THE AMERICAN MINERALOGIST, VOL. 52, SEPTEMBER–OCTOBER, 1967

COMPUTER PROGRAM FOR REFINING CELL PARAMETERS FROM WEISSENBERG PHOTOGRAPHS AND ITS APPLICATION TO INDEXING THE POWDER PATTERN OF GAYLUSSITE

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Introduction

In a recent paper Main and Woolfson (1963) proposed a technique for obtaining accurate lattice parameters from zero-level Weissenberg photographs. The method derives refined cell constants by applying least-squares analysis to $\alpha_1-\alpha_2$ separations of high-angle reflections. For theory of the Main-Woolfson method, the reader is referred to their original paper. We have written a Fortran code, following the Main-Woolfson scheme, and have tested it by obtaining accurate lattice parameters of gaylussite which are then used to index its powder pattern. The cell parameters of gaylussite are also obtained independently from its powder pattern using a least squares program written by Argonne National Laboratory.

Computer Program

A FORTRAN IV program has been written following the scheme set forth by Main and Woolfson. The program is applicable to any of the seven crystal systems. The data consist of camera diameter, K_{α_1} and $(K_{\alpha_1}-K_{\alpha_2})$ wavelengths, approximate cell parameters and a code number for crystal system followed by Miller indices and their $\alpha_1-\alpha_2$ separations. Observations may be weighted according to Eq. (7) in the Main-Woolfson paper. The output gives the coefficient matrix of the normal equations, shifts of the lattice parameters after each cycle, and finally, the refined parameters with standard deviations. Iterations are terminated when shifts are less than the pre-determined values. Program listings and copies of the FORTRAN source deck can be obtained from the authors.

Gaylussite

The ASTM X-ray powder data file shows two cards, 2–0122 and 9–482, on gaylussite, $Na_2Ca(CO_3)_2\cdot 5H_2O$. Both are unindexed and not mutually in agreement. No other published data are available.

The crystallographic data for gaylussite (locality: Searles Lake, San Bernardino, California) was obtained from precession photographs and are summarized in Table 1. A body-centered cell was chosen to conform to the morphological data (Palache, Berman and Frondel, 1951). The matrix for transformation to a *C* face-centered cell is:

$$\begin{bmatrix} 1 & 0 & \bar{1} \\ 0 & 1 & 0 \\ 0 & 0 & 1 \end{bmatrix}$$

Experimental Procedure

For the purpose of obtaining accurate cell parameters, ordinary zero-level Weissenberg photographs around three axes were taken and indexed. The separations of $\alpha_1-\alpha_2$ doublets of 15 suitable reflections (2θ angles between 114° and 152°) were measured with a travelling

Table 1. Crystallographic Data for Gaylussite

	This Study	Pratt[1]	Dana (morphological)[1]
nα	1.445±0.002	1.4435	
nβ	1.516±0.002	1.5156	
nγ	1.522±0.002	1.5233	
a	11.593±0.024Å		
b	7.779±0.011Å		
c	11.211±0.024Å		
β	102°00′±30′		101°33′
Space Group	$I\,2/a$		$2/m$
Z	4		
Axial Ratio	1.4903:1:1.4412		1.4897:1:1.4441
Dx	1.989 g/cm³		
Dm[2]	1.995± .01 g/cm³		1.991 g/cm³

[1] As quoted in Dana's *System of Mineralogy*, 7th ed., Vol. II, (John Wiley and Sons, 1951) p. 234.

[2] Sink-float method.

microscope, and averages of two measurements for each separation were taken. Using approximate cell parameters obtained from precession photographs, the cell parameters were refined by the program previously described. Convergence was achieved in two cycles and yielded the cell constants listed in Table 1.

In order to determine "goodness of fit" the parameters were used to index a 1/8°/min diffractometer scan of gaylussite internally calibrated with spinel and using CuKα radiation. The indexed powder pattern is shown in Table 2. As can be noted, the matching between d(obs) and d(calc) is remarkable.

Cell parameters were then independently obtained by use of an Argonne National Laboratory least-squares program[1] which minimizes the difference between d(calc) and d(obs) using unequivocally indexed powder lines. The parameters thus obtained—a = 11.589±0.003 Å, b = 7.779±0.002 Å, c = 11.207±0.003 Å and B = 101°58′±0.9′—compare favorably with those herein reported, namely, a = 11.593±0.024 Å, b = 7.779±0.011 Å, c = 11.211±0.024 Å and β = 102°00′±30′. The higher standard deviations shown by the $\alpha_1-\alpha_2$ method are mainly due to the small number of observations used.

[1] Program B106 by J. Gvildys, 1964.

TABLE 2. INDEXED POWDER DATA FOR GAYLUSSITE

d(obs)	I/I_0	d(calc)	hkl	d(obs)	I/I_0	d(calc)	hkl
6.407	57	6.415	110	2.139	7	2.138	330
6.344	10	6.345	011	1.999	5	1.998	422
5.668	7	5.670	200	1.991	6	1.992	$52\bar{1}$
5.489	5	5.483	002	1.923	8	1.924	$42\bar{4}$
4.505	16	4.506	$21\bar{1}$	1.918	9	1.917	332
4.426	14	4.428	$20\bar{2}$	1.907	5	1.908	233
3.944	4	3.945	112			1.899	$14\bar{1}$
3.890	1	3.890	020	1.899	6	1.900	$51\bar{4}$
3.561	3	3.561	$12\bar{1}$			1.898	512
3.417	5	3.419	121	1.856	3	1.856	$20\bar{6}$
3.307	5	3.308	013	1.829	5	1.830	125
3.205	100	3.207	220	1.827	6	1.828	006
3.165	3	3.167	$31\bar{2}$	1.815	3	1.815	$11\bar{6}$
3.123	5	3.125	$21\bar{3}$	1.791	2	1.791	$33\bar{4}$
2.921	9	2.922	$22\bar{2}$	1.753	1	1.752	$31\bar{6}$
2.726	51	2.728	$32\bar{1}$			1.754	611
2.696	11	2.698	$20\bar{4}$	1.733	3	1.734	$60\bar{4}$
2.684	9	2.685	$12\bar{3}$			1.734	$34\bar{1}$
2.635	54	2.635	$11\bar{4}$	1.723	8	1.723	$14\bar{3}$
2.510	26	2.510	123	1.705	1	1.706	116
2.419	5	2.420	114			1.706	$40\bar{6}$
2.385	2	2.386	$41\bar{3}$	1.680	3	1.680	$23\bar{5}$
2.360	3	2.361	$31\bar{4}$	1.674	5	1.674	143
2.352	4	2.352	$32\bar{3}$	1.624	2	1.624	$34\bar{3}$
2.328	3	2.328	402	1.611	1	1.611	523
2.291	5	2.291	420			1.540	051
2.267	3	2.266	231	1.540	2	1.541	150
2.257	6	2.256	132			1.541	235
2.217	7	2.217	$22\bar{4}$	1.489	4	1.490	$54\bar{1}$
2.177	4	2.177	510			1.488	$72\bar{3}$
		2.178	$51\bar{2}$				

CONCLUSION

The method of obtaining accurate lattice parameters from Weissenberg photographs is simple, rapid, and quite accurate, when the accuracy needed does not justify more time-consuming methods.

REFERENCES

MAIN, P. AND M. M. WOOLFSON (1963) Accurate lattice parameters from Weissenberg photographs. *Acta Crystallogs.* 16,, 731–733.

PALACHE, C., H. BERMAN AND C. FRONDEL (1951) *The System of Mineralogy*. Vol. 2, 7th ed. John Wiley and Sons, New York.