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CHROMATOGRAPHIC BEHAVIOR OF HALIDE SALTS OF ALKALOIDS ON ALUMINA

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In medical practice, alkaloids are usually used in the form of salts of mineral acids. These salts are checked for foreign impurities, mainly accompanying alkaloids, by chromatographic analysis. The amount of impurities is evaluated from the sensitivity of their detection and the amount of substance in the sample deposited. With a low sensitivity of detection (50-100 μg), 1000 and more micrograms of substance is deposited in a single point on the starting line of the chromatogram in order to detect the minimum amount of impurities.

We have observed that in this case the halide salts of alkaloids, after chromatography on a nonfixed layer of alumina are revealed by the Dragendorff reagent in the form of two spots: a main spot and a spot at the start the shape and size of which are equal to the deposited spot. The longer the chromatogram with the deposited sample was dried the clearer did the spot at the start become. In view of the fact that alumina is an active ion-exchange material [1], we assumed that during the drying of the deposited sample a reaction takes place between the salt of the alkaloid and the solvent. The alkaloid formed as the result of the reaction migrates on the plate in the form of the free base and the halide ion remains at the start and is revealed by the Dragendorff reagent in the form of a yellow spot. This assumption was confirmed by a series of experiments, which showed the following facts. The halide salts of alkaloids (hydrochlorides, hydrobromides) and the bases obtained from these salts have the same R_f values.

The reaction between the salt of an alkaloid and the sorbent takes place quantitatively. To show this, a section of the sorbent from the start after chromatography and the elimination of the solvents was eluted with water until the reaction for the halide ion was negative. The amount of halide ions was determined in the eluate by the mercurimetric method. A control experiment was performed in parallel. In all cases an amount of halide ions equivalent to the amount deposited was found at the start. The decomposition of the alkaloid salts takes place on "for chromatography" alumina with different pH values of a 10% aqueous suspension: 4-5, 6.7-7.5, and 9-10.

The sensitivity of the detection of chloride and bromide ions by the Dragendorff reagent is 500 μg .

We used systems of solvents containing chloroform, acetone, and ethanol in various proportions. The above-mentioned observations were checked on the following salts: deoxypeganine hydrochloride, lycorine hydrochloride, and galanthamine hydrobromide.

Thus, when halide salts of alkaloids in amounts greater than 500 μg are chromatographed on alumina, the decomposition of the salt and the appearance of an additional spot of the halide ions at the start must be taken into account.

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