Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2,2′-Dinitro-5,5′-dithiodibenzoic acid

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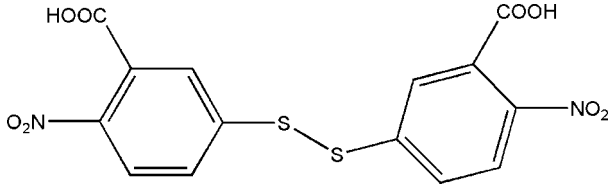
Received 23 September 2008; accepted 30 September 2008

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.007 Å; R factor = 0.051; wR factor = 0.171; data-to-parameter ratio = 11.7.

In the title compound, $C_{14}H_8N_2O_8S_2$, the asymmetric unit contains two independent 2,2′-dinitro-5,5′-dithiodibenzoic acid (Dina) molecules with roughly the same conformation. In the crystal structure, strong intermolecular O—H···O hydrogen bonds link the organic molecules into a one-dimensional zigzag chain along the a axis. The dihedral angles between the two aromatic rings [109.3 (2) and 103.1 (3)°] are larger than that (88.95°) observed in a structure of the compound with a solvent water molecule [Shefter & Kalman (1969), *J. Chem. Soc. D*, p. 1027]. Such a difference may be explained by the occurrence of O—H···O hydrogen bonds involving the water molecule in the previously reported structure.

Related literature

For general background, see: Gudbjarlson *et al.* (1991); Li *et al.* (2006); Luo *et al.* (2007); Ye *et al.* (2005). For a related structure, see: Shefter & Kalman (1969)



Experimental

Crystal data

$C_{14}H_8N_2O_8S_2$
M_r = 396.34
Triclinic, $P\overline{1}$
a = 7.875 (2) Å
b = 14.695 (4) Å
c = 15.116 (5) Å
α = 111.480 (5)°
β = 101.182 (5)°
γ = 90.572 (5)°
V = 1590.6 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.39 mm^{-1}
T = 298 (2) K
0.21 × 0.17 × 0.15 mm

Data collection

Bruker APEXII area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.921, T_{max} = 0.942
8041 measured reflections
5555 independent reflections
3077 reflections with $I > 2\sigma(I)$
R_{int} = 0.031

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.171
S = 0.92
5555 reflections
473 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.47 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O11—H1A···O22^{i}	0.82	1.83	2.635 (4)	166
O21—H2A···O12^{ii}	0.82	1.88	2.688 (4)	169
O31—H3A···O42^{i}	0.82	1.85	2.666 (4)	171
O41—H4A···O32^{ii}	0.82	1.83	2.618 (4)	160

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996), *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

The author is grateful to Lishui University for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2385).

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supplementary materials

Acta Cryst. (2008). E**64**, o2051 [**doi:10.1107/S1600536808031620**]

2,2'-Dinitro-5,5'-dithiodibenzoic acid

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Comment

Until now, various flexible carboxylate ligands can often be employed due to their versatile coordination modes and high structural stability. In particular, multi-benzenecarboxylate ligands have been shown to be good building blocks in the design of metal-organic materials (Li *et al.*, 2006; Luo *et al.*, 2007; Gudbjarlson *et al.*, 1991). On the other hand, relative strong hydrogen bondings play an important role in the formation of the ultimate netwrok (Ye *et al.*, 2005). Originally, we attempted to synthesize a complex in the presence of metal salt. However, we only obtain the starting organic material (I).

In the title compound, $[C_{14}H_8N_2O_8S]_2$, the asymmetric unit is built up by two independent 5,5'-dithiobis(2-nitrobenzcic-acid) (Dina) molecules having roughly the same conformation (Fig. 1). The geometric parameters for (I) are in the usual range. The dihedral angles between the two aromatic rings in the molecules are 109.3 (2)° and 103.1 (3)°, respectively, which indicate a twisted conformation between phenyl rings. A similar $[C_{14}H_8N_2O_8S, H_2O]$ compound, has been previously published (Shefter & Kalman, 1969). In this previous structure, the occurrence of the water molecule which acts as donor and acceptor, links the organic molecules to build up a sheet whereas in the title compound, the O—H···O hydrogen bonds form chains which develop parallel to the *a* axis (Table 1, Fig.2). In the previously reported compound, $[C_{14}H_8N_2O_8S, H_2O]$, the dihedral angles between the two aromatic rings is slightly smaller (88.95°) than in the title compound. Such difference might result from the occurrence of the water molecule which interconnects the oragnic molecule.

Experimental

5,5'-dithiobis(2-nitrobenzcic-acid) (21 mg,0.05 mmol), CoSo4 (13 mg, 0.06 mmol). were added in methanol. The mixture was refluxed under stirring for six hours. After cooling the resulting mixture to room temperature, some single crystals appeared within two weeks.

Refinement

All H atoms attached to C and O atoms were fixed geometrically and treated as riding with C—H = 0.93 Å (aromatic) and O—H = 0.82 Å with $U_{iso}(H) = 1.2U_{eq}(C, O)$.

Figures

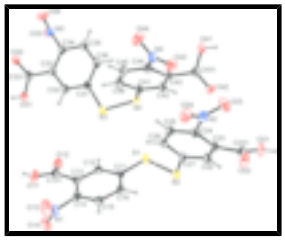


Fig. 1. Molecular view of (I) with the atom-labeling scheme. scheme. Ellipsoids are drawn at the 30% probability level. H atoms are represented as small spheres of arbitrary radii..

Fig. 2. Partial packing view showing the formation of the chain though O—H···O hydrogen bonds. H bonds are shown as dashed line. H atoms not involved in H bonds have been omitted for clarity. [Symmetry codes: (i) *x*, *y* + 1, *z*; (ii) *x*, *y* - 1, *z*.]

2,2'-Dinitro-5,5'-dithiodibenzoic acid

Crystal data

$C_{14}H_8N_2O_8S_2$	$Z = 4$
$M_r = 396.34$	$F_{000} = 808$
Triclinic, $P\overline{1}$	$D_x = 1.655$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 7.875$ (2) Å	Cell parameters from 5555 reflections
$b = 14.695$ (4) Å	$\theta = 1.5$–25.1º
$c = 15.116$ (5) Å	$\mu = 0.39$ mm^{-1}
$\alpha = 111.480$ (5)º	$T = 298$ (2) K
$\beta = 101.182$ (5)º	Block, colourless
$\gamma = 90.572$ (5)º	0.21 × 0.17 × 0.15 mm
$V = 1590.6$ (8) Å^3	

Data collection

Bruker APEXII area-detector diffractometer	5555 independent reflections
Radiation source: fine-focus sealed tube	3077 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.031$
$T = 298$(2) K	$\theta_{max} = 25.1$º
φ and ω scans	$\theta_{min} = 1.5$º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -9 \rightarrow 9$
$T_{min} = 0.921$, $T_{max} = 0.942$	$k = -17 \rightarrow 16$
8041 measured reflections	$l = -18 \rightarrow 15$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.051$	H-atom parameters constrained
$wR(F^2) = 0.171$	$w = 1/[\sigma^2(F_o^2) + (0.1043P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.92$	$(\Delta/\sigma)_{max} < 0.001$
5555 reflections	$\Delta\rho_{max} = 0.37$ e Å^{-3}
473 parameters	$\Delta\rho_{min} = -0.46$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	−0.01726 (16)	0.70300 (8)	0.33908 (9)	0.0468 (4)
S2	−0.04932 (15)	0.59689 (8)	0.39126 (9)	0.0433 (3)
N1	0.3900 (5)	1.0562 (3)	0.6593 (3)	0.0487 (11)
N2	0.5648 (5)	0.3610 (3)	0.3567 (3)	0.0430 (10)
O11	0.3554 (4)	1.1261 (2)	0.5124 (3)	0.0592 (10)
H1A	0.3428	1.1749	0.4984	0.089*
O12	0.1174 (4)	1.0668 (2)	0.3939 (3)	0.0524 (9)
O13	0.5428 (5)	1.0529 (3)	0.6900 (3)	0.0876 (15)
O14	0.3084 (5)	1.1242 (3)	0.6944 (3)	0.0700 (12)
O21	0.1194 (4)	0.2278 (2)	0.3491 (3)	0.0538 (10)
H2A	0.1308	0.1771	0.3597	0.081*
O22	0.3681 (4)	0.2775 (2)	0.4591 (3)	0.0584 (10)
O23	0.5385 (5)	0.2744 (2)	0.3064 (3)	0.0657 (11)
O24	0.7108 (4)	0.4035 (2)	0.3973 (3)	0.0579 (10)
C11	0.1118 (5)	0.8035 (3)	0.4363 (3)	0.0316 (10)
C12	0.1199 (5)	0.8905 (3)	0.4200 (3)	0.0332 (10)
H12	0.0591	0.8927	0.3617	0.040*
C13	0.2177 (5)	0.9741 (3)	0.4898 (3)	0.0326 (10)
C14	0.2998 (5)	0.9692 (3)	0.5775 (3)	0.0332 (10)
C15	0.2940 (5)	0.8827 (3)	0.5935 (3)	0.0364 (11)
H15	0.3536	0.8807	0.6521	0.044*
C16	0.2006 (5)	0.7996 (3)	0.5234 (3)	0.0330 (10)
H16	0.1969	0.7413	0.5342	0.040*
C21	0.1340 (5)	0.5290 (3)	0.3762 (3)	0.0340 (11)
C22	0.1208 (6)	0.4352 (3)	0.3793 (3)	0.0403 (12)
H22	0.0154	0.4094	0.3831	0.048*
C23	0.2633 (5)	0.3803 (3)	0.3768 (3)	0.0339 (11)
C24	0.4171 (5)	0.4200 (3)	0.3685 (3)	0.0345 (11)
C25	0.4336 (6)	0.5117 (3)	0.3644 (3)	0.0380 (11)
H25	0.5388	0.5362	0.3589	0.046*
C26	0.2938 (5)	0.5661 (3)	0.3684 (3)	0.0359 (11)
H26	0.3041	0.6282	0.3660	0.043*
C131	0.2281 (5)	1.0620 (3)	0.4637 (3)	0.0342 (10)

C231	0.2520 (5)	0.2874 (3)	0.3953 (3)	0.0377 (11)
S3	0.08887 (17)	0.82074 (8)	0.15577 (9)	0.0454 (4)
S4	0.02988 (15)	0.67435 (8)	0.09932 (9)	0.0406 (3)
N3	0.2790 (5)	0.9447 (3)	−0.1503 (3)	0.0395 (9)
N4	0.6705 (5)	0.4718 (3)	0.1507 (3)	0.0427 (10)
O31	0.2018 (4)	1.1464 (2)	0.1163 (2)	0.0482 (9)
H3A	0.2036	1.2030	0.1186	0.072*
O32	0.3339 (4)	1.1224 (2)	−0.0084 (2)	0.0509 (9)
O33	0.1633 (5)	0.9729 (3)	−0.1961 (3)	0.0640 (10)
O34	0.4258 (4)	0.9351 (3)	−0.1681 (2)	0.0581 (10)
O41	0.3935 (5)	0.3091 (2)	0.0261 (3)	0.0535 (9)
H4A	0.3956	0.2535	0.0265	0.080*
O42	0.2379 (4)	0.3353 (2)	0.1412 (2)	0.0441 (8)
O43	0.6680 (4)	0.4104 (2)	0.1883 (3)	0.0530 (9)
O44	0.7995 (4)	0.4923 (3)	0.1241 (3)	0.0688 (11)
C31	0.1522 (6)	0.8507 (3)	0.0627 (3)	0.0363 (11)
C32	0.1794 (5)	0.9510 (3)	0.0841 (3)	0.0377 (11)
H32	0.1671	0.9959	0.1441	0.045*
C33	0.2250 (5)	0.9841 (3)	0.0158 (3)	0.0330 (10)
C34	0.2427 (5)	0.9141 (3)	−0.0736 (3)	0.0331 (10)
C35	0.2196 (6)	0.8161 (3)	−0.0944 (4)	0.0430 (12)
H35	0.2338	0.7712	−0.1540	0.052*
C36	0.1747 (6)	0.7836 (3)	−0.0259 (3)	0.0412 (12)
H36	0.1596	0.7166	−0.0394	0.049*
C41	0.2267 (6)	0.6214 (3)	0.1214 (3)	0.0360 (11)
C42	0.2104 (6)	0.5236 (3)	0.1103 (3)	0.0355 (11)
H42	0.1002	0.4925	0.0981	0.043*
C43	0.3533 (5)	0.4716 (3)	0.1167 (3)	0.0314 (10)
C44	0.5177 (6)	0.5213 (3)	0.1368 (3)	0.0389 (11)
C45	0.5381 (6)	0.6202 (3)	0.1518 (3)	0.0457 (13)
H45	0.6484	0.6521	0.1665	0.055*
C46	0.3911 (6)	0.6708 (3)	0.1446 (3)	0.0422 (12)
H46	0.4023	0.7372	0.1552	0.051*
C331	0.2575 (6)	1.0908 (3)	0.0403 (4)	0.0390 (12)
C431	0.3248 (6)	0.3645 (3)	0.0970 (3)	0.0354 (11)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0637 (8)	0.0279 (6)	0.0414 (7)	−0.0014 (6)	−0.0062 (6)	0.0134 (6)
S2	0.0457 (7)	0.0277 (6)	0.0567 (8)	0.0010 (5)	0.0101 (6)	0.0167 (6)
N1	0.054 (3)	0.035 (2)	0.050 (3)	−0.009 (2)	0.002 (2)	0.013 (2)
N2	0.047 (2)	0.044 (3)	0.045 (2)	0.002 (2)	0.007 (2)	0.026 (2)
O11	0.067 (2)	0.045 (2)	0.071 (2)	−0.0140 (18)	0.0039 (19)	0.0339 (19)
O12	0.053 (2)	0.044 (2)	0.069 (2)	0.0025 (16)	0.0034 (18)	0.0375 (19)
O13	0.060 (3)	0.074 (3)	0.100 (3)	−0.018 (2)	−0.026 (2)	0.021 (3)
O14	0.095 (3)	0.037 (2)	0.058 (3)	−0.003 (2)	0.005 (2)	0.001 (2)
O21	0.058 (2)	0.0334 (19)	0.071 (2)	−0.0089 (16)	0.0007 (18)	0.0273 (18)

O22	0.057 (2)	0.051 (2)	0.072 (2)	−0.0115 (17)	−0.0105 (19)	0.040 (2)
O23	0.073 (2)	0.033 (2)	0.080 (3)	0.0124 (18)	0.014 (2)	0.009 (2)
O24	0.0402 (19)	0.066 (2)	0.074 (3)	0.0021 (17)	0.0096 (18)	0.035 (2)
C11	0.040 (2)	0.022 (2)	0.033 (2)	0.0046 (19)	0.009 (2)	0.0091 (19)
C12	0.037 (2)	0.032 (2)	0.035 (2)	0.0096 (19)	0.012 (2)	0.016 (2)
C13	0.031 (2)	0.032 (2)	0.038 (3)	0.0060 (19)	0.010 (2)	0.016 (2)
C14	0.027 (2)	0.030 (2)	0.043 (3)	0.0017 (18)	0.012 (2)	0.012 (2)
C15	0.044 (3)	0.033 (3)	0.034 (3)	0.007 (2)	0.008 (2)	0.016 (2)
C16	0.037 (2)	0.025 (2)	0.041 (3)	0.0098 (18)	0.011 (2)	0.015 (2)
C21	0.041 (2)	0.025 (2)	0.035 (3)	−0.0025 (19)	0.009 (2)	0.009 (2)
C22	0.051 (3)	0.028 (2)	0.039 (3)	−0.006 (2)	0.003 (2)	0.014 (2)
C23	0.041 (2)	0.026 (2)	0.035 (3)	−0.001 (2)	0.005 (2)	0.015 (2)
C24	0.037 (2)	0.032 (2)	0.038 (3)	0.003 (2)	0.008 (2)	0.017 (2)
C25	0.045 (3)	0.032 (3)	0.038 (3)	−0.005 (2)	0.007 (2)	0.016 (2)
C26	0.047 (3)	0.021 (2)	0.040 (3)	0.002 (2)	0.014 (2)	0.010 (2)
C131	0.037 (2)	0.028 (2)	0.042 (3)	0.003 (2)	0.018 (2)	0.013 (2)
C231	0.040 (2)	0.028 (2)	0.047 (3)	−0.002 (2)	0.013 (2)	0.016 (2)
S3	0.0688 (8)	0.0302 (6)	0.0422 (7)	0.0033 (6)	0.0186 (6)	0.0161 (6)
S4	0.0442 (7)	0.0307 (6)	0.0500 (7)	−0.0005 (5)	0.0062 (6)	0.0207 (6)
N3	0.046 (2)	0.038 (2)	0.037 (2)	0.0009 (19)	0.010 (2)	0.0160 (19)
N4	0.043 (2)	0.032 (2)	0.048 (2)	−0.0015 (19)	0.008 (2)	0.011 (2)
O31	0.063 (2)	0.0243 (17)	0.057 (2)	0.0022 (16)	0.0247 (18)	0.0081 (17)
O32	0.078 (2)	0.0269 (17)	0.053 (2)	−0.0034 (16)	0.0255 (19)	0.0158 (16)
O33	0.068 (2)	0.076 (3)	0.064 (2)	0.019 (2)	0.014 (2)	0.044 (2)
O34	0.058 (2)	0.072 (3)	0.051 (2)	0.0025 (19)	0.0209 (18)	0.026 (2)
O41	0.080 (2)	0.0263 (18)	0.061 (2)	−0.0009 (18)	0.030 (2)	0.0173 (18)
O42	0.0558 (19)	0.0312 (17)	0.055 (2)	−0.0011 (15)	0.0257 (17)	0.0209 (16)
O43	0.054 (2)	0.048 (2)	0.067 (2)	0.0120 (16)	0.0143 (18)	0.0325 (19)
O44	0.043 (2)	0.074 (3)	0.109 (3)	0.0074 (19)	0.035 (2)	0.047 (2)
C31	0.047 (3)	0.027 (2)	0.035 (3)	0.003 (2)	0.004 (2)	0.015 (2)
C32	0.042 (2)	0.029 (2)	0.040 (3)	−0.002 (2)	0.006 (2)	0.012 (2)
C33	0.031 (2)	0.027 (2)	0.040 (3)	−0.0004 (18)	0.005 (2)	0.013 (2)
C34	0.033 (2)	0.031 (2)	0.038 (3)	−0.0006 (19)	0.009 (2)	0.015 (2)
C35	0.051 (3)	0.034 (3)	0.043 (3)	0.001 (2)	0.013 (2)	0.012 (2)
C36	0.049 (3)	0.027 (2)	0.051 (3)	−0.001 (2)	0.010 (2)	0.019 (2)
C41	0.048 (3)	0.027 (2)	0.032 (2)	−0.005 (2)	0.003 (2)	0.014 (2)
C42	0.046 (3)	0.023 (2)	0.036 (3)	−0.007 (2)	0.004 (2)	0.012 (2)
C43	0.036 (2)	0.024 (2)	0.035 (2)	−0.0032 (19)	0.0035 (19)	0.015 (2)
C44	0.044 (3)	0.035 (3)	0.039 (3)	−0.002 (2)	0.003 (2)	0.019 (2)
C45	0.049 (3)	0.038 (3)	0.049 (3)	−0.014 (2)	−0.001 (2)	0.022 (2)
C46	0.047 (3)	0.027 (2)	0.054 (3)	−0.004 (2)	0.004 (2)	0.021 (2)
C331	0.048 (3)	0.025 (2)	0.041 (3)	0.000 (2)	0.002 (2)	0.012 (2)
C431	0.041 (3)	0.028 (2)	0.033 (3)	−0.002 (2)	0.002 (2)	0.010 (2)

Geometric parameters (Å, °)

S1—C11	1.782 (4)	S3—C31	1.773 (5)
S1—S2	2.0218 (16)	S3—S4	2.0133 (16)
S2—C21	1.766 (5)	S4—C41	1.770 (4)

N1—O14	1.204 (5)	N3—O33	1.205 (4)
N1—O13	1.211 (5)	N3—O34	1.235 (4)
N1—C14	1.467 (5)	N3—C34	1.461 (5)
N2—O23	1.211 (4)	N4—O43	1.231 (4)
N2—O24	1.233 (4)	N4—O44	1.235 (5)
N2—C24	1.457 (5)	N4—C44	1.433 (5)
O11—C131	1.274 (5)	O31—C331	1.302 (5)
O11—H1A	0.8200	O31—H3A	0.8200
O12—C131	1.256 (5)	O32—C331	1.238 (5)
O21—C231	1.263 (5)	O41—C431	1.303 (5)
O21—H2A	0.8200	O41—H4A	0.8200
O22—C231	1.245 (5)	O42—C431	1.216 (5)
C11—C16	1.387 (6)	C31—C36	1.388 (6)
C11—C12	1.391 (5)	C31—C32	1.393 (5)
C12—C13	1.390 (5)	C32—C33	1.393 (6)
C12—H12	0.9300	C32—H32	0.9300
C13—C14	1.386 (6)	C33—C34	1.400 (6)
C13—C131	1.489 (6)	C33—C331	1.480 (5)
C14—C15	1.379 (5)	C34—C35	1.359 (5)
C15—C16	1.374 (5)	C35—C36	1.388 (6)
C15—H15	0.9300	C35—H35	0.9300
C16—H16	0.9300	C36—H36	0.9300
C21—C22	1.400 (5)	C41—C42	1.385 (5)
C21—C26	1.409 (6)	C41—C46	1.394 (6)
C22—C23	1.386 (6)	C42—C43	1.374 (6)
C22—H22	0.9300	C42—H42	0.9300
C23—C24	1.386 (6)	C43—C44	1.403 (5)
C23—C231	1.496 (5)	C43—C431	1.498 (5)
C24—C25	1.378 (5)	C44—C45	1.388 (6)
C25—C26	1.366 (6)	C45—C46	1.390 (6)
C25—H25	0.9300	C45—H45	0.9300
C26—H26	0.9300	C46—H46	0.9300
C11—S1—S2	106.97 (15)	C31—S3—S4	105.94 (15)
C21—S2—S1	105.54 (15)	C41—S4—S3	106.38 (14)
O14—N1—O13	123.9 (4)	O33—N3—O34	123.4 (4)
O14—N1—C14	119.1 (4)	O33—N3—C34	119.0 (4)
O13—N1—C14	116.9 (4)	O34—N3—C34	117.5 (4)
O23—N2—O24	123.8 (4)	O43—N4—O44	122.6 (4)
O23—N2—C24	118.8 (4)	O43—N4—C44	118.9 (4)
O24—N2—C24	117.4 (4)	O44—N4—C44	118.5 (4)
C131—O11—H1A	109.5	C331—O31—H3A	109.5
C231—O21—H2A	109.5	C431—O41—H4A	109.5
C16—C11—C12	120.1 (4)	C36—C31—C32	120.0 (4)
C16—C11—S1	124.8 (3)	C36—C31—S3	125.5 (3)
C12—C11—S1	115.1 (3)	C32—C31—S3	114.6 (4)
C13—C12—C11	120.8 (4)	C31—C32—C33	120.1 (4)
C13—C12—H12	119.6	C31—C32—H32	119.9
C11—C12—H12	119.6	C33—C32—H32	119.9
C14—C13—C12	117.9 (4)	C32—C33—C34	118.2 (4)

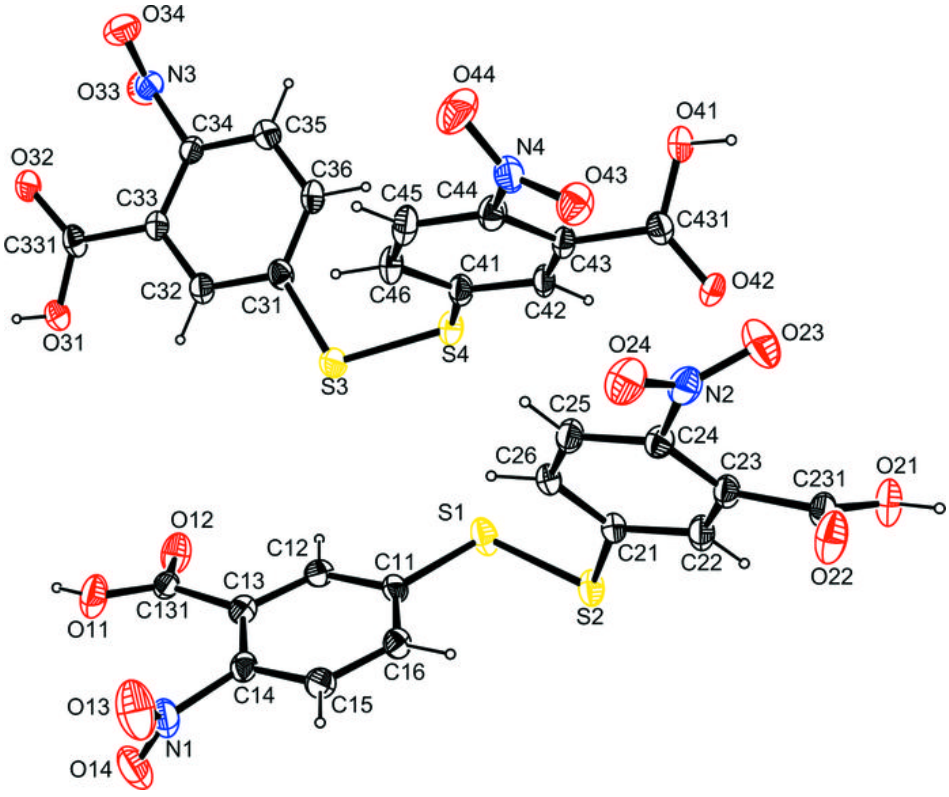
C14—C13—C131	124.9 (4)	C32—C33—C331	119.6 (4)
C12—C13—C131	117.1 (4)	C34—C33—C331	122.3 (4)
C15—C14—C13	121.3 (4)	C35—C34—C33	122.1 (4)
C15—C14—N1	116.8 (4)	C35—C34—N3	117.4 (4)
C13—C14—N1	121.8 (4)	C33—C34—N3	120.4 (4)
C16—C15—C14	120.5 (4)	C34—C35—C36	119.4 (4)
C16—C15—H15	119.8	C34—C35—H35	120.3
C14—C15—H15	119.8	C36—C35—H35	120.3
C15—C16—C11	119.3 (4)	C35—C36—C31	120.2 (4)
C15—C16—H16	120.4	C35—C36—H36	119.9
C11—C16—H16	120.4	C31—C36—H36	119.9
C22—C21—C26	118.9 (4)	C42—C41—C46	120.1 (4)
C22—C21—S2	116.9 (3)	C42—C41—S4	115.9 (3)
C26—C21—S2	124.1 (3)	C46—C41—S4	124.0 (3)
C23—C22—C21	120.8 (4)	C43—C42—C41	121.5 (4)
C23—C22—H22	119.6	C43—C42—H42	119.2
C21—C22—H22	119.6	C41—C42—H42	119.2
C24—C23—C22	118.0 (4)	C42—C43—C44	117.8 (4)
C24—C23—C231	122.4 (4)	C42—C43—C431	118.4 (3)
C22—C23—C231	119.1 (4)	C44—C43—C431	123.7 (4)
C25—C24—C23	122.5 (4)	C45—C44—C43	121.9 (4)
C25—C24—N2	117.7 (4)	C45—C44—N4	118.3 (4)
C23—C24—N2	119.7 (4)	C43—C44—N4	119.6 (4)
C26—C25—C24	119.2 (4)	C44—C45—C46	119.0 (4)
C26—C25—H25	120.4	C44—C45—H45	120.5
C24—C25—H25	120.4	C46—C45—H45	120.5
C25—C26—C21	120.6 (4)	C45—C46—C41	119.7 (4)
C25—C26—H26	119.7	C45—C46—H46	120.2
C21—C26—H26	119.7	C41—C46—H46	120.2
O12—C131—O11	124.3 (4)	O32—C331—O31	124.1 (4)
O12—C131—C13	119.0 (3)	O32—C331—C33	121.2 (5)
O11—C131—C13	116.6 (4)	O31—C331—C33	114.6 (4)
O22—C231—O21	125.0 (4)	O42—C431—O41	125.6 (4)
O22—C231—C23	117.6 (3)	O42—C431—C43	121.3 (4)
O21—C231—C23	117.2 (4)	O41—C431—C43	113.0 (4)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O11—H1A···O22^{i}	0.82	1.83	2.635 (4)	166
O21—H2A···O12^{ii}	0.82	1.88	2.688 (4)	169
O31—H3A···O42^{i}	0.82	1.85	2.666 (4)	171
O41—H4A···O32^{ii}	0.82	1.83	2.618 (4)	160

Symmetry codes: (i) *x*, *y*+1, *z*; (ii) *x*, *y*−1, *z*.

Fig. 1


O34
N3
O33
C34
C35
O32
C36
C33
C331
O31
C32
C31
S3
S4
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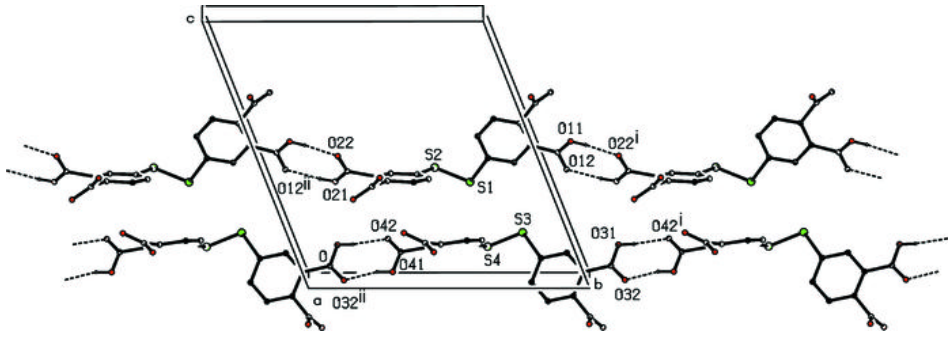
N4
O43
C45
C44
C41
C43
C46
C42
O41
C431
O42
O24
O23
N2
C25
C24
C26
C23
C231
O21
O22
S1
C21
C22
S2
O12
C12
C11
C13
C131
O11
C16
C14
C15
O13
N1
O14

Fig. 2


c
O22
S2
O12ii
O21
S1
O11
O22i
O12
O42
S3
O
O41
S4
O31
O42i
a
O32ii
b
O32