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catena-Poly[[aqua(*cis*-*syn*-*cis*-dicyclohexyl-18-crown-6)lead(II)]-μ-thiocyanato-κ^2N:S-[dithiocyanatopalladium(II)]-μ-thiocyanato-κ^2S:N]

Yun-Ming Sun,[a,b] Feng-Ying Dong,[a,b] Jian-Min Dou,[a]* Xi-Ke Gao,[a] Da-Cheng Li[a] and Da-Qi Wang[a]

[a]Department of Chemistry, Liaocheng University, Liaocheng 252059,People's Republic of China, and [b]Department of Plant Protection, Laiyang Agricultural College, Qingdao 266109, People's Republic of China

Correspondence e-mail: jmdou@lctu.edu.cn

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Key indicators

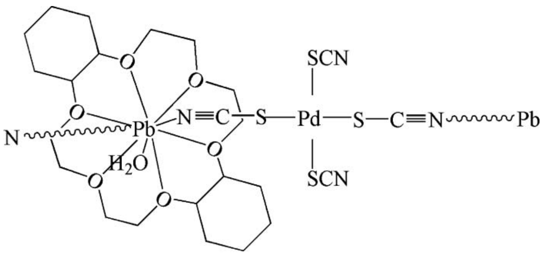
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.009 Å
R factor = 0.033
wR factor = 0.046
Data-to-parameter ratio = 15.5

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

The title polymeric complex, $[PbPd(NCS)_4(C_{20}H_{36}O_6)(H_2O)]_n$ or $\{[Pb(DC18C6\text{-}A)(H_2O)][Pd(SCN)_4]\}_n$ (DC18C6-A = *cis*-*syn*-*cis*-dicyclohexyl-18-crown-6), has been synthesized by the reaction of dicyclohexyl-18-crown-6 with $PdCl_2$ and $Pb(SCN)_2$. The SCN groups bridge $[Pb(DC18C6\text{-}A)(H_2O)]^{2+}$ complex cations and $[Pd(SCN)_4]^{2-}$ complex anions to form infinite polymeric chains.

Comment

In recent years, the synthesis and characterization of supramolecular architectures designed from metal-based coordination has expanded drastically (Reineke *et al.*, 1999; Ferey, 2001). This may be attributed largely to the potential applications of coordination polymers in many areas (Chen & Suslick, 1993; Lu, 2003). Our interest is focused on synthesizing some organic–inorganic hybrid crystalline solids containing crown ether cation building blocks (Dou *et al.*, 2004; Dong *et al.*, 2005). The title complex, (I), is reported here.



(I)

The molecular structure of (I) is shown in Fig. 1. The asymmetric unit consists of one $[Pb(DC18C6\text{-}A)(H_2O)]^{2+}$ complex cation (DC18C6-A = *cis*-*syn*-*cis*-dicyclohexyl-18-crown-6) and one $[Pd(SCN)_4]^{2-}$ complex anion. In the

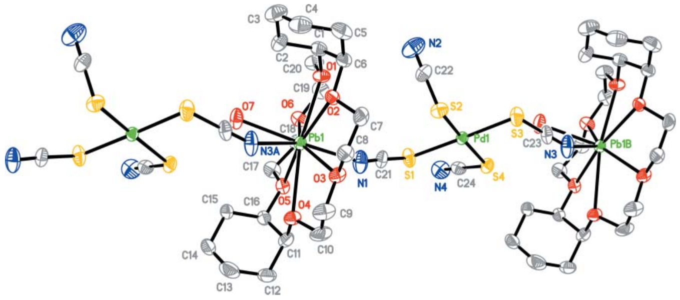


Figure 1
The molecular structure of (I), shown with 30% displacement ellipsoids, H atoms have been omitted for clarity [symmetry codes: (A) *x*, *y* + 1, *z*; (B) *x*, *y* − 1, *z*].

$[Pd(SCN)_4]^{2-}$ complex anion, the Pd atom is coordinated by four S atoms from SCN groups and exhibits distorted square-planar coordination geometry. The bond lengths of Pd—S, S—C and C≡N are in the ranges 2.3082 (16)–2.3347 (17), 1.648 (6)–1.678 (7) and 1.128 (6)–1.160 (7) Å, respectively, which are consistent with the corresponding values found in the complex $[Na(B18C6)]_2[Pd(SCN)_4](H_2O)$ (Li *et al.*, 2003). In the $[Pb(DC18C6\text{-}A)(H_2O)]^{2+}$ complex cation, the Pb atom lies in the crown ether and is coordinated by nine atoms, six O atoms of a crown ether, one O atom from a water molecule and two N atoms from two $[Pd(SCN)_4]^{2-}$ complex anions. The distances of Pb—O(crown ether) are in the range 2.666 (4)–2.839 (4) Å. The Pb1—N1 and Pb1—N3*A* bond lengths [symmetry code: (*A*: *x*, *y* + 1, *z*] are 2.542 (5) and 2.927 (5) Å, respectively. Similar values can be found in the literature (Bailey *et al.*, 1983; Engelhardt *et al.*, 1989; Metz & Weiss, 1974). As shown in Fig. 1, the $[Pb(DC18C6\text{-}A)(H_2O)]^{2+}$ complex cations and $[Pd(SCN)_4]^{2-}$ complex anions are assembled into an infinite chain *via* SCN groups.

The coordinated water molecule forms two hydrogen bonds, *viz.* an intrachain O—H···S and an interchain O—H···N hydrogen bond (Table 1).

Experimental

All chemicals were commercially available and were used without further purification. The microanalytical data were obtained from a Perkin–Elmer 2400 II analyser. The title complex was prepared by adding an aqueous mixture (10 ml) of $PdCl_2$ (0.25 mmol) and $Pb(SCN)_2$ (5 mmol) to a solution of dicyclohexyl-18-crown-6 (1 mmol) in 1,2-dichloroethane (10 ml). The reaction mixture was stirred for 3 h at room temperature. The organic phase was separated from the reaction solution. Crystals of (I) were obtained from a 4:1 diethyl ether–1,2-dichloroethane solution over a period of 20 d.

Crystal data

$[PbPd(NCS)_4(C_{20}H_{36}O_6)(H_2O)]$
M_r = 936.41
Monoclinic, $P2_1/n$
a = 14.177 (5) Å
b = 12.967 (4) Å
c = 18.885 (6) Å
β = 106.237 (5)°
V = 3333.4 (19) Å^3
Z = 4
D_x = 1.866 Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 4334 reflections
θ = 1.9–25.0°
μ = 5.88 mm^{-1}
T = 298 (2) K
Block, red
0.25 × 0.23 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.241, T_{max} = 0.259
17097 measured reflections
5836 independent reflections
3801 reflections with $I > 2\sigma(I)$
R_{int} = 0.052
θ_{max} = 25.0°
$h = -15 \rightarrow 16$
$k = -15 \rightarrow 15$
$l = -20 \rightarrow 22$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.046
S = 0.95
5836 reflections
376 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0003P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 1.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.95 e Å^{-3}

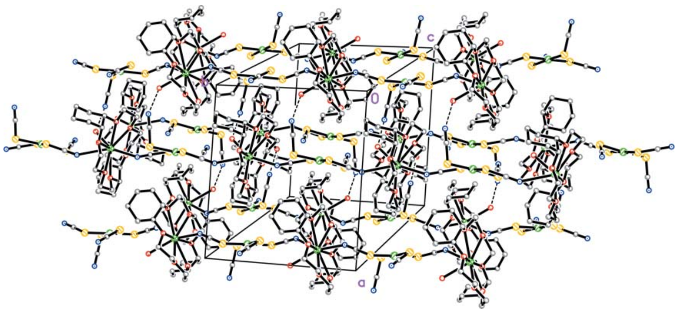


Figure 2
The unit-cell packing of (I), with dashed lines indicating the hydrogen bonds.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O7—H21···N2^{i}	0.90 (3)	1.93 (3)	2.805 (7)	165 (6)
O7—H22···S4^{ii}	0.87 (2)	2.54 (3)	3.349 (4)	154 (5)

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

Aqua H atoms were located in a difference map, the O—H distances were restrained to 0.88 (2) Å and the U_{iso}(H) values were fixed at 0.08 Å^2. Other H atoms were placed in idealized positions and constrained to ride on their parent atoms, with C—H = 0.97 Å and $U_{iso}(H) = 1.2U_{eq}(C)$. The highest electron-density difference peak was located 1.03 Å from atom Pd1.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997a); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997a); molecular graphics: *SHELXTL* (Sheldrick, 1997b); software used to prepare material for publication: *SHELXTL*.

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