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PREPARATION OF ACETIC ESTERS OF SOME HIGHER ALCOHOLS

JOHN W. SWANSON AND JAMES COSS

The preparation of the lower esters of acetic acid using the acid and alcohol, has been utilized for a long time. It has also been known that the velocity of esterification is greatly increased by the addition of hydrogen ions. Esters have also been prepared by the passing of the vapors of acetic acid and alcohol over a heated catalyst such as Al_2O_3 , ThO_2 or TiO_2 . Several theories have been advanced for the mechanism of the reaction, especially catalysis, by the hydrogen ion in the liquid phase reactions.

In the present work several of the higher esters were prepared using acetic anhydride and the alcohol. The yields were noted and some ideas about the mechanism of the reactions and the function of the catalyst brought out.

The esters were prepared by using acetic anhydride obtained from the Carbon Carbide Co. and Merck C. P. grade alcohols. These were not purified further.

One-half mole each of the reacting substances was employed throughout the experimentation. The alcohol was placed in a 500cc. pyrex reaction flask along with the acid catalyst. A few pieces of clay plate or a little glass wool was added to prevent bumping. To this was attached a reflux condenser through which the anhydride was slowly added. After all the anhydride was added, the mixture was refluxed for one hour in an oil bath. Upon cooling to the room temperature, the ester was washed three times with cold saturated salt solution, NaHCO_3 being added to neutralize any residual acid. After thorough drying the ester was decanted or filtered off and distilled.

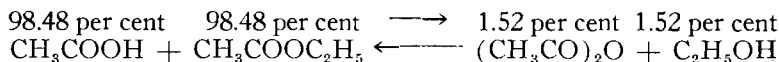
The catalyst used in the preparation of Allyl Acetate was 2cc. of concentrated H_2SO_4 and the yield was 90.9% of theoretical. The ester combined with CaCl_2 when it was added as a drying agent and anhydrous CuSO_4 decomposed part of the ester and gave it a yellow color. Anhydrous K_2CO_3 was found to be satisfactory.

It was found necessary to use 2cc. of H_3PO_4 as a catalyst for making Benzyl Acetate because HCl combined with benzyl alcohol to form benzyl chloride and H_2SO_4 makes a resin of the alcohol.

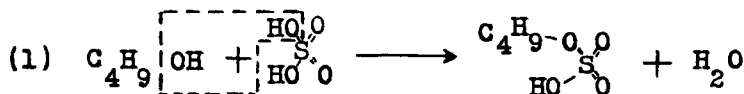
The yield was 86 per cent of the theoretical. The ester combined with CaO when it was added as a dehydrating agent and it finally set to a gel. Anhydrous CuSO₄ was a satisfactory drying agent if not allowed to remain too long in contact with the ester.

The catalyst used for both the Butyl Acetate and the Isoamyl acetate was 2cc. of concentrated H₂SO₄. If the Isoamyl ester was refluxed more than one hour, the reaction mixture became very dark on account of oxidation by H₂SO₄. The yield of Isoamyl acetate was 88.2 per cent and that of the Butyl ester 86.2 per cent of the theoretical. Anhydrous CuSO₄ was used as a dehydrating agent for the Butyl ester and CaCl₂ for the Isoamyl ester. The latter combined with CaCl₂ on prolonged standing.

An attempt was made to find the extent of the reverse reaction by refluxing for one hour one-half mole of each of ethyl acetate and glacial acetic acid and titrating the amount of acetic anhydride formed. It was found by this method that the reverse reaction takes place to the extent of about 1.5 to 2 per cent.

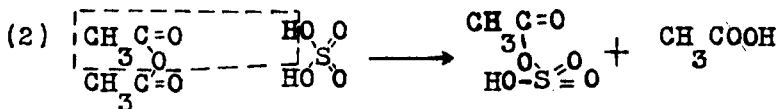


It has been a known and accepted fact for many years that alcohols react with sulfuric acid thus:



and that the Butyl sulphuric acid boiled with H₂O will in turn produce the alcohol and sulfuric acid again.

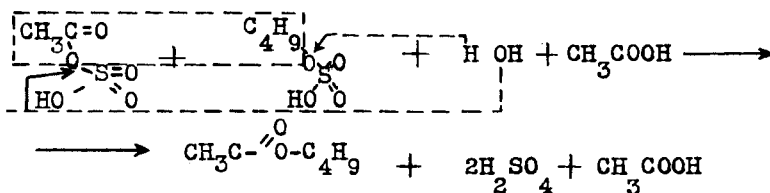
Now it is probable that a similar reaction takes place between H₂SO₄ and acetic anhydride.



This was tried out by using one quarter mole each of H₂SO₄ and acetic anhydride. The mixture was distilled and 66 per cent of the theoretical CH₃COOH distilled over with a boiling point of 116-117° C. Upon dilution of the syrupy residue in the distilling flask with water there was evidence that acetic acid was formed although this will be checked by further experimentation.

Putting these two reactions together we have the probable

mechanism of the reaction between the H_2SO_4 catalyst, the alcohol and the acetic anhydride.



This method of preparing esters should lend itself very well to the preparation of these esters in quantity for the laboratory since Acetic Anhydride is on the market at a relatively low price.

We wish to express our thanks to the Carboa Carbide Co. for the acetic anhydride used in these experiments.

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