Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(β-Alanine)dibromolead(II)

Kathleen Reynolds,[a] Roger D. Willett[a]* and Brendan Twamley[b]

[a]Department of Chemistry, Washington State University, Pullman, WA 99164, USA, and [b]University Research Office, University of Idaho, Moscow, ID 83844, USA

Correspondence e-mail: rdw@mail.wsu.edu

Key indicators

Single-crystal X-ray study
T = 82 K
Mean σ(C–C) = 0.008 Å
R factor = 0.018
wR factor = 0.042
Data-to-parameter ratio = 16.9

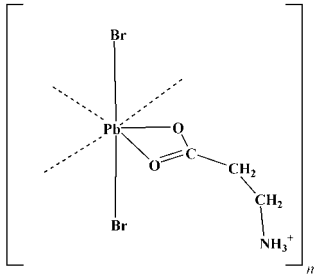
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 6 May 2003
Accepted 27 May 2003
Online 10 June 2003

The structure of the title compound, $[PbBr_2(C_3H_7NO_2)]$, contains Pb^{2+} ions, Br^- ions and β-alanine molecules in their zwitterion form. Each lead(II) ion has a seven-coordinate geometry, with four sites occupied by Br^- ions, two by a bidentate carboxylate group and the last by a single O atom. The singly-bridging Br^- ions link the Pb^{II} ions into layers that are further aggregated into a three dimensional array by the formation of Pb—O bonds and hydrogen bonds involving the $-NH_3^+$ groups.

Comment

The sevenfold local coordination for the lead(II) ion is shown in Fig. 1, and can conveniently be viewed as a severely distorted octahedron in which one site of the octahedron is occupied by the bidentate carboxylate group. The four Pb—Br bond lengths range from 2.9918 (6) to 3.1731 (5) Å. The carboxylate group in the zwitterion form of the β-alanine molecule coordinates in one octahedral site in a bidentate fashion, while the sixth site is occupied by an O atom from a β-alanine molecule of an adjacent octahedron. The Pb—O distances are 2.533 (3) and 2.600 (4) Å for the O atoms in the bidentate group, and 2.754 (4) Å for the bridging O atom. As anticipated, the angular distortions imposed by the presence of the bidentate group are significant. As is also seen in Fig. 1, the backbone of the β-alanine molecule assumes a *gauche* conformation. As discussed below, this allows the formation of an intramolecular hydrogen bond as well as several other interactions.



(I)

Corner-sharing, through the bromide ions on adjacent octahedra, leads to the formation of a two-dimensional structure, as shown in Fig. 2. This layer structure may be viewed as a (110) section of the parent cubic AMX_3 structure. This is a single metal halide layer of the type in the multiple layer $(NH_2ClNH_2)_2(CH_3NH_3)_{n-1}Sn_nI_{3n+1}$ (110) sections reported by Mitzi *et al.* (1995). This is in contrast to the typical (001) section formed by $(RNH_3)_2MX_4$ layer perovskite compounds, such as in (β-alaninium)$_2CuX_4$ salts (X = Cl^- and Br^-) (Willett *et al.*, 1981, 1983).

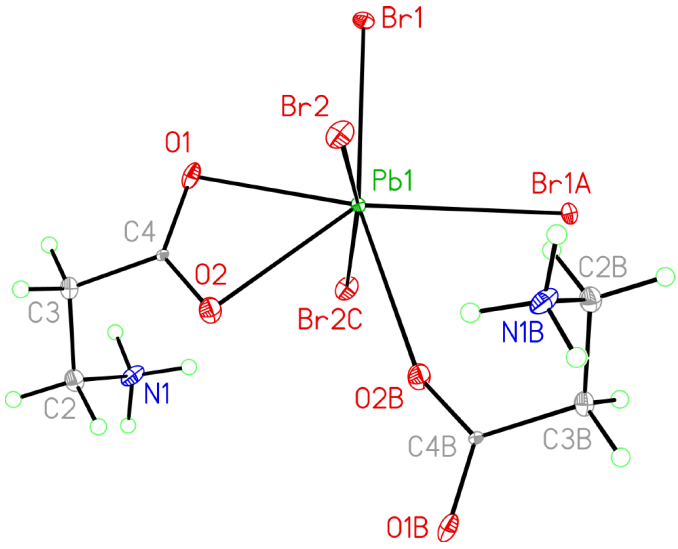


Figure 1
Illustration of the lead(II) ion coordination. Displacement ellipsoids are drawn at the 70% probability level. Suffix letters denote symmetry-generated atoms: *A* $x, \frac{1}{2}-y, z-\frac{1}{2}$; *B* $-x, 1-y, -z$; *C* $1+x, y, z$.

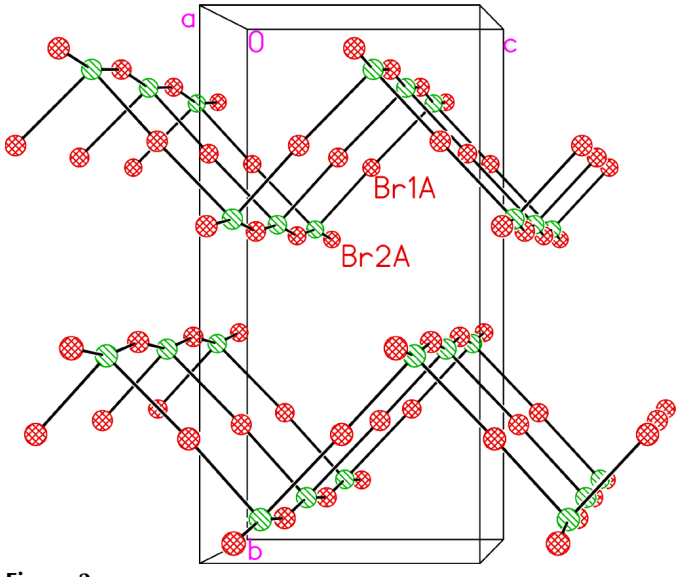


Figure 2
Illustration of the (110) $PbBr_2$ layer.

These metal halide layers are linked together *via* double Pb—O—Pb bridges (dashed lines, Fig. 3), as well as N—H···O and N—H···Br hydrogen bonds (dashed lines, inset, Fig. 3). These Pb—O bonds are 0.15 Å longer than those in the bidentate linkage. The bridging Pb—O—Pb angle is 109.0 (1)°. The $-NH_3^+$ group forms one asymmetric bifurcated hydrogen bond (to two different Br2 atoms, see Table 1) and two normal hydrogen bonds (to Br1 and O1). The intramolecular N—H···Br hydrogen bond is nearly 0.2 Å longer than the one between layers, presumably because of steric constraints on the conformation of the *β*-alanine molecule. In addition, there is an electrostatic interaction of the $-NH_3^+$ group with an O2 atom from the adjacent layer, in which the C—N···O angle is close to linear.

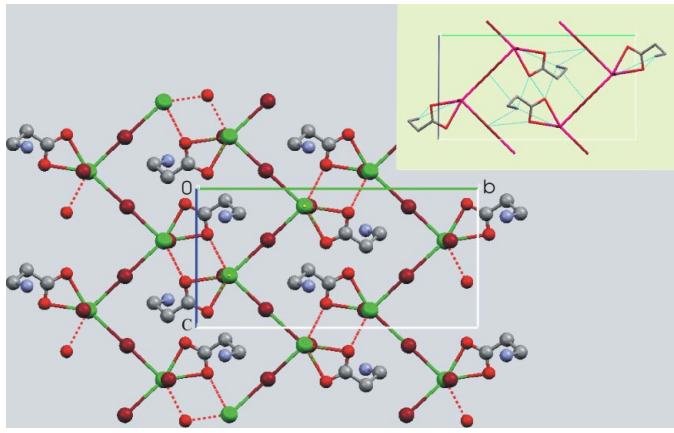


Figure 3
Illustration of the interconnection of the $PbBr_2$ layers, viewed down the **a** direction. The *b* axis is horizontal. The bridging Pb—O bonds are shown as dashed lines. The inset shows the hydrogen-bonding contacts.

Experimental

Crystals of the title compound were prepared by slow evaporation of a solution obtained by dissolving 0.9282 g $PbBr_2$ (0.002 mmol) and 0.4550 g *β*-alanine (0.005 mmol) in 80 ml deionized water that had been acidified with 5 drops of concentrated HBr.

Crystal data

$[PbBr_2(C_3H_7NO_2)]$	D_x = 3.737 Mg m^{-3}
M_r = 456.11	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 2506 reflections
a = 6.0073 (4) Å	θ = 2.5–28.3°
b = 16.5286 (10) Å	μ = 30.60 mm^{-1}
c = 8.3057 (5) Å	T = 82 (2) K
β = 100.56 (1)°	Rhomboid, colorless
V = 810.71 (9) Å^3	0.11 × 0.08 × 0.06 mm
Z = 4	

Data collection

Bruker–Siemens SMART APEX diffractometer	1423 independent reflections
ω scans	1366 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)	R_{int} = 0.034
T_{min} = 0.066, T_{max} = 0.159	θ_{max} = 25.0°
7447 measured reflections	$h = -7 \rightarrow 7$
	$k = -19 \rightarrow 19$
	$l = -9 \rightarrow 9$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + 2.9938P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.018	$(\Delta/\sigma)_{max}$ = 0.001
$wR(F^2)$ = 0.042	$\Delta\rho_{max}$ = 1.11 e Å^{-3}
S = 1.20	$\Delta\rho_{min}$ = −0.68 e Å^{-3}
1423 reflections	Extinction correction: *SHELXL*97
84 parameters	Extinction coefficient: 0.00191 (12)
H-atom parameters constrained	

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O1^{i}	0.91	1.93	2.843 (6)	177
N1—H1B···Br1^{ii}	0.91	2.55	3.422 (5)	162
N1—H1C···Br2^{iii}	0.91	2.75	3.604 (5)	156
N1—H1C···Br2^{iv}	0.91	2.98	3.490 (4)	118

Symmetry codes: (i) $1-x, 1-y, 1-z$; (ii) $1-x, \frac{1}{2}+y, \frac{1}{2}-z$; (iii) $1+x, y, z$; (iv) $-x, 1-y, -z$.

H atoms were positioned geometrically and refined using a riding model, with U_{iso} for the methylene C—H groups constrained to be $1.2U_{eq}$ of the carrier atom, while those of the N—H H atoms were set at $1.5U_{eq}$. There is a large residual of 1.11 e Å^{-3} *ca* 0.92 Å from Pb1.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

References

Bruker (2000). *SHELXTL*. Version 6.10. Bruker AXS Inc., Madison, Wisconsin, USA.

Bruker (2001). *SADABS* (Version 2.03) and *SAINT-Plus* (Version 6.22). Bruker AXS Inc., Madison, Wisconsin, USA.

Bruker (2002). *SMART*. Version 5.626. Bruker AXS Inc., Madison, Wisconsin, USA.

Mitzi, D. B., Wang, S., Feild, C. A., Chess, C. A. & Guloy, A. M. (1995). *Science*, **267**, 1473–1476.

Willett, R. D., Jardine, F. H., Rouse, I., Wong, R. J., Landee, C. P. & Numata, M. (1981). *Phys. Rev.* **24**, 5372–5381.

Willett, R. D., Wong, R. & Numata, M. (1983). *Inorg. Chem.* **22**, 3189.