

Experimental

The general methods of preparation of the various intermediates have been described in previous articles in this series.

Summary

1. A number of dialkyl acetic acids having 12, 13 and 14 carbon atoms in the acid molecule have been prepared and tested for bactericidal action toward *B. Leprae*.

2. The dodecanoic acids have no bactericidal action, the tridecanoic acids practically no action, and the tetradecanoic acids have slight bactericidal action. This indicates as in previous researches that the molecular weight of the molecule plays an important role.

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[CONTRIBUTION FROM THE CHEMICAL LABORATORY OF THE UPJOHN COMPANY]

MICRO-DETERMINATION OF SULFUR BY FUSION

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In the course of several sulfur determinations by the Pregl method, the question was raised as to the possibility of finding a method which would require less apparatus and manipulation.

The method of Récsei¹ was unsatisfactory because of the tendency of sodium peroxide to deteriorate on standing, also the danger accompanying the use of it, the action at times taking place with explosive violence. It was found that the macro method employing potassium nitrate as an oxidizing agent could be modified for use as a micro method.

Experimental

Prepare the fusion mixture by mixing four parts by weight of sodium carbonate and three parts of potassium nitrate and grinding the mixture to a fine powder. This can be kept indefinitely.

Weigh a finely powdered sample equivalent to approximately 0.5 mg. of sulfur and mix thoroughly with 100 times its weight of the fusion mixture in a 20-cc. nickel crucible. Sprinkle a thin layer of the mixture over the top to prevent sulfur fumes from escaping. Place a close-fitting cover on the crucible and set it in a porcelain crucible of convenient size.

With a Bunsen burner apply a very low flame, gradually increasing it during ten minutes until the maximum is reached. Continue heating for fifteen minutes, then shut off the flame and allow the crucible to cool.

Dissolve the contents of the crucible in 5 cc. of warm water and filter into a 30-cc. beaker. Wash the crucible and filter with 10 cc. more water and add to the solution. Acidify with hydrochloric acid and heat to boiling,

¹ Andor Récsei, *Chem.-Ztg.*, **50**, 785 (1926).

Precipitate with 1 cc. of a 10% solution of barium chloride and let the mixture stand for at least one hour, after which the precipitated barium sulfate may be weighed on a micro Neubauer crucible. To prepare the crucible, wash it with chromic acid and water and then, after closing the crucible with the lid and lower cap, ignite it on a crucible cover and weigh when cool.

Filter the precipitate in the same manner as with a Gooch crucible in the macro method and again ignite and weigh the crucible as before. A few results are given in Table I.

TABLE I
PERCENTAGE OF SULFUR IN ORGANIC COMPOUNDS

Compound	Sample, mg.		BaSO ₄ , mg.		Sulfur, %		
	Calcd.	Found	Calcd.	Found	Calcd.	Found	Found
Cystine	4.472	7.039	8.516	13.412	26.06	26.15	26.16
Methyl orange	9.125	6.988	6.390	4.910	9.8	9.62	9.65
Aminonaphthol sulfonic acid	5.807	6.698	5.729	6.646	13.47	13.55	13.62
Thymol blue	8.230	8.698	3.960	4.185	6.87	6.61	6.61
Potassium methyl sulfonate	4.646	7.200	8.152	12.562	23.89	24.09	23.96
Sulfanilic acid	5.604	5.366	7.598	7.243	18.52	18.62	18.53

Summary

By this method the sulfur content of any sample can be determined very accurately. It requires very little manipulation and apparatus and is economical in respect to time and material.

KALAMAZOO, MICHIGAN

NEW BOOKS

Physikalisch-chemisches Praktikum. (Laboratory Manual of Physical Chemistry.)

By Professor Dr. K. FAJANS, and Dr. J. WÜST, of the University of Munich. Akademische Verlagsgesellschaft m. b. H., Schlossgasse 9, Leipzig C 1, Germany, 1929. xvi + 217 pp. 74 figs. 16 × 24 cm. Price, unbound, M. 12; bound, M. 13.50.

The outstanding feature of this laboratory manual is the great variety of experiments presented. In addition to the experiments commonly found in laboratory guides, we have here chapters on Adsorption from Solution, Coagulation of Sols by Electrolytes, Metallography, Rate of Radioactive Disintegration, Adsorption of Light, Ultraviolet Spectroscopy, Electrochemical Preparations and the Lead Accumulator. The experiments are not as formidable as this array of topics might suggest, being carried out for the most part with quite simple apparatus.

The experiments have been used at the Munich laboratory for the most part, the rest having been tested at Würzburg, Karlsruhe, or Erlangen. The volume contains sufficient material to occupy a student's entire time for one semester. Each chapter begins with a theoretical discussion, complete enough to enable the student to understand the experiment.