

## Notes

### Shusaku Sakamoto and Tsutomu Kaneko: Automatic Combustion of Sulfur.

(Pharmaceutical Institute, Medical Faculty, University of Tokyo\*)

As a part of studies on the automatization of elemental microanalysis, automatic combustion method was devised for the quantitative determination of sulfur in organic compounds. The principle of this method follows that of Stragand<sup>1)</sup> in which a sample is burned in oxygen stream using a platinum contact and the sulfur trioxide formed by combustion is made to absorb on silver wire. By weighing this silver wire, the amount of sulfur is calculated from the increase in weight. Reëxamination of this method by hand manipulation has already been reviewed<sup>2)</sup>.

Determination of sulfur was attempted, using the automatic combustion apparatus given in the previous paper<sup>3)</sup>, with some modification of the hand manipulated method. The attempt gave some promising results.

The oxygen used was a moist one, and the speed of its flow rate was controlled by a pressure controller to 5~8 cc. per minute during analysis.

The temperature of the fixed furnace was kept at 700°, fixed furnace around the silver wire portion at 500~530°, and the movable furnace at 800~820°.

The analysis was carried out by the following procedure:

When the apparatus was set completely, platinum contact and silver wire were placed in their proper places. The furnace was brought to the desired temperature and heating kept up for 20 minutes in oxygen stream. Silver wire was then taken out, allowed to stand near the balance for 10 minutes and weighed, until a constant weight of  $\pm 0.020$  mg. has been obtained. About 3 mg. of the sample was weighed into a platinum boat, and silver wire and the sample in boat placed in the combustion tube. The cam was adjusted so that the movable furnace reached the fixed furnace in 30 minutes, and the burning began. When the movable furnace had reached the fixed furnace, it was left in that place for 10 minutes, and then the electric switch was cut off for the movable furnace. After allowing this to be left for five minutes, the silver wire was taken out, allowed to stand near the balance for 10 minutes, and weighed. The difference in weight of the silver wire was taken as the increase in weight of  $\text{SO}_4$ .

When the analyses were carried out continuously, the temperature of the fixed furnace and that over the silver wire portion were kept at the necessary limit, with the oxygen stream continuously passed through.

Some of the results obtained by the automatic combustion are given in the following Table.

Sample Compound	Sample mg.	$\text{SO}_4$ mg.	S, Calcd. %	S, Found %	Difference
Sulfadiazine	3.579	1.354	12.81	12.52	-0.19
	3.215	1.220		12.66	-0.15
Sulfacetamide	3.130	1.386	14.96	14.78	-0.18
	3.128	1.384		14.77	-0.19
Sulfanilamide	3.094	1.711	18.82	18.45	-0.17
	3.165	1.748		18.43	-0.19
Sulfathiazole	2.690	2.021	25.12	25.07	-0.05
	3.389	2.463		24.82	-0.30
Sulfonal	3.048	2.519	28.08	28.37	+0.29
	3.364	2.812		27.90	-0.18

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\* Motofuji-cho, Bunkyo-ku, Tokyo (阪本秀策, 金子 力).

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2) J. Pharm. Soc. Japan, 70, 287 (1950).

3) S. Sakamoto: J. Pharm. Soc. Japan, 72, 509 (1952)