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An Investigation of Sundry Reactions for Application as Confirmatory Tests in Qualitative Analysis

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AN INVESTIGATION OF SUNDRY REACTIONS FOR

APPLICATION AS CONFIRMATORY TESTS IN

QUALITATIVE ANALYSIS

By

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PREFACE

Is the present "scheme" of qualitative analysis satisfactory? For many years the answer has been, No! In the present "scheme" it has frequently been necessary to strike a compromise between accuracy and simplicity. One solution to the problem can be found in the availability of a number of specific reagents, one for each substance to be identified, giving unmistakable tests and positive results. It would greater simplify the situation if a single inorganic salt could be used as a reagent for all the metallic ions.

One of the most successful reagents used was potassium mercuric thiocyanate. The water soluble salts of sodium and potassium were employed because of their reactivity in acid solutions.

The author wishes to acknowledge her gratitude to the members of the Marquette University Chemistry Department for their time and interest, and especially to Dr. Scott L. Kittsley under whom this research was done, for his directions and ever available help.

I N T R O D U C T I O N

All analytical procedures may be grouped under two headings, qualitative and quantitative. Qualitative work is concerned only with the separation and identification of all metallic ions and acid radicals. The metals are divided into five groups, namely...the silver, copper-tin, aluminum-iron, barium, and the magnesium groups.

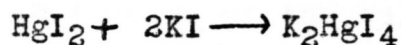
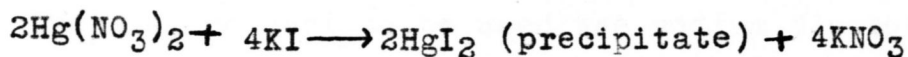
In analyzing a solution, the groups must first be separated usually by precipitation, then each group is analyzed separately for the metals. If it is desired to know if a certain metal is present or not, its group must first be separated, and the rest of the metals of that group excluded before the test can be made. Quantitative procedures are much along this same line. A certain substance is to be analyzed to find out how much of a given constituent it contains.

It has long been the desire of the analysts to use a reagent which will indicate immediately the presence of the metal for which they are testing; and it is only within recent years that their desire has partially become a reality.

EXPERIMENTAL RESULTS

Preparation of Reagents:

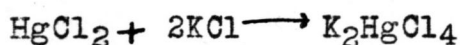
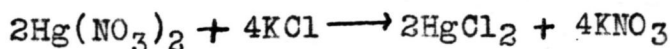
The first reagent to be prepared was potassium mercuric iodide. A solution of potassium iodide was added to a known amount of mercuric nitrate until a red-orange precipitate was formed. Then an excess of potassium iodide was added until the precipitate disappeared, and the solution became clear. The equation for the reaction is as follows:



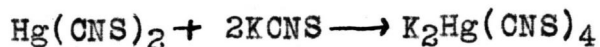
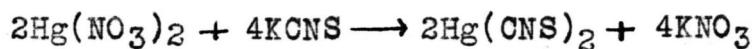
The reagent was filtered to remove any residue or impurities. A trial was made with a water blank to detect any color change of the reagent in an aqueous solution. No color change was detected in the case of the reagents employed here.

In the preparation of all the reagents used, the volumes of the constituents and their respective concentrations were known. The amount of reagent produced in all cases is equal to and cannot exceed 0.1032 moles.

The preparation of the second and third reagents is comparable to the method used for the preparation of the first. The second reagent used was potassium mercuric chloride. Instead of using potassium iodide as was used in the preparation of the first reagent, potassium chloride was used in this case. The equation for the preparation of potassium mercuric chloride is as follows:



The third reagent does not contain a halogen. Although the same method was employed in the preparation of it, potassium sulfocyanate was used. The reagent is potassium mercuric sulfocyanate and the equation for the reaction is as follows:



The last reagent to be used was sodium fluoborate, (NaBF_4). 2.4 grams of the salt were weighed and dissolved in 200 milliliters of distilled water to give a 0.1032 molar solution.

Procedure:

To five milliliters of the known solution, containing the metal or metallic ion, was added a small amount of reagent. If on addition of more reagent, no precipitate occurred, the solution was heated and cooled gradually. If a precipitate was not formed, then the solution was allowed to stand for 24 hours to detect if any precipitation or color change would take place.

The test solutions used were nitrates of the metals, with a few exceptions. The metal chlorides used were those of arsenic, antimony and tin. The metal sulfate used was that of iron (ferrous); and the chromate salt used was potassium dichromate.

TABLE I

Test Solutions

These test solutions used are of known concentration.

The test solutions are arranged in the order of the groups.

Metal	Formula of the dry salt	Moles of salt per liter of soln.	Special pre- cautions in preparing the soln.
Silver	AgNO_3	0.0776	
Mercury (ous)	HgNO_3	0.1256	use 150cc. of 5N HNO_3 /l.
Lead	$\text{Pb}(\text{NO}_3)_2$	0.1036	use 5cc. of 5N HNO_3 /l.
Bismuth	$\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$	0.0957	dissolve in 500cc. of N HNO_3 and dilute to 1 liter.
Mercury (ic)	$\text{Hg}(\text{NO}_3)_2$	0.1032	use 50cc. of 5N HNO_3 /l.
Copper	$\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$	0.3136	use 5cc. of 5N HNO_3 /l.
Cadmium	$\text{Cd}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$	0.1711	use 5cc. of 5N HNO_3 /l.
Arsenic (ous)	As_2O_3	0.1029	dissolve in hot water containing 20cc. of 5N HCl , and dilute to 1 liter.
Arsenic (ic)	As_2O_5	0.1305	dissolve in hot water, adding HCl to form a clear soln., and dilute to 1 liter.
Antimony (ous)	SbCl_3	0.1642	dissolve in 400cc. of 5N HCl and dilute to 1 liter.

Table I continued:

Metal	Formula of the dry salt	Moles of salt per liter of soln.	Special precautions in preparing the soln.
Antimony (ic)	SbCl_5	0.1500	dissolve in 500cc. of 5N HCl and dilute to 1 liter.
Tin (ous)	$\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$	0.2115	dissolve the salt with 100cc. of conc. HCl, let stand 24 hours, then dilute to 1 liter. Filter if necessary.
Tin (ic)	$\text{SnCl}_4 \cdot 5\text{H}_2\text{O}$	0.2705	dissolve in 400cc. of 5N HCl and dilute to 1 liter.
Iron (ous)	$\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$	0.3555	use 25cc. of 5N H_2SO_4 per liter.
Iron (ic)	$\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$	0.3706	use 50cc. of 5N HNO_3 per liter.
Aluminum	$\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$	0.7405	use 25cc. of 5N HNO_3 per liter.
Chromium (ic)	$\text{Cr}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$	0.3920	use 50cc. of 5N HNO_3 per liter.
Chromium (ate)	$\text{K}_2\text{Cr}_2\text{O}_7$	0.3870	
Nickel	$\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$	0.3396	use 25cc. of 5N HNO_3 per liter.
Cobalt	$\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$	0.3466	use 25cc. of 5N HNO_3 per liter.
Manganese (ous)	$\text{Mn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$	0.3624	use 5cc. of 5N HNO_3 per liter.
Zinc	$\text{Zn}(\text{NO}_3)_2$	0.5503	
Barium	$\text{Ba}(\text{NO}_3)_2$	0.1515	
Calcium	$\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$	0.4987	
Magnesium	$\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$	0.7725	use 5cc. of 5N HNO_3 per liter.

Table I continued:

Metal	Formula of the dry salt	Moles of salt per liter of soln.	Special pre- cautions in preparing the soln.
Sodium	NaNO_3	0.8695	
Potassium	KNO_3	0.5091	
Ammonium	NH_4NO_3	0.3669	

TABLE II

The Effect of Potassium Mercuric Iodide
on the Metals and Metallic Ions.

The concentration of the reagent is 0.1032 moles per liter of solution.

Metal	Amount of Reagent Used	Description of Precipitates Formed
Ag ⁺	2cc.	Fine yellow precipitate; turns white on standing
Hg ⁺	2cc.	Heavy yellow precipitate
Pb ⁺⁺	2cc.	Fine, granular orange precipitate
Bi ⁺⁺⁺	2cc.	Violet-brown precipitate
Hg ⁺⁺	2cc.	Orange-white, suspension like precipitate
Cu ⁺⁺	5cc.	Heavy, orange-brown precipitate
	additional 5cc.	Heavy, orange precipitate
Cd ⁺⁺	2cc.	Fine, orange precipitate
As ⁺⁺⁺	----	No precipitate formed
As ⁺⁺⁺⁺⁺	----	No precipitate formed
Sb ⁺⁺⁺	1cc.	Soln. turns yellow
	additional 3cc.	Flocculent, yellow-white precipitate
Sb ⁺⁺⁺⁺⁺	3cc.	Flocculent, deep yellow precipitate
Sn ⁺⁺	----	No precipitate formed
Sn ⁺⁺⁺⁺	----	No precipitate formed
Fe ⁺⁺	----	No precipitate formed
Fe ⁺⁺⁺	----	No precipitate formed

Table II continued:

Metal	Amount of Reagent Used	Description of Precipitates Formed
Al +++	----	No precipitate formed
Cr +++	----	No precipitate formed
Cr ++++++	----	No precipitate formed
Ni ++	----	No precipitate formed
Co ++	----	No precipitate formed
Mn ++	----	No precipitate formed
Zn ++	----	No precipitate formed
Ba ++	----	No precipitate formed
Ca ++	----	No precipitate formed
Mg ++	----	No precipitate formed
Na +	----	No precipitate formed
K +	----	No precipitate formed
NH ₄ +	3cc.	Small deposit of yellow crystals

TABLE III

The Effect of Potassium Mercuric Chloride
on the Metals and Metallic Ions.

The concentration of the reagent is 0.1032 moles per liter of solution.

Metal	Amount of Reagent Used	Description of Precipitates Formed
Ag +	1cc.	Heavy white precipitate
Hg +	1cc.	Fine white precipitate
Pb ++	2cc.	White crystalline precipitate
Bi +++	----	No precipitate formed
Hg ++	----	No precipitate formed
Cu ++	----	No precipitate formed
Cd ++	----	No precipitate formed
As +++	----	No precipitate formed
As +++++	----	No precipitate formed
Sb +++	8cc.	White flocculent precipitate; disappears on standing
Sb +++++	6cc.	White flocculent precipitate; disappears on standing
Sn ++	5cc.	Heavy white precipitate; turns gray on standing
Sn +++++	5cc.	Heavy white precipitate; turns gray on standing
Fe ++	----	No precipitate formed
Fe +++	----	No precipitate formed
Al +++	----	No precipitate formed
Cr +++	----	No precipitate formed

Table III continued:

Metal	Amount of Reagent Used	Description of Precipitates Formed
Cr ++++++	----	No precipitate formed
Ni ++	----	No precipitate formed
Co ++	----	No precipitate formed
Mn ++	----	No precipitate formed
Zn ++	----	No precipitate formed
Ba ++	----	No precipitate formed
Ca ++	----	No precipitate formed
Mg ++	----	No precipitate formed
Na +	----	No precipitate formed
K +	----	No precipitate formed
NH ₄ +	----	No precipitate formed

TABLE IV

The Effect of Potassium Mercuric Thiocyanate
on the Metals and Metallic Ions.

The concentration of the reagent is 0.1032 moles per liter of solution.

Metal	Amount of reagent used	Description of precipitates formed
Ag ⁺	1cc.	Heavy white precipitate
Hg ⁺	1cc.	Fine, granular gray precipitate
Pb ⁺⁺	----	No precipitate formed
Bi ⁺⁺⁺	8cc.	Slight effervescence; color change in soln. from light red to orange to brown-orange; soln. clears and turns to green; small deposit of green precipitate is formed
Hg ⁺⁺	3cc.	Fine, white flocculent precipitate
Cu ⁺⁺	3cc.	Color of soln. changes from blue to green; heavy, green-yellow precipitate is formed
Cd ⁺⁺	----	No precipitate formed
As ⁺⁺⁺	5cc.	Soln. turns pink; color disappears on standing; no precipitate formed
As ⁺⁺⁺⁺⁺	5cc.	Soln. turns pink; color disappears on standing; no precipitate formed
Sb ⁺⁺⁺	5cc.	White suspension formed; disappears immediately
	additional 15cc.	Fine, white precipitate formed; settles on standing

Table IV continued;

Metal	Amount of reagent used	Description of precipitates formed
Sb +++++	5cc.	White suspension formed; disappears immediately
	additional 15cc.	Fine, white precipitate formed; settles on standing
Sn ++	----	No precipitate formed
Sn +++++	----	No precipitate formed
Fe ++	3cc.	Fine, red-brown precipitate
Fe +++	2cc.	Fine, orange-brown precipitate
Al +++	----	No precipitate formed
Cr +++	----	No precipitate formed
Cr ++++++	----	No precipitate formed
Ni ++	----	No precipitate formed
Co ++	5cc.	Fine, purple-blue precipitate; settles on standing
Mn ++	----	No precipitate formed
Zn ++	5cc.	Fine, white precipitate; settles on standing
Ba ++	----	No precipitate formed
Ca ++	----	No precipitate formed
Mg ++	----	No precipitate formed
Na +	----	No precipitate formed
K +	----	No precipitate formed
NH ₄ ⁺	2cc.	Fine, yellow precipitate; settles on standing

TABLE V

The Effect of Sodium Fluoborate
on the Metals and Metallic Ions.

The concentration of the reagent is 0.1032 moles per liter of solution.

Metal	Amount of reagent used	Description of precipitates formed
Ag ⁺	----	No precipitate formed
Hg ⁺	----	No precipitate formed
Pb ⁺⁺	----	No precipitate formed
Bi ⁺⁺⁺	----	No precipitate formed
Hg ⁺⁺	----	No precipitate formed
Cu ⁺⁺	----	No precipitate formed
Cd ⁺⁺	----	No precipitate formed
As ⁺⁺⁺	----	No precipitate formed
As ⁺⁺⁺⁺⁺	----	No precipitate formed
Sb ⁺⁺⁺	5cc.	White suspension formed; disappears immediately
	additional 5cc.	Fine, white precipitate formed
	additional 1cc.	Precipitate disappears; soln. is colorless
Sb ⁺⁺⁺⁺⁺	5cc.	White suspension formed; disappears immediately
	additional 5cc.	Fine, white precipitate formed
	additional 2cc.	Precipitate disappears; soln. is colorless
Sn ⁺⁺	----	No precipitate formed
Sn ⁺⁺⁺⁺	----	No precipitate formed

Table V continued:

Metal	Amount of reagent used	Description of precipitates formed
Fe ⁺⁺	----	No precipitate formed
Fe ⁺⁺⁺	----	No precipitate formed
Al ⁺⁺⁺	----	No precipitate formed
Cr ⁺⁺⁺	----	No precipitate formed
Cr ⁺⁺⁺⁺⁺⁺	----	No precipitate formed
Ni ⁺⁺	----	No precipitate formed
Co ⁺⁺	----	No precipitate formed
Mn ⁺⁺	----	No precipitate formed
Zn ⁺⁺	----	No precipitate formed
Ba ⁺⁺	----	No precipitate formed
Ca ⁺⁺	----	No precipitate formed
Mg ⁺⁺	----	No precipitate formed
Na ⁺	----	No precipitate formed
K ⁺	----	No precipitate formed
NH ₄ ⁺	----	No precipitate formed

S U M M A R Y

The silver group, including silver mercury(ous) and lead, produced the most clearly defined precipitates, in so far as color reactions were concerned. The reagent used gave more successful conformatory tests with respect to the silver group, than any other group tested.

In the second group, namely the copper-tin, precipitates of the metals occurred frequently, but the precipitates were not distinguishable. By not distinguishable, is meant that the color of the precipitates of the different cations were alike. So similar were the colors, that the identity of the metal was hard to discern. Tin was precipitated only by the reaction of potassium mercuric chloride as a reagent. In no case was arsenic precipitated.

Of the reagents employed in these tests only potassium mercuric thiocyanate was successful as a precipitating agent for the aluminum-iron group. Those metallic ions precipitated by this reagent were iron (ferrous and ferric), cobalt and zinc.

These water soluble salts proved unsuccessful as precipitating agents for both the barium and magnesium groups.

A confirmatory test for the presence of ammonium resulted only when potassium mercuric iodide and potassium mercuric thiocyanate were used.

SUGGESTIONS FOR FURTHER RESEARCH

The work of this research may be continued by testing the effect of still other water soluble salts on the cations. An excellent research would be to investigate the reactions of other fluoborate compounds for application as confirmatory tests in qualitative analysis.

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Date May 17, 1948

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Date May 13, 1948