

Table III—Effect of Calcium Lactate on the Whipping Ability of Ice Cream Mix

Time in freezer Minutes	(Per cent overrun)		
	Control	AGED 48 HOURS	
		0.1 per cent calcium lactate	0.2 per cent calcium lactate
1	14.0	6.5	6.0
2	30.5	14.0	12.5
3	42.0	19.0	16.0
4	47.0	24.5	22.0
5	47.0	33.5	32.0
6	48.0	42.5	42.0
7	53.0	54.0	56.0
8	63.0	66.0	65.0
9	70.0	73.0	72.0
10	78.0	77.5	76.0
11	85.0	81.0	82.0
12	95.0	84.0	85.0
13	...	90.0	89.0
14	...	90.0	87.0
15	...	90.5	89.0

In order to determine whether the sodium citrate was operative through its effect on the milk proteins alone, on the gelatin alone, or on both, the experiment reported in Table IV was conducted. Two batches of mix were prepared alike in every respect except that one contained 0.4 per cent gelatin and the other contained no gelatin. These two batches were in turn divided into two groups each, and to one of each group 0.4 per cent sodium citrate was added prior to aging. The mix containing both gelatin and sodium citrate whipped up considerably faster than the others, indicating that the sodium citrate is operative through its effect on both the gelatin and the milk proteins.

Table IV—Effect of Sodium Citrate on the Whipping Ability of Ice Cream Mix with and without Gelatin

Time in freezer Minutes	(Aged 48 hours)			
	WITH 0.5 PER CENT GELATIN		WITHOUT GELATIN	
	Without sodium citrate	0.4% sodium citrate	Without sodium citrate	0.4% sodium citrate
1	19.0	21.0	42.5	37.0
2	42.0	63.0	64.0	64.0
3	61.0	92.5	73.0	81.0
4	72.0	112.0	80.0	92.0
5	74.5	120.0	79.0	99.0
6	74.0	117.0	77.0	100.0
7	72.0	120.0	77.0	100.0
8	73.0	123.0	83.0	105.0
9	78.0	127.0	83.0	112.0
10	84.0	130.0	93.0	...
11	88.0	...	98.0	...
12	94.0

Conclusion

This preliminary report points out the importance of the milk salts as a factor in determining the whipping ability of ice cream mixes, a factor that had hitherto been overlooked. These results indicate an explanation for variations in the whipping ability of mixes that are otherwise uniform in their composition, variations which always have been very puzzling and up to the present time inexplicable.

Work in this field is being continued by the authors to determine what other salts have such an effect, the manner in which the salts exert this effect, and the application of the results to commercial practice. The possibility of affecting an economy in the manufacture of ice cream by the use of sodium citrate is being considered.

Catalytic Removal of Oxygen from Gas Mixtures Containing Hydrogen¹

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ALTHOUGH absorption methods for the removal of free oxygen from gas mixtures are known, it is often more convenient to effect its removal by causing its catalytic combination with hydrogen to form water. This is particularly true in working with the large volumes of gas for the commercial synthesis of ammonia. For the preparation of hydrogen-nitrogen mixtures for this purpose, by-product nitrogen resulting from the production of oxygen by the fractionation of air may be used to advantage where it is available. Such nitrogen contains oxygen in amounts which will be dependent in general upon the degree of purity sought for the oxygen. The concentration of oxygen in such waste nitrogen is too low to permit free combustion with hydrogen, but the reaction can be made to go readily in the presence of suitable catalysts.

Among the readily available catalyst materials for water synthesis, copper appears to be the most promising and, therefore, a brief study has been made of the effectiveness of various forms of this metal.

Experimental

The catalysts tested were (1) copper shot, (2) copper reduced from Baker and Adamson C. P. granular copper oxide, (3) copper reduced from fused copper oxide which was prepared by E. C. White of this laboratory by heating copper in an oxidizing atmosphere with an oxyhydrogen flame. All the catalysts were 10 to 14 mesh. The reduction of the copper oxides was started at 310° C. in 3H₂:N₂ and allowed to continue without further application of heat. After reduction had ceased under these conditions, the temperature was maintained at 310° C. until reduction was complete.

¹ Received April 16, 1926.

In making the oxygen removal tests, purified 3H₂:N₂ gas and oxygen, after passing through calibrated flowmeters, were mixed and entered the catalyst contained in a glass reaction tube mounted in a constant temperature vapor bath. After leaving the catalyst, the gas passed in succession through a water trap, concentrated sulfuric acid, fused potassium hydroxide, and phosphorus pentoxide. All or an aliquot part of this dry effluent gas was then analyzed for free oxygen by passage over platinumized asbestos mounted in a constant-temperature vapor bath (310° C.) and collecting the water formed in a weighed U-tube containing phosphorus pentoxide.

Before beginning each test the entire system was flushed with 3H₂:N₂, and blank tests were made on the oxygen content, which gave results of 0.001 to 0.003 per cent O₂ by volume. The activity of the platinumized asbestos in the analyzing system was also checked at intervals by admitting a known quantity of oxygen in 3H₂:N₂ directly to the platinum catalyst and weighing the water formed.

The results for various rates of flow are given in Table I.

Table I—Removal of Oxygen from 3H₂:N₂ by Copper Catalysts

Material	Temperature ° C.	Space velocity	VOLUME, PER CENT O ₂	
			Inlet gas	Exit gas
Copper shot	310	12,000	0.89	0.105
	444	12,000	3.05	0.006
		60,000	2.00	0.01
Copper oxide (Baker and Adamson)	310	120,000	4.00	0.009
		180,000	2.00	0.059
			4.00	0.084
	60,000	2.00	0.083	
		4.00	0.081	
		2.00	0.006	
		4.00	0.006	
		120,000	2.00	0.016
		4.00	0.011	
Copper oxide (fused)	310	180,000	2.00	0.029
		240,000	4.00	0.016
			2.00	0.073

The volume per cent of oxygen remaining in the effluent gas is given as a measure of the catalytic efficiency. Although the highest accuracy was not sought in these experiments, the results of blank and reproducibility tests indicate that the maximum error is equivalent to less than 0.01 per cent oxygen in the exit gas results. The results show a decided advantage for reduced oxide catalysts over massive copper. Copper from the fused oxide was the most active, although both of the reduced catalysts are capable of effecting practically complete removal of 2 or 4 per cent of oxygen at 60,000 space velocity, which corresponds to a flow of 1000 liters per minute per liter of catalyst volume.

It will be noted that the oxygen leakage at equal rates of flow is less for gas containing 4 per cent oxygen than it is for 2 per cent of oxygen. This is readily explainable when it is

remembered that the temperatures given in the table are those of the vapor baths used and that the actual temperature of the catalyst was higher than this in proportion to the exothermicity of the reaction, which in this case, of course, increased with the oxygen content.

In conjunction with these experiments, a few tests were made employing 80-mesh platinum gauze as a catalyst. Extensive tests were not made, since a preliminary study indicated that it was much less promising than the reduced copper in granular form. It may be of interest, however, to state that a single layer of this gauze heated electrically to 750° C. was able to decrease the oxygen concentration from 2 or 4 per cent, to about 0.01 per cent, with a gas flow of 1 liter per minute per square centimeter of gauze area.

The Suitability of Chloroform for Alkaloidal Assay¹

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The usual purity tests to determine the suitability of chloroform for alkaloidal assay are inadequate. Automatic continuous extraction affords easy and certain means of determining such suitability.

THE presence and character of contaminating impurities in chloroform have been exhaustively studied by Baskerville and Hamor² in their general investigations of anesthetics. In the last article of the series these authors present a scheme for testing chloroform, which forms the basis of present Pharmacopeial and analytical reagent tests now generally employed in the examination of this product.

In this paper it is proposed to show that the ordinary tests employed for examining chloroform are not adequate to ascertain its suitability for alkaloidal assay. This report deals with the subject in a more or less empiric way, and no attempt is made to determine the origin or nature of contaminants.

Chloroform is doubtless the most widely used of all solvents in pharmaceutical analysis. While it is well known that a high-grade chloroform is necessary in such analysis, the extent and manner of purification necessary to render it suitable for alkaloidal assay are hardly appreciated. The error that might be introduced by the use of faulty chloroform where the alkaloid extraction is carried out in the cold by the hand-shaken separatory funnel is variable, depending on a number of factors not easily controlled. Automatic, continuous extraction,³ however, where contact of solvent (hot) with the alkaline aqueous layer and with the alkaloid can be conveniently prolonged and controlled, affords a satisfactory means of ascertaining the effect of the solvent on the alkaloid being extracted. The character of the errors introduced indicates that in the main partial neutralization, and to some extent destruction also, has occurred. In effect, however, a low titration result is obtained.

Solvents Used

Chloroforms 1, 2, 3, 4, and 9 made by different manufacturers, all labeled "U. S. P." and found upon examination to meet the Pharmacopeial requirements.

Reagent Chloroforms R-5, R-6, R-7, R-8, obtained from different manufacturers of chemical reagents and labeled as reagents, sold as of analytical reagent grade.

Chloroforms T-3 and T-4 are chloroforms 3 and 4, respectively, which have been given a purification treatment involving refluxing for several hours with brucine and then removing from the alkaloid by distillation.

Experimental Results

VARIATION IN CHLOROFORM—The fact that some U. S. P. and reagent chloroforms may be unsuitable for quantitative alkaloidal extractions was forced to the writers' attention by the low and variable results obtained in the course of an investigation, when a change was made in the brand of chloroform being used (Table I).

Table I—Nux Vomica Preparations

No.	Method of assay	ALKALOIDS PER 10 CC. FLUID EXTRACT	
		Chloroform 1 Gram	Chloroform 2 Gram
46671	U. S. P.	0.2293	0.2380
	U. S. P. modified ^a	0.2284, 0.2193	0.2370
50803	Automatic extractor		0.2417, 0.2417
	U. S. P. modified ^a	0.1984, 0.2029, 0.2018	0.2155, 0.2164,
	U. S. P.	0.2133	
	Automatic extractor		0.2155
Sol. X ^b	Automatic extractor	0.0895, 0.0976, 0.0946	0.1088
Sol. XX	Automatic extractor	0.1274	0.1463

^a *J. Am. Pharm. Assoc.*, **13**, 691 (1924).

^b Sol. X gave with benzene as solvent—0.1085 gram alkaloid per 10 cc. fluid extract.

This showed the need for investigating the various brands of reagent chloroform on the market. Chloroform 1, which, as shown in Table I, gave erroneous results, was subjected to an even more rigid examination for compliance with reagent requirements by the usual reagent tests and found to be superior, if anything, to chloroform 2. The alkaloid extractions were made, as indicated in the table, by several different methods, both in the hot continuous method and cold hand-shaken separatory funnel method.

Considerable variations are obtained with the two different chloroforms. It is also apparent that even in cold extraction (U. S. P. method) the error is appreciable. Incidentally, it may be observed that the better chloroform (No. 2) on prolonged extraction with hot solvent, using automatic extractor, not only showed no loss of alkaloid, but the results so obtained were even higher than the highest by the separatory

¹ Received May 10, 1926.

² *THIS JOURNAL*, **4**, 212, 278, 362, 422, 499, 571 (1912).

³ Palkin, Murray, and Watkins, *Ibid.*, **16**, 612 (1925).