

Lead-tellurium oxysalts from Otto Mountain near Baker, California: VI. Telluroperite, $\text{Pb}_3\text{Te}^{4+}\text{O}_4\text{Cl}_2$, the Te analog of perite and nadorite

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ABSTRACT

Telluroperite, $\text{Pb}_3\text{Te}^{4+}\text{O}_4\text{Cl}_2$, is a new tellurite from Otto Mountain near Baker, California. The new mineral occurs on fracture surfaces and in small vugs in brecciated quartz veins in direct association with acanthite, bromine-rich chlorargyrite, caledonite, cerussite, galena, goethite, and linarite. Various other secondary minerals occur in the veins, including six new tellurates, housleyite, markcooperite, paratimroseite, ottoite, thorneite, and timroseite. Telluroperite is orthorhombic, space group *Bmmb*, $a = 5.5649(6)$, $b = 5.5565(6)$, $c = 12.4750(14)$ Å, $V = 386.37(7)$ Å³, and $Z = 2$. The new mineral occurs as rounded square tablets and flakes up to 0.25 mm on edge and 0.02 mm thick. The form {001} is prominent and is probably bounded by {100}, {010}, and {110}. It is bluish-green and transparent, with a pale bluish-green streak and adamantine luster. The mineral is non-fluorescent. Mohs hardness is estimated to be between 2 and 3. The mineral is brittle, with a curved fracture and perfect {001} cleavage. The calculated density based on the empirical formula is 7.323 g/cm³. Telluroperite is biaxial (–), with very small $2V$ (~10°). The average index of refraction is 2.219 calculated by the Gladstone-Dale relationship. The optical orientation is $X = c$ and the mineral exhibits moderate bluish-green pleochroism; absorption: $X < Y = Z$. Electron microprobe analysis provided PbO 72.70, TeO₂ 19.26, Cl 9.44, O≡Cl –2.31, total 99.27 wt%. The empirical formula (based on O+Cl = 6) is $\text{Pb}_{2.79}\text{Te}_{1.03}\text{O}_{3.72}\text{Cl}_{2.28}$. The six strongest powder X-ray diffraction lines are [d_{obs} in Å (hkl) I]: 3.750 (111) 58, 2.857 (113) 100, 2.781 (020, 200) 43, 2.075 (024, 204) 31, 1.966 (220) 30, and 1.620 (117, 313, 133) 52. The crystal structure ($R_1 = 0.056$) is based on the Sillén X_1 structure-type and consists of a three-dimensional structural topology with lead-oxide halide polyhedra linked to tellurium/lead oxide groups. The mineral is named for the relationship to perite and the dominance of Te (with Pb) in the Bi site of perite.

Keywords: Telluroperite, new mineral, tellurite, crystal structure, perite, nadorite, Sillén X_1 , Otto Mountain, California

INTRODUCTION

Telluroperite, $\text{Pb}_3\text{Te}^{4+}\text{O}_4\text{Cl}_2$, is one of seven new secondary lead-tellurium minerals discovered recently at Otto Mountain near Baker, California. It is the only one of the seven new minerals that is a tellurite (i.e., contains the Te^{4+} cation); all of the others are tellurates (i.e., contain the Te^{6+} cation). Detailed information on the mining history, geology, mineralogy, and mineral paragenesis of the deposit, as well as the discovery of the new minerals, is provided in Kampf et al. (2010b).

Telluroperite is isostructural with perite, PbBiO_2Cl , and nadorite, PbSbO_2Cl . As described below, charge balance requires that Te^{4+} and Pb^{2+} jointly occupy the $\text{Bi}^{3+}/\text{Sb}^{3+}$ site. The name “telluroperite” was chosen in preference to “telluronadorite” because perite also occurs at this locality and because the cell parameters of the new mineral are much closer to those of perite and their powder-diffraction patterns are almost identical. The new mineral

and mineral name have been approved by the Commission on New Minerals, Nomenclature and Classification of the International Mineralogical Association (IMA 2009-044). Four cotype specimens are deposited in the Natural History Museum of Los Angeles County, catalog numbers 62513 and 62514.

OCCURRENCE

Telluroperite was found in the Bird Nest drift on the southwest flank of Otto Mountain, approximately 2 km northwest of Baker, San Bernardino County, California. The Bird Nest drift (35° 16.606'N, 116° 05.956'W) is located 0.7 km northwest of the Aga mine (35° 16.399'N, 116° 05.665'W).

Telluroperite is very rare and occurs mostly on fracture surfaces and in small vugs in quartz veins. Species observed in direct association with the new mineral include acanthite, bromine-rich chlorargyrite, caledonite, cerussite, galena, goethite, and linarite. Other species identified in the Otto Mountain assemblages include: anglesite, atacamite, boleite, brochantite,

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burckhardtite, calcite, celestine, chalcopyrite, chrysocolla, devilline, diabloite, fluorite, fornicite, gold, hessite, iodargyrite, jarosite, khinite-4*O*, kuranakhite, malachite, mimetite, mottramite, munakataite, murdochite, muscovite, perite, phosphohedyphane, plumbojarosite, pyrite, schieffelinite, vanadinite, vauquelinite, wulfenite, and six other new minerals: housleyite [Pb₆Cu²⁺Te₄⁶⁺O₁₈(OH)₂] [IMA2009-024; Kampf et al. (2010c)], markcooperite [Pb₂(UO₂)Te⁶⁺O₆] [IMA2009-045; Kampf et al. (2010d)], ottoite [Pb₂Te⁶⁺O₅] [IMA2009-063; Kampf et al. (2010b)], paratimroseite [Pb₂Cu₄²⁺(Te⁶⁺O₆)₂(H₂O)₂] [IMA2009-065; Kampf et al. (2010e)], thorneite [Pb₆(Te₂⁶⁺O₁₀)(CO₃)Cl₂(H₂O)] [IMA2009-023; Kampf et al. (2010a)], and timroseite [Pb₂Cu₅²⁺(Te⁶⁺O₆)₂(OH)₂] [IMA2009-064; Kampf et al. (2010e)]. Other potentially new species are still under investigation.

Telluroperite and most the other secondary minerals of the quartz veins are interpreted as having formed from the partial oxidation of primary sulfides (e.g., galena) and tellurides (e.g., hessite) during or following brecciation of the quartz veins. The Cl may be sourced in part from primary phases; however, it is most likely from salty brines interacting with primary tellurides. Porter and Halasyamani (2003) synthesized telluroperite by combining Pb₃O₂Cl₂ with TeO₂ at 575 °C for 1 day, which resulted in the production of an off-white powder. Similar temperatures have been used to synthesize other tellurium-bearing oxysalts (e.g., Mill' 2009a, 2009b; Mills et al. 2010); however, natural telluroperite almost certainly crystallized at ambient temperatures.

PHYSICAL AND OPTICAL PROPERTIES

Telluroperite commonly occurs as thin somewhat rounded square plates and flakes up to 0.25 mm on edge and 0.02 mm thick (Fig. 1). The form {001} is prominent and is bounded by several other forms; {100}, {010}, and {110} are most likely based upon the visual examination of SEM images.

Telluroperite is bluish-green and transparent, with a pale bluish-green streak and adamantine luster. The mineral is non-fluorescent. The Mohs hardness is estimated at between 2 and 3. The tenacity is brittle and the fracture is curved. Cleavage is perfect on {001}. The density could not be measured because it is greater than those of available high-density liquids and there is insufficient material for physical measurement. The calculated density is 7.323 g/cm³ for the empirical formula. In dilute HCl acid, telluroperite immediately decomposes, turning white and opaque, and then dissolves slowly.

Telluroperite is biaxial (-); however, the indices of refraction are higher than the available high-density reference liquids. The measured $2V$ is very small ($\sim 10^\circ$), indicating only a small departure from tetragonal symmetry (see crystal structure discussion below). The Gladstone-Dale relationship (Mandarino 1981) predicts $n_{av} = 2.219$. The observed retardation (using a Berek compensator) and thickness in the X optical direction (acute bisectrix), provides the β - γ birefringence of about 0.005. Based on the foregoing, the calculated indices of refraction are $\alpha = 2.091$, $\beta = 2.237$, and $\gamma = 2.242$. The optical orientation of telluroperite is $X = c$; however, because of the near equivalence of the a and b cell dimensions, it was not possible to unambiguously assign the Y and Z optical directions. Telluroperite exhibits moderate bluish-green pleochroism with absorption: $X < Y = Z$. No dispersion was observed.

CHEMICAL COMPOSITION

Four chemical analyses were carried out using a JEOL8200 electron microprobe (WDS mode, 15 kV, 10 nA, focused beam) at the Division of Geological and Planetary Sciences, California Institute of Technology. No other elements other than Te, Pb, and Cl were detected in EDS analyses and in the microprobe S, Cr, Cu, Zn, and U were below detection limits. Crystal-structure and bond-valence considerations (see below) indicate the absence of H₂O and OH in the structure. The averages (and ranges) of the analyses are PbO 72.70 (72.13–73.17), TeO₂ 19.26 (18.88–19.72), Cl 9.44 (9.38–9.48), O=Cl –2.13, total 99.27 wt%. Standards used were PbS, ZnO, Te metal, and sodalite (for Cl).

The empirical formula (based on O+Cl = 6) is Pb_{2.79}Te_{1.03}O_{3.72}Cl_{2.28}. The simplified formula is Pb₃Te⁴⁺O₄Cl₂, which requires PbO 75.74, TeO₂ 18.05, Cl 8.02, O=Cl –1.81, total 100.00 wt%.

X-RAY CRYSTALLOGRAPHY AND STRUCTURE DETERMINATION

Both powder and single-crystal X-ray diffraction data were obtained on a Rigaku R-Axis Spider curved imaging plate microdiffractometer utilizing monochromatized MoK α radiation. The powder data presented in Table 1 show good agreement with the pattern calculated from the structure determination.

The Rigaku CrystalClear software package was used for processing the structure data, including the application of an

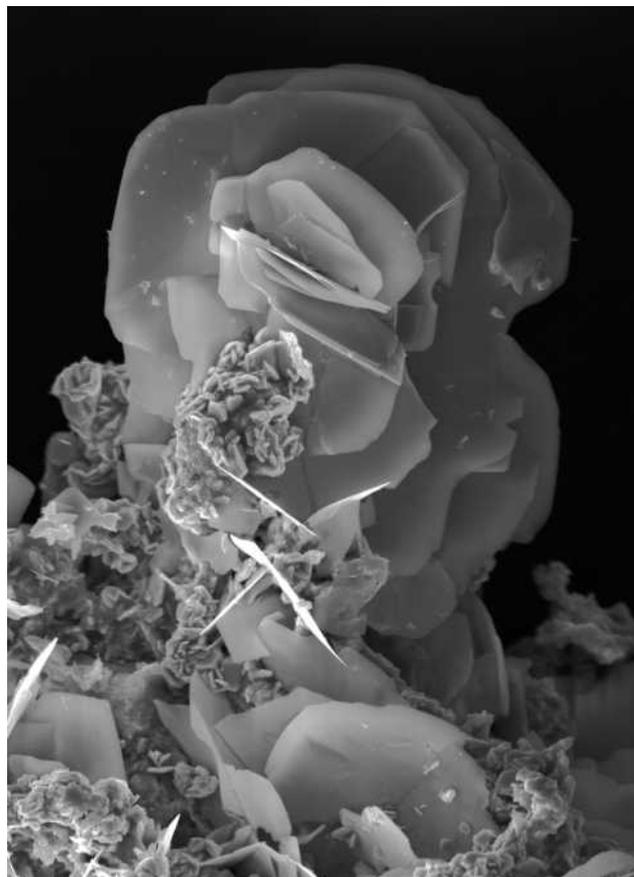


FIGURE 1. SEM image of telluroperite (FOV 165 μ m across).

empirical absorption correction. The structure was solved by direct methods using SHELXS-97 and refined, with neutral atom scattering factors, using SHELXL-97 (Sheldrick 2008). Large electron density residuals suggested some disorder in the structure. The largest residual is $8.356 e/\text{\AA}^3$ at (0, 1/4, 0.029), 0.84 Å from the Te/Pb site. Efforts to model this residual as a partially occupied Te site were unsuccessful. The refinement problems may, in part, be the result of an imperfect empirical absorption correction, another effect of which might be the very anisotropic nature of the Cl ellipsoid [$U_{33} = 0.000(4)$].

After the structure was solved we noticed the relationship to the so-called Sillén X_1 -type lead tellurium oxyhalides, of formula $\text{Pb}_3\text{Te}^{4+}\text{O}_4\text{X}_2$ (Porter and Halasyamani 2003; Charkin et al. 2006), as well as to the minerals perite (Gillberg 1960) and nadorite (Giuseppetti and Tadani 1973). Porter and Halasyamani (2003) refined the structure of synthetic $\text{Pb}_3\text{Te}^{4+}\text{O}_4\text{Cl}_2$ in the orthorhombic space group $Bmmb$ using the Rietveld method; however, Charkin et al. (2006) reported the tetragonal space group $I4/m$ in their study. Notably Charkin et al. (2006) were only able to synthesize single crystals of $\text{Pb}_3\text{Te}^{4+}\text{O}_4\text{Br}_2$ suitable for structure work. Their synthesized crystals of $\text{Pb}_3\text{Te}^{4+}\text{O}_4\text{Cl}_2$ always exhibited twinning. We were unable to solve and refine the structure of telluroperite in $I4/m$; however, we note that natural telluroperite is pseudotetragonal, as noted by the optics and similar a and b cell dimensions. The atomic positions in our structure determination closely match those determined by Porter and Halasyamani (2003) for synthetic $\text{Pb}_3\text{Te}^{4+}\text{O}_4\text{Cl}_2$; the minor differences are probably ascribable to the differences between the Rietveld and single-crystal methods.

The details of the data collection and the final structure refinement are provided in Table 2. The final atomic coordinates and displacement parameters are in Table 3. Selected interatomic distances are listed in Table 4 and bond valences in Table 5. CIF and structure factors on deposit¹.

DESCRIPTION OF THE STRUCTURE

Telluroperite possesses the Sillén X_1 structure-type (Sillén and Melander 1941) in which fluorite-like $\text{Pb}(\text{Pb}, \text{Te}^{4+})\text{O}_4$ – $\text{Pb}(\text{Pb}, \text{Te}^{4+})$ layers alternate with Cl_2 layers (Fig. 2). The Pb and Te/Pb sites lay between sheets of O and Cl atoms, forming slightly different PbO_4Cl_4 and $(\text{Te}^{4+}, \text{Pb})\text{O}_4\text{Cl}_4$ coordination polyhedra.

To consider the relationship between the stoichiometries of telluroperite, perite and nadorite, the formulas of all three minerals must be expressed in terms of two formula units per unit

¹ Deposit item AM-10-048, CIF and structure factors. Deposit items are available two ways: For a paper copy contact the Business Office of the Mineralogical Society of America (see inside front cover of recent issue) for price information. For an electronic copy visit the MSA web site at <http://www.minsocam.org>, go to the *American Mineralogist* Contents, find the table of contents for the specific volume/issue wanted, and then click on the deposit link there.

TABLE 1. X-ray powder-diffraction data for telluroperite

l_{obs}	d_{obs}	d_{calc}	l_{calc}	hkl
12	6.243	6.238	10	002*
58	3.750	3.753	48	111*
3	3.318	3.331	2	103
12	3.118	3.119	13	004*
100	2.857	2.858	100	113*
43	2.781	2.783	23	020*
		2.782	23	200*
		2.079	5	006
31	2.075	2.076	13	024*
		2.076	13	204*
30	1.966	1.968	23	220*
3	1.877	1.876	2	222
15	1.741	1.743	6	131*
		1.743	6	311*
		1.666	7	026*
21	1.665	1.666	7	206*
		1.664	8	224*
		1.623	12	117
52	1.620	1.621	17	133*
		1.621	17	313*
4	1.430	1.429	5	226
9	1.390	1.391	3	040*
		1.391	3	400*
3	1.357	1.360	1	028
		1.360	1	208
4	1.304	1.305	2	331
3	1.270	1.271	2	044
		1.271	2	404
		1.252	4	137*
17	1.250	1.252	4	317*
		1.251	6	333*

Notes: l_{obs} based upon peak heights. l_{calc} calculated from the crystal structure using Powder Cell (Kraus and Nolze 1996). d_{calc} based on the cell from the crystal-structure determination. Refinement of the cell from the powder data (*) using UnitCell (Holland and Redfern 1997) provides: $a = 5.5713(6)$, $b = 5.5713(6)$, $c = 12.486(2)$ Å, $V = 387.54(6)$ Å³.

TABLE 2. Data collection and structure refinement details for telluroperite

Diffractionmeter	Rigaku R-Axis Spider
X-ray radiation/power	MoK α ($\lambda = 0.71075$ Å)/50 kV, 40 mA
Temperature	298(2) K
Structural formula	$\text{Pb}_3(\text{PbTe}^{4+})\text{O}_4\text{Cl}_2$
Space group	$Bmmb$
Unit-cell dimensions	$a = 5.5649(6)$ Å $b = 5.5565(6)$ Å $c = 12.4750(14)$ Å
Z	2
Volume	$386.37(7)$ Å ³
Density (for above formula)	7.599 g/cm ³
Absorption coefficient	69.543 mm ⁻¹
$F(000)$	728
Crystal size	$40 \times 40 \times 10$ µm
θ range	3.27 to 27.42°
Index ranges	$-7 \leq h \leq 7$, $-7 \leq k \leq 7$, $-16 \leq l \leq 16$
Reflections collected/unique	4515/258 [$R_{\text{int}} = 0.0657$]
Completeness to $\theta = 27.50^\circ$	95.6%
Refinement method	Full-matrix least-squares on F^2
Parameters refined	21
GoF	1.280
Final R indices [$F_o > 4\sigma F$]	$R_1 = 0.056$, $wR_2 = 0.121$
R indices (all data)	$R_1 = 0.057$, $wR_2 = 0.121$
Largest diff. peak/hole	$8.356/-6.048$ e/Å ³

Notes: $R_{\text{int}} = \sum |F_o - F_c(\text{mean})| / \sum |F_o|$. GoF = $S = \{ \sum [w(F_o^2 - F_c^2)^2] / (n - p) \}^{1/2}$. $R_1 = \sum |F_o - F_c| / \sum |F_o|$. $wR_2 = \{ \sum [w(F_o^2 - F_c^2)^2] / \sum [w(F_o^2)^2] \}^{1/2}$. $w = 1 / [\sigma^2(F_o^2) + (aP)^2 + bP]$ where a is 0, b is 160, and P is $[2F_o^2 + \text{Max}(F_o, 0)] / 3$.

TABLE 3. Atomic positions and displacement parameters for telluroperite

	x	y	z	U_{eq}	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Pb	0.0	0.25	0.3892(2)	0.021(1)	0.016(1)	0.018(1)	0.028(1)	0.0	0.0	0.0
Te/Pb*	0.0	0.25	0.0961(2)	0.025(1)	0.013(1)	0.010(1)	0.052(2)	0.0	0.0	0.0
Cl	0.0	0.25	0.7473(7)	0.013(2)	0.019(4)	0.018(4)	0.000(4)	0.0	0.0	0.0
O	0.763(4)	0.0	0.0	0.018(4)	0.016(10)	0.021(10)	0.018(11)	-0.001(9)	0.0	0.0

* Occupancy: 0.50(3)/0.50(3).

TABLE 4. Selected bond lengths (Å) in telluroperite

Pb-O (×4)	2.447(14)	Te/Pb-O (×4)	2.261(13)
Pb-Cl (×2)	3.262(9)	Te/Pb-Cl (×2)	3.362(9)
Pb-Cl (×2)	3.336(9)	Te/Pb-Cl (×2)	3.400(9)

TABLE 5. Bond-valence analysis for telluroperite

	O	Cl	Sum
Pb	0.37	0.14 ×2↓→	1.98
	×2↓ ×4→	0.11 ×2↓→	
Te/Pb	0.52	0.09 ×2↓→	2.42
	×2↓ ×4→	0.08 ×2↓→	
Sum	1.78	0.84	

Notes: Values are expressed in valence units. Multiplicity is indicated by ×→↓; Pb²⁺-O bond strengths from Krivovichev and Brown (2001); Te⁴⁺-O bond strengths from Sidey (2009); Pb²⁺-Cl and Te⁴⁺-Cl bond strengths from Brese and O'Keeffe (1991); bond strength for Te/Pb site based on occupancy by 0.5 Te⁴⁺ and 0.5 Pb²⁺ (see discussion).

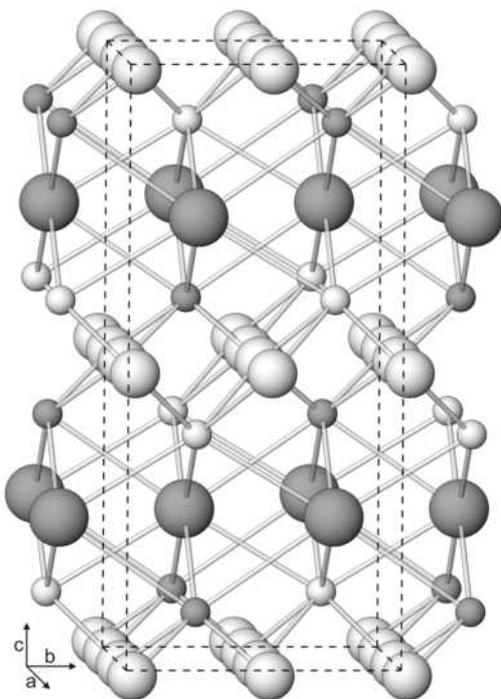


FIGURE 2. Structure of telluroperite. Small white spheres are Pb, small gray spheres are Te/Pb, large white spheres are O, and large gray spheres are Cl.

cell ($Z = 2$). The replacement of the two Bi³⁺ atoms in perite, Pb₂Bi₂O₄Cl₂, or the two Sb³⁺ atoms in nadorite, Pb₂Sb₂O₄Cl₂, with one Pb²⁺ and one Te⁴⁺ yields Pb₂PbTeO₄Cl₂, which has a mixed Te/Pb site, ideally with a 50:50 ratio of Te to Pb. In our refinement of the telluroperite structure, the occupancy of the Te/Pb site did indeed refine to 50(3):50(3).

As noted above, both orthorhombic and tetragonal polymorphs of the Sillén X₁ structure-type are known. Naturally occurring perite, PbBiO₂Cl, and nadorite, PbSbO₂Cl, both have orthorhombic cells, as does telluroperite; however, the synthetic equivalents of perite (Ketterer and Krämer 1985) and telluroperite (Charkin et al. 2006) were reported to be tetragonal. Furthermore, Deschanvres et al. (1970) showed that by increasing the Sb:Bi ratio in synthetic nadorite, the structure transitioned from orthorhombic to tetragonal. It is possible that examining many various natural samples of perite, nadorite, and telluroperite will show that both orthorhombic and tetragonal polymorphs exist in nature; however, investigations to date suggest that orthorhombic variants are preferred under ambient conditions.

The cations Pb²⁺ and Te⁴⁺ both have lone-electron pairs and typically are positioned off-center in their coordination polyhedra (e.g., Moore 1988; Cooper and Hawthorne 1996; Back et al. 1999). In telluroperite, the PbO₄Cl₄ and (Te,Pb)O₄Cl₄ coordination polyhedra are modified square antiprisms with short bonds to four O atoms and much longer bonds to four Cl atoms (Table 4; Fig. 3). The lone-pair electrons of Pb²⁺ and Te⁴⁺ clearly point toward the center of the square formed by the four Cl atoms of each polyhedron.

While the unusual joint occupancy of the Te/Pb site by ½ Te⁴⁺ and ½ Pb²⁺ provides an effective charge of +3, the bond-valence sum (BVS) (Table 5) for the site is only 2.42. The very low BVS can be attributed to the placement of the Te⁴⁺ on the same site as Pb²⁺, as this does not allow it to participate in the very short bond lengths typical of Te⁴⁺-O coordinations with stereo-active lone-pair electrons. For example, in walfordite [(Fe³⁺,Te⁶⁺)Te₃O₈] (Back et al. 1999), Te⁴⁺ forms bonds to O atoms of 1.880 (×2), 2.125 (×2), and 2.904 Å (×2) and in spiroffite [Mn₂Te₃O₈] (Cooper and Hawthorne 1996), there are two Te⁴⁺-O coordinations; one with Te⁴⁺-O bonds of 1.857 (×2), 2.104 (×2), and 3.83 Å (×2) and the other with Te⁴⁺-O bonds of 1.868, 1.908, 1.943, 2.390, 2.862, and 3.134 Å. Clearly, Pb²⁺-O bond lengths shorter than about 2.2 Å are highly unlikely. This argues for the placement

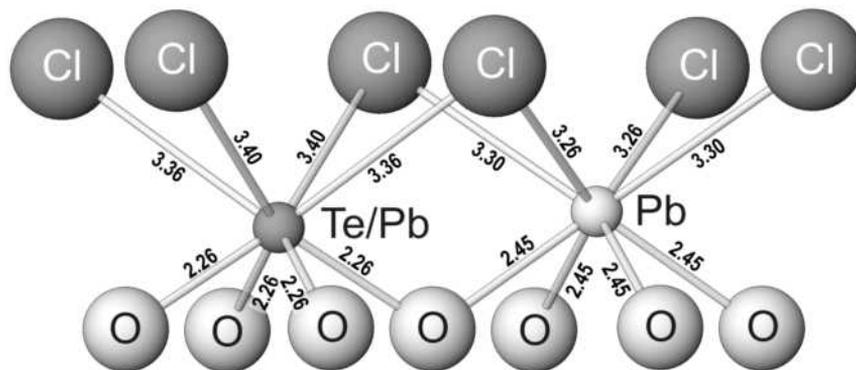


FIGURE 3. Coordinations of Pb and Te/Pb atoms in telluroperite. The lopsided distributions of bond lengths are attributable to the localization of the lone-pair electrons. Bond lengths are given in angstroms.

of the Te^{4+} closer to the O atoms and the large electron-density residuals, noted above, deserve further consideration in this respect. The largest residual, 0.84 Å from the Te/Pb site, is 1.95 Å from four O atoms and is 3.89 Å ($\times 2$) and 3.94 Å ($\times 2$) from four Cl atoms. Placing Te at this site yields a BVS of 4.16 v.u. In spite of our failure to refine this residual as a partially occupied Te site, if we assume that Te is split between this site and the Te/Pb site, bond-valence summations can be reconciled.

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