A STUDY OF SODIUM LANTHANUM CARBONATES

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A STUDY OF SODIUM LANTHANUM CARBONATES

By

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A THESIS

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INTRODUCTION

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The literature is lacking of information on sodium lanthanum carbonate. This research problem has been concerned mainly with the solubility of lanthanum carbonate in sodium carbonate solutions and the preparation of sodium lanthanum carbonate. The dehydration of lanthanum carbonate was also studied.

Lanthanum carbonate occurs in nature as lanthanite, $La_2(CO_3)_3 \cdot 8H_2O$ (15). Rare earth carbonates have been prepared by three methods, two of which are not satisfactory for pure crystalline compounds. Precipitation of rare earth carbonates by the addition of alkali carbonates (16, 17) or alkali bicarbonates (12) to rare earth salt solutions yields a product usually contaminated with alkali carbonates. A second method, that of passing a stream of carbon dioxide through a suspension of rare earth hydroxide in water, was used by Cleve (2, 3). The rare earth carbonates obtained contain hydroxide impurities due to incomplete conversion of one solid to another. A more recent method is that of Salutsky (13). In the latter study pure rare earth carbonates were prepared by decomposition of rare earth trichloroacetates from hot water solutions. Since no interfering ions are introduced, pure normal carbonates are obtained. The work is based on the original observations by Dumas (4) that trichloroacetates in hot water solution decompose to liberate chloroform and carbon dioxide. Kinetic studies for the reaction were made by Verhoek (18, 19).

Meyer (9) found that rare earth carbonates form double salts with alkali carbonates. While the ammonium and sodium double carbonates were found to be rather insoluble, the potassium salt was readily soluble in concentrated potassium carbonate. Differences in degree of solubility of the potassium double carbonates have been used by various investigators (1, 8, 9, 10) to obtain pure lanthanum carbonate. Meyer obtained $K_2La_2(CO_3)_4 \cdot 12\ H_2O$, $(NH_4)_2La_2(CO_3)_4 \cdot 4\ H_2O$, and $Na_6La_4(CO_3)_9 \cdot 20\ H_2O$ upon the addition of concentrated lanthanum salt solutions to the respective alkali carbonate solutions. Sodium lanthanum carbonate, a dense gelatinous precipitate would not crystallize even after weeks of contact with the mother liquor. Furthermore, it was observed that the formula given for the sodium salt could not be proven because of its instability.

EXPERIMENTAL

EXPERIMENTAL

I. Solubility of Lanthanum Carbonate in Sodium Carbonate

A study was made to determine the solubility of lanthanum carbonate in sodium carbonate solutions and the solid phases thus formed.

Lanthanum carbonate was prepared by decomposition of lanthanum trichloroacetate in a hot water solution. Approximately 15 g. of lanthanum oxide was dissolved in slight excess of 25 per cent trichloroacetic acid. The solution was diluted to 750 ml. and heated on a steam bath for 10 hours after the first carbonate appeared. After cooling to room temperature the solution was filtered with suction. The precipitate was washed with water and air dried.

Experimental determinations of the per cent lanthanum oxide and per cent carbon dioxide were made. Weighed samples of the carbonate were ignited in a muffle furnace at 950°C. to obtain the per cent lanthanum oxide. The per cent carbon dioxide was determined by treating weighed samples in a gas evolution apparatus (11). The carbon dioxide liberated by dilute hydrochloric acid was collected in ascarite filled absorption tubes and weighed. The per cent water was determined by difference ($100\% - \%\ La_2O_3 - \%\ CO_2$).

The per cent composition thus obtained was La_2O_3, 57.41; CO_2, 23.21; and H_2O, 19.38. From the per cent composition were calculated the mole ratios $\frac{CO_2}{La_2O_3}$, 3.00; and $\frac{H_2O}{La_2O_3}$, 6.11.

Solutions which were 1 M., 2 M., and 3 M. in sodium carbonate were prepared and placed in 50 ml. mixing cylinders. To each was added sufficient $La_2(CO_3)_3 \cdot 6.11\ H_2O$ to make the solutions 0.1 M. in $La_2(CO_3)_3$. The cylinders were placed in a thermostat at 25°C. and rotated for six days. Duplicate samples were prepared and rotated for 18 days. After the above mentioned times had passed the cylinders were removed and the solutions filtered with suction. The unwashed solid phases were air dried.

Portions, 25 ml., of each filtrate were acidified with nitric acid and evaporated to dryness. The solid was dissolved in the minimum amount of distilled water, heated to boiling, and oxalic acid added to precipitate any lanthanum in solution. The solutions remained water clear in all cases, indicating insufficient lanthanum in solution to precipitate as lanthanum oxalate.

Analyses were made to determine the solid phases formed. The per cent carbon dioxide was obtained by the gas evolution method used to analyze the lanthanum carbonate. The resulting hydrochloric acid solutions were evaporated to dryness and converted to nitrate solutions which were diluted, heated to boiling, and oxalic acid added. The precipitated lanthanum oxalate was well washed to remove any sodium oxalate (7). Ignition of lanthanum oxalate at 950°C. yielded lanthanum oxide. Nitric acid was added to the filtrates from the lanthanum oxalate precipitations and the solutions evaporated to dryness. The resulting nitrate residues were dissolved in distilled water and converted to the sulfate by addition of sulfuric acid and evaporation.

After evaporation to dryness the residues were ignited at 900°C. (6). The resulting sodium sulfate was weighed and the per cent sodium oxide calculated.

From the per cent composition were calculated the mole ratios $\frac{CO_2}{La_2O_3}$ and $\frac{Na_2O}{La_2O_3}$. The results indicated normal carbonates;* thus the per cent water was found by difference and the ratio $\frac{H_2O}{La_2O_3}$ calculated. The results obtained are listed in Table I, and will be considered in the discussion.

TABLE I

COMPOSITION OF SOLID PHASES FROM SOLUBILITY STUDY

Conc. of Na_2CO_3	Time Rotated	% La_2O_3	% Na_2O	% CO_2	% H_2O	$\frac{Na_2O}{La_2O_3}$	$\frac{CO_2}{La_2O_3}$	$\frac{H_2O}{La_2O_3}$
1 M.	6 days	45.64	9.05	25.02	19.27	1.04	4.06	7.64
1 M.	18 days	46.57	9.16	25.63	18.64	1.03	4.08	7.25
2 M.	6 days	36.93	18.04	27.87	17.16	2.57	5.59	8.41
2 M.	18 days	37.57	19.90	27.94	14.59	2.79	5.51	7.03
3 M.	6 days	37.40	24.61	31.80	6.19	3.46	6.30	3.00
3 M.	18 days	37.06	24.50	31.71	6.73	3.48	6.34	3.29

Approximately 1 g. samples of the double carbonate obtained from the 2 M. and 3 M. sodium carbonate solutions were each added to 250 ml. of distilled water and stirred for 24 hours. The precipitates were allowed to settle, washed with distilled water, filtered by suction, and air dried. Analyses yielded results of $\frac{CO_2}{La_2O_3}$ ratios of 2.98 and 3.01,

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* Double

indicating the complete removal of sodium carbonate and the formation of normal lanthanum carbonate, $La_2\cdot(CO_3)_3\cdot x H_2O$.

II. Preparation of Sodium Lanthanum Carbonate

Sodium lanthanum carbonate was prepared by two methods: decomposition of sodium trichloroacetate and lanthanum trichloroacetate in hot water solutions, and the addition of lanthanum trichloroacetate solution to sodium carbonate solution.

Solutions containing 2 g., 10 g., 30 g., and 50 g., respectively, of sodium carbonate dissolved in the stoichiometric amount of trichloroacetic acid were prepared. To each was added a lanthanum trichloroacetate solution containing 4 g., of lanthanum oxide dissolved in slight excess trichloroacetic acid. Each volume was diluted to 600 ml. and the solutions warmed on a steam plate at 80°C. for 24 hours after the first precipitate appeared. At first the white precipitate formed slowly and settled rapidly; but during the last 12 hours of heating a gelatinous white foam was produced. The solutions were allowed to stand at room temperature for 24 hours, the precipitates settling completely in this time as finely divided crystalline solids.

When filtered by suction each precipitate was divided, one-half being thoroughly washed with distilled water and the other half left unwashed. Analyses of the air dried solids were made in the same manner as in the solubility study experiments. Results of these analyses are given in Table II. Washing with water decreased the $Na_2CO_3:La_2(CO_3)_3$

ratio in all cases of the double carbonate. The double salt was not precipitated from the solution lowest in sodium ion concentration.

TABLE II

COMPOSITION OF PRECIPITATES FROM DECOMPOSITION OF SODIUM TRICHLOROACETATE LANTHANUM TRICHLOROACETATE SOLUTIONS

Filtration Conditions	Grams Na_2CO_3 Added	% La_2O_3	% Na_2O	% CO_2	% H_2O	$\frac{Na_2O}{La_2O_3}$	$\frac{CO_2}{La_2O_3}$	$\frac{H_2O}{La_2O_3}$
Unwashed	2	52.53	*	21.46	26.01	*	3.03	8.97
Washed	2	53.79	*	21.91	24.30	*	3.02	8.18
Unwashed	10	52.62	7.23	26.29	13.86	0.72	3.70	4.77
Washed	10	55.52	5.07	26.01	13.40	0.48	3.47	4.37
Unwashed	30	48.75	10.03	28.11	13.11	1.08	4.27	4.87
Washed	30	55.63	10.01	29.31	5.05	0.95	3.90	1.64
Unwashed	50	43.61	12.01	27.36	17.02	1.45	4.65	7.07
Washed	50	56.35	10.01	29.76	3.88	0.93	3.91	1.25

* Per cent Na_2O assumed negligible since normal $La_2(CO_3)_3 \cdot x\ H_2O$ formed.

Attempts to decompose sodium trichloroacetate lanthanum trichloroacetate solutions at 95°C. brought about sudden effervescense and foaming when the first precipitate appeared in the water clear solutions. This resulted in substantial loss of both solution and solid.

In the second method of obtaining sodium lanthanum carbonate, 15 g. of lanthanum oxide was dissolved in slight excess of 25 per cent trichloroacetic acid and the solution added slowly, with constant stirring, to one liter of 2 M. sodium carbonate. A gelatinous white precipitate was formed immediately. The solution was heated on a steam plate at 80°C. for eight hours and allowed to stand at room temperature for 48 hours. Although the precipitate settled completely in this time, little solid settled during the first 24 hours.

One half of the precipitate was washed with distilled water and the other with three per cent sodium carbonate. Both portions were filtered by suction and air dried.

Analyses were made as in the previous experiments, the results being listed in Table III. Using different wash solutions produced a noticeable difference in the $Na_2CO_3:La_2)CO_3)_3$ ratios.

TABLE III

COMPOSITION OF PRECIPITATE FROM MIXING LANTHANUM TRICHLOROACETATE AND SODIUM CARBONATE

Wash Solution	% La_2O_3	% Na_2O	% CO_2	% H_2O	$\frac{Na_2O}{La_2O_3}$	$\frac{CO_2}{La_2O_3}$	$\frac{H_2O}{La_2O_3}$
Distilled H_2O	47.33	15.83	30.93	5.91	1.76	4.84	2.26
3% Na_2CO_3	42.55	20.26	32.18	5.01	2.50	5.60	2.13

A sample of 5 g. of the sodium lanthanum carbonate washed with distilled water (Table III) was added to 500 ml. of distilled water and stirred for 24 hours. The solution was allowed to settle for 24 hours and filtered by suction while washing with distilled water. Analyses of the solid for lanthanum oxide and carbon dioxide gave a $\frac{CO_2}{La_2O_3}$ ratio of 2.97:1 indicating decomposition to normal lanthanum carbonate, $La_2(CO_3)_3 \cdot x H_2O$.

III. Dehydration of Hydrated Lanthanum Carbonate

The effects of temperature and drying agents on hydrated lanthanum carbonate were studied. The lanthanum carbonate used in these studies was prepared by decomposition of lanthanum trichloroacetate in hot water solution. The per cent composition of the air dried carbonate was obtained by the same methods as used for the lanthanum carbonate in the solubility study experiments. The results are listed in Table IV.

Samples of approximately 4 g. of the above carbonate were heated under various conditions. Analyses of the resulting solids were made in the same manner as before. The per cent composition and mole ratios obtained in this study are found in Table IV. While a temperature of 250°C. was found to completely dehydrate the carbonate, it is entirely possible that more lengthy heating at 200°C. would produce anhydrous lanthanum carbonate.

A sample of the carbonate inadvertently heated at 400°C. lost more weight than that calculated as the water of hydration. This indicates decomposition at or before this temperature. No analyses of the sample were made.

Weighed two gram samples of the air dried lanthanum carbonate were placed in desiccators containing sulfuric acid in one and fresh anhydrone in the other. At different time intervals the samples were reweighed

TABLE IV

EFFECT OF TEMPERATURE ON HYDRATED LANTHANUM CARBONATE

Conditions	% La_2O_3	% CO_2	% H_2O	$\frac{CO_2}{La_2O_3}$	$\frac{H_2O}{La_2O_3}$
Room Temperature (air-dried)	54.48	21.99	23.53	2.97	7.78
110° for 1 hr.	60.48	24.36	15.16	2.99	4.60
3 hrs.	64.01	25.86	10.13	3.00	2.87
5 hrs.	64.53	26.07	9.40	2.99	2.63
24 hrs.	66.34	26.71	6.95	2.98	1.90
48 hrs.	67.32	27.08	5.60	2.98	1.51
130° for 24 hrs.	68.13	27.48	4.41	2.99	1.17
48 hrs.	68.42	27.86	3.72	3.02	0.99
150° for 24 hrs.	68.18	27.29	4.53	2.97	1.12
48 hrs.	66.82	27.68	3.50	2.98	0.92
72 hrs.	68.94	28.01	3.05	3.01	0.80
120 hrs.	69.09	27.96	2.95	2.99	0.77
200° for 4 hrs.	69.93	28.23	1.84	2.99	0.48
6 hrs.	70.02	28.25	1.73	2.99	0.45
18 hrs.	70.30	28.47	1.23	3.00	0.32
250° for 4 hrs.	70.73	28.59	0.68	2.99	0.17
8 hrs.	70.92	28.71	0.37	3.01	0.09
Anhydrous (theoretical)	71.18	28.82	0.00	3.00	0.00

and the per cent water remaining calculated from loss in weight. Table V contains the results of these drying studies. Since the experiment was started late in this work, the drying time is not too lengthy. Further drying would undoubtedly lead to lesser hydrated lanthanum carbonates.

TABLE V

DRYING OF HYDRATED LANTHANUM CARBONATE

	Over H_2SO_4		Over Anhydrone	
Time	% of H_2O Remaining	H_2O/La_2O_3	% of H_2O Remaining	H_2O/La_2O_3
0 hrs.	100.0	7.78	100.0	7.78
2 hrs.	99.4	7.73	99.2	7.72
10 hrs.	97.4	7.50	95.6	7.44
33 hrs.	86.5	6.73	84.7	6.59
44 hrs.	77.1	6.01	75.0	5.84
120 hrs.	63.2	4.91	62.5	4.86
210 hrs.	62.0	4.82	59.9	4.66
370 hrs.	61.7	4.80	56.9	4.43

DISCUSSION

DISCUSSION

In this study the compositions of the lanthanum carbonates and of the sodium lanthanum carbonates were determined from the mole ratios $\frac{CO_2}{La_2O_3}$ and $\frac{Na_2O}{La_2O_3}$ as calculated from the percentages of carbon dioxide, sodium oxide, and lanthanum oxide obtained from analyses. In the case of lanthanum carbonates containing neither ammonium or other metal ions the $\frac{CO_2}{La_2O_3}$ ratio is 3 only for the normal carbonates. A value less than 3 is obtained for basic carbonates and greater than 3 for acid carbonates. For example, the mole ratios $\frac{CO_2}{La_2O_3}$ would be 2 and 6, respectively, for $R(OH)CO_3$ and $R(HCO_3)_3$. For double carbonates a mole ratio $\frac{CO_2}{La_2O_3}$ equal to the mole ratio $\frac{Na_2O}{La_2O_3}$ plus 3. A value less than the mole ratio $\frac{Na_2O}{La_2O_3}$ plus 3 would indicate some basic carbonate formed; a value greater than the mole ratio $\frac{Na_2O}{La_2O_3}$ plus 3 would indicate some acid carbonate formed. Since the mole ratios indicated normal carbonates the percentages of water were obtained by difference and the moles ratios $\frac{H_2O}{La_2O_3}$ calculated. As examples of double carbonates the results listed in Table I would give $Na_2CO_3 \cdot La_2(CO_3)_3 \cdot x\ H_2O$, $2.5\ Na_2CO_3 \cdot La_2(CO_3)_3 \cdot x\ H_2O$, and $3.5\ Na_2CO_3 \cdot La_2(CO_3)_3 \cdot x\ H_2O$, respectively, for the solid phases formed in 1 M., 2 M., and 3 M. sodium carbonate solutions.

Attempts to determine the solubility of lanthanum carbonate in concentrated sodium carbonate solutions showed no lanthanum in solution. Various sodium lanthanum carbonates (Table I) were formed in the solubility studies. The double carbonates were found to decompose in large volumes of water. The lack of solubility of lanthanum carbonate in sodium carbonate solutions contrasts the known solubility of rare earth carbonates in concentrated potassium carbonate solutions as first discovered by Meyer (9).

The precipitation of sodium lanthanum carbonate by the decomposition of the trichloroacetates of sodium and lanthanum produced compounds of varying composition. The more concentrated the solution in sodium ions, the higher the $Na_2CO_3:La_2(CO_3)_3$ ratio in the double salt. Washing the precipitate with water decreased the ratio in all cases. It is noted (Table II) that the normal lanthanum carbonate is formed from a solution containing a small amount of sodium ions.

The precipitate formed by the addition of lanthanum trichloroacetate solution to a concentrated sodium carbonate solution and heating yielded double carbonates of high $Na_2CO_3:La_2(CO_3)_3$ ratios. Although washing with dilute sodium carbonate and water did not completely remove the sodium carbonate from the double salt (Table IV), lengthy stirring in a large volume of water gave a product of normal lanthanum carbonate composition.

In all cases studied the sodium lanthanum carbonate settled out. This differs from the double salt formed by Meyer (9) which formed a thick gelatinous precipitate even after months of standing.

In this study $La_2(CO_3)_3 \cdot 6.11\ H_2O$ and $La_2(CO_3)_3 \cdot 7.78\ H_2O$ were prepared by the trichloroacetate method. Salutsky (13) reported the formation of $La_2(CO_3)_3 \cdot 5.51\ H_2O$. The monohydrate, trihydrate, and octahydrate are listed in the literature (15). Thus far no satisfactory explanation has been put forth for the variations in the amount of hydration. One possible explanation is variable adsorption or occlusion by the crystals during precipitation. A second possibility is the transition from one hydrate to another within the range of room temperature. Failure to obtain constant composition might also be due to differences in drying conditions such as temperature, time, humidity, etc. Additional research is needed to find the answer to this question.

Anhydrous lanthanum carbonate, $La_2(CO_3)_3$, was obtained by heating $La_2(CO_3)_3 \cdot 7.78\ H_2O$ at 250° C. for eight hours. Heating at 100°C. for 50 hours to obtain the monohydrate has been reported (13). This is in disagreement with results from this study whereby 1.5 moles of water per mole of carbonate remained after 48 hours at 110° C. Heating at 130° C. for 48 hours gave the monohydrate.

Lanthanum carbonate was found to decompose at 400° C. The resulting products were not determined. Although the mechanism of the thermal decomposition of lanthanum carbonate is not completely known (5, 12, 14), ignition at 900° C. yields lanthanum oxide, La_2O_3.

After 370 hours of drying over concentrated sulfuric acid, $La_2(CO_3)_3 \cdot 7.78\ H_2O$ had lost less than 50 per cent of its water of hydration. The same was true with anhydrone as the desiccant. Preiss (12) reports that drying of octahydrated rare earth carbonates over concentrated sulfuric acid for 50 days yielded the dihydrate. Since the above experiment was started late in this study, it was not carried out for sufficient time to compare results with those of Preiss.

SUMMARY

SUMMARY

1. Lanthanum carbonate is insoluble in 1 M., 2 M., and 3 M. sodium carbonate solutions. The solid phase, sodium lanthanum carbonate, is decomposed by water to lanthanum carbonate.

2. Sodium lanthanum carbonate is precipitated upon decomposition of lanthanum trichloroacetate and sodium trichloroacetate in hot water solution.

3. Sodium lanthanum carbonate of high $Na_2CO_3:La_2(CO_3)_3$ ratio is precipitated upon addition of lanthanum trichloroacetate solution to concentrated sodium carbonate solution and heating. The double carbonate is decomposed to lanthanum carbonate upon lengthy mixing in a large volume of water.

4. Anhydrous lanthanum carbonate is produced by heating the hydrated lanthanum carbonates at 250° C. for eight hours.

5. $La_2(CO_3)_3 \cdot 7.78\ H_2O$ desiccated over concentrated sulfuric acid and anhydrone for 370 hours lost less than 50% of its water of hydration in both cases.

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AN ABSTRACT

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THESIS ABSTRACT

The high degree of solubility of lanthanum carbonate in concentrated potassium carbonate solutions has been used in the past to separate lanthanum from other rare earths. In this study lanthanum carbonate was found to be insoluble in 1 M., 2 M., and 3 M. sodium carbonate. Sodium lanthanum carbonates were formed as the solid phase. A large excess of water decomposed the double salt to lanthanum carbonate.

Precipitation of sodium lanthanum carbonate by decomposition of the trichloroacetates of sodium and lanthanum in hot water solution produced compounds of varying composition. The more concentrated the solution in sodium ions, the higher the $Na_2CO_3:La_2(CO_3)_3$ ratio in the double salt.

The precipitate formed by the addition of lanthanum trichloroacetate solution to a concentrated sodium carbonate solution and heating yielded double carbonates of high $Na_2CO_3:La_2(CO_3)_3$ ratios. Although washing with water did not completely remove the sodium carbonate from the double salt, lengthy stirring in a large volume of water gave a product of normal lanthanum carbonate composition.

Anhydrous lanthanum carbonate was obtained by heating $La_2(CO_3)_3 \cdot 8\ H_2O$ at 250° for eight hours. After 370 hours of drying over concentrated sulfuric acid, $La_2(CO_3)_3 \cdot 8\ H_2O$ had lost less than 50 per cent of its water of hydration. The same was true with anhydrone as the dessicating agent.