

scriptions are studied and the student is able to apply the qualitative work to this course with a degree of understanding. He is more able to realize the care needed in filling prescriptions containing alkaloids and their salts. Numerous other problems of a similar nature could be cited.

There are many situations and experiences gained in the beginning courses which can be related to those used in the advanced work in pharmacy. A more complete correlation of techniques could be worked out if a series of tests or measurements were made and then applied to both the beginning student and those of advanced training.

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#### CALCIUM DETERMINATION IN BLOOD SERUM.\*

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(SUGGESTED FOR U. S. P. ADOPTION.)

Place 2 cc. of clear blood serum, 2 cc. of distilled water and 1 cc. of a 4 per cent ammonium oxalate solution in a graduated, 15-cc. centrifuge tube with an outside diameter of from 6 to 7 mm. at the 0.1-cc. mark and mix. Mixing is facilitated by giving the tube a quick, jerky, whirling motion. Allow it to stand for thirty minutes. Again mix the contents and centrifuge for about five minutes at 1500 revolutions per minute. Carefully pour off the supernatant liquid and, while the tube is still inverted, allow it to drain in a rack for five minutes, resting the mouth of the tube on a pad of filter paper. Wipe the mouth of the tube dry with a soft cloth. Stir up the precipitate and wash the sides of the tube with 3 cc. of dilute ammonia (2 cc. of stronger ammonia T.S. to 98 cc. of distilled water) directed in a very fine stream from a wash bottle. Centrifuge the suspension and drain again as before. Add 2 cc. of approximately normal sulfuric acid by blowing it from a pipette directly upon the precipitate so as to break up the mat and facilitate solution. Place the tube and contents in a bath of boiling water for about one minute and titrate with one hundredth-normal potassium permanganate to a definite pink color which persists for at least one minute. During the course of the titration the contents of the tube must be maintained at a temperature of from 70° to 75° C. A micro-burette, graduated in 0.02 cc., should be used.

*Hundredth-Normal Potassium Permanganate.*—It is essential that the potassium permanganate solution be carefully prepared and standardized as directed below:

Dissolve approximately 4 Gm. of reagent potassium permanganate in 1000 cc. of redistilled water in a thoroughly clean Florence flask. Insert a funnel, covered with a watch glass as a condenser and digest for several hours at approximately the boiling point. Cool, allow it to stand over night and filter with gentle suction through a 3-inch Buchner funnel lined with ignited asbestos. Transfer this to a perfectly clean glass-stoppered bottle and keep it in a dark place. This serves as the stock solution. From this stock solution approximately hundredth-normal potassium permanganate is prepared by dilution and standardized against hundredth-normal sodium oxalate which should keep for several months.

*Hundredth-Normal Sodium Oxalate.*—Dry reagent sodium oxalate in an oven at from 100° to 105° C. for twelve hours. Dissolve exactly 0.67 Gm. of this oxalate in redistilled water, add 5 cc. of reagent sulfuric acid and dilute to 1000 cc. Mix well. Transfer exactly 25 cc. of this solution to a 100-cc. Erlenmeyer flask, add 1 cc. of reagent sulfuric acid, warm to about 70° C. and titrate with the permanganate solution.

The permanganate solution should be frequently restandardized. A fresh solution of hundredth-normal potassium permanganate should be prepared from the stock solution by dilution, each time the reagent is to be used.

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\* See page 202.