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Reprinted from American Mineralogist, 45: (1960)
Pages 241-243

THE AMERICAN MINERALOGIST, VOL. 45, JANUARY-FEBRUARY, 1960

THE CELL CONSTANTS OF ARTIFICIAL SIDERITE*

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The artificial siderite used to measure the cell constants was prepared in the following way. Equal molar quantities of solid NaHCO_3 and $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ were placed in a 120 ml. capacity stainless steel bomb which was half filled with water. The sealed bomb was then heated to 200°C . After reaching 200°C ., CO_2 was pumped into the bomb until a total pressure of 500 bars had been reached. The bomb was maintained at 200°C . and 500 bars for 3 days. This procedure served to stabilize the precipitated FeCO_3 . The white precipitate was then removed, filtered, and dried in an oven at 100°C . It partly oxidized to a yellow-brown

* Contribution No. 165, Institute of Geophysics.

color while drying. The dried material was placed in a "simple squeezer" high pressure apparatus, Kennedy and Griggs (1956), and heated to 600° C. at a pressure of 15 kb. The final product used in the x-ray work consisted of siderite together with a small amount of magnetite.

Earlier attempts to prepare siderite were less successful. Mixtures of $\text{FeCl}_2 \cdot 4\text{H}_2\text{O} + \text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$ and $\text{FeSO}_4 \cdot 4\text{H}_2\text{O} + \text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$ with total $\text{CO}_2 + \text{H}_2\text{O}$ pressures of 50–200 bars in a bomb resulted only in forming magnetite. A small amount of siderite was, however, prepared by precipitating amorphous FeCO_3 and $\text{Fe}(\text{OH})_2$ from solutions of FeSO_4 and Na_2CO_3 at atmospheric pressure. The solutions were kept covered with

TABLE 1. X-RAY POWDER DATA FOR ARTIFICIAL SIDERITE

Indices	$d_{\text{obs.}}$	$d_{\text{calc.}}$	100 I/I ₀
01.2	3.591	3.591	25
10.4	2.789	2.791	100
11.0	2.341	2.345	20
11.3	2.131	2.132	27
20.2	1.962	1.964	30
02.4	1.794	1.796	15
01.8	1.735	1.737	35
11.6	1.730	1.730	44
12.2	1.505	1.506	19
21.4	1.425	1.426	16
20.8	1.395	1.396	7
03.0	1.353	1.354	20
12.8	1.199	1.199	17
21.10	1.086	1.086	17
13.4	1.081	1.081	26
22.6	1.066	1.066	17

kerosene to prevent oxidation from the air. The precipitate was allowed to settle and the excess solution decanted off. The remaining water was removed by adding acetone and filtering. The material was dried in a vacuum oven at 110° C. However, most of the material still oxidized. This material was subsequently run as above in the "simple squeezer" at 500° C. and 15 kb. The resulting product was a mixture of hematite and siderite.

An x-ray powder diffraction pattern of the siderite-magnetite mixture, finely ground and mixed with vaseline, was obtained at 25° C. in a Norelco high angle recording diffractometer using $\text{FeK}\alpha$ radiation ($\lambda = 1.9373 \text{ \AA}$) and a Mn filter, with a scanning speed of $\frac{1}{3}^\circ (2\theta)$ per minute. High purity pre-calibrated NaCl was used as an internal standard.

The hexagonal unit-cell belonging to the space group $R\bar{3}c$ was obtained by a least squares treatment. The hexagonal cell constants are:

$$a_0 = 4.690 \pm 0.002 \text{ \AA}, \quad c_0 = 15.370 \pm 0.003 \text{ \AA}, \quad Z = 6, \quad \text{axial ratio } c/a = 3.277.$$

This compares closely with the values for natural material given in Dana's System of Mineralogy (1951) p. 167.

$$\begin{aligned} a_0 &= 4.71 \text{ kX}, & c_0 &= 15.43 \text{ kX}; \\ a_0 &= 4.677 \text{ kX}, & c_0 &= 15.267 \text{ kX}. \end{aligned}$$

The calculated rhombohedral dimensions are: $a_{rh} = 5.795 \text{ \AA}$, $\alpha = 47^\circ 45'$. The d-spacings and the corresponding indices are given in Table 1.

The calculated density is 3.941 which compares with the measured density of $3.96 \pm .01$, Dana's System of Mineralogy (1951) p. 168.

ACKNOWLEDGMENTS

I wish to thank Drs. Carl Pistorius and Julian Goldsmith for their help and encouragement during the preparation of this manuscript.

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Note added in proof—

The lattice constants of artificial siderite have also recently been determined by D. L. Graf (personal communication) using the same method. His results, found by extrapolation on back reflection measurements are:

$$a_0 = 4.6887 \text{ \AA}, \quad c_0 = 15.373 \text{ \AA}; \quad a_{rh} = 5.7954 \text{ \AA}, \quad \alpha = 47^\circ 43.3'$$

