

Yedlinite, a New Mineral from the Mammoth Mine, Tiger, Arizona

W. JOHN McLEAN,

*Department of Geosciences, University of Arizona,
Tucson, Arizona 85721*

RICHARD A. BIDEAUX,

1242 West Pelaar, Tucson, Arizona 85705

AND RICHARD W. THOMSEN

2138 Camino El Ganado, Tucson, Arizona 85718

Abstract

Yedlinite is a new hydrated oxychloride of lead and chromium found associated with diaboileite, quartz, wulfenite, diopside, phosgenite, and wherryite on specimens from the Mammoth Mine, Tiger, Arizona. Yedlinite occurs as prismatic crystals up to one millimeter long which are red-violet, transparent to translucent and somewhat sectile, with white streak, Mohs' hardness of about 2 1/2, and observed density of 5.85 g/cc. Crystals show rhombohedral symmetry with forms {1120}, {1101}, {0001}, {1010} and {2021} in order of decreasing prominence. Crystals are occasionally doubly terminated and exhibit distinct {1120} cleavage. The morphological axial ratio is $c/a = 0.763(2)$. Yedlinite is optically uniaxial negative and dichroic with $\omega = 2.125$ (pale cobalt blue) and $\epsilon = 2.059$ (lavender); X-ray diffraction shows space group $R\bar{3}$ or $R\bar{3}$ ($R\bar{3}$ is indicated by morphology and confirmed by structure determination), $a = 12.868(2)$ Å, $c = 9.821(2)$ (hexagonal axes), $c/a = 0.7632(3)$. The most intense powder diffraction lines in order d (I) (hexagonal hkl) are: 2.952 **100** $3\bar{1}\bar{4}1$, 2.622 **68** 3142 and $13\bar{4}2$, 4.506 **65** $01\bar{1}2$, 6.44 **32** $11\bar{2}0$ and 2.473 **27** $32\bar{5}1$ and $03\bar{3}3$. Electron probe analysis combined with structural information yields the chemical formula $Pb_xCl_yCrX_zY_2$ with $X = O$ or (OH) and $Y = H_2O$ or (O,OH). The hexagonal unit cell content, $Z = 3$, and the calculated density is 5.80 g/cc. The name honors Mr. Neal Yedlin.

Introduction

The mineral herein described was first noted in 1967 by Mr. Neal Yedlin of New Haven, Connecticut, a well-known amateur mineralogist, collector, lecturer, and writer on micromounting in mineralogy, in whose honor it has been named. Mr. Yedlin first observed the species on material obtained from Schortmann's Minerals, Easthampton, Massachusetts, and deposited several fragments in the National Museum of Natural History. Other specimens were later noted in the NMNH collection and were included in an ongoing study by one of us (RAB) of the Mammoth Mine suite. All specimens seem to date from collections made at the mine in 1940–41 (Palache, 1941). About 150 crystals are presently known. The name and species have been approved by the IMA Commission on New Minerals and Mineral Names.

Occurrence

Yedlinite is known only from the Tiger locality and is found sparingly on a few specimens; perhaps the most notable is NMNH R-8171. It is associated with the most complex paragenesis yet observed in Mammoth Mine material. Minute, doubly-terminated quartz crystals, which replaced primary galena, formed a framework for deposition of diaboileite. This was later replaced by phosgenite and rarely matlockite, and altered to wherryite. Yedlinite crystals are commonly found growing upon and partly surrounded by diaboileite or in intimate contact with phosgenite. Wulfenite, diopside, cerussite, mimetite, willemite, hemimorphite, fluorite, and quartz were later superimposed. The latter assemblage is usually observed separately on other specimens from the mine. Yedlinite is rarely observed perched on diopside and among the fluorite and drusy quartz. Crys-

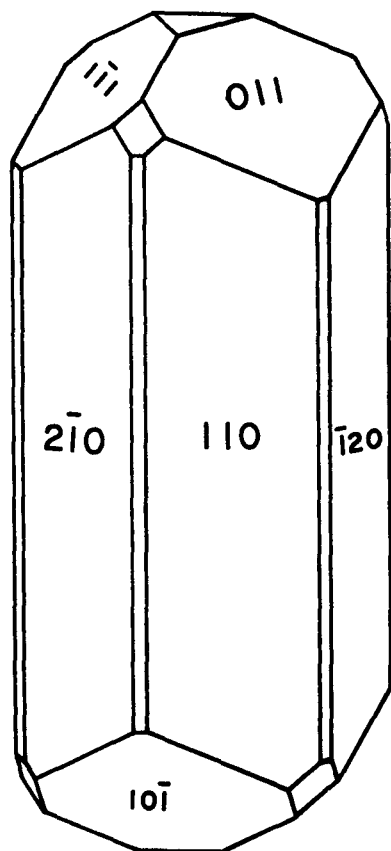


FIG. 1. Axonometric projection of an idealized crystal of yedlinite. The c axis is vertical, and a_2 is to the right. Faces of the two most important forms are indexed with the superfluous i index omitted.

tals of other minerals are never observed to be implanted on yedlinite. A little earthy hematite is occasionally present.

Chromium, essential to yedlinite, is apparently an extremely minor element in the Mammoth Mine ores. Exceptional fornacite crystals are rarely locally abundant. Crocoite has been reported, but cannot be confirmed by our observations. Sparsely occurring yellow-green cerussite and wherryite, yellow leadhillite, and brilliant red wulfenite may owe their color to this element.

Physical, Crystal and Optical Properties

Crystals of yedlinite are transparent to translucent hexagonal prisms up to about one millimeter in length and are generally about 0.4 as thick as they are long. The color is red-violet, and may vary both in hue and intensity within a single crystal. The

streak is white. The Mohs' scale hardness is about two and one half, and the mineral is generally not brittle and somewhat sectile. The densities, determined by weighing two single crystals and calculating their volumes from microscopic measurements, were 5.88 g/cc for a 21 μ g crystal and 5.81 for a 45 μ g crystal; the observed density is thus about 5.85 g/cc.

Crystal morphology suggests point group $\bar{3}2/m$ and is dominated by the second order hexagonal prism $\{11\bar{2}0\}$ and the relatively flat rhombohedron $\{1101\}$. Usually also present are the basal pinacoid $\{0001\}$, the first order prism $\{10\bar{1}0\}$, and sometimes the rhombohedron $\{20\bar{2}1\}$. The crystals are occasionally doubly terminated. The goniometric axial ratio using hexagonal axes is $c/a = 0.763 \pm 0.002$ based on measurement of 17 faces of the $\{11\bar{1}01\}$ form ($\rho = 41.4^\circ$) and seven faces of the $\{20\bar{2}1\}$ form ($\rho = 60.6^\circ$) on four crystals. An idealized crystal drawing is shown in Figure 1. Distinct prismatic cleavage, $\{11\bar{2}0\}$, is detectable.

Optically, yedlinite is uniaxial negative with $\omega = 2.125$ and $\epsilon = 2.059$ ($\lambda = 570$ nm). Crystals are

TABLE 1. Indexed Powder Pattern for Yedlinite*

$I_{rel.}$	$d_{obs.}$	$d_{calc.}$	hkl	$I_{rel.}$	$d_{obs.}$	$d_{calc.}$	hkl
9	7.375	7.368	101	4	1.530	1.527	621
32	6.440	6.434	110	2	1.502	1.498	306, 036
11	4.861	4.846	021	5	1.475	1.474	622, 262
65	4.506	4.494	012	2	1.461	1.459	226, 226
23	3.879	3.871	211, 121	1	1.439	1.436	245
11	3.719	3.715	300			1.402	155
11	3.287	3.274	003	6	1.399	1.397	164, 614
12	3.206	3.217	220			1.392	
		3.197	122			1.340	802
100	2.952	2.948	311	5	1.341	1.340	345
6	2.683	2.680	401			1.336	534
68	2.622	2.616	312, 132	4	1.289	1.290	633
27	2.473	2.474	321			1.285	165, 615
11	2.436	2.456	033	2	1.238	1.237	535
15	2.297	2.432	140, 410	1	1.215	1.215	372
11	2.297	2.295	223, 223	1	1.215	1.215	265
11	2.126	2.121	214	1	1.207	1.206	526, 526
5	1.953	1.952	413, 143	5	1.159	1.160	191, 561
		1.936	242, 422			1.158	093
15	1.935	1.934	015			1.156	740
		1.922	314	3	1.137	1.136	562, 192
		1.857	600			1.134	464
8	1.855	1.853	152	2	1.104	1.107	328
		1.842	404			1.103	832
4	1.804	1.804	431	1	1.091	1.090	473
		1.784	250			1.082	167
19	1.787	1.780	215	2	1.031	1.030	930
4	1.719	1.716	342, 432			1.030	842
2	1.679	1.675	611, 161			.988	906
6	1.609	1.606	162	1	.981	.983	933
		1.605	045			.980	835
		1.571	531			.978	1.0.10
14	1.570	1.567	523, 253, 253	1	.894	.894	188
		1.557	325			.892	4.10.0

* Taken in a 114.6 mm Debye-Scherrer camera with $\text{CuK}\alpha$ radiation. A pseudo-powder pattern taken with a Gandolfi-type device contained an additional 33 lines, indicating extensive structure damage in producing a powder.

The superfluous i index is omitted from all hkl reflection indices.

moderately dichroic with ω pale cobalt blue and ϵ lavender and more strongly colored.¹

X-Ray Diffraction Study

Yedlinite single crystals were examined by the oscillation and Weissenberg techniques using $\text{CuK}\alpha$ ($\lambda = 1.5418 \text{ \AA}$) radiation. Systematic absences on Weissenberg films coupled with diffraction symmetry 3 showed the space group to be either $R\bar{3}$ or $R\bar{3}$. The morphological $\bar{3}$ axis indicated the space group to be $R\bar{3}$ and this was verified by the crystal structure determination (Wood, McLean, and Laughon, 1974). Measurements of 2θ for 29 Weissenberg reflections were used to refine the unit cell dimensions by the least squares method yielding $a = 12.868(2) \text{ \AA}$ and $c = 9.821(2)$ for the hexagonal cell ($a = 8.119 \text{ \AA}$ and $\alpha = 104.84^\circ$ for the rhombohedral cell). The X-ray axial ratio is $c/a = 0.7632(3)$.

A powder diffraction pattern was prepared using $\text{CuK}\alpha$ radiation and a 114.6 mm Debye-Scherrer camera. Relative intensities were visually estimated, and the lines were indexed (Table 1) using the hexagonal unit cell parameters and intensities calculated from the crystal structure.

Chemical Composition

Crystals of yedlinite were analyzed using an ARL electron microprobe. Wavelength scans disclosed only Pb, Cr, Mn, and Cl in major amounts and trace amounts of Cu and Fe. Quantitative data were collected using a one micron diameter beam at 15 kV, $0.15 \mu\text{A}$ and ten second counting periods. Ten peak readings were obtained for each of the four major elements, and averaged. Comparison with standards prepared from matlockite, hemihedrite, diaboiteite, and manganese metal was used to arrive at values for Pb, Cr, Cl, and Mn, respectively. Stoichiometry was assumed for these mineral species.

It was recognized from bubbling on the surface of yedlinite when exposed to the electron beam that an unknown quantity of volatiles was escaping and, consequently, that the counts obtained for non-

TABLE 2. Chemical Analysis of Yedlinite

Element	Probe	$\text{Pb}_6\text{Cl}_6\text{CrO}_6 \cdot 2\text{H}_2\text{O}$	$\text{Pb}_6\text{Cl}_6\text{Cr}(\text{OH})_6(\text{O},\text{OH})_2$
Mn	.7 wt. %	--	--
Pb	79.4	75.8 wt. %	75.7 wt. %
Cr	3.8	3.2	3.2
Cl	7.5	13.0	13.0
O	8.6*	7.8	7.8
H	--	.2	.3
	100.0	100.0	100.0

* By difference.

volatile elements would be erroneously high. The chemical formula derived from the microprobe analysis was therefore treated only as an approximation. As there is insufficient material to use other common analytical methods, it was necessary to determine the details of the composition by determining the crystal structure. The crystal structure analysis (Wood *et al.*, 1974) shows the hexagonal unit cell to contain $3[\text{Pb}_6\text{Cl}_6\text{CrO}_6\text{O}_2]$ with hydrogen undetermined. Depending on the valence of Cr, 4 or 7 hydrogens are required for neutrality. Many compositions are possible, but the most likely are $\text{Pb}_6\text{Cl}_6\text{CrO}_6 \cdot 2\text{H}_2\text{O}$, $\text{Pb}_6\text{Cl}_6\text{Cr}(\text{O},\text{OH})_6(\text{O},\text{OH})_2$, and $\text{Pb}_6\text{Cl}_6\text{Cr}(\text{OH})_6(\text{O},\text{OH})_2$. These compositions result in a calculated density of 5.80 which compares well with the measured density of 5.85 g/cc. Application of the rule of Gladstone and Dale with the measured optical data and the composition $3(\text{PbO}) + 3(\text{PbCl}_2) + \text{CrO}_3 + 2(\text{H}_2\text{O})$ indicates a density of 5.74. Analytical results are shown in Table 2.

Acknowledgments

We are grateful to the U. S. National Museum and to Mr. Neal Yedlin for the loan of specimen material. We are also indebted to Dr. Sidney A. Williams of Phelps Dodge Corporation for the nonroutine determination of the optical properties.

References

- PALACHE, C. (1941) Diaboiteite from Mammoth Mine, Tiger, Arizona. *Am. Mineral.* **26**, 605-612.
 WOOD, MICHAEL M., W. JOHN MCLEAN, AND ROBERT B. LAUGHON (1974) The crystal structure and composition of yedlinite. *Am. Mineral.* **59**, 1160-1165.

Manuscript received, December 13, 1973; accepted for publication, June 25, 1974.

¹Optical data were determined by Dr. Sidney A. Williams of Phelps Dodge Corporation.

The Crystal Structure and Composition of Yedlinite

MICHAEL M. WOOD,

Department of Earth Sciences, California State University,
Hayward, California 94542

W. JOHN McLEAN,

Department of Geosciences, University of Arizona,
Tucson, Arizona 85721

AND ROBERT B. LAUGHON

Johnson Space Center, Houston, Texas 77058

Abstract

The crystal structure of the new mineral yedlinite has been determined using the Patterson method and refined to $R = 0.14$. The structure indicates the composition to be $Pb_6Cl_6CrX_6Y_2$ where X is O^{2-} or OH^- , and Y is O^{2-} , OH^- , or H_2O . The structure contains a continuous three-dimensional framework of irregular $Pb(Cl_6X_2Y)$ polyhedra and CrX octahedra. Chromium seems to be in the $6+$ valence state, and the structure of yedlinite appears to contain the first recognized occurrence of an octahedrally coordinated Cr^{6+} ion.

Introduction

Several small crystals of yedlinite, a new mineral from the Mammoth Mine, Tiger, Arizona, were obtained from U.S. National Museum Specimen R-8171 for a study of the crystal structure. Yedlinite has been described by McLean, Bideaux, and Thomsen (1974) who report crystallographic data as follows: $a = 12.868$, $c = 9.821$ Å, space group = $R\bar{3}$, $Z = 3$, density (obs) = 5.85 gm/cc.

The chemical composition of yedlinite was in doubt at the inception of the structure study because determination of oxygen, hydroxyl, and water and of the valence of chromium could not be made from the chemical analysis. The elucidation of the structural formula of yedlinite was expected to be a contribution of the investigation.

Experimental

The process of preparing a sphere of the mineral for data collection was complicated by a distinct prismatic cleavage and a lack of brittleness. Intensity data were finally obtained using a very small spheroid of mean diameter about 0.045 mm and absorption coefficient, μR , 1.36 for Mo radiation. Data were collected on an automated Picker diffractometer using monochromatized Mo radiation,

and intensities less than 2σ were considered unobserved. Both $hkil$ and $hkil$ portions of the reflection sphere were collected and corrected for the usual geometrical factors. Approximate absorption corrections were applied using the spherical correction factors from pages 302-305 of Vol. 2 of the *International Tables for X-Ray Crystallography*. The structure factor values of symmetrically equivalent reflections within the data set were averaged. A general agreement of structure factors from equivalent planes increased confidence in much of the data, but systematic differences for a number of equivalent reflections, with $hkil$ generally having greater F than the equivalent $hkil$ reflection, gave evidence of sig-

TABLE 1. Position and Temperature Parameters in Yedlinite*

Atom	x	y	z	B
Pb	.1829(5)*	.1379(5)	.2121(5)	2.27(14)
Cl	.0869(31)	.2886(30)	.1050(37)	2.14(68)
Cr	.0000	.0000	.5000	.60(77)
X**	.133(8)	.091(8)	.604(9)	2.1(1.8)
Y**	.0000	.0000	.108(16)	3.6(3.2)

* Numbers in parentheses are estimated standard deviations and refer to the last places.

** $X=O^{2-}$ or OH^- ; $Y=O^{2-}$, OH^- , or H_2O

nificant uncorrected absorption error. No attempt was made to correct for the trend except through the averaging process. Only 290 observed structure factors were recorded. The weakness of the diffraction spectrum can be attributed to the very small crystal size, the substantial absorption of the crystal, and the soft and non-brittle nature of the mineral which promotes structure damage to the surficial parts of the crystal during grinding. Crystals after grinding gave notably weaker diffraction results than unground crystals of similar size. Because of the dearth of material, it seemed unlikely that a better sphere could be obtained. Attempts to collect data on unground crystal fragments proved unsatisfactory and were abandoned.

TABLE 2. Observed and Calculated Structure Factors for Yedlinite*

h	k	l	FO	FC	h	k	l	FO	FC	h	k	l	FO	FC
3	0	0	369	-324	2	0	2	121	45	1	0	4	183	224
6	0	0	335	293	5	0	2	72	-19	4	0	4	349	-348
9	0	0	235	-209	8	0	2	339	320	7	0	4	148	128
1	1	0	288	265	0	1	2	169	-515	10	0	4	328	-316
4	1	0	360	-325	3	1	2	838	858	2	1	4	528	-571
7	1	0	171	-158	9	1	2	127	143	5	1	4	117	98
2	2	0	286	-260	1	2	2	334	368	8	1	4	106	-122
5	2	0	181	-199	4	2	2	297	305	0	2	4	122	269
8	2	0	142	116	7	2	2	55	-31	3	2	4	372	-389
3	3	0	128	106	10	2	2	113	-102	6	2	4	184	184
6	3	0	195	-162	2	3	2	181	155	1	3	4	160	-171
9	3	0	381	347	5	3	2	92	82	4	3	4	178	198
1	4	0	454	413	8	3	2	364	-358	7	3	4	155	175
4	4	0	45	12	0	4	2	107	-90	2	4	4	268	272
7	4	0	350	322	3	4	2	436	-415	8	4	4	198	191
2	5	0	806	738	6	4	2	67	79	0	5	4	256	241
5	5	0	207	-183	9	4	2	161	-163	3	5	4	54	89
8	5	0	168	171	1	5	2	488	-472	6	5	4	157	-166
3	6	0	169	161	4	5	2	242	242	1	6	4	311	327
1	7	0	166	169	2	6	2	274	-258	4	6	4	421	-416
4	7	0	211	-171	5	6	2	351	336	5	7	4	171	-187
2	8	0	197	-148	3	7	2	327	285	1	9	4	175	192
3	9	0	242	234	1	8	2	55	1	2	10	4	115	138
1	0	1	79	156	3	10	2	295	-272	2	0	5	92	109
4	0	1	475	433	0	0	3	822	-730	5	0	5	187	142
7	0	1	117	-80	3	0	3	286	-253	0	1	5	156	418
10	0	1	305	262	6	0	3	209	-204	3	1	5	92	-120
2	1	1	370	373	9	0	3	151	-124	6	1	5	338	-320
5	1	1	191	-181	1	1	3	193	-352	1	2	5	174	-189
8	1	1	67	63	4	1	3	140	-96	4	2	5	162	-216
11	1	1	226	-221	7	1	3	262	239	7	2	5	208	-187
0	2	1	270	-296	2	2	3	302	-302	2	3	5	83	-8
3	2	1	438	430	5	2	3	444	428	5	3	5	409	-395
6	2	1	570	-535	8	2	3	170	-140	8	3	5	270	266
9	2	1	119	94	0	3	3	470	588	0	4	5	280	-336
1	3	1	122	86	3	3	3	212	-226	3	4	5	204	270
4	3	1	450	-419	6	3	3	358	343	1	5	5	395	435
7	3	1	67	-134	9	3	3	172	-177	2	6	5	202	211
2	4	1	154	131	1	4	3	173	-171	3	7	5	82	-93
5	4	1	174	-167	4	4	3	114	87	1	8	5	259	-239
8	4	1	72	109	7	4	3	253	-213	0	10	5	261	-288
0	5	1	123	-114	2	5	3	437	-425	0	0	6	205	-178
3	5	1	52	89	0	6	3	259	-197	3	0	6	380	407
6	5	1	236	198	3	6	3	182	-198	9	0	6	349	319
1	6	1	155	110	6	6	3	72	-60	1	1	6	109	184
4	6	1	302	292	4	7	3	144	-176	4	1	6	275	289
7	6	1	84	-138	2	8	3	294	256	7	1	6	81	-114
2	7	1	75	-89	0	9	3	391	360	2	2	6	371	433
0	8	1	365	-360	3	9	3	72	-127	5	2	6	329	-328
1	9	1	475	-438	1	10	3	133	155					

* Scale factor is 1.287.

TABLE 2, Continued

h	k	l	FO	FC	h	k	l	FO	FC	h	k	l	FO	FC
0	3	6	168	-277	-4	6	2	529	-446	-6	9	6	150	-186
3	3	6	66	105	-3	7	2	304	284	-5	10	6	111	-137
6	3	6	194	-205	-6	7	2	576	-475	-8	10	6	190	186
1	4	6	75	-75	-5	8	2	276	-224	-7	11	6	209	-231
4	4	6	69	-84	-4	9	2	185	176	-5	6	7	322	-351
5	5	6	249	231	-7	9	2	150	115	-4	7	7	269	-312
4	7	6	311	318	-9	10	2	409	351	-3	8	7	167	181
0	9	6	248	-230	-2	11	2	232	-182	-6	8	7	268	-249
1	0	7	379	-402	-4	12	2	349	-293	-8	9	7	114	104
4	0	7	112	102	-9	13	2	159	-180	-5	7	9	210	244
2	1	7	256	272	-4	5	3	371	-330	-2	10	0	124	116
3	2	7	147	157	-3	6	3	69	33	-2	9	1	195	201
6	2	7	144	138	-5	7	3	538	-452	-2	9	4	160	-171
1	3	7	85	77	-4	8	3	167	-111	-2	8	8	138	-175
2	4	7	296	-345	-7	8	3	184	-184	-2	8	5	170	-174
0	5	7	88	-159	-3	9	3	165	-181	-2	8	2	619	545
3	5	7	178	-196	-6	9	3	120	95	-2	7	0	235	-199
1	6	7	362	-395	-5	10	3	320	273	-2	7	3	260	-203
4	6	7	96	168	-4	11	3	148	-185	-2	7	6	378	372
0	8	7	94	122	-7	11	3	487	392	-2	7	9	140	-223
3	1	8	281	-321	-10	11	3	111	-102	-2	6	7	106	125
6	1	8	275	263	-3	12	3	307	-283	-2	6	1	72	-56
2	3	8	70	-102	-6	12	3	112	129	-2	5	2	108	-61
5	3	8	330	319	-9	12	3	156	-154	-2	5	5	352	-387
0	4	8	248	316	-5	13	3	194	-145	-2	5	8	259	355
1	5	8	92	-126	-3	5	4	211	183	-2	4	6	268	-352
0	0	9	339	392	-5	6	4	375	341	-2	4	3	569	587
3	0	9	267	-255	-4	7	4	246	219	-2	4	0	331	-261
6	0	9	126	128	-3	8	4	246	-476	-2	3	7	119	115
4	1	9	246	-245	-6	8	4	151	141					
2	2	9	250	-263	-8	9	4	356	-313	-1	2	0	334	265
1	4	9	110	139	-3	11	4	250	245	-1	2	3	103	97
2	5	9	227	238	-6	11	4	244	204	-1	3	8	161	337
1	0	10	219	240	-9	11	4	135	-114	-1	3	5	279	-424
					-8	12	4	114	127	-1	3	2	128	133
										-1	4	1	986	-934
										-1	4	4	337	352
										-1	4	7	137	198
										-1	4	10	179	-326
										-1	7	4	397	-373
										-1	7	1	479	406
										-1	8	0	157	-158
										-1	8	3	76	-77
										-1	8	6	193	186
										-1	9	5	180	-158
										-1	9	2	139	104
										-1	10	1	252	-222
										-1	10	4	182	147
										-1	12	2	158	206

Structure Determination and Refinement

Diffraction symmetry and systematic absences indicate the space group of yedlinite is either $R\bar{3}$ or $R3$. The structure was attempted in $R\bar{3}$ because the morphological symmetry of the crystals appears centric. Hexagonal axes were used throughout. A Patterson synthesis was used to locate the 18 lead atoms in a general position. This gave a conventional R factor of 0.21 after one cycle of full matrix least-squares refinement using a modified version of ORFLS (Busing, Martin, and Levy, 1962). The three chromium atoms were then located in a special position at $0,0,\frac{1}{2}$ from a Fourier difference map. Two additional occupied general positions were also detected, one with approximately twice the electron density of the other. The greater electron density was attributed to chlorine, and oxygen was assigned to

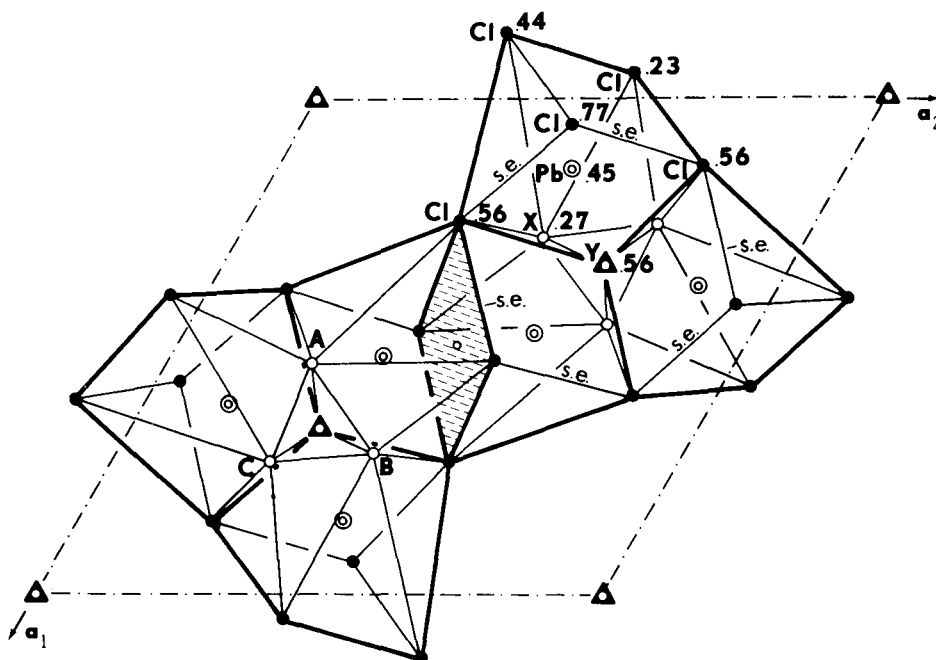


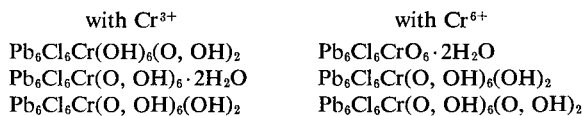
FIG. 1. A part of the structure of yedlinite viewed down the c axis. Shown are two pinwheels, each composed of three lead polyhedra. The linkage of the two adjacent pinwheels by face sharing (shaded face) is shown. Not explicitly shown are the pinwheels at the $\bar{3}$ axis passing through the cell origin and the linkage of center-of-symmetry related pinwheels along the $\bar{3}$ axis by sharing of Cl-Cl edges (s.e.) and through the Cr octahedron, one face of which is the equilateral triangle ABC.

the sites of lower electron density which were in octahedral coordination about the chromium atoms. Another Fourier difference map at this stage disclosed only the presence of oxygen-size peaks at a six-fold special position on the $\bar{3}$ axes. Additional least-squares refinement using isotropic temperature factors reduced R to 0.14, and subsequent difference maps failed to reveal significant electron density in the cell. Attempted refinements using anisotropic temperature factors reduced R to 0.11, but the tem-

perature factors appeared unrealistic, presumably because of unresolved absorption error. Position and isotropic temperature parameters are shown in Table 1, and observed and calculated structure factors are in Table 2.

Crystal Chemistry

The crystal structure of yedlinite strongly endorses the chemical formula, $\text{Pb}_6\text{Cl}_6\text{CrX}_6\text{Y}_2$, where X and Y are the oxygen species, O, OH, or H_2O . Since charge equilibrium can be achieved in the formula by juggling the combinations of oxygen species in the X and Y sites coupled with variation of the valence of chromium from three to six, there are many possibilities consistent with the general formula. Several of the more likely that we have considered are:



The Cr-O interatomic distance in yedlinite (Table 3) is 1.82(10) Å. One would predict a Cr^{3+} -O dis-

TABLE 3. Interatomic Distances and Angles in Yedlinite*

i	j	d_{ij}	i	j	d_{ij}	i	j	d_{ij}
Cr	X**	1.82(10)*	Pb	Cl	3.68(4)	Y	Y	2.12(32)
Pb	Cl	2.96(4)	Pb	Cl	4.41(4)	Y	X	3.20(13)
Pb	Cl	3.20(4)	Pb	X	2.70(10)	Cl	Y	3.30(4)
Pb	Cl	3.33(4)	Pb	X	2.53(10)			
Pb	Cl	3.38(4)	Pb	Y**	2.36(5)			

i	j	k	Angle $_{ijk}$	i	j	k	Angle $_{ijk}$
X	Cr	X	91.5(4.3)	X	Cr	X	88.5(4.3)

* Numbers in parentheses are estimated standard deviations and refer to the last places.

** X=O²⁻ or OH⁻; Y=O²⁻, OH⁻, or H₂O

tance of 2.00 Å, based on ionic radii for 6-fold coordination (Shannon and Prewitt, 1969), or about 1.98(8) Å from *International Tables for X-Ray Crystallography* (Vol. 3, 1968). A Cr^{6+} -O distance in 4-fold coordination would be 1.67 Å based both on the ionic radii of Shannon and Prewitt and the *International Tables*. A Cr^{6+} -O distance based on extrapolation of the values of Shannon and Prewitt would be about 1.81 Å for 6-fold coordination. The distance of 1.82 Å in yedlinite is consistent with a valence state of chromium of 6+, as is the occurrence of yedlinite in an oxidized assemblage.

Minimization of disorder favors the formula, $\text{Pb}_6\text{Cl}_6\text{CrO}_6 \cdot 2\text{H}_2\text{O}$. However, electrostatic neutrality of the oxygen sites is best achieved in $\text{Pb}_6\text{Cl}_6\text{Cr}(\text{O}, \text{OH})_6(\text{O}, \text{OH})_2$ where the *X* site deviates from neutrality by about 0.04 (not corrected for hydrogen bonding) and the *Y* site by about -0.10, well within normal limits. This, to our knowledge, would be the first recognized occurrence of Cr^{6+} in octahedral coordination. Minimal disorder and satisfactory oxygen site charge balance is achieved in $\text{Pb}_6\text{Cl}_6\text{Cr}(\text{OH})_6(\text{O}, \text{OH})_2$ with Cr^{3+} although a Cr^{3+} -O bond distance

is not in as good agreement with the observed Cr-O distance. A refinement of the structure to resolve these variances is impractical until a satisfactory crystal for data collection can be prepared.

Discussion of the Structure

The most pervasive and continuous part of the structure of yedlinite is the coordination framework around the lead atoms. The bond distance and angle information in Table 3 shows that lead is coordinated by two *X* oxygen atoms, one *Y* oxygen atom, and five chlorine atoms, forming an irregular polyhedron. A sixth chlorine at a distance of 4.41 Å is not included in the coordinating group, as bonding would be minimal at that distance.

Three symmetry-related lead polyhedra share faces—each face consisting of one chlorine, one *X* oxygen, and one *Y* oxygen—to form a planar three member “pinwheel” centered on a $\bar{3}$ axis (Fig. 1). Each pinwheel shares six Cl-Cl edges (e.g., s.e. in Fig. 1) with the centrosymmetrically related pinwheel above or below on this $\bar{3}$ axis. These double pinwheel groups are linked to form stacks parallel to

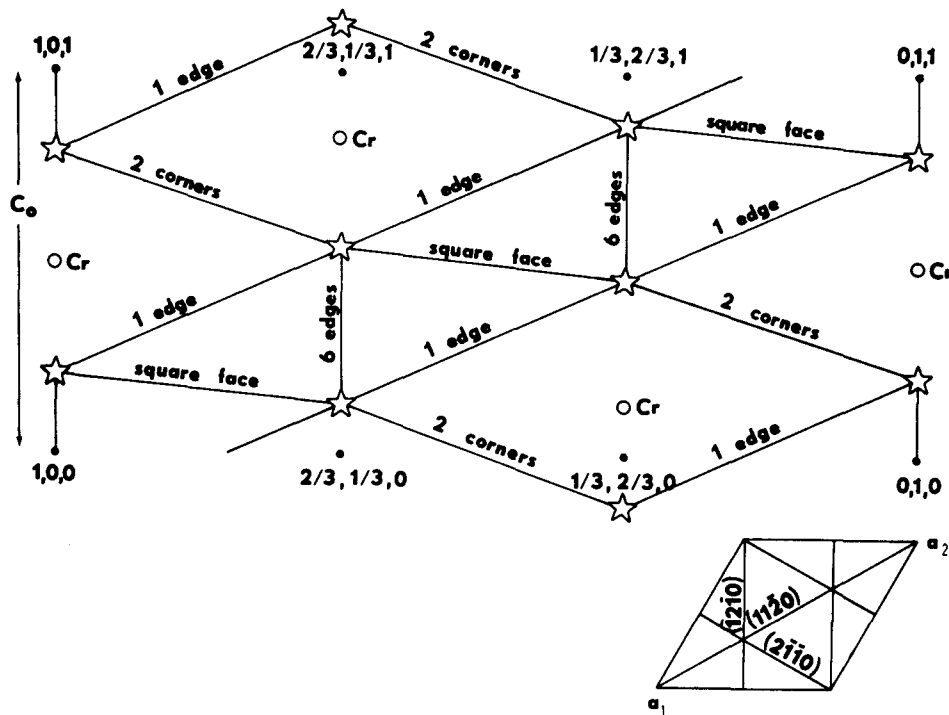


FIG. 2. A schematic view of the $(11\bar{2}0)$ section of the structure of yedlinite showing the distribution of shared elements among lead-coordination pinwheel groups. All sharing elements shown as lines involve only chlorine apices and Cl-Cl edges. The star symbol represents a single three-member pinwheel. The insert at the lower right shows the locations of similar sections in the cell.

