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Synthesis and Thermal Spectred Analysis of Barium Iodate (And Strontium Iodate (BA and SR) Crystals

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Abstract – Single crystals of iodates of barium and strontium grown by gel method were accounted for. Optimum conditions for good quality single crystals after synthesis were analysed thermally IR spectra of the synthesis.

Keywords: Barium Iodate, Strontium Iodate, Single Crystals

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INTRODUCTION

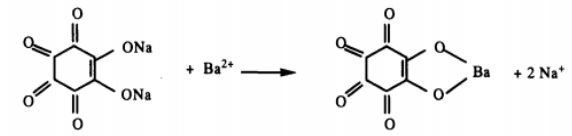
Barium iodate is an inorganic chemical compound with the chemical formula $Ba(IO_3)_2$. It is a white, granular substance. Barium iodate can be inferred either as a product of a reaction of iodine and barium hydroxide or by joining barium chlorate with potassium iodate. The compound is stable on a temperature up to roughly 580 °C (1,076 °F). In the event that the temperature is higher than that esteem, the accompanying reaction, known as Rammelsberg's reaction, happens:

$$5Ba(IO_3)_2 \longrightarrow Ba_5(IO_6)_2 + 9O_2 + 4I_2$$

Barium

1. Test with sodium rhodizonate

One drop of the neutral or slightly acid test solution is put on a filter paper and afterward a drop of an aqueous 0.2 % solution of sodium rhodizonate. A red-brown stain of barium rhodizonate is gotten.



2. Test with potassium chromate

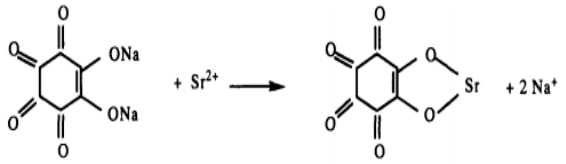
One drop of the test solution and a drop of dil. acetic acid set on a filter paper. A drop of 2.0 % aqueous solution of potassium chromate is included. A yellow precipitate of barium chromate is gotten**.**

$$Ba^{2+} + K_2CrO_4 \longrightarrow BaCrO_4 + 2K^+$$

Strontium

1. Test with sodium rhodizonate

One drop of the test solution is put on a filter paper impregnated with a saturated solution of potassium chromate. After a min. a drop of an aqueous 0.2 % solution of sodium rhodizonate is set on the spot. A brown-red bit or hover of strontium rhodizonate is framed.



Cation Identification Tests

1. Strontium Ions:

Strontium can be distinguished, in the absence of calcium, by accelerating its sulfate. To the solution include 0.1 M H_2SO_4 drop wise. The formation of a finely-partitioned, crystalline, white precipitate indicates the nearness of Sr_2+. (Ba_2+ must be missing, obviously.) To affirm, dissolve the precipitate in concentrated HCl and fire test.

2. Barium Ions:

Barium ions can be recognized by precipitation of its insoluble yellow $BaCrO_4$ salt. On the off chance that Ca_2+ or Sr_2+ are available they will likewise

precipitate within the sight of high concentrations of $CrO4_2$-. In any case, the chromates of Ca_2+ and Sr_2+ are respectably dissolvable; their precipitation can be forestalled by addition of acetic acid. This weak acid gives adequate hydronium ions to lower the $CrO4_2$- concentration enough to keep $CaCrO_4$ and $SrCrO_4$ in solution yet to permit the $BaCrO_4$ to precipitate.

$$2CrO_4^{2-}(aq) + 2H^+(aq) \rightarrow Cr_2O_7^{2-}(aq) + H_2O$$

The flame test on the strong chromate is significant for confirmation.To around 1 mL of solution include 10 drops of 6 M CH_3COOH. At that point include a couple of drops of 0.5 M K_2CrO_4 solution. The presence of a yellow precipitate indicates the nearness of Ba2+. To affirm, dissolve the precipitate in concentrated HCl and flame test.

Reaction with barium nitrate

Include HNO3 drop wise until solution is acidic, heat up the solution for two minutes, at that point test with litmus paper. Keep including and bubbling until solution remains acidic in the wake of bubbling. Cool the solution and include a couple of drops of Ba(NO3)2 and watch any reaction. A precipitate will shape with anions that structure an insoluble barium compound (aside from the ones destroyed by acid as in the above test).

1. Confirmation of iodate ions

To a drop of neutral solution under test, a drop of starch solution and a drop of 1 percent solution of hypo phosphorous acid were included. A transitory lived blue color indicated the nearness of iodate ions.

2. Confirmation of barium ions in barium iodates crystals

A drop of neutral solution was taken on a segment of filter paper. By including 5 percent solution of sodium rhodizonate gave red color which indicated barium

3. Confirmation of strontium ions in strontium iodates crystals

A filter paper was impregnated with a saturated solution of potassium chromate and afterward dried. A drop of neutral solution under test was set on it. Under this condition strontium chromate was framed. Following a moment a drop of 1 percent solution of sodium rhodizonate was included. A brown red color gave the proof of strontium.

4. Confirmation of calcium ions in calcium iodates crystals

To a drop of neutral solution 3 drops of saturated aqueous ammonium ferro cyanide and a drop of alcohol were included. White crystalline precipitates were framed which endorse presence of calcium.

ELECTRON MICROPROBE ANALYSIS

Electron test is a helpful technique for deciding constituent elements in a specimen. By scanning the outside of the specimen locally focused impurities as low as of the request for 100 ppm can be distinguished. Hence in homogeneity in the crystal composition can be concentrated by electron small scale test analysis. Various examples of the grown crystals were mounted on aluminum stubs and carbon coating of around 300 £ thickness was made on the crystal surfaces. Tests were analyzed on sound system scan 5-4-10 electron microscope and results acquired.

From these outcomes it tends to be said that fairly transparent crystals don't have any noticeable impurity. In hazy crystals of barium iodate impurities of Si, Al, Fe, Ca and K are discovered present. Occasionally a couple of transparent crystals of strontium iodate and calcium iodate of light yellow coloruerfe acquired. Electron microprobe analysis indicated the presence of Si, Al, Mg and Fe in these crystals. This suggests color formation is because of these foreign elements. A few portions of platy strontium iodate crystals and calcium iodate crystals were discovered murky.

Examination of these hazy regions indicated presence of Na and Si ions, while transparent regions of a similar crystal didn't show presence of such impurities. This demonstrates misty regions are because of the incorporation of gel in the crystals during their growth. The test is helpful for distinguishing a specific inclusion in jam measured zones in a matrix, and in conjunction with the scanning electron microscope, this can be imagined too. One such typical case. Here, haphazardly disseminated impurity spots of silicon are seen on s 135s growth highlights of strontium iodate crystal surface.

THERMAL ANALYSIS

Thermal analysis includes techniques, for example, thermo gravimetric analysis (TGA) and differential thermal analysis (DTA). It was carried out on MOM Derive to diagram. Unequivocal measure of .the example ,e.g., 200 mg, was taken and warming was carried out from room temperature to 1000° C at a pace of 10° C every moment. Thermal spectra of iodate crystals of barium, strontium and calcium. TGA bends indicate changes in weight with increment in temperature and DTA bends show, the nature of reaction - regardless of whether it is endothermic or exothermic, encephalitic changes or stage changes, and so on.

Barium iodate crystals

TGA curve for barium iodate crystals shows that the compound is stable upto 160° C. It loses 4 % weight in the temperature range 160 - 240° C. The weight

reduction is finished at 240° C and there is no further weight reduction upto 640° C. The 4 percent weight reduction is because of dehydration of 1 particle of water, demonstrating that barium iodate crystals are mono hydrated.

$$Ba(IO_3)_2 \cdot H_2O \rightarrow Ba(IO_3)_2 + H_2O\uparrow$$

The anhydrous barium iodate remains stable upto 640 C and afterward it decomposes in the temperature range 640 - 760° C. The 54 percent weight reduction in this "emperature range indicates that this decomposition ought to follow the reaction

$$5\ Ba(IO_3)_2 \rightarrow Ba_5(IO_6)_2 + 4I_2\uparrow + 9O_2\uparrow$$

Qualitative chemical analysis of the residue shows presence of Ba and I in it. Reaction is analogous 4) to the aftereffects of Bousquet and Varmande ; DTA curve of this compound shows two peaks at 160 - 240° C and 640 - 760° C. These peaks indicate that the reactions and are endothermic. Identical pyrolysis curves are gotten for both transparent and opaque crystals. This implies transparent just as opaque crystals of barium iodate are monohydrate.

Strontium iodate crystals

Thermal curves indicated two varieties of strontium iodate crystals found in the current work. Prismatic, platy and lamellar needles give the thermal curves while prismatic bipyramidal and prismatic needle type crystals give the thermal curves . TGA curve 4.5 percent weight reduction in the temperature range 180 - 210° C which indicates that prismatic, platy and lamellar needle type crystals of strontium iodate are monohydrate. They are stable upto 180° C and dehydrate in the above temperature range forming an anhydrous strontium iodate compound.

$$Sr(IO_3)_2 \cdot H_2O \rightarrow Sr(IO_3)_2 + H_2O\uparrow$$

TGA curve indicates 20 percent weight reduction in the temperature range 50 - 100° C. Calculations shows that this weight reduction must be because of dehydration of hexa hydrate strontium iodate crystals.

$$Sr(IO_3)_2 \cdot 6H_2O \rightarrow Sr(IO_3)_2 + 6H_2O\uparrow$$

The anhydrous strontium iodate remains stable upto 590° C, at that point it decomposes in the temperature range 590 - 700° C, 58 percent weight loos in this temperature range predicts the decomposition reaction as

$$5\ Sr(IO_3)_2 \rightarrow Sr_5(IO_6)_2 + 4I_2\uparrow + 9O_2\uparrow$$

TGA curve of indicates comparative reaction in the temperature range 560 - 700° C. Chemical analysis of the residue in both the cases show presence of Sr and I in it. DTA curves show endothermic peaks when dehydration and decomposition happen in both the cases

QUALITATIVE ANALYSIS

Qualitative analysis is a method of Analytical chemistry that manages the determination of natural composition of inorganic salts. It is predominantly worried about the detection of ions in an aqueous solution of the salt.The basic procedure for testing any obscure example is to make its solution and test this solution with various reagents for the ions present in it. Testing with various reagents gives trademark reaction of specific ions, which might be a color change, a strong formation or some other noticeable changes. There are independent procedures for recognizing cations and anions, called the Cation Analysis and Anion Analysis.

Qualitative Analysis of Cations

Some preliminary tests need to be done before doing the analysis of cations.

(a) Physical Appearance: Colour and Smell

The physical examination of the obscure salt includes the investigation of color, smell and density. The test isn't a lot of reliable, however absolutely supportive in recognizing some coloredcations. Characteristicsmell assists with recognizing a few ions like ammonium ion.

(b) Charcoal Cavity Test

This test depends on the way that metallic carbonates when warmed in a charcoal cavity decompose to give comparing oxides. The oxides show up as colored incrustation or residue in the cavity. In specific cases, the oxides shaped mostly go through reduction to the metallic state creating metallic beads or scales.

Group V comprise of three radicals: Ba_2+, Sr_2+ and Ca_2+. These cations are precipitated as their carbonates. Group reagent for this group may be (NH4)2CO3 in the presence of NH_4Cl and NH4OH.

(c) Chemical Reaction involved in Group V Analysis

When $(NH_4)_2CO_3$ is added to salt solution containing NH_4Cl and NH_4OH, the carbonates of Ba^2+, Sr^2+ and Ca^2+ are precipitated.

$$BaCl_2 + (NH_4)_2CO_3 \rightarrow BaCO_3 + 2NH_4Cl$$

$$SrCl_2 + (NH_4)_2CO_3 \rightarrow SrCO_3 + 2NH_4Cl$$

$$CaCl_2 + (NH_4)_2CO_3 \rightarrow CaCO_3 + 2NH_4Cl$$

(d) Confirmation of Barium (II) ion (Ba^{2+})

The white precipitate of barium carbonate framed in the group analysis dissolves in hot dil. acetic acid because of the formation of dissolvable barium acetic acid derivation.

$$BaCO_3 + 2CH_3COOH \rightarrow (CH_3COO)_2Ba + CO_2\uparrow H_2O$$

Barium acetate

(a) Potassium chromate test Barium acetic acid derivation (shaped by dissolving barium carbonate in dil. acetic acid) reacts with potassium chromate to frame yellow precipitate of barium chromate.

$$(CH_3COO)_2Ba + K_2CrO_4 \rightarrow 2CH_3COOK + BaCrO_4\downarrow$$

Barium chromate (Yellow precipitate)

(b) Flame test Barium grants a verdant green color to the flame.

(e) Confirmation of Strontium (II) ion (Sr^{2+})

The white precipitate of strontium carbonate shaped in the group analysis dissolves in hot dil. acetic acid because of the formation of solvent strontium acetic acid derivation.

$$SrCO_3 + 2CH_3COOH \rightarrow (CH_3COO)_2Sr + CO_2\uparrow H_2O$$

Strontium acetate

(a) Ammonium sulfate test Strontium acetic acid derivation (shaped by dissolving Strontium carbonate in dil. acetic acid) reacts with ammonium sulfate to shape a white precipitate o strontium sulfate..

$$(CH_3COO)_2Sr + (NH_4)_2SO_4 \rightarrow 2CH_3COONH_4 + SrSO_4\downarrow$$

Strontium sulphate (White precipitate)

(b) Flame test Strontium imparts a ruby red color to the flame.

(f) Confirmation of Calcium (II) ion (Ca^{2+})

The white precipitate of calcium carbonate framed in the group analysis dissolves in hot dil. acetic acid because of the formation of solvent calcium acetic acid derivation.

$$CaCO_3 + 2CH_3COOH \rightarrow (CH_3COO)_2Ca + CO_2\uparrow H_2O$$

Calcium acetate

(a) Ammonium oxalate test Calcium acetic acid derivation (shaped by dissolving calcium carbonate in dil. acetic acid) reacts with ammonium oxalate to frame a white precipitate o calcium oxalate.

$$(CH_3COO)_2Ca + (NH_4)_2C_2O_4 \rightarrow 2CH_3COONH_4 + CaC_2O_4\downarrow$$

Calcium oxalate (White precipitate)

(b) Flame test Calcium grants block red color to the flame.

INFRARED SPECTRAL ANALYSIS

The measurement of infrared absorption spectra of mixes is finding - wide spread use since stable, business, infrared spectrophotometers have opened up The infrared spectrophotometer is likely the most generally utilized technique for distinguishing and recognizing obscure organic and inorganic mixes just as limited quantity of impurities and, for the elucidation of the structure of molecules. It is additionally utilized for quantitative measurements of concentration. At the point when a particle ingests radiation, its energy increments in proportion to the energy of the photon. The expansion in energy might be as electronic, vibration or rotational energy of the atom. On the off chance that the radiation is in the medium infrared region, both the vibration and rotational energy of the atom change. The infrared spectrum of the molecules results from transition among vibration and rotational energy levels

For little amplitude of vibration, the vibration is treated as harmonic and for a diatomic system, the recurrence of vibration of a particle can be communicated as

$$\nu = \frac{1}{2\pi c}\sqrt{\frac{F}{\mu}}$$

where c = velocity of light

F « force binding the vibrating atoms together

$$\mu = \frac{m_1 m_2}{m_1 + m_2}$$ = reduced mass of the system

For a nonlinear molecular system having particles more than two, the number of ordinary modes of vibration is equivalent to 3n-6 where n is the number of iotas in the atom. Every ordinary mode of vibration can happen autonomously of different modes, and the absorption band for every mode wherein the vibration quantum number changes by one is known as key band or central frequency.

Groups of weak power are gotten when vibration quantum number changes by more noteworthy than one. Such groups are known as suggestion groups. Also a few vibration modes can happen together which give combination ends

CONCLUSION

Opaque crystals of barium iodate and yellow colored transparent crystals of strontium iodate and calcium iodate have impurities of Si, Al, Fe, K, and so on. Gel inclusion is found in platy crystals of strontium iodate and in all habits of calcium iodate crystals. 2, Crystals of barium iodate, either transparent or opaque, are discovered monohydrate. Prismatic, platy and lamellar needles of strontium iodate are monohydrate, while prismatic pyramidal and prismatic needle type crystals are hexahydrate. Stable prismatic pyramidal crystals of calcium iodate are monohydrate, while prismatic and prismatic pyramidal crystals are hexahydrate. Monohydrate crystals of iodates of barium, strontium and calcium are stable. Then again, hexahydrate crystals of strontium iodate and calcium iodate are unstable even at room temperature as it were that they show the phenomenon of efflorescence

REFERENCES

1. S. Bhanumati, Int. News. Chem.Educ, 45, 11(1997) and references there in

2. F. Feigl, V. Anger and R. E. Oesper (1972). Spot Tests in Inorganic Analysis, G*^ edn, Elsevier Publishing Company, New York.

3. Vogel, Macro and Semimicro Qualitative Inorganic Analysis, edn, Orient Longman Ltd, New Delhi, 1953.

4. N. D. Cheronis, A. R. Ronzio and T. S. Ma (1954). Micro ande Semimicro Methods, Interscience Publishers, Inc., New York.

5. T. S. Ma and V. Horak (1976). Microscale Manipulations in Chemistry, John Wiley and Sons, New York.

6. Z. Szafran, R. M. Pike and M. M. Singh (1991). Microscale Inorganic Chemistry: A Comprehensive Laboratory Experience, Wiley, New York.

7. D. W. Mayo, R. M. Pike, S. S. Butcher and P. K. Trumper, Microscale Organic Chemistry, 3'* edn, Wiley, New York, 1995.

8. S. L. Kelkar and D. D. Dhavale, Report On The Scheme Sanctioned Under Innovative Programme "Development of Microscale Experiments in Chemistry", University of Pune, Pune, 1996.

9. Knoll, G. F. (2000). Radiation Detection and Measurement. Wiley. p. 505. ISBN 978-0-471-07338-3.

10. Padmanabhan, T. (2001). "Stellar Nucleo synthesis". Theoretical Astrophysics, Volume II: Stars and Stellar Systems. Cambridge University Press. pp. 230–236. ISBN 978-0-521-56631-5.

11. Rolo of. “Wichtige Nachrichtfür Aerzte und Apoteker – Entdeckungeines Arsenikgehalts in der Zinkblume und des Zinkvitriols in Tartarusvitriolis". Journal des practischen Arzneykunde und Wundarzneykunst (Hufelands Journal) (2 Februar Stück): pp. 110.

12. Hermann, C. S. (1818). "Nocheinschreibenüber das neueMetall". Annalen der Physik. 59 (5): pp. 113–116.

13. Bibcode:1818AnP....59..113H. doi:10.1002/andp.18180590511.

14. Waterston, W.; Burton, J. H. (1844). Cyclopædia of commerce, mercantile law, finance, commercial geography and navigation. H. G. Bohn. p. 122.

15. Crenshaw, M.A. (1972). Inorganic composition of molluscanextrapallial fluid. Biological Bulletin 143: pp. 506-512.

16. Dauphin, Y., Cuif, J.P., Doucet, J., Salomé, M., Susini, J. and Williams, C.T., 2003. In situ mapping of growth lines in the calcitic prismatic layers of mollusc shells using X-ray absorption near-edge structure (XANES) spectrometry at the sulphur K-edge. Mar. Biol., 142, pp. 299-304.

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