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MONOHYDRATES OF STRONTIUM AND BARIUM HYDROXIDE

THEIR PREPARATION AND X-RAY POWDER PATTERNS

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The monohydrates of strontium and barium hydroxide have been prepared by decomposition, under vacuum, of the corresponding octahydrates. X-ray powder data for both compounds are reported, together with that for anhydrous strontium hydroxide; the latter is included in order to clarify an apparent anomaly in the literature.

In previous work [1, 2], results obtained by the application of various thermoanalytical techniques to the thermal decomposition of strontium and barium hydroxide octadydrates have been described. In two cases, namely thermogravimetry (TG) ane dilatometry, the data suggested that the dehydration reaction occurred via the formation and subsequent decomposition of the corresponding monohydrate; evidence for the existence of these compounds has been reported elsewhere [3]. However, under the experimental conditions used (i.e. with a continuously evacuated system and a constant rate of rise of temperature), it was not found possible to show conclusively the presence of the monohydrates.

The monohydrate of barium hydroxide is sufficiently well known for its X-ray powder pattern to be listed in the standard tables [4]. Little information, however, appears to be available on the corresponding monohydrate of strontium hydroxide. No X-ray powder pattern is listed in the standard tables although two conflicting sets of data are reported for anhydrous strontium hydroxide [5, 6].

This paper presents a simple method for the preparation of the monohydrates of strontium and barium hydroxide, together with their X-ray powder data.

Experimental

The samples of barium and strontium hydroxide octahydrate used in this investigation were General Purpose Reagent grade, with a percentage purity claimed to be not less than 98%.

Isothermal weight loss determinations were carried out under low vacuum (ca. 10^{-1} torr), using a quartz spring balance. Samples (ca. 500 mg) were contained in nickel buckets (15 mm × 13 mm dia.) and evacuated at room temperature.

X-ray photographs were taken in a Debye–Scherrer camera using Cu Kα radiation. Samples were packed into Lindemann glass capillaries of 0.5 mm diameter, which were sealed to prevent reaction of the sample with water vapour in the atmosphere.

Figures 1 and 2 show the results obtained on evacuating samples of barium and strontium hydroxide octahydrate, respectively, at room temperature.

In the case of barium hydroxide (Fig. 1), it can be seen that constant weight is achieved after five hours; the observed weight loss (39.2%) agrees well with the theoretical value (39.9%) for the loss of $7H_2O$. No further weight loss occurred on pumping for a further twenty-two hours, indicating the high stability of the monohydrate. When the temperature was increased to 100° a further loss in weight occurred, so that the final overall weight loss (45.0%) corresponded to that required for the formation of the anhydrous hydroxide (45.7%).

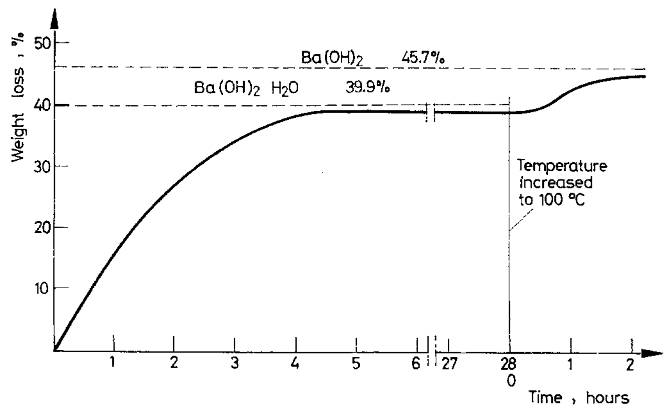

Fig. 1. Isothermal (room temperature) decomposition, under vacuum, of barium hydroxide octahydrate

The X-ray powder pattern obtained for the sample of barium hydroxide monohydrate, prepared as above, is given in Table 1 and compared with the data listed in ASTM file No. 1–0306 [4]. Good agreement is found between the two sets of *d*-spacings with the exception of the line at $d = 6.40$ Å which was not found in our data. The absence of this line can probably be explained by the marked darkening of the X-ray film which occurred with this sample at low angles of diffraction.

Decomposition of strontium hydroxide octahydrate (Fig. 2) follows a similar pattern, with constant weight being effectively reached after evacuating at room temperature for 6 hours. However, evacuation overnight for fifteen hours led to a further small weight loss (ca. 2.5%) indicating that the monohydrate is less stable than the corresponding barium compound. On heating at 100°, the sample once more lost weight until the final weight loss (54.0%) agreed well with that expected for formation of the anhydrous hydroxide (54.6%).

No data are available in the literature for strontium hydroxide monohydrate although, as mentioned previously, two sets of X-ray powder patterns are given

Table 1

X-ray powder data for $Ba(OH)_2H_2O$

d, Å (expt.)	Intensity	d Å, (Lit.)	Intensity $\left(\frac{I}{I_1}\right)$
—	—	6.40	48
4.67	s	4.70	100
3.85	w	3.90	40
3.66	w	3.72	8
3.45	m	3.49	48
3.30	w	3.32	40
3.19	w	—	—
2.98	m	3.00	64
2.90	w	2.90	48
2.65	w	—	—
2.63	w	—	—
2.58	m	2.60	64
2.45	w	2.46	13
2.38	w	2.41	32
2.32	m	2.33	80
2.28	w	—	—
2.24	w	—	—
2.17	w	2.18	26
—	—	2.12	14
2.04	w	2.03	13
2.01	w	—	—
1.94	w	1.95	20
1.89	w	1.91	20
1.86	w	1.87	20
1.80	w	1.81	40
1.76	w	1.74	11
1.68	m	1.70	40
1.64	w	1.65	20

Intensities were estimated visually, and are expressed as follows:
s = strong, m = medium, w = weak.

for the anhydrous hydroxide. The X-ray data obtained for the monohydrate (prepared by evacuating the octahydrate at room temperature for seven hours) were therefore compared with the published powder patterns for the anhydrous hydroxide. It was found that good agreement was obtained between the experimental d-spacings and those for the anhydrous hydroxide reported by Mercer and Miller [5]. These results are summarized in Table 2.

The method of obtaining the anhydrous hydroxide reported by these workers is to heat the octahydrate, at a constant rate of rise of temperature, to 100°. If the decomposition was carried out in air, then it is unlikely that this heat treatment would have produced the anhydrous hydroxide, since it has been shown previously [1, 2] that, even under vacuum, decomposition is not complete by 200°.

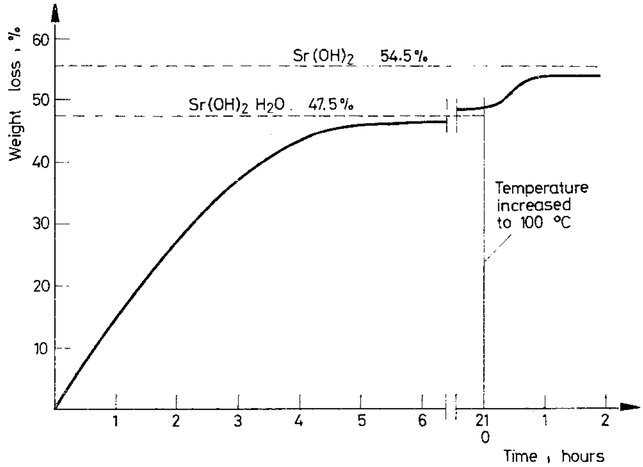

Fig. 2. Isothermal (room temperature) decomposition, under vacuum, of strontium hydroxide octahydrate

Table 2

X-ray powder data for $Sr(OH)_2H_2O$

d, Å (expt.)	Intensity	d, Å (Lit.)	Intensity $\left(\frac{I}{I_1}\right)$
6.18	s	6.19	100
4.54	s	4.53	100
3.63	m	3.65	75
3.56	w	—	—
3.43	m	3.35	100
3.19	m	3.14	75
2.92	w	2.84	75
2.79	s	2.81	75
2.50	s	2.47	75
2.35	w	—	—
2.31	s	2.29	100
2.25	s	2.22	100
2.10	m	2.10	50
2.07	w	2.06	10
1.97	m	1.97	30
1.82	m	1.82	50
1.71	w	1.73	30
1.67	w	1.69	30
1.60	m	1.62	50
1.59	w	1.58	30
1.53	w	1.55	10

The second set of data for the anhydrous hydroxide are reported by Barnighausen and Weilden [6], who obtained their sample by heating the octahydrate at 350°, by which temperature the dehydration process should be complete. We

Table 3

X-ray powder data for $Sr(OH)_2$

d, Å (expt.)	Intensity	*d*, Å (Lit.)	Intensity $\left(\frac{I}{I_1}\right)$
5.21	w	5.22	8
4.92	m	4.95	70
3.86	m	3.85	80
3.30	m	3.30	80
3.10	s	3.13	100
2.89	m	2.92	60
—	—	2.90	80
2.79	w	2.75	20
—	—	2.60	10
2.47	w	2.47	60
2.35	m	2.34	80
2.31	w	2.29	40
2.22	w	2.24	20
2.08	m	2.09	70
—	—	1.96	70
1.94	m	1.95	80
1.88	w	1.88	60
1.80	w	—	—

therefore prepared a sample of the anhydrous hydroxide by heating the octahydrate, under vacuum, in the thermobalance, at 140°, until the weight loss corresponded to the losss of $8H_2O$. The X-ray data obtained with the sample are reported in Table 3 and compared with the data of Barnighausen and Weilden. As can be seen, good agreement is obtained.

It seems therefore reasonable to suppose that the data of Mercer and Miller do not refer to the anhydrous hydroxide but to strontium hydroxide monohydrate.

Conclusion

The monohydrates of both strontium and barium hydroxide have been prepared by room temperature evacuation of samples of the corresponding octahydrate. Barium hydroxide monohydrate is formed as a stable decomposition product and the final molecule of water can only be removed by increasing the temperature at which evacuation is carried out. Strontium hydroxide monohydrate is less stable and there is evidence to suggest that it can be decomposed to the anhydrous hydroxide by prolonged evacuation at room temperature.

From the X-ray powder data obtained it is suggested that the data for anhydrous strontium hydroxide, reported by Mercer and Miller, does, in fact, refer to strontium hydroxide monohydrate.

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RÉSUMÉ — Préparation des hydroxydes de strontium et de baryum monohydratés par décomposition dans le vide des octohydrates correspondants. Etude par rayons X de ces composés ainsi que de l'hydroxyde de strontium anhydre, dans le but, pour ce dernier, d'élucider certaines anomalies de la littérature.

ZUSAMMENFASSUNG — Es wurden die Monohydrate von Strontium und Bariumhydroxid im Vakuum aus den entsprechenden Octahydraten hergestellt. Röntgenographische Daten für beide Verbindungen sowie für wasserfreies Strontiumhydroxid wurden gegeben; für Letzteres, um gewisse Anom alien in der Literatur zu klären.

Резюме — Приготовлены одноводные гидроокиси стронция и бария путем распада в вакууме кристаллогидратов соответствующих солей, содержащих по восемь молекул воды. Описаны результаты рентгенографического исследования этих соединений, а также безводной окиси стронция; изучение безводной соли было проведено с целью выяснения кажущегося отклонения, отмеченного в литературе.