Improved Apparatus for the Gravimetric Determination of Carbon Dioxide

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THE USUAL APPARATUS for the determination of carbon dioxide is arranged horizontally in a train of components comprising a flask for decomposition of the sample, a bubble counter, sulfuric acid drying bulbs, and an absorption tube for recovery of the evolved carbon dioxide. Thus much bench space is occupied, and quite often it presents a "straggly" appearance.

Description of Apparatus

Figure 1 shows the details of a new apparatus for the determination of carbon dioxide in soap and synthetic detergent products. This apparatus requires little bench space.

Since the apparatus contains only one sulfuric acid drying bulb, it is important that air be slowly drawn through the apparatus, *i.e.*, at a rate that discrete bubbles rise through the acid. An electric mantle or gas flame may be used to heat the apparatus. However, when analyzing synthetic detergents for carbonate content, it is preferable to employ a gas flame for heating as this permits better heat control in the event of excessive foaming.

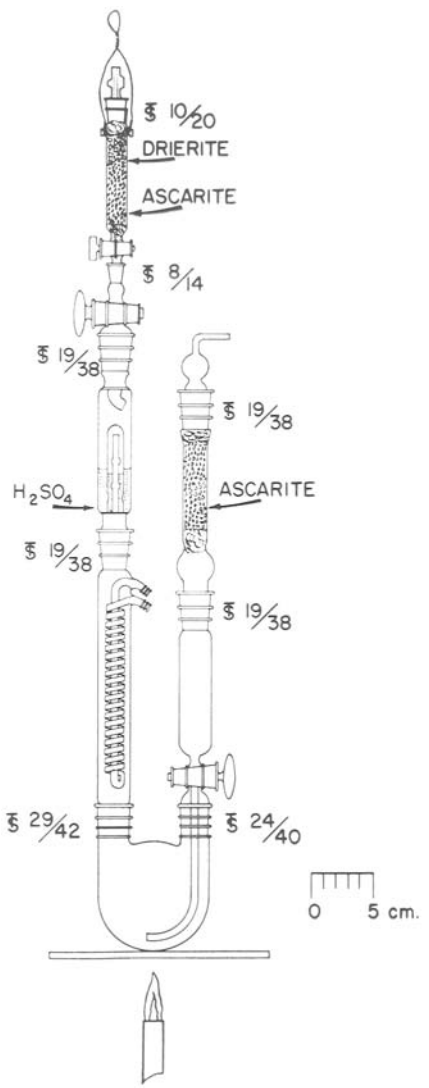

FIG. 1.

Method of Use

The procedure for use of the apparatus is similar to that described in the literature for the customary form of train (1, 2). The apparatus is also suited for the determination of bicarbonates (3).

A weight of sample sufficient to yield *ca.* 0.15 g. of CO_2 should be taken for analysis. The sample is decomposed with 20 ml. of dilute HCl (equal volumes of concentrated acid and water), and the contents of the sample flask are brought to a boil over a period of 15 to 20 min. Continue boiling for 1 to 2 min. Discontinue heating but continue aspirating until the flask has cooled to room temperature (approximately 30 min.). Remove the CO_2 absorbing tube and weigh. Replace in the train and reweigh at successive 5-min. intervals of aspiration until constant weight is attained.

Experimental Results

Table I presents recovery results for pure, dry sodium carbonate obtained by use of the new type of apparatus. Table II gives a comparison of the values obtained in the analysis of sprayed soap product and synthetic detergent products with the new apparatus and with the apparatus described in the A.O.C.S. Official Method Da 19a-42.

TABLE I

Recovery Results Obtained by Use of Improved CO_2 Train

Material	Na_2CO_3 taken	Na_2CO_3 recovered
	gms.	*gms.*
Na_2CO_3	0.0762	0.0754
Na_2CO_3	0.2627	0.2626
Na_2CO_3	0.5591	0.5589
Na_2CO_3	1.1316	1.1316

TABLE II

Comparison of Results Obtained by Use of Improved Train and A.O.C.S. Official Method Da 19a-42

Material	Wt. sample	Percentage Na_2CO_3 content	
		New apparatus	A.O.C.S. Method
	gms.		
Sprayed soap	2.4374	2.18	2.28
Sprayed detergent product	5.5498	5.90	5.92
Mixed detergent product	0.9738	38.82	38.91

Acknowledgment

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REFERENCES

1. Official and Tentative Methods of the American Oil Chemists' Society, Official Method Da 19a-42 (1955).
2. Hillebrand and Lundell, "Applied Inorganic Analysis," 2nd ed., p. 768, John Wiley and Sons Inc., New York (1953).
3. Kelley and Blank, J. Am. Oil Chemists' Soc., *26*, 685 (1949).

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