

A NOTE ON THE RAPID ASSAY OF EXTRACT OF IPECACUANHA AND OF IPECACUANHA ROOT

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Received August 16, 1951

EXTRACT OF IPECACUANHA

THE B.P. method for the assay of extract of ipecacuanha consists in the initial removal of colouring matter with chloroform, the washing of this chloroform with acid and the extraction of the alkaloids from the combined acid liquors with chloroform after the addition of excess of ammonia. The solvent is then distilled off, the residual alkaloids are dissolved in excess of standard acid and the solution back-titrated with standard alkali using methyl red as indicator. In practice this method is often very long and tedious owing to the formation of very stable emulsions, which cannot be broken down even by the addition of large volumes of chloroform. Time is also wasted during the distillation, while again there is the danger that the delicate alkaloids may be partly decomposed during the final evaporation and drying.

These difficulties may all be overcome by at once making alkaline with ammonia, extracting the alkaloids with ether, washing the ether free from ammonia with water, shaking with excess of standard acid, running out the acid and back-titrating with standard alkali.¹ In this way the necessity for distillation and removal of colouring matter is avoided and no troublesome emulsions are produced, so that the whole assay can be completed in 30 to 40 minutes, the results being identical with those given by B.P. method, which often takes several hours.

Details are as follows:—Transfer 5 ml. of the extract to a separator with a pipette. Add 3 ml. of strong solution of ammonia. Shake with successive quantities of about 50 ml. each of ether until the ethereal extract is free from alkaloid. About four extractions are usually sufficient. Wash the combined ethereal extracts with successive quantities, each of about 10 ml., of water, shaking each after separation with the same quantity of about 50 ml. of ether until the ether-washed water no longer turns red litmus paper blue. Mix the ethereal extracts. Add 10 ml. of 0.1N sulphuric acid, shake well and run the acid into a beaker. Shake the ethereal layer with further quantities of water until the last washing no longer turns blue litmus paper red, adding these washings to the acid solution in the beaker. Titrate with 0.1N sodium hydroxide using methyl red as indicator.

IPECACUANHA ROOT

By using a procedure similar to the above it is possible to dispense with the extraction of colouring matter and the distillation of solvent in the B.P. assay of this substance, thus saving a considerable amount of time and tedious work. The modified method is as follows. Extract the powdered root, as in the official method, using ammonia and a mixture of chloroform and ether. Shake the solvent with successive quantities,

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each of about 15 ml., of water, shaking each water extract after separation with the same 50 ml. of ether, contained in a second separator, until the ether-washed water no longer turns red litmus paper blue. Add 25 ml. of 0.1N sulphuric acid, shake well and run into a beaker. Wash the ether with further quantities of water until the washings no longer turn blue litmus paper red, adding each washings to the acid solution in the beaker. Titrate with 0.1N sodium hydroxide using methyl red as indicator.

My thanks are due to the Directors of Messrs. Whiffen & Sons, Ltd., for permission to publish this note.

REFERENCE

1. Lowdell, *Pharm. J.*, 1946, **156**, 141.