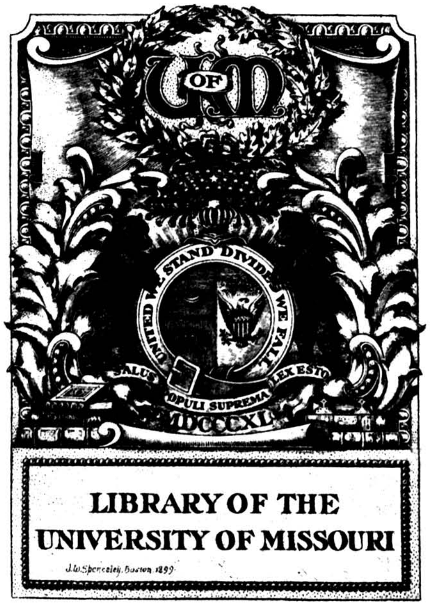
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AN IMPROVED METHOD FOR THE QUALITATIVE SEPARATION AND DETERMINATION OF THE LEAD, BARIUM, AND STRONTIUM IONS.

by

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AN IMPROVED METHOD FOR THE QUALITATIVE SEPARATION AND DETERMINATION OF THE LEAD, BARIUM, AND STRONTIUM IONS.

More accurate and distinct tests in Qualitative analysis are continually sought. Since L. J. Curtman and E. Frankel showed the source of the inaccuracies attending the qualitative determination of the barium ion, new methods have been suggested, of which this one hopes to prove its advantages and probable superiority.

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A common characteristic of the elements, lead, barium, strontium, calcium, and in a peculiar way, bismuth, is the fact that their ions are precipitated by the addition of the sulfate ion. This tendency is strongest with barium and least with calcium. The precipitation of bismuth is prevented by the presence of hydrochloric acid. The carbonates of lead, barium, strontium, and calcium are also quite insoluble. The usual method of Qualitative Analysis uses the insolubility of the lead sulfate in identifying the lead ion and the insolubility of the carbonates of barium, strontium, and calcium in determining their presence. Unfortunately the determination of this carbonate group is in the latter part of the analysis and therefore these ions are quite often exposed to the eliminating action of sulfate ions which appear unintentionally and unavoidably in the course of analysis. These sulfate ions which enter and the carbonate ions obtained from the carbon dioxide of the air mean a loss of certain amounts of barium, strontium and calcium. Calcium and strontium are removed to a lesser degree than barium because of the greater solubility of their sulfates.

The difficulty of detecting small amounts of barium in the usual quantitative procedure had long been noted. The source of these inaccuracies was first located and investigated by L. J. Curtman and E. Frankel.(1) The inability to detect fifty milligrams of barium by the ammonium carbonate method was shown. The factors causing the removal of this amount of barium were shown to be four. The addition of hydrogen sulfide in the precipitation of the copper group, the addition of ammonium sulfide to precipitate the aluminum-zinc group, the sensitiveness of the group and confirmatory tests, and the presence of sulfates in the reagents were all found to be eliminating factors.

The hydrogen sulfide precipitation of the copper group produced a loss of one and one-half milligrams of barium when one hundred milligrams of the latter were present. Curtman and Frankel attribute this loss and other similar losses to the formation of the sulfate ion; others hold that barium has a tendency to be occluded when present during the precipitation of various salts. Strength is lent to the former explanation by the fact that the presence of oxidizing agents at these points augment this loss. One might infer from the last statement that the removal of these oxidizing agents would remove the difficulty. This is possible in some cases, e.g. the nitrate ion may be expelled, but others like ferric chloride cannot be removed. Where ferric chloride is present the loss was shown to reach fourteen milligrams.

At the ammonium sulfide step the losses were due to two causes; the absorbed carbon dioxide in the alkaline reagent and again the oxidized sulfide. During the forty minute separation of one hundred milligrams of ferric chloride and one hundred milligrams of barium chloride with pure ammonium hydroxide, the barium loss was fourteen milligrams. About two and two-tenths milligrams of barium are lost as a result of the oxidized sulfide at this point.

The sensitiveness of the group test depends upon the presence of other ions. If ten grams of ammonium chloride are present less than ten milligrams of barium can not be precipitated by reagent ammonium carbonate.

The loss due to sulfates in reagents is of course a variable source of error. Curtman and Frankel say, "Many of the so called "highest purity" reagents sold by prominent manufacturers often contain enough sulfates to make the test for barium in the fourth group a practical impossibility even though appreciable amounts of the element are present."

Appreciating the possibility of these large losses, Curtman and Frankel(2) and others including Prof. J. W.

(3)
Marden of the University of Missouri have devised various schemes for the determination of lead barium and strontium in the earlier part of the analysis.

The schemes proposed are alike to the extent that the lead, barium, strontium and calcium ions are precipitated as sulfates immediately after the removal of the silver group. They differ in the means of separating the lead sulfate from the other sulfates. Some of the solvents for lead sulfate which have been proposed are the acetate, nitrate, citrate, tartrate, and thiosulfate of ammonia and certain alkaline solutions. After the removal of the lead sulfate, the others are converted to carbonates by boiling with twice normal sodium carbonate solution. These carbonates are dissolved in acetic acid solution. The barium is removed by the addition of acid ammonium chromate solution. The strontium is removed by making the filtrate which is a result of the barium removal alkaline with ammonium hydroxide. Calcium is finally precipitated as an oxalate.

There are several sources of error when the above scheme is carried out. The first is the incomplete precipitation of the lead as sulfate if sufficient hydrochloric acid is present to keep the ions other than those whose precipitation are desired in solution. This objection is mitigated by the fact that lead may again be tested for in the copper group if traces are suspected.

A more serious objection is the solvent action of the alkali salt solutions, which are intended to dissolve the lead sulfate, on the other sulfates. Ammonium acetate solution, so far considered the best solvent for lead sulfate, will dissolve large amounts of strontium and calcium sulfates also. This makes the tests for small amounts of strontium and calcium impossible, and since this dissolved strontium and calcium goes with the lead, the identification of the lead is made less certain.

Experience with students using this scheme in Qualitative Analysis showed that there was some trouble in determining when all the lead sulfate was removed. The result is that "barium" tests are reported when lead is responsible for the "barium" chromate precipitate. There is a difference in the color of lead and barium chromates which one may distinguish. Yet one can not infer absence of barium when the chromate precipitate resembles lead chromate.

The data offered by J. W. Marden illustrates to what degree these other sulfates are dissolved when lead sulfate is removed.(4)

Concentration of	Solubility of Sulfates in Am. Ac.				
Ammonium Acetate	.00	2.13	3.34	10.68	21.37
Grams of $Sr.SO_4$	.0151	.0451	.0732	.0942	.115
Grams of $BaSO_4$	.000235	—	—	—	.016

Temperature 25°C

Data is lacking for the high concentration (30.8) of the four-normal ammonium acetate used in the University

Laboratory but it would seem from this data that at least thirty milligrams of strontium sulfate and four milligrams of barium sulfate would be removed by the use of twenty-five c.c. of the solvent. Work on the effect of the presence of soluble sulfates in the ammonium acetate has shown that these losses can be reduced.(5)

In view of experience with the above outlined method now in use at the University of Missouri, Prof. J. A. Gibson suggested that other solvents might be experimented with for the purpose of securing an easier solution of the lead sulfate and a less solvent action on the other sulfates. Other work is in progress which tends to show that other solvents like ammonium citrate, while capable of dissolving lead sulfate, dissolved the other sulfates to a degree even greater than ammonium acetate.(6)

After considering the trend of the work on this point, the writer suggested that the removal of the lead sulfate might be dispensed with, and thought it advisable to try a conversion of all the sulfates to carbonates and the removal of the lead from an acetate solution as the sulfide. This method brought a conviction that a greater degree of accuracy in the determination of barium and strontium would result. The question as to whether lead sulfate could be transferred to the carbonate as readily as the other sulfates arose: also, can lead be quantitatively separated from barium by the use of hydrogen sulfide when the ions are present in an acetate solution. The latter question presenting the greater interest was treated first.

Preparatory to testing of the sulfide separation, solutions of lead and barium acetates were made such that five grams of the solutions contained about one hundred milligrams of the basic ion. These solutions were analysed by precipitating the bases as sulfates, evaporating to expell other ions, and then diluting with one hundred c.c. of a 1-1 mixture of alcohol and water. The precipitate was filtered onto a Gooch crucible, and after drying two hours at 200°C it was weighed. From the weight of the sulfates the amount of lead and barium per sample was determined and from this the amount of lead and barium per five grams of these solutions determined. The amount of lead per five grams was .0997 grs., of barium there were .1046 grs.

In testing the hydrogen sulfide separation of lead from barium in acetate solution, beakers were prepared containing about five grams of each of the above named solutions. The mixtures were heated and hydrogen sulfide passed in until complete precipitation. The lead sulfide was filtered out. The filtrate was boiled to expell hydrogen sulfide, and reserved for the barium determination.

The lead sulfide was dissolved in warm dilute nitric acid, and after the removal of some free sulfur, the lead in the nitric acid solution determined as sulfate. The amounts of barium in the first filtrate was also determined.

The amounts of lead per five gram recovered were:

.0997 gr. .0999 gr. .0994 gr. .0994 gr.

The amounts of barium per five gram recovered were:

.1043 gr. .1052 gr. .1047 gr. .1051 gr.

The average of lead taken	.09967
The average of lead recovered	.09961
The average of barium taken	.1046
The average of barium recovered	.1048

From the above results little concern as to the sulfide separation remained.

To get an idea of the applicability of the entire scheme, known amounts of barium and lead acetate solutions were mixed and taken through the various steps and at the final steps determined as sulfates. The barium results were a recovery of about the same amount as that taken. The average amount of barium taken was .1046 grams and the analyses of two of the recovered amounts were .1035 gr. and .1047 gr. The lead results were very disappointing. .09967 grams of lead were taken and the analyses at the final step revealed only:-

.05678 gr. .04649 gr. .04807 gr. .04222 gr.

respectively for four samples which had gone through the system of analysis. The place at which this large lead loss might take place was apparently at the point where the lead sulfate is converted to lead carbonate.

Authoritative statements regarding the conversion of lead sulfate to lead carbonate by boiling with sodium carbonates solution were found which gave no intimation of the source of error. Storer cites the following statements; (7)

"Decomposed when boiled with an aquous solution of the carbonate of potash, soda, and ammonia, with formation of insoluble carbonates of lead" (Perez, Chem Molec., p 384)

"Completely decomposed even at ordinary temperatures by solutions of MONO and bicarbonates of the alkalis" (H. Rose Pogg Ann 95:426) "An equivalent of sulfate of lead may be completely decomposed by an equivalent of an alkaline carbonate. in aqueus solution. (Malaguti, Ann Ch. et Phys. (3) 51, 347.)

A. A. Noyes and Wm. Bray publish the following statement;(8) "The boiling with Na_2CO_3 converts into carbonates the sulfates of lead, calcium, strontium, and bismuth completely, and at least 80% of the sulfate of barium, even when large quantities of them are present."

Gardner and Schaeffer in their "Analysis of Paints" (9) say that total sulfates may be obtained as follows: "Mix in a beaker ½ gram sample with 3 grams sodium carbonate. Add 30c.c. of water and boil gently for ten minutes. Allow to stand four hours. Dilute with hot water, filter and wash until filtrate is about 200 c.c. in valume. Reject the residue. By this reaction all the lead sulphate is changed to carbonate and the filtrate contains sodium sulphate." The sulfate in the filtrate is weighed as $BaSO_4$.

A. M. Comey states that lead sulfate is "decomposed by boiling with K_2CO_3, Na_2CO_3, and $(NH_4)_2CO_3$+Aq."(1)

The word "decomposed" conveys the idea that there is a metathesis in which lead carbonate and sodium sulfate result. The first citation explains the word by the statement of the product.

To test this conversion of lead sulfate to lead carbonate, weighed samples of lead sulfate were taken and covered with about fifty c.c. of 2N. Sodium carbonate solution, After boiling about five minutes, the mixture was allowed to stand on the waterbath three or four hours, and then to stand in the cold about two hours. After washing by decantation the residual carbonate was dissolved in thirty percent acetic acid. The lead contained in the acetic acid solution was determined as sulfate. The results were:-

$PbSO_4$ taken	740.7 mg	470.5 mg	544.9 mg	450.2 mg
" recovered	717.1 "	441.8 "	521.9 "	414.9 "
" lost	23.6 "	28.7 "	23. "	35. "

Volumes were not kept absolutely equal in these determinations as no such losses were anticipated. When these losses were noticed, the marked solubility of the lead was even then not suspected, and a more exact method was sought. Noyes and Braye's statement was followed. "Transfer the residue insoluble in H_2SO_4(P.5) to a casserole, add about 25 ccm. saturated Na_2CO_3 solution, cover the casserole, and boil gently for 10 minutes." A saturated solution of sodium carbonate was prepared and the proceedure followed with the following results:

$PbSO_4$ taken	548.5 mg	588.3 mg	680.5 mg
" found	497. "	529.2 "	628.1 "
" lost	51.5 "	59.1 "	52.4 "

This greater loss with more concentrate sodium carbonate solution suggested the idea that lead sulphate was quite soluble in concentrate sodium carbonate solutions. This means that there is probably a double salt produced which dissolves in water.

The method of Gardner and Schaeffer was next tried where the strength of the carbonate solution is three grams of the salt and thirty cc. of water except that three grams of sodium carbonate in fifty cc. of water was used. This is about 1.2 normal sodium carbonate. The results were:

$PbSO_4$ taken	534.6 mg	547.9 mg	484.3 mg
" recovered	523.5 "	538.9 "	476.7 "
" lost	11.1 "	9 "	7.6 "

Since more dilute solutions of the carbonate solutions were giving better results, a solution of two grams of salt in fifty c.c. was tried.

Lead sulfate taken	583.5 mg	415.4 mg	510.6 mg	633.7 mg
" " recov.	577.8 "	410.1 "	505.2 "	627.4 "
" " lost	5.7 "	5.3 "	5.4 "	6.3 "

A place will be attained in using more dilute solutions of the carbonate where the concentration of the carbonate ion is in-sufficient to replace the sulfate and maintain an equilibrium. But considering the statement of Abegg,[13] "Bleicarbonat ist im verhaltnis zum sulfat so viel schwerer löslich (die Löslichkeiten in reinem Wasser verhalten sich wie 1:600) daz es beim Behandeln mit Alkalisulfatlösung nicht umgesetzt wird, the use of equal molecular solutions should be sufficient to make the conversion."

Barium sulfate conversion to carbonate does not seem to present a difficulty similar to that of lead. This may be due to a lack of that general tendency which the lead compounds possess of producing water soluble compounds with alkali salts. This lead sulfate conversion is still capable of improvement.

In the work of Curtman and Frankel a minimum of twenty-five milligrams of lead seems to be the limit of the test for the lead ion.[14] One of the facts leading to this seems to be the inefficiency of the precipitation of lead as sulfate. The table cited in their paper indicates that the presence of 5 m.g. of ammonium nitrate increased the efficiency of the sulfate precipitation. This seems remarkable because of the solvent action of solutions of this salt on lead sulfate. An attempt was made to s/p corraborate this statement by using a slightly different procedure. The method given in the paper was to set up solutions containing varying amounts of lead in twenty-five c.c. of solution containing five c.c. of con. hydrochloric acid and equal amounts of alcohol. To half were added five grams of ammonium nitrate and to a similar set none of the salt. Eight c.c. of 20%

sulfuric acid were added to all and a note made of those showing a precipitate in five minutes.

Without the ammonium nitrate the only solutions to give a precipitate in five minutes were those containing fifteen or more milligrams of lead. With ammonium nitrate present solutions containing as little as ten milligrams would give a precipitate. The presence of this salt apparently enabled smaller concentrates of lead to precipitate as sulfate.

In trying this effect of the nitrate the writer tried another scheme. Solutions were made up containing five hundred milligrams of lead nitrate, five c.c. of concentrate hydrochloric acid, five gr. of ammonium nitrate, in twenty-five c.c. of solution. After boiling, eight c.c. of 20% sulfuric acid were added and the volume doubled by the addition of alcohol. After twenty-hours the sulfate precipitate was filtered out and weighed. From this weight the amount of lead nitrate recovered was found. Then the same procedure was followed with another set of samples which did not contain the five grams of ammonium nitrate. By comparing the losses in the two methods the effect of the ammonium nitrate was made apparent.

Without the ammonium nitrate:

Lead	nitrate	taken	618.2 mg	671.7 mg	5044 mg
"	"	recov.	616.2 "	669.0 "	5016 "
"	"	lost	2. "	2.7 "	2.8 "

With ammonium nitrate the following results were obtained:

Lead nitrate	taken	533.8 mg	4847 mg
" "	recovered	228.8 "	4801 "
" "	lost	5.0 "	4.6 "

From these results it would seem that after standing twenty-four hours a greater amount of the lead salt can be removed by filtration. and that ammonium nitrate has the effect of lowering the sulfate yield. If the sulfate is filtered after five or ten minutes the loss is somewhat greater, approximating the figures of Curtman and Frankel, i.e. about ten milligrams. There is a tendency for the lead sulfate to "run through" which can be corrected to some extent by pouring the filtrate through the filter till clear. Unless sufficient time is allowed, the lead removal at this point is not so accurate as it might be. If though the ammonium salts are removed by evaporating to dryness and the solution allowed to stand after precipitation with sulfuric acid, the lead loss at this point is less than two milligrams. If evaporation after precipitation is possible the removal may be made quantitative.

Twice normal sulfuric acid had been used in a former scheme as the group precipitant with the hope of a small removal of calcium at this point. This strength acid was tried as a precipitant of lead with results that would indicate its impracticability. One hundred milligrams of lead were dissolved in five grams of dilute nitric acid, twenty c.c. of water added, and the solution neutralized with ammonium hydroxide. Five c.c. of con. hydrochloric acid were added and after heating to boiling twice normal sulfuric acid was added. No amount of the sulfuric acid would cause a precipitate in the hot solution and in only one case did a precipitate form after cooling the liquids. The concentration of the sulfate ion in this solution is apparently too small to act as a lead precipitant under the conditions which exist at this point. The sulfuric acid used to precipitate the lead or sulfate group should be at least 20% in strength. This concluded the tests on the points in which the proposed method would be interested.

All new conditions involved in the proposed scheme have been quantitatively tested. The barium and strontium follow the old route except for their subjection to the action of hydrogen sulphide when in solution as acetate. One would not expect any loss here as their sulphides are soluble and there is little danger of oxidation in acetic acid solution. The test on the separation of lead and barium under these conditions confirmed the supposition.

The lead has been followed through each step; the precipitation as sulfate, the conversion to carbonate, the solution as acetate, the precipitation as sulphide and lastly the sulfate confirmatory test. The results point to more exact methods in all cases and qualitative results for barium and strontium.

The following method for the quantitative separation of lead, barium and strontium is proposed.

Lead Group

Procedure 1. To two c.c. of the filtrate from the silver group which contains all the lead and 5 c.c. of con.

hydrochloric acid (the Ag. group precipitant) add 1 c.c. dilute sulfuric acid, and 3 c.c. of 95% alcohol. Let stand ten minutes. A white precipitate indicates the presence of the lead group. If the group is present use procedure 2.

Procedure 2. Evaporate filtrate from silver group to dryness, take up with a solution of 5 c.c. con. hydrochloric acid and 25 c.c. water, add ten c.c. of 20% sulfuric acid and 95% alcohol to double the volume. For most accurate test allow to stand twnety-four hours before decanting through a quantitative filter. Wash the precipitate twice by decantation and treat by P.3.

Procedure 3. Transfer residue 2 to a porcelain dish and add 2 gr. of sodium carbonate and 50 c.c. of water. Boil gently ten minutes and allow to stand 3-4 hours. Dilute with warm water and filter. Wash carefully. Reserve filtrate for P. 5. Pour 5 c.c. of warm 30% acetic acid through the filter. Warm the acetic acid and again pour it through the filter. If the residue does not dissolve completely, boil the undissolved residue with sodium carbonate solution and again filter and wash. Again pour 5 c.c. of warm acetic acid through the filter. Mix the acetic acid solutions and saturate with hydrogen sulphide. A brown or black precipitate indicates the presence of the lead ion. If the lead ion is found, filter and confirm by P.4. If the lead ion is not found use. P. 6.

Procedure 4. Dissolve precipitate 3 in 5 c.c. of warm dilute nitric acid. Boil to expell hydrogen sulphide and after cooling add potassium iodide drop by drop. A yellow precipitate confirms the presence of the lead ion.

Procedure 5. Acidify the alkaline filtrate with dilute sulfuric acid and evaporate to dryness. A white precipitate insoluble in water indicates the lead ion.

Procedure 6. Saturate filtrate 3 with hydrogen sulfide and filter if necessary. Reject the precipitate. Boil the filtrate vigorously to expell H_2S, and add 5 c.c. of acid ammonium ch~~l~~romate solution. A pale, yellow precipitate indicates the presence of the barium ion. Filter and treat the clear filtrate by P.7.

Procedure 7. To the filtrate 6 add ammonium hydroxide solution till the solution just turns yellow and then add ten drops more. An immediate precipitate shows the presence of the strontium ion in considerable quantity. A slowly forming precipitate shows a small amount of the strontium ion. Add enough 60% alcohol to double the volume. This precipitates all the strontium as chromate. Let stand 15 minutes to detect traces. Filter and treat filtrate by P.8.

Procedure 8. Evaporate filtrate 7 to 5 c.c. and add ammonium oxalate. A white crystaline precipitate indicates the calcium ion.

A copy of the above scheme was given to a class of students in qualtitative analysis, who had completed the work on the bases, together with two unknown solutions each containing members of this group. Without further instructions they were told to analyze the solutions. The method was cheerfully accepted, and only one error in this group was found in the reports of the fifteen students. The concentration of the basic ions in these solutions was less than that of unknowns usually given; and the number of errors considerably less than that previously found.

CONCLUSIONS:

The use of sulfuric acid more concentrated than twice normal is necessary to precipitate lead as sulfate under the conditions which exist at the beginning of the sulfate group. The presence of ammonium nitrate at this point does not increase the efficiency of the sulfate precipitation of lead. If after the precipitation of the sulfate group the mixture is allowed to stand twenty-four hours, all but four milligrams of lead may be filtered out as sulfate.

The conversion of lead sulfate to lead carbonate by boiling with sodium carbonate solution produces a solution of some of the lead. This solubility of the lead decreases with the dilution of the sodium carbonate solution used. The solubility of lead sulfate in fifty c.c. of sodium carbonate solution decreases from fifty m.g. in a saturated solution to about five m.g. in a four percent solution. This drop would indicate that more dilute solutions of sodium carbonate than four percent would give still better conversion, i.e. less of the soluble lead compound would be found. These four milligrams of lead which dissolve in the four percant sodium carbonate solution can, by the use of another step, be recovered and tested for.

The results obtained in the method of determining total sulfates suggested by Gardner and Schaeffer must be slightly inaccurate. The sulfate weighed and from which the amount of sulfate present is calculated is not entirely barium sulfate but some lead sulfate formed from the lead which had dissolved in the sodium carbonate solution used. The lead sulfate present will probably be about eight milligrams under the conditions outlined in the text.

When lead is separated from barium by passing hydrogen sulfide into their acetate solution there is no appreciable loss of lead or of barium due to the formation of sulfates or to other causes. If barium is not lost strontium and calcium will also be uneffected, as the formation of their sulfates is less likely than that of barium sulfate.

The degree of accuracy for the determination of lead depends entirely upon the initial precipitation of the lead or sulfate group. The completeness of the lead removal immediately after the silver group is dependent upon the presence of other ions and the time available for making the removal. If antimony or bismuth and ammonium salts are present the amount of lead to pass to the copper group may be twelve milligrams. If evaporation is possible after the sulfate precipitation all the lead may be tested for by the method proposed. In this scheme amounts of lead above four milligrams will be found after the sulfide separation of lead from the other basic ions. The other four milligrams or less can be detected in the sodium carbonate filtrate which contains besides the sodium, carbonate, and sulfate ions the small amount of lead. The old scheme will not detect lead when present to an extent of less than twenty-four milligrams.

Allowing twalve milligrams for incomplete precipitation at the beginning of the sulfate group, the loss during the process of separation and identification must be an equal amount. This last source of error can be avoided. The method proposed should give a test for all the lead precipitated with the group.

By the scheme proposed the determination of barium, strontium and calcium should be more exact. Their route is similar to that of the methods already proposed except that they are not exposed to the action of ammonium acetate while sulfates, and that as acetates they are present while the lead is removed as sulfide. As before stated this last variance from the old procedure produces no appreciable loss. The first variance can have but one possible result, i.e. more strontium and calcium where their identification is sought.

The new method lacks the possibility of confusing the ions, i.e. there seems to be no possibility of strontium appearing with the lead or vice versa. When the lead ion is removed from the others, its removal will be complete and have no possibility of taking any barium, strontium or calcium with it. The tests should be clear cut.

(1) Journal of the American Chemical Society
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(2) Journal of the American Chemical Society
XXXIV, 1493 (1912)

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(5) Journal of the American Chemical Society

(6) Work being done by J. W. Marden and C. Loeb, U. of Mo.

(7) Dictionary of the solubilities of Chemical substances
p. 605

(8) Journal of the American Chemical Society (Reprint)
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(9) "Analysis of Paints" p.17 (1911)

(10) Dictionary of Chemical Solubilities - Morgan
p. 437 (1896)

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(12) "Analysis of Paint" p.17 (1911)

(13) Handbuch der Anorganishen Chem. 111, 2, 726

(14) Journal of American Chemical Society
XXXIV, 1493 (1912)

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