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RELATIVE DETERMINATION OF THE ATOMIC WEIGHT OF CHLORINE IN BAMLE APATITE

By Margot Dorenfeldt

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Introduction

This work was undertaken at the suggestion of Dr. Ellen Gleditsch in connection with an investigation carried out by Gleditsch and Samdahl,[1] the object being to determine whether, in an old mineral which has not been in contact with water, the two chlorine isotopes of atomic weights 35 and 37 occur in the same proportion as in ordinary chlorine. The mode of attack was to compare the specific gravities of saturated solutions of sodium chloride, one solution being made from ordinary sodium chloride and the other from Bamle apatite, the method being similar to that described by Fajans and Lembert.[2]

Apparatus and Materials

The pycnometer was an exact copy of one described by Fajans and Lembert,[2] a modified Sprengel-Ostwald type. The temperature variations of the thermostat cannot be given accurately but a 0.1° thermometer showed no change. The glass test-tubes in which the solutions were rotated in the thermostat were of thick glass, 14 cm. long, and of 22 mm. internal diameter. The clean tubes and the rubber stoppers were soaked in warm brine solution for 12 hours, after which treatment they were carefully rinsed. The balance was a Bünge with a sensitiveness of 0.4 mg.; no mirror was used but $^1/_4$ divisions could easily be read.

The "ordinary sodium chloride" was a commercial highly purified preparation which was further purified. A saturated solution was treated with chlorine, the excess chlorine removed by boiling, the salt precipitated with hydrogen chloride, recrystallized

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[1] Gleditsch and Samdahl, *Compt. rend.*, **174,** 746 (1922). *Arch. Math. Naturvidensk.*, **38** (1923).

[2] Fajans and Lembert, *Z. anorg. Chem.*, **95**, 297 (1916).

several times, ignited, and then recrystallized several more times. All final crystallizations were made in platinum with twice-distilled water, the second time from alkaline permanganate.

The apatite was from the Ödegaarden mines in Bamle and was comparatively rich in chlorine, but the presence of considerable fluorine made it difficult to obtain a preparation free from silica. It was heated with 70% C. P. sulfuric acid and the acid distillate of hydrochloric, hydrofluoric and sulfuric acids and silicon tetrafluoride was neutralized with a highly purified commercial sodium carbonate. The precipitated silica was removed, and after 3 crystallizations the salt was divided into 2 portions, from 1 of which was made a saturated brine solution and from the other was generated a stream of hydrogen chloride which was brought to the surface of the brine solution. The precipitate, dried by suction, was heated to redness in platinum, dissolved, filtered, recrystallized 7 times, ignited again, dissolved and filtered, and then recrystallized 5 times.

Specific-Gravity Determinations of the Saturated Solutions

The two salt solutions, one from ordinary sodium chloride, the other from apatite, were evaporated until crystallization began. The warm solutions were transferred to the tubes, and when cool the tubes were tightly closed with rubber stoppers.

After 3 days' rotation the first weighings of the filled pycnometer were made, No. 1 in the table below. The "apatite sodium chloride" solution was slightly cloudy, the numerous crystallizations having failed to remove all the silica, whose constantly recurring opalescence must have been due to traces of fluoride. Baryta water was added, whereupon a distinct turbidity appeared, and the solution was filtered. The excess of barium hydroxide was precipitated by carbon dioxide gas, the excess of carbon dioxide driven out by boiling, and the solution filtered. The filtrate was neutralized with a few drops of "apatite hydrochloric acid," evaporated to saturation and precipitated with alcohol. The ignited powder was reprecipitated with alcohol and then crystallized several times. The "ordinary sodium chloride" was evaporated to dryness, heated to redness, dissolved, filtered, and recrystallized several times.

After 3 days' rotation another weighing was made, No. 2 in the table. The specific gravity of the "ordinary sodium chloride" being unchanged shows the preparation to be pure, but the "apatite sodium chloride" was again treated as above, giving value No. 3. This solution was then

TABLE I

SPECIFIC GRAVITIES OF SATURATED SOLUTIONS[a]

Expt.	"Ordinary Sodium Chloride"		"Apatite Sodium Chloride"	Expt.	"Ordinary Sodium Chloride"	"Apatite Sodium Chloride"	
			Temp., 18°				
1	1.202791	1.202791	1.204691	4	......	1.202791	
2	1.202790		1.203698	5	......	1.202857	1.202855
3	......		1.203597	6	1.202867	1.202852	

[a] Vacuum corrections were applied to all weighings taking 8.4 as the specific gravity of the brass weights by the usual formulas.

evaporated to dryness, ignited, and recrystallized thrice. Weighing gave value No. 4, 1.202791, which agrees with that found for "ordinary sodium chloride," 1.202791 and 1.202790.

After the work had been abandoned for a time the "apatite sodium chloride" solution was evaporated, ignited, recrystallized two or three times, and the specific gravity again determined (No. 5). Another evaporation, ignition and recrystallization gave value No. 6, the specific gravity of "ordinary sodium chloride" being determined at the same time.

The slight difference between the first and the last determinations for "ordinary sodium chloride" may be accounted for by a new setting of the thermostat regulator. The measurements taken at the same time under identical conditions are in good agreement. Thus the average for "ordinary sodium chloride" under the earlier conditions agrees precisely with the value for the purest "apatite sodium chloride" (No. 4), while the later value, 1.202867, for "ordinary sodium chloride" agrees closely with the average, 1.202855, of the later values for the still further purified "apatite sodium chloride."

A sample was then taken from each of the two solutions by means of the pycnometer and ignited to constant weight in platinum, all precautions being observed. When corrected to vacuum the "ordinary sodium chloride" weighed 3.31262 while the "apatite sodium chloride" weighed 3.31267 or 0.05 mg. more, a difference less than the errors of weighing (0.1 mg.). It follows that the atomic weight of the apatite chlorine does not differ from that of ordinary chlorine by an appreciable amount. Even if the solubilities found (26.357% at 18° or 35.790 g. per 100 g. of water) were not absolutely correct, the atomic weight 35.46 calculated from the relative values would still hold good.

Incidentally, we have compared the solubilities we found with those calculated from the formula of Andreae[3] which are in good agreement with many recent determinations.[4] The calculated value is 26.363%, while we found 26.357% which is in excellent agreement.

Summary

The chlorine isotopes in Bamle apatite occur in the same proportion as in ordinary sodium chloride.

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[3] Andreae, *J. prakt. Chem.*, **29**, 467 (1884).

[4] Taylor, *J. Phys. Chem.*, **1**, 718 (1896–1897). Berkeley, *Phil. Trans.*, **203**, 189 (1904). Fontein, *Z. physik. Chem.*, **73**, 212 (1910). Cohen, *ibid.*, **75**, (1912). Schreinemakers, *Arch. Neer. sci. nat.*, **15**.