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Solubility of Calcium Bromate in Water

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The solubility of calcium bromate monohydrate in water from (−20 to +100) °C has been determined, along with a differential thermal analysis curve indicating loss of water from (130 to 150) °C and decomposition to calcium bromide from (270 to 300) °C. The effect of calcium chloride on the solubility at room temperature is also reported.

Introduction

Calcium bromate appears to be one of the least studied of the common alkaline-earth halate compounds. No solubility data on it are given in work by Seidell and Linke,[1] though data from Linke[2] on magnesium bromate are reported. Calcium bromate is not mentioned in the latest issues of the *CRC Handbook of Chemistry and Physics*,[3] but earlier editions had listed the monohydrate as very soluble in cold or hot water, with conversion to the anhydrous form at 130 °C. Dean[4a] gave a value of 230 parts $Ca(BrO_3)_2 \cdot H_2O$ in 100 parts of water, but this was omitted from the next edition.[4b] Radford[5] quoted solubilities of group II metal bromates in grams per 100 g of solution at 20 °C, without giving a source. This was probably from a much earlier paper by Kohlrausch and Sitzb,[6] but the latter had expressed the same numerical values as grams per 100 mL of solution at 18 °C. Because the density of a saturated calcium bromate solution at 20 °C is about 1.72 ($g \cdot cm^{-3}$), Radford's data are seriously in error. Unfortunately, Kohlrausch and Siztb's figure of 63 for calcium bromate, when converted to grams per 100 g of solution, becomes only 36. Much more recently, the present authors[7] reported a preliminary value of about 118 g/100 g of water, equivalent to 54 g/100 g of solution.

A fairly recent paper[8] gives physical properties of calcium bromate and iodate, including crystal structures, X-ray, IR, and Raman spectra, and thermoanalytical data. Our thermal results differ slightly from theirs.

Experimental Section

Most of the calcium bromate used was a nearly anhydrous product obtained from Alfa Aesar. Some was prepared from calcium carbonate and bromic acid (obtained from equivalent amounts of barium bromate and sulfuric acid). All were recrystallized from water and dried to the monohydrate form in air. Typical products assayed around 99.4 to 99.5%, with most of the balance likely being moisture. One sample dried at 70 °C assayed 99.89%. Other reagents used were ACS grade where available.

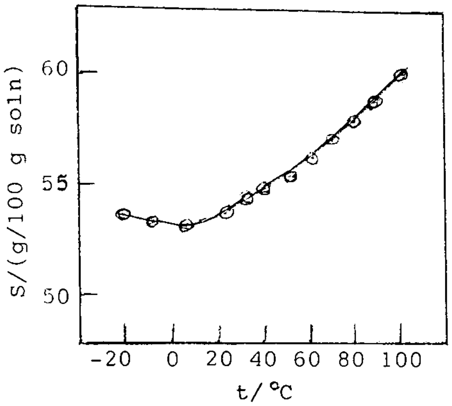

Figure 1. Solubilities of calcium bromate in water.

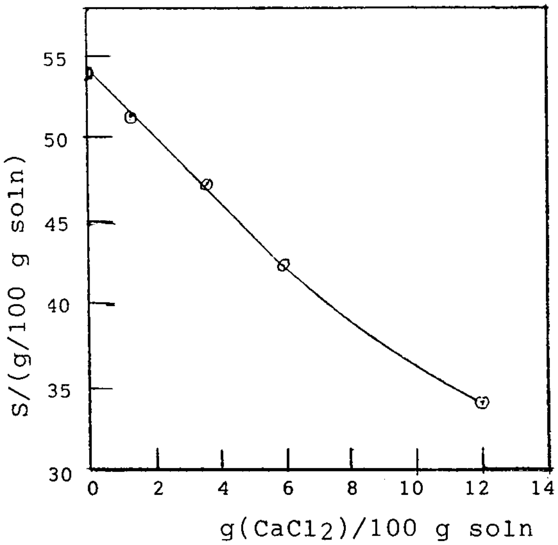

Figure 2. Solubilities of calcium bromate in calcium chloride solutions at 22.7 °C.

The solubility determinations were made using a refrigerator, room-temperature chamber, water bath, or oven for the higher temperatures. Maximum temperature variations were about ±0.5 °C. The solutions were prepared with about 5 mL of water and an excess of solid, in 25-mL vials. At least 24 h was allowed for saturation, with occasional mixing and a settling period of at least 2 h. Precaution had to be taken to avoid either under- or supersaturation. Samples of 0.50 or 1.00 mL were taken (with a precooled or warmed 1.00-mL pipet graduated in 0.01 mL) and weighed in covered 20-mL beakers. Following dilution to

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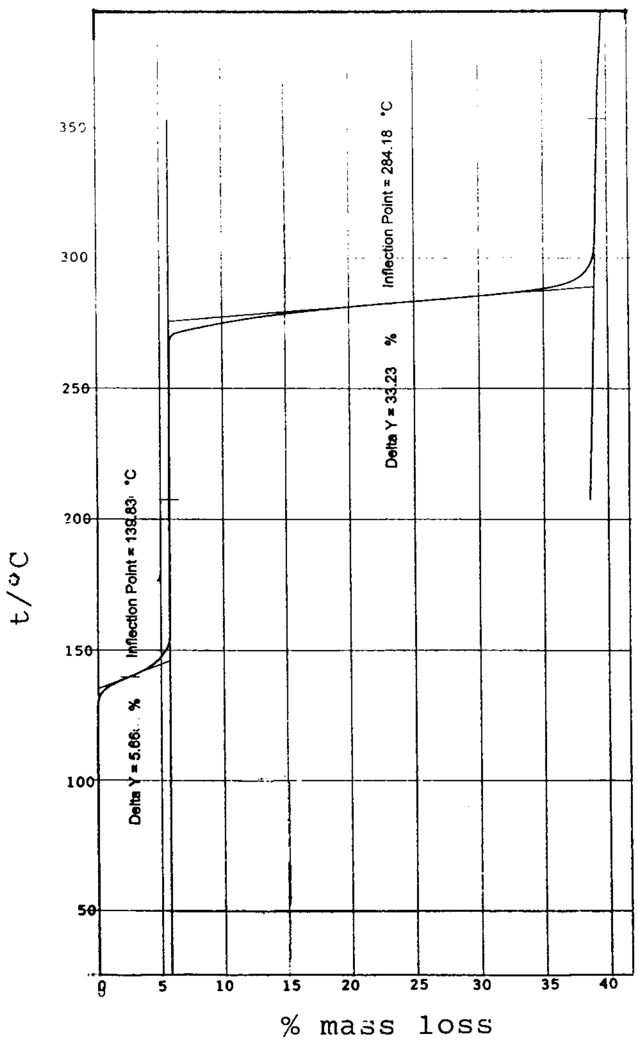

Figure 3. DTA diagrams of $Ca(BrO_3)_2 \cdot H_2O$.

volume, aliquots were titrated iodometrically with 0.1 M sodium thiosulfate.

When calcium chloride was added, the chloride in an aliquot was buffered with potassium hydrogen phthalate and titrated potentiometrically with 0.025 M silver nitrate.

Thermogravimetric analyses (TGA) were carried out at the Michigan Molecular Institute with a Perkin-Elmer TGA-6 instrument with Pyris-1 software. Finely divided crystals were weighed into ceramic pans and slow heat rate experiments were run at 2.5 °C/min from (25 to 400) °C in a flow of pure nitrogen.

Table 1. Solubility of $Ca(BrO_3)_2$ in H_2O and $H_2O + CaCl_2$

	saturated solution		
t/°C	$CaCl_2$/(mass %)	$Ca(BrO_3)_2$/(mass %)	density/(g·cm^{-3})
−20	0	53.4 ± 0.2	1.71
−8		53.3 ± 0.2	1.71
+6		53.2 ± 0.25	1.70
22.7		53.8 ± 0.2	1.72
32		54.6 ± 0.2	1.73
40		54.9 ± 0.3	1.73
50		55.4 ± 0.3	1.74
60		56.2 ± 0.3	1.75
70		57.0 ± 0.4	1.75
80		57.9 ± 0.4	1.76
90		58.4 ± 0.4	1.76
100		59.8 ± 0.4	1.78
22.7	1.24	51.4 ± 0.2	1.69
22.7	3.55	47.5 ± 0.2	1.66
22.7	5.94	42.5 ± 0.2	1.63
22.7	12.0	34.1 ± 0.2	1.53

Results

The data obtained are shown in Table 1 and Figures 1−3. The solubility curve is nearly flat from (−20 to +10) °C and rises gradually at higher temperatures. Added calcium chloride lowers the solubility, as would be expected from the common ion effect. The TGA show water loss beginning at about 137 °C and reaching completion at 150 °C. Oxygen loss begins at about 270 °C, and conversion to calcium bromide is complete at 300 °C. The inflection point for dehydration varies with the rate of heating. At 1 °C/min, it is about 130 °C.

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