Possible Applications of the Cell

The preliminary results which have been presented show that the cell is peculiarly adapted to the study of such environmental factors as temperature, *P*H and oxygen tension on the growth and activity of the proteolytic, ammonia-producing anaerobes. There is no reason why the cell might not be successfully applied to the effects of antiseptics, carbon dioxide tension, antagonistic action of salts or the stimulating effect of extractives on this type of organism.

The cell may possibly be applied to the study of any metabolic process involving the production of conducting substances from slightly or non-conducting substrates, for example, the rate of acid production from carbohydrates. It should also lend itself to investigations of the rate of disappearance of conducting substances as, for example, the conversion of nitrate into nitrogen by denitrifying bacteria.

Summary

A new method of studying biochemical activities of certain organisms involving a combined culture conductivity cell has been developed.

This has been successfully applied to the study of biochemical activities of anaerobes at different *P*H's, temperatures and oxygen tensions.

Further possible applications of the new cell have been enumerated.

OMAHA, NEBRASKA

[CONTRIBUTION FROM THE CHEMICAL LABORATORY OF THE UNIVERSITY OF WISCONSIN]

THE PREPARATION AND STUDY OF TWO AMMONIUM MOLYBDOTELLURATES

BY V. W. MELOCHE AND WILLARD WOODSTOCK

RECEIVED SEPTEMBER 10, 1928 PUBLISHED JANUARY 8, 1929

Considerable work has been done by Denigès[1] on the adaptation of the complex phosphomolybdate to the colorimetric determination of small amounts of phosphorus. His method depended upon the formation of a blue color upon the addition of stannous chloride to an acid solution of the phosphomolybdate. Since Gibbs[2] and Klein[3] have already mentioned the formation of complex molybdotellurites and tungstotellurites and Pechard[4] has isolated complex molybdoselenites, it was thought that a study of the possible combinations between telluric acid and molybdic acid might furnish the basis for a delicate determination of tellurium similar to the one used for small amounts of phosphorus. The following

[1] M. G. Denigès, *Compt. rend.*, **171**, 802 (1920).

[2] Gibbs, *Am. Chem. J.*, **17**, 177 (1895); *Ber.*, **18**, 1089 (1895).

[3] Klein, *Bull. soc. chim.*, **42**, 169 (1884).

[4] Pechard, *Compt. rend.*, **117**, 104 (1893).

contribution includes the isolation and analysis of two new molybdotellurates, together with a discussion of some of their properties.

Materials Used

Telluric Acid.—Telluric acid was prepared by the Staudenmeier[5] method. This procedure depends on the chromic acid oxidation of pure tellurium dioxide in dilute nitric acid solution. The product was recrystallized from water solution until it was free from nitric acid, chromic acid and chromic nitrate.

Molybdic Oxide.—Two types of C. P. molybdic oxide were used, the one being 99.9% MoO_3 and the other 85% MoO_3. The latter variety is sometimes referred to as H_2MoO_4 but in this instance it contained variable amounts of ammonia and nitrate. None of the samples contained sodium, potassium or sulfate.

Ammonium Nitrate.—Ordinary C. P. ammonium nitrate was recrystallized from aqueous solution and used without further treatment.

Preparation

The isolation of two different ammonium molybdotellurates was accomplished by the preparation of aqueous solutions containing varying amounts of telluric acid, molybdic acid and ammonium molybdate and subsequent separation of the resulting products by evaporation and crystallization. When an aqueous solution was prepared which contained equimolar portions of telluric acid and molybdic acid and sufficient ammonia to satisfy three of the hydrogens of the H_6TeO_6, clear white crystals separated upon evaporation and cooling which had the formula $3(NH_4)_2O\cdot2TeO_3\cdot6MoO_3\cdot10H_2O$. If the above proportion was so changed that the solution contained a considerable excess of molybdic acid in the ratio of one mole of telluric acid to four moles of molybdic acid, and ammonia was present in sufficient quantity to satisfy the telluric acid completely, the solution upon evaporation and cooling would yield clear white crystals having the formula $3(NH_4)_2O\cdot TeO_3\cdot6MoO_3\cdot7H_2O$. Each of the two different compounds was recrystallized six times and the complete analysis of samples taken from the last crystallization was compared with that of samples taken from the first and second crystallizations. In the instances where the pure molybdic anhydride was used for the preparation, it was found necessary to use aqua ammonia to facilitate the solution of the molybdic oxide. Variations of the general procedure showed that the ammonium ion could also be provided by the addition of ammonium molybdate or ammonium tellurate.

In order to prepare samples for analysis, the crystals were dried by successive washing with alcohol and ether.

Methods of Analysis

Tellurium.—The tellurium content of the compounds was determined by the Lenher-Homberger[6] method in which elementary tellurium was precipitated by the

---

[5] Staudenmeier, *Z. anorg. Chem.*, **10**, 189 (1895).

[6] Lenher and Homberger, THIS JOURNAL, **30**, 387 (1908).

combined action of saturated sulfur dioxide water and hydrazine hydrochloride solution upon a dilute hydrochloric acid solution of the sample. It was found desirable to digest the sample in concentrated hydrochloric acid solution before attempting to precipitate the tellurium in the dilute acid solution.

Molybdic Oxide.—The filtrate from the determination of tellurium was evaporated to small bulk on a water-bath and was then fumed with about twenty cubic centimeters of concentrated sulfuric acid. The resulting sample was diluted until the acid concentration was about 6% and the molybdenum was determined by reduction in a Jones reductor and titration with potassium permanganate. The titration was corrected by a blank which was run on the reagents used. Standardization of the permanganate was made upon both sodium oxalate and pure molybdic oxide.

Ammonia.—The original sample was treated with an excess of sodium hydroxide and the liberated ammonium hydroxide distilled into a standard solution of hydrochloric acid. The excess acid was titrated with standard sodium hydroxide and the results were calculated in terms of $(NH_4)_2O$.

Water.—On account of the presence of the ammonium group in the compounds, it was found necessary to modify the ordinary method which is used for the determination of water. Instead of heating the sample and collecting the liberated water in a weighed U-tube which contained concentrated sulfuric acid, the sample was heated in a stream of oxygen in an organic combustion furnace. The porcelain tube contained the usual reduced copper spiral and the finely divided copper oxide. The water evolved was collected in a weighed tube which contained phosphorus pentoxide. Since the oxygen available contained appreciable amounts of hydrogen, it was necessary to pass the oxygen over hot copper oxide and remove the water formed by passing the gas through phosphorus pentoxide before using it in the combustion furnace. The total water obtained in the determination was corrected for that amount due to the oxidation of the hydrogen from the ammonia. This amount of hydrogen was calculated from the results of the ammonia determination already described.

TABLE I

RESULTS OF ANALYSES

For $3(NH_4)_2O \cdot 2TeO_3 \cdot 6MoO_3 \cdot 10H_2O$			For $3(NH_4)_2O \cdot TeO_3 \cdot 6MoO_3 \cdot 7H_2O$		
	Calcd.	Found		Calcd.	Found
TeO_3	22.62	22.61	TeO_3	13.28	13.20
MoO_3	55.70	55.38	MoO_3	65.36	65.20
$(NH_4)_2O$	10.07	10.18	$(NH_4)_2O$	11.82	12.02
H_2O	11.61	11.75	H_2O	9.54	9.49
	100.00	99.92		100.00	99.91

Properties

Both compounds decompose upon heating at 550° to give a residue containing tellurium dioxide and a small amount of molybdic oxide. This does not constitute a method for the separation of the two oxides since the removal of the molybdic oxide is generally incomplete. If alcohol is added to aqueous solutions of the compounds, finely divided crystals are thrown out of solution. Analysis shows these crystals to have exactly the same composition as those obtained from water solutions by evaporating and cooling. If the dry crystals are allowed to stand over fused calcium chloride in a desiccator, they disintegrate rapidly. The loss of water in the desiccator is not accompanied by the loss of ammonia. The original compounds are readily soluble in water and insoluble in alcohol or ether.

When the ditellurate was recrystallized from a solution containing a large excess of ammonium nitrate, the composition of the compound was unchanged. However, when an excess of ammonium hydroxide was added to the solution and the excess ammonia was driven off by evaporation until a clear solution was obtained, crystals were deposited which upon analysis were shown to have the composition of the monotellurate.

Although it was possible quantitatively to determine the molybdenum in the ammonium molybdotellurates by reduction with stannous chloride,[1] and comparison of the blue color with that obtained from a standard ammonium molybdate solution, it was not found possible to adapt this procedure to the determination of small amounts of tellurium. In the determination of phosphorus the stannous chloride reduces the molybdenum of the ammonium phosphomolybdate, producing the molybdenum blue, while the molybdenum of the excess ammonium molybdate reagent is unaffected. Under similar conditions for tellurium not only is the molybdenum of the molybdotellurate reduced but also the molybdenum of the ammonium molybdate reagent and the tellurium originally present as tellurate.

Summary

Two new ammonium molybdotellurates have been isolated. The analyses of the compounds have been described and formulas assigned on interpretation of these analyses. The formula for the monotellurate was given as $3(NH_4)_2O{\cdot}TeO_3{\cdot}6MoO_3{\cdot}7H_2O$, while that of the ditellurate was $3(NH_4)_2O{\cdot}2TeO_3{\cdot}6MoO_3{\cdot}10H_2O$.

Some of the general properties of the compounds have been described. It was mentioned that although molybdotellurates were shown to exist, it was not possible to adapt them to the determination of small amounts of tellurium in a procedure similar to that used for the determination of small amounts of phosphorus.

MADISON, WISCONSIN