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THE EFFECT OF CONTINUED GRINDING ON WATER OF
CRYSTALLIZATION.

(Second Paper.)

BY NICHOLAS KNIGHT.

In the proceedings of the "Iowa Academy of Science," XVII, 131, we described a series of experiments to show the effect of long grinding on water of crystallization. Some of the results were rather surprising, and we decided to repeat the work, and also to include a larger number of hydrates in the experiments. The conditions under which the work was done were as uniform as possible. After grinding, the specimens were kept from the air in ground glass stoppered bottles of about 20 c.c. capacity. Gram portions were weighed out for all determinations. In finding the water content of the unground specimens, the coarse crystals were merely crushed, and the fine crystals were used without further division. Usually about three grams were taken for the grinding, which was effected in a large highly polished agate mortar. The grinding was continued for two hours. In some cases it might be hard to decide whether the loss of water is due to efflorescence or to the heat caused by the continual grinding. Both causes doubtless contributed to some of the results.

In our work on barium chloride, the chemically pure products of two manufacturers were used. The sample was taken from the original package, and the amount of water determined, and another portion was ground two hours, and the water content estimated in the same way. The two specimens were then crystallized once from water, and coarse and fine crystals were obtained. They were dried between filter-papers and at once placed in the glass stoppered bottles. The water was determined as in the original specimens. The results are as follows:

KAHLBAUM'S.

	Per cent.	Crystals	
		Coarse (per cent)	Fine (per cent)
Unground	15.03	14.86	14.84
Ground	14.79	14.80	14.80
Loss	0.24	0.06	0.04

MERCK'S.

Unground	14.89	14.78	14.78
Ground	14.38	14.74	14.77
Loss	0.51	0.04	0.01

It will be noticed that the loss is greater in the original substance than after one crystallization from water. It is also greater in the coarse than in the fine

crystals. The increase in weight, and the stickiness noticed in the case of barium chloride in our first paper were probably due to the presence of the very deliquescent calcium chloride as an impurity.

To ascertain whether it was superficial water that the grinding removed, or the water of crystallization, portions of one of the original specimens, and of the coarse and fine crystallizations, were weighed out, heated a few moments, and the loss of water determined. The residue was then ground for two hours, and portions weighed out and again heated, and the loss by the grinding determined. The results were as follows:

	First determination. (per cent)	Second determination. (per cent)
Original specimen, 14.89% water.		
Removed by first heating.....	2.31	14.62
Removed by second heating.....	12.42	0.26
	<hr/>	<hr/>
Total	14.73	14.88
Loss	0.16	0.01
Purified crystals, 14.78% water.		
Removed by first heating.....	4.75	14.11
Removed by second heating.....	10.01	0.68
	<hr/>	<hr/>
Total	14.76	14.79
Loss	0.02	0.01

It requires a comparatively low temperature to remove 10 or 12 per cent of the water. It was also observed that the specimen in the crucible could be placed very near the flame without decomposition. Results that differed from one another by no more than one-hundredth of a per cent were easily attainable. The results with barium chloride seem to indicate that most of the water removed by grinding is superficial, although a small portion of the water of crystallization can also be removed.

To learn the effect of the atmosphere upon barium chloride before and after grinding, portions of each were left in the air a few days. The unground specimens underwent no change, but the ground portions took up the greater part of the water that had been removed. The fine crystals after grinding would take up slightly more water than the coarse ones.

Removed by grinding (Per cent)	Days in air	Regained (Per cent)	Kind of crystals
0.04	1	0.007	Fine
0.04	3	0.03	Fine
0.06	1	0.01	Coarse
0.06	3	0.04	Coarse
0.24	1	0.06	Uncrystallized
0.24	3	0.16	Uncrystallized

Potassium Alum, $\text{AlK}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$.

Ammonium Alum, $\text{AlNH}_4(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$.

These salts require more care in heating than barium chloride as they are quite easily decomposed. The commercial products were purified by crystallization, and coarse and fine crystals were obtained. The water was determined in

both ground and unground portions. The grinding could not be carried on in a porcelain mortar on account of the tendency to cling to the sides of the dish. The portion for the determination was placed as nearly as possible in the center of the crucible, where it fused to a round ball, thus avoiding the tendency to creep over the sides. The tests failed to show any decomposition of the salts by heating. The water was also determined in an air bath at a temperature of from 105 to 110 degrees, but equally good results were attainable in the ordinary way.

	Coarse (Per cent)	Fine (Per cent)
AlNH₄(SO₄)₂. 12H₂O.		
Unground	47.13	46.98
Ground	44.60	44.75
Loss	2.53	2.23
AlK(SO₄)₂. 12H₂O.		
Unground	45.48	45.44
Ground	42.97	43.42
Loss	2.51	2.02
Strontium Chloride, SrCl₂. 6H₂O.		

Only a small quantity of the water is removed at a low temperature. It decomposes more easily than barium chloride, and the heating must be longer continued and at a lower temperature.

	Uncrystallized (per cent)	Coarse (per cent)	Crystallized Fine (per cent)
Unground	39.94	40.41	39.63
Ground	38.92	38.42	38.10
Loss	1.02	1.99	1.53
Borax, Na₂B₄O₇. 10H₂O.			

This salt is quite efflorescent, and as the specimen at hand had been in contact with the atmosphere for some time, no determination was made before crystallizing. The work was pushed as rapidly as possible before any change in the crystals could take place. A part of the loss is undoubtedly due to efflorescence.

	Coarse (per cent)	Fine (per cent)
Unground	42.39	42.15
Ground	38.66	38.82
Loss	3.73	3.33
Disodium Phosphate, Na₂HPO₄. 12H₂O.		

This salt was the most difficult to handle of any of the series. After grinding for about an hour, it became pasty, adhering quite firmly to the sides of the agate mortar. If constantly scraped loose, it soon becomes a dry powder again. It was difficult to secure uniform results owing, no doubt, to the effect of atmospheric changes upon the crystals. The results given, which seem somewhat erratic and surprisingly high, are considered a fair sample of those obtained.

	Original sample (per cent)	Crystallized.	
		Coarse (per cent)	Fine (per cent)
Unground	61.13	62.45	60.67
Ground	<u>33.21</u>	<u>32.35</u>	<u>32.01</u>
Loss	27.94	30.10	28.66
Manganese Chloride, MnCl ₂ · 4H ₂ O.			

A very pure specimen of the salt (Kahlbaum's) was at hand. No attempt was made to crystallize it, as the number of molecules of water of crystallization it may contain depends upon the temperature. The crystals become pure white when ground.

	Per Cent
Unground	34.03
Ground	<u>32.11</u>
Loss	1.92

I desire to express my hearty thanks to Mr. C. E. Gillette for making the analyses, and for grinding the specimens as described in this paper.