VII. DETERMINATION OF VOLATILE SUBSTANCES.

By GEORGE GAZENKO.

From Prof. Fromholdt's Laboratory of Propedeutics, State University, Moscow.

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THE quantitative determination of volatile substances in solution cannot be always considered satisfactory for many reasons.

Some methods of measuring the amount of ammonia (Schloesing) require some days for their execution. Folin's method of blowing air through the solution in the cold is attended with some risk of losing a part of the substance with too rapid an air current. The same danger is observed in the measurement of preformed acetone by the formation of iodoform.

Starting from Folin's method, it was decided, in order to eliminate all possibility of loss of the substance to be determined, to enclose the whole system, as is done in Regnault's respiration-chamber. A special apparatus was constructed based on the following principle. Through the liquor to be analysed air is passed, by means of an air-pump, which carries away the volatile substance and enters, in the form of small bubbles, the receiver containing a standard solution in which it is absorbed. After passing through the standard solution the air is again pumped through the liquid by the same air-pump and circulates indefinitely.

In this way the volatile substance, if not absorbed at first in the receiver, is repeatedly brought into contact with the standard solution and is finally completely absorbed. The circulation being effected in a closed system, the possibility of any loss of substance is eliminated.

An experiment with our apparatus was executed on a standard solution of $(\text{NH}_4)_2\text{SO}_4$.

The amount of ammonia had previously been determined by means of

simple distillation with sodium hydroxide and titration. 10 cc. standard solution neutralised 18.7 and 18.8 cc. of $N/10$ acid.

Distilled	Time of distillation (min.)	Amount of $N/10$ H_2SO_4 neutralised
10 cc. of standard solution	(1) 35	18.7
$(NH_4)_2SO_4$ + 20 cc. 33 %	(2) 20	18.7
NaOH without heating	(3) 15	18.7
in our apparatus	(4) 10	18.7

The figures for the determination of ammonia in three different urines by means of our apparatus and the method of Krueger-Reich-Schittenhelm are as follows.

	$N/10$ H_2SO_4 neutralised	
	Krueger method	With our apparatus
25 cc. urine + 10.0 g. NaCl + 1.0 g. Na_2CO_3 . Schittenhelm adds some alcohol; we have used vaselin oil	(1) 5.7	5.9
		5.6
		5.6
	(2) 15.7	15.9
		15.7
		16.1
	(3) 13.7	13.8
		13.8

A series of experiments with varying quantities of alkali and with alcohol (as in the method of Krueger-Reich-Schittenhelm) did not show any advantage. Prolonged distillation (more than 20 minutes) with our apparatus did not increase the amount of H_2SO_4 neutralised.

Excellent results were obtained with the apparatus in the determination of acetone. 2 cc. of a standard solution of acetone in water required 3.8 cc. of $N/10$ iodine. From the same quantity of standard solution in our apparatus we have obtained the same result (3.8 cc.) in 10 minutes. (We have also obtained correct results with a micro-apparatus.)

According to this, we are entitled to claim that the apparatus described above, in spite of its technical deficiencies, can be of great use owing to the simplicity of the methods used and the almost automatic character of its work, greatly shortening the time required for many analyses.

There is no doubt that the closed circuit system can be utilised in other ways in work with volatile substances.