ASSAY OF CINCHONA BARK.

By A. PETIT.

PROLLIUS has shown that the whole of the alkaloids of cinchona bark may be obtained in solution by treating, say 40 grammes of the powdered bark with 800 gms. of a mixture composed of

Alcohol, 95 per cent.	67 parts.
Ether, sp. gr. 0.724	733 „
Ammonia	32 „

Comparative experiments have shown me that the bark must be in as fine powder as possible, and that, if the mixture be shaken every five minutes, the exhaustion is as complete after one hour, as it will be after five or six hours if merely macerated.

The next step is to pour off 600 gms. of the liquid, corresponding to three-fourths of the alkaloids contained in the bark, that is, representing 30 gms. of the latter.

Now add to the ethereal liquid enough of a solution containing one-fourth of its weight of sulphuric acid, so that the aqueous layer which separates shall be just acid. In general, about 20 cubic centimetres will be sufficient.

This aqueous layer contains all the alkaloid of the ethereal liquid.

The layer is separated by a suitable funnel (in fact the ethereal liquid should be in a separating funnel when treated with the acid), and the ethereal liquid again agitated with 5 c.c. of the diluted acid and 15 c.c. of water. This portion is likewise separated, and added to the former.

Now heat the aqueous liquid on a water-bath in order to get rid of the dissolved ether, then dilute it with two volumes of water, and precipitate with caustic soda in excess. On stirring with a glass rod, the alkaloids coalesce together in a mass. The same result may also be obtained by warming the liquid on the water-bath.

Transfer the alkaloids to a tared capsule and dry them at a temperature of 100°C . (212°F .).

If the liquid is not perfectly clear, it is passed through a tared filter, and the gain in weight of the latter when dried at 100°C . added to the alkaloids in the capsule.

We have now the weight of the total alkaloids contained in the 30 gm. of bark, and from this we may calculate the quantity contained in one kilogramme.

The next step is to ascertain the proportion of alkaloids soluble in ether. Proceed as follows:—

Dissolve the total alkaloids in a slight excess of sulphuric acid. Add 25 c.c. of ether (sp. gr. 0.724) and 5 c.c. of ammonia, and shake. The alkaloids soluble in ether are thereby taken up. Decant the ether: shake again with 10 c.c. of ether and decant again. Unite the ethereal solutions, let stand 15 minutes, so that the alkaloids which are but little soluble in this menstruum may deposit; decant again, and shake the clear, decanted ethereal liquid with 10 c.c. of diluted sulphuric acid (1 in 20). Separate the aqueous liquid; agitate the ethereal solution with 5 c.c. more of the dilute acid, and add the second aqueous layer to the first.

Dilute the united liquids with water to 25 c.c., heat to boiling, and saturate with pure diluted ammonia (1 in 5). As soon as the reaction is faintly alkaline, the heating is interrupted.

The sulphate of quinine will now separate in fine needle-shaped crystals.

When completely cold, collect it upon a tared filter, and wash it with a cold saturated solution of sulphate of quinine; finally dry it at 100°C . (212°F .), until the weight remains constant.

We now have the weight of sulphate of quinine obtainable from 30 gms. of bark, and, therefore, by a simple calculation, that contained in one kilogramme.

In order to prove that the sulphate of quinine thus obtained is pure, the salt is dissolved with the aid of sulphuric acid, and examined by the polariscope.

If the rotary power does not approach sufficiently close to -238.3 , with sodium light, at a temperature of 15°C ., the salt must be purified by a renewed treatment with ether and ammonia and recrystallization.

According to my experience, the polarimetric deviation is proportional to the quantity of salt dissolved; the amount of sulphuric acid does not influence this deviation, provided it is present in at least sufficient quantity to form bisulphate of quinine.

In practice, I prefer a solution containing one gm. of basic sulphate dried at 100° C., dissolved in two c.c., of one-tenth per cent. sulphuric acid, and enough distilled water to make twenty c.c. Under these conditions the polariscope deviation is to -110° (for pure sulphate of quinine at 15° C.). According to my experiments, it is necessary to add to the observed degree about one degree for every four degrees of temperature above 15° C.

These different treatments by acid, and the separations of the ether, are very rapidly performed if the operator has had some previous practice in these manipulations. A few hours are sufficient to make a complete assay of cinchona by this process.—*Repertoire de Pharmacie.*