

10408 COMPETITIVE PROBLEMS IN THE DRUG INDUSTRY

6505-890-2010 (P. D. No. 3)

4.4.3 Alcohol content. Adjust the temperature of the sample to 25° C. and transfer exactly 100 cc to a distilling flask. Dilute with 150 cc purified water and distill, collecting about 90 cc distillate in a 100 cc volumetric flask immersed in an ice bath. Rinse the distillate into a separator, add 10 grams NaCl and 30 cc petroleum ether. Shake the mixture to extract volatile oils and CHCl₃. Draw off the water-alcohol layer into a distilling flask. Wash the petroleum ether solution in the separator with two 40 cc portions of saturated NaCl solution, adding the aqueous washings to the distilling flask. Distill, collecting about 90 cc distillate in a 100 cc volumetric flask immersed in an ice bath. Adjust the temperature of the distillate to 25° C. and dilute to the mark with purified water at the same temperature. Add about 5 Gm talc, mix, and filter. Determine the specific gravity of the filtrate at 25° C. in a pycnometer. Calculate percent required amount of alcohol.

4.4.4 Chloroform assay.

REAGENTS:

Cyclohexane - B.P. 80° - 81° C.

Chloroform - Suitable for Spectrophotometric use.

STANDARD CHLOROFORM SOLUTION:

Transfer exactly 5 ml of chloroform to a 250 ml volumetric flask containing about 150 ml of cyclohexane. Dilute to the mark with cyclohexane and mix well. Transfer exactly 20 ml of this solution to a 100 ml volumetric flask containing 50 ml cyclohexane. Dilute to the mark with cyclohexane and mix well (Chloroform concentration at this dilution is 0.40 ml/100 cc).

PROCEDURE: ALL TRANSFERS SHALL BE MADE AS RAPIDLY AS POSSIBLE.

Transfer about 20 ml of sample accurately measured to a suitable glass stoppered centrifuge tube (or other suitable vessel), add 4 glass beads and exactly 20 ml of cyclohexane. Stopper securely and shake vigorously for 5 minutes. Centrifuge at 3000 rpm for 5 minutes. Determine the absorbance of the cyclohexane solution in the sample and standard in 0.1 mm NaCl cells, using a suitable infrared spectrophotometer.

Calculate percent required amount of chloroform.