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Synthesis and Characterization of Dawsonite

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19

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Synthesis and Characterization of Dawsonite

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SYNTHESIS AND CHARACTERIZATION OF DAWSONITE

by

J. Jackson, Jr.,[1] C. W. Huggins,[1] and S. G. Ampian[2]

ABSTRACT

Synthetic dawsonite [$NaAl(OH)_2CO_3$] has been prepared with crystallinity similar to that of dawsonite found in oil shale deposits of the Piceance Creek area of Colorado. Optimum experimental conditions were as follows: Na/Al atomic ratio, 43; temperature, 175° to 200° C; pCO_2, 15 psig; and reaction time, 5 hr. Chemical and spectrographic analysis revealed that the synthetic dawsonite was of high purity and was essentially stoichiometric. Similarity of the synthetic to natural dawsonite, demonstrated by X-ray diffraction, infrared spectrophotometry, and electron microscopy, suggests its suitability as a standard.

INTRODUCTION

This research represents part of a program designed to develop processes for efficiently recovering alumina and soda from spent retorted oil shales from the saline zone, Piceance Creek Basin, Colorado. Our objective was to synthesize and characterize a well-crystallized dawsonite [$NaAl(OH)_2CO_3$] suitable for use in either the internal standard-calibration or known-addition technique for quantitative X-ray diffractometry (4, 6, 12).[3] A lack of a suitable standard, essentially comparable in crystallinity[4] with that of dawsonite found in the Piceance Creek shales, has deterred development of suitable quantitative X-ray diffraction (10, 15, 17) and infrared (7) techniques. The only presently available methods are based on selective solubilities, and these methods can have systematic errors if specific sodium-bearing minerals are present (18).

The X-ray peak intensity for the strongest dawsonite reflection, the (110) at 5.69A, had an angular half-maximum breadth ($\beta_{\frac{1}{2}}$) of 0.175°2θ and

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[1]Research chemist.

[2]Physical scientist, now with Division of Nonmetallic Minerals, Bureau of Mines, Arlington, Va.

[3]Underlined numbers in parentheses refer to items in the reference list at the end of the report.

[4]Crystallinity on this work is determined by measurement of the breadth of the 5.69A peak at half-maximum.

registered 2,500 counts per second (cps) on material microscopically separated from dawsonite veins in Colorado oil shale. The available Olduvai Gorge dawsonite[5] has a $\beta\frac{1}{2}$ of 0.300°2θ and a peak intensity of 1,750 cps and, even after extensive beneficiation, is of insufficient purity to qualify as an internal standard.

Synthetic dawsonite was made by Bader (2) in 1938, but he reported that crystallization was not complete based on his X-ray data. Bader (3) again synthesized dawsonite in 1944 and reported the crystallinity of the material as nearly equal to that of natural dawsonite found in Albania. His best material was prepared at a temperature of 95° C and a carbon dioxide partial pressure (pCO_2) of 5 atm. In 1968, Van Nordstrand (20) patented a process for synthesizing dawsonite using conditions similar to the optimum conditions reported by Bader for his best crystallization. However, Van Nordstrand's best synthetic dawsonite, as evaluated for this report, registered only 800 cps, which was inferior to the natural Olduvai dawsonite. None of the available synthetic or natural dawsonite evaluated by the Bureau of Mines has proved satisfactory as a standard. Accurate dawsonite values are critical for meaningful processing research and cost-evaluation data. This report presents data that will resolve the problem by providing well-crystallized dawsonite.

EXPERIMENTAL PROCEDURES

The dawsonite syntheses were carried out in a commercial 2-liter, teflon-lined, stainless steel Parr[6] autoclave equipped with a stirrer, pressure gage, in-out valve, and a thermocouple well. The reactor-heater was automatically programed and controlled to within ±5° C. The actual reaction temperature, measured ½-inch from the liner bottom by a Chromel-Alumel thermocouple in a thermowell, was controlled to ±1° C. The starting material for these syntheses was poorly crystallized commercial dawsonite, reagent-grade soda ash (Na_2CO_3), and sodium hydroxide (NaOH). Commercial dawsonite was used in this study because it was readily available.[7]

The reactor solution was prepared by dissolving the commercial dawsonite with a Na_2CO_3-NaOH solution containing sufficient carbonate and hydroxide, not only to dissolve the dawsonite but also to provide the desired Na/Al atomic ratio for each run. Digestion was accomplished by slowly adding the measured dawsonite to the preheated alkaline solution maintained at 70°±2° C, while stirring magnetically for 15 min. The resulting solution was transferred to the autoclave, heated to the desired temperature, and then carbonated at a selected pCO_2. A typical recorder trace of the heating and cooling cycle and operating highlights for the reactor are illustrated in figure 1.

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[5]The African dawsonite was supplied by Dr. Charles Milton of the George Washington University, Washington, D.C.

[6]Reference to specific products is made to facilitate description and does not imply endorsement by the Bureau of Mines.

[7]Although this report is limited to recrystallizing dawsonite from commercially available material, comparable well-crystallized dawsonite can also be prepared from sodium aluminate-sodium carbonate-sodium hydroxide solutions.

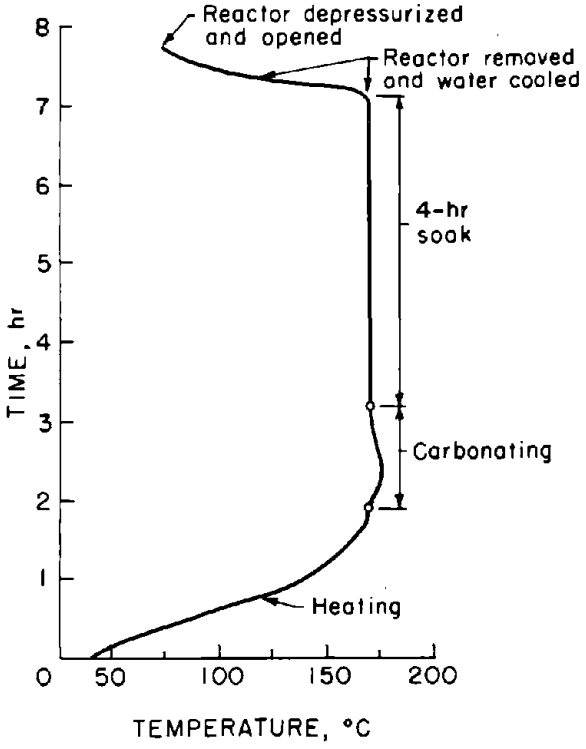


FIGURE 1. - Typical Recorder Trace for Reactor Showing Operation Highlights for Recrystallizing Dawsonite.

The reactor was brought to thermal equilibrium and pressurized with the desired pCO_2. The total reaction time for each experiment was measured from the thermal equilibrium point indicated by the lower circle in the thermogram of figures 1, 3, and 4. Upon conclusion of the run the reactor was removed, cooled to ≈75° C, and depressurized. The recrystallized material was separated from the mother liquor by vacuum filtration. The water-soluble carbonate was removed from the dawsonite by three hot-water (70° C) washings.

The measure of crystallinity of dawsonite, along with other data presented in this work, was determined on washed samples oven-dried at 90° C for 24 hr. The recrystallized dawsonite was evaluated first by X-ray diffraction to ascertain its crystallinity, and was further evaluated by infrared spectroscopy, differential thermal analysis, and electron microscopy. Chemical and spectrographic analyses were also done on selected samples of the recrystallized dawsonite.

RESULTS AND DISCUSSION

Selected experimental data on the syntheses of dawsonite are listed in table 1. The entire range of variables investigated were as follows: recrystallization temperature, 70° to 200° C; Na/Al atomic ratio, 27 to 43; pCO_2, 10 to 25 psig; and recrystallization time, 1 to 5 hr. Table 1 also contains the X-ray peak intensities for the strongest dawsonite reflection, the (110) at 5.69A.

The initial screening tests in the dawsonite synthesis included the atmospheric test conditions outlined in the Van Nordstrand (20) patent. Dawsonite synthesis by the patented process involves flooding with CO_2 the surface of a preheated (95° C) sodium aluminate-carbonate-hydroxide solution, containing an approximate Na/Al atomic ratio of 41, until precipitation occurs. This precipitation, although instantaneous upon CO_2 contact, takes approximately 1 hr for completion owing to the slow diffusion of CO_2. Variations of the Van Nordstrand patent procedure resulted consistently in poorly crystallized material that was comparable with the commercial dawsonite illustrated in figure 2. The (110) reflections from this dawsonite and others recorded from Olduvai Gorge, Colorado oil shale, and Bureau of Mines synthetic material are also shown.

TABLE 1. - Selected experimental data for dawsonite syntheses[1]

Run No.	Dissolving solution			Recrystallization[2]		Peak intensity,[3] cps
	Na_2CO_3, g	NaOH, g	Na/Al atomic ratio	Temp, ° C	pCO_2, psig	
9.....	80	60	43	95	15	770
10.....	80	60	43	110	15	1,100
12.....	80	60	43	140	15	1,260
13.....	80	60	43	160	15	1,780
14.....	80	60	43	175	15	2,460
28A.....	80	60	43	200	15	2,700
23.....	80	30	33	175	15	1,540
21.....	80	15	27	175	15	1,620
24A.....	80	60	43	175	10	1,220
25A.....	80	30	33	175	10	2,450
26A.....	80	60	43	175	25	2,000
28.....	80	30	33	175	25	2,275
27A.....	80	15	27	175	25	2,060

[1]Starting solution contained 10 g of commercial dawsonite and 500 ml of distilled water.

[2]Total heating plus carbonating time = 7 hr.

[3]Peak intensities from (110) reflection determined with a copper target X-ray tube operated at 40 kv and 15 ma using a goniometer scan speed of 1/8° 2θ per min on samples dried at 90° C that were free of soluble carbonates.

Subsequent investigations were limited to recrystallizing selected solutions containing dissolved commercial dawsonite under arbitrarily selected autoclaving conditions. Selected data from these experiments are listed in table 1. The listed data reveal that under conditions of 1 atm pCO_2 and Na/Al atomic ratio of 43, dawsonite crystallinity increased steadily with increasing temperature. As indicated by the (110) reflection, the 95° C low-temperature-synthesized dawsonite, run 9, accumulated 770 cps for this reflection, whereas the highest temperature experiment (run 28A, 200° C) produced material that recorded a maximum cps of 2,700 during these studies. Deviations from the optimized parameters, listed in table 1 for run 28A, produced dawsonites that were either comparable in counts per second with the oil shale dawsonite (≃2,500 cps) or lower.

The influence of temperature on the crystallinity of the synthetic dawsonite is explained readily with the aid of figure 3. These three traces reveal a marked dissimilarity in the exothermic carbonating portion. The reactor thermogram shows a stronger exothermic carbonating reaction at lower temperatures compared with that of higher temperatures. These differences are due to the decreasing solubility of CO_2 as the temperature increases, which retards the conversion of sodium aluminate to dawsonite sufficiently to enhance grain growth. These thermograms are simply copies of recorder traces from the high-output, Chromel-Alumel reactor thermocouple. The traces in figure 3 are from experiments run at 160°, 175°, and 200° C with invariant conditions of pCO_2 (15 psig) and Na/Al (43), which gave the best crystallized dawsonite.

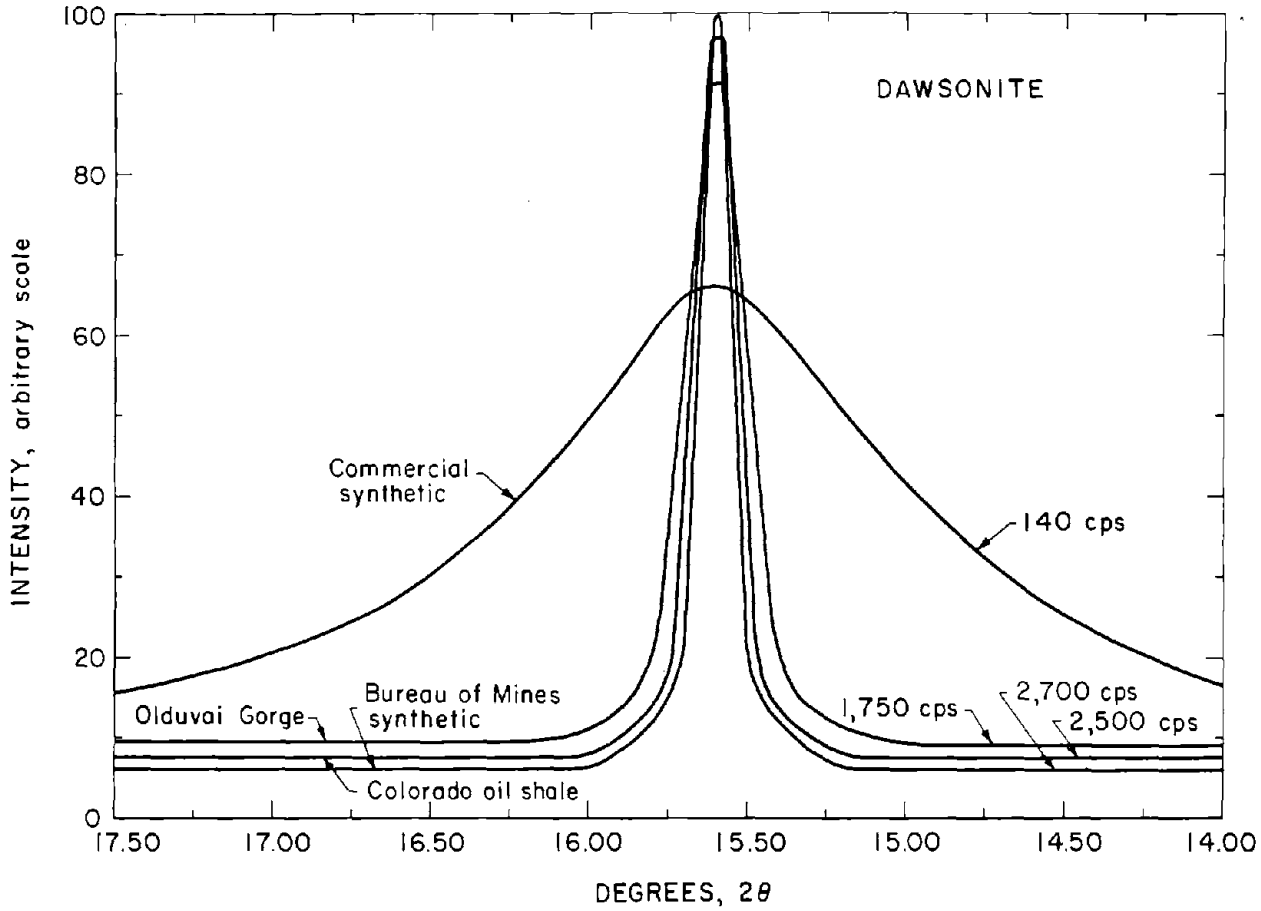


FIGURE 2. - Diffraction Maximums for 5.69A (110) Reflections From Selected Synthetic and Natural Dawsonites. The commercial dawsonite maximum is vertically exaggerated.

The influence of pCO_2 and OH^- concentrations on the crystallinity of synthetic dawsonite is also shown by the data in table 1. Run 25A showed that grain growth comparable with run 14 was achieved by lowering both the pCO_2 and the OH^- concentration at 175° C. This product gave 2,450 cps, compared with 1,540 cps for run 23 in which only the Na/Al atomic ratio was reduced. A reduction in the pressure alone at 175° C (run 24A) gave a product that showed only 1,220 cps, compared with 2,460 for run 14. An increase in pressure to 25 psig at 175° C (run 26A) also showed a decrease in counts per second (2,000) compared with run 14 (2,460 cps). Figure 4 shows the superimposed thermograms from runs 25A and 26A that indicate the increased exothermic carbonation reaction at 25 psig pCO_2 (run 26A), compared with 10 psig pCO_2 (run 25A). The dawsonite sample from the low carbon dioxide partial pressure experiment also contained some boehmite. The monohydrate is the result of an incomplete sodium-aluminate conversion reaction brought about by the low carbon dioxide partial pressure, which reduces the availability of the CO_3^{--} formed during carbonation.

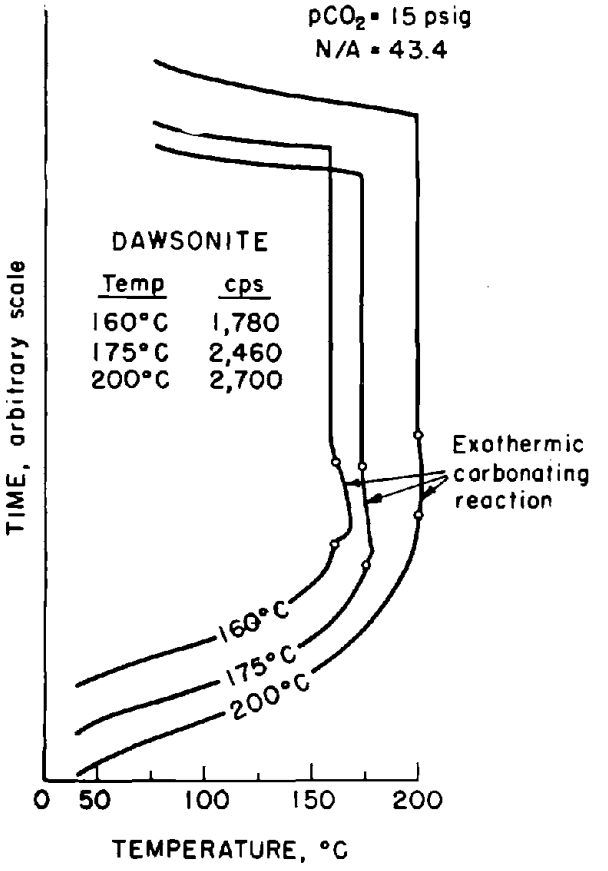


FIGURE 3. - Recorder Traces Showing Effect of Temperature on Carbonation Reaction.

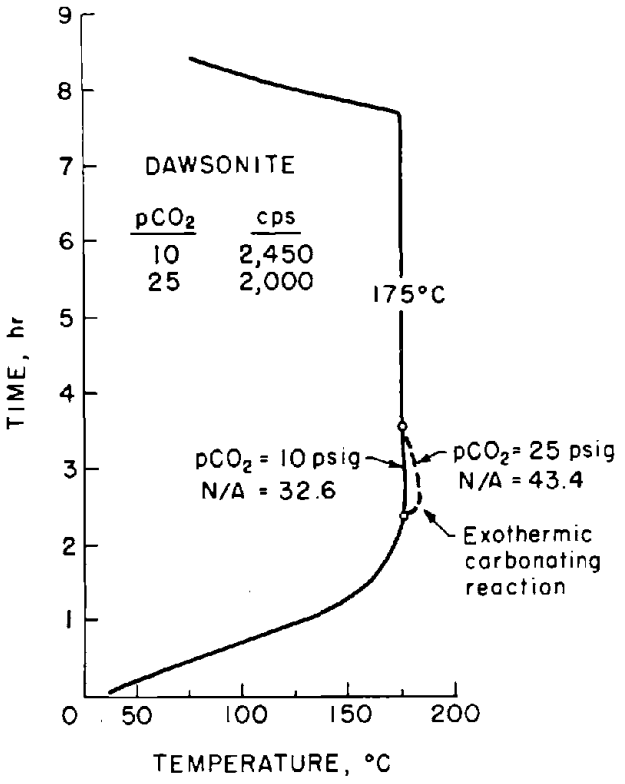


FIGURE 4. - Recorder Traces Showing Effect of Na/Al and pCO_2 on Carbonation Reaction at 175° C.

Most of the dawsonite syntheses, however, were restricted to 175° C. This was less than the optimum temperature of 200° C, because of teflon's dimensional instability at >200° C. These lower temperature dawsonites (runs 14 and 25A), although inferior in crystallinity (≈2,500 compared with 2,700 cps) to the higher 200° C temperature material (run 28A), are ideally suited as an internal standard because of their similarity to the natural oil shale material in line-broadening characteristics.

The comparability and suitability of run 14 dawsonite, as an internal standard, are indicated by the $\beta\frac{1}{2}$ and peak intensity data listed in table 2, which also affords a comparison between the oil shale, African, commercial, and Bureau of Mines runs 14 and 28A dawsonites. The $\beta\frac{1}{2}$ of 0.175°2θ for the oil shale and run 14 dawsonite and their near equality in peak intensity, 2,460 versus 2,500 cps, clearly established this comparability, and hence, the suitability of the run 14 material. This run was omitted from figure 2 because its line-broadening characteristics are identical to that of the oil shale material.

TABLE 2. - $\beta\frac{1}{2}$ and peak intensities for selected natural and synthetic dawsonites

Type	$\beta\frac{1}{2}$, °2θ	Peak intensity, cps
Commercial.....	1.263	140
Olduvai Gorge.....	.300	1,750
Oil shale.....	.175	2,500
Bureau of Mines, No. 14.....	.175	2,460
Bureau of Mines, No. 28A.....	.150	2,700

[1]From (110) reflection, 5.69A, scanned at 1/8° 2θ/min at 25°±1° C; copper target X-ray tube operated at 40 kv and 15 ma.

The dissimilarity in crystallinity and/or grain size between the present synthesized and Olduvai Gorge dawsonite is demonstrated by figure 5, which compares typical DTA data. The thermograms reveal essentially similar decomposition endotherms with peak centers at ≃375° C, but differing in shape at the onset of decomposition. The gentler onset decomposition trace of the African Olduvai Gorge dawsonite, compared with the more abrupt and clearly defined change in the synthetic material at the constant heating rate of 10° C/min, may be attributed to differences in crystallite size (9). This difference in crystallite size is readily apparent in the electron micrographs (fig. 6). These show the growth in crystallite size from the poorly crystallized commercial dawsonite (A), to better crystallized material (B), to well-crystallized material (C) that is nearly identical to the natural dawsonite (D) from the Piceance Creek area of Colorado.

The X-ray powder data for the best crystallized 28A dawsonite, both calculated and observed, and its unit-cell dimensions obtained by least-squares refinement (19) of the diffractometer data are listed in tables 3 and 4, respectively. The published lattice constants for natural dawsonite from Montreal, Canada (1, 14), are also included. The optical microscopic values for 28A dawsonite and those published for the Canadian material (13) are listed in table 5. The lower indices of refraction for the synthesized dawsonite, compared with the Canadian material, are probably due to the presence of impurities in the natural material. The Canadian material, along with other dawsonites, is reported to be intimately associated with calcareous minerals (16).

Infrared data obtained using Cole's procedure (5) for comparing the Piceance Creek and Bureau of Mines synthetic dawsonite are illustrated in figure 7. The comparability of these materials is again indicated by the similarity of the data. The published infrared data for the Piceance Creek (7, 11) and Canadian (8) dawsonites are also in close agreement.

TABLE 3. - Powder X-ray diffraction data for Bureau of Mines synthetic dawsonite[1]

hkl	2θ,[2] observed	2θ, calculated	Δ θ	I	d, A, observed	d, A, calculated
110.....	15.585	15.615	-0.030	>100	5.685	5.674
200.....	26.360	26.354	+.005	82	3.380	3.381
121.....	26.826	26.848	-.022	7	3.322	3.320
130.....	28.871	28.876	-.005	8	3.092	3.092
211.....	32.084	32.109	-.025	>100	2.789	2.787
040.....	34.407	34.380	+.027	22	2.606	2.608
112.....	35.830	35.837	-.007	7	2.506	2.506
231.....	40.502	40.570	-.068	4	2.227	2.223
202.....	41.955	41.955	.000	15	2.153	2.153
240.....	43.796	43.845	-.049	3	2.067	2.065
150.....	45.518	45.508	+.009	17	1.993	1.993
051.....	46.469	46.474	-.005	6	1.954	1.954
312.....	52.924	52.928	-.004	15	1.730	1.730
400.....	54.276	54.248	+.027	30	1.690	1.691
242.....	55.333	55.333	.000	7	1.660	1.660
420.....	57.291	57.278	+.012	3	1.608	1.608
260.....	59.838	59.825	+.012	3	1.545	1.545
350.....	60.450	60.466	-.016	2	1.531	1.531
440.....	65.830	65.832	-.002	2	1.418	1.418
422.....	67.135	67.160	-.025	2	1.394	1.393
352.....	70.184	70.090	+.093	5	1.340	1.342

[1]Data obtained from run 28A.
[2]Values are for CuK_α (1.54178 A).

TABLE 4. - Unit cell parameters for dawsonite

Type	a, A	b, A	c, A	b/a	b/c	Dx, g/cm^3	Dm, g/cm^3	V, A^3
Bureau of Mines synthesized[1].	6.763	10.428	5.585	0.649	0.536	2.427	2.418	393.9
Natural[2].....	6.77	10.40	5.58	.651	.537	2.434	2.44	392.9

[1]Run 28A; measured at 25°±1° C.
[2]Type locality, McGill University Campus, Montreal, Canada.

TABLE 5. - Optical data for dawsonite[1]

	Synthetic[2]	Natural[3] (13)
α.....	1.458	1.466
β.....	1.535	1.542
γ.....	1.586	1.596
Optic sign.....	Negative	Negative
2 V.....	74° (est.)	77°

[1]Optical data by M. V. Denny, College Park Metallurgy Research Center, College Park, Md.
[2]Run 28A; measured at 27°±3° C.
[3]Type locality, McGill University Campus, Montreal, Canada; similar optical values listed on ASTM X-ray diffraction cards 12-449 and 19-1175 (1) and in Dana's System of Mineralogy, 7th ed., v. 2 (16).

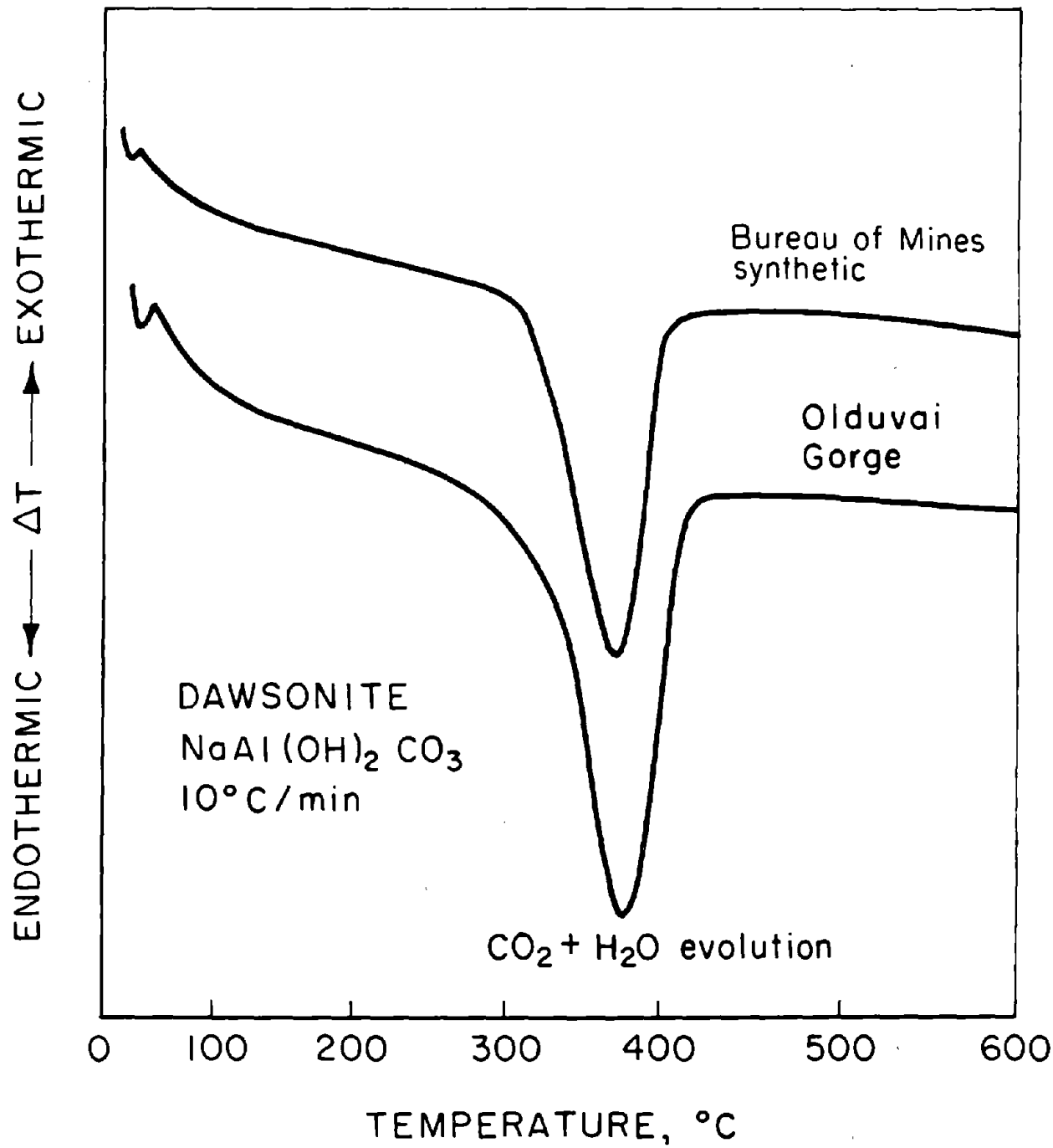
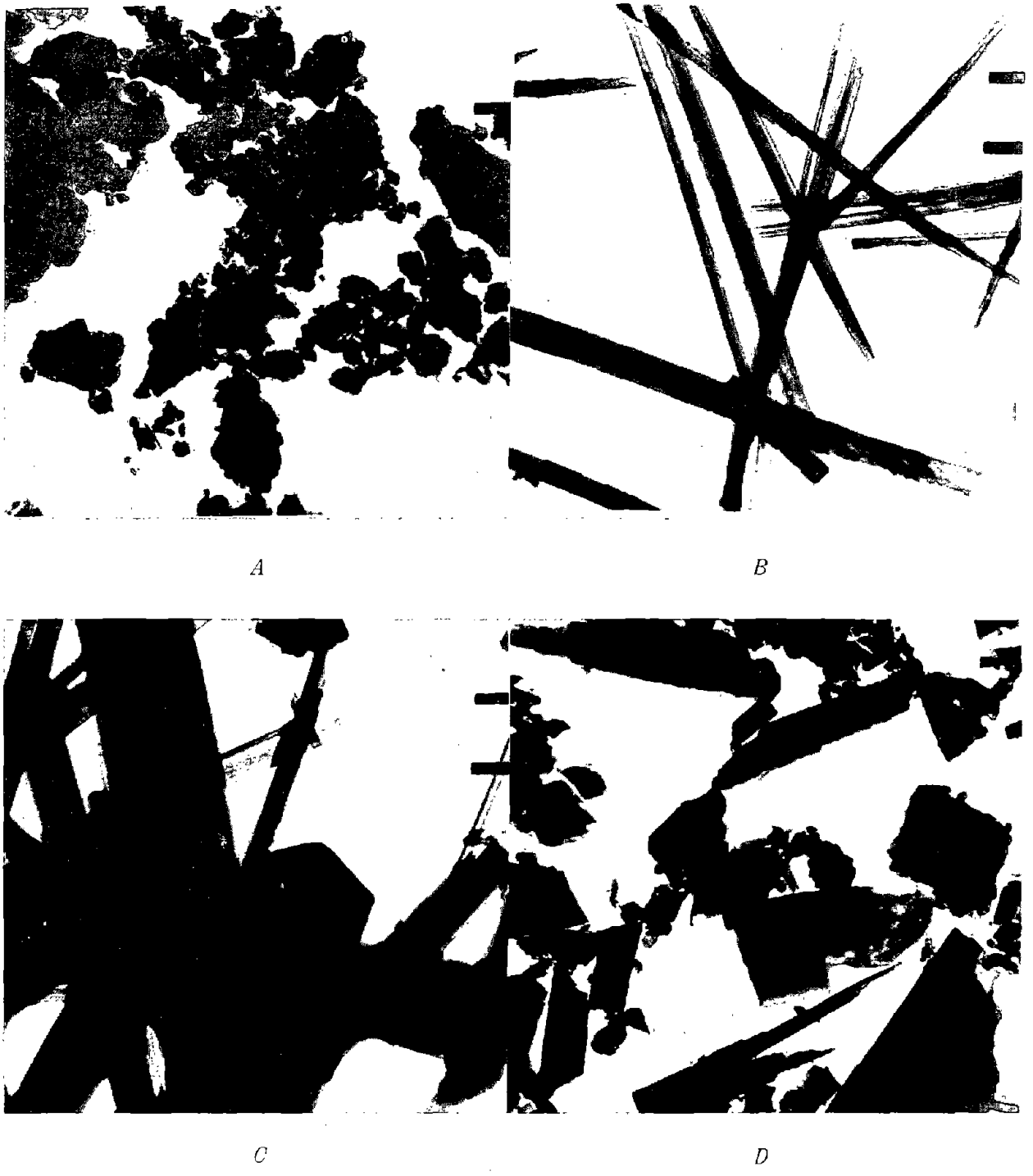


FIGURE 5. - Typical DTA Traces of Bureau of Mines Synthetic and African Dawsonite.



A *B*

C *D*

FIGURE 6. - Selected Electron Micrographs of *(A)* Commercial, *(B)* Bureau of Mines Synthetic No. 10, *(C)* Bureau of Mines No. 28A, and *(D)* Colorado Oil Shale Dawsonite (X 9,000).

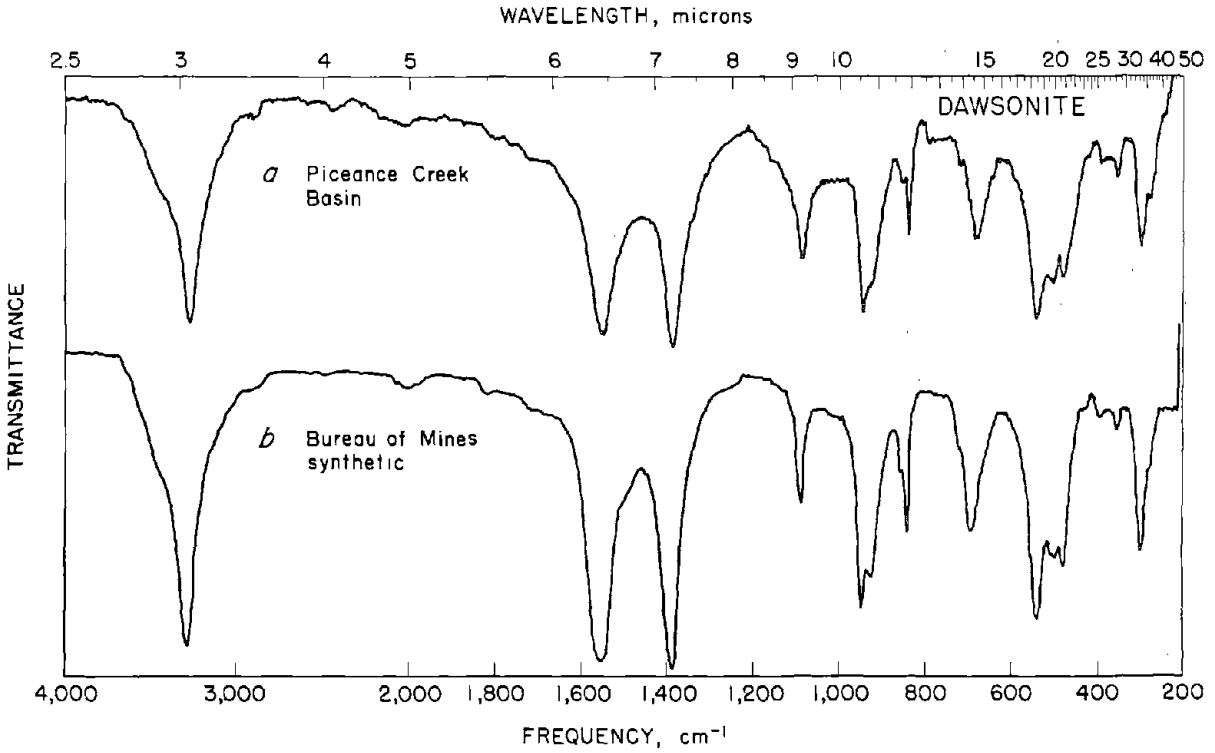


FIGURE 7. - Infrared Spectra of Natural Dawsonite From Piceance Creek Basin of Colorado and Bureau of Mines Synthetic Dawsonite.

Chemical and spectrographic analyses of the synthetic dawsonite (28A) and theoretical composition for the chemical formula [$NaAlCO_3(OH)_2$] are given in table 6. The synthetic dawsonite is of high purity, and the composition was determined to be stoichiometric within the limits of the analytical procedure used.

TABLE 6. - Composition of synthetic dawsonite, weight-percent

Components	Theoretical composition	Synthetic dawsonite
Na_2O.....	21.53	21.56
Al_2O_3.....	35.40	34.57
CO_2.....	30.56	30.52
H_2O.....	12.51	13.71
CaO.....	-	.06
Fe_2O_3.....	-	.04
MgO.....	-	.01
Total.....	100.00	[1]100.47

[1]Additional trace impurities reported by emission spectrography were Si <0.05, Zr <0.02, Ti <0.002, and Mn <0.05.

CONCLUSION

Pure, well-crystallized dawsonite, suitable for use in the internal standard calibration or known-addition technique for quantitative X-ray

diffractometry, was synthesized and characterized. Experimental autoclave conditions were as follows: Na/Al atomic ratio, 43; temperature, 175° to 200° C; pCO_2, 15 psig; and reaction time, 5 hr.

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REFERENCES[8]

1. American Society for Testing and Materials. Inorganic Index to the Powder Diffraction File. Philadelphia, Pa., 1971, File Nos. 12-449 and 19-1175.

2. Bader, Erich. Uber bie Bildung und Konstitution des Dawsonits und seine synthetische Dorstellung (On the Formation and Constitution of Dawsonite and Its Synthetic Preparation). Neues Jahrb. Mineral. Geol., Beil. A., v. 74, 1938, pp. 449-465.

3. Bader, Erich, and U. Esch. Versuche zur Drucksynthese des Dawsonits (Experiments on the Pressure Synthesis of Dawsonite). Z. Elektrochem., v. 50, 1944, pp. 266-268.

4. Brindley, G. W. Quantitative Analysis of Clay Mixtures. Ch. in the X-Ray Identification and Crystal Structures of Clay Minerals, ed. by G. Brown. Mineral. Soc. (Clay Minerals Group), London, 1961, pp. 489-516.

5. Coles, J. L. A Study of Some Synthetic Apatites. Ph.D. Thesis, Dept. of Min., Univ. of Utah, Salt Lake City, Utah, June 1963, pp. 16-18.

6. Cullity, B. D. Elements of X-Ray Diffraction. Addison-Wesley Publishing Co., Inc., Reading, Mass., 1956, 514 pp.

7. Estep, P. A., and C. Karr, Jr. The Infrared Spectrum of Dawsonite. Am. Miner., v. 53, Nos. 1-2, January-February 1968, pp. 305-309.

8. Frueh, A. J., Jr., and J. P. Golightly. The Crystal Structure of Dawsonite $NaAl(CO_3)(OH)_2$. Can. Miner., v. 9, pt. 1, 1967, pp. 51-56.

9. Harris, L. A., W. Ernst, and V. J. Tennery. A High-Temperature X-Ray and Thermal Analysis Study of Synthetic Dawsonite. Am. Miner., v. 56, Nos. 5-6, May-June 1971, pp. 1111-1113.

10. Hite, R. J., and J. R. Dyni. Potential Resources of Dawsonite and Nahcolite in the Piceance Creek Basin, Northwest Colorado. Quart. Colo. Sch. Mines, v. 62, No. 3, July 1967, pp. 25-38.

11. Karr, C., Jr., and J. J. Kovach. Far-Infrared Spectroscopy of Minerals and Inorganics. Appl. Spectry., v. 23, No. 3, May-June 1969, pp. 219-223.

12. Klug, H. P., and L. E. Alexander. X-Ray Diffraction Procedures for Polycrystalline and Amorphous Materials. John Wiley & Sons, Inc., New York, 1954, 716 pp.

[8]Titles enclosed in parentheses are translations from the language in which the item was published.

13. Larsen, E. S., and H. Berman. The Microscopic Determination of Nonopaque Minerals. U.S. Geol. Survey Bull. 848, 2d ed., 1934, p. 157.

14. Mandarino, J. A., and D. C. Harris. New Canadian Mineral Occurrences: I. Eucryptite, Pollucite, Rozenite, Epsomite, Dawsonite, Fairchildite and Bütschlüte. Can. Miner., v. 8, pt. 3, 1965, pp. 377-381.

15. Müller-Vonmoos, M., and R. Bach. Thermoanalytical--Mass Spectrometrical Investigation of an Oil Shale Containing Dawsonite. Paper in Thermal Analysis: Inorganic Materials and Physical Chemistry, ed. by R. F. Schwenker, Jr., and P. D. Garn. Academic Press, New York, v. 2, 1969, pp. 1229-1238.

16. Palache, C., H. Berman, and C. Frondel. Dana's System of Mineralogy. John Wiley & Sons, Inc., New York, v. 2, 7th ed., 1951, pp. 276-278.

17. Smith, J. W., and C. Milton. Dawsonite in the Green River Formation of Colorado. Econ. Geol., v. 61, No. 6, September-October 1966, pp. 1029-1042.

18. Smith, J. W., and N. B. Young. Determination of Dawsonite and Nahcolite in Green River Formation Oil Shales. BuMines Rept. of Inv. 7286, 1969, 20 pp.

19. Stewart, J. M. X-Ray 67 Program System for X-Ray Crystallography. Tech. Rept. 67-58, Univ. of Maryland, College Park, Md., December 1967, 181 pp.

20. Van Nordstrand, R. A. (assigned to Sinclair Research, Inc.). Process for the Recovery of Aluminum Values From Retorted Shale and Conversion of Sodium Aluminate to Sodium Aluminum Carbonate Hydroxide. U.S. Pat. 3,389,975, June 25, 1968.

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