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3,3-Dinitroazetidinium 2-hydroxybenzoate

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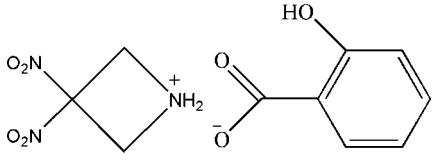
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.040; wR factor = 0.128; data-to-parameter ratio = 12.1.

In the title *gem*-dinitroazetidinium 2-hydroxybenzoate salt, $C_3H_6N_3O_4^+\cdot C_7H_5O_3^-$, the azetidine ring is virtually planar, with a mean deviation from the plane of 0.0242 Å. The dihedral angle between the two nitro groups is 87.5 (1)°.

Related literature

For related literature on 1,3,3-trinitroazetidine and compounds prepared from its derivative 3,3-dinitroazetidine, see: Archibald *et al.* (1990); Gao *et al.* (2009); Hiskey *et al.* (1992); Ma, Yan, Li, Guan *et al.* (2009); Ma, Yan, Li, Song & Hu (2009); Ma, Yan, Song *et al.* (2009); Ma *et al.* (2010); Yan *et al.* (2009, 2010).



Experimental

Crystal data

$C_3H_6N_3O_4^+\cdot C_7H_5O_3^-$
M_r = 285.22
Monoclinic, $P2_1/n$
a = 11.174 (3) Å
b = 7.013 (2) Å
c = 16.661 (5) Å
β = 105.960 (5)°
V = 1255.3 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.13 mm^{-1}
T = 296 K
0.36 × 0.26 × 0.19 mm

Data collection

Bruker APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2000)
T_{min} = 0.955, T_{max} = 0.976
6012 measured reflections
2222 independent reflections
1504 reflections with $I > 2\sigma(I)$
R_{int} = 0.027

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.128
S = 0.99
2222 reflections
183 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1C···O6	0.90	2.38	2.922 (2)	118
N1—H1C···O7	0.90	1.81	2.708 (2)	179
N1—H1D···O7^{i}	0.90	1.96	2.720 (2)	141

Symmetry code: (i) $-x+1, -y+2, -z$.

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); 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: NG5053).

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supporting information

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3,3-Dinitroazetidinium 2-hydroxybenzoate

Rong Gao, Biao Yan, Tao Mai, Ying Hu and Yu-Lei Guan

S1. Comment

Dinitro- and trinitro-derivatives of azetidine are of interest because they contain strained ring systems. This makes them good candidates for energetic materials (propellants or explosives). Azetidine-based explosives, such as 1,3,3-trinitroazetidine (TNAZ) (Archibald *et al.*, 1990) demonstrate excellent performance partly because of the high strain associated with the four-membered ring. As one of the important derivates of TNAZ, 3,3-dinitroazetidine (DNAZ) (Hiskey *et al.*, 1992;)can prepare a variety of solid energetic materials with high oxygen-balance (Ma, Yan, Li, Guan *et al.*, 2009; Gao *et al.*, 2009; Ma, Yan, Li, Song & Hu, 2009; Ma, Yan, Song *et al.*, 2009; Yan *et al.*, 2009; Yan *et al.*, 2010; Ma *et al.*, 2010). This paper reports synthesis and crystal structure of the title DNAZ salt.

S2. Experimental

A solution of DNAZ (0.30 g, 2.04 mmol), salicylic acid (0.28 ml, 2.04 mmol) in trichloromethane (15.0 ml) was stirred for 2 h. The reaction mixture was concentrated *in vacuo*, then a white solid began to precipitate. The solid product was washed with ethanol and purified by recrystallization from trichloromethane to give the pure colorless compound in 90.5% yield. The title compound (43 mg,0.15 mmol) was dissolved in chloroform (15 ml). Colorless crystals were isolated after several days. Elemental analysis calculated for $C_{10}H_{11}N_3O_7$: C 42.11, N 14.73, H 3.89%; found: C 47.44, N 14.80, H 3.89%. IR (KBr, cm^{-1}): 3060.25, 1647.33, 1579.29, 1298.39, 1485.78, 1454.23, 706.64.

S3. Refinement

H atoms were placed at calculated idealized positions and refined using a riding model, with C—H distances in the range 0.93–0.97 Å, N—H distances 0.90 Å and O—H distances 0.82 Å.

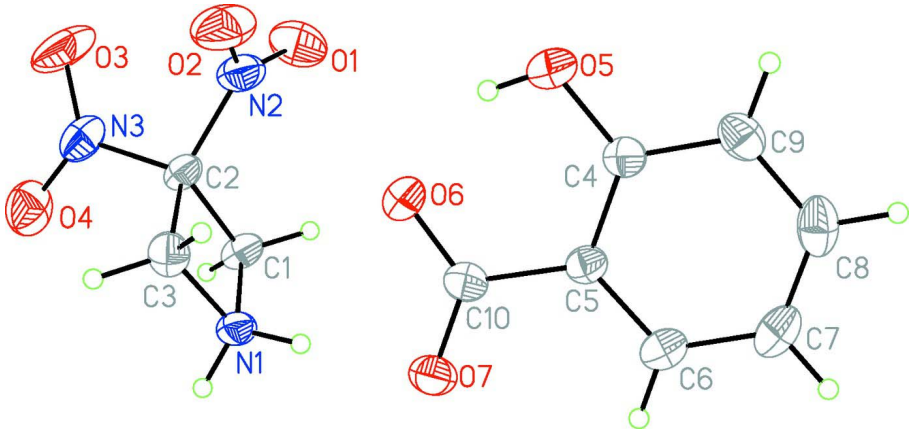

Figure 1

The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are drawn as spheres of arbitrary radius.

3,3-Dinitroazetidinium 2-hydroxybenzoate

Crystal data

$C_3H_6N_3O_4^+·C_7H_5O_3^-$
M_r = 285.22
Monoclinic, $P2_1/n$
Hall symbol: -P2yn
a = 11.174 (3) Å
b = 7.013 (2) Å
c = 16.661 (5) Å
β = 105.960 (5)°
V = 1255.3 (6) Å^3
Z = 4
$F(000)$ = 592
D_x = 1.509 Mg m^{-3}
Melting point: 379.4 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1275 reflections
θ = 2.5–21.9°
μ = 0.13 mm^{-1}
T = 296 K
Block, colorless
0.36 × 0.26 × 0.19 mm

Data collection

Bruker APEX CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2000)
T_{min} = 0.955, T_{max} = 0.976
6012 measured reflections
2222 independent reflections
1504 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 25.1°, θ_{min} = 2.0°
h = −12→13
k = −8→8
l = −18→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.128
S = 0.99
2222 reflections
183 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0797P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.008 (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 $F^2 > \sigma(F^2)$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O7	0.58576 (13)	0.7941 (2)	0.06388 (8)	0.0563 (4)
O6	0.46538 (12)	0.5959 (2)	0.10843 (9)	0.0629 (4)
O5	0.53637 (13)	0.2679 (2)	0.15624 (10)	0.0619 (4)
H5	0.4878	0.3566	0.1405	0.093*
C4	0.67334 (16)	0.4982 (3)	0.12215 (10)	0.0380 (5)
N1	0.36966 (14)	0.9772 (2)	0.05657 (9)	0.0447 (4)
H1D	0.3660	1.0893	0.0297	0.054*
H1C	0.4417	0.9169	0.0593	0.054*
C5	0.65122 (17)	0.3202 (3)	0.15274 (11)	0.0407 (5)
N3	0.10748 (16)	0.9419 (3)	0.10825 (12)	0.0580 (5)
C10	0.56810 (18)	0.6383 (3)	0.09534 (11)	0.0439 (5)
C3	0.34209 (17)	0.9937 (3)	0.13919 (11)	0.0471 (5)
H3B	0.3181	1.1211	0.1514	0.056*
H3A	0.4075	0.9437	0.1854	0.056*
C2	0.23289 (16)	0.8586 (3)	0.10855 (11)	0.0412 (5)
C6	0.7486 (2)	0.1923 (3)	0.18100 (11)	0.0511 (5)
H6	0.7340	0.0722	0.2000	0.061*
C1	0.25748 (18)	0.8552 (3)	0.02338 (11)	0.0479 (5)
H1A	0.2765	0.7293	0.0061	0.057*
H1B	0.1928	0.9160	−0.0201	0.057*
C9	0.79343 (18)	0.5449 (3)	0.12150 (12)	0.0501 (5)
H9	0.8089	0.6630	0.1010	0.060*
N2	0.24179 (17)	0.6717 (3)	0.15310 (13)	0.0585 (5)
O4	0.08623 (14)	1.0972 (3)	0.07553 (11)	0.0771 (5)
C7	0.8681 (2)	0.2457 (4)	0.18067 (12)	0.0595 (6)
H7	0.9340	0.1620	0.2011	0.071*
O3	0.03912 (17)	0.8537 (3)	0.13847 (15)	0.0995 (7)
C8	0.89002 (19)	0.4198 (4)	0.15066 (13)	0.0594 (6)
H8	0.9703	0.4534	0.1500	0.071*
O1	0.19839 (17)	0.5345 (3)	0.11130 (12)	0.0861 (6)
O2	0.29006 (19)	0.6747 (3)	0.22818 (11)	0.0891 (6)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O7	0.0650 (10)	0.0485 (9)	0.0622 (9)	0.0038 (7)	0.0288 (7)	0.0121 (7)
O6	0.0415 (8)	0.0663 (11)	0.0854 (11)	0.0040 (7)	0.0248 (7)	0.0167 (8)
O5	0.0492 (9)	0.0573 (10)	0.0826 (11)	−0.0109 (7)	0.0237 (8)	0.0107 (8)
C4	0.0390 (10)	0.0420 (11)	0.0340 (9)	−0.0047 (8)	0.0115 (7)	−0.0025 (8)
N1	0.0432 (9)	0.0451 (10)	0.0531 (9)	0.0044 (7)	0.0256 (7)	0.0105 (7)
C5	0.0418 (11)	0.0429 (11)	0.0363 (9)	−0.0046 (9)	0.0087 (8)	−0.0011 (8)
N3	0.0432 (10)	0.0605 (13)	0.0751 (12)	−0.0041 (9)	0.0245 (9)	−0.0147 (10)
C10	0.0500 (12)	0.0425 (12)	0.0400 (10)	−0.0027 (9)	0.0140 (9)	0.0011 (9)
C3	0.0407 (11)	0.0577 (13)	0.0467 (10)	−0.0116 (9)	0.0185 (8)	−0.0046 (9)
C2	0.0330 (9)	0.0492 (12)	0.0447 (10)	−0.0038 (8)	0.0162 (8)	−0.0006 (9)
C6	0.0633 (14)	0.0489 (12)	0.0395 (10)	0.0057 (10)	0.0116 (9)	0.0042 (9)
C1	0.0484 (11)	0.0557 (13)	0.0401 (10)	0.0045 (10)	0.0131 (8)	−0.0021 (9)
C9	0.0450 (11)	0.0535 (13)	0.0546 (12)	−0.0094 (10)	0.0183 (9)	−0.0029 (10)
N2	0.0551 (11)	0.0572 (13)	0.0725 (13)	−0.0050 (9)	0.0333 (10)	0.0088 (10)
O4	0.0646 (11)	0.0716 (12)	0.0967 (13)	0.0169 (9)	0.0250 (9)	−0.0032 (10)
C7	0.0537 (14)	0.0751 (17)	0.0443 (11)	0.0211 (12)	0.0041 (10)	−0.0059 (11)
O3	0.0672 (11)	0.0869 (14)	0.173 (2)	−0.0139 (10)	0.0806 (13)	−0.0085 (13)
C8	0.0367 (11)	0.0786 (17)	0.0633 (13)	−0.0060 (11)	0.0147 (10)	−0.0108 (12)
O1	0.0896 (13)	0.0543 (11)	0.1241 (16)	−0.0227 (9)	0.0458 (11)	−0.0050 (10)
O2	0.1054 (14)	0.1075 (15)	0.0614 (11)	0.0001 (11)	0.0347 (10)	0.0311 (10)

Geometric parameters (Å, º)

O7—C10	1.251 (2)	C3—H3B	0.9700
O6—C10	1.261 (2)	C3—H3A	0.9700
O5—C5	1.351 (2)	C2—N2	1.496 (3)
O5—H5	0.8200	C2—C1	1.518 (3)
C4—C9	1.384 (3)	C6—C7	1.388 (3)
C4—C5	1.396 (2)	C6—H6	0.9300
C4—C10	1.503 (3)	C1—H1A	0.9700
N1—C1	1.493 (2)	C1—H1B	0.9700
N1—C3	1.495 (2)	C9—C8	1.372 (3)
N1—H1D	0.9000	C9—H9	0.9300
N1—H1C	0.9000	N2—O1	1.208 (2)
C5—C6	1.389 (3)	N2—O2	1.219 (2)
N3—O3	1.196 (2)	C7—C8	1.367 (3)
N3—O4	1.212 (2)	C7—H7	0.9300
N3—C2	1.517 (2)	C8—H8	0.9300
C3—C2	1.518 (3)		
C5—O5—H5	109.5	N2—C2—C1	116.46 (16)
C9—C4—C5	118.95 (17)	N3—C2—C1	114.04 (15)
C9—C4—C10	121.59 (17)	N2—C2—C3	116.25 (16)
C5—C4—C10	119.37 (16)	N3—C2—C3	114.66 (16)
C1—N1—C3	91.18 (13)	C1—C2—C3	89.36 (14)

C1—N1—H1D	113.4	C7—C6—C5	119.3 (2)
C3—N1—H1D	113.4	C7—C6—H6	120.3
C1—N1—H1C	113.4	C5—C6—H6	120.3
C3—N1—H1C	113.4	N1—C1—C2	89.66 (13)
H1D—N1—H1C	110.7	N1—C1—H1A	113.7
O5—C5—C6	118.36 (17)	C2—C1—H1A	113.7
O5—C5—C4	121.62 (16)	N1—C1—H1B	113.7
C6—C5—C4	120.02 (17)	C2—C1—H1B	113.7
O3—N3—O4	125.8 (2)	H1A—C1—H1B	110.9
O3—N3—C2	119.7 (2)	C8—C9—C4	121.03 (19)
O4—N3—C2	114.47 (17)	C8—C9—H9	119.5
O7—C10—O6	122.29 (18)	C4—C9—H9	119.5
O7—C10—C4	119.66 (17)	O1—N2—O2	126.9 (2)
O6—C10—C4	118.00 (17)	O1—N2—C2	116.75 (19)
N1—C3—C2	89.56 (14)	O2—N2—C2	116.36 (19)
N1—C3—H3B	113.7	C8—C7—C6	120.8 (2)
C2—C3—H3B	113.7	C8—C7—H7	119.6
N1—C3—H3A	113.7	C6—C7—H7	119.6
C2—C3—H3A	113.7	C7—C8—C9	119.87 (19)
H3B—C3—H3A	111.0	C7—C8—H8	120.1
N2—C2—N3	105.92 (15)	C9—C8—H8	120.1

C9—C4—C5—O5	178.87 (17)	O5—C5—C6—C7	−177.77 (16)
C10—C4—C5—O5	2.3 (2)	C4—C5—C6—C7	1.8 (3)
C9—C4—C5—C6	−0.7 (3)	C3—N1—C1—C2	−3.71 (15)
C10—C4—C5—C6	−177.31 (16)	N2—C2—C1—N1	−115.59 (16)
C9—C4—C10—O7	7.2 (3)	N3—C2—C1—N1	120.51 (16)
C5—C4—C10—O7	−176.32 (16)	C3—C2—C1—N1	3.65 (14)
C9—C4—C10—O6	−170.31 (17)	C5—C4—C9—C8	−0.3 (3)
C5—C4—C10—O6	6.2 (2)	C10—C4—C9—C8	176.18 (17)
C1—N1—C3—C2	3.71 (14)	N3—C2—N2—O1	87.1 (2)
O3—N3—C2—N2	−1.8 (2)	C1—C2—N2—O1	−40.8 (2)
O4—N3—C2—N2	178.59 (17)	C3—C2—N2—O1	−144.22 (18)
O3—N3—C2—C1	127.6 (2)	N3—C2—N2—O2	−91.5 (2)
O4—N3—C2—C1	−52.0 (2)	C1—C2—N2—O2	140.53 (18)
O3—N3—C2—C3	−131.4 (2)	C3—C2—N2—O2	37.1 (2)
O4—N3—C2—C3	49.0 (2)	C5—C6—C7—C8	−1.9 (3)
N1—C3—C2—N2	115.79 (17)	C6—C7—C8—C9	0.9 (3)
N1—C3—C2—N3	−119.95 (17)	C4—C9—C8—C7	0.2 (3)
N1—C3—C2—C1	−3.65 (14)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*C*···O6	0.90	2.38	2.922 (2)	118

N1—H1*C*···O7	0.90	1.81	2.708 (2)	179
N1—H1*D*···O7^{i}	0.90	1.96	2.720 (2)	141

Symmetry code: (i) −*x*+1, −*y*+2, −*z*.