# Quantitative Determination of Dehydrocholic Acid in Tablets

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**Quantitative recovery of dehydrocholic acid from the tablets can be accomplished by first extraction with chloroform and subsequent titration as given in the U.S.P. XVI. Results are reported to indicate that the residue obtained is of pure acid.**

THE U.S.P. XVI (1) procedure for the estimation of dehydrocholic acid appears to be satisfactory for pure samples of acid but reproducible results are obtained only with difficulty for some of the commercial preparations. Some samples require stirring for periods up to 1 hour on the magnetic stirrer before solutions are effected in order to obtain quantitative recoveries; without stirring there is a marked tendency to obtain premature end points which are difficult to distinguish from the true end point. The addition of 100 ml. of water as prescribed shortly before the end point is reached may also lead to low results if added too soon. This is probably caused by some of the acid going out of solution. Crisafio and Chatten (2) of the National Food and Drug Laboratories, Ottawa (Ontario) have reported similar difficulties.

## EXPERIMENTAL

Not less than ten tablets were reduced to a fine powder and a portion of the powder, representing about 300 mg. of dehydrocholic acid was accurately weighed and transferred to a dry 100-ml. volumetric flask with the aid of reagent grade chloroform. Sufficient chloroform was added to make exactly 100 ml. of the mixture and the flask was agitated. The mixture was allowed to stand for 1 minute and rapidly filtered through dry rapid filter paper. The first 20 ml. was rejected and the next 50 ml. of the filtrate was collected in a dry 50-ml. volumetric flask which was transferred to a 250-ml. Erlenmeyer flask or a 100-ml. tared beaker if the residue was to be weighed. The flask was washed with two 5-ml. portions of chloroform and mixed with the filtrate. Chloroform was removed by evaporation; 2 ml. of dehydrated alcohol was then added and also removed by evaporation. The residue was cooled and assayed by U.S.P. method (1) for dehydrocholic acid, using 0.02 *N* sodium hydroxide. Each milliliter of 0.02 *N* sodium hydroxide is equivalent to 8.05 mg of anhydrous dehydrocholic acid. If the tablets were known to contain no chloroform-soluble excipients such as stearic acid, then the residue was dried at 100° for 15 minutes, cooled in a desiccator, and weighed. Twice the weight of the residue represented the amount of dehydrocholic acid contained in the portion of the powder taken. The melting point of the residue and the U.S.P. method (1) for determining the dehydrocholic acid confirmed that the residue was a pure form of the dehydrocholic acid. Results are recorded in Table I.

## SUMMARY AND CONCLUSION

1. A study was designed to develop a method for 100% recovery of dehydrocholic acid from the tablets.

2. The method described required 20 minutes (actual working time) for each analysis with 100% recovery. The solvent used is available in every laboratory.

3. Assay results appear to confirm the accuracy of the method.

TABLE I.—ASSAY RESULTS FROM DEHYDROCHOLIC ACID TABLETS

| Sample No. | Recovery, U.S.P. Method | Recovery, Proposed Method | M.p. of Residue | Percentage Found in the Residue by U.S.P. Method |
|---|---|---|---|---|
| 1 | 100.5 | 102.2 | 238–241° | 102.2 |
| | 100.3 | 102.3 | as above | 102.1 |
| | 100.3 | 102.5 | as above | 102.2 |
| 2 | 98.1 | 100.5 | as above | 100.1 |
| | 98.0 | 100.5 | as above | 100.1 |
| | 97.7 | 100.7 | as above | 100.2 |
| 3 | 99.2 | 101.2 | as above | 100.9 |
| | 99.5 | 101.5 | as above | 101.4 |
| | 99.7 | 101.7 | as above | 101.4 |
| 4 | 103.7 | 105.9 | as above | 105.4 |
| | 102.5 | 105.8 | as above | 105.4 |
| | 102.8 | 105.8 | as above | 105.4 |
| 5 | 97.4 | 99.7 | as above | 99.5 |
| | 97.8 | 99.9 | as above | 99.5 |
| | 97.3 | 99.2 | as above | 99.0 |
| 6 | 98.2 | 100.1 | as above | 99.8 |
| | 97.5 | 100.3 | as above | 99.9 |
| | 97.2 | 100.3 | as above | 99.8 |

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## REFERENCES

(1) "United States Pharmacopeia," 16th rev., Mack Publishing Company, Easton, Pa., 1960, p. 200.

(2) Crisafio, R., and Chatten, L. G., THIS JOURNAL, **44**, 529(1955).