dichromate.*

A. The balanced equation is obtained by writing the half-reaction for the oxidation of 2-butanol:

$$CH_3CHOHCH_2CH_3 \rightarrow CH_3COCH_2CH_3 + 2H^+ + 2e^-.$$

The half-reaction for the reduction of dichromate is written $Cr_2O_7{}^{2-} + 14H^+ + 6e^- \rightarrow 2Cr^{3+} + 7H_2O$. Inspection of these half-reactions shows that multiplying the butanol half-reaction by 3 and adding the half-reactions gives the overall equation:

$$3CH_3CHOHCH_2CH_3 + Cr_2O_7{}^{2-} + 8H^+ \rightarrow 3CH_3COCH_2CH_3 + 2Cr^{3+} + 7H_2O$$

B. Calculating from the equation, the proper amounts of the reagents are weighed out. With ½ mole or 37 grams of 2-butanol as the starting material, ⅓ as many moles of chromate are needed.

$$\frac{1 \text{ mole butanol}}{2 \quad \text{(used)}} \times \frac{1 \text{ mole } Na_2Cr_2O_7}{3 \text{ mole butanol}} \times \frac{298 \text{ g}}{1 \text{ mole } Na_2Cr_2O_7 \bullet 2H_2O} = 50 \text{ g}$$

For every 3 moles of 2-butanol, 8 H^+ ions must be supplied from the H_2SO_4. Remember that each H_2SO_4 supplies $2H^+$ ions.

$$\frac{1 \text{ mole butanol}}{2 \quad \text{(used)}} \times \frac{8 \text{ mole } H^+}{3 \text{ mole butanol}} \times \frac{1 \text{ mole } H_2SO_4}{2 \text{ mole } H^+} \times \frac{98 \text{ g}}{1 \text{ mole } H_2SO_4} = 65 \text{ g}$$

C. The various steps in the synthesis are carried out. First sulfuric acid is diluted by pouring it slowly into water. Sodium dichromate is dissolved in the acid solution forming the oxidizing solution. Ether is added to the 2-butanol to dilute the mixture which is then placed in the reaction flask. The oxidizing solution is slowly added to the ether solution with vigorous stirring. A water bath removes the heat of the reaction.

2. *Purification of the 2-butanone product.*

A. The ether layer, containing the product, is separated from the aqueous layer in a separatory funnel. The aqueous layer is extracted with ether to remove any traces of ketone. The ether wash is added to the original ether layer. This removal of ketone dissolved in the aqueous layer is necessary to ensure a good yield. Butanone-2 is appreciably soluble in water (37g/100g H_2O).

B. The ether layer is purified first by adding $NaHCO_3$ solution to neutralize any excess acid: $H^+ + HCO_3^- \rightarrow H_2O + CO_2(g)$. Repeated washings with distilled water remove inorganic material. Anhydrous Na_2SO_4 removes traces of water, leaving a clear, colorless solution. This drying agent is chosen because it is a salt that has a great affinity for water of crystallization. Solid $Na_2SO_4 \bullet 10\,H_2O$ is formed.

C. The ether layer is distilled to separate the ether from the 2-butanone. The remaining ketone is transferred to a small distilling apparatus. The purified ketone is then collected in a weighed flask and weighed.

D. The yield is calculated. The weight of the product, 30 g, compared to the theoretical yield of ½ mole, or 36 g, gives a percent yield of 30/36 x 100 or 83%.

3. *Identification of the product.*

A. The infrared spectrum is used for identification by taking the spectrum of the product and comparing it to the spectrum of a known sample of 2-butanone.

B. Nuclear magnetic resonance spectroscopy, NMR, is also used to characterize the compound. This technique involves the absorption of radiowaves by a sample placed in a strong magnetic field. The energies that the protons in the sample absorb are characteristic of their chemical environment—that is, the proximity in the molecule of other specific atoms or groups of atoms. Comparison of this NMR spectrum with spectra of protons in known environments in various molecules can clearly identify the product.

SUPPLEMENTARY MATERIAL:

2-butanone is also known as methyl ethyl ketone, or MEK, a common industrial solvent.

The weighing technique (to 0.1 gram) is typical of that for organic reactions. Often a large excess of one reagent (usually the least expensive one) is added to cause additional formation of product as predicted by Le Chatelier's Principle.

The experiment actually requires 3-4 hours, and is typical of experiments done in a beginning course in organic chemistry.

Students must be cautioned not to try this type of experiment until they have a full understanding of and availability to safety procedures.

20½ MINUTES IN COLOR

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