

THE PREPARATION AND IDENTIFICATION
OF RUBIDIUM TELLURO-MOLYBDATE AND
OF CESIUM TELLURO-MOLYBDATE

SEP 27 1938

THE PREPARATION AND IDENTIFICATION OF
RUBIDIUM TELLURO-MOLYBDATE AND OF
CESIUM TELLURO-MOLYBDATE

By

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Bachelor of Science

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Springfield, Missouri

1936

Submitted to the Department of Chemistry
Oklahoma Agricultural and Mechanical College
In Partial Fulfillment of the Requirements
For the degree of
MASTER OF SCIENCE

1938

RECEIVED
AGRICULTURAL EXPERIMENT STATION
SEP 27 1938

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ACKNOWLEDGMENT

The author wishes to acknowledge the valuable advice and assistance of Dr. Sylvan R. Wood, under whose direction this work was done.

Acknowledgment is also made of the many helpful suggestions and cordial cooperation of Dr. H. M. Trimble.

The author wishes to express his sincere appreciation to the Oklahoma Agricultural and Mechanical College for financial assistance in the form of a graduate assistantship in the Department of Chemistry during the school years 1936-37 and 1937-38.

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THE PREPARATION AND IDENTIFICATION OF RUBIDIUM TELLURO-MOLYBDATE AND
OF CESIUM TELLURO-MOLYBDATE

I. INTRODUCTION

The preparation and study of the complex salts of molybdenum and tellurium are comparatively recent developments in inorganic chemistry. Complex salts of molybdenum and tellurium were first prepared in 1929 at the University of Wisconsin.

Woodstock and Meloche (1), of the University of Wisconsin, prepared and studied two complex salts of ammonium telluro-molybdates. These salts were prepared by vaporization and crystallization of aqueous solutions containing varying mole quantities of the reagents. They determined the empirical formula for the ditellurate to be $3(\text{NH}_4)_2\text{O} \cdot 2\text{TeO}_3 \cdot 6\text{MoO}_3 \cdot 10 \text{H}_2\text{O}$, and for the monotellurate to be $3(\text{NH}_4)_2\text{O} \cdot \text{TeO}_3 \cdot 6\text{MoO}_3 \cdot 7 \text{H}_2\text{O}$.

According to J. B. Dyess (2), the composition of lithium telluro-molybdate was found to correspond to the molecular formula $3\text{Li}_2\text{O} \cdot \text{TeO}_3 \cdot 6\text{MoO}_3 \cdot 13 \text{H}_2\text{O}$.

S. M. Thomsen (3) described the preparation of sodium telluro-molybdate, which has the empirical formula $3\text{Na}_2\text{O} \cdot \text{TeO}_3 \cdot 6\text{MoO}_3 \cdot 22 \text{H}_2\text{O}$, as determined by Thomsen and Wood (4).

H. F. Hansen (5) described the preparation of the potassium salt of the telluro-molybdate. He determined the empirical formula to be $3\text{K}_2\text{O} \cdot \text{TeO}_3 \cdot 6\text{MoO}_3 \cdot 8 \text{H}_2\text{O}$.

Since the telluro-molybdates of three alkali metals and of ammonium have been prepared and found to form a homologous series, with the general formula $3\text{M}_2\text{O} \cdot \text{TeO}_3 \cdot 6\text{MoO}_3 \cdot X \text{H}_2\text{O}$, it is thought that the other

two alkali metals, namely, rubidium and cesium, would form compounds with the same general formulas.

The purpose of this work is to prepare the rubidium and the cesium salts of the telluro-molybdate complex, to determine their empirical formulas, and thus to complete the alkali series of the telluro-molybdates.

II. MATERIALS USED

The reagents used in the preparation of rubidium telluro-molybdate and of cesium telluro-molybdate were of the purest grade obtainable.

MOLYBDIC ACID - Baker's Analyzed molybdic anhydride of 99.9% purity was used.

TELLURIC ACID - Pure telluric acid was prepared from metallic tellurium by a modification of the Staudenmeyer method (6). The tellurium is pulverized and heated in a casserole with repeated small portions of dilute nitric acid until oxidation of the tellurium is complete. This residue was taken up with a small amount of concentrated hydrochloric acid, and fumed to drive off all nitrates, and to convert the tellurium to the chloride. This was then dissolved in concentrated hydrochloric acid to form a solution of TeCl_4 . The latter is poured into a large volume of water, whereupon hydrolysis of the TeCl_4 causes a very voluminous precipitate of hydrated TeO_2 to form. Practically all the tellurium is thus deposited. On standing for several hours, the bulky, flocculent precipitate undergoes syneresis to yield a white, granular powder. The liquor is then decanted and the precipitate washed several times by decantation. To insure the purest grade of telluric acid, the TeO_2 thus obtained is dissolved in dilute hydrochloric acid, and the tellurium precipitated by means of 10% hydrazine hydrate and saturated sulfur dioxide solution (7).

The acidity should not be over 10% to insure complete precipitation of the tellurium. The mixture is boiled to coagulate the black colloidal tellurium which first precipitates. The latter is then separated from the liquid by decantation, and the tellurium washed. It is then oxidized

with nitric acid, repeating the previous process to prepare the tellurium dioxide. The latter is dissolved in concentrated hydrochloric acid. Very pure tellurium dioxide is then produced by hydrolysis of the resulting TeCl_4 solution. The TeO_2 is then separated and washed, dissolved in hot dilute nitric acid, and treated with a hot saturated solution of potassium dichromate in concentrated nitric acid. It is undesirable to add more than sufficient dichromate to fully oxidize the tellurous acid to telluric acid. The use of a spot-plate and careful observance of the color of the liquor will indicate when a slight excess of dichromate is present. The hot solution is then set aside to cool, whereupon sparingly soluble telluric acid crystals are deposited. The mother liquor is decanted off and the crystals drained. They are then washed with alcohol to remove remaining liquor, and dissolved in the minimum amount of hot water. On cooling, white crystals of telluric acid, H_6TeO_6 , are deposited. Six recrystallizations were carried out to insure a very pure product. Further crops are obtained by concentration of the acid liquor, and from solutions used in recrystallization. Analysis of the acid thus prepared gave the following results: (in terms of % Te)

Sample 1: 55.46% Te	Sample 2: 55.41% Te
Calculated % Te in H_6TeO_6 : 55.54%	

CESIUM NITRATE - Very pure cesium nitrate was obtained from the University of Wisconsin.

RUBIDIUM HYDROXIDE - The rubidium hydroxide was prepared by treating pure rubidium chloride, obtained from the University of Wisconsin, with the stoichiometrically calculated amount of pure silver oxide. The precipitated silver chloride was filtered off, leaving rubidium hydroxide in solution.

III. PREPARATION OF RUBIDIUM TELLURO-MOLYBDATE

In the preparation of this salt, the reagents are dissolved in water in the ratio of 0.06 moles of rubidium hydroxide to 0.01 moles of pure telluric acid to 0.06 moles of molybdic trioxide. The following procedure is very satisfactory.

6.15 grams of rubidium hydroxide in about 200 ml. of water was heated to boiling. To the hot alkali solution is added 1.76 grams of pure telluric acid. When this has dissolved, add with stirring, successive small portions of pure molybdic trioxide, until a total of 8.64 grams have been added. The hot solution was filtered as soon as the reaction was complete in order to separate any of the unreacted molybdic oxide. The clear solution was then evaporated to a small volume on a water bath. On cooling, colorless crystals of rubidium telluro-molybdate appeared.

Recrystallization of the salt is readily carried out, as its solubility in water increases rapidly with temperature. The salt was purified by recrystallization from water six times.

In appearance, the salt is composed of crystals of very definite and characteristic appearance. The crystals in the pure state are colorless, hexagonal rhombohedrons as shown in the pictures, which were taken of the freshly prepared salt.



Figure 1. Crystals of Rubidium Telluro-molybdate (100 X)

IV. METHODS OF ANALYSIS

In the analysis of rubidium, tellurium and molybdenum, samples were used in which fresh hydrated salt was pulverized and all water expelled by heating over night at 110° C. To gain familiarity with the technique involved, preliminary analyses were carried out on compounds containing tellurium and molybdenum before engaging in the actual analysis of the salt, choosing substances of known composition for the purpose. This work was carried out on pure samples of telluric acid and molybdic acid until satisfactory results were obtained. Several preliminary analyses were then carried out on the complex salt to ascertain whether analysis of the combined constituents offered any undue complications. Some difficulty was experienced in the molybdenum analysis, but this was overcome by fuming with concentrated sulfuric acid for a longer period of time, to be sure that all reducing agents had been removed.

TELLURIUM - The method of Lenher and Homberger (7, 8) was used. Samples of 0.5 grams were taken for analysis. These were refluxed for three hours with 100 ml. of 6 N hydrochloric acid, in order to reduce the tellurium to the quadrivalent state.

The solution is heated to boiling, 15 ml. of a saturated solution of sulfur dioxide is added, then 10 ml. of a ten per cent solution of hydrazine hydrate followed by 25 ml. of sulfur dioxide solution. Dilute to 10 per cent acidity.

Tellurium begins to precipitate in the elementary form as a more or less colloidal black deposit. The boiling is continued until the precipitate settles in such a way that it can be easily washed, which requires about five minutes. The precipitated tellurium is brought on a previously

weighed Gooch crucible, washed with hot water until all the chlorides are removed, after which the water is displaced by alcohol, and the crucibles and contents dried to constant weight at 110° C.

The percentage of tellurium trioxide in the sample is then calculated from the following expression:

$$\text{Per cent of TeO}_3 = \frac{\text{wt. of ppt. Te X chem. factor } \frac{\text{TeO}_3}{\text{Te}} \text{ X 100}}{\text{Wt. of Sample}}$$

Table I gives the results of the analysis of tellurium found in the salt.

TABLE I

Determination of Tellurium Trioxide in
Rubidium Telluro-Molybdate

	No. 1	No. 2	No. 3	No. 4
Weight sample and tube	25.3824	24.9268	24.4450	23.9015
	24.9268	24.4450	23.9015	23.4433
Weight sample	0.4556	0.4818	0.5435	0.4582
Weight crucible and tellurium	15.2439	17.4907	19.3317	18.7793
Constant weights	15.2430	17.4907	19.3313	18.7793
	15.2429		19.3313	
Weight crucible	15.2072	17.4526	19.2912	18.7432
Constant weight	15.2072	17.4526	19.2889	18.7432
			19.2888	
Weight tellurium	0.0357	0.0381	0.0425	0.0361
Log weight tellurium	8.5527	8.5809	8.6284	8.5575
Log factor $\frac{\text{TeO}_3}{\text{Te}} \text{ X 100}$	2.1387	2.1387	2.1387	2.1387
Colog sample	0.3414	0.3172	0.2648	0.3389
	1.0328	1.0368	1.0324	1.0351
TeO ₃ in Sample Number	10.78%	10.88%	10.76%	10.84%
Average				10.82%

MOLYBDENUM - The amount of molybdenum was determined by indirect volumetric methods, making use of the Jones Reductor (9, 10).

The filtrate and washings from the tellurium analysis is evaporated to dryness on a hot plate and fumed with 10 ml. of concentrated sulfuric acid, in order to convert the molybdenum from the chloride to the sulfate, and to remove any reducing agents, which might be present. The fuming was continued for at least an hour, after which the residue was put into solution with water and diluted to 150 ml.

The receiving flask of the Jones Reductor is charged with about 30 ml. of 10 per cent ferric alum and 4 ml. of 85 per cent phosphoric acid. The ferric alum solution contained 100 grams of ferric ammonium sulfate, 25 ml. of concentrated sulfuric acid, and 40 ml. of 85 per cent phosphoric acid in a liter of solution.

Through the 40 cm. of amalgamated zinc in the reductor are passed in succession, 100 ml. of 2.5 per cent sulfuric acid, the molybdenum solution, followed by 250 ml. of 2.5 per cent sulfuric acid and 100 ml. of water, to wash out the remaining molybdenum.

The reduced green molybdic acid upon coming in contact with the ferric alum solution produces a bright red color. The entire solution is titrated with standard potassium permanganate solution. The potassium permanganate approximately N/10 was standardized against a standard molybdic acid solution.

Blanks were run until two successive tests required less than 0.2 ml. of 0.1 N potassium permanganate to give a pink color to the acid solution. In running a blank, 100 ml. of 1.5 N sulfuric acid was substituted for the molybdenum solution.

The molybdenum is reduced completely to the trivalent condition by the amalgamated zinc. The ferric alum solution does not change the reduced molybdenum entirely back to the 6-valent condition, but it does oxidize it sufficiently to prevent the effect of the atmosphere, and for every equivalent of molybdenum oxidized, an equivalent of reduced iron is formed, so that the solution titrates exactly as if a perfect reduction was obtained without the use of the ferric salt.

The percentage of MoO_3 in the sample is then calculated from the following expression:

$$\text{Per cent of MoO}_3 = \frac{\text{ml. of KMnO}_4 \times \frac{0.0144}{3} \times \frac{N}{0.1} \times 100}{\text{Sample}}$$

In this equation the factor $\frac{0.0144}{3}$ is the number of grams of MoO_3 equivalent to one ml. of $N/10 \text{ KMnO}_4$ solution.

The results of the determination are given in Table II.

TABLE II
Results of the Molybdenum Trioxide
Determinations

Sample No.	Weight of Sample in grams	Ml. 0.1163 N KMnO_4 used	Molybdenum Trioxide %
1	0.4556	44.14	54.08
2	0.4818	46.52	53.92
3	0.5435	52.46	53.88
4	0.4582	44.10	53.71
Average			53.90

RUBIDIUM - 0.5 gram samples were taken for the determination of rubidium in rubidium telluro-molybdate. The sample was placed in a previously weighed porcelain boat, and the boat placed inside a combustion tube. It was then heated at 500° C. for several hours in a stream of dry hydrogen chloride gas. The temperature of the reaction was observed by the use of a thermo-electric pyrometer.

Tellurium and molybdenum are thus converted to chlorides, which are volatile at 500° C., and sublime away to the cooler portions of the tube, leaving only rubidium chloride in the boat. At the beginning of the reaction, the sample became yellow in color, but as the molybdenum and tellurium chlorides passed off, the sample whitened, until finally, when only the rubidium chloride remained, the sample was completely white.

The rubidium chloride was weighed, and the per cent of rubidium oxide was calculated as follows:

$$\text{Per cent of Rb}_2\text{O} = \frac{\text{wt. RbCl} \times \text{Chem. Factor} \frac{\text{Rb}_2\text{O}}{2\text{RbCl}} \times 100}{\text{Sample}}$$

Table III presents the results of the analysis.

TABLE III
Results of the Rubidium Oxide Determinations

Sample No.	Weight in grams			RbCl	Rubidium Oxide per cent
	Sample	Container	Container and RbCl		
1	0.5778	3.4965	3.7578	0.2613	34.93
2	0.6438	3.4789	3.7711	0.2922	35.09
3	0.4904	3.4965	3.7178	0.2213	34.87
4	0.5480	3.4789	3.7264	0.2475	34.90
Average					34.95

WATER OF HYDRATION - 1-2 gram portions of fresh hydrated salt are removed from the mother liquor. Small crystals of 1-2 mm. size are chosen. Adhering liquor is removed by absorption on filter paper. The crystals are then transferred to a dry filter paper placed in a small Buchner funnel fitted to a suction flask. Using slight suction, the crystals are washed four to five times with about 5 ml. portions of absolute alcohol, to remove the last traces of mother liquor. Excessive washing is to be avoided. Adhering alcohol is then removed by washing with three or four 5 ml. portions of ether, which has been dried over sodium. Allow to air dry for several minutes with suction to remove the ether. The crystals are then transferred to a tared weighing bottle and weighed. Dry for at least ten hours, or over night in the oven at 110° C. to constant weight. Cool in desiccator and weigh at once.

$$\text{Per cent of water} = \frac{\text{loss in weight of sample}}{\text{weight of sample}} \times 100$$

Table IV gives the results of the water of hydration determinations.

TABLE IV

Results of the Water of Hydration Determinations
of Rubidium Salt

Sample No.	Weight in grams			Water	Water Per cent
	Sample	Hydrated Sample and Container	Dried Sample and Container		
1	1.8968	21.4758	21.3583	0.1175	6.19
2	1.2215	20.3640	20.2857	0.0783	6.41
3	1.4298	20.6554	20.5682	0.0872	6.20
4	1.2553	20.9242	20.8445	0.0797	6.35
Average					6.26

V. CALCULATION OF FORMULA

After having determined the percentage composition of the compound, the empirical formula was calculated from the means of the percentages of the several constituents.

The percentage composition was converted to the wet basis as follows:

$$\text{Per cent (wet basis)} = \frac{\text{per cent (dry basis)} \times (100 - \text{per cent water})}{100}$$

Table V gives the results of the calculations.

TABLE V

Calculation of Mole Ratios of the Constituents
in Rubidium Telluro-molybdate

Constituents	Quantity present dry basis per cent	Quantity present wet basis per cent	Mole Ratio	Moles Present
TeO ₃	10.82	10.15	0.05795	1
Rb ₂ O	34.95	32.77	0.1753	3
MoO ₃	53.90	50.53	0.3509	6
H ₂ O		6.26	0.3478	6

Results of the above analyses indicate that the empirical formula of the compound is: 3Rb₂O·TeO₃·6MoO₃·6 H₂O.

Results of the analyses are shown below in comparison with the calculated percentages based on the formula, 3Rb₂O·TeO₃·6MoO₃·6 H₂O.

TABLE VI

Constituents	Moles	Quantity Calculated per cent	Quantity Found per cent
TeO ₃	1	10.27	10.15
Rb ₂ O	3	32.82	32.77
MoO ₃	6	50.58	50.53
H ₂ O	6	6.33	6.26
Total		100.00	99.71

VI. PREPARATION OF CESIUM TELLURO-MOLYBDATE

The preparation of cesium telluro-molybdate was carried out in substantially the same manner as that of the rubidium salt. The reagents are dissolved in water in the ratio of 0.06 mole of cesium nitrate, 0.01 mole of pure telluric acid, and 0.06 mole of molybdic trioxide.

Cesium telluro-molybdate is obtained in well-defined crystals whose general appearance is similar to that of the rubidium salt, although they tend to crystallize out as somewhat larger crystals. The crystals are colorless. The pictures of the crystals were taken of the freshly prepared salt.

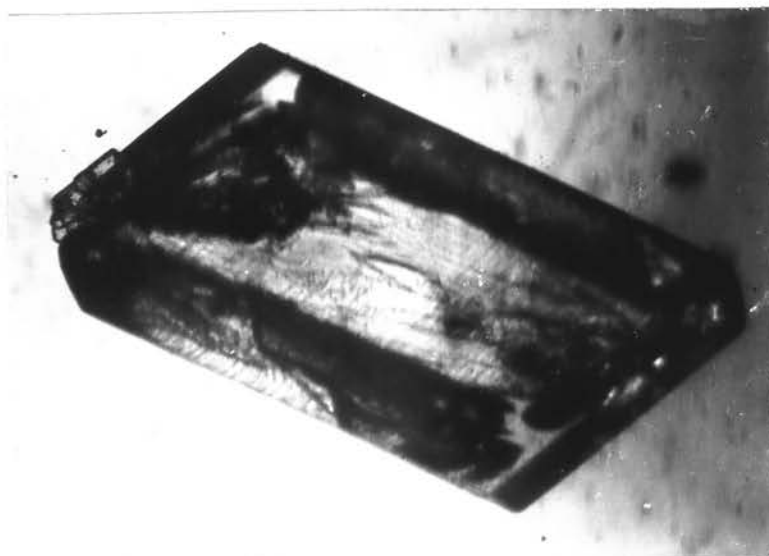


Figure 2. Crystals of Cesium Telluro-molybdate (100 X)

VII. RESULTS OF ANALYSIS

The analysis of cesium telluro-molybdate was carried out in identically the same manner as the rubidium salt.

TABLE VII

Determination of Tellurium Trioxide
in Cesium Telluro-molybdate

Sample No.	Weight in grams		Sample	Tellurium	TeO ₃ per cent
	Gooch Crucible	Gooch Crucible and Tellurium			
1	15.1448	15.1949	0.7536	0.0501	9.15
2	17.4321	17.4848	0.7832	0.0527	9.26
3	16.9228	16.9644	0.6268	0.0416	9.13
4	19.9286	19.9709	0.6317	0.0423	9.22
Average					9.19

TABLE VIII

Results of the Molybdenum Trioxide
Determinations

Sample No.	Weight of Sample in grams	Ml. 0.1163 N KMnO ₄ used	Molybdenum Trioxide %
1	0.4455	36.63	45.90
2	0.4582	37.52	45.69
3	0.6268	51.45	45.81
4	0.6317	51.74	45.72
Average			45.78

TABLE IX

Results of the Cesium Oxide Determinations

Sample No.	Weight in grams				Cesium Oxide per cent
	Sample	Container	Container and CsCl	CsCl	
1	0.7481	3.4789	3.8809	0.4020	44.96
2	0.6640	3.4965	3.8519	0.3554	44.79
3	0.4743	3.4789	3.7326	0.2537	44.76
4	0.4987	3.4965	3.7642	0.2677	44.92
Average					44.86

TABLE X

Results of the Water of Hydration Determinations of Cesium Salt

Sample No.	Weight in grams				Water per cent
	Sample	Hydrated Sample and Container	Dried Sample and Container	Water	
1	2.5818	20.5030	20.3388	0.1642	6.36
2	3.9966	24.4852	24.2394	0.2458	6.15
3	3.1018	21.4007	21.2087	0.1920	6.19
4	3.9979	24.1212	23.8689	0.2523	6.31
Average					6.25

VIII. CALCULATION OF FORMULA

TABLE XI

Calculation of Mole Ratios of the Constituents
in Cesium Telluro-molybdate

Constituents	Quantity present dry basis per cent	Quantity present wet basis per cent	Mole Ratio	Moles Present
TeO ₃	9.19	8.62	0.04912	1
Cs ₂ O	44.86	42.06	0.1494	3
MoO ₃	45.78	42.92	0.2981	6
H ₂ O	--	6.25	0.3472	7

Results of the above analysis indicate that the empirical formula of the compound is: $3\text{Cs}_2\text{O} \cdot \text{TeO}_3 \cdot 6\text{MoO}_3 \cdot 7 \text{H}_2\text{O}$.

Results of the analysis are shown below in comparison with the calculated percentages based on the formula, $3\text{Cs}_2\text{O} \cdot \text{TeO}_3 \cdot 6\text{MoO}_3 \cdot 7 \text{H}_2\text{O}$.

TABLE XII

Constituents	Moles	Quantity Calculated per cent	Quantity Found per cent
TeO ₃	1	8.73	8.62
Cs ₂ O	3	42.02	42.06
MoO ₃	6	42.98	42.92
H ₂ O	7	6.27	6.25
Total		100.00	99.85

IX. SUMMARY AND CONCLUSIONS

1. Pure rubidium telluro-molybdate has been prepared and subjected to complete chemical analysis.
2. Results of this analysis indicate that the empirical formula of the compound is: $3\text{Rb}_2\text{O}\cdot\text{TeO}_3\cdot 6\text{MoO}_3\cdot 6\text{H}_2\text{O}$.
3. Pure cesium telluro-molybdate has been prepared and subjected to complete chemical analysis.
4. Results of this analysis indicate that the empirical formula of the compound is: $3\text{Cs}_2\text{O}\cdot\text{TeO}_3\cdot 6\text{MoO}_3\cdot 7\text{H}_2\text{O}$.
5. The preparation of these two salts completes the alkali series of the telluro-molybdates.
6. Analyses of these two salts show that the telluro-molybdates of the alkali metals form a homologous series with the general formula, $3\text{M}_2\text{O}\cdot\text{TeO}_3\cdot 6\text{MoO}_3\cdot \text{X}\text{H}_2\text{O}$.

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XI. AUTOBIOGRAPHY

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I, Henry Arthur Carlson, was born on a farm in Spring River Township, near Verona, Missouri, County of Lawrence, on the fifth day of April, 1914. I received my elementary education at Verona, Missouri. My high school education was obtained in the public schools of Verona and Aurora, Missouri. In September, 1932, I enrolled in Monett Junior College, Monett, Missouri. After graduating from the Junior College, I enrolled in Drury College, Springfield, Missouri, September, 1934. I received my Bachelor of Science degree in chemistry from that institution in June, 1936.

In the fall of 1936, I enrolled in the Graduate School of the Oklahoma Agricultural and Mechanical College. I expect to complete the requirements for the degree of Master of Science in Chemistry in May, 1938.

1936
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M. S. IN CHEMISTRY

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