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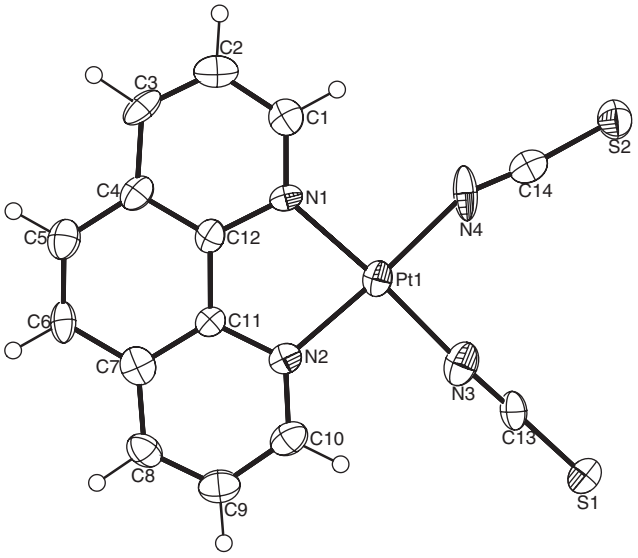
Crystal structure of (1,10-phenanthroline-κ²*N*,*N*′) bis(thiocyanato-κ*N*)platinum(II), $C_{14}H_8N_4PtS_2$



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Abstract

$C_{14}H_8N_4PtS_2$, monoclinic, $P2_1/n$ (no. 14), $a = 7.3318(7)$ Å, $b = 15.1300(15)$ Å, $c = 12.5924(13)$ Å, $\beta = 90.379(2)°$, $V = 1396.8(2)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0349$, $wR_{ref}(F^2) = 0.0668$, $T = 183(2)$ K.

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The crystal structure is shown in the figure. Tables 1–3 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

To a solution of K_2PtCl_4 (0.199 g, 0.48 mmol) and KSCN (0.938 g, 9.65 mmol) in H_2O (10 mL) was added, 1,10-phenanthroline (phen; 0.0958 g, 0.532 mmol) and stirred for 7 h at room temperature. The formed precipitate was separated by filtration, washed with H_2O and EtOH, and dried at 50 °C, to give a bright yellow powder (0.1578 g). Crystals suitable for X-ray diffraction analysis were obtained by slow evaporation from a dimethyl sulfoxide (DMSO) solution at 90 °C.

Table 1: Data collection and handling.

Crystal:	Yellow, rod, size 0.06×0.08×0.13 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	103.40 cm^{-1}
Diffractometer, scan mode:	Bruker SMART 1000 CCD, φ and ω scans
$2\theta_{max}$:	52.03°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	8392, 2708
$N(param)_{refined}$:	190
Programs:	SHELX [8], WinGX [9], Platon [10]

Table 2: Atomic coordinates and isotropic displacement parameters (Å^2).

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1)	4*e*	0.6906	0.5980	0.3176	0.046
H(2)	4*e*	0.7473	0.5650	0.1423	0.057
H(3)	4*e*	0.8695	0.4312	0.0935	0.049
H(5)	4*e*	0.9994	0.2795	0.1567	0.052
H(6)	4*e*	1.0667	0.1841	0.2938	0.045
H(8)	4*e*	1.0663	0.1539	0.4945	0.047
H(9)	4*e*	1.0004	0.2043	0.6635	0.052
H(10)	4*e*	0.8689	0.3429	0.6867	0.046

Experimental details

Hydrogen atoms were positioned geometrically and allowed to ride on their parent atoms with d(C—H) = 0.95 Å and $U_{iso}(H) = 1.2U_{eq}(C)$. The highest peak (1.26 e Å^{-3}) and the deepest hole (−0.91 e Å^{-3}) in the difference Fourier map are located 0.86 Å and 0.74 Å from the atom Pt1, respectively.

Discussion

This contribution is part of my continuing interest in the structural chemistry of phenanthroline (phen) containing platinum complexes [1–4]. The color of title complex

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Table 3: Atomic coordinates and displacement parameters (Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Pt(1)	4*e*	0.74476(3)	0.49674(2)	0.53547(2)	0.0279(1)	0.0349(2)	0.0330(2)	−0.0073(1)	0.0013(1)	−0.0075(1)
S(1)	4*e*	0.6906(3)	0.5143(1)	0.9085(2)	0.058(1)	0.049(1)	0.033(1)	0.0039(9)	0.0061(9)	−0.0018(8)
S(2)	4*e*	0.5106(3)	0.7827(1)	0.5854(2)	0.046(1)	0.046(1)	0.046(1)	0.0036(8)	−0.0006(9)	−0.0083(9)
N(1)	4*e*	0.7757(6)	0.4849(3)	0.3790(4)	0.017(2)	0.030(3)	0.025(3)	−0.008(2)	−0.006(2)	0.001(2)
N(2)	4*e*	0.8532(7)	0.3751(3)	0.5348(4)	0.023(3)	0.036(3)	0.028(3)	−0.009(2)	0.001(2)	0.000(2)
N(3)	4*e*	0.7216(8)	0.4995(4)	0.6911(5)	0.048(4)	0.051(4)	0.037(4)	−0.008(3)	0.009(3)	−0.008(3)
N(4)	4*e*	0.6417(8)	0.6189(4)	0.5285(5)	0.031(3)	0.045(4)	0.067(5)	−0.010(3)	0.005(3)	−0.033(3)
C(1)	4*e*	0.7409(9)	0.5419(5)	0.3005(6)	0.031(4)	0.039(4)	0.045(5)	−0.004(3)	−0.003(3)	0.000(3)
C(2)	4*e*	0.775(1)	0.5223(5)	0.1953(6)	0.042(4)	0.061(5)	0.038(5)	−0.006(4)	−0.009(3)	0.013(4)
C(3)	4*e*	0.8474(9)	0.4438(5)	0.1662(5)	0.033(4)	0.071(5)	0.017(4)	−0.012(4)	−0.007(3)	−0.001(3)
C(4)	4*e*	0.8893(9)	0.3813(5)	0.2451(5)	0.031(4)	0.048(4)	0.027(4)	−0.009(3)	−0.003(3)	−0.005(3)
C(5)	4*e*	0.9720(9)	0.2967(5)	0.2274(6)	0.033(4)	0.049(5)	0.046(5)	−0.002(3)	0.000(3)	−0.014(4)
C(6)	4*e*	1.0130(9)	0.2398(5)	0.3086(6)	0.032(4)	0.038(4)	0.043(5)	0.000(3)	−0.001(3)	−0.016(3)
C(7)	4*e*	0.9752(9)	0.2642(4)	0.4148(6)	0.027(3)	0.032(4)	0.048(5)	−0.008(3)	0.001(3)	−0.006(3)
C(8)	4*e*	1.0132(9)	0.2106(4)	0.5039(6)	0.033(4)	0.029(4)	0.055(5)	−0.006(3)	0.003(3)	0.001(3)
C(9)	4*e*	0.974(1)	0.2402(5)	0.6035(6)	0.044(4)	0.043(4)	0.043(5)	−0.001(3)	−0.005(4)	0.007(3)
C(10)	4*e*	0.8945(9)	0.3229(5)	0.6169(6)	0.038(4)	0.048(4)	0.029(4)	−0.007(3)	0.003(3)	0.003(3)
C(11)	4*e*	0.8935(8)	0.3461(4)	0.4346(5)	0.026(3)	0.033(4)	0.025(4)	−0.005(3)	0.002(3)	−0.003(3)
C(12)	4*e*	0.8507(8)	0.4045(4)	0.3499(5)	0.027(3)	0.031(4)	0.032(4)	−0.010(3)	−0.001(3)	−0.004(3)
C(13)	4*e*	0.7085(9)	0.5063(4)	0.7820(6)	0.031(3)	0.029(4)	0.045(5)	−0.007(3)	0.003(3)	−0.016(3)
C(14)	4*e*	0.5863(9)	0.6862(5)	0.5560(6)	0.028(4)	0.046(4)	0.037(4)	−0.009(3)	−0.006(3)	0.002(3)

$[Pt(NCS)_2(phen)]$ is yellow, whereas the related 2,2′-bipyridine (bipy) complex $[Pt(NCS)_2(bipy)]$ is red [5, 6].

In the complex $[Pt(NCS)_2(phen)]$, the central Pt(II) ion is four-coordinated in a slightly distorted square-planar environment defined by two N atoms derived from a chelating phen ligand and two mutually *cis*-positioned N atoms from two SCN^- anionic ligands. The main contribution to the distortion is the tight N1-Pt1-N2 chelate angle of 82.3(2)°, which results in non-linear *trans* axes (<N1-Pt1-N3 = 175.8(2)° and <N2-Pt1-N4 = 177.0(2)°). The Pt-N(phen) and Pt-N(NCS) bond lengths are nearly equivalent (d(Pt-N) = 1.968(6)–2.005(5) Å) and in the typical ranges [7]. The thiocyanato ligands are almost linear displaying N-C-S bond angles of 179.2(7)° and 175.7(7)°, and the N atoms are slightly bent coordinated to the Pt atom with the Pt-N-C(NCS) bond angles of 176.1(6)° and 159.9(7)°, characteristic of an N-bonded conformation. In the crystal, the nearly planar complexes are stacked in columns along [100]. When viewed down the *a* axis, the successive complexes are related by an inversion center and thus stack in the opposite direction with the relatively short Pt···Pt distances of 3.6941(5) Å and 3.8549(5) Å.

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