Separation of cocaine, benzoylecgonine and ecgonine by paper chromatography

The hydrolytic decomposition of cocaine leads to formation of benzoylecgonine and ecgonine. Both compounds are intermediates in cocaine production; also benzoylecgonine is associated with cocaine in coca leaves.

Thus detection of benzoylecgonine and ecgonine is of great importance in the purity control of cocaine and cocaine salts and accordingly we have developed a paper chromatographic purity test of cocaine hydrochloride for pharmacopoeial purposes.

Only a small number of communications have been published on the paper chromatography of the decomposition products of coca alkaloids. BÜCHI AND SCHUMACHER[1] studied the separation of cocaine and tropacocaine, but not their hydrolysis products. KLEMENTSCHITZ AND MATHES[2] separated cocaine (R_F 1.00), tropacocaine (R_F 0.75), benzoylecgonine (R_F 0.53) and ecgonine (R_F 0.00) in a solvent system consisting of methyl ethyl ketone–water–pyridine–glycol monoethyl ether–ligroin. For the purpose of purity control this method has the disadvantage that cocaine travels with the front and ecgonine remains on the starting line.

For the separation of cocaine, benzoylecgonine and ecgonine we found the solvent system methyl ethyl ketone–dimethylformamide–water (2:1:1) more advantageous. We used the ascending technique on Schleicher-Schüll 2043/b chromatographic paper, development time being 15–16 h.

This method enables traces of benzoylecgonine and/or ecgonine to be separated from a relatively large amount of cocaine (400 μg).

The solvent system may be used for 6–8 days, and variations of temperature between 18° and 25° do not influence the R_F values which are: cocaine (hydrochloride) 0.90, benzoylecgonine 0.81, and ecgonine 0.50.

For the detection of the spots, VÁGUJFALVY's[3] new process was found suitable. The developed chromatograms are sprayed with a modified Dragendorff reagent*, air dried, and drawn through 0.05 *N* sulfuric acid. The alkaloids appear as quickly fading orange-coloured spots against a brown background. For spots which remain visible for longer than 2 min the limit of detection is 2 μg of benzoylecgonine or ecgonine, otherwise the limits are < 1 μg.

Our method is useful for purity control[4] and also for stability studies of cocaine preparations.

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* The preparation of the modified Dragendorff reagent is as follows: boil 2.6 g bismuth carbonate and 7 g sodium iodide for a few minutes with 25 ml of glacial acetic acid. Allow to stand overnight. Filter off the precipitated sodium acetate and add 80 ml of ethyl acetate to 20 ml of the filtrate. This solution should be protected from light. Before using dilute 10 ml of it with 25 ml of glacial acetic acid and 60 ml of ethyl acetate.