Esterification: Isoamyl acetate

Macroscale

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 OH + OH \rightarrow OOH

Chemicals: Isoamylalkohol: Mr. Frenzel acetic acid:

Procedure:

In a 250-ml round-bottom flask place 8.8 g (100 mmole) isoamyl alcohol, 23 ml (24 g, 400 mmole) of glacial acetic acid und a few boiling chips. Add 2 ml of concentrated sulfuric acid, swirl to dissolve, fit the flask with a reflux condenser, and boil the mixture gently, using a heating bath, for two hours. Remove the bath and after some cooling add 50 g of ice; the mixture than has to be cooled to below 25 °C.

Isolation and purification:

Transfer the cooled mixture to a separatory funnel, add 50 ml of diethyl ether, using some of the ether to rinse the boiling flask. Thoroughly mix the contents of the separatory funnel, allow the layers to separate, and draw off the lower, aqueous, layer. Wash the ether layer with a 50 ml portion of cold water and then extract the ether layer with a solution of 3 g of sodium carbonate in 50 ml of water, using this solution in two 25-ml portions. Take care not to build up excessive pressure in the separatory funnel! Dry the ether extract over anhydrous magnesium sulfate, filter off the drying agent and distill off the ether in a rotary evaporator (normal pressure!). Destill the residue at normal pressure to isolate the isoamyl acetate (high bath temperature needed, ca. 170-180 °C).

Literature yield: ca. 8 to 9 g Literature boiling point: 142 °C.

Literature n_D: 1.4010