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GAMMA SCALE CHEMISTRY PROGRESS REPORT

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GAMMA SCALE CHEMISTRY GROUP

M. Economides, E. Estabrook, E. F. Joy, and A. Martin

This report considers the work done during the year ending June 30, 1948, present work being done and future plans on the determination of formulas, methods of preparation, and properties of as many compounds of postum as possible. An experimental approach to such a research problem on the element postum requires that procedures which may be used deal with ultramicro quantities of material. Such procedures on an ultramicro or gamma scale require special technique by personnel trained in manipulating these small quantities of radioactive material. Equipment which may be used varies with the experiment considered. Often new apparatus must be developed or equipment previously developed and used in some other experiment must be modified. This generalized research problem is subdivided in the "Research Problems Outline". The presentation of a survey of these research problems with reference to the outline for the year ending June 30, 1948 is a critical review of the work done by the Gamma Scale Chemistry Group as well as a consideration of future plans. The course which these future plans may follow will depend upon information which may be obtained when carrying out planned experiments.

RESEARCH PROBLEMS OUTLINE

For Year Ending June 30, 1948.

I. OXIDES OF POSTUM

A. Quantitative Preparation of Dioxide, O_2

Completed experimental work reported in Information Meeting Paper No. 4, "Determination of the Formula of an Oxide of Postum", August 6, 1947, by Alonzo W. Martin.(1)

B. Preparation and Isolation of Oxides of Postum

Oxides of postum have been studied in their crystalline forms by means of X-ray diffraction powder patterns. Further work will be done on the dioxide, and on the preparation and separation of oxides other than the dioxide. Present methods of preparation of X-ray samples are not too good. Inadequate interpretation of X-ray diffraction powder photographs; also present facilities inadequate.

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C. Physical and Chemical Properties of Oxides

Some information on the yellow oxide has been obtained; practically none on other possible oxides. This problem should be thoroughly investigated.

D. Rate of Reaction

A study is being made of the effect of temperature and gaseous pressure, with special attention to the effect of room temperature, on the rate of combination of moisture free oxygen gas and metallic postum. Some information is available; however, these data are incomplete and not too precise. This problem requires a thermostatically controlled system. Such a system is being considered when installing equipment at the Mound Laboratory. Information obtained on this rate of reaction problem will be of interest to production processing.

E. Qualitative and Quantitative Studies in the Preparation of Compounds Which May Be Formed When the Oxide of Postum Reacts With Substances Listed Below

1. Reactions Already Attempted

- a. Water vapor
- b. Chlorine, hydrogen chloride, and carbon tetrachloride
- c. Hydrogen gas
- d. Helium gas
- e. Helium gas saturated with water vapor

These studies are incomplete; further investigation is contemplated.

2. Suggested Reactions not as yet Attempted

- a. Bromine and hydrogen bromide
- b. Nitrogen gas
- c. Oxides of Nitrogen
- d. Sulfur and oxides of sulfur

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e. Hydrogen sulfide

f. Ammonia

These studies are planned for future investigation. Unless personnel becomes available, such experiments will not be attempted in the near future.

F. Vapor Pressure Measurements on the Yellow Dioxide of Postum

This problem is suggested for the Physics Group in connection with their work on vapor pressure measurements of postum.

II. DENSITY OF POSTUM

An approximate value for the density of metallic postum has been obtained. "Density of Metallic Postum", Ad Interim Report, May 18, 1948, by B. Brody.⁽⁷⁾ The two values obtained are not in quantitative agreement. The purity of the metallic postum used is in question. This work was interrupted because of a lack of personnel. Mr. Brody is to continue work on this problem during the summer months of 1948.

III. CHLORIDES OF POSTUM

Completed experimental work reported in Information Meeting Paper No. 5, "The Preparation and Properties of the Chlorides of Postum", August 11, 1947, by E. F. Joy.⁽⁵⁾

A. Preparation and Purification of Tetrachloride of Postum

This investigation has established quantitatively the compound QCl_4 . Some work needs to be done to complete this problem.

B. Preparation and Purification of Dichloride of Postum

Quantitative data have been obtained which established the formation of QCl_2 . Some work remains to be done in order to confirm these data.

C. Physical and Chemical Properties of Chlorides of Postum, X-Ray Diffraction Data on Chlorides of Postum

Considerable information has been obtained and reported,

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however, this work is not complete. A temporary transfer of personnel interrupted the work on this problem.

D. Dissociation Pressure Studies

This suggested investigation conforms to work being done by the Physics Group on vapor pressure measurements, and is therefore, a suggested problem for that Group.

IV. BROMIDES OF POSTUM

Such phases as preparation and purification with physical and chemical properties of the bromides of postum are being studied.

Preliminary work, Progress Report for May 1948,⁽⁹⁾ indicates the formation of postum bromides when bromine is reacted with postum. No comprehensive quantitative study has as yet been made. Personnel recently available has been assigned an investigation of this problem.

V. IODIDES OF POSTUM

This is a future problem considering possible compounds formed in reaction of iodine and postum. It has been indicated by L. E. Marchi, Final Report No. 38, "The Volatility of Polonium Iodide", March 1, 1945⁽¹⁰⁾, also Final Report No. 35, "The Polonium/Iodine Ratio",⁽¹¹⁾ that iodides of postum are possible of formation. After information on the bromides of postum becomes available and personnel to do this work is available the study of iodides of postum will be undertaken.

VI. FLUORIDES OF POSTUM

In view of the information on compounds of postum, it should be possible to prepare fluorides of postum. See "Polonium Fluorides" by C. L. Rollison and L. E. Marchi, November 13, 1944⁽¹²⁾ and "Proposed Procedure for the Study of the Polonium-Fluorine System" by L. V. Coulter and F. J. Leitz, December 13, 1944⁽¹⁴⁾ Interest in other preparations by present personnel has delayed any study or investigation of postum fluorides.

VII. POSTUM WITH METALS; e.g., ZINC, BARIUM, MERCURY, AND SODIUM

The isolation of compounds, determination of the physical and chemical properties of the compounds and X-ray diffraction studies are suggested. This investigation has been worked on; however, no conclusive results have been obtained. This problem has been temporarily laid aside because of a transfer of personnel. When personnel becomes available this problem is to be continued.

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VIII. PREPARATION OF POTASSIUM POSTUM BROMIDE

This problem was suggested by the analogous tellurium compound K_2TeBr_6 . Preliminary investigation shows some promise for the preparation of K_2QBr_6 . Such a compound containing a ratio by weight of one part of postum to 3.7 parts of compound might be quantitatively analyzed by weight determinations. The preparation of K_2QBr_6 is under investigation at the present time.

IX. NITRATE OF POSTUM

A white residue is obtained when the liquid phase is removed from a solution of nitric acid and postum. This white residue on ignition yields a yellow crystalline substance which is in appearance identical with the yellow dioxide of postum. Further investigation of this white residue is planned in the near future.

X. PREPARATION OF SOURCES AS REQUESTED

Frequently, specified preparations are requested. This is a service which will be continued.

XI. INVESTIGATION OF COUNTING TECHNIQUE

Considerable time and experimental work has gone into a thorough investigation of discrepancies in counting technique. A forthcoming paper on a phase of the work completed will appear in Information Report No. 1 on Counting Technique by M. Economides. This work will be continued.

XII. PREPARATION OF TELLURIUM COMPOUNDS

These preparations are for the purpose of obtaining information in preparing analogous postum compounds.

A. Preparation and Quantitative Analysis of Tellurium Nitrate

One phase of this work has been completed. More experimental work remains to be done.

B. Solubility of Tellurium Nitrate

Planning of the experimental work on this problem depends on the outcome of C below, a rapid titration method for quantitative determination of tellurium.

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C. Titration of Telluric Acid with Titanium Trichloride

An attempt is being made to use this oxidation-reduction titration method on a micro-scale as an accurate quantitative measure of tellurium in solution. Work is under way at the present time and is to be continued.

DISTRIBUTION RESEARCH PROBLEMS

Refer to "Research Problem Outline", for year ending June 30, 1948.

Considered as Complete, Further Experimental Work Suggested	Problems Being Investigated at the Present Time	Problems Which Would be Active if Facilities Were Available	Problems Which Would Be Assigned if Personnel Were Available	Problems Suggested for Another Research Group
I A	IV	I B	II (Personnel indicated for summer 1948)	I F
I Elc	VIII	I C		III D
I Eld	IX		V	
I Ele	X		VI	
III A	XI		VII	
III B	XII C		I B	
III C			I C	
XI A			I Ela	
			I Elb	
			I E2a	
			I E2b	
			I E2c	
			I E2d	
			I E2e	
			I E2f	

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SURVEY OF RESEARCH PROBLEMS

For Year Ending June 30, 1948.

Numbers and letters underlined below refer to "Research Problems Outline". Progress Reports already submitted for the year contain the detailed experimental information.

I A

Research studies for new compounds of postum and the physical and chemical properties of these compounds led to the preparation of an oxide of postum by direct combination of moisture free oxygen gas and metallic postum. A Bourdon gauge apparatus was designed and constructed for this oxide preparation.⁽¹⁾ Quantitative experiments used in the preparation of this oxide of postum established the formula of the yellow dioxide of postum as QO_2 , thus an oxidation state of plus four for postum. It was necessary in this preparation to devise a procedure for purification of metallic postum. This purification procedure has been found useful for other preparations involving reactions with metallic postum. The apparatus and procedure used to determine the formula of the yellow oxide of postum might also be used for the preparation of other compounds of postum with gaseous reactants. This completed work on the "Determination of the Formula of an Oxide of Postum" was presented in Information Meeting Paper No. 4, August 6, 1947, by Alonzo W. Martin.⁽¹⁾

I B

In order to extend the information on the properties of this yellow dioxide of postum, preparation of X-ray capillaries containing samples of the yellow oxide crystals was undertaken. Considerable difficulty was encountered in preparation of samples suitable for X-ray powder diffraction studies. X-ray diffraction photographs however, were taken on three different samples of the yellow dioxide preparations. Complete interpretation of these photographs has not been made. X-ray diffraction studies on the yellow oxide of postum indicates a conversion from the one crystalline form of a freshly prepared yellow oxide of postum to another crystalline form upon standing at room temperature. This conversion is noticeable two weeks after preparation of this yellow oxide of postum. Information such as the density of the crystals and an X-ray pattern of a single crystal is needed for interpretation of the X-ray diffraction powder pattern. This work on the crystal structure of the dioxide crystals needs careful study, both in interpretation of the photographs already obtained and in improvement of the procedure for preparation of X-ray samples.

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Note: During preparation of this report, and after writing the material included in the above paragraph, a memorandum on a "Conference with Dr. Zachariassen" written by H. P. Knauss(2) was received. Topics 3 and 4 of this memorandum are of particular significance.

"3. The diffraction patterns of the oxide, in comparison with tellurium dioxide, show the compound undoubtedly is the dioxide. Comparison with a superimposed sodium chloride pattern shows that the spacing is about $5.60 \text{ \AA}$. The disappearance of patterns after several weeks is not too surprising, since plutonium trichloride crystals go to pieces in some months."

"4. Thorium could be used as a "stand in" because its size is the same, i. e., $5.59 \text{ \AA}$. Comparison with tellurium dioxide was also suggested."

I C

The information furnished from the X-ray diffraction powder photographs on the yellow oxide of postum indicated the possible formation of, or the presence of more than one crystalline phase. It was reasonable to believe that there was present more than one oxide corresponding to more than one oxidation state of postum in the prepared samples. An investigation of a means of formation, or of separation of the different crystalline phases of these oxides of postum was undertaken.(3) Volatilization studies on the yellow dioxide of postum under varying amounts of oxygen gas present gave results which indicated the yellow form of the oxide is present only in an excess of moisture free oxygen. This yellow oxide volatilizes and recondenses at a furnace temperature of about 670°C . to 700°C . A second phase of orange-red crystals will form in the absence of an excess of oxygen gas, and this phase volatilizes and recondenses at a furnace temperature in the neighborhood of 800°C . In these volatilization studies, at a furnace temperature above 800°C ., there appeared a metallic deposit identical in appearance to metallic postum. These volatilization experiments attempting a separation of the oxides were not carried to completion as planned because of the cracking of the quartz volatilization tube caused by the activity present. No diffraction photographs could be made of the orange-red crystals. This work needs further investigation; however, some study as to the procedure to be followed and the material to be used for containing the X-ray samples of the active material should be made.

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I D

Interest in the experimental information obtained in the preparation of the dioxide of postum when oxygen combines with postum (1) resulted in a discussion with personnel from production process. The data available from these experiments were useful but were not complete. Data are needed on the rate at which oxygen reacts with postum at various temperatures. The temperature range of most interest is room temperature. In order to carry out such experiments and obtain reliable data, it will be necessary to modify the present apparatus so as to have a constant and known temperature of the gas in the system. A thermostatically controlled system is planned for installation at the Mound Laboratory.

I E1a

When a closed tube containing the yellow dioxide of postum, CO_2 , in an excess of dry oxygen gas is opened to the atmosphere a rapid change takes place, the yellow crystalline dioxide going to a white crystalline substance. Experiments using oxygen gas saturated with water vapor, at room temperature, reacting with metallic postum were run. These experiments followed essentially the same procedure as was used in preparations of the yellow dioxide of postum with moisture free oxygen.⁽¹⁾ Interpretation of these data quantitatively was not practicable because of the presence of too many unknown factors. There was, however, conclusive evidence that the dioxide of postum is formed, and that this dioxide readily reacts with water vapor as well as taking up water. Therefore, it was postulated that the yellow dioxide of postum forms a hydrated oxide with water, the formula of this hydrate not being determinable from the data of these experiments. Further experimental work on the reaction of water and the dioxide of postum is planned.

I E1b

Some of the earlier work of T. J. LaChapelle, et. al.⁽⁴⁾, on the chloride of postum made use of a procedure whereby a residue of postum which, interpreted from the information now known was probably the dioxide of postum. There is added experimental evidence that the dioxide of postum when treated with hydrogen chloride yields a mixture of di- and tetra chlorides of postum.⁽⁵⁾ Likewise, the dioxide of postum with carbon tetrachloride plus heat forms a mixture of di- and tetra chlorides of postum.⁽⁵⁾ No experiments have as yet been performed using chlorine gas with the dioxide directly. This study is incomplete. Further quantitative experimental data are needed for a complete interpretation of these reactions. Future plans include carrying out such experiments. At the present time, however, it is not desirable to

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contaminate the vacuum line now in use with chlorine.

I Elc

In the purification of metallic postum for use in preparing its compounds, one of the steps frequently used was to admit oxygen-free dry hydrogen gas to an evacuated tube containing metallic postum. Hydrogen gas under approximately one atmosphere of pressure in this tube containing the postum was heated by means of a tubular furnace at a temperature of 200 to 250°C. for several hours. The hydrogen gas was removed by evacuation, and the tube containing the postum sealed off from the vacuum system. Volatilization of the hydrogen treated metallic postum yields a metallic mirror uniform in appearance. This purification step with hydrogen indicates a possible reduction of the oxides of postum.

Quantitative measurements of the change in the total number of gaseous molecules present when oxygen-free dry hydrogen gas is confined in a Bourdon gauge apparatus and over metallic postum indicated no change in the number of gas molecules in the confined volume.

Experiments using a prepared sample of the yellow dioxide of postum with oxygen free dry hydrogen gas in a Bourdon gauge apparatus were run. These experiments, in which the total number of gaseous molecules confined in a definite volume within the system could be followed, indicated conversion of the dioxide to metal. Conclusive quantitative data were not obtained; however, this conversion of the yellow dioxide deposit indicated a reduction of the dioxide of postum to metallic postum. It may be advisable to repeat these experiments, but such repetition is not planned at the present time.

I El (d and e)

In order to confirm an observation, namely, the reaction already referred to, I Ela, that of oxide formation with water vapor, helium gas at atmospheric pressure was saturated at room temperature with water vapor and the gases confined in a definite volume with metallic postum.(6) A Bourdon gauge apparatus was used and the number of gaseous molecules present determined from pressure measurements. The metallic mirror of postum changed in appearance to a yellow residue similar to the yellow dioxide of postum. This yellow residue on standing changing to a white residue. There was no change in the total number of gaseous molecules until the appearance of the white residue. Explanation of the experimental data was on the basis of a reaction between water and postum to first form the dioxide of postum, the dioxide in turn taking up water to form a hydrated oxide. An experiment

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was run duplicating the above procedure, but substituting dry helium gas for the helium gas saturated with water vapor which was previously used. There was no observed reaction between dry helium gas and metallic postum. A repetition of these experiments should be undertaken.

I E2 (a,b,c,d,e, and f)

The search for possible reactions which would yield new compounds of postum suggests as possible reactions certain gases with metallic postum and with the dioxide of postum. Using the Bourdon gauge apparatus and a definite and known volume of gas and following the change in the number of molecules present from pressure measurements, it is planned to try the following gases both on metallic postum and with the prepared dioxide of postum: bromine, hydrogen bromide, nitrogen, oxides of nitrogen, sulfur and oxides of sulfur, hydrogen sulfide, and ammonia.

I F

Experiments dealing with the preparation and properties of the yellow dioxide of postum suggests the existence of an equilibrium between two forms of the oxide, namely, a monoxide and a dioxide--the dioxide existing only in the presence of an excess dry oxygen. There is an added possibility of an equilibrium between the dioxide of postum, monoxide of postum, metallic postum and oxygen. Other oxides of postum may be present. Information on dissociation pressure of the oxide is highly desirable. Vapor pressure measurements on a prepared yellow oxide of postum should be attempted. This problem is suggested for the Physics Section in connection with their work on vapor pressure measurements of postum.

II

An accurate value for the density of metallic postum is needed. This valuable physical constant, if accurately known, would aid in the interpretation of other physical data which have already been obtained on this element. An approximate value for the density of metallic postum has been obtained.(7) The values experimentally measured to date do not represent a quantitative check. Mr. E. B. Brody gave considerable attention to this problem when working with the research staff. No work has been done on this density problem for several months due to a lack of trained personnel. It is planned to have Mr. Brody continue the work of this problem during the summer months of 1948.

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III (A, B, and C)

The earlier work of B. M. Abraham, B. B. Brody, J. J. Howland, L. W. Niedrach, A. W. Martin, and T. J. LaChapelle was summarized in Report No. 31, "Preliminary Work on Oxidation States of Postum and Preparation of Some of Its Compounds", by T. J. LaChapelle, et. al.(4) A continuation of this problem has resulted in the preparation of the two chlorides of postum. A detailed report on "The Preparation and Properties of the Chlorides of Postum", by E. F. Joy, August 11, 1947, Information Meeting Paper No. 5,(5) summarizes the work on postum chlorides. Additional quantitative experimental data have been obtained since presentation of this Information Paper(5), establishing definite valence states of 2 and 4 for postum in the lower and higher chlorides respectively. There remains some suggested experimental work to complete the information on the postum chlorides.

Note: Reference is made in a memorandum(2), by H. P. Knauss on a "Conference with Dr. Zachariasen", June 16, 1948, and in particular to No. 7 of this memorandum, "Dr. Zachariasen suggests that we study the crystal structure of the halides." Several X-ray diffraction powder photographs on both the yellow tetrachloride and red dichloride of postum have been made. Data and interpretation of these X-ray diffraction patterns which have been reported by D. H. Reynolds(8) are also included in the Information Meeting Paper.(5)

III D

A similar statement as given under I F, namely, the need of vapor pressure data on the chlorides of postum is applicable here. This is a problem suggested for the Physics Section in connection with their vapor pressure measurements on postum.

IV

Preparation of what should be bromides of postum by reacting bromine with metallic postum was reported in the Progress Report of this group for May 1-30, 1948.(9) Indications are that the same experimental procedures used in the problem with postum chlorides should be applicable in the case of the postum bromides. This problem has been assigned to E. F. Joy.

V

Another possible halide of postum is the halide which might be formed with iodine and postum. Reference is made to earlier attempted

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preparation of postum iodide.(10)(11) This is a future problem and it is suggested that it follow the experimental work on postum bromides with assignment to the same personnel if possible.

VI

In order to complete an investigation of all the halogens which may combine with postum to form postum halides, preparation of the fluorides of postum should be attempted. Some earlier attempts to prepare and isolate compounds of fluorine and postum are not conclusive.(12)(13)(14) An investigation of these fluorides would require the use of apparatus other than quartz or glass. Present procedures in the preparation of compounds of the halogens and postum make use of a vacuum system of pyrex glass, pyrex, or quartz volatilization tubes, microscopic examination of samples in pyrex or quartz tubes and preparation of X-ray samples in pyrex or quartz capillaries. A careful study of the problem of preparation of fluorides of postum and the apparatus which may be used in such preparations is planned if and when trained personnel becomes available.

VII

Considerable experimental work has gone into an attempted preparation of some compound of postum and some other element in which postum is present with a negative valence. Among the more positive elements tried were zinc and barium. Zinc and postum yielded a crystalline product. X-ray analysis showed these crystals to be in the cubic system. Procedures for chemical analysis of the zinc and postum residue were worked on but not carried to completion. An attempted preparation of compound formation of barium and postum was tried. A residue was obtained but there has been no attempted analysis of this residue for barium. Additional experimental work is needed to confirm the results obtained. Personnel working on this program was assigned to other duties. At present, there is no personnel available for this program.

VIII

Information available on the dioxide and chlorides of postum indicates that their preparation and separation into a single pure compound is difficult. Apparently in an oxide preparation, the yellow dioxide of postum exists only in an excess of moisture free oxygen gas and even under these conditions is unstable. Likewise, the yellow tetrachloride of postum exists only in the presence of an excess of dry chlorine. Separation of the dioxide, tetrachloride, or dichloride

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in a pure form has not been entirely successful. The preparation of a compound of postum in a pure form would be desirable. Preparation of a compound, such as K_2QBr_6 analogous to K_2TeBr_6 has been attempted. Indications are that such a compound may have been prepared.(9)

Preparation of potassium postum bromide, K_2QBr_6 , would be a compound in which the ratio by weight for postum, 210, to the total molecular weight, 768, would be desirable in a quantitative determination. Experimental investigation on the preparation of K_2QBr_6 is being worked on at the present time.

IX

In the one attempted preparation of potassium postum bromide, a nitric acid postum residue was ignited to produce a yellow residue which is probably the yellow dioxide of postum. This white residue was obtained by evaporation of the liquid phase from a solution of postum in nitric acid. The formula of this white residue from postum in nitric acid is not known. Preparation and analysis of this white residue, an intermediate in the preparation of potassium postum bromide, is to be investigated and will receive a priority over the preparation of potassium postum bromide.

X

A survey of the work being done in and planned for the Gamma Scale Chemistry Group includes that work being done by M. Economides on preparation of sources as requested. Since these requests vary as to specifications and the amount of time required to make up such preparations, a definite statement as to future requirements cannot be made. These requests receive a priority depending on the urgency of preparation. Included in the work being done by M. Economides, is a service as expeditor for postum and precious metals for Unit 3.

XI

A thorough investigation on counting technique has been undertaken in order to determine the cause of discrepancies occurring between calorimetric standards and slide counting with instruments now in use. One phase of this investigation involves the various aspects of slide preparation. A large amount of statistical experimental data has accumulated and is being collected for presentation

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in Information Report No. 1 on Counting Technique. This investigation is being continued.

XII (A, B, and C)

As a starting point for information in the preparation and the determination of the properties of possible compounds of postum the analogy to tellurium and tellurium compounds is of interest. Since first hand information on tellurium compounds is desirable, personnel has been assigned the investigation of tellurium compounds related to the postum compounds which are under consideration by the Gamma Scale Chemistry Group. The nitrate of tellurium is being studied at the present time. This problem concerns a rapid oxidation-reduction micro-titration method as an accurate means of quantitatively determining the tellurium content of a solution. Such a micro-titration is to be used in connection with solubility determinations of the nitrate of tellurium. This work will be continued.

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