Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-Carboxyquinolinium–2,4,6-trinitrobenzenesulfonate–quinolinium-2-carboxylate (1/1/1)

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Received 20 November 2007; accepted 23 November 2007

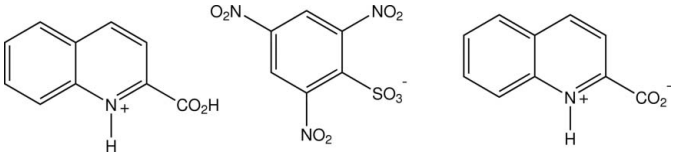
Key indicators: single-crystal X-ray study; T = 130 K; mean σ(C–C) = 0.003 Å; R factor = 0.034; wR factor = 0.090; data-to-parameter ratio = 10.6.

The structure of the title adduct compound, $C_{10}H_8NO_2^+\cdot C_6H_2N_3O_9S^-\cdot C_{10}H_7NO_2$, from the reaction of 2,4,6-trinitrobenzenesulfonic acid (picrylsulfonic acid) with quinoline-2-carboxylic acid (quinaldic acid) in 2-propanol–water, has been determined at 130 (2) K. The cation and the adduct species form a twisted cyclic hydrogen-bonded $R_2^2(10)$ pseudo-dimer which is extended into a one-dimensional chain structure through short head-to-tail carboxylic acid O—H···$O_{carboxyl}$ associations [O···O = 2.4711 (19) Å]. The picrylsulfonate anions are attached peripherally by single N—H···$O_{sulfonate}$ hydrogen bonds [N···O = 2.8643 (19) Å].

Related literature

For other related picrylsulfonate and quinaldic acid structures, see: Russell & Ward (1997); Smith *et al.* (2004); Smith, Wermuth & Healy (2006); Smith, Wermuth & White (2006); Smith, Wermuth, Healy & White (2007); Smith, Wermuth & White (2007); Dobrzyńska & Jerzykiewicz (2004).

For graph-set nomenclature, see: Etter *et al.* (1990).



Experimental

Crystal data

$C_{10}H_8NO_2^+\cdot C_6H_2N_3O_9S^-\cdot C_{10}H_7NO_2$
M_r = 639.51
Triclinic, $P\overline{1}$
a = 7.8872 (6) Å
b = 12.4753 (10) Å
c = 14.6617 (12) Å
α = 66.227 (1)°
β = 74.997 (2)°
γ = 82.191 (2)°
V = 1274.42 (18) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.21 mm^{-1}
T = 130 (2) K
0.40 × 0.30 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)
T_{min} = 0.93, T_{max} = 0.98
6761 measured reflections
4446 independent reflections
3779 reflections with $I > 2\sigma(I)$
R_{int} = 0.039

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.090
S = 1.00
4446 reflections
419 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1A—H1A···O21A	0.87 (2)	2.31 (2)	2.690 (2)	106.3 (18)
N1A—H1A···O21B	0.87 (2)	1.94 (2)	2.761 (2)	155 (2)
N1B—H1B···O13	0.85 (2)	2.23 (2)	2.8643 (19)	132 (2)
N1B—H1B···O21A	0.85 (2)	2.14 (3)	2.746 (2)	128 (2)
N1B—H1B···O21B	0.85 (2)	2.30 (3)	2.683 (2)	108 (2)
O22A—H22A···O22B^{i}	0.97 (2)	1.50 (2)	2.4711 (19)	179 (3)
C4B—H4B···O41^{ii}	0.95	2.37	3.240 (2)	152
C5B—H5B···O12^{iii}	0.95	2.44	3.339 (2)	158
C8A—H8A···O21B	0.95	2.39	3.122 (2)	134

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON*.

The authors acknowledge financial support from the School of Physical and Chemical Sciences, Queensland University of Technology, the School of Biomolecular and Physical Sciences, Griffith University, and the School of Chemistry, University of Melbourne.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SF2012).

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supplementary materials

Acta Cryst. (2008). E**64**, o132-o133 [**doi:10.1107/S1600536807062757**]

2-Carboxyquinolinium-2,4,6-trinitrobenzenesulfonate-quinolinium-2-carboxylate (1/1/1)

G. Smith, U. D. Wermuth and J. M. White

Comment

Picrylsulfonic acid (2,4,6-trinitrobenzenesulfonic acid) reacts with certain Lewis bases to form 1:1 proton-transfer salts and the structures of a small number of these are known: with guanidine (Russell & Ward, 1997) and quinoline (Smith, Wermuth & Healy, 2006). However, the 1:1 reaction with quinoline-2-carboxylic acid in 80% 2-propanol-water resulted in the adduct salt 2-carboxyquinolinium-2,4,6-trinitrobenzenesulfonate-quinolinium- 2-carboxylate (1/1/1) (I) and the structure is reported here. In (I), the asymmetric unit comprises a protonated quinaldic acid cation (A), a picrylsulfonate anion and a zwitterionic quinaldic acid adduct molecule (B) (Fig. 1). The cation and the adduct species form a pseudo-dimer through a twisted cyclic hydrogen-bonded duplex N–H···O association [graph set $R^2_2(10)$ (Etter *et al.*, 1990]. This pseudo-dimer incorporates a cyclic $R^2_2(4)$ association and two intramolecular S(5) *N*–*H*···$O_{carboxyl}$ associations and is similar to that found in the zwitterionic parent acid (Dobrzyńska & Jerzykiewicz, 2004), the 1:2 *L*-tartaric acid-quinaldic acid adduct (Smith, Wermuth & White, 2006) and in the analogous (1:1:1) protonated quinaldic acid-zwitterionic adduct compounds with 5-sulfosalicylic acid (Smith *et al.*, 2004) and 4,5-dichlorophthalic acid (Smith, Wermuth & White, 2007). However, in the 1:1 compound with 3,5-dinitrosalicylic acid (Smith, Wermuth, Healy & White, 2007), this dimer is not found.

In (I), the pseudo-dimers are extended into one-dimensional chain structures through short head-to-tail carboxylic acid O–H··· $O_{carboxyl}$ associations [O···O, 2.4711 (19) Å] (Fig. 2). The picrylsulfonate anions are attached peripherally by single *N*–*H*···$O_{sulfonate}$ hydrogen bonds (Table 1).

All nitro groups of the anion are rotated out of the plane of the benzene ring, particularly those which are *ortho* to the sulfonate group [torsion angle C1–C2–N2–O22, −139.19 (17) °; C5–C6–N6–O62, 115.70 (17) °], compared to the *para*- related group [torsion angle C3–C4–N4–O42, 165.79 (16) °].

Experimental

The title compound was synthesized by heating under reflux 1 mmol quantities of 2,4,6-trinitrobenzenesulfonic acid (picrylsulfonic acid) and quinoline-2-carboxylic acid (quinaldic acid) in 50 ml of 80% 2-propanol-water for 10 minutes. After concentration to *ca* 30 ml, partial room temperature evaporation of the hot-filtered solution gave pale yellow flat prisms of (I) [m.pt. 495–496 K].

Refinement

Interactive hydrogen atoms were located by difference methods and their positional and isotropic displacement parameters were refined. The aromatic ring H atoms were included in the refinement in calculated positions (C–H = 0.95 Å) using a riding model approximation, with $U_{iso}(H) = 1.2U_{eq}(C)$.

Figures

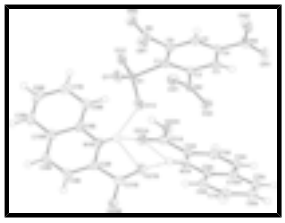


Fig. 1. Molecular configuration and atom naming scheme for the cation, anion and the zwitterionic adduct species in (I). Inter-species hydrogen-bonding interactions are shown as dashed lines. Non-H atom displacement ellipsoids are drawn at the 50% probability level.

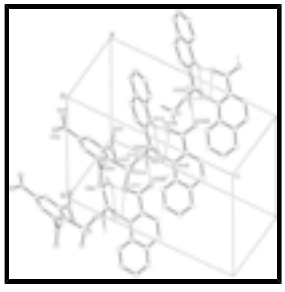


Fig. 2. A perspective view of the one-dimensional head-to-tail cation-adduct dimer extension and peripheral N–H···$O_{sulfonate}$ associations in (I). Non-interactive H-atoms are omitted while hydrogen bonds are shown as dashed lines. Symmetry code (iv): $x + 1, y, z$. For other symmetry codes see Table 1.

2-Carboxyquinolinium–2,4,6-trinitrobenzenesulfonate– quinolinium-2-carboxylate (1/1/1)

Crystal data

$C_{10}H_8NO_2^+·C_6H_2N_3O_9S^-·C_{10}H_7NO_2$	$Z = 2$
$M_r = 639.51$	$F_{000} = 656$
Triclinic, $P\overline{1}$	$D_x = 1.667$ Mg m^{-3}
Hall symbol: -P 1	Melting point: 495–496 K
$a = 7.8872$ (6) Å	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$b = 12.4753$ (10) Å	Cell parameters from 3558 reflections
$c = 14.6617$ (12) Å	$\theta = 2.7$–27.5º
$\alpha = 66.227$ (1)º	$\mu = 0.21$ mm^{-1}
$\beta = 74.997$ (2)º	$T = 130$ (2) K
$\gamma = 82.191$ (2)º	Plate, colourless
$V = 1274.42$ (18) Å^3	0.40 × 0.30 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer	4446 independent reflections
Radiation source: sealed tube	3779 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.039$
$T = 130$(2) K	$\theta_{max} = 25.0$º
φ and ω scans	$\theta_{min} = 1.6$º
Absorption correction: multi-scan (SADABS; Bruker, 1999)	$h = -9 \rightarrow 9$
$T_{min} = 0.93$, $T_{max} = 0.98$	$k = -14 \rightarrow 14$
6761 measured reflections	$l = -10 \rightarrow 17$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.034$

$wR(F^2) = 0.090$

$S = 1.00$

4446 reflections

419 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0504P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.26$ e Å^{-3}

$\Delta\rho_{min} = -0.36$ e Å^{-3}

Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0035 (9)

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

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 $F^2 > 2$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}
O21A	0.58977 (15)	0.59940 (10)	0.39221 (10)	0.0254 (4)
O21B	1.00107 (15)	0.64175 (10)	0.34714 (10)	0.0241 (4)
O22A	0.38775 (15)	0.50663 (10)	0.36987 (9)	0.0228 (4)
O22B	1.16069 (15)	0.60475 (11)	0.46423 (10)	0.0259 (4)
N1A	0.84672 (18)	0.51549 (12)	0.27485 (11)	0.0189 (4)
N1B	0.74154 (18)	0.73921 (12)	0.45119 (11)	0.0185 (5)
C2A	0.6823 (2)	0.48449 (14)	0.29343 (13)	0.0195 (5)
C2B	0.8899 (2)	0.70550 (14)	0.48319 (13)	0.0180 (5)
C3A	0.6451 (2)	0.41203 (14)	0.24976 (14)	0.0223 (5)
C3B	0.9103 (2)	0.72614 (14)	0.56645 (13)	0.0213 (5)
C4A	0.7771 (2)	0.37568 (15)	0.18609 (14)	0.0236 (6)
C4B	0.7771 (2)	0.78111 (15)	0.61378 (14)	0.0232 (5)
C5A	1.0908 (2)	0.38090 (16)	0.09579 (14)	0.0274 (6)
C5B	0.4742 (2)	0.86973 (15)	0.62801 (14)	0.0246 (6)
C6A	1.2556 (2)	0.41723 (16)	0.07874 (14)	0.0284 (6)
C6B	0.3237 (2)	0.89788 (15)	0.59302 (15)	0.0267 (6)

C7A	1.2868 (2)	0.48655 (16)	0.12761 (14)	0.0265 (6)
C7B	0.3095 (2)	0.87352 (15)	0.50922 (14)	0.0248 (6)
C8A	1.1553 (2)	0.51932 (15)	0.19338 (13)	0.0224 (5)
C8B	0.4452 (2)	0.82010 (14)	0.46165 (14)	0.0217 (5)
C9A	0.9842 (2)	0.48298 (14)	0.21127 (13)	0.0200 (5)
C9B	0.6008 (2)	0.79115 (14)	0.49680 (13)	0.0176 (5)
C10A	0.9496 (2)	0.41196 (14)	0.16341 (14)	0.0217 (6)
C10B	0.6178 (2)	0.81513 (14)	0.58089 (13)	0.0202 (5)
C21A	0.5445 (2)	0.53589 (14)	0.35874 (13)	0.0198 (5)
C21B	1.0288 (2)	0.64568 (14)	0.42510 (14)	0.0206 (5)
S1	0.67941 (6)	0.96877 (4)	0.18966 (3)	0.0221 (1)
O11	0.79371 (16)	1.05360 (11)	0.18376 (10)	0.0275 (4)
O12	0.49579 (16)	0.99398 (11)	0.22231 (9)	0.0270 (4)
O13	0.73845 (16)	0.84886 (11)	0.23828 (9)	0.0288 (4)
O21	0.48489 (16)	0.78413 (11)	0.16864 (10)	0.0287 (4)
O22	0.69059 (17)	0.69083 (10)	0.09636 (10)	0.0296 (4)
O41	0.89200 (16)	0.91344 (11)	−0.26263 (10)	0.0280 (4)
O42	0.90947 (17)	1.10219 (11)	−0.31897 (9)	0.0328 (4)
O61	0.92941 (16)	1.23709 (11)	−0.02903 (10)	0.0295 (4)
O62	0.64569 (16)	1.23611 (11)	0.02533 (10)	0.0295 (4)
N2	0.61840 (19)	0.78074 (13)	0.10551 (11)	0.0222 (5)
N4	0.87752 (18)	1.00599 (13)	−0.25023 (11)	0.0224 (5)
N6	0.78729 (19)	1.19400 (12)	−0.00650 (11)	0.0219 (5)
C1	0.7125 (2)	0.98489 (15)	0.05707 (13)	0.0182 (5)
C2	0.6957 (2)	0.89262 (14)	0.02920 (13)	0.0183 (5)
C3	0.7470 (2)	0.89921 (14)	−0.07008 (13)	0.0189 (5)
C4	0.8198 (2)	1.00040 (15)	−0.14463 (13)	0.0187 (5)
C5	0.8392 (2)	1.09627 (15)	−0.12484 (13)	0.0194 (5)
C6	0.7815 (2)	1.08587 (14)	−0.02403 (13)	0.0189 (5)
H1A	0.863 (3)	0.5625 (18)	0.3025 (16)	0.036 (6)*
H1B	0.737 (3)	0.7269 (19)	0.3989 (18)	0.047 (7)*
H3A	0.52850	0.38810	0.26420	0.0270*
H3B	1.01600	0.70210	0.59000	0.0260*
H4A	0.75210	0.32560	0.15700	0.0280*
H4B	0.79170	0.79670	0.66950	0.0280*
H5A	1.07080	0.33450	0.06200	0.0330*
H5B	0.48240	0.88690	0.68420	0.0300*
H6A	1.34990	0.39550	0.03350	0.0340*
H6B	0.22730	0.93430	0.62530	0.0320*
H7A	1.40250	0.51120	0.11450	0.0320*
H7B	0.20370	0.89460	0.48550	0.0300*
H8A	1.17860	0.56560	0.22640	0.0270*
H8B	0.43400	0.80300	0.40590	0.0260*
H22A	0.298 (3)	0.5460 (19)	0.4062 (18)	0.057 (7)*
H3	0.73240	0.83520	−0.08650	0.0230*
H5	0.88950	1.16590	−0.17750	0.0230*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O21A	0.0235 (7)	0.0263 (7)	0.0307 (8)	−0.0017 (5)	−0.0044 (6)	−0.0160 (6)
O21B	0.0219 (7)	0.0299 (7)	0.0259 (7)	−0.0004 (5)	−0.0044 (6)	−0.0169 (6)
O22A	0.0186 (7)	0.0258 (7)	0.0267 (7)	−0.0008 (5)	−0.0045 (6)	−0.0131 (6)
O22B	0.0215 (7)	0.0315 (7)	0.0275 (7)	0.0055 (5)	−0.0079 (6)	−0.0148 (6)
N1A	0.0204 (8)	0.0189 (7)	0.0192 (8)	−0.0005 (6)	−0.0049 (6)	−0.0090 (7)
N1B	0.0215 (8)	0.0199 (8)	0.0162 (8)	−0.0008 (6)	−0.0036 (6)	−0.0094 (7)
C2A	0.0225 (9)	0.0155 (8)	0.0176 (9)	0.0001 (7)	−0.0066 (8)	−0.0025 (7)
C2B	0.0198 (9)	0.0145 (8)	0.0174 (9)	−0.0026 (7)	−0.0043 (7)	−0.0031 (7)
C3A	0.0227 (9)	0.0185 (9)	0.0246 (10)	−0.0028 (7)	−0.0050 (8)	−0.0068 (8)
C3B	0.0228 (9)	0.0216 (9)	0.0190 (9)	−0.0024 (7)	−0.0062 (8)	−0.0060 (8)
C4A	0.0301 (10)	0.0191 (9)	0.0246 (10)	−0.0015 (8)	−0.0070 (8)	−0.0106 (8)
C4B	0.0306 (10)	0.0237 (9)	0.0180 (9)	−0.0064 (8)	−0.0054 (8)	−0.0092 (8)
C5A	0.0334 (11)	0.0268 (10)	0.0238 (10)	0.0017 (8)	−0.0044 (9)	−0.0136 (9)
C5B	0.0326 (11)	0.0208 (9)	0.0202 (10)	−0.0043 (8)	0.0018 (8)	−0.0113 (8)
C6A	0.0278 (10)	0.0302 (10)	0.0237 (10)	0.0040 (8)	−0.0005 (8)	−0.0116 (9)
C6B	0.0261 (10)	0.0208 (9)	0.0275 (11)	−0.0007 (8)	0.0048 (8)	−0.0103 (8)
C7A	0.0217 (10)	0.0296 (10)	0.0244 (10)	0.0000 (8)	−0.0031 (8)	−0.0080 (9)
C7B	0.0218 (10)	0.0218 (9)	0.0279 (11)	−0.0018 (7)	−0.0016 (8)	−0.0086 (8)
C8A	0.0224 (9)	0.0240 (9)	0.0210 (10)	−0.0007 (8)	−0.0061 (8)	−0.0081 (8)
C8B	0.0234 (9)	0.0210 (9)	0.0206 (10)	−0.0035 (7)	−0.0032 (8)	−0.0081 (8)
C9A	0.0239 (9)	0.0179 (9)	0.0155 (9)	0.0030 (7)	−0.0049 (7)	−0.0046 (7)
C9B	0.0203 (9)	0.0147 (8)	0.0165 (9)	−0.0021 (7)	−0.0003 (7)	−0.0065 (7)
C10A	0.0255 (10)	0.0178 (9)	0.0208 (10)	0.0020 (7)	−0.0065 (8)	−0.0065 (8)
C10B	0.0260 (10)	0.0156 (8)	0.0171 (9)	−0.0050 (7)	−0.0004 (8)	−0.0057 (7)
C21A	0.0225 (9)	0.0160 (8)	0.0190 (9)	−0.0001 (7)	−0.0054 (8)	−0.0046 (8)
C21B	0.0197 (9)	0.0190 (9)	0.0221 (10)	−0.0035 (7)	−0.0031 (8)	−0.0069 (8)
S1	0.0203 (2)	0.0327 (3)	0.0160 (2)	0.0025 (2)	−0.0048 (2)	−0.0128 (2)
O11	0.0244 (7)	0.0413 (8)	0.0250 (7)	−0.0010 (6)	−0.0066 (6)	−0.0205 (6)
O12	0.0215 (7)	0.0408 (8)	0.0223 (7)	0.0018 (6)	−0.0029 (5)	−0.0179 (6)
O13	0.0304 (7)	0.0362 (8)	0.0181 (7)	0.0055 (6)	−0.0078 (6)	−0.0094 (6)
O21	0.0224 (7)	0.0347 (7)	0.0228 (7)	−0.0028 (6)	−0.0002 (6)	−0.0072 (6)
O22	0.0380 (8)	0.0202 (7)	0.0280 (7)	0.0022 (6)	−0.0056 (6)	−0.0087 (6)
O41	0.0296 (7)	0.0351 (8)	0.0272 (7)	−0.0040 (6)	−0.0021 (6)	−0.0217 (6)
O42	0.0425 (8)	0.0357 (8)	0.0172 (7)	−0.0152 (6)	−0.0019 (6)	−0.0057 (6)
O61	0.0277 (7)	0.0296 (7)	0.0372 (8)	−0.0040 (6)	−0.0095 (6)	−0.0166 (6)
O62	0.0289 (7)	0.0296 (7)	0.0350 (8)	0.0063 (6)	−0.0075 (6)	−0.0196 (6)
N2	0.0228 (8)	0.0248 (8)	0.0200 (8)	0.0008 (6)	−0.0081 (7)	−0.0082 (7)
N4	0.0209 (8)	0.0300 (9)	0.0175 (8)	−0.0058 (7)	−0.0023 (6)	−0.0101 (7)
N6	0.0229 (9)	0.0238 (8)	0.0225 (8)	0.0009 (7)	−0.0072 (7)	−0.0116 (7)
C1	0.0151 (8)	0.0244 (9)	0.0168 (9)	0.0037 (7)	−0.0057 (7)	−0.0096 (8)
C2	0.0172 (9)	0.0182 (8)	0.0185 (9)	0.0016 (7)	−0.0056 (7)	−0.0059 (7)
C3	0.0183 (9)	0.0202 (9)	0.0213 (9)	0.0030 (7)	−0.0067 (7)	−0.0110 (8)
C4	0.0169 (9)	0.0249 (9)	0.0160 (9)	0.0012 (7)	−0.0040 (7)	−0.0100 (8)
C5	0.0171 (9)	0.0208 (9)	0.0203 (10)	0.0006 (7)	−0.0053 (7)	−0.0075 (8)

C6	0.0174 (9)	0.0214 (9)	0.0228 (10)	0.0037 (7)	−0.0078 (8)	−0.0129 (8)

Geometric parameters (Å, °)

S1—C1	1.8232 (18)	C5A—C10A	1.412 (3)
S1—O13	1.4442 (15)	C5A—C6A	1.363 (2)
S1—O11	1.4440 (15)	C5B—C6B	1.361 (2)
S1—O12	1.4367 (14)	C5B—C10B	1.410 (3)
O21A—C21A	1.215 (2)	C6A—C7A	1.404 (3)
O21B—C21B	1.239 (2)	C6B—C7B	1.413 (3)
O22A—C21A	1.288 (2)	C7A—C8A	1.363 (3)
O22B—C21B	1.265 (2)	C7B—C8B	1.368 (3)
O22A—H22A	0.97 (2)	C8A—C9A	1.408 (2)
O21—N2	1.218 (2)	C8B—C9B	1.400 (2)
O22—N2	1.228 (2)	C9A—C10A	1.421 (3)
O41—N4	1.226 (2)	C9B—C10B	1.422 (2)
O42—N4	1.223 (2)	C3A—H3A	0.9500
O61—N6	1.218 (2)	C3B—H3B	0.9500
O62—N6	1.229 (2)	C4A—H4A	0.9500
N1A—C2A	1.332 (2)	C4B—H4B	0.9500
N1A—C9A	1.368 (2)	C5A—H5A	0.9500
N1B—C9B	1.367 (2)	C5B—H5B	0.9500
N1B—C2B	1.326 (2)	C6A—H6A	0.9500
N1A—H1A	0.87 (2)	C6B—H6B	0.9500
N1B—H1B	0.85 (2)	C7A—H7A	0.9500
N2—C2	1.480 (2)	C7B—H7B	0.9500
N4—C4	1.472 (2)	C8A—H8A	0.9500
N6—C6	1.480 (2)	C8B—H8B	0.9500
C2A—C3A	1.396 (3)	C1—C2	1.399 (3)
C2A—C21A	1.509 (3)	C1—C6	1.393 (3)
C2B—C21B	1.513 (3)	C2—C3	1.377 (2)
C2B—C3B	1.396 (2)	C3—C4	1.370 (3)
C3A—C4A	1.368 (3)	C4—C5	1.375 (3)
C3B—C4B	1.366 (3)	C5—C6	1.386 (2)
C4A—C10A	1.407 (2)	C3—H3	0.9500
C4B—C10B	1.413 (2)	C5—H5	0.9500
O12—S1—O13	115.62 (8)	C5B—C10B—C9B	118.43 (15)
O12—S1—C1	105.96 (8)	C4B—C10B—C5B	123.52 (16)
O13—S1—C1	102.31 (8)	O21A—C21A—C2A	118.93 (15)
O11—S1—C1	103.56 (8)	O22A—C21A—C2A	113.06 (15)
O11—S1—O12	114.08 (9)	O21A—C21A—O22A	127.99 (17)
O11—S1—O13	113.42 (8)	O21B—C21B—C2B	117.66 (15)
C21A—O22A—H22A	113.0 (15)	O22B—C21B—C2B	114.26 (16)
C2A—N1A—C9A	123.24 (16)	O21B—C21B—O22B	128.07 (17)
C2B—N1B—C9B	124.03 (15)	C2A—C3A—H3A	120.00
C9A—N1A—H1A	120.2 (16)	C4A—C3A—H3A	120.00
C2A—N1A—H1A	116.5 (16)	C2B—C3B—H3B	120.00
C9B—N1B—H1B	119.9 (17)	C4B—C3B—H3B	120.00
C2B—N1B—H1B	116.0 (17)	C10A—C4A—H4A	120.00

O21—N2—C2	118.58 (16)	C3A—C4A—H4A	120.00
O22—N2—C2	116.42 (14)	C10B—C4B—H4B	120.00
O21—N2—O22	124.89 (16)	C3B—C4B—H4B	120.00
O41—N4—C4	117.43 (15)	C6A—C5A—H5A	120.00
O41—N4—O42	124.80 (15)	C10A—C5A—H5A	120.00
O42—N4—C4	117.77 (16)	C6B—C5B—H5B	120.00
O61—N6—C6	118.03 (15)	C10B—C5B—H5B	120.00
O62—N6—C6	116.65 (15)	C5A—C6A—H6A	120.00
O61—N6—O62	125.23 (16)	C7A—C6A—H6A	120.00
C3A—C2A—C21A	123.84 (15)	C5B—C6B—H6B	120.00
N1A—C2A—C21A	116.27 (16)	C7B—C6B—H6B	120.00
N1A—C2A—C3A	119.84 (16)	C6A—C7A—H7A	119.00
N1B—C2B—C3B	119.65 (16)	C8A—C7A—H7A	119.00
C3B—C2B—C21B	123.91 (15)	C6B—C7B—H7B	119.00
N1B—C2B—C21B	116.44 (15)	C8B—C7B—H7B	119.00
C2A—C3A—C4A	119.76 (16)	C7A—C8A—H8A	121.00
C2B—C3B—C4B	119.51 (16)	C9A—C8A—H8A	121.00
C3A—C4A—C10A	120.41 (17)	C7B—C8B—H8B	121.00
C3B—C4B—C10B	120.86 (17)	C9B—C8B—H8B	121.00
C6A—C5A—C10A	120.34 (18)	S1—C1—C2	123.06 (14)
C6B—C5B—C10B	120.03 (17)	S1—C1—C6	121.54 (14)
C5A—C6A—C7A	120.44 (17)	C2—C1—C6	114.77 (16)
C5B—C6B—C7B	120.70 (17)	N2—C2—C1	121.68 (15)
C6A—C7A—C8A	121.67 (16)	N2—C2—C3	115.28 (16)
C6B—C7B—C8B	121.20 (16)	C1—C2—C3	123.04 (16)
C7A—C8A—C9A	118.51 (17)	C2—C3—C4	118.32 (17)
C7B—C8B—C9B	118.57 (17)	N4—C4—C3	118.03 (17)
C8A—C9A—C10A	120.87 (16)	N4—C4—C5	119.17 (16)
N1A—C9A—C8A	120.92 (16)	C3—C4—C5	122.80 (16)
N1A—C9A—C10A	118.21 (15)	C4—C5—C6	116.46 (16)
N1B—C9B—C8B	121.07 (16)	N6—C6—C1	120.05 (15)
N1B—C9B—C10B	117.84 (15)	N6—C6—C5	115.32 (15)
C8B—C9B—C10B	121.08 (16)	C1—C6—C5	124.54 (17)
C4A—C10A—C5A	123.37 (17)	C2—C3—H3	121.00
C5A—C10A—C9A	118.16 (16)	C4—C3—H3	121.00
C4A—C10A—C9A	118.47 (16)	C4—C5—H5	122.00
C4B—C10B—C9B	118.05 (16)	C6—C5—H5	122.00
O13—S1—C1—C6	−138.21 (15)	C3A—C4A—C10A—C9A	2.5 (3)
O12—S1—C1—C2	−89.31 (16)	C3B—C4B—C10B—C5B	178.54 (18)
O11—S1—C1—C2	150.32 (15)	C3B—C4B—C10B—C9B	−0.4 (3)
O11—S1—C1—C6	−20.10 (17)	C6A—C5A—C10A—C4A	−178.72 (18)
O12—S1—C1—C6	100.27 (15)	C6A—C5A—C10A—C9A	0.9 (3)
O13—S1—C1—C2	32.21 (16)	C10A—C5A—C6A—C7A	−0.4 (3)
C9A—N1A—C2A—C21A	−175.27 (16)	C6B—C5B—C10B—C9B	0.2 (3)
C9A—N1A—C2A—C3A	2.3 (3)	C10B—C5B—C6B—C7B	−0.3 (3)
C2A—N1A—C9A—C10A	−0.5 (3)	C6B—C5B—C10B—C4B	−178.74 (18)
C2A—N1A—C9A—C8A	179.27 (17)	C5A—C6A—C7A—C8A	0.3 (3)
C9B—N1B—C2B—C3B	−2.1 (3)	C5B—C6B—C7B—C8B	0.7 (3)
C2B—N1B—C9B—C10B	2.9 (3)	C6A—C7A—C8A—C9A	−0.6 (3)

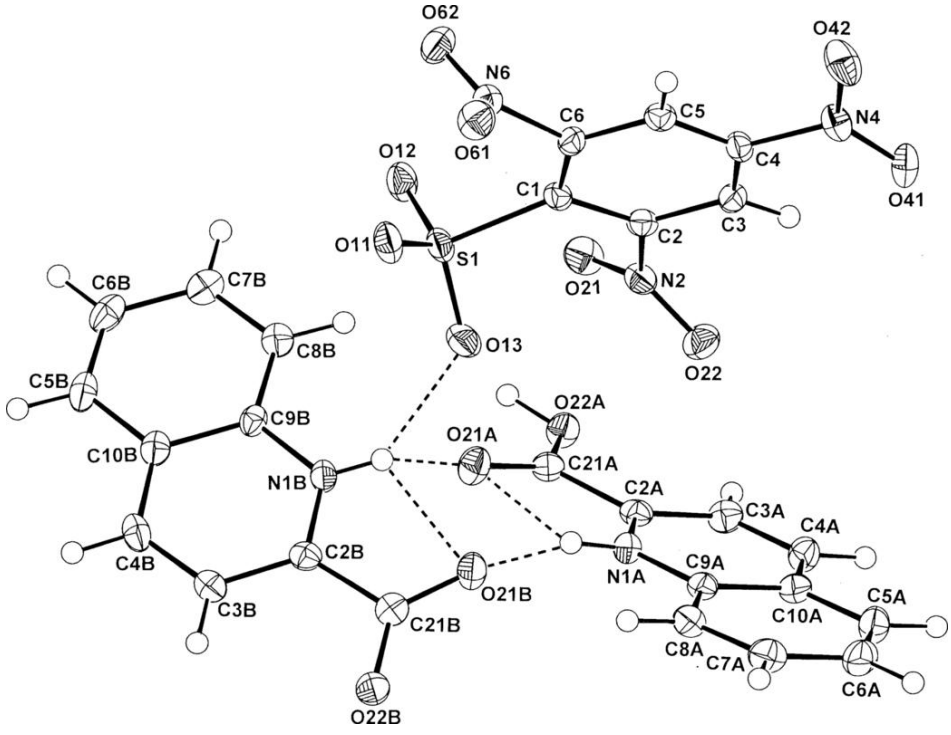
C9B—N1B—C2B—C21B	177.93 (16)	C6B—C7B—C8B—C9B	−0.9 (3)
C2B—N1B—C9B—C8B	−177.26 (17)	C7A—C8A—C9A—C10A	1.1 (3)
O21—N2—C2—C3	−134.74 (17)	C7A—C8A—C9A—N1A	−178.59 (17)
O21—N2—C2—C1	44.5 (2)	C7B—C8B—C9B—N1B	−179.04 (17)
O22—N2—C2—C1	−139.19 (17)	C7B—C8B—C9B—C10B	0.8 (3)
O22—N2—C2—C3	41.6 (2)	N1A—C9A—C10A—C4A	−1.9 (3)
O41—N4—C4—C3	−15.1 (2)	C8A—C9A—C10A—C5A	−1.3 (3)
O42—N4—C4—C5	−13.6 (2)	N1A—C9A—C10A—C5A	178.43 (16)
O41—N4—C4—C5	165.55 (16)	C8A—C9A—C10A—C4A	178.37 (17)
O42—N4—C4—C3	165.79 (16)	N1B—C9B—C10B—C4B	−1.6 (3)
O61—N6—C6—C1	122.44 (18)	N1B—C9B—C10B—C5B	179.39 (16)
O62—N6—C6—C1	−61.0 (2)	C8B—C9B—C10B—C4B	178.57 (17)
O62—N6—C6—C5	115.70 (17)	C8B—C9B—C10B—C5B	−0.4 (3)
O61—N6—C6—C5	−60.9 (2)	S1—C1—C2—N2	11.3 (2)
N1A—C2A—C3A—C4A	−1.6 (3)	S1—C1—C2—C3	−169.48 (14)
C21A—C2A—C3A—C4A	175.71 (17)	C6—C1—C2—N2	−177.64 (15)
C3A—C2A—C21A—O22A	−0.4 (2)	C6—C1—C2—C3	1.5 (3)
N1A—C2A—C21A—O21A	−1.6 (2)	S1—C1—C6—N6	−15.7 (2)
N1A—C2A—C21A—O22A	177.05 (15)	S1—C1—C6—C5	167.94 (14)
C3A—C2A—C21A—O21A	−178.99 (17)	C2—C1—C6—N6	173.10 (15)
N1B—C2B—C3B—C4B	−0.2 (3)	C2—C1—C6—C5	−3.2 (3)
C21B—C2B—C3B—C4B	179.87 (17)	N2—C2—C3—C4	−179.94 (15)
N1B—C2B—C21B—O21B	6.2 (2)	C1—C2—C3—C4	0.8 (3)
C3B—C2B—C21B—O22B	6.6 (3)	C2—C3—C4—N4	178.82 (15)
C3B—C2B—C21B—O21B	−173.77 (17)	C2—C3—C4—C5	−1.8 (3)
N1B—C2B—C21B—O22B	−173.36 (16)	N4—C4—C5—C6	179.65 (15)
C2A—C3A—C4A—C10A	−0.8 (3)	C3—C4—C5—C6	0.3 (3)
C2B—C3B—C4B—C10B	1.3 (3)	C4—C5—C6—N6	−174.09 (15)
C3A—C4A—C10A—C5A	−177.87 (18)	C4—C5—C6—C1	2.4 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1A—H1A···O21A	0.87 (2)	2.31 (2)	2.690 (2)	106.3 (18)
N1A—H1A···O21B	0.87 (2)	1.94 (2)	2.761 (2)	155 (2)
N1B—H1B···O13	0.85 (2)	2.23 (2)	2.8643 (19)	132 (2)
N1B—H1B···O21A	0.85 (2)	2.14 (3)	2.746 (2)	128 (2)
N1B—H1B···O21B	0.85 (2)	2.30 (3)	2.683 (2)	108 (2)
O22A—H22A···O22B^{i}	0.97 (2)	1.50 (2)	2.4711 (19)	179 (3)
C4B—H4B···O41^{ii}	0.95	2.37	3.240 (2)	152
C5B—H5B···O12^{iii}	0.95	2.44	3.339 (2)	158
C8A—H8A···O21B	0.95	2.39	3.122 (2)	134

Symmetry codes: (i) *x*−1, *y*, *z*; (ii) *x*, *y*, *z*+1; (iii) −*x*+1, −*y*+2, −*z*+1.

Fig. 1


O62
N6
O61
O42
N4
O41
C6
C5
C4
C1
C3
C2
O12
O11
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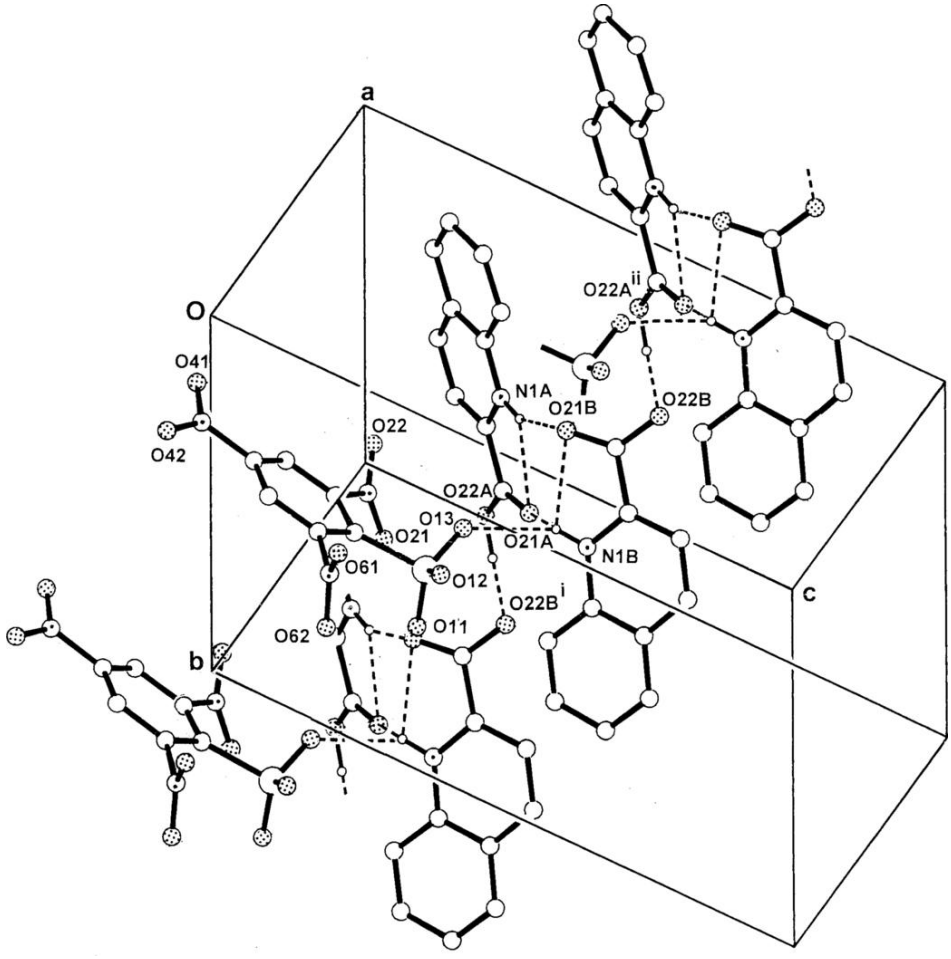
O21
N2
O22
O13
C6B
C7B
C8B
C5B
C9B
C10B
N1B
C4B
C2B
C3B
C21B
O22B
O22A
O21A
C21A
C2A
C3A
C4A
N1A
C9A
C10A
C5A
C8A
C7A
C6A
O21B

Fig. 2


a
O
O41
O42
O22
O22A^{ii}
N1A
O21B
O22B
O22A
O13
O21
O21A
N1B
O61
O12
O22B^{i}
O62
O11
b
c