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1-Hydroxyethyl-2-methyl-5-nitroimidazolium 3-carboxy-4-hydroxybenzenesulfonate

Bo Yang

Wuhan Grand Pharmaceutical Group Co. Ltd, No. 5 Gu Tian Road, Wuhan 430035, People's Republic of China
Correspondence e-mail: b_yang58@126.com

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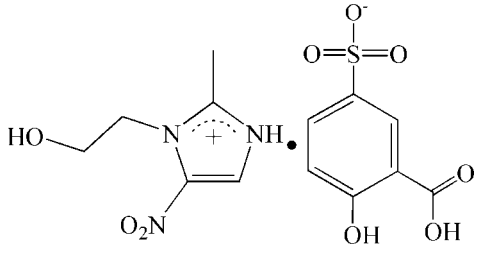
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.003 Å; disorder in main residue; R factor = 0.042; wR factor = 0.119; data-to-parameter ratio = 13.4.

Cocrystallization of 1-hydroxyethyl-2-methyl-5-nitroimidazole (metronidazole) and 5-sulfosalicylic acid (5-H_2SSA) from methanol solution yields the title salt, $C_6H_{10}N_3O_3^+\cdot C_7H_5O_6S^-$. In the crystal structure, the ions are linked by a combination of intermolecular O—H···O, N—H···O and C—H···O hydrogen bonds, forming a three-dimensional framework. The hydroxyl group of the cation is disordered over two sites in a 0.860 (4):0.140 (4) ratio.

Related literature

For related literature, see: Athar *et al.* (2005); Bharti *et al.* (2002); Castelli *et al.* (2000); Cohen-Jonathan *et al.* (2001); Crozet *et al.* (2002); Galván-Tejada *et al.* (2002); Hodgkiss (1998); Kennedy *et al.* (2006); Meng *et al.* (2007); Skupin *et al.* (1997); Wu *et al.* (2003).



Experimental

Crystal data

$C_6H_{10}N_3O_3^+\cdot C_7H_5O_6S^-$
M_r = 389.34
Monoclinic, $P2_1/n$
a = 8.8438 (3) Å
b = 13.0249 (4) Å
c = 14.148 (5) Å
β = 100.413 (1)°
V = 1602.9 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.26 mm^{-1}
T = 294 (2) K
0.35 × 0.26 × 0.20 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)
T_{min} = 0.904, T_{max} = 0.950
17399 measured reflections
3503 independent reflections
3156 reflections with $I > 2\sigma(I)$
R_{int} = 0.020

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.118
S = 1.06
3503 reflections
262 parameters
4 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3A···O2	0.83 (3)	1.86 (3)	2.618 (2)	151 (3)
N2—H2···O6	0.81 (2)	1.97 (2)	2.7557 (19)	163 (2)
C13—H13D···O7^{i}	0.97	2.46	3.280 (3)	142
C11—H11C···O4^{ii}	0.96	2.55	3.457 (3)	157
O9—H9A···O5^{iii}	0.82 (1)	2.129 (13)	2.940 (2)	170 (4)
O9′—H9′···O2^{iv}	0.82 (1)	2.258 (13)	2.830 (2)	127 (2)
O1—H1···O4^{v}	0.86 (3)	1.73 (3)	2.5801 (19)	171 (3)

Symmetry codes: (i) $-x, -y, -z+1$; (ii) $-x+\frac{3}{2}, y-\frac{1}{2}, -z+\frac{3}{2}$; (iii) $x-\frac{1}{2}, -y+\frac{1}{2}, z-\frac{1}{2}$; (iv) $-x, -y+1, -z+1$; (v) $x-1, y, z$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2643).

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supplementary materials

Acta Cryst. (2008). E**64**, o1338-o1339 [**doi:10.1107/S1600536808018643**]

1-Hydroxyethyl-2-methyl-5-nitroimidazolium 3-carboxy-4-hydroxybenzenesulfonate

B. Yang

Comment

1-Hydroxyethyl-2-methyl-5-nitrimidazole (metronidazole) is often used in the treatment of anaerobic protozoan and bacterial infections (Castelli *et al.*, 2000; Cohen-Jonathan *et al.*, 2001; Hodgkiss *et al.*, 1998). However, the low solubility in water makes its absorption in human body much less than expected. Recently, many efforts have been devoted to developing some new substitutes for the medicine, *i.e.* i) metal-organic coordination compounds (Kennedy *et al.*, 2006; Galván-Tejada *et al.*, 2002; Athar *et al.*, 2005; Bharti *et al.*, 2002; Wu *et al.*, 2003), ii) organic substitute derivatives (Crozet *et al.*, 2002, Skupin *et al.*, 1997) and iii) pharmaceutical co-crystals. In this paper, we report the 1:1 molecular adduct formed by metronidazole 5-sulfosalicylic acid (5-H_2SSA), (I).

In (I), the H atom is transferred from the sulfonic acid group to the imidazole N atom (Fig.1) forming an 1:1 organic adduct, which is similar to the analogous organic adducts reported (Meng *et al.*, 2007). The hydroxyl O atom is disordered at two sites with occupancy being 0.86 (1)/0.14 (1) for the major and minor components, respectively.

In the crystal packing, the component ions are linked by a combination of O—H···O, N—H···O and C—H···O hydrogen bonds (Table 1), forming a three-dimensional network (Fig.2). There are no other interactions (*e.g.* C—H···π and π-π) observed in the crystal structure by using *PLATON* (Spek, 2003).

Experimental

All the reagents and solvents were used as obtained without further purification. Equivalent molar amount of metronidazole and 5-sulfosalicylic acid dihydrate were dissolved in methanol (10 ml). The mixture was stirred for ten minutes at 300 K and then filtered. Block colorless crystals of (I) suitable for single-crystal X-ray diffraction analysis were grown by slow evaporation of the solution at the bottom of the vessel in two days.

Refinement

H atoms bonded to C atoms were positioned geometrically [C–H = 0.93Å (aromatic), 0.97(methylene) and 0.96(methyl)] and refined in riding modes [U_{iso}(H) = 1.2U_{eq}(aromatic and methylene C) and 1.5U~eq~(methyl C]. H atoms bonded to N and O atoms were found in Fourier difference maps with the constraints of N—H = 0.86 (2) Å,*O*—H = 0.82 (2) Å, and U_{iso}(H) = 1.2U_{eq}(N) or 1.5U_{eq}(O)]. The hydroxyl O atom is disordered at two sites with the occupancy being 0.86 (1):0.14 (1) for the major and minor components, respectively.

Figures

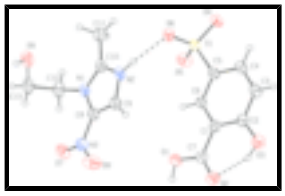

Fig. 1. Molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H-bonds are shown in dashed lines.

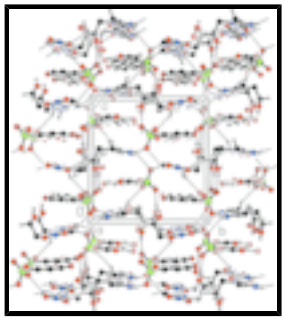

Fig. 2. Part of the crystal structure of (I), showing the formation of the three-dimensional framework structure. Hydrogen bonds are shown as dashed lines. For the sake of clarity, H atoms not involved in the motif have been omitted from the drawing.

1-Hydroxyethyl-2-methyl-5-nitroimidazolium 3-carboxy-4-hydroxybenzenesulfonate

Crystal data

$C_6H_{10}N_3O_3^+ \cdot C_7H_5O_6S^-$	F_{000} = 808
M_r = 389.34	D_x = 1.613 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 9747 reflections
a = 8.8438 (3) Å	θ = 2.5–28.2º
b = 13.0249 (4) Å	μ = 0.26 mm^{-1}
c = 14.148 (5) Å	T = 294 (2) K
β = 100.4130 (10)º	Block, colorless
V = 1602.9 (6) Å^3	0.35 × 0.26 × 0.20 mm
Z = 4	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	3503 independent reflections
Radiation source: fine focus sealed Siemens Mo tube	3156 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.020
T = 294(2) K	θ_{max} = 27.0º
0.3° wide ω exposures scans	θ_{min} = 2.1º
Absorption correction: multi-scan (SADABS; Sheldrick, 1997)	h = −11→11
T_{min} = 0.905, T_{max} = 0.950	k = −16→16
17399 measured reflections	l = −18→18

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.042$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.118$	$w = 1/[\sigma^2(F_o^2) + (0.0695P)^2 + 0.4716P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.07$	$(\Delta/\sigma)_{max} < 0.001$
3503 reflections	$\Delta\rho_{max} = 0.32$ e Å^{-3}
262 parameters	$\Delta\rho_{min} = -0.30$ e Å^{-3}
4 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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}	Occ. (<1)
C1	0.26714 (18)	0.68882 (13)	0.68052 (11)	0.0389 (3)	
C2	0.3234 (2)	0.78986 (13)	0.68903 (12)	0.0441 (4)	
C3	0.4814 (2)	0.80629 (14)	0.69525 (14)	0.0520 (4)	
H3	0.5198	0.8729	0.7007	0.062*	
C4	0.5807 (2)	0.72533 (14)	0.69348 (13)	0.0462 (4)	
H4	0.6851	0.7376	0.6965	0.055*	
C5	0.52498 (18)	0.62468 (13)	0.68713 (11)	0.0380 (3)	
C6	0.36914 (18)	0.60747 (13)	0.68103 (11)	0.0380 (3)	
H6	0.3320	0.5405	0.6772	0.046*	
C7	0.1012 (2)	0.67003 (14)	0.67435 (13)	0.0433 (4)	
C8	0.1649 (2)	0.27776 (14)	0.56001 (12)	0.0443 (4)	
C9	0.2283 (2)	0.36913 (14)	0.54592 (13)	0.0474 (4)	
H9	0.1782	0.4318	0.5342	0.057*	
C10	0.41167 (19)	0.25196 (13)	0.56990 (11)	0.0405 (4)	
C11	0.5662 (2)	0.20700 (17)	0.58081 (16)	0.0591 (5)	
H11A	0.6376	0.2584	0.5680	0.089*	

H11B	0.5650	0.1512	0.5363	0.089*	
H11C	0.5969	0.1820	0.6452	0.089*	
C12	0.2655 (3)	0.09157 (14)	0.58498 (13)	0.0546 (5)	
H12A	0.1935	0.0764	0.6273	0.065*	
H12B	0.3647	0.0633	0.6137	0.065*	
C13	0.2110 (3)	0.04186 (16)	0.48829 (16)	0.0625 (6)	
H13A	0.2000	−0.0315	0.4965	0.075*	0.860 (4)
H13B	0.1113	0.0694	0.4597	0.075*	0.860 (4)
H13C	0.2960	0.0404	0.4535	0.075*	0.140 (4)
H13D	0.1834	−0.0288	0.4987	0.075*	0.140 (4)
N1	0.27916 (17)	0.20437 (10)	0.57531 (10)	0.0415 (3)	
N2	0.38028 (18)	0.35061 (12)	0.55237 (11)	0.0441 (3)	
H2	0.447 (3)	0.3933 (19)	0.5509 (15)	0.055 (6)*	
N3	0.0052 (2)	0.26065 (16)	0.56153 (12)	0.0582 (4)	
O1	0.06220 (15)	0.57238 (10)	0.66817 (11)	0.0551 (4)	
H1	−0.029 (3)	0.567 (2)	0.6806 (19)	0.083*	
O2	0.00803 (16)	0.73868 (11)	0.67490 (13)	0.0644 (4)	
O3	0.23266 (19)	0.87220 (10)	0.69337 (12)	0.0612 (4)	
H3A	0.144 (4)	0.849 (3)	0.688 (2)	0.092*	
O4	0.80111 (14)	0.55223 (12)	0.72322 (11)	0.0577 (4)	
O5	0.59335 (15)	0.44064 (11)	0.74685 (10)	0.0552 (4)	
O6	0.62653 (14)	0.48356 (9)	0.58626 (9)	0.0447 (3)	
O7	−0.0358 (2)	0.17473 (15)	0.58045 (15)	0.0874 (6)	
O8	−0.0796 (2)	0.33520 (18)	0.54514 (15)	0.0865 (6)	
O9	0.3138 (2)	0.06004 (17)	0.42931 (13)	0.0725 (7)	0.860 (4)
H9A	0.260 (4)	0.065 (3)	0.3759 (14)	0.109*	0.860 (4)
O9'	0.1003 (12)	0.0853 (9)	0.4339 (7)	0.066 (4)	0.140 (4)
H9'	0.13 (3)	0.135 (12)	0.408 (15)	0.099*	0.140 (4)
S1	0.64580 (4)	0.51687 (3)	0.68609 (3)	0.03782 (14)	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0401 (8)	0.0393 (8)	0.0371 (8)	−0.0030 (7)	0.0064 (6)	−0.0011 (6)
C2	0.0514 (10)	0.0369 (8)	0.0437 (9)	−0.0007 (7)	0.0078 (7)	0.0033 (7)
C3	0.0565 (11)	0.0377 (9)	0.0610 (11)	−0.0127 (8)	0.0084 (9)	0.0039 (8)
C4	0.0425 (9)	0.0445 (9)	0.0514 (10)	−0.0126 (7)	0.0077 (7)	0.0038 (7)
C5	0.0367 (8)	0.0395 (8)	0.0381 (8)	−0.0048 (6)	0.0070 (6)	0.0012 (6)
C6	0.0380 (8)	0.0358 (8)	0.0404 (8)	−0.0065 (6)	0.0075 (6)	−0.0027 (6)
C7	0.0412 (8)	0.0417 (9)	0.0472 (9)	0.0003 (7)	0.0088 (7)	−0.0034 (7)
C8	0.0451 (9)	0.0486 (10)	0.0391 (8)	−0.0103 (7)	0.0073 (7)	−0.0059 (7)
C9	0.0491 (9)	0.0401 (9)	0.0517 (10)	−0.0048 (7)	0.0058 (8)	−0.0027 (7)
C10	0.0464 (9)	0.0394 (8)	0.0343 (8)	−0.0087 (7)	0.0036 (6)	−0.0016 (6)
C11	0.0510 (10)	0.0617 (12)	0.0620 (12)	0.0035 (9)	0.0035 (9)	0.0017 (10)
C12	0.0836 (14)	0.0359 (9)	0.0447 (9)	−0.0151 (9)	0.0128 (9)	0.0021 (7)
C13	0.0888 (16)	0.0447 (10)	0.0561 (12)	−0.0255 (11)	0.0188 (11)	−0.0107 (9)
N1	0.0520 (8)	0.0365 (7)	0.0351 (7)	−0.0120 (6)	0.0060 (6)	−0.0017 (5)
N2	0.0465 (8)	0.0371 (7)	0.0480 (8)	−0.0133 (6)	0.0066 (6)	−0.0014 (6)

N3	0.0501 (9)	0.0760 (12)	0.0500 (9)	−0.0176 (9)	0.0134 (7)	−0.0135 (8)
O1	0.0359 (6)	0.0428 (7)	0.0875 (10)	−0.0024 (5)	0.0135 (6)	−0.0028 (6)
O2	0.0482 (7)	0.0479 (8)	0.0985 (12)	0.0063 (6)	0.0172 (7)	−0.0104 (7)
O3	0.0614 (8)	0.0360 (7)	0.0852 (11)	0.0023 (6)	0.0104 (8)	0.0021 (7)
O4	0.0333 (6)	0.0637 (9)	0.0728 (9)	−0.0072 (6)	0.0014 (6)	−0.0126 (7)
O5	0.0534 (7)	0.0544 (8)	0.0578 (8)	0.0007 (6)	0.0105 (6)	0.0214 (6)
O6	0.0486 (7)	0.0396 (6)	0.0456 (7)	−0.0045 (5)	0.0079 (5)	−0.0006 (5)
O7	0.0759 (11)	0.0821 (12)	0.1140 (15)	−0.0409 (10)	0.0429 (10)	−0.0266 (11)
O8	0.0517 (9)	0.1110 (16)	0.0978 (13)	0.0069 (10)	0.0161 (9)	0.0071 (11)
O9	0.0805 (13)	0.0892 (14)	0.0503 (10)	−0.0317 (11)	0.0186 (9)	−0.0189 (9)
O9'	0.082 (8)	0.066 (7)	0.047 (6)	0.001 (6)	0.006 (5)	−0.013 (5)
S1	0.0307 (2)	0.0400 (2)	0.0417 (2)	−0.00536 (14)	0.00351 (15)	0.00333 (15)

Geometric parameters (Å, °)

C1—C6	1.391 (2)	C11—H11B	0.9600
C1—C2	1.405 (2)	C11—H11C	0.9600
C1—C7	1.475 (2)	C12—N1	1.482 (2)
C2—O3	1.348 (2)	C12—C13	1.512 (3)
C2—C3	1.400 (3)	C12—H12A	0.9700
C3—C4	1.376 (3)	C12—H12B	0.9700
C3—H3	0.9300	C13—O9'	1.265 (8)
C4—C5	1.398 (2)	C13—O9	1.361 (3)
C4—H4	0.9300	C13—H13A	0.9700
C5—C6	1.384 (2)	C13—H13B	0.9700
C5—S1	1.7662 (17)	C13—H13C	0.9700
C6—H6	0.9300	C13—H13D	0.9700
C7—O2	1.217 (2)	N2—H2	0.81 (2)
C7—O1	1.317 (2)	N3—O7	1.221 (3)
C8—C9	1.346 (2)	N3—O8	1.223 (3)
C8—N1	1.379 (2)	O1—H1	0.86 (3)
C8—N3	1.434 (2)	O3—H3A	0.83 (3)
C9—N2	1.352 (2)	O4—S1	1.4539 (12)
C9—H9	0.9300	O5—S1	1.4437 (13)
C10—N2	1.328 (2)	O6—S1	1.4579 (14)
C10—N1	1.340 (2)	O9—H13C	0.4764
C10—C11	1.469 (3)	O9—H9A	0.82 (1)
C11—H11A	0.9600	O9'—H9'	0.82 (1)
C6—C1—C2	119.60 (15)	C13—C12—H12B	109.4
C6—C1—C7	120.76 (15)	H12A—C12—H12B	108.0
C2—C1—C7	119.61 (15)	O9'—C13—O9	94.5 (6)
O3—C2—C3	118.11 (16)	O9'—C13—C12	116.4 (5)
O3—C2—C1	123.01 (16)	O9—C13—C12	109.93 (18)
C3—C2—C1	118.87 (16)	O9'—C13—H13A	115.5
C4—C3—C2	120.97 (16)	O9—C13—H13A	109.7
C4—C3—H3	119.5	C12—C13—H13A	109.7
C2—C3—H3	119.5	O9—C13—H13B	109.7
C3—C4—C5	120.09 (16)	C12—C13—H13B	109.7
C3—C4—H4	120.0	H13A—C13—H13B	108.2

C5—C4—H4	120.0	O9'—C13—H13C	106.6
C6—C5—C4	119.46 (16)	C12—C13—H13C	108.5
C6—C5—S1	117.90 (12)	H13A—C13—H13C	98.3
C4—C5—S1	122.63 (13)	H13B—C13—H13C	121.6
C5—C6—C1	120.98 (15)	O9'—C13—H13D	109.2
C5—C6—H6	119.5	O9—C13—H13D	118.2
C1—C6—H6	119.5	C12—C13—H13D	108.4
O2—C7—O1	122.75 (17)	H13B—C13—H13D	100.4
O2—C7—C1	123.05 (17)	H13C—C13—H13D	107.4
O1—C7—C1	114.20 (15)	C10—N1—C8	107.09 (14)
C9—C8—N1	108.85 (15)	C10—N1—C12	123.38 (16)
C9—C8—N3	125.28 (18)	C8—N1—C12	129.19 (16)
N1—C8—N3	125.85 (17)	C10—N2—C9	110.91 (15)
C8—C9—N2	105.54 (16)	C10—N2—H2	122.7 (16)
C8—C9—H9	127.2	C9—N2—H2	126.1 (16)
N2—C9—H9	127.2	O7—N3—O8	124.99 (19)
N2—C10—N1	107.62 (16)	O7—N3—C8	118.5 (2)
N2—C10—C11	124.35 (17)	O8—N3—C8	116.46 (19)
N1—C10—C11	128.03 (17)	C7—O1—H1	108 (2)
C10—C11—H11A	109.5	C2—O3—H3A	105 (2)
C10—C11—H11B	109.5	C13—O9—H9A	104 (3)
H11A—C11—H11B	109.5	H13C—O9—H9A	119.2
C10—C11—H11C	109.5	C13—O9'—H9'	109 (10)
H11A—C11—H11C	109.5	O5—S1—O4	112.76 (9)
H11B—C11—H11C	109.5	O5—S1—O6	112.23 (8)
N1—C12—C13	111.03 (16)	O4—S1—O6	112.44 (8)
N1—C12—H12A	109.4	O5—S1—C5	106.37 (8)
C13—C12—H12A	109.4	O4—S1—C5	106.18 (8)
N1—C12—H12B	109.4	O6—S1—C5	106.24 (7)
C6—C1—C2—O3	−176.99 (16)	C11—C10—N1—C8	−179.79 (17)
C7—C1—C2—O3	1.0 (3)	N2—C10—N1—C12	174.03 (15)
C6—C1—C2—C3	1.7 (2)	C11—C10—N1—C12	−6.0 (3)
C7—C1—C2—C3	179.71 (16)	C9—C8—N1—C10	−0.29 (19)
O3—C2—C3—C4	178.52 (17)	N3—C8—N1—C10	−178.63 (16)
C1—C2—C3—C4	−0.2 (3)	C9—C8—N1—C12	−173.61 (17)
C2—C3—C4—C5	−1.2 (3)	N3—C8—N1—C12	8.0 (3)
C3—C4—C5—C6	1.1 (3)	C13—C12—N1—C10	−96.5 (2)
C3—C4—C5—S1	−179.15 (14)	C13—C12—N1—C8	75.8 (2)
C4—C5—C6—C1	0.4 (2)	N1—C10—N2—C9	−0.10 (19)
S1—C5—C6—C1	−179.38 (12)	C11—C10—N2—C9	179.92 (17)
C2—C1—C6—C5	−1.8 (2)	C8—C9—N2—C10	−0.1 (2)
C7—C1—C6—C5	−179.78 (15)	C9—C8—N3—O7	−175.94 (19)
C6—C1—C7—O2	179.04 (18)	N1—C8—N3—O7	2.1 (3)
C2—C1—C7—O2	1.1 (3)	C9—C8—N3—O8	2.8 (3)
C6—C1—C7—O1	−1.0 (2)	N1—C8—N3—O8	−179.15 (17)
C2—C1—C7—O1	−178.93 (16)	C6—C5—S1—O5	−42.03 (15)
N1—C8—C9—N2	0.23 (19)	C4—C5—S1—O5	138.20 (15)
N3—C8—C9—N2	178.58 (16)	C6—C5—S1—O4	−162.37 (13)
N1—C12—C13—O9'	−44.9 (7)	C4—C5—S1—O4	17.86 (17)

N1—C12—C13—O9	61.0 (3)	C6—C5—S1—O6	77.73 (14)
N2—C10—N1—C8	0.23 (18)	C4—C5—S1—O6	−102.04 (15)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3A···O2	0.83 (3)	1.86 (3)	2.618 (2)	151 (3)
N2—H2···O6	0.81 (2)	1.97 (2)	2.7557 (19)	163 (2)
C13—H13D···O7^{i}	0.97	2.46	3.280 (3)	142
C11—H11C···O4^{ii}	0.96	2.55	3.457 (3)	157
O9—H9A···O5^{iii}	0.82 (1)	2.129 (13)	2.940 (2)	170 (4)
O9'—H9'···O2^{iv}	0.82 (1)	2.258 (13)	2.830 (2)	127 (2)
O1—H1···O4^{v}	0.86 (3)	1.73 (3)	2.5801 (19)	171 (3)

Symmetry codes: (i) −*x*, −*y*, −*z*+1; (ii) −*x*+3/2, *y*−1/2, −*z*+3/2; (iii) *x*−1/2, −*y*+1/2, *z*−1/2; (iv) −*x*, −*y*+1, −*z*+1; (v) *x*−1, *y*, *z*.

Fig. 1

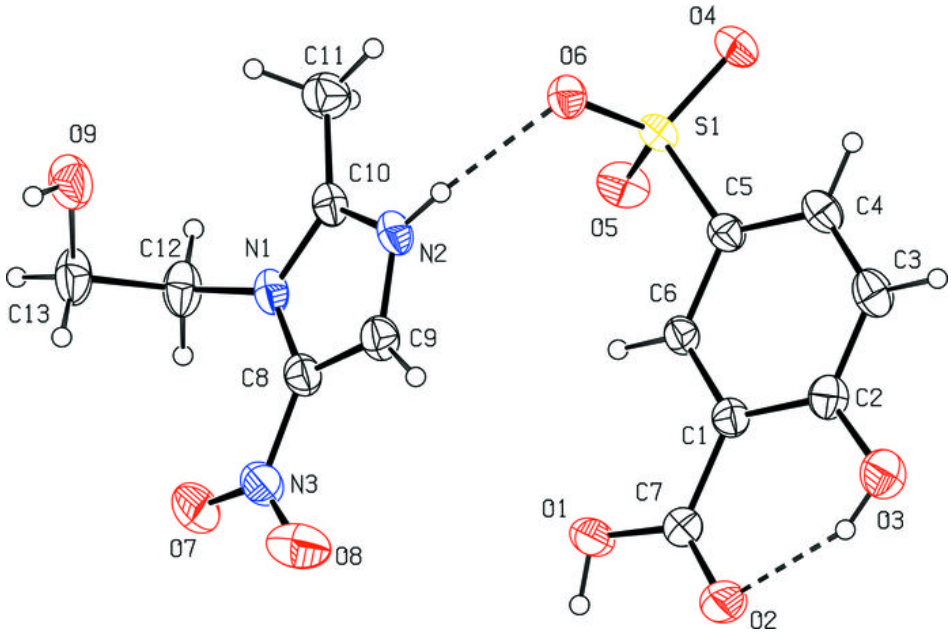
O4
C11
O6
S1
O9
C10
C5
C4
O5
C12
N1
N2
C3
C13
C6
C9
C8
C2
C1
N3
C7
O1
O3
O7
O8
O2

Fig. 2