and the secondary reaction is:

$$CH_3COCH_2CH_3 + 5I_2 + 4NaOH = 2CHI_3 + CH_3COOH + 4NaI + 3H_2O \quad (4)$$

Cassar found that the iodoform method yielded about 110.6% of theory and he believes that the high results may be attributed to the secondary reaction and its subsequent consumption of more iodine.

SUMMARY

1. There are three general methods for assaying acetone: the mercury complex method, the oxime method and the iodoform method.

2. The mercury method gives the lowest results, the oxime method high results and both give variable results.

3. The iodoform method gave the most uniform results, but the results are high. It is believed that these high results are due to a secondary reaction involving formate and consuming ten rather than six atoms of iodine.

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Decomposition Rate of Ethyl Nitrite in Brown Mixture, U. S. P.

By Edward Greenfield and H. Walter Kuhl**

Several samples of Brown Mixture, U. S. P. XI, known to be approximately six months old, were tested for their ethyl nitrite content. Analysis showed none of these samples to contain any ethyl nitrite. In view of this, experiments were made to determine the rate of decomposition of this ingredient in the mixture.

EXPERIMENTAL

A modified Peter Griess (1) method for the determination of nitrous acid was employed in these experiments. This colorimetric method is extremely sensitive, depending upon the formation of intensively colored azo dyes (2) and detecting as little as 0.001 mg. HONO in a liter. It was assumed that the ethyl nitrite was hydrolyzed to nitrous acid by the action of the acetic acid in the reagent. A standard solution of sodium nitrite was used for quantitative colorimetric comparison.

Procedure.—A well-shaken sample of Brown Mixture was diluted to 4000 volumes to yield a theoretical concentration of 0.3 mg. C_2H_5ONO per liter in a practically colorless solution. Fifty cc. of this latter solution, theoretically containing 0.015 mg. C_2H_5ONO, was used for the actual test. After negative results were obtained with the original samples of Brown Mixture, the theoretical amount of Spirit of Nitre, as per U. S. P., was added to a composite of these samples. At the same time a fresh batch of Brown Mixture was prepared by the official U. S. P. method. These two samples were individually assayed each day, after preparation, up to a month. The composite results follow:

RATE OF DECOMPOSITION OF ETHYL NITRITE IN BROWN MIXTURE

Time after Manufacture of Mixture	Ethyl Nitrite Remaining, in Per Cent
0	100.0
4 Hours	66.5
28 Hours	39.5
2 Days	33.3
5 Days	13.3
6 Days	6.7
1 Month	Negligible

From the above results, it is evident that the ethyl nitrite begins to decompose as soon as it is added to the preparation. It is doubtful whether it has any therapeutic (3), (4) or other value in the preparation unless it is used immediately after manufacture. It has been shown (5) that on deterioration ethyl nitrite develops no new kind of pharmacologic activity.

CONCLUSION

The decomposition rate of ethyl nitrite in Brown Mixture U. S. P. is so rapid and extensive as to render it valueless in the latter preparation.

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