Ethanol *[64-17-51* **M 46.1, b 78.3O, d15 0.79360, d5 0.78506, n 1.36139, pKZ5 15.93.** Usual impurities of fermentation alcohol are fusel oils (mainly higher alcohols, especially pentanols), aldehydes, esters, ketones and water. With synthetic alcohol, likely impurities are water, aldehydes, aliphatic esters, acetone and diethyl ether. Traces of *benzene are present in ethanol that has been dehydrated by azeotropic distillation with *benzene. Anhydrous ethanol is very ***hygroscopic.*** Water (down to 0.05%) can be detected by formation of a voluminous ppte when aluminium ethoxide in *benzene is added to a test portion, Rectified spirit (95% ethanol) is converted to ***absolute*** (99.5%) ethanol by refluxing with freshly ignited CaO (250gL) for 6h, standing overnight and distilling with precautions to exclude moisture.

Numerous methods are available for further drying of ***absolute*** ethanol for making "Super dry ethanol". Lund and Bjermm *[Chem* Ber ***64*** 210 *1931]* used reaction with magnesium ethoxide, prepared by placing 5g of clean dry magnesium turnings and 0.5g of iodine (or a few drops of CCl4), to activate the Mg, in a 2L flask, followed by 50-75 mL of ***absolute*** ethanol, and warming the mixture until a vigorous reaction occurs. When this subsides, heating is continued until all the magnesium is converted to magnesium ethoxide. Up to 1L of ethanol is added and, after an hour's reflux, it is distd off. The water content should be below 0.05%. Walden, Ulich and Laun [Z ***Phys Chem*** **114** 275 *1925]* used amalgamated aluminium chips, prepared by degreasing aluminium chips (by washing with Et20 and drying in a vac to remove grease from machining the Al), treating with alkali until hydrogen was vigorously evolved, washing with H20 until the washings were weakly alkaline and then stirring with 1% HgC12 soln. After 2min, the chips were washed quickly with H20, then alcohol, then ether, and dried with filter paper. (The amalgam became warm.) These chips were added to the ethanol, which was then gently warmed for several hours until evolution of hydrogen ceased. The alcohol was distd and aspirated for some time with pure dry air. Smith ***[J Chem Soc*** 1288 **19271** reacted 1L of ***absolute*** ethanol in a 2L flask with 7g of clean dry sodium, and added 25g of pure ethyl succinate 27g of pure ethyl phthalate was an alternative), and refluxed the mixture for 2h in a system protected from moisture, and then distd the ethanol. A modification used 40g of ethyl formate, instead, so that sodium formate separated out and, during reflux, the excess of ethyl formate decomposed to CO and ethanol.

Drying agents suitable for use with ethanol include Linde type 4A molecular sieves, calcium metal, and CaH2. The calcium hydride (2g) was crushed to a powder and dissolved in lOOmL ***absolute*** ethanol by gently boiling. About 7OmL of the ethanol were distd off to remove any dissolved gases before the remainder was poured into 1L of ***ca*** 99.9% ethanol in a still, where it was boiled under reflux for 20h, while a slow stream of pure, dry hydrogen (better use nitrogen or Ar) was passed through. It was then distd [Ruber Z ***Elektrochem*** **29** 334 *1923].* If calcium was used for drying, about ten times the theoretical amount should be taken, and traces of ammonia (from some calcium nitride in the Ca metal) would be removed by passing dry air into the vapour during reflux.

Ethanol can be freed from traces of basic materials by distn from a little 2,4,6-trinitrobenzoic acid or sulfanilic acid. *Benzene can be removed by fractional distn after adding a little water (the *benzene/water/ethanol azeotrope distils at 64.90); the alcohol is then redried using one of the methods described above. Alternatively, careful fractional distn can separate *benzene as the *benzene/ethanol azeotrope **(b** 68.2O). Aldehydes can be removed from ethanol by digesting with 8-log of dissolved KOH and 5-log of aluminium or zinc per L, followed by distn. Another method is to heat under reflux with KOH (20g/L) and AgN03 (lOg/L) or to add 2.5-3g of lead acetate in 5mL of water to 1L of ethanol, followed (slowly and without stirring) by 5g of KOH in 25mL of ethanol: after lhr the flask is shaken thoroughly, then set aside overnight before filtering and distilling.

The residual water can be removed by standing the distillate over activated aluminium amalgam for 1 week, then filtering and distilling. Distn of ethanol from Raney nickel eliminates catalyst poisons. Other purification procedures include pre-treatment with conc H2SO4 (3mL/L) to eliminate amines, and with KMnO4 to oxidise aldehydes, followed by refluxing with KOH to resinify aldehydes, and distilling to remove traces of H3P04 and other acidic impurities after passage through silica gel, and drying over CaS04. Water can be removed by azeotropic distn with dichloromethane (azeotrope boils at 38.1O and contains 1.8% water) or 2,2,Ctrimethylpentane.

Rapid purification: Place degreased Mg turnings (grease from machining the turnings is removed by washing with dry EtOH then Et20, and drying in a vac) (5g) in a dry 2L round bottomed flask fitted with a reflux condenser (protect from air with a drying tube filled with $CaC1_2$ or KOH pellets) and flush with dry **N2.**Then add iodine crystals (0.5g) and gently warm the flask until iodine vapour is formed and coats the turnings. Cool, then add EtOH **(50mL)** and carefully heat to reflux until the iodine disappears. Cool again then add more EtOH (to 1L) and reflux under **N_2** for several hours. Distil and store over 3A molecular sieves (pre-heated at 300^{o} -350^{o} for several hours and cooled under dry N_2 or argon).