Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

Poly[bis(benzotriazolato)di-μ-oxo-dilead(II)]

V. C. Sharma,[a] U. Lal,[a] Tej Singh,[a] Deepak Chopra[b] and T. N. Guru Row[b]*

[a]National Research Laboratory for Conservation of Cultural Property, E/3 Aliganj, Lucknow 226 024, Uttar Pradesh, India, and [b]Solid State and Structural Chemistry Unit, Indian Institute of Science, Bangalore 560 012, Karnataka, India

Correspondence e-mail:
ssctng@sscu.iisc.ernet.in

Key indicators

Single-crystal X-ray study
T = 290 K
Mean σ(C–C) = 0.022 Å
R factor = 0.056
wR factor = 0.188
Data-to-parameter ratio = 13.7

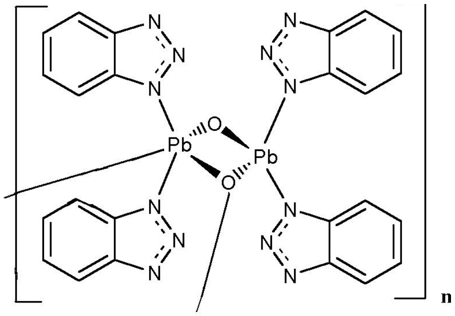
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 21 March 2005
Accepted 5 May 2005
Online 14 May 2005

The title compound, $[Pb_2O_2(C_6H_4N_3)_2]_n$, exhibits anti-corrosion properties. The coordination around each Pb atom is different, with coordination numbers of four and five. The compound forms a polymeric chain with pairs of oxo bridges between metal centers.

Comment

Benzotriazole (BTAH), possessing three vicinal N atoms, is used as an antifouling and antiwear reagent (Sha *et al.*, 1996). Several studies on the interaction of BTA with copper and the characterization of copper compounds of BTA have been reported (Brostoff, 1997). Transition metal complexes having benzotriazole as a ligand have also been synthesized and characterized by single-crystal diffraction studies (Sotofte & Nielsen, 1984*a*,*b*; Li *et al.*, 2002). Sharma *et al.* (2003) found that a neutral solution of BTA is an excellent corrosion inhibitor for lead and have used it to prevent preferential corrosion of lead in ancient leaded bronze sculptures. The chemistry involved in the interactions between lead and the BTA ligand and the nature of lead–BTA complexes are not well characterized. In this paper, we present the crystal structure of the title polymeric complex, (I).



(I)

This structure is unique, with no hydration associated with the compound, such hydration being a common feature in

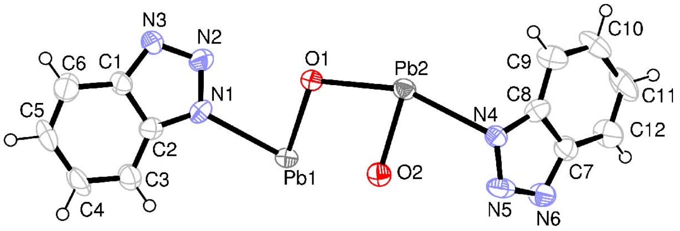


Figure 1
The asymmetric unit of (I). Displacement ellipsoids are drawn at the 50% probability level.

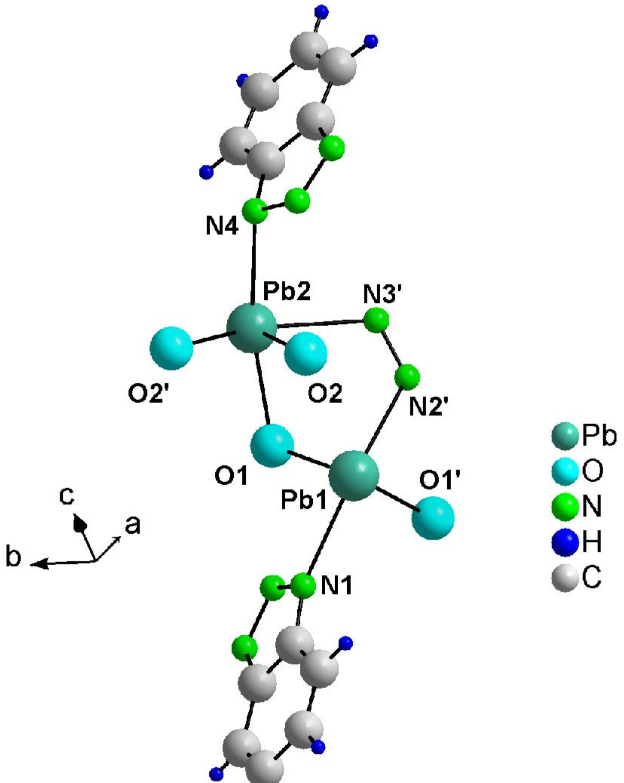


Figure 2
The coordination environments around the two Pb atoms. The primes indicate symmetry-equivalent atoms; for details, see Table 1.

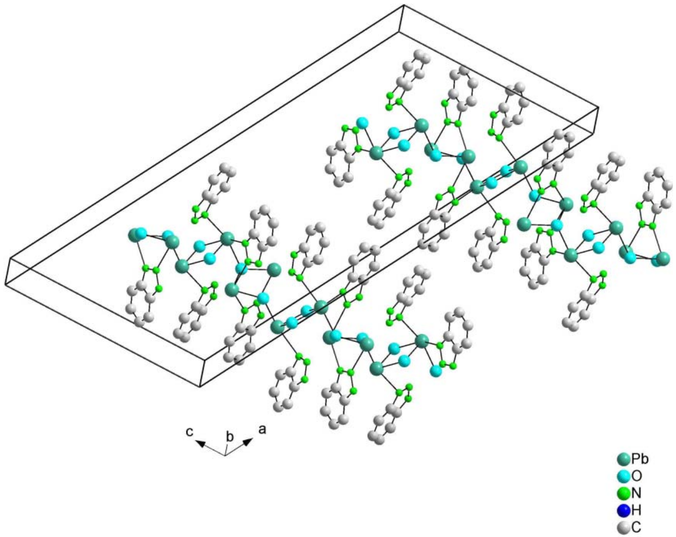


Figure 3
Packing diagram of (I), viewed approximately down the *b* axis.

most of the known metal-substituted corrosion inhibitors containing BTA. Fig. 1 shows the asymmetric unit of the title compound. The one-sided coordination of the Pb atoms is due to the presence of the 6*s* lone pair of electrons. The Pb atoms have different primary coordination environments: Pb1 is four-coordinate, while Pb2 is five-coordinate (Table 1 and Fig. 2). However, in addition, a secondary Pb1—O2 contact of 2.771 (8) Å is present, making Pb1 also five-coordinate if this is included

Van der Waals interactions stabilize the crystal structure with its polymeric assembly of aromatic surfaces, which are responsible for generating a hydrophobic environment. This might provide a protective coating on the surface of a substrate material, thus preventing corrosion (Fig. 3).

Experimental

Crystals of suitable size and quality for single-crystal diffraction were grown in neutral BTA solution (1.0%), which was prepared by adding 0.2 g of solid calcium carbonate to 200 ml of BTA solution with occasional stirring. The solution was filtered after 6 h when it attained a pH of 7.0. Two lead plates were immersed in this neutral solution and crystals were obtained after a period of 16 d (m.p. 629 K). ^{1}H NMR (DMSO, 300 MHz): δ 7.32 (4H), 7.87 (4H).

Crystal data

$[Pb_2O_2(C_6H_4N_3)_2]$
$M_r = 682.64$
Monoclinic, $C2/c$
$a = 35.035$ (6) Å
$b = 5.8335$ (10) Å
$c = 16.049$ (3) Å
$\beta = 116.837$ (2)°
$V = 2926.8$ (9) Å^3
$Z = 8$
$D_x = 3.114$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 625 reflections
$\theta = 1.4$–25.2°
$\mu = 23.11$ mm^{-1}
$T = 290$ (2) K
Thick plate, colorless
0.18 × 0.12 × 0.06 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.033$, $T_{\max} = 0.250$
12665 measured reflections
2724 independent reflections
2523 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.041$
$\theta_{\max} = 25.5°$
$h = -42 \rightarrow 42$
$k = -7 \rightarrow 7$
$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.056$
$wR(F^2) = 0.188$
$S = 1.13$
2724 reflections
199 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1502P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 2.96$ e Å^{-3}
$\Delta\rho_{\min} = -4.15$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Pb1—O1	2.338 (7)	Pb2—O2^{ii}	2.442 (8)
Pb1—O1^{i}	2.492 (8)	Pb2—O1	2.463 (8)
Pb1—N1	2.591 (9)	Pb2—N3^{i}	2.625 (11)
Pb1—N2^{i}	2.730 (12)	Pb2—N4	2.646 (10)
Pb2—O2	2.289 (9)		
O1—Pb1—O1^{i}	97.07 (18)	O2^{ii}—Pb2—O1	91.7 (3)
O1—Pb1—N1	75.9 (3)	O2—Pb2—N3^{i}	74.7 (3)
O1^{i}—Pb1—N1	76.4 (3)	O2^{ii}—Pb2—N3^{i}	146.0 (3)
O1—Pb1—N2^{i}	75.0 (3)	O1—Pb2—N3^{i}	82.4 (3)
O1^{i}—Pb1—N2^{i}	68.3 (3)	O2—Pb2—N4	80.6 (3)
N1—Pb1—N2^{i}	130.3 (4)	O2^{ii}—Pb2—N4	82.2 (3)
O2—Pb2—O2^{ii}	71.3 (3)	O1—Pb2—N4	158.5 (3)
O2—Pb2—O1	77.9 (3)	N3^{i}—Pb2—N4	91.1 (4)

Symmetry codes: (i) $\frac{1}{2} - x, y - \frac{1}{2}, \frac{1}{2} - z$; (ii) $\frac{1}{2} - x, \frac{3}{2} - y, 1 - z$.

All the H atoms were placed in idealized positions and constrained to ride on their parent atoms, with C—H = 0.93 Å and $U_{\rm iso}$(H) = $1.2U_{\rm eq}$(C). The largest residual electron-density peak is 0.88 Å from atom Pb1 and the deepest hole is 1.23 Å from the same atom.

Acta Cryst. (2005). E**61**, m1108–m1110 V. C. Sharma *et al.* • $[PbO_2(C_6H_4N_3)_2]$ **m1109**

Data collection: *SMART* (Bruker, 2000); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2000); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *CAMERON* (Watkin *et al.*, 1993); software used to prepare material for publication: *PLATON* (Spek, 2003).

The authors thank the Department of Science and Technology, India, for data collection on the CCD facility set up under the IRHPA–DST program. The authors are thankful to the Sophisticated Instrumentation Center for Applied Research and Testing, Vallabh Vidyanagar, Anand, Gujarat, and the Regional Sophisticated Instrumentation Center, Central Drug Research Institute, Lucknow, for the use of some of their instrumental facilities. D. Chopra thanks Saikat Sen for advice and CSIR, India, for a Junior Research Fellowship.

References

Altomare, A., Cascarano, G., Giacovazzo, C. & Guagliardi, A. (1993). *J. Appl. Cryst.* **26**, 343–350.

Brostoff, L. B. (1997). *Metal 95*, edited by I. D. Macleod, S. L. Pennec and L. Robbiola. Auxois, France: James and James.

Bruker (2000). *SMART* (Version 5.628) and *SAINT* (Version 6.02). Bruker AXS Inc., Madison, Wisconsin, USA.

Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.

Li, L., Wang, X., Shen, G., Wang, R. & Shen, D. (2002). *Inorg. Chem. Commun.* **5**, 453–457.

Sha, G., Wang, W. & Ren, T. (1996). *Mocha Xuebao*, **16**, 344–350.

Sharma, V. C., Lal, U. S. & Singh, T. (2003). *Studies in Conservation*, **48**, 203–209.

Sheldrick, G. M. (1996). *SADABS.* University of Göttingen, Germany.

Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.

Sotofte, I. & Nielsen, K. (1984*a*). *Acta Chem. Scand. Ser. A*, **38**, 253–256.

Sotofte, I. & Nielsen, K. (1984*b*). *Acta Chem. Scand. Ser. A*, **38**, 257–260.

Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

Watkin, D. M., Pearce, L. & Prout, C. K. (1993). *CAMERON.* Chemical Crystallography Laboratory, University of Oxford, England.

m1110 V. C. Sharma *et al.* • $[PbO_2(C_6H_4N_3)_2]$ *Acta Cryst.* (2005). E**61**, m1108–m1110