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CHEMICAL COMPOSITION AND POSSIBLE INDUSTRIAL APPLICATIONS OF CELESTITE FROM THE LOCH LOMOND AREA OF NOVA SCOTIA

Prepared
by

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SUMMARY

A 150 pound sample from the celestite deposits in the Loch Lomond area was received from the Lura Corporation Ltd. on December 4, 1967.

The sample was found to contain 87.43% of strontium sulfate, 0.40% of barium sulfate, 0.36% of calcium sulfate and 11.81% of combined water, carbonates, silica, alumina and other impurities.

Partial separation of silicates by flotation increased the content of strontium sulfate to about 95%.

By treatment with hot 5% hydrochloric acid the sample was upgraded to contain about 98.6% of strontium sulfate, 0.5% of barium sulfate and less than 1% of impurities.

Strontium carbonate and other strontium compounds were prepared from the sample by the usual chemical procedures. Chemical purity of the products depended on the treatment of the celestite before chemical processing and on subsequent chemical purification of the products. For the preparation of technical grade strontium salts, the upgrading of celestite and the purification of the products was necessary in a minor extent only and for some applications it was not necessary.

A part of the original sample, without any upgrading or purification, was used for preparation of strontium carbonate. Strontium hexaferrite prepared from this strontium carbonate was suitable for fabrication of permanent ceramic magnets.

According to the results of chemical analyses and other tests and experiments performed in the course of this investigation, the deposits of celestite represented by the samples received from the Lura Corporation Ltd. on December 4, 1967 are recommended as a source of strontium sulfate suitable

for preparation of other strontium compounds.

The economic aspects of a potential commercial exploitation of the celestite deposits were not studied in the course of this investigation and no recommendation on these aspects can be given.

IDENTIFICATION

This investigation was performed by the Laboratory for Investigation of Minerals at the Nova Scotia Technical College, Halifax, Nova Scotia.

The investigation was requested by the Deputy Minister of Mines, Dr. J. P. Nowlan, Nova Scotia Department of Mines.

Advisory body: Committee on Laboratory Investigation of Minerals:
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E. F. J. Flemming
Dr. K. V. Gow
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1. INTRODUCTION

1.1. General

The purpose of this investigation was to evaluate the quality of the deposits of the mineral celestite reported from the Loch Lomond area in Cape Breton, Nova Scotia. This qualitative evaluation should include a complete chemical analysis of a representative sample of the deposits and a tentative exploration of the suitability of these deposits for manufacturing of strontium compounds.

The mineral celestite is basically strontium sulfate, often accompanied by variable amounts of impurities. As strontium in the form of strontium sulfate has a very limited scope of direct practical uses only, it is necessary to transform strontium sulfate to some other, more suitable strontium compound, usually strontium carbonate of the desired degree of purity. Strontium carbonate then can be applied for some purposes directly as such, or it can be easily transformed to other strontium compounds. Preparation of pure strontium carbonate from the Loch Lomond celestite was the major objective of this investigation.

Special features of this investigation were the preparation of strontium hexaferrite from celestite and the testing of the antiscumming effect of strontium nitrate in fired clays. The application of celestite for these two purposes allowed a fairly large amount of impurities, which, in the case of the hexaferrite, were reported to have a beneficial effect. This was contrary to the preparation of strontium carbonate and other strontium compounds in which all impurities were definately undesirable.

The investigation was limited to laboratory tests only as it was not supposed to include evaluation of the economic aspects of a potential commercial exploitation of the celestite deposits. The geological survey of the deposits was done by the Lura Corporation Ltd., the results of the survey are not a part of this report.

1.2. Brief history of strontium chemistry

Strontium was discovered as the last one of the three alkaline earths elements, calcium - strontium - barium. The most abundant of the three elements, calcium, was known and used in the form of calcium oxide as a mortar by Egyptians and later by Romans, the name calcium was derived from the Latin word "calx" meaning "lime". Heavy spar containing barium was discovered at the beginning of the 17th century by the Italian alchemist Casciorolus who called it "lapis solaris" because of its glow when heated. Galla mentioned the same mineral in 1612. The name barium orginated from the Greek "barys", i.e. "heavy".

Strontium remained undiscovered until 1790 when Crawford, a Scottish physician, was trying to prepare barium chloride from a local mineral which was believed to be identical with witherite (barium carbonate, discovered by Withering in 1783). As the chloride prepared by Crawford was distinctively different from barium chloride, both in its water solubility and in the appearance of the crystals, presence of a new alkaline earth had to be assumed. A name for the new mineral was suggested by Sulzer in a letter to Blumenbach in 1791, the suggestion was "strontianite", after the name of the locality where it was found for the first time, the town of Strontian in Scotland. However, a certain degree of confusion between strontianite and witherite prevailed until 1797 when Pelletier made a positive distinction between the two minerals.

The mineral celestite, strontium sulfate, was discovered by Klaproth in 1797. Some deposits of celestite were pale blue in color, the name celestite was derived from the Latin word "caelestis" meaning "heavenly".

Berthier prepared strontium sulfide by reduction of strontium sulfate with carbon in 1823, and in 1855, Rose prepared strontium carbonate from strontium sulfate by conversion in an alkali carbonate solution. Industrial production of strontium carbonate and other strontium compounds still is, in major part, based on these two reactions.

1.3. Application of strontium compounds

The major part of strontium is used by the industry in the form of strontium compounds. Only a minor part is used as strontium metal, either pure or in the form of alloys with other metals.

Strontium carbonate and strontium nitrate are the two most important strontium compounds manufactured from celestite, strontium chloride ranks third in importance while other strontium salts are manufactured and used in minor quantities only.

By far the largest part of the whole production of strontium salts is used in pyrotechnics, flares and fireworks. This is based on the unique ability of strontium to impart a brilliant crimson color to a flame.

Strontium compounds are used also:
as additives to glasses, enamels and ceramics,
as additives to dehydrogenation catalysts,
as additives to greases and plastics,
in medicine (strontium is relatively harmless compared with the highly toxic soluble barium salts), and
for preparation of phoshorescent materials based on strontium sulfide.

Two relatively new fields for industrial application of strontium are the preparation of strontium hexaferrite and the use of strontium carbonate or strontium nitrate as an antiscumming agent in fired clays.

One major potential application of strontium salts is the production of strontium titanate, now used only in minor amounts as an additive modifying the electrical properties of barium titanate. Stoichiometric strontium titanate is an insulator while partly reduced strontium titanate is a semiconductor which at very low temperatures becomes superconductive. A large scale use of superconductors for transfer of electrical energy may become feasible in future.

Strontium metal and its alloys are used for removing traces of gas from vacuum tubes and as a scavenger in metallurgy.

2. CELESTITE FROM THE LOCH LOMOND AREA

2.1. Chemical composition

The results of all chemical analyses and other tests and experiments reported here apply only to the sample received from the Lura Corporation Ltd. on December 4, 1967. According to a letter from the Consulting Geologist, Dr. J. H. Morgan, of December 19, 1967 the sample represented sampling of celestite deposits in the Loch Lomond area.

From the original 150 pound sample consisting mainly of large rocks weighing between one and five pounds, a representative sample was obtained by crushing and quarting. A five pound lot of the representative sample was dried and ground to pass a 100 mesh sieve. This final sample was used for all chemical analyses and other tests and experiments in the course of this investigation.

Results of chemical analysis:

Loss on Ignition at 1000°C	2.11%	
SiO_2	6.70%	
Fe_2O_3	0.34%	
Al_2O_3	1.61%	
CaO	0.15%	
SrO	49.31%	(equivalent to 87.43% $SrSO_4$)
BaO	0.26%	
MgO	0.15%	
Na_2O	0.13%	
K_2O	0.16%	
SO_3	38.83%	
P_2O_5	0.01%	

Minor amounts and traces detected by spectrographic analysis:
Ni, Mn, Pb, B, Ti, Ag.

2.2. Upgrading

Two methods of upgrading were tested on the sample:

(a) flotation with water - to separate the low density impurities from the havier strontium and barium sulfates, after this treatment the sample was dried and analyzed,

(b) chemical treatment - 100 grams of the sample after flotation was stirred in one liter of 5% hydrochloric acid at 95 - 100°C for one hour, then the sample was decanted, washed with water, dried and analyzed.

Results of chemical analyses:

	original sample	after flotation	after chemical treatment
$SrSO_4$	87.43%	95.04%	98.56%
$BaSO_4$	0.40%	0.43%	0.47%
other	12.17%	4.53%	0.97%
YIELD	100%	86%	79%
		100%	92%

2.3. Conversion to strontium carbonate

The conversion of strontium sulfate to strontium carbonate is based on the difference in their respective solubilities.

	$SrSO_4$	+ Na_2CO_3	= $SrCO_3$	+ Na_2SO_4
solubility in 100 ml water at 20°C	0.0113g	30g	0.0011g	48g

Potassium carbonate, ammonium carbonate or ammonia and carbon dioxide under pressure may be used instead of sodium carbonate. The byproducts of the conversion then are potassium sulfate or ammonium sulfate. The marketability of the byproducts would be an important consideration in commercial evaluation or various reagents for conversion.

Experimental:

233 grams of sodium carbonate was dissolved in 4 liters of water, 420 grams of celestite powder (original sample without any upgrading) was added and the mixture was stirred and heated continuously. The degree of conversion was checked in five-minute intervals by titration of the unreacted sodium carbonate in 5 ml samples taken out from the mixture. An equilibrium at 80°C was reached after 65 minutes (X). The heating was discontinued and the mixture was stirred until it cooled to room temperature, then it was allowed to sediment. The clear solution was decanted and the insoluble part was stirred in 4 liters of water, let to sediment and decanted. This was repeated until the pH of the solution was less than 7.5.

It was observed that the insoluble part divided into two different fractions during sedimentation, a whitish coarse fraction sedimenting very fast and a beige very fine fraction sedimenting slowly. By repeated decantation it was possible to separate the fine fraction from the coarse fraction almost quantitatively. After drying at 105°C both fractions were weighed and analyzed.

(X) Note: Kinetics of this reaction could not be studied within the very limited scope of this investigation. Such a study would be both interesting theoretically and important for industrial application. The study should include the effects of the grain size of cellestite, the $NaCO_3/SrSO_4$ ratio, the reaction temperature and the concentration.

Results of chemical analyses:

	fine fraction	coarse fraction
Loss on Ignition at 1000°C	23.05%	26.13%
SiO_2.....	7.78%	4.56%
Fe_2O_3.....	0.27%	0.16%
Al_2O_3.....	2.35%	1.25%
CaO.....	0.49%	0.52%
SrO.....	53.81%	56.94%
BaO.....	0.97%	0.68%
MgO.....	0.32%	0.40%
Na_2O.....	0.82%	0.81%
K_2O.....	0.13%	0.11%
SO_3.....	5.28%	3.98%
YIELD (100 parts of celestite).....	42.8	and 37.4 parts

2.4. Purification of strontium carbonate

Two modifications of a method of purification were tested. The first modification included the following four steps:

(1) 100 grams of strontium carbonate prepared by the conversion of celestite as described in 2.3. was mixed with one liter of water. The mixture was stirred continuously while hydrochloric acid was added to the mixture drop by drop, until the development of carbon dioxide ceased. The stirring was continued and the mixture was heated to about 80°C. The acidity of the solution was kept around pH 3 by further additions of hydrochloric acid when necessary. The heating and stirring was discontinued after ten minutes and the mixture was allowed to sediment. The solution was decanted and filtered.

The purpose of this step was to separate the major part of unreacted sulfates and insoluble silica and silicates.

(2) The clear filtrate from this step (1) was heated to about 70°C. The solution was stirred continuously and ammonium hydroxide was added slowly until the pH 7 - 8 was reached. Stirring and heating was continued for about five minutes, then the precipitate was let to sediment and the solution was filtered.

The purpose of the step (2) was to separate the aluminum and iron from their soluble salts and all soluble phosphates.

(3) The concentration of calcium was determined in a small sample of the filtrate resulting from the step (2) and the theoretical amount of ammonium oxalate, necessary for precipitation of the calcium present in the whole amount of the filtrate, was calculated. 150% of the theoretical amount of ammonium oxalate was dissolved in a small amount of water and the solution was added to the filtrate preheated to about 80°C. The mixture was stirred and heated for five minutes and then it was let to sediment at room temperature overnight. The clear solution was decanted and filtered.

The purpose of the step (3) was to separate the major part of calcium. Approximately 60% of the calcium was separated by the oxalate precipitation described above. However, after filtration it was possible to repeat the step (3) as many times as necessary to reduce the calcium content to the desired level.

(4) The clear filtrate from the step (3) was mixed with saturated solution prepared from 100 grams of ammonium carbonate. The mixture was stirred and heated to 50°C and then it was let to sediment. The clear solution was decanted and the precipitate was stirred in one liter of water containing about 5 grams of ammonium oxalate. The sedimentation, decantation and stirring in water with ammonium oxalate was repeated three times. Finally, the precipitate was separated by filtration and dried by heating at 400°C for one hour.

This modification of the purification method was tested separately on both fractions of the strontium carbonate prepared by conversion of celestite and no significant differences in the composition of the purified strontium carbonate were found.

Strontium carbonate purified by this method contained in average:

$SrCO_3$.....	98.54%	
$CaCO_3$.....	0.59%	(after only one precipitation with ammonium oxalate)
$BaCO_3$.....	0.38%	
$MgCO_3$.....	0.07%	
Na_2CO_3.....	0.24%	
other impurities	0.18%	

By repetition of the whole purification process, including an addition of sulfuric acid in the amount necessary to precipitate barium during the step (1), it should be possible to prepare strontium carbonate better than 99.5% pure.

The purity of strontium carbonate prepared by the first modification described above was much higher than the purity necessary for many technical applications of strontium carbonate. Therefore, a simplified method, the second modification, was devised, which should, in a faster and cheaper way, produce strontium carbonate still pure enough for technical applications. The second modification was based on the steps (1), (2) and (4) as described in the first modification, but the filtration in the step (1) was omitted and the ammonium hydroxide precipitation followed immediately after the treatment with hydrochloric acid, without separation of the undissolved matter. The mixture was filtered only after the precipitation with ammonium hydroxide in the step (2) and the clear filtrate was then treated as described in the step (4). The step (3) was omitted completely.

This simplified modification was tested on a mixture of both fractions of the strontium carbonate resulting from the conversion of celestite.

Strontium carbonate purified by the second (simplified) modification contained:

$SrCO_3$	96.64%
$CaCO_3$	2.44%
$BaCO_3$	0.40%
$MgCO_3$	0.07%
Na_2CO_3	0.25%
other impurities	0.20%

Note: No attempts have been made to study in detail the composition of the "other impurities" included in the analytical results on page 9 and on page 10. However, it was established that the content of either SiO_2, Al_2O_3, Fe_2O_3 or SO_3 in any of the purified strontium carbonate samples was less than 0.05%.

2.5. Reduction of celestite to strontium sulfide

The method of preparation of barium sulfide by the reduction of barium sulfate with carbon is well known and it is used in a large scale by industry. It was assumed that the same technology could be applied to celestite for commercial preparation of strontium sulfide. Under this assumption, and because of the limited time which could have been devoted to this investigation, the reduction of celestite and the subsequent treatment of the strontium sulfide was tested in a few small scale runs only. The data obtained from these tests were sufficient for a general qualitative evaluation of the method, but no attempts for a quantitative evaluation could be made on that basis.

The reaction

$SrSO_4 + 4\ C = SrS + 4\ CO$

proceeded smoothly when a mixture of celestite and carbon powders was heated in a covered crucible on a Bunsen burner. In all tests 50% excess of carbon over the theoretical amount was applied.

The reaction seemed to proceed faster when coal dust (origin unknown) was used instead of the pure carbon prepared from sucrose.

The mixture resulting from the reduction contained strontium sulfide and small amounts of unreacted sulfates and the impurities introduced with celestite and coal dust. The mixture was cooled to room temperature and then it was transferred into an Erlemeyer flask and mixed with water in the ratio of 10 ml of water for each gram of celestite used for reduction.

Two reactions of strontium sulfide with water took place, at room temperature the prevailing reaction was:

$2\ SrS + 2\ H_2O = Sr(HS)_2 + Sr(OH)_2$

and on heating

$Sr(HS)_2 + 2\ H_2O = Sr(OH)_2 + 2\ H_2S$

The mixture was boiled until the development of hydrogen sulfide ceased. The hot solution of strontium hydroxide was separated from the insoluble matter by filtration.

The solution was used either

(a) directly for preparation of strontium hexaferrite (see 3.2.),

(b) for preparation of strontium carbonate by precipitation with ammonium carbonate as described in the step (4) on page 9, or

(c) for preparation of strontium chloride or strontium nitrate by neutralization of the strontium hydroxide solution with the mineral acid, after which the solution was evaporated to the saturation point and the salt was separated by crystallization.

Strontium carbonate prepared by this method contained in average (semiquantitative spectroscopic analyses) 92% of strontium carbonate, 3% of calcium carbonate, less than 2% of barium carbonate and more than 3% of impurities, mainly alumina and silica.

It is possible to precipitate strontium carbonate directly from the strontium hydroxide solution without a preliminary separation of the insoluble matter. All these insoluble impurities are then contained in the product (for a typical chemical analysis see reference 4.)

Strontium chloride and strontium nitrate prepared from the filtered strontium hydroxide solution contained relatively less calcium and aluminum impurities than the carbonate and after recrystallization their purity increased to over 96% of the strontium salt.

Note: The possibility to utilize the hydrogen sulfide should be seriously considered in any economic study of a potential industrial application of this method.

3. APPLICATIONS

3.1. General considerations

There was not appreciable domestic production of strontium minerals in United States and Canada since 1959. Practically all requirements for strontium compounds were covered by import of celestite from United Kingdom, Mexico and Spain. The total amount of the celestite imported to United States flucuated between 9,000 and 21,000 short tons annually. Strontium carbonate and strontium nitrate were the principal compounds manufactured from the imported celestite and sold by producers.

Considering these facts, domestic production of celestite and manufacturing of strontium compounds seems desirable and commercially feasible.

The celestite from the Loch Lomond area may be either

(a) marketed as a raw material, or

(b) used for production of strontium carbonate and other strontium compounds by any of the methods described in 2.3., 2.4. and 2.5.

Two other possibilities are discussed in 3.2. and 3.3.

The sample of celestite did not meet the specification of the largest consumer, E. I. du Pont de Nemours & Co., Inc., Grasselli, N.J., namely minimum 95% of strontium sulfate, maximum 2% of calcium sulfate, maximum 2% of calcium sulfate, maximum 2% of barium sulfate and maximum 1% of moisture. This specification was easily met after upgrading of the sample as described in 2.2.

One of the large consumers of strontium carbonate is the Pemco Corporation in Baltimore, Maryland. Their specification for strontium carbonate requires:

$SrCO_3$	minimum	90.0%
$BaCO_3$	maximum	1.5%
$CaCO_3$	maximum	4.00%
Insoluble in HCl	maximum	2.50%
Total alkali	maximum	1.50%
SO_3	maximum	0.50%
Fe_2O_3	maximum	0.15%

From a comparison of these values with the results reported in Chapter 2. it is obvious that the specification can be met only after a certain degree of purification of the strontium carbonate prepared from the original celestite by

either of the two tested methods, i.e. by conversion or by reduction. Upgrading of the celestite before conversion or reduction should make the subsequent purification of strontium carbonate unnecessary.

3.2. Preparation of strontium hexaferrite

Strontium hexaferrite was reported to be superior to barium hexaferrite as a material for production of permanent magnets. The only major reason why strontium hexaferrite was not produced in larger quantities seemed to be the price of strontium carbonate, which was much higher than the price of barium carbonate.

Impurities contained in some deposits of celestite were believed to improve the magnetic properties of strontium hexaferrite prepared from strontium carbonate in which the impurities, and a small amount of unreacted celestite, had been left. To test the Loch Lomond celestite from this point of view, several batches of strontium hexaferrite were prepared.

In an attempt to avoid any duplication of the preparation methods already patented, the following preparation procedure was used:

The coarse fraction of the strontium carbonate prepared by conversion was mixed with ferric oxide in the ratio of 5.75 moles of ferric oxide per one mole of the combined strontium + barium + calcium carbonates according to analysis. The mixture was precalcined at 900 - 1000°C for one hour and after cooling it was ground to pass 100 mesh. The powder was calcined at 1200°C for one hour and the crushing and grinding was repeated. Finally, the calcined powder was sintered at 1300°C for two hours and the hexaferrite was crushed to pass 30 mesh. Further testing, including ball milling, pressing in magnetic field, firing and measuring of magnetic properties, was done by the Mineral Sciences Division of the Department of Energy, Mines and Resources in Ottawa.

The samples for testing were processed in only four different combinations of the milling time and firing temperature and for this reason the results of magnetic meaxurements cannot be considered as conclusive. The results were $(BH)_{max} = 2.9 \times 10^6$ G.Oe, $B_R = 3650$ G and ${}_iH_C = 2100$ Oe. Samples prepared under the optimum processing conditions, which could not be found within the small number of tested combinations, should exhibit better magnetic properties.

The solution of strontium hydroxide prepared by hydrolysis of strontium sulfide, as described in 2.5., was applied as a source of strontium for preparation of strontium hexaferrite too. The solution was mixed with ferric oxide and the mixture was dried. Further processing was the same as in the method with strontium carbonate.

The developmeng of the magnetoplumbite structure was checked by X-ray diffraction analysis. Magnetoplumbite was the only phase identified in the samples after sintering at 1300°C, no foreign phases were found. The same results were obtained on the samples prepared from the coarse fraction of strontium carbonate as on the samples prepared from the hydroxide solution.

3.3. Antiscumming effect of strontium compounds on fired clays

Barium carbonate is used as an additive to clays for fabrication of face bricks and other fired clays where the preservation of the bright color is desirable. Barium carbonate forms barium sulfate with the sulfates present is the clay, this prevents the formation of the greyish white scum when the clay is fired.

Laboratory tests with strontium confirmed our speculation that the antiscumming effect of strontium would be similar to that of barium.

A solution of strontium nitrate was prepared from the strontium carbonate and added to a face-brick clay in concentrations equivalent to additions of barium carbonate in the range from 0 to 34 pounds per 1000 bricks. The samples were fired at 1125°C.

A distinctive difference was observed between the color of the sample with no strontium added and the color of the samples with strontium, the color of the former being dull because of a thin deposit of scum. No difference was observed between the color of the samples to which the strontium equivalent of 8.5 or more pounds of barium carbonate per 1000 bricks was added.

The prices of strontium carbonate and strontium nitrate are higher than the prices of barium salts, from this point of view the use of strontium compounds as antiscumming agents is not attractive. However, this may be a profitable possibility for utilization of the low grade strontium batches resulting as byproducts from production of higher purity strontium compounds. Impurities like iron, aluminum and silica would not impair the effect of strontium and barium, the only undersirable impurities from celestite would be calcium sulfate and alkali salts.

Arrangements for a series of large scale tests in a plant of the L. E. Shaw Ltd., Halifax, were not completed at the time of this report.

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