centrifuge tube. To the 4 g. of concentrate add 12 ml. of distilled water. Hot, 75°C., 95 percent ethyl alcohol is added to a volume of 40 ml. and the mixture heated for 10 min. in a water bath at 85°C. with occasional stirring, using a glass rod. The stirring rod is then rinsed off with 95 percent alcohol and the volume of the mixture made up to 50 ml. in the tube. Centrifuge the tube at 2,300 r.p.m. (1,150 × gravity) for 15 min. and after decanting discard the supernatant solution. Repeat the leaching with hot 63 percent alcohol for 10 min. in a water bath at 85°C., centrifuge and again decant and discard the supernatant solution.

Add ca. 5 ml. distilled water to the tube and disperse the precipitate with a rubber policeman. Rinse policeman with distilled water, make contents to a volume of 35 ml. and stir vigorously and continuously for 10 min. This is accomplished either by a mechanical stirrer or by bubbling air through the mixture in the tube. The bubbling device is made by connecting to a source of air a capillary tube about six in. long which is inserted into the centrifuge tube. The stirrer or capillary tube is rinsed with approximately 5 ml. distilled water increasing the volume to 40 ml., the tube centrifuged at 2,300 r.p.m. for 15 min. and the liquid decanted into a 100 ml. volumetric flask. Repeat the water extraction and after centrifuging decant into the same volumetric flask. Add 5 ml. 1N sodium hydroxide to the water extract and dilute to volume. Mix and let stand 15 min. before beginning the colorimetric procedure.

To the residue in the centrifuge tube add 5 ml. of 0.75 percent ammonium oxalate solution and disperse the precipitate with a rubber policeman. Rinse policeman with oxalate solution, make contents to a volume of 35 ml., and stir vigorously and continuously for 10 min. as described above. Make to volume of 40 ml. with oxalate solution. Centrifuge as before and decant into a 100 ml. volumetric flask. Repeat the oxalate extraction, centrifuge and decant into the same 100 ml. flask. Add 5 ml. 1N sodium hydroxide to the oxalate extract and dilute to volume. Mix and let stand for 15 min. before beginning the colorimetric procedure.

The residue remaining in the centrifuge tube is washed into a 100 ml. volumetric flask, 5 ml. 1N sodium hydroxide is added, and the contents made to volume with distilled water. Mix, let stand 15 min. with occasional shaking, and filter.