Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Bis(1,10-phenanthroline-5,6-dione) diaminoglyoxime

Megha S. Deshpande,[a] Christian Näther,[b]* Wolfgang Bensch[b] and Avinash S. Kumbhar[a]

[a]Department of Chemistry, University of Pune, Pune 411 007, India, and [b]Institut für Anorganische Chemie, Christian-Albrechts-Universität Kiel, Olshausenstrasse 40, D-24098 Kiel, Germany

Correspondence e-mail: cnaether@ac.uni-kiel.de

Key indicators

Single-crystal X-ray study
T = 220 K
Mean σ(C–C) = 0.003 Å
R factor = 0.048
wR factor = 0.136
Data-to-parameter ratio = 11.0

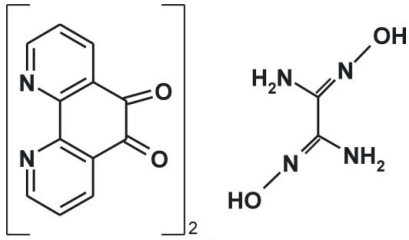
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 15 September 2006
Accepted 17 November 2006

In the crystal structure of the title cocrystal, $2C_{12}H_6N_2O_2\cdot C_2H_6N_4O_2$, the diaminoglyoxime molecules are located on inversion centres, while the 1,10-phenanthroline-5,6-dione molecules are located in general positions. Both molecules are connected through intermolecular N—H···O hydrogen bonds.

Comment

Crystals of the title compound, (I), were obtained as one of the products from the reaction of 1,10-phenanthroline-5,6-dione with diaminoglyoxime (see *Experimental*). For the accurate identification of this compound, a single-crystal structure determination was performed.



(I)

The crystal structure of (I) consists of 1,10-phenanthroline-5,6-dione molecules, which occupy general positions, and planar, *trans*-configured diaminoglyoxime molecules, which are located on inversion centres (Fig. 1). For the 1,10-phenanthroline-5,6-dione molecule, bond lengths and angles are not significantly different from those reported for the free

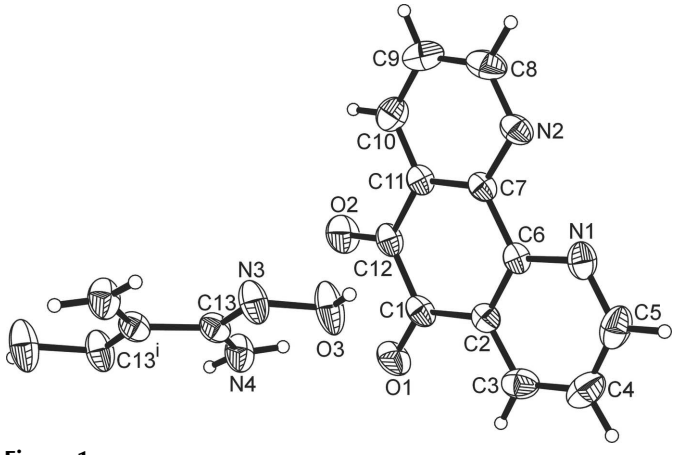


Figure 1
Molecular structure of (I), with labelling and displacement ellipsoids for non-H atoms drawn at the 50% probability level. [Symmetry code: (i) $1-x, 1-y, 1-z$.]

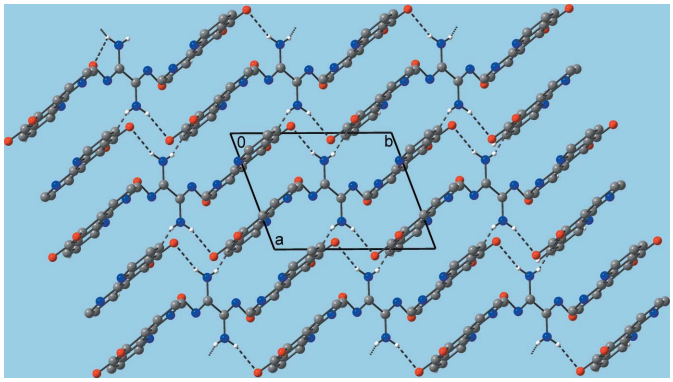


Figure 2
Crystal structure of (I), viewed along the *c* axis. Hydrogen bonds are shown as dashed lines. H atoms not involved in the hydrogen-bonding scheme have been omitted.

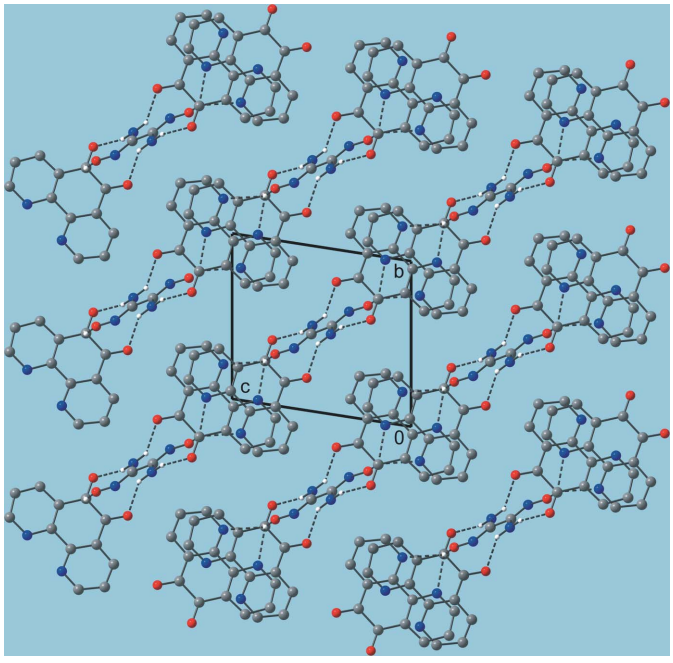


Figure 3
Crystal structure of (I), viewed along the *a* axis. Hydrogen bonds are shown as dashed lines. H atoms not involved in the hydrogen-bonding scheme have been omitted.

molecule (Calderazzo *et al.*, 1999) or its metal complexes (*e.g.* Fujihara *et al.*, 2003, 2004; Larsson & Öhrström 2004). The structural parameters of the diaminoglyoxime molecule are also very similar to those observed in pure diaminoglyoxime (Chertanova *et al.*, 1989) as well as in its metal complexes (*e.g.* Endres *et al.*, 1980), in which this ligand consistently shows a *trans* configuration.

In the crystal structure, both molecules are arranged in columns running along the *a* axis (Fig. 2). Within these columns two neighbouring 1,10-phenanthroline-5,6-dione molecules are rotated by 180° and are slightly shifted relative to each other. The shortest distance between the least-squares plane calculated through two neighbouring molecules is *ca* 3.3 Å. Both components of the cocrystal are connected into chains *via* two bifurcated O—H···N hydrogen bonds involving the hydroxy H atom at O3 and N atoms N1 and N2 (Figs. 2 and 3). The H···N separations and the O—H···N angles indicate that these interactions are rather weak (Table 1). Finally, 1,10-phenanthroline-5,6-dione molecules are further connected to diaminoglyoxime molecules *via* N—H···O hydrogen bonds between the carbonyl O atoms O1 and O2 and both amino H atoms which are attached to N4 (Table 1 and Fig. 3).

Experimental

1,10-Phenanthroline-5,6-dione was synthesized according to a literature procedure (Yamada *et al.*, 1992) and diaminoglyoxime was obtained from ACROS Organics. 1,10-Phenanthroline-5,6-dione (0.3 g, 1.43 mmol) and diaminoglyoxime (0.19 g, 1.64 mmol) were refluxed in 20 ml of methanol for 4 h. The solution was then cooled and allowed to evaporate for a day, giving (I) as red crystals, and white crystals which were not identified.

Crystal data

$2C_{12}H_6N_2O_2\cdot C_2H_6N_4O_2$
$M_r = 538.48$
Triclinic, $P\overline{1}$
$a = 7.1513\ (8)$ Å
$b = 9.3637\ (14)$ Å
$c = 9.6271\ (12)$ Å
$\alpha = 78.712\ (16)^\circ$
$\beta = 81.427\ (15)^\circ$
$\gamma = 68.269\ (15)^\circ$
$V = 585.18\ (13)$ Å^3
$Z = 1$
$D_x = 1.528$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.11$ mm^{-1}
$T = 220\ (2)$ K
Block, red
0.30 × 0.15 × 0.15 mm

Data collection

Stoe IPDS-1 diffractometer
φ scans
Absorption correction: none
3472 measured reflections
1991 independent reflections
1395 reflections with $I > 2\sigma(I)$
$R_{int} = 0.040$
$\theta_{max} = 25.0^\circ$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.136$
$S = 0.98$
1991 reflections
181 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0876P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.20$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H1O1···N2^{i}	0.83	2.27	2.984 (2)	144
O3—H1O1···N1^{i}	0.83	2.34	3.043 (3)	143
N4—H1N3···O1	0.87	2.32	3.166 (3)	166
N4—H2N3···O2^{ii}	0.87	2.42	3.149 (3)	142

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

The C-bonded H atoms were placed in idealized positions and refined with C—H bond lengths constrained to 0.94 Å and U_{iso}(H) = 1.2U_{eq}(carrier C). H atoms bonded to heteroatoms were located in a difference map and, starting from their initial positions, bond lengths were constrained to ideal values: O—H = 0.83 Å and N—H = 0.87 Å. Isotropic displacement parameters for these H atoms were fixed to U_{iso}(H) = 1.5U_{eq}(carrier atom).

Data collection: *IPDS Program Package* (Stoe & Cie, 1998); cell refinement: *IPDS Program Package*; data reduction: *IPDS Program Package*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998) and *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *SHELXTL*.

MSD acknowledges DAAD (German Academic Exchange Service) for financial support.

References

Brandenburg, K. (1999). *DIAMOND*. Release 2.1c. Crystal Impact GbR, Bonn, Germany.

Bruker (1998). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.

Calderazzo, F., Marchetti, F., Pampaloni, G. & Passarelli, V. (1999). *J. Chem. Soc. Dalton Trans.* pp. 4389–4396.

Chertanova, L. F., Yanovskii, A. I., Struchkov, Yu.-T., Sopin, V. F., Rakitin, O. A., Godovikova, T. I. & Khmel'nitskii, L. I. (1989). *J. Struct. Chem.* **30**, 129–133.

Endres, H., Jannack, T. & Prickner, B. (1980). *Acta Cryst.* B**36**, 2230–2233.

Fujihara, T., Okamura, R., Wada, T. & Tanaka, K. (2003). *J. Chem. Soc. Dalton Trans.* pp. 3221–3226.

Fujihara, T., Wada, T. & Tanaka, K. (2004). *J. Chem. Soc. Dalton Trans.* pp. 645–652.

Larsson, K. & Öhrström, L. (2004). *Inorg. Chim. Acta*, **357**, 657–664.

Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

Stoe & Cie (1998). *IPDS Program Package*. Version 2.89. Stoe & Cie, Darmstadt, Germany.

Yamada, M., Tanaka, Y., Yoshimoto, Y., Kuroda, S. & Shimao, I. (1992). *Bull. Chem. Soc. Jpn*, **65**, 1006–1011.