Crystal structure of tetraaqua-bis(1,10-phenanthroline)-barium(II) dithiocyanate — 1,10-phenanthroline (1:2), $[\text{Ba}(\text{H}_2\text{O})_4(\text{C}_{12}\text{H}_8\text{N}_2)_2](\text{NCS})_2 \cdot 2\text{C}_{12}\text{H}_8\text{N}_2$

Li Han^{*I}, Jing-Yuan Zhang^{II}, Wei-Shan Zhu^I and Wen-Xia Ma^I

^I Tianjin University of Commerce, Department of Gemology, Tianjin 300134, P. R. China

^{II} Tianjin University, School of Material Science and Engineering, Tianjin 300072, P. R. China

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the colorless prism-like crystals of the title compound were obtained.

Experimental details

After checking their presence in the difference map, all H atoms were allowed to ride on their attached atoms, with $d(\text{C}—\text{H}) = 0.93 \text{ \AA}$ and $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$.

Discussion

1,10-Phenanthroline (phen) is a well known neutral bidentate ligand [1]. The nucleus of the title compound forms barium with two bidentate phen ligands and four water molecules (figure, top), similarly as reported for the analog structures [2,3]. The bond lengths and angles of title compound are similar to the reported in the literature. The eight-vertex coordination sphere of the barium ions has C_{2h} point-group symmetry, the two-fold axis being perpendicular to the planes formed by the chelated phens and the metal. The oxygens from the four water molecules lie in an plane perpendicular to the plane containing the phens and the barium ion. The title compound has three planar rings (figure, top). There is a fourteen-membered ring (N3/N4/C13/C14/C15/C16/C17/C18/C19/C20/C21/C22/C23/C24), with the maximum deviations from its least-squares plane of $0.046(2) \text{ \AA}$ for N4. There is a fifteen-membered ring of (Ba1/N1/N2/C1/C2/C3/C4/C5/C6/C7/C8/C9/C10/C11/C12), with the maximum deviation from its least-squares plane of $0.078(2) \text{ \AA}$ for Ba1. In fact, the molecule shows a quite planar conformation. There are several intermolecular contacts shorter than the sum of the van der Waals radii (figure, bottom). The distances of $\text{O1} \cdots \text{N5}$ ($1+x, y, z$), $\text{O1} \cdots \text{N4}$ ($2-x, 1-y, -z$) and $\text{O2} \cdots \text{N3}$ ($1-x, 1-y, -z$) are $2.871(4)$, $2.923(3)$ and $2.982(3) \text{ \AA}$, respectively.

Table 1. Data collection and handling.

Crystal:	colorless prism, size $0.10 \times 0.17 \times 0.48 \text{ mm}$
Wavelength:	Mo K_{α} radiation ($0.71069 \text{ \AA}$)
μ :	10.08 cm^{-1}
Diffractionmeter, scan mode:	Bruker Smart APEX II 4K CCD, ω
$2\theta_{\text{max}}$:	55.14°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	17123, 5311
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 5189
$N(\text{param})_{\text{refined}}$:	308
Programs:	SHELXS-97 [4], SHELXL-97 [5]

Abstract

$\text{C}_{50}\text{H}_{40}\text{BaN}_{10}\text{O}_4\text{S}_2$, triclinic, $P\bar{1}$ (no. 2), $a = 6.981(5) \text{ \AA}$, $b = 12.148(5) \text{ \AA}$, $c = 14.320(5) \text{ \AA}$, $\alpha = 76.397(5)^{\circ}$, $\beta = 87.013(5)^{\circ}$, $\gamma = 77.940(5)^{\circ}$, $V = 1154.3 \text{ \AA}^3$, $Z = 1$, $R_{\text{gof}}(F) = 0.029$, $wR_{\text{ref}}(F^2) = 0.075$, $T = 293 \text{ K}$.

Source of material

1,10-Phenanthroline (phen, 0.1 g), ammonium thiocyanate (0.1 g) and barium nitrate (0.1 g) were dissolved in water (20 ml), ethanol (20 ml) and acetone (10 ml), respectively. The mixture was placed in a beaker and was slowly evaporated at 293 K , then

* Correspondence author (e-mail: lihan369@yahoo.com.cn)

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U _{iso}
H(1)	2i	1.1919	0.1872	0.0554	0.068
H(2)	2i	1.2572	0.0081	0.1610	0.081
H(3)	2i	1.2017	-0.0057	0.3211	0.080
H(5)	2i	1.0941	0.0940	0.4556	0.082
H(6)	2i	0.9792	0.2569	0.5061	0.084
H(8)	2i	0.8524	0.4728	0.4668	0.086
H(9)	2i	0.7969	0.6441	0.3549	0.089
H(10)	2i	0.8654	0.6448	0.1950	0.073
H(13)	2i	0.4496	0.4950	0.1669	0.066
H(14)	2i	0.4072	0.4830	0.3289	0.069

Table 2. Continued.

Atom	Site	x	y	z	U _{iso}
H(15)	2i	0.4809	0.3047	0.4346	0.064
H(17)	2i	0.5975	0.0886	0.4598	0.066
H(18)	2i	0.7003	-0.0676	0.3974	0.071
H(20)	2i	0.8005	-0.1519	0.2530	0.098
H(21)	2i	0.8391	-0.1235	0.0894	0.115
H(22)	2i	0.7763	0.0622	-0.0039	0.094
H(102)	2i	1.2110	0.6774	0.0909	0.080
H(101)	2i	1.2020	0.7180	-0.0050	0.090
H(106)	2i	0.5716	0.6707	0.0633	0.100
H(105)	2i	0.5645	0.6551	-0.0347	0.140

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
Ba(1)	1c	0	½	0	0.0482(1)	0.0445(1)	0.02940(9)	-0.00902(6)	-0.00359(6)	-0.00156(6)
C(1)	2i	1.1673(3)	0.1829(2)	0.1205(2)	0.053(1)	0.056(1)	0.060(1)	-0.0063(9)	-0.005(1)	-0.015(1)
C(2)	2i	1.2082(4)	0.0741(2)	0.1840(2)	0.057(1)	0.046(1)	0.096(2)	-0.006(1)	-0.014(1)	-0.011(1)
C(3)	2i	1.1758(4)	0.0661(2)	0.2782(2)	0.053(1)	0.048(1)	0.087(2)	-0.0109(9)	-0.018(1)	0.013(1)
C(4)	2i	1.1027(3)	0.1662(2)	0.3118(2)	0.038(1)	0.062(1)	0.054(1)	-0.0171(9)	-0.0117(8)	0.014(1)
C(5)	2i	1.0682(4)	0.1643(3)	0.4107(2)	0.056(1)	0.086(2)	0.049(1)	-0.026(1)	-0.011(1)	0.024(1)
C(6)	2i	0.9996(4)	0.2609(3)	0.4408(2)	0.058(1)	0.114(2)	0.031(1)	-0.033(1)	-0.0038(9)	0.009(1)
C(7)	2i	0.9569(3)	0.3710(2)	0.3741(2)	0.042(1)	0.088(2)	0.036(1)	-0.026(1)	0.0006(8)	-0.011(1)
C(8)	2i	0.8801(4)	0.4741(3)	0.4023(2)	0.068(2)	0.113(2)	0.046(1)	-0.034(2)	0.008(1)	-0.030(2)
C(9)	2i	0.8464(4)	0.5750(3)	0.3366(2)	0.082(2)	0.084(2)	0.070(2)	-0.023(1)	0.012(1)	-0.041(2)
C(10)	2i	0.8874(4)	0.5743(2)	0.2398(2)	0.069(1)	0.057(1)	0.057(1)	-0.014(1)	0.004(1)	-0.017(1)
C(11)	2i	0.9901(3)	0.3781(2)	0.2748(1)	0.0337(8)	0.059(1)	0.0329(9)	-0.0166(7)	-0.0029(7)	-0.0027(8)
C(12)	2i	1.0642(2)	0.2730(2)	0.2428(1)	0.0300(8)	0.051(1)	0.039(1)	-0.0129(7)	-0.0059(7)	0.0021(8)
C(13)	2i	0.4810(3)	0.4226(2)	0.2087(2)	0.048(1)	0.045(1)	0.066(2)	-0.0081(8)	-0.003(1)	-0.002(1)
C(14)	2i	0.4542(3)	0.4165(2)	0.3064(2)	0.046(1)	0.054(1)	0.078(2)	-0.0102(9)	0.001(1)	-0.023(1)
C(15)	2i	0.4978(3)	0.3110(2)	0.3689(2)	0.046(1)	0.071(1)	0.047(1)	-0.0162(9)	0.0019(9)	-0.018(1)
C(16)	2i	0.5685(3)	0.2124(2)	0.3328(2)	0.0338(9)	0.053(1)	0.043(1)	-0.0146(7)	-0.0028(7)	-0.0048(8)
C(17)	2i	0.6126(3)	0.0983(2)	0.3937(2)	0.059(1)	0.065(1)	0.036(1)	-0.019(1)	-0.0045(9)	0.005(1)
C(18)	2i	0.6753(4)	0.0056(2)	0.3566(2)	0.070(1)	0.049(1)	0.049(1)	-0.015(1)	-0.011(1)	0.011(1)
C(19)	2i	0.7046(3)	0.0170(2)	0.2559(2)	0.059(1)	0.044(1)	0.053(1)	-0.0153(9)	-0.004(1)	-0.0010(9)
C(20)	2i	0.7723(6)	-0.0775(2)	0.2142(3)	0.122(3)	0.042(1)	0.076(2)	-0.015(1)	-0.000(2)	-0.009(1)
C(21)	2i	0.7969(7)	-0.0610(3)	0.1177(3)	0.151(3)	0.058(2)	0.084(2)	-0.022(2)	0.014(2)	-0.030(2)
C(22)	2i	0.7574(5)	0.0514(3)	0.0621(2)	0.115(2)	0.071(2)	0.054(2)	-0.025(2)	0.012(2)	-0.022(1)
C(23)	2i	0.6663(3)	0.1277(2)	0.1932(2)	0.042(1)	0.044(1)	0.042(1)	-0.0147(8)	-0.0022(8)	-0.0018(8)
C(25)	2i	0.3676(4)	0.7375(2)	0.2507(2)	0.081(2)	0.051(1)	0.057(2)	-0.011(1)	-0.006(1)	-0.014(1)
C(24)	2i	0.5929(2)	0.2277(2)	0.2328(1)	0.0316(8)	0.045(1)	0.040(1)	-0.0124(7)	-0.0035(7)	0.0000(8)
N(1)	2i	0.9550(2)	0.4800(2)	0.2085(1)	0.0484(9)	0.0499(9)	0.0382(9)	-0.0113(7)	-0.0009(7)	-0.0068(7)
N(2)	2i	1.0955(2)	0.2804(1)	0.1483(1)	0.0439(8)	0.0452(9)	0.0407(9)	-0.0080(7)	-0.0044(7)	-0.0038(7)
N(3)	2i	0.5480(2)	0.3327(2)	0.1711(1)	0.0449(9)	0.0442(9)	0.047(1)	-0.0106(7)	-0.0031(7)	0.0010(7)
N(4)	2i	0.6946(3)	0.1443(2)	0.0971(1)	0.069(1)	0.055(1)	0.042(1)	-0.0180(9)	0.0046(8)	-0.0083(8)
N(5)	2i	0.3938(5)	0.7302(3)	0.1722(2)	0.133(2)	0.094(2)	0.060(2)	-0.015(2)	-0.016(2)	-0.028(1)
O(1)	2i	1.1561(3)	0.6734(2)	0.0398(1)	0.071(1)	0.071(1)	0.0441(9)	-0.0331(8)	-0.0070(7)	-0.0007(8)
O(2)	2i	0.6309(2)	0.6260(2)	0.0218(1)	0.0523(9)	0.102(1)	0.047(1)	0.0060(9)	0.0042(7)	0.0074(9)
S(1)	2i	0.3427(2)	0.74532(8)	0.36240(6)	0.1364(8)	0.0820(5)	0.0551(4)	0.0012(5)	0.0214(4)	-0.0181(4)

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