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2-Hydroxypyridinium 2,4,6-trinitrophenolate

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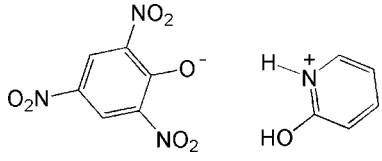
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.005 Å; R factor = 0.067; wR factor = 0.170; data-to-parameter ratio = 12.2.

The title proton-transfer compound, $C_5H_6NO^+\cdot C_6H_2N_3O_7^-$, consists of a 2,4,6-trinitrophenolate anion and a protonated 2-hydroxypyridinium cation. Intermolecular O—H···O and N—H···O hydrogen bonds link ions into dimers. The formation of hydrogen bonds with 2-pyridinol decreases the bitterness of 2,4,6-trinitrophenol.

Related literature

For related literature, see: Allen *et al.* (1987); Harrison *et al.* (2007); Hofmann (1999); In *et al.* (1997); Näther *et al.* (1997); Saminathan & Sivakumar (2007*a*,*b*); Shaw *et al.* (1984); Soriano-García *et al.* (1990); Suzuki *et al.* (2002, 2004).



Experimental

Crystal data

$C_5H_6NO^+\cdot C_6H_2N_3O_7^-$
M_r = 324.21
Monoclinic, $P2_1/c$
a = 12.360 (3) Å
b = 3.7323 (10) Å
c = 27.575 (7) Å
β = 93.128 (4)°
V = 1270.2 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.15 mm^{-1}
T = 298 (2) K
0.45 × 0.03 × 0.03 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.936, T_{max} = 0.996
9449 measured reflections
2633 independent reflections
1468 reflections with $I > 2\sigma(I)$
R_{int} = 0.081

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.067
$wR(F^2)$ = 0.170
S = 1.02
2633 reflections
215 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O3	0.86 (3)	2.45 (3)	2.937 (3)	117 (3)
O1—H1···O2	0.86 (3)	1.73 (2)	2.539 (3)	157 (4)
N1—H1*A*···O8	0.906 (10)	2.18 (3)	2.953 (4)	142 (3)
N1—H1*A*···O2	0.906 (10)	1.96 (3)	2.707 (4)	138 (3)

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1998); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: KP2123).

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supplementary materials

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2-Hydroxypyridinium 2,4,6-trinitrophenolate

Z.-L. You, W.-M. Dai and Y.-Q. Hu

Comment

Much effort has been made to decrease the bitterness of food and medicines (Suzuki *et al.*, 2002, 2004; Hofmann, 1999; Shaw *et al.*, 1984). 2,4,6-Trinitrophenol is a bitter compound; in order to investigate the influence of the hydrogen bonds on the bitterness of the compound, the title proton-transfer compound was synthesized and characterized.

The proton-transfer compound (I) (Fig. 1) consists of an unprotonated 2,4,6-trinitrophenolate anion and a protonated 2-hydroxypyridinium cation. The H atom of O2 is transferred to N1. The bond lengths and angles are within normal ranges (Allen *et al.*, 1987) and comparable with the values observed in other similar compounds (Saminathan & Sivakumar, 2007*a*,b; Näther *et al.*, 1997; In *et al.*, 1997; Harrison *et al.*, 2007; Soriano-García *et al.*, 1990). The dihedral angles between C6—C11 phenyl ring and N2/O3/O4, N3/O5/O6, N4/O7/O8 planes are 32.6 (3), 10.5 (3) and 17.6 (3)°, respectively. The C6—C11 phenyl ring and the N1/C1—C5 pyridine ring form dihedral angle of 10.3 (3)°. Intramolecular O—H···O and N—H···O hydrogen bonds connect molecules into dimers (Fig. 2, Table 1). Both protons of cation are involved into bifurcated hydrogen bonds. Each of them acts as a donor to two acceptors of anion (Table 2).

Experimental

All the reagents were of commercial grade and were used without further purification. 2,4,6-Trinitrophenol (0.1 mmol, 23.0 mg) and 2-pyridinol (0.1 mmol, 9.5 mg) were dissolved in MeOH/H_2O (10 ml, v:v = 1:1). The mixture was stirred at room temperature for 30 min to give a clear yellow solution. After keeping the solution in air for 10 d, yellow needle-shaped crystals were formed. Analysis found: C 40.62, H 2.54, N 17.37; calculated for $C_{11}H_8N_4O_8$: C 40.75, H 2.49, N 17.28%.

Refinement

Atoms H1 and H1A were located in a difference Fourier map and refined isotropically, with the O–H distance restrained to 0.85 (1) Å, N–H distance restrained to 0.90 (1) Å, and with U_{iso}(H) values fixed at 0.08 Å^2. The other H atoms were placed in idealized positions and constrained to ride on their parent atoms with C—H distances of 0.93 Å, and with U_{iso}(H) set to 1.2U_{eq}(C).

Figures

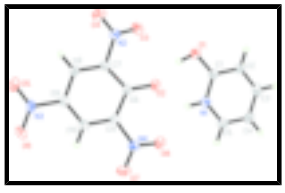

Fig. 1. The structure of (I) with the 30% probability displacement ellipsoids and the atom-numbering scheme.

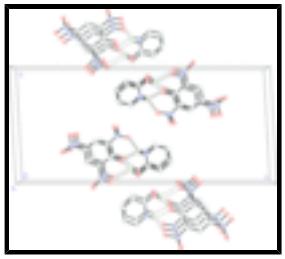

Fig. 2. Molecular packing of the compound, viewed along the *b* axis. Intramolecular hydrogen bonds are shown as dashed lines.

2-Hydroxypyridinium 2,4,6-trinitrophenolate

Crystal data

$C_5H_6NO^+·C_6H_2N_3O_7^-$	F_{000} = 664
M_r = 324.21	D_x = 1.695 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 12.360 (3) Å	Cell parameters from 718 reflections
b = 3.7323 (10) Å	θ = 2.2–24.4º
c = 27.575 (7) Å	μ = 0.15 mm^{-1}
β = 93.128 (4)º	T = 298 (2) K
V = 1270.2 (6) Å^3	Needle, yellow
Z = 4	0.45 × 0.03 × 0.03 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer	2633 independent reflections
Radiation source: fine-focus sealed tube	1468 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.081
T = 298(2) K	θ_{max} = 26.5º
ω scans	θ_{min} = 1.5º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −15→15
T_{min} = 0.936, T_{max} = 0.996	k = −4→4
9449 measured reflections	l = −34→34

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.067	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.170	$w = 1/[\sigma^2(F_o^2) + (0.0695P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.02	$(\Delta/\sigma)_{max} < 0.001$
2633 reflections	$\Delta\rho_{max}$ = 0.26 e Å^{-3}

215 parameters	$\Delta\rho_{min} = -0.28$ e Å^{-3}
2 restraints	Extinction correction: SHELXL97, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.012 (2)

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 $F^2 > 2\text{sigma}(F^2)$ 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}
O1	0.08847 (19)	0.7094 (8)	0.47182 (8)	0.0498 (7)
O2	0.19824 (18)	0.3631 (7)	0.41230 (8)	0.0510 (7)
O3	0.02968 (19)	0.7159 (7)	0.36716 (9)	0.0560 (8)
O4	−0.01971 (19)	0.4424 (8)	0.30070 (9)	0.0646 (9)
O5	0.2730 (2)	0.3254 (9)	0.19155 (9)	0.0718 (10)
O6	0.4134 (2)	0.0296 (8)	0.21813 (9)	0.0625 (9)
O7	0.4813 (2)	−0.1360 (8)	0.38769 (9)	0.0628 (8)
O8	0.3973 (2)	0.1652 (9)	0.43928 (9)	0.0698 (9)
N1	0.2547 (2)	0.5368 (8)	0.50557 (9)	0.0375 (7)
N2	0.0490 (2)	0.5260 (8)	0.33235 (10)	0.0401 (8)
N3	0.3295 (2)	0.1891 (9)	0.22435 (10)	0.0445 (8)
N4	0.4074 (2)	0.0611 (8)	0.39779 (10)	0.0396 (7)
C1	0.1554 (3)	0.6779 (9)	0.51021 (11)	0.0333 (8)
C2	0.1262 (3)	0.7854 (9)	0.55578 (11)	0.0385 (9)
H2	0.0580	0.8824	0.5599	0.046*
C3	0.1985 (3)	0.7474 (9)	0.59448 (12)	0.0450 (10)
H3	0.1792	0.8195	0.6251	0.054*
C4	0.3011 (3)	0.6021 (10)	0.58887 (12)	0.0451 (10)
H4	0.3503	0.5773	0.6154	0.054*
C5	0.3270 (3)	0.4985 (9)	0.54424 (12)	0.0426 (9)
H5	0.3948	0.3999	0.5398	0.051*
C6	0.2276 (2)	0.3217 (9)	0.36964 (11)	0.0316 (8)
C7	0.1595 (2)	0.3997 (9)	0.32664 (11)	0.0316 (8)
C8	0.1906 (2)	0.3560 (9)	0.28060 (11)	0.0332 (8)
H8	0.1437	0.4126	0.2542	0.040*
C9	0.2940 (3)	0.2251 (9)	0.27358 (11)	0.0338 (8)
C10	0.3629 (3)	0.1285 (9)	0.31213 (11)	0.0343 (8)
H10	0.4308	0.0340	0.3068	0.041*

C11	0.3303 (2)	0.1730 (9)	0.35853 (11)	0.0331 (8)
H1A	0.271 (3)	0.451 (11)	0.4762 (7)	0.080*
H1	0.123 (3)	0.645 (12)	0.4472 (9)	0.080*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0418 (15)	0.073 (2)	0.0338 (14)	0.0173 (13)	−0.0020 (11)	−0.0015 (14)
O2	0.0495 (15)	0.0755 (19)	0.0279 (13)	0.0175 (14)	0.0009 (11)	−0.0038 (13)
O3	0.0482 (16)	0.073 (2)	0.0474 (15)	0.0188 (14)	0.0067 (12)	−0.0105 (14)
O4	0.0418 (15)	0.098 (2)	0.0530 (17)	0.0125 (15)	−0.0116 (13)	−0.0109 (16)
O5	0.075 (2)	0.109 (3)	0.0312 (14)	0.0229 (18)	0.0043 (14)	0.0140 (16)
O6	0.0513 (17)	0.092 (2)	0.0461 (16)	0.0201 (16)	0.0166 (13)	−0.0088 (15)
O7	0.0494 (16)	0.077 (2)	0.0610 (18)	0.0301 (15)	−0.0064 (13)	−0.0058 (15)
O8	0.0608 (18)	0.115 (3)	0.0329 (15)	0.0300 (17)	−0.0056 (13)	−0.0064 (16)
N1	0.0374 (16)	0.0421 (19)	0.0334 (16)	0.0037 (14)	0.0048 (13)	0.0026 (14)
N2	0.0352 (17)	0.050 (2)	0.0350 (16)	0.0052 (15)	0.0000 (14)	0.0050 (15)
N3	0.0465 (19)	0.057 (2)	0.0307 (16)	−0.0029 (16)	0.0091 (14)	−0.0016 (15)
N4	0.0342 (16)	0.047 (2)	0.0372 (17)	0.0021 (15)	−0.0028 (13)	−0.0009 (15)
C1	0.0356 (19)	0.031 (2)	0.0332 (18)	−0.0004 (16)	0.0003 (15)	0.0027 (15)
C2	0.041 (2)	0.039 (2)	0.0355 (19)	0.0042 (16)	0.0064 (16)	−0.0029 (16)
C3	0.057 (2)	0.048 (2)	0.0309 (19)	−0.005 (2)	0.0072 (17)	−0.0098 (17)
C4	0.042 (2)	0.056 (3)	0.036 (2)	−0.0053 (19)	−0.0078 (16)	0.0041 (18)
C5	0.0341 (19)	0.045 (2)	0.048 (2)	0.0061 (17)	−0.0020 (17)	0.0097 (19)
C6	0.0357 (19)	0.0324 (19)	0.0270 (17)	−0.0027 (15)	0.0040 (14)	−0.0026 (15)
C7	0.0274 (17)	0.031 (2)	0.0364 (19)	0.0033 (14)	0.0047 (14)	0.0011 (15)
C8	0.0338 (19)	0.037 (2)	0.0289 (17)	−0.0019 (16)	−0.0016 (14)	0.0049 (15)
C9	0.040 (2)	0.036 (2)	0.0262 (17)	−0.0033 (16)	0.0063 (14)	−0.0026 (15)
C10	0.0310 (18)	0.033 (2)	0.0390 (19)	−0.0003 (15)	0.0078 (15)	−0.0016 (16)
C11	0.0335 (19)	0.035 (2)	0.0303 (17)	0.0011 (15)	−0.0020 (14)	0.0019 (16)

Geometric parameters (Å, °)

O1—C1	1.313 (4)	C2—C3	1.361 (4)
O1—H1	0.86 (3)	C2—H2	0.9300
O2—C6	1.260 (3)	C3—C4	1.395 (5)
O3—N2	1.227 (3)	C3—H3	0.9300
O4—N2	1.224 (3)	C4—C5	1.345 (4)
O5—N3	1.223 (4)	C4—H4	0.9300
O6—N3	1.216 (3)	C5—H5	0.9300
O7—N4	1.217 (3)	C6—C11	1.434 (4)
O8—N4	1.221 (3)	C6—C7	1.446 (4)
N1—C1	1.348 (4)	C7—C8	1.356 (4)
N1—C5	1.361 (4)	C8—C9	1.391 (4)
N1—H1A	0.906 (10)	C8—H8	0.9300
N2—C7	1.462 (4)	C9—C10	1.373 (4)
N3—C9	1.456 (4)	C10—C11	1.372 (4)
N4—C11	1.463 (4)	C10—H10	0.9300
C1—C2	1.385 (4)		

C1—O1—H1	107 (3)	C5—C4—H4	120.8
C1—N1—C5	121.9 (3)	C3—C4—H4	120.8
C1—N1—H1A	118 (3)	C4—C5—N1	120.5 (3)
C5—N1—H1A	119 (3)	C4—C5—H5	119.7
O4—N2—O3	123.2 (3)	N1—C5—H5	119.7
O4—N2—C7	117.2 (3)	O2—C6—C11	123.4 (3)
O3—N2—C7	119.6 (3)	O2—C6—C7	123.8 (3)
O6—N3—O5	123.8 (3)	C11—C6—C7	112.7 (3)
O6—N3—C9	118.7 (3)	C8—C7—C6	124.1 (3)
O5—N3—C9	117.5 (3)	C8—C7—N2	117.0 (3)
O7—N4—O8	121.5 (3)	C6—C7—N2	118.8 (3)
O7—N4—C11	118.0 (3)	C7—C8—C9	118.8 (3)
O8—N4—C11	120.4 (3)	C7—C8—H8	120.6
O1—C1—N1	119.7 (3)	C9—C8—H8	120.6
O1—C1—C2	121.4 (3)	C10—C9—C8	121.3 (3)
N1—C1—C2	118.9 (3)	C10—C9—N3	119.5 (3)
C3—C2—C1	119.3 (3)	C8—C9—N3	119.2 (3)
C3—C2—H2	120.4	C11—C10—C9	119.3 (3)
C1—C2—H2	120.4	C11—C10—H10	120.4
C2—C3—C4	121.0 (3)	C9—C10—H10	120.4
C2—C3—H3	119.5	C10—C11—C6	123.6 (3)
C4—C3—H3	119.5	C10—C11—N4	116.3 (3)
C5—C4—C3	118.4 (3)	C6—C11—N4	120.0 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O3	0.86 (3)	2.45 (3)	2.937 (3)	117 (3)
O1—H1···O2	0.86 (3)	1.73 (2)	2.539 (3)	157 (4)
N1—H1A···O8	0.906 (10)	2.18 (3)	2.953 (4)	142 (3)
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Fig. 1

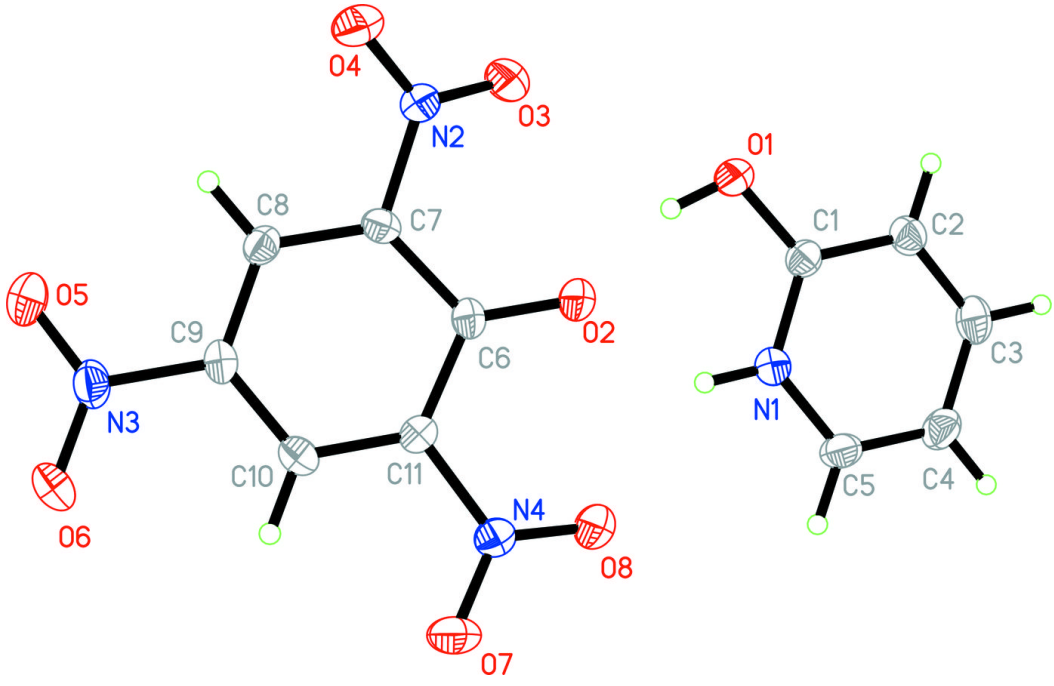

Fig. 2