

## Chapter 8

### SUMMARY OF THE EXPERIMENTAL WORKS

#### 1. Spectrophotometric Determination of Uranium(VI) with Benzoylacetanilide (Section 5.1)

The yellow coloured uranium(VI)-benzoylacetanilide complex has been formed at pH 6.5 or above using pyridine to adjust the acidity. The complex has then been extracted into isobutyl methyl ketone. By measuring the absorbance of this extract at 390 nm against pure solvent, 1 to 40  $\mu\text{g}$  of uranium(VI) per ml of the organic solvent has been determined. The estimation has also been done in the presence of  $\text{Ca}^{2+}$ ,  $\text{Sr}^{2+}$ ,  $\text{Ba}^{2+}$ ,  $\text{Mg}^{2+}$ ,  $\text{Hg}^{2+}$ ,  $\text{Cu}^{2+}$ ,  $\text{Cd}^{2+}$ ,  $\text{Zn}^{2+}$ ,  $\text{Mn}^{2+}$ ,  $\text{Co}^{2+}$ ,  $\text{Ni}^{2+}$ ,  $\text{Pb}^{2+}$ ,  $\text{Bi}^{3+}$ ,  $\text{Fe}^{3+}$ ,  $\text{Al}^{3+}$ ,  $\text{Th}^{4+}$ ,  $\text{Zr}^{4+}$ ,  $\text{VO}^{2+}$ ,  $\text{CrO}_4^{2-}$  or  $\text{MoO}_4^{2-}$  in quantities up to five moles per mole of uranium. In the determination of uranium in such mixtures magnesium-EDTA has been used as the masking agent. The relative standard deviation is  $\pm 0.70\%$ , while the practical sensitivity is  $0.19 \mu\text{g}$  of uranium(VI) per  $\text{cm}^2$ .

#### 2. Gravimetric Determination of Beryllium with Benzoyl-m-nitroacetanilide (Section 6.1)

Small amounts of beryllium (2.0 to 3.0 mg) have been determined gravimetrically with benzoyl-m-nitroacetanilide. The yellow complex is precipitated from solutions having pH between 6.5 and 9.0 and weighed directly as  $\text{Be}(\text{C}_{15}\text{H}_{11}\text{O}_4\text{N}_2)_2$  containing 1.567% of the metal

after drying at  $120^{\circ}\text{C}$ . Using thioglycolic acid, magnesium-EDTA and /or  $\text{H}_2\text{O}_2$  as masking agents, separation of  $\text{Be}^{2+}$  from considerable quantities of  $\text{Cu}^{2+}$ ,  $\text{Pb}^{2+}$ ,  $\text{Co}^{2+}$ ,  $\text{Mn}^{2+}$ ,  $\text{Ni}^{2+}$ ,  $\text{Zn}^{2+}$ ,  $\text{Al}^{3+}$ ,  $\text{Cr}^{3+}$ ,  $\text{Fe}^{3+}$ ,  $\text{Ce}^{4+}$ ,  $\text{Th}^{4+}$ ,  $\text{TlO}^{2+}$ ,  $\text{VO}^{2+}$  or  $\text{UO}_2^{2+}$  has been effected. The standard deviation is only  $\pm 0.27\%$ . Using magnesium-EDTA as the masking agent for the various foreign ions present, this method has also been applied to the estimation of the beryllium content of beryl.

### 3. Spectrophotometric Determination of Beryllium with Benzoyl-m-nitroacetanilide (Section 6.2)

The complex formed by the reaction between beryllium and benzoyl-m-nitroacetanilide in the aqueous medium of pH 4.4 or above has been extracted into chloroform and the absorbance of the extract has been measured at 400 nm against a reagent blank. Thus, beryllium in quantities 100 to 600  $\mu\text{g}$  has been determined with a relative standard deviation of  $\pm 2.4\%$  provided the measurements are carried out within 30 minutes after the extraction. The determination of beryllium is possible in the presence of  $\text{Cu}^{2+}$ ,  $\text{Pb}^{2+}$ ,  $\text{Co}^{2+}$ ,  $\text{Ni}^{2+}$ ,  $\text{Zn}^{2+}$ ,  $\text{Al}^{3+}$ ,  $\text{Cr}^{3+}$  and  $\text{Fe}^{3+}$  when magnesium-EDTA is used as the masking agent.

### 4. Gravimetric Determination of Uranium(VI) with Benzoyl-m-nitroacetanilide (Section 6.3)

Uranium(VI) (25 to 45 mg) has been quantitatively precipitated with benzoyl-m-nitroacetanilide by adjusting the pH in three different ways. By controlling the pH to 5.8 or above with pyridine, a vermilion-red complex,  $\text{UO}_2(\text{C}_{15}\text{H}_{11}\text{O}_4\text{N}_2)_2 \cdot 2\text{H}_2\text{O}$  containing 27.30%

of the metal, is obtained. This complex is formed homogeneously in the presence of magnesium-EDTA or sulphosalicylic acid. But from solutions of pH 6.8 to 7.8 buffered with ammonia-ammonium acetate,  $\text{UO}_2(\text{C}_{15}\text{H}_{11}\text{O}_4\text{N}_2)_2$ ,  $\text{NH}_3$ ,  $\text{H}_2\text{O}$  is precipitated. In a third method, the latter complex is obtained homogeneously when the pH is raised with boiling urea solution. Using magnesium-EDTA, sulphosalicylic acid or iodide ion as masking agent separation of uranium(VI) from  $\text{Pb}^{2+}$ ,  $\text{Ni}^{2+}$ ,  $\text{Be}^{2+}$ ,  $\text{Al}^{3+}$ ,  $\text{Cr}^{3+}$ ,  $\text{Fe}^{3+}$ ,  $\text{Ce}^{3+}$ ,  $\text{Th}^{4+}$ ,  $\text{Zr}^{4+}$  or  $\text{VO}^{2+}$  is possible. The relative standard deviation is  $\pm 0.24\%$ .

5. Spectrophotometric Determination of Uranium(VI) with Benzoyl-m-nitroacetanilide (Section 6.4)

The complex formed by the reaction between uranium(VI) and benzoyl-m-nitroacetanilide in aqueous medium at pH 5.25 or above, buffered by pyridine solution, has been extracted into isobutyl methyl ketone. The absorbance of the extract is measured at 395 or 415 nm against a reagent blank within 2 hours. The relative standard deviation is  $\pm 0.21\%$ . Sulphosalicylic acid, triethanolamine, magnesium-EDTA and iodide do not interfere with the determination. Using magnesium-EDTA, as the masking agent the estimation is also possible in the presence of  $\text{Ni}^{2+}$ ,  $\text{Al}^{3+}$ ,  $\text{Cr}^{3+}$ ,  $\text{Fe}^{3+}$ ,  $\text{Ce}^{3+}$  and  $\text{Th}^{4+}$ .

6. Gravimetric Determination of Mercury(II) with Benzoyl-m-nitroacetanilide (Section 6.5)

Benzoyl-m-nitroacetanilide has been used to determine 12 to 32 mg of mercury(II) gravimetrically. The complex is precipitated quantitatively within the pH range, 2.5 to 9.0. Using

citrate, tartrate or fluoride as the masking agent, mercury(II) has been separated from  $\text{Cu}^{2+}$ ,  $\text{Pb}^{2+}$ ,  $\text{Co}^{2+}$ ,  $\text{Ni}^{2+}$ ,  $\text{Mn}^{2+}$ ,  $\text{Bi}^{3+}$ ,  $\text{Al}^{3+}$ ,  $\text{Fe}^{3+}$ ,  $\text{Cr}^{3+}$ ,  $\text{UO}_2^{2+}$  or  $\text{WO}_4^{2-}$ . No masking agent is required to effect separation from  $\text{Ag}^+$ ,  $\text{Tl}^+$  or  $\text{Cd}^{2+}$ . The precipitate is weighed after drying as  $\text{Hg}(\text{C}_{15}\text{H}_{11}\text{O}_4\text{N}_2)_2$  containing 26.19% of mercury as it is stable up to  $220^\circ\text{C}$ . The relative standard deviation is  $\pm 0.13\%$ .

7. Gravimetric Determination of Beryllium with Cyclopentanone-2-carboxyanilide (Section 7.1)

Beryllium (1.4 to 4.0 mg) has been estimated gravimetrically using cyclopentanone-2-carboxyanilide in the pH range, 5.9 to 8.1 within  $\pm 0.26\%$  relative standard deviation. The complex,  $\text{Be}(\text{C}_{12}\text{H}_{12}\text{NO}_2)_2$ , containing 2.181% of the metal is weighed directly after drying at  $120^\circ\text{C}$  as it is thermally stable up to  $250^\circ\text{C}$ . Using disodium-EDTA as the masking agent, the determination of beryllium is possible in the presence of at least 10 to 15 times its weight of  $\text{Cu}^{2+}$ ,  $\text{Pb}^{2+}$ ,  $\text{Co}^{2+}$ ,  $\text{Ni}^{2+}$ ,  $\text{Zn}^{2+}$ ,  $\text{Al}^{3+}$ ,  $\text{Cr}^{3+}$ ,  $\text{Fe}^{3+}$ ,  $\text{Ce}^{3+}$ ,  $\text{Th}^{4+}$ ,  $\text{TiO}^{2+}$ ,  $\text{VO}^{2+}$  or  $\text{UO}_2^{2+}$ . A simple and rapid procedure for the determination of beryllium in beryl has also been developed.

8. Titrimetric Determination of Beryllium through the Cyclopentanone-2-carboxyanilide complex (Section 7.2)

Beryllium has been precipitated with cyclopentanone-2-carboxyanilide in the pH range 5.2 to 8.1 buffered with ammonium acetate-ammonia. The complex, after washing, is dissolved in hydrochloric acid and treated with sufficient amounts of potassium

bromide and a standard potassium bromate solution. Potassium iodide was added and the iodine equivalent of the unreacted bromine is determined titrimetrically with a standard sodium thiosulphate solution. From the amount of thiosulphate equivalent of the bromine consumed by cyclopentanone-2-carboxyanilide, beryllium is calculated. Thus, 1 ml of 0.1 N thiosulphate (or bromine or bromate) is equivalent to 0.1126 mg of beryllium.

9. Spectrophotometric Determination of Beryllium with Cyclopentanone-2-carboxyanilide (Section 7.3.)

Beryllium (2.0 to 6.5  $\mu\text{g}$ ) has been extracted into isobutyl methyl ketone as its cyclopentanone-2-carboxyanilide chelate at pH 7.0 to 10.5 in the presence of magnesium-EDTA. The metal is determined by measuring absorption of this extract at 332 nm against the pure solvent. The relative standard deviation is  $\pm 1.08\%$ .

Under the conditions stated above, large quantities of  $\text{Ag}^+$ ,  $\text{Tl}^+$ ,  $\text{Cd}^{2+}$ ,  $\text{Cu}^{2+}$ ,  $\text{Co}^{2+}$ ,  $\text{Pb}^{2+}$ ,  $\text{Hg}^{2+}$ ,  $\text{Ni}^{2+}$ ,  $\text{Bi}^{3+}$ ,  $\text{Al}^{3+}$ ,  $\text{Ga}^{3+}$ ,  $\text{In}^{3+}$ ,  $\text{Cr}^{3+}$ ,  $\text{Fe}^{3+}$ ,  $\text{Ce}^{3+}$ ,  $\text{Th}^{4+}$ ,  $\text{Zr}^{4+}$ ,  $\text{TiO}^{2+}$ ,  $\text{VO}^{2+}$ ,  $\text{MoO}_2^{2+}$ ,  $\text{UO}_2^{2+}$  or  $\text{WO}_4^{2-}$  do not interfere with the estimation of beryllium. The beryllium contents of different samples of beryl have also been determined.

10. Gravimetric Determination of Uranium(VI) with Cyclopentanone-2-carboxyanilide (Section 7.4.)

Uranium(VI) (30 to 45 mg) has been precipitated as the brownish-red complex,  $\text{UO}_2 (\text{C}_{12}\text{H}_{12}\text{NO}_2)_{2.2} \cdot 2\text{H}_2\text{O}$  with cyclopentanone-2-carboxyanilide in the pH range, 6.2 to 8.0. The pH is adjusted

in two stages, first with aqueous ammonia and finally with pyridine(20%) solution. The complex containing 33.52% of the metal is weighed directly after drying at 120°C. Uranium(VI) has also been quantitatively separated and determined in the presence of  $\text{Cu}^{2+}$ ,  $\text{Cd}^{2+}$ ,  $\text{Co}^{2+}$ ,  $\text{Hg}^{2+}$ ,  $\text{Pb}^{2+}$ ,  $\text{Ni}^{2+}$ ,  $\text{Zn}^{2+}$ ,  $\text{Fe}^{3+}$ ,  $\text{Al}^{3+}$ ,  $\text{Ce}^{4+}$ ,  $\text{Th}^{4+}$ ,  $\text{VO}^{2+}$  or  $\text{CrO}_4^{2-}$ , when these foreign ions are masked with magnesium-EDTA. The relative standard deviation is  $\pm 0.4\%$ .

11. Gravimetric Determination of Mercury(II) with Cyclopentanone-2-carboxyanilide (Section 7.5)

Mercury(II) in quantities 25 to 40 mg has been determined gravimetrically by precipitating it as the cyclopentanone-2-carboxyanilide complex,  $\text{Hg}(\text{C}_{12}\text{H}_{12}\text{O}_2\text{N})_2$  containing 33.18% of the metal. The precipitation is quantitative in the pH range, 5.5 to 8.6. The complex can be weighed directly if it is carefully dried first below 105°C for half an hour and then at 110 - 115°C. Mercury(II) has also been quantitatively separated from solutions containing  $\text{Cu}^{2+}$ ,  $\text{Cd}^{2+}$ ,  $\text{Zn}^{2+}$ ,  $\text{Co}^{2+}$ ,  $\text{Ni}^{2+}$ ,  $\text{Bi}^{3+}$ ,  $\text{Fe}^{3+}$  or  $\text{Al}^{3+}$ . In these separations citric acid-sodium citrate was used as masking agent. The buffering action of this mixture was also helpful in controlling the pH. The relative standard deviation is  $\pm 0.32\%$ .