PLATTNERITE, A DESCRIPTION OF THE SPECIES FROM NATURAL CRYSTALS

by

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All of the specimen material used in this study came from the mineralogical collections of the United States National Museum.

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ABSTRACT

Plattnerite is redescribed from a study of natural crystals. A semiquantitative spectrographic analysis showed the crystals to be essentially pure PbO_2. The density is 9.564 ± 0.074 g/cm^3 (calculated 9.558). The symmetry is 4/m2/m2/m; ditetragonal dipyramidal. The ditetragonal dipyramid L $\{131\}$ is a newly reported form and the $\{011\}$ twin is described. The axial ratio from morphology is a:c = 1:0.6830. X-ray examination gave unit cell dimensions of a = 4.959 Å and c = 3.379 Å with an axial ratio of 1:0.6814.

INTRODUCTION

Plattnerite is natural lead dioxide, a tetragonal mineral which is isostructural with rutile and typically is found in oxidized ore deposits.

The purpose of this paper is to present a unification and refinement of data gathered in an extensive study of plattnerite. Existing data are not of high caliber; most were obtained from poor quality, natural material and synthetic crystals. New x-ray, morphological, chemical, and physical property determinations are reported. Good quality, natural crystals from the Ojuela mine, Mapimi, Durango, Mexico, were used for most of the work.

HISTORICAL BACKGROUND

Lead dioxide was first described as a mineral species in 1837 by August Breithaupt who named it schwerbleierz. The mineral later was given the name plattnerit by Haidinger (1845). Over the years numerous workers have contributed to the data relating to plattnerite but because of the lack of measureable natural crystals and good clean material, a great deal of this information is inaccurate, incomplete, and contradictory. Breithaupt's type material, believed to have come from Leadhills, Scotland, was poor and his description of it was so inadequate that Greg and Lettsom (1858) and Dana (1868) relegated plattnerite to the status of "a doubtful species." Not only were Breithaupt's "hexagonal" crystals of the mineral thought to be pseudomorphs after pyromorphite but later workers believed that the specific gravity was too high for lead dioxide.

In 1889, Yeates (1892) identified as plattnerite a specimen from the You Like lode, Hunter mining district, three miles northwest of Mullan, Idaho. This material was mostly massive but included crystals on which goniometry was done by Edward F. Ayres (Yeates and Ayres, 1892). The poor quality of these measurements was admitted by Ayres.

The crystals were good enough to establish that the mineral is tetragonal, however.

Structural data were added by Darbyshire (1932) through x-ray powder pictures of artificial crystals. His unit cell dimensions are those given for plattnerite in Palache, Berman, and Frondel (1944). Davidson (1941) contributed morphology measurements obtained from synthetic crystals. He recognized four forms, only one of which, {011}, has been observed on natural crystals. The {011} twin was first reported by Davidson; the angle of the twin is given as 68°.

No significant refinement of the data has appeared in the literature since the work of Darbyshire and Davidson.

OCCURRENCE AND ASSOCIATIONS

Plattnerite from the Ojuela mine is developed on soft, porous limonite-goethite as druses and sprays of sharp crystals. The most common associated minerals are hemimorphite and calcite but cerussite, rosasite, aurichalcite, and hydrozincite have been observed with plattnerite on some specimens. Many of the secondary minerals at the Ojuela mine have been described by Foshag (1937), Mrose (1948), and Desautels (1963). A similar occurrence of plattnerite at Goodsprings, Nevada, has been described by Takahashi (1960).

The stability field of plattnerite has been delineated by Garrels (1960), Takahashi (1960) and others. The mineral forms under very highly oxidizing conditions, just below the decomposition threshold of H_2O, in neutral to strongly alkaline environments. All of the specimens examined exhibit characteristics and mineral associations compatible with these conditions.

Breithaupt (1837) first described plattnerite from material believed by him to have come from Leadhills, Scotland. Kinch (1886) described another specimen known to have come from Leadhills, and Heddle (1889) wrote about a find from the Belton Grain vein, Wanlockhead, Scotland. In 1889, Yeates (1892) identified and described plattnerite from the You Like lode, Mullan, Idaho.

For a long period of time no new occurrences were reported until Palache, Berman, and Frondel (1944) cited three more localities in Idaho and one at Tsumeb, South-West Africa. Ojuela mine specimens began to appear in the late 1950's. Bariand (1959) wrote a short note about the discovery of crystals at the Tchach-Mille and Dare-Zandjir mines, Yezd, central Iran. In the same year Golovanov (1959) reported on the Kurgashinkan occurrence, followed by Takahashi's (1960) Goodsprings, Nevada, find. Bideaux, Williams, and Thomssen (1960) list seven new occurrences in Arizona, in addition to the one at the Glove mine, Santa Cruz County, cited by Galbraith and Brennan (1959).

Additional associated minerals from these localities include pyromorphite, leadhillite, malachite, azurite, murdochite, wulfenite, smithsonite, vanadinite, bayldonite, minium, and massicot.

COMPOSITION

Plattnerite is the tetragonal dimorph of lead dioxide, having a unit cell content of 2[PbO_2]. It is the β PbO_2 phase; the orthorhombic dimorph αPbO_2 described by Zaslavskii, et al. (1950, 1952) is not known to occur in nature.

Because plattnerite is such a simple compound, it was believed that a wet chemical analysis would contribute little useful information so none was performed. A sample of approximately 500 mg. was submitted to the U. S. Geological Survey for a semiquantitative spectographic analysis. The small size of the crystals necessitated a partial crushing and hand-picking, under the microscope, of the crystals and crystal fragments that appeared to be clean. The greatest problem was removing the limonite-goethite which is the material upon which the Ojuela mine plattnerite crystals have developed. The Frantz Magnetic Separator was partially successful. Many crystals, however, contained cavities at the attached end which were filled with limonite-goethite. All of the crystals seen to contain such cavities were discarded from the selected sample. This process was used in preparing material for a density determination and an x-ray powder pattern as well.

The results of the analysis are presented in Table I. The total amount of trace constituents is less than 0.2 weight per cent. The largest contributor is iron at 0.1 percent which is almost certainly due to admixed limonite-goethite.

TABLE I

SEMIQUANTITATIVE SPECTROGRAPHIC ANALYSIS OF
PLATTNERITE FROM THE OJUELA MINE,
MAPIMI, DURANGO, MEXICO

Constituent	Percentage	Constituent	Percentage
Pb	Major	Mn	0.002
Fe	0.1	Ag	0.0001
Si	0.015	B	< 0.003
Al	0.01	Ba	0.0003
Mg	0.003	Cr	0.0001
Ca	0.03	Cu	0.005
Na	0.01	Mo	0.02
Ti	0.005	Tl	0.005

Analyst - Helen Worthing, U.S.G.S., Washington, D.C.

PHYSICAL PROPERTIES

Plattnerite is jet-black with a brilliant luster which dulls somewhat upon exposure. Extremely thin platy crystals from White Pine County, Nevada, are translucent yellow to red in color under the petrographic microscope (Fig. 3). The powder is deep chestnut brown. The mineral is brittle and it breaks conchoidally. No cleavage was detected.

Refractive indices were not determined because of the lack of immersion media of high enough index. Larsen (1924) obtained an index of 2.30 ± 0.05 on You Like lode material using lithium light. He remarked that the mineral was "very unsatisfactory material for optical study, clouded and nearly opaque" (p. 121). He noted that no birefringence could be recognized.

The small size of the crystals made a precise hardness determination impossible. Crystals were rubbed between glass alides but the results were inconclusive. Attempts by the author to measure the hardness using the Vicker's Microhardness Tester were unsuccessful because of the brittle nature of the crystals. Hardness is one property on which most of the prior investigators concur. Yeates (1892) obtained a Moh's scale hardness of about 5 and

Schneiderhohn (1931) measured 5-5.5, both on massive material from the You Like lode. Golovanov (1959) observed that crystals from Kurgashinkan would scratch glass and are, therefore, harder than 5.

Density was determined on a Berman density balance, using toluene of known density. An average of eight measurements gave a density of 9.564 ± 0.074 g/cm^3. One reading was made with the pycnometer, using toluene, which gave a density of 9.57. These values compare favorably with a theoretical density of 9.558 calculated by the author. The small size of the crystals comprising the measured samples contributed to the wide variance of the Berman balance measurements. Table II includes densities obtained by a number of investigators.

TABLE II

A COMPARISON OF DENSITIES FOR PLATTNERITE BY VARIOUS WORKERS

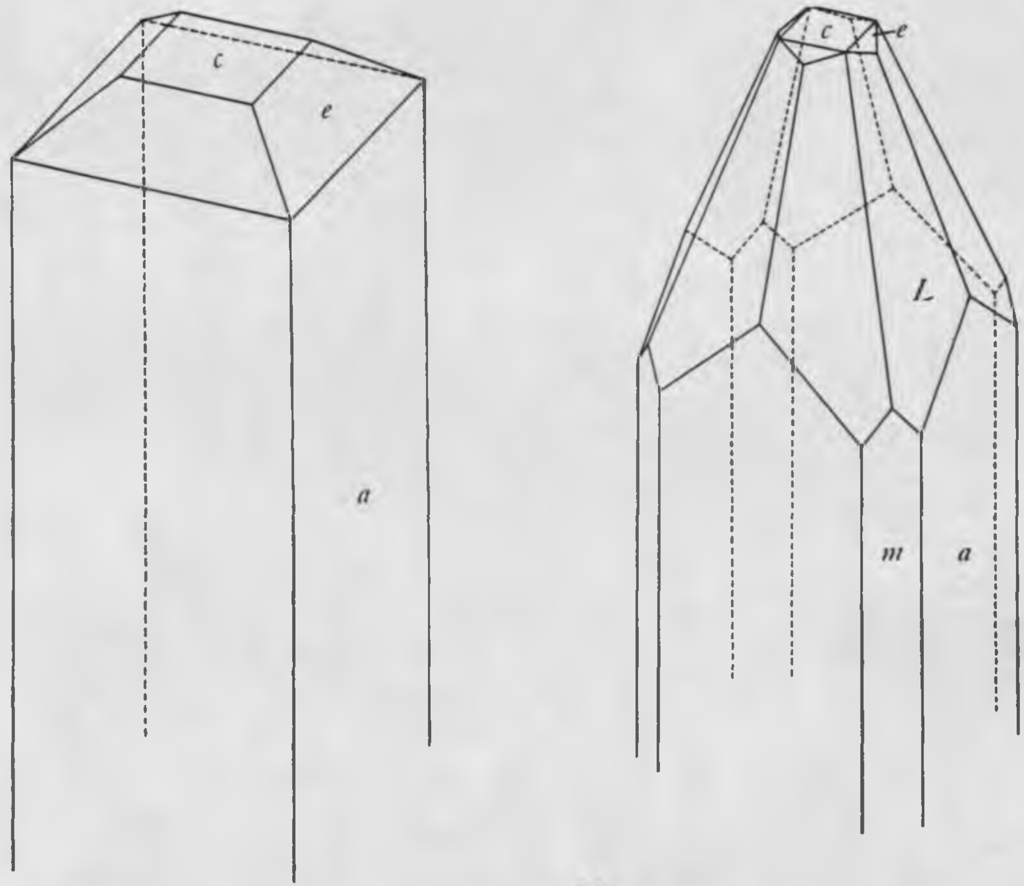
Density	Locality	Author
9.392* 9.448	Leadhills(?), Scotland	Breithaupt (1837)
9.4	Leadhills, Scotland	Greg and Lettsom(1858)
9.411	Idaho	Wheeler (1889)
8.56	You Like lode, Idaho	Yeates and Ayres(1892)
8.5	You Like lode, Idaho	Schneiderhohn (1931)
8.5-9.45	Synthetic crystals	Davidson (1941)
8.95	Kurgashinkan, USSR	Golovanov (1959)
9.564 ± .074	Ojuela mine, Mexico	White (this paper)
9.558 (Calc)	Ojuela mine, Mexico	White (this paper)

*Two determinations

CRYSTALLOGRAPHY

The class symmetry of plattnerite is ditetragonal dipyramidal (4/m2/m2/m). Crystals from the Ojuela mine are prismatic, elongated parallel to [001], and rarely exceed 1 mm. in length. Some are doubly terminated and many are cavernous. The faces are sharp and brilliant producing excellent signals for goniometric measurement. The crystals are relatively simple; no more than five forms were recognized. Most exhibit the five forms: first order prism a {010}, second order prism m {110}, ditetragonal dipyramid L {131}, tetragonal dipyramid e {011}, and pinacoid c {001}. Many exhibit only the second order dipyramid and prism in combination. The two most common habits are shown in Fig. 1a,b. A tendency toward unequal development is noted such that the symmetry of the terminal forms is not usually obvious. Most of the crystals on a single specimen have developed the same habit and approximate size.

The crystals from the You Like lode described by Ayres (Yeates and Ayres, 1892) were substantially different. Most displayed only the second order prism and a steep second order dipyramid {031}, with and without the pinacoid. Ayres recognized the dipyramid {011} and identified a first order dipyramid {332} on some crystals. The measurements of Ayres are included in Table III.



1a. 1b.

Fig. 1. Crystals of Plattnerite, Ojuela mine, Mapimi, Durango, Mexico.

TABLE III

PLATTNERITE ANGLE TABLE

(a) White* Symmetry 4/m2/m2/m a:c = 1:0.6830

Forms:		Phi	Rho	A	$\overline{M}$
c	001	---	0°00'	90°00'	90°00'
a	010	0°00'	90 00	90 00	45 00
m	110	45 00	90 00	45 00	90 00
e	011	0 00	34 20	90 00	66 30
L	131	18 26	65 11	73 19	66 03

*weighted mean of measurements on the nine best crystals from the Ojuela mine, Mapimi, Durango, Mexico.

(b) Ayres (Yeates and Ayres, 1892) You Like lode, Mullan, Idaho

a:c = 1:0.67643

Forms:

c	001	----	0°00'
a	010	0°00'	90 00
e	011	0 00	35 20½
v	031	0 00	63 46
x	332	----	----- (shown on crystal drawing but no angle data are given)

(c) Davidson (1941) - synthetic crystals a:c = 1:0.6785

Forms:		Meas. (Arith. Mean)		Computed	
e	011	0°00'	34°09½'	0°00'	34°09½'
-	111	44 30	43 30	45 00	43 49
-	121	63 30	56 30	63 26	56 38
-	230	32 32	85 00	33 40	90 00

Davidson (1941) was able to synthesize plattnerite crystals but these crystals did not exhibit faces which could be reliably measured--"the chief difficulty was caused by the minuteness of the pyramid faces. Although these crystals sometimes grow to a length of nearly 3 mm. their ends are usually mere points . . . . No normal signal was obtainable on any of the surfaces, but because of the small size of the pyramid faces it was possible to use a 'pinhole' image in measuring the angles."

The axial ratio derived from goniometric measurements of nine crystals from the Ojuela mine is a:c = 0.6830. This does not compare well with the axial ratio a:c = 1:0.6814 calculated from the powder pattern and 1:0.6816 from single crystal x-ray precession pictures. This discrepancy is apparently due to growth distortion of the prism faces which made it difficult to obtain consistent readings of the dipyramidal angles. In every case the dipyramid faces were overdeveloped on one side of the crystals so that their corresponding faces on the other side of the crystals were nearly undeveloped and produced poor signals. The readings varied by as much as $\pm 10'$ from the average. The goniometer was checked for accuracy.

Twinning

Plattnerite twins on $\{011\}$ as do rutile and cassiterite of the rutile group. Crystals exhibit both contact and penetration twins, the former predominating at the Ojuela mine. Polysynthetic twins were observed by the author but they are rare. Like rutile, the twins are usually flattened on (100) and are deeply furrowed parallel to the elongation so that accurate measurement of the twin angle is impossible. Twinned crystals rarely have visible terminal modifications, but taper to a narrow, irregular edge. The two best developed twins found were measured on the two-circle reflecting goniometer:

(1) Twin angle of 69°53' from reflections from two similar prism faces (010); good quality reflections.

(2) Twin angle of 68°50' from reflections from two pinacoids; poor quality reflections.

Considering the distorted nature of the twins, these angles compare very well with the twin angle of 68°32' obtained by doubling the (011) rho angle calculated from the powder data, establishing that these are twins and not merely randomly intergrown crystals. Measurements were also made of the twin angle from photomicrographs of Ojuela mine crystals. Again the distortion of the crystals precluded high precision but a value of approximately 68°30' was obtained, in good agreement with the other data. One of the photomicrographs taken for this purpose is shown in Fig. 2.

Davidson (1941) was the first to observe twinned plattnerite. He reported that long, tapering synthetic crystals formed crosses at angles of 68°.

Twinned, tabular, branching crystals were observed by the author on a specimen in the United States National Museum (USNM No. R16109) labelled only White Pine County, Nevada (Fig. 3). The extremely small size of these crystals (<0.2 mm.) required viewing under high magnification with the petrographic microscope. The twin angle was measured by aligning one set of the twin branches along a crosshair and rotating the stage until the other set came into alignment with the same crosshair. The angle measured was 79°(±30'), which is about 10° more than it should be were the crystals twinned on {011} and flattened on (100). However, if it is assumed that the flattening is parallel to the (130) plane, then the proper twin angle of 68°32' might seem to increase 10° to an angle of 79½°. The (130) face has not been ovserved on crystals, natural or synthetic, but the powder pattern reveals the presence of a (130) plane which produces a strong reflection.

The {011} twin is thus the only law demonstrated for plattnerite. The author investigated the possibility that

Fig. 2. Plattnerite Crystal Twinned on {011}; Ojuela mine, Mapimi, Durango, Mexico (100X).

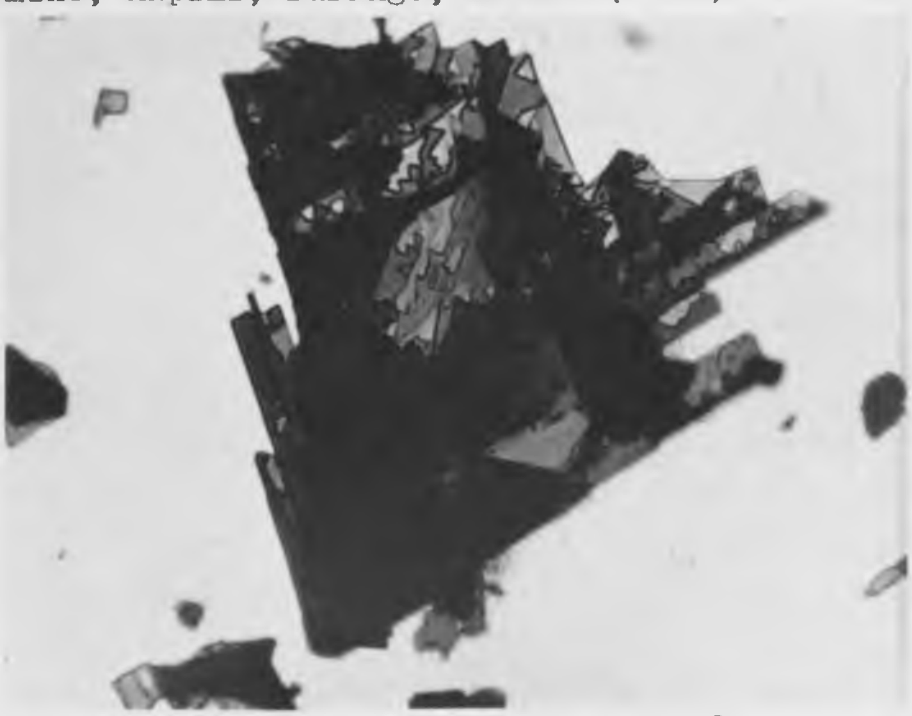


Fig. 3. Plattnerite Crystal Twinned on {011} and Flattened on (130); White Pine County, Nevada.

the excessively high angle could be explained by twinning on another prominent plane, and found that no proper angular relationship exists to permit such an explanation.

X-RAY STUDY

An x-ray diffraction photograph was taken of powdered plattnerite from the Ojuela mine by the Debye-Scherrer method using a Wilson film positioning and CuK radiation with a nickel filter. Forty-eight lines were measured and indexed (Table IV) and unit cell parameters determined. The powder data of Darbyshire (1932) and Golovanov (1959) are included for comparison. Darbyshire's data appear on card 8-185 of the Powder Diffraction File prepared by the American Society for Testing Materials.

The film was measured for shrinkage which was found to be negligible. The relative intensities of the lines on the powder pattern were measured from a graph of the pattern made with a Siemens recording photometer. The largest peak was assigned an intensity of 100 and the other lines scaled proportionally. Refinement of the unit cell parameters by extrapolation to 90° and the least squares method (Figs. 4, 5) gave dimensions of a=4.959 Å and c = 3.379 Å which yielded an axial ratio of 1:0.6814. Darbyshire obtained parameters of a = 4.931 and c = 3.367 and an axial ratio of 1:0.6828 by the powder method on synthetic crystals.

Single crystal precession pictures were taken with crystals mounted so as to precess about the 001 and 100 axes. Systematic extinctions confirmed that the space group is the rutile type, P4/mnm, as previously noted by numerous investigators (van Arkel, 1925; Ferrari, 1925; Goldschmidt, 1926; and Zaslavskii, 1952). Unit cell dimensions obtained were a = 4.953 Å and c = 3.376 Å which give an axial ratio of 1:0.6816. These parameters are in good accord with those obtained from the powder pattern.

TABLE IV

X-RAY POWDER DATA FOR PLATTNERITE

OJUELA MINE				YOU LIKE LODE[2]				KURGASHINKAN[3]	
Measured		Calculated		Measured		Calculated		Measured	
I[1]	d_{hkl}	d_{hkl}	hkl	I	d_{hkl}	d_{hkl}	hkl	I	d_{hkl}
*5	3.892		(110)						
100	3.500	3.500	110	10	3.51	3.494	110	6	3.49
*5	3.100		(011)					1	2.99
94	2.793	2.791	011	8	2.80	2.787	011	6	2.79
40	2.469	2.472	020	4	2.47	2.471	020	4	2.48
6	2.438	2.432	111						
5	2.224	2.217	120					2	2.06
*5	2.060		(121)					1	1.94
80	1.855	1.853	121	9	1.852	1.849	121	10	1.85
17	1.752	1.752	220	3	1.749	1.746	220	4	1.74
13	1.692	1.689	002	1	1.689	1.687	002	1	1.69
19	1.568	1.568	130	4	1.566	1.562	130	4	1.57
23	1.524	1.522	112	4	1.523	1.519	112	5	1.52
19	1.486	1.484	031	4	1.484	1.480	031	5	1.490
14	1.398	1.396	022	2	1.395	1.393	022	4	1.410
19	1.274	1.273	231	4	1.274	1.270	231	2	1.265
7	1.240	1.239	040	1	1.238	1.235	040	1	1.252
11	1.218	1.216	222	2	1.215	1.213	222	6	1.232
7	1.169	1.168	330	1	1.168	1.164	330	8	1.187
14	1.151	1.149	132	3	1.148	1.146	132		
14	1.133	1.133	141	3	1.133	1.129	141		
9	1.109	1.108	240	2	1.108	1.105	240	5	1.119
9	1.102	1.104	331	1	1.100	1.101	331		
△12	1.005	1.004	123	4	1.005	1.002	123	5	1.010
+6	1.001		(123)						
6	0.972	0.972	150	1	0.972	0.969	150		
13	0.961	0.961	332	1	0.960	0.958	332		
15	0.951	0.951	{051, 341}	4	0.952	0.948	{051, 341}		
7	0.934	0.934	151	4	0.929	0.931	151		
△10	0.927	0.927	242						
+7	0.927		(242)						
12	0.887	0.888	251	4	0.888	0.886	251		
6	0.877	0.876	440	½	0.877	0.874	440		
△9	0.872	0.871	233	3	0.872	0.870	233		
+6	0.872		(233)						
△6	0.851	0.850	350	1	0.851	0.853 0.848	342 350		
+4	0.849		(350)						
8	0.842	0.844	004	4	0.842	0.844	004		
△20	0.823	0.822	413	5	0.822	0.824	060		
+14	0.822		(413)						
8	0.802	0.799	024	1	0.800	0.799	024		
+6	0.802		(024)						
△11	0.793	0.793	161	3	0.792	0.790	161		
+8	0.792		(161)						
△9	0.784	0.784	260	1	0.784	0.782	260		
+7	0.784		(260)						
△12	0.778	0.778	442	2	0.778	0.776	442		
+10	0.778		(442)						

*Copper $K\beta$ line
△Copper $K\alpha_1$ line
+Copper $K\alpha_2$ line

(Camera diameter 114.59 mm., Nickel filter, $CuK\alpha$ = 1.5418 Å, Copper $K\beta$ = 1.3922 Å, Copper $K\alpha_1$ = 1.5405 Å, Copper $K\alpha_2$ = 1.5443 Å)

1. Relative intensities measured from Siemens recording photometer trace.

2. Darbyshire (1932)

3. Golovanov (1959)

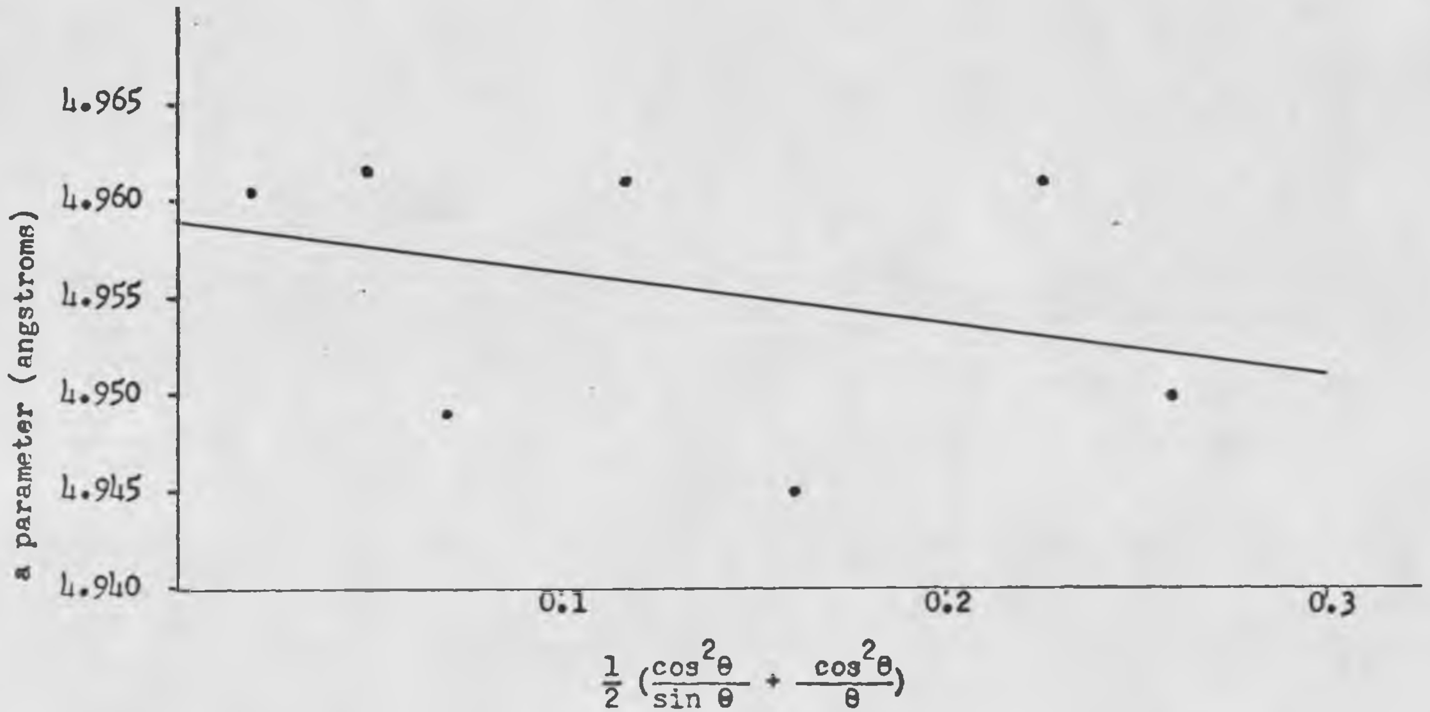


Fig. 4.a Unit Cell Dimension Refinement Using the Nelson-Riley Function and Least Squares Method.

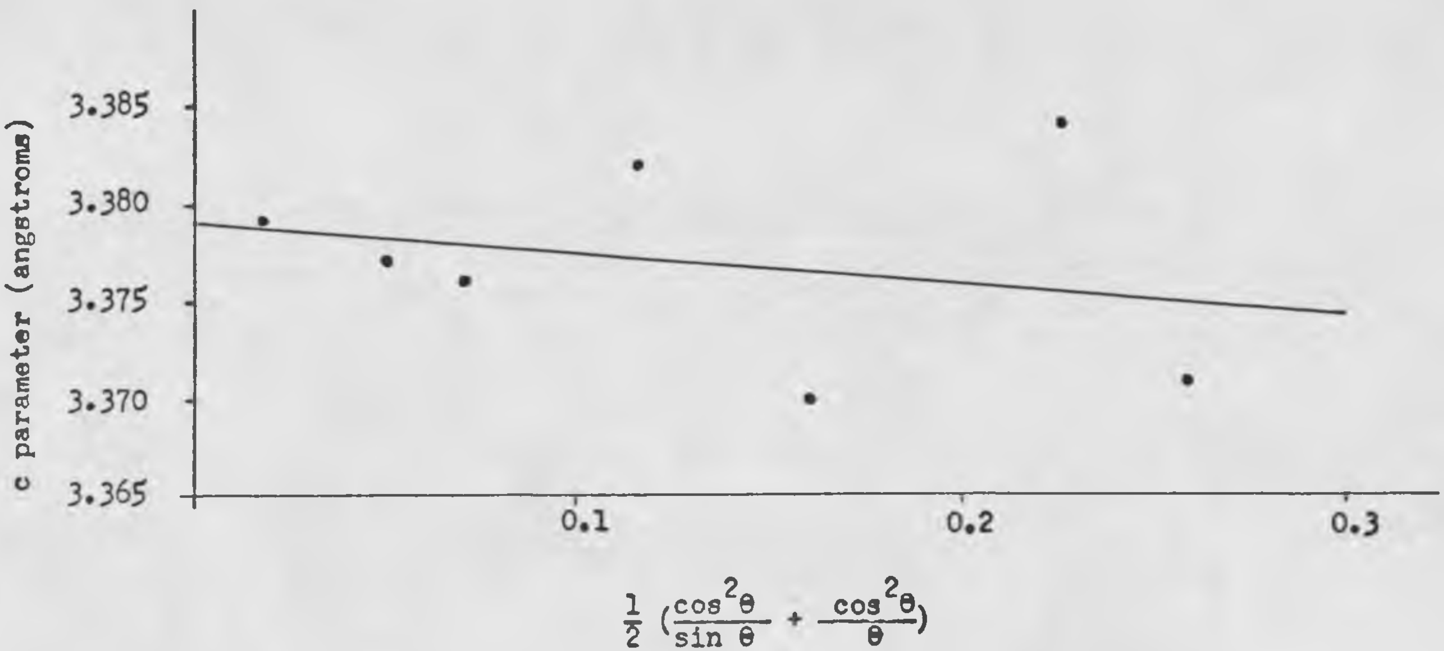


Fig. 5. c Unit Cell Dimension Refinement Using the Nelson-Riley Function and Least Squares Method.

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