Analysis: Theor. for $C_{22}H_{15}O_4N$: 3.92% N.
Found: 4.13% N.

The *m*-hydroxymandelonitrile dibenzoate is a white crystalline compound. It was obtained after some difficulty by recrystallization as fine needles from toluene, cooled in a dry ice-alcohol bath. Its melting point is 118.5–119.5°.

Analysis: Theor. for $C_{22}H_{15}O_4N$: 3.92% N.
Found: 3.98% N.

SUMMARY

The synthesis of the dibenzoates of the three isomeric hydroxymandelonitriles has been investigated. They may be obtained:

(1) by treating the benzoate of the hydroxybenzaldehyde with an equivalent of alkali cyanide and benzoyl chloride;

(2) by treating the hydroxybenzaldehyde with an equivalent of pyridine, an equivalent of alkali cyanide and two equivalents of benzoyl chloride.

Of the three isomeric dibenzoates, the *o*- and the *m*- compounds have been, so far as known, prepared for the first time. The benzoate of *m*-hydroxybenzaldehyde is also reported for the first time.

The reduction behavior of these hydroxymandelonitrile dibenzoates will be investigated.

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Christian Eijkman (1858–1930), discoverer of the cause of beriberi, shared the Nobel Prize for medicine in 1929 with Sir Frederick Hopkins for extensive vitamin research. To his satisfaction, the antineuritic vitamin was isolated as a pure crystalline substance by his countrymen, Janses and Donath, in the laboratory in Batavia which had risen out of his own primitive research quarters.

The Analysis of Sodium Acetate

By R. M. Hitchens, G. W. Ashworth* and W. H. Demaree†*

One common method for the determination of acetic acid in acetates has consisted of distillation from a strongly acid solution and titration of the distillate. Hurd and Fiedler (1) have reported encountering considerable difficulty in the assay of sodium acetate by this process. They found it necessary to maintain the volume of liquid being distilled at 200 cc. and to carry out the distillation over a period of ten hours to prevent high results from carryover of phosphoric acid.

These observations seem surprising to us since we have long used this method as a routine assay method for sodium acetate, both alone and in the presence of sodium carbonate and sodium chloride. In view of the troubles which Hurd and Fiedler experienced we are describing below the apparatus, procedure and the results obtained with known amounts of sodium acetate and acetic acid.

EXPERIMENTAL

Apparatus.—The distillation apparatus employed is illustrated in Fig. 1. It consists of a 250-cc., round-bottom flask connected by means of a ground-glass joint to a spray column filled with small glass helices which in turn leads to a condenser. A small reservoir is fitted to the top of the column to allow addition of water to the flask. The receiver is a 250-cc. Volhard absorption flask. This simple, compact still allows rapid distillation with no carryover. It is widely applicable to a variety of quantitative distillations.

Procedure.—Sufficient sample to contain about 0.02 Gm. mol of sodium acetate is added to the flask, along with 50 cc. of water and 15 cc. of 85% phosphoric acid. Twenty cc. of water are added to the receiver to act as a seal. The solution is distilled to a volume of about 20 cc., the flame removed, 20 cc. of water run in through the reservoir and the solution again distilled to 20 cc. For safety a second 20-cc. portion of water is added and distilled as before. The distillate is titrated with carbonate-free 0.5*N* sodium hydroxide solution, using phenolphthalein or thymol blue as indicator. The entire distillation consumes only 20 minutes.

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The whole analysis can be carried out in an elapsed time of 45 minutes.

Results with Known Amounts of Acetic Acid.—An approximately 0.4*N* solution of acetic acid was prepared by diluting glacial acid with distilled water. Exactly 50.00-cc. portions of this solution were added to the distilling flask and to a 250-cc. Erlenmeyer flask. The former were distilled as above and titrated with a given, carbonate-free, 0.5*N* sodium hydroxide solution; the latter were titrated directly. In this way it was possible to detect any errors introduced by the distillation. The results were as follows:

Table I

Sample	Cc. 0.5*N* Sodium Hydroxide
Not distilled	42.83
Not distilled	42.82
Distilled	42.82
Distilled	42.81
Distilled	42.82

Thus, within the limits of the experimental error of reading a burette no loss occurs in the distillation. All distillates were tested for phosphates by acidifying with nitric acid and adding ammonium molybdate reagent. No yellow precipitate was ever obtained, and, in fact, no yellow coloring even observed. This indicates the absence of phosphoric acid carryover.

Standardization against Pure Sodium Acetate.—Three samples of sodium acetate, A. R., were dried thoroughly at 130° C. About 2 Gm. of each were weighed into the distilling flask, care being taken to avoid pickup of water during weighing. The results of these analyses are given in Table II.

Table II.—Assay of Pure Sodium Acetate

Sample	Weight of Sample, Gm.	Cc. 0.5*N* Sodium Hydroxide	Factor for Sodium Hydroxide	Per Cent Acetate
A	1.8824	45.78	1.0020	100.0
B	1.9571	45.59	1.0020	99.9
C	2.4249	58.68	1.0070	100.0

Once again, within 1 part in 1000 or within the experimental error of volumetric analysis, recovery of acetic acid is quantitative.

This method is applicable in the presence of chloride if a slight excess of silver oxide is added to precipitate the chloride as silver chloride. It is likewise applicable in the presence of carbonates if the distillate is subjected to reduced pressure and a slow stream of carbon dioxide free air led through it for a minute or so before titration.

We have found this distillation procedure to be more precise than the conventional U. S. P. XI method which involves ashing to sodium carbonate, leaching and titrating the sodium carbonate produced. It does away with the errors caused by incomplete washing and by occlusion of carbonates by the unburned carbon. It reduces manipulative losses since it involves less handling. It has proved to give more consistently reproducible results in the hands of the average analyst.

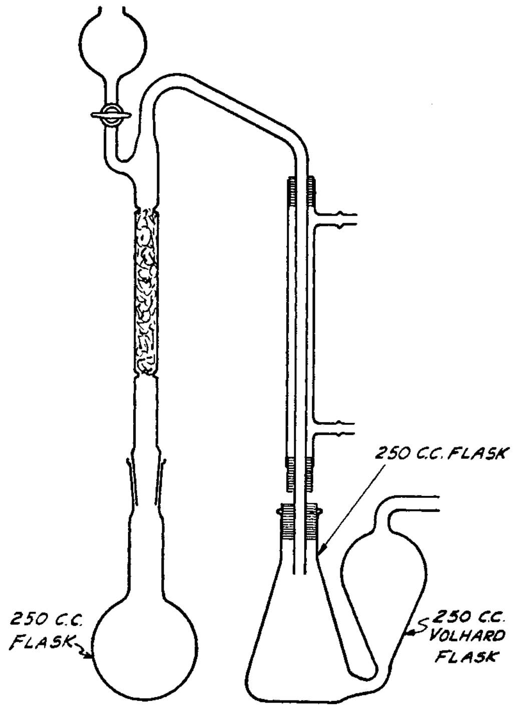

Fig. 1.—Distillation Apparatus.

CONCLUSIONS

A simple, compact, all-glass still which eliminates spray in quantitative distillations is described.

Its use in the quantitative determination of sodium acetate by adding phosphoric acid, distilling and titrating the acetic acid is described.

The method can be adapted to eliminate the effect of chlorides and carbonates.

The errors observed by Hurd and Fiedler are the result of faulty apparatus.

The method has certain definite advantages, notably speed and precision, over the U. S. P. XI ashing procedure.

REFERENCE

(1) Hurd, C. B., and Fiedler, W., Jr., *Ind. Eng. Chem.*, *Anal. Ed.*, 9 (1937), 116.

"It is provided in the nature of things that from any fruition of success shall come forth something that shall make a greater effort necessary."—WALT WHITMAN.