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# (*RS*)-Dimethylammonium 2-*sec*-butyl-4,6-dinitrophenolate

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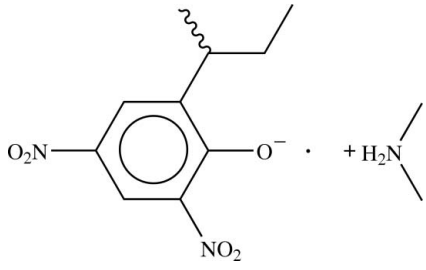
Key indicators: single-crystal X-ray study; $T$ = 105 K; mean $\sigma$(N–C) = 0.002 Å; disorder in main residue; $R$ factor = 0.034; $wR$ factor = 0.094; data-to-parameter ratio = 13.3.

The title compound, $C_2H_8N^+\cdot C_{10}H_{11}N_2O_5^-$, is a highly toxic herbicide known as dinoseb. The *sec*-butyl group is disordered [occupancy ratio 0.828 (3):0.172 (3)], while the nitro group in the 6 position is twisted by 25° with respect to the ring plane. Pairs of $-O^-\cdots H-N^+-H\cdots{}^-O-$ bridges between phenolic O atoms generate eight-membered hydrogen-bonded rings.

## Related literature

For toxicicity information, see: EXTOXNET (1996). Related structures have been described by Smith *et al.* (2002, 2005); Lynch & McClenaghan (2004); West-Nielsen *et al.* (2006).




## Experimental

### *Crystal data*

$C_2H_8N^+\cdot C_{10}H_{11}N_2O_5^-$
$M_r$ = 285.30
Monoclinic, $C2/c$
$a$ = 16.804 (4) Å
$b$ = 9.1446 (17) Å
$c$ = 19.223 (4) Å
$\beta$ = 104.555 (6)°
$V$ = 2859.2 (10) Å$^3$
$Z$ = 8
Mo $K\alpha$ radiation
$\mu$ = 0.10 mm$^{-1}$
$T$ = 105 K
0.70 × 0.09 × 0.05 mm

### *Data collection*

Siemens SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.832, $T_{max}$ = 0.995
10444 measured reflections
2909 independent reflections
2382 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.026

### *Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.094
$S$ = 1.05
2909 reflections
219 parameters
45 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.21 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.20 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1$B$—H1$B$···O1$A$ | 0.904 (16) | 1.914 (16) | 2.7585 (15) | 154.7 (14) |
| N1$B$—H2$B$···O1$A^{i}$ | 0.902 (17) | 1.868 (17) | 2.7173 (16) | 156.2 (15) |

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

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus* ; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL* software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PK2194).

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# supporting information

*Acta Cryst.* (2009). E**65**, o2630 [https://doi.org/10.1107/S1600536809038677]

## (*RS*)-Dimethylammonium 2-*sec*-butyl-4,6-dinitrophenolate

**Carl Henrik Görbitz**

### S1. Comment

Large amounts of a highly volatile yellow powder of unknown origin was discovered below the floor planks in the attic of a private home, and a small quantity was forwarded to the University of Oslo for analysis. Single crystals were easily obtained, and subsequent structure determination by X-ray diffraction identified the sample as a dimethylammonium salt of 2-*sec*-butyl-4,6-dinitrophenol, with common name dinoseb (I). This herbicide has been used in soybeans, vegetables, fruits and nuts, citrus, and other field crops for the selective control of grass and broadleaf weeds (*e.g.*, in corn). It was also used as an insecticide in grapes and as a seed crop drying agent. Product names for pesticides containing dinoseb include Basanite, Caldon, Chemox, Chemsect DNBP, Dinitro, Dynamyte, Elgetol, Gebutox, Hel-Fire, Kiloseb, Nitropone, Premerge, Sinox General, Subitex, and Vertac Weed Killer. The use of dinoseb was prohibited in the U.S. in 1986, an action based on the potential risk of birth defects and other adverse health effects (EXTOXNET, 1996).

2-*sec*-butyl-4,6-dinitrophenol has a chiral C-atom at C7, and the title salt is racemic, space group *C*2/c. For simplicity, the *R*-form alone is shown in Fig. 1, but there is actually disorder at each phenolate position giving a 0.828 (3):0.172 (3) distribution between the *R*-form and the S-form, as in Fig. 2, or *vice versa*. Both ammonium H-atoms form strong hydrogen bonds to charged phenolate O atom acceptors, one interaction is visible in Fig. 1.

Related structures include achiral analogues with *tert*-butyl (Lynch & McClenaghan, 2004) and acetyl (= 2-hydroxy-3,5-dinitroacetophenone; West-Nielsen *et al.*, 2006) substituents rather than *sec*-butyl at C2 (both obtained as neutral molecules) as well as a series of proton-transfer complexes of 3,5-dinitrosalicylic acid with aliphatic amines (Smith *et al.*, 2002; Smith *et al.*, 2005).

### S2. Experimental

Crystals in the shape of fine, yellow needles were grown by slow evaporation of a diethylether solution of the title complex.

### S3. Refinement

Normal anisotropic refinement except for atoms with low occupancy (minor disorder component), which were either refined isotropically (*sec*-butyl group) or constrained to have the same thermal parameters as the near-by atom of the major disorder component (C1, C2, C3). The geometries of the minor and the major component, in terms of covalent bond lengths and bond angles, were restrained to be more or less similar by a *SHELXTL* SAME command. Positional parameters were refined for the two ammonium H atoms involved in hydrogen bonding, other H atoms were positioned with idealized geometry and C–H distances fixed in the range 0.95 to 0.99 Å. $U_{iso}$ values were $1.2U_{eq}$ of the carrier atom, or $1.5U_{eq}$ for the methyl groups.

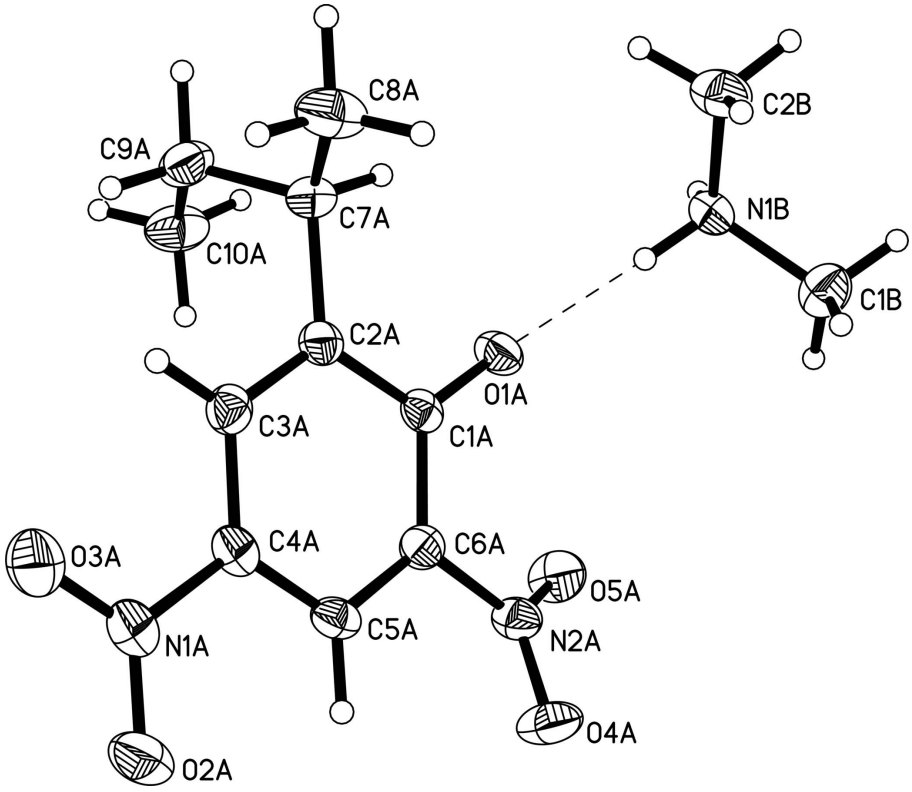


**Figure 1**

The molecular structure of (I) (major disorder component). Displacement ellipsoids are shown at the 50% probability level and H-atoms are shown as spheres of arbitrary size.

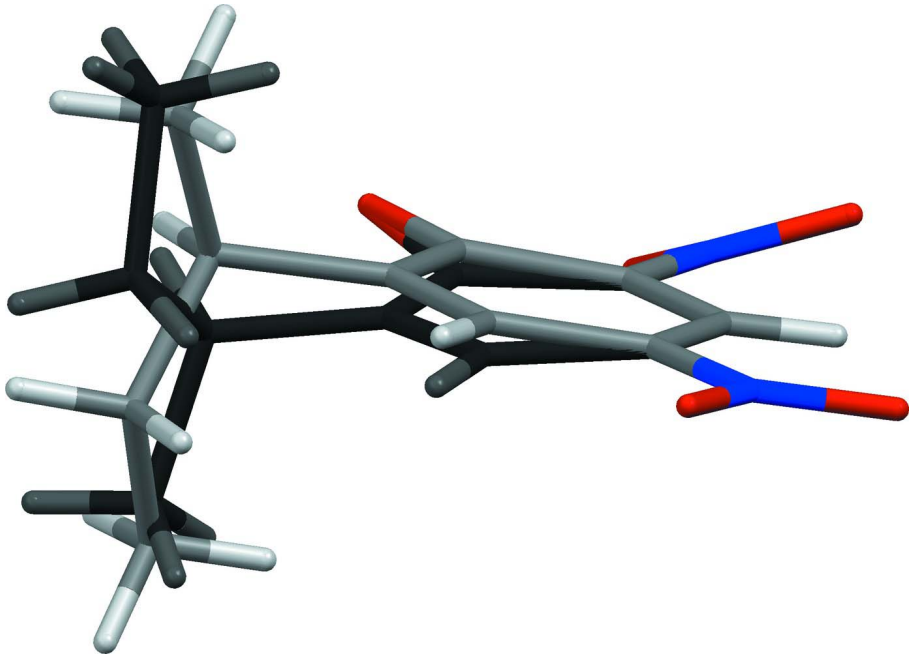

**Figure 2**

Disorder for the *sec*-butyl group and neighbouring atoms. Normal, light colour is used for the major component with *R*-configuration at C7, the minor component with *S*-configuration appears in darker colour.

### (*RS*)-Dimethylammonium 2-*sec*-butyl-4,6-dinitrophenolate

*Crystal data*

$C_2H_8N^+\cdot C_{10}H_{11}N_2O_5^-$
$M_r = 285.30$
Monoclinic, *C*2/*c*
$a = 16.804\ (4)$ Å
$b = 9.1446\ (17)$ Å
$c = 19.223\ (4)$ Å
$\beta = 104.555\ (6)°$
$V = 2859.2\ (10)$ Å$^3$
$Z = 8$
$F(000) = 1216$
$D_x = 1.326$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4501 reflections
$\theta = 2.2–26.4°$
$\mu = 0.10$ mm$^{-1}$
$T = 105$ K
Needle, yellow
0.70 × 0.09 × 0.05 mm

*Data collection*

Siemens SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 8.3 pixels mm$^{-1}$
$\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.832$, $T_{max} = 0.995$
10444 measured reflections
2909 independent reflections
2382 reflections with $I > 2\sigma(I)$
$R_{int} = 0.026$
$\theta_{max} = 26.4°$, $\theta_{min} = 2.2°$
$h = -20\rightarrow20$
$k = -11\rightarrow11$
$l = -23\rightarrow22$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.094$
$S = 1.05$
2909 reflections
219 parameters
45 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0469P)^2 + 1.2758P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.21$ e Å$^{-3}$
$\Delta\rho_{min} = -0.20$ e Å$^{-3}$

*Special details*

**Experimental**. Crystallized from diethyl ether.
Three sets of frames each taken over 0.3° $\omega$ rotation with 20 s exposure time. Detector set at $2\theta = 27°$, crystal-to-detector distance 5.00 cm.
**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 on $F^2$ against ALL reflections.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| O1A | 0.09291 (5) | 0.27723 (10) | 0.31483 (5) | 0.0280 (2) | 0.828 (3) |
| O2A | 0.43766 (6) | 0.16711 (12) | 0.52414 (6) | 0.0379 (3) | 0.828 (3) |
| O3A | 0.37950 (6) | −0.04279 (12) | 0.52952 (6) | 0.0417 (3) | 0.828 (3) |
| O4A | 0.27707 (6) | 0.55996 (11) | 0.39749 (6) | 0.0391 (3) | 0.828 (3) |
| O5A | 0.18713 (6) | 0.50849 (11) | 0.29870 (6) | 0.0372 (3) | 0.828 (3) |
| N1A | 0.37863 (7) | 0.08255 (13) | 0.50556 (6) | 0.0289 (3) | 0.828 (3) |
| N2A | 0.23146 (7) | 0.47265 (12) | 0.35751 (6) | 0.0272 (3) | 0.828 (3) |
| C1A | 0.15919 (8) | 0.23247 (15) | 0.35793 (10) | 0.0211 (4) | 0.828 (3) |
| C2A | 0.16612 (9) | 0.08529 (15) | 0.38681 (9) | 0.0229 (4) | 0.828 (3) |
| C3A | 0.23721 (9) | 0.03893 (16) | 0.43364 (10) | 0.0248 (4) | 0.828 (3) |
| H31A | 0.2404 | −0.0576 | 0.4524 | 0.030* | 0.828 (3) |
| C4A | 0.30549 (7) | 0.13271 (15) | 0.45422 (7) | 0.0245 (3) | 0.828 (3) |
| C5A | 0.30269 (7) | 0.27426 (15) | 0.42947 (7) | 0.0235 (3) | 0.828 (3) |
| H51A | 0.3483 | 0.3380 | 0.4452 | 0.028* | 0.828 (3) |
| C6A | 0.23184 (8) | 0.32142 (14) | 0.38106 (7) | 0.0234 (3) | 0.828 (3) |
| C7A | 0.09301 (9) | −0.01813 (17) | 0.36283 (9) | 0.0273 (4) | 0.828 (3) |
| H71A | 0.0486 | 0.0358 | 0.3279 | 0.033* | 0.828 (3) |
| C8A | 0.05870 (16) | −0.0639 (4) | 0.42727 (16) | 0.0404 (6) | 0.828 (3) |
| H81A | 0.0112 | −0.1283 | 0.4104 | 0.061* | 0.828 (3) |
| H82A | 0.0418 | 0.0235 | 0.4494 | 0.061* | 0.828 (3) |
| H83A | 0.1014 | −0.1157 | 0.4628 | 0.061* | 0.828 (3) |
| C9A | 0.11577 (11) | −0.15163 (18) | 0.32438 (10) | 0.0328 (5) | 0.828 (3) |
| H91A | 0.1610 | −0.2049 | 0.3577 | 0.039* | 0.828 (3) |
| H92A | 0.0678 | −0.2180 | 0.3114 | 0.039* | 0.828 (3) |

| | | | | | |
|---|---|---|---|---|---|
| C10A | 0.1424 (2) | −0.1119 (3) | 0.25677 (14) | 0.0392 (7) | 0.828 (3) |
| H11A | 0.1603 | −0.2004 | 0.2362 | 0.059* | 0.828 (3) |
| H12A | 0.1881 | −0.0421 | 0.2689 | 0.059* | 0.828 (3) |
| H13A | 0.0961 | −0.0678 | 0.2217 | 0.059* | 0.828 (3) |
| O1C | 0.09291 (5) | 0.27723 (10) | 0.31483 (5) | 0.0280 (2) | 0.172 (3) |
| O2C | 0.43766 (6) | 0.16711 (12) | 0.52414 (6) | 0.0379 (3) | 0.172 (3) |
| O3C | 0.37950 (6) | −0.04279 (12) | 0.52952 (6) | 0.0417 (3) | 0.172 (3) |
| O4C | 0.27707 (6) | 0.55996 (11) | 0.39749 (6) | 0.0391 (3) | 0.172 (3) |
| O5C | 0.18713 (6) | 0.50849 (11) | 0.29870 (6) | 0.0372 (3) | 0.172 (3) |
| N1C | 0.37863 (7) | 0.08255 (13) | 0.50556 (6) | 0.0289 (3) | 0.172 (3) |
| N2C | 0.23146 (7) | 0.47265 (12) | 0.35751 (6) | 0.0272 (3) | 0.172 (3) |
| C1C | 0.1637 (2) | 0.2281 (3) | 0.3484 (4) | 0.0211 (4) | 0.172 (3) |
| C2C | 0.1743 (2) | 0.0761 (3) | 0.3713 (3) | 0.0229 (4) | 0.172 (3) |
| C3C | 0.2435 (3) | 0.0322 (3) | 0.4213 (4) | 0.0248 (4) | 0.172 (3) |
| H31C | 0.2499 | −0.0682 | 0.4342 | 0.030* | 0.172 (3) |
| C4C | 0.30549 (7) | 0.13271 (15) | 0.45422 (7) | 0.0245 (3) | 0.172 (3) |
| C5C | 0.30269 (7) | 0.27426 (15) | 0.42947 (7) | 0.0235 (3) | 0.172 (3) |
| H51C | 0.3483 | 0.3380 | 0.4452 | 0.028* | 0.172 (3) |
| C6C | 0.23184 (8) | 0.32142 (14) | 0.38106 (7) | 0.0234 (3) | 0.172 (3) |
| C7C | 0.1084 (3) | −0.0307 (4) | 0.3324 (3) | 0.032 (2)* | 0.172 (3) |
| H7C | 0.0566 | 0.0271 | 0.3158 | 0.038* | 0.172 (3) |
| C8C | 0.1287 (9) | −0.0929 (13) | 0.2640 (5) | 0.052 (7)* | 0.172 (3) |
| H81C | 0.1447 | −0.0128 | 0.2365 | 0.078* | 0.172 (3) |
| H82C | 0.0802 | −0.1424 | 0.2345 | 0.078* | 0.172 (3) |
| H83C | 0.1741 | −0.1629 | 0.2778 | 0.078* | 0.172 (3) |
| C9C | 0.0903 (4) | −0.1504 (6) | 0.3806 (4) | 0.035 (2)* | 0.172 (3) |
| H91C | 0.1422 | −0.2010 | 0.4038 | 0.041* | 0.172 (3) |
| H92C | 0.0531 | −0.2229 | 0.3507 | 0.041* | 0.172 (3) |
| C10C | 0.0509 (13) | −0.0946 (15) | 0.4387 (8) | 0.066 (7)* | 0.172 (3) |
| H11C | 0.0340 | −0.1778 | 0.4637 | 0.099* | 0.172 (3) |
| H12C | 0.0027 | −0.0352 | 0.4166 | 0.099* | 0.172 (3) |
| H13C | 0.0907 | −0.0349 | 0.4731 | 0.099* | 0.172 (3) |
| C1B | −0.05882 (9) | 0.53181 (16) | 0.34180 (8) | 0.0342 (3) | |
| H11B | −0.0275 | 0.5864 | 0.3137 | 0.051* | |
| H12B | −0.0330 | 0.5441 | 0.3932 | 0.051* | |
| H13B | −0.1153 | 0.5689 | 0.3310 | 0.051* | |
| N1B | −0.05977 (7) | 0.37513 (12) | 0.32293 (7) | 0.0251 (3) | |
| H1B | −0.0076 (10) | 0.3408 (17) | 0.3347 (8) | 0.030* | |
| H2B | −0.0796 (9) | 0.3654 (17) | 0.2750 (9) | 0.030* | |
| C2B | −0.11157 (9) | 0.28503 (17) | 0.35852 (9) | 0.0365 (4) | |
| H21B | −0.1073 | 0.1819 | 0.3460 | 0.055* | |
| H22B | −0.1690 | 0.3167 | 0.3423 | 0.055* | |
| H23B | −0.0926 | 0.2970 | 0.4107 | 0.055* | |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1A | 0.0194 (4) | 0.0320 (5) | 0.0310 (5) | 0.0004 (4) | 0.0034 (4) | 0.0035 (4) |

| | | | | | | |
|---|---|---|---|---|---|---|
| O2A | 0.0232 (5) | 0.0452 (6) | 0.0406 (6) | −0.0027 (4) | −0.0006 (4) | −0.0006 (5) |
| O3A | 0.0355 (6) | 0.0370 (6) | 0.0474 (7) | 0.0063 (5) | 0.0006 (5) | 0.0110 (5) |
| O4A | 0.0376 (6) | 0.0310 (6) | 0.0437 (6) | −0.0147 (4) | 0.0011 (5) | 0.0020 (5) |
| O5A | 0.0393 (6) | 0.0334 (6) | 0.0339 (6) | −0.0018 (4) | −0.0004 (5) | 0.0087 (5) |
| N1A | 0.0243 (6) | 0.0345 (7) | 0.0278 (6) | 0.0046 (5) | 0.0065 (5) | −0.0006 (5) |
| N2A | 0.0242 (5) | 0.0283 (6) | 0.0296 (6) | −0.0040 (5) | 0.0076 (5) | 0.0019 (5) |
| C1A | 0.0201 (6) | 0.0256 (7) | 0.0194 (8) | 0.0007 (5) | 0.0084 (5) | −0.0024 (5) |
| C2A | 0.0217 (7) | 0.0243 (7) | 0.0247 (9) | −0.0007 (5) | 0.0095 (6) | −0.0047 (6) |
| C3A | 0.0262 (7) | 0.0237 (7) | 0.0264 (9) | 0.0017 (5) | 0.0102 (6) | −0.0006 (6) |
| C4A | 0.0207 (6) | 0.0303 (7) | 0.0223 (7) | 0.0034 (5) | 0.0054 (5) | −0.0013 (5) |
| C5A | 0.0189 (6) | 0.0295 (7) | 0.0229 (7) | −0.0033 (5) | 0.0069 (5) | −0.0037 (5) |
| C6A | 0.0240 (6) | 0.0236 (6) | 0.0242 (7) | −0.0009 (5) | 0.0092 (5) | −0.0003 (5) |
| C7A | 0.0232 (8) | 0.0256 (9) | 0.0324 (10) | −0.0048 (6) | 0.0057 (7) | −0.0015 (7) |
| C8A | 0.0333 (12) | 0.0502 (13) | 0.0409 (13) | −0.0134 (11) | 0.0153 (10) | −0.0074 (12) |
| C9A | 0.0350 (9) | 0.0255 (9) | 0.0377 (10) | −0.0074 (7) | 0.0089 (8) | −0.0044 (7) |
| C10A | 0.0408 (13) | 0.0430 (13) | 0.0348 (13) | −0.0144 (11) | 0.0114 (11) | −0.0094 (10) |
| O1C | 0.0194 (4) | 0.0320 (5) | 0.0310 (5) | 0.0004 (4) | 0.0034 (4) | 0.0035 (4) |
| O2C | 0.0232 (5) | 0.0452 (6) | 0.0406 (6) | −0.0027 (4) | −0.0006 (4) | −0.0006 (5) |
| O3C | 0.0355 (6) | 0.0370 (6) | 0.0474 (7) | 0.0063 (5) | 0.0006 (5) | 0.0110 (5) |
| O4C | 0.0376 (6) | 0.0310 (6) | 0.0437 (6) | −0.0147 (4) | 0.0011 (5) | 0.0020 (5) |
| O5C | 0.0393 (6) | 0.0334 (6) | 0.0339 (6) | −0.0018 (4) | −0.0004 (5) | 0.0087 (5) |
| N1C | 0.0243 (6) | 0.0345 (7) | 0.0278 (6) | 0.0046 (5) | 0.0065 (5) | −0.0006 (5) |
| N2C | 0.0242 (5) | 0.0283 (6) | 0.0296 (6) | −0.0040 (5) | 0.0076 (5) | 0.0019 (5) |
| C1C | 0.0201 (6) | 0.0256 (7) | 0.0194 (8) | 0.0007 (5) | 0.0084 (5) | −0.0024 (5) |
| C2C | 0.0217 (7) | 0.0243 (7) | 0.0247 (9) | −0.0007 (5) | 0.0095 (6) | −0.0047 (6) |
| C3C | 0.0262 (7) | 0.0237 (7) | 0.0264 (9) | 0.0017 (5) | 0.0102 (6) | −0.0006 (6) |
| C4C | 0.0207 (6) | 0.0303 (7) | 0.0223 (7) | 0.0034 (5) | 0.0054 (5) | −0.0013 (5) |
| C5C | 0.0189 (6) | 0.0295 (7) | 0.0229 (7) | −0.0033 (5) | 0.0069 (5) | −0.0037 (5) |
| C6C | 0.0240 (6) | 0.0236 (6) | 0.0242 (7) | −0.0009 (5) | 0.0092 (5) | −0.0003 (5) |
| C1B | 0.0378 (8) | 0.0301 (7) | 0.0324 (8) | 0.0000 (6) | 0.0046 (6) | −0.0019 (6) |
| N1B | 0.0198 (5) | 0.0283 (6) | 0.0271 (6) | 0.0006 (4) | 0.0057 (5) | 0.0006 (5) |
| C2B | 0.0300 (7) | 0.0399 (8) | 0.0411 (9) | −0.0055 (6) | 0.0117 (6) | 0.0058 (7) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1A—C1A | 1.2770 (16) | C10A—H13A | 0.9800 |
| O2A—N1A | 1.2375 (15) | C1C—C2C | 1.455 (2) |
| O3A—N1A | 1.2339 (15) | C2C—C3C | 1.369 (2) |
| O4A—N2A | 1.2326 (15) | C2C—C7C | 1.524 (3) |
| O5A—N2A | 1.2318 (15) | C3C—H31C | 0.9500 |
| N1A—C4A | 1.4440 (17) | C7C—C9C | 1.514 (3) |
| N2A—C6A | 1.4546 (17) | C7C—C8C | 1.547 (4) |
| C1A—C6A | 1.4412 (18) | C7C—H7C | 1.0000 |
| C1A—C2A | 1.4493 (19) | C8C—H81C | 0.9800 |
| C2A—C3A | 1.3700 (19) | C8C—H82C | 0.9800 |
| C2A—C7A | 1.5269 (19) | C8C—H83C | 0.9800 |
| C3A—C4A | 1.4073 (19) | C9C—C10C | 1.520 (4) |
| C3A—H31A | 0.9500 | C9C—H91C | 0.9900 |

| | | | |
|---|---|---|---|
| C4A—C5A | 1.3758 (19) | C9C—H92C | 0.9900 |
| C5A—C6A | 1.3830 (18) | C10C—H11C | 0.9800 |
| C5A—H51A | 0.9500 | C10C—H12C | 0.9800 |
| C7A—C9A | 1.525 (2) | C10C—H13C | 0.9800 |
| C7A—C8A | 1.549 (4) | C1B—N1B | 1.4771 (18) |
| C7A—H71A | 1.0000 | C1B—H11B | 0.9800 |
| C8A—H81A | 0.9800 | C1B—H12B | 0.9800 |
| C8A—H82A | 0.9800 | C1B—H13B | 0.9800 |
| C8A—H83A | 0.9800 | N1B—C2B | 1.4845 (18) |
| C9A—C10A | 1.521 (3) | N1B—H1B | 0.904 (16) |
| C9A—H91A | 0.9900 | N1B—H2B | 0.902 (17) |
| C9A—H92A | 0.9900 | C2B—H21B | 0.9800 |
| C10A—H11A | 0.9800 | C2B—H22B | 0.9800 |
| C10A—H12A | 0.9800 | C2B—H23B | 0.9800 |

| | | | |
|---|---|---|---|
| O3A—N1A—O2A | 122.63 (12) | C9C—C7C—C8C | 112.1 (3) |
| O3A—N1A—C4A | 118.58 (11) | C2C—C7C—C8C | 111.4 (3) |
| O2A—N1A—C4A | 118.79 (12) | C9C—C7C—H7C | 106.5 |
| O5A—N2A—O4A | 122.53 (12) | C2C—C7C—H7C | 106.5 |
| O5A—N2A—C6A | 119.53 (11) | C8C—C7C—H7C | 106.5 |
| O4A—N2A—C6A | 117.93 (11) | C7C—C8C—H81C | 109.5 |
| O1A—C1A—C6A | 123.52 (12) | C7C—C8C—H82C | 109.5 |
| O1A—C1A—C2A | 121.27 (11) | H81C—C8C—H82C | 109.5 |
| C6A—C1A—C2A | 115.20 (11) | C7C—C8C—H83C | 109.5 |
| C3A—C2A—C1A | 120.79 (12) | H81C—C8C—H83C | 109.5 |
| C3A—C2A—C7A | 120.62 (13) | H82C—C8C—H83C | 109.5 |
| C1A—C2A—C7A | 118.59 (12) | C7C—C9C—C10C | 113.5 (3) |
| C2A—C3A—C4A | 120.73 (13) | C7C—C9C—H91C | 108.9 |
| C2A—C3A—H31A | 119.6 | C10C—C9C—H91C | 108.9 |
| C4A—C3A—H31A | 119.6 | C7C—C9C—H92C | 108.9 |
| C5A—C4A—C3A | 121.43 (12) | C10C—C9C—H92C | 108.9 |
| C5A—C4A—N1A | 118.98 (12) | H91C—C9C—H92C | 107.7 |
| C3A—C4A—N1A | 119.49 (12) | C9C—C10C—H11C | 109.5 |
| C4A—C5A—C6A | 118.36 (12) | C9C—C10C—H12C | 109.5 |
| C4A—C5A—H51A | 120.8 | H11C—C10C—H12C | 109.5 |
| C6A—C5A—H51A | 120.8 | C9C—C10C—H13C | 109.5 |
| C5A—C6A—C1A | 123.42 (12) | H11C—C10C—H13C | 109.5 |
| C5A—C6A—N2A | 116.20 (11) | H12C—C10C—H13C | 109.5 |
| C1A—C6A—N2A | 120.23 (11) | N1B—C1B—H11B | 109.5 |
| C9A—C7A—C2A | 111.19 (13) | N1B—C1B—H12B | 109.5 |
| C9A—C7A—C8A | 111.13 (16) | H11B—C1B—H12B | 109.5 |
| C2A—C7A—C8A | 111.02 (16) | N1B—C1B—H13B | 109.5 |
| C9A—C7A—H71A | 107.8 | H11B—C1B—H13B | 109.5 |
| C2A—C7A—H71A | 107.8 | H12B—C1B—H13B | 109.5 |
| C8A—C7A—H71A | 107.8 | C1B—N1B—C2B | 113.36 (12) |
| C10A—C9A—C7A | 112.75 (19) | C1B—N1B—H1B | 109.0 (10) |
| C10A—C9A—H91A | 109.0 | C2B—N1B—H1B | 109.6 (10) |
| C7A—C9A—H91A | 109.0 | C1B—N1B—H2B | 109.0 (10) |

| | | | |
|---|---|---|---|
| C10A—C9A—H92A | 109.0 | C2B—N1B—H2B | 107.9 (10) |
| C7A—C9A—H92A | 109.0 | H1B—N1B—H2B | 107.9 (14) |
| H91A—C9A—H92A | 107.8 | N1B—C2B—H21B | 109.5 |
| C3C—C2C—C1C | 120.69 (19) | N1B—C2B—H22B | 109.5 |
| C3C—C2C—C7C | 122.7 (2) | H21B—C2B—H22B | 109.5 |
| C1C—C2C—C7C | 116.4 (2) | N1B—C2B—H23B | 109.5 |
| C2C—C3C—H31C | 119.2 | H21B—C2B—H23B | 109.5 |
| C9C—C7C—C2C | 113.3 (3) | H22B—C2B—H23B | 109.5 |

| | | | |
|---|---|---|---|
| O1A—C1A—C2A—C3A | −179.26 (17) | O1A—C1A—C6A—N2A | 2.4 (3) |
| C6A—C1A—C2A—C3A | 1.1 (2) | C2A—C1A—C6A—N2A | −177.99 (13) |
| O1A—C1A—C2A—C7A | 1.9 (2) | O5A—N2A—C6A—C5A | 154.19 (12) |
| C6A—C1A—C2A—C7A | −177.74 (16) | O4A—N2A—C6A—C5A | −24.80 (17) |
| C1A—C2A—C3A—C4A | −0.7 (2) | O5A—N2A—C6A—C1A | −30.0 (2) |
| C7A—C2A—C3A—C4A | 178.17 (17) | O4A—N2A—C6A—C1A | 151.04 (15) |
| C2A—C3A—C4A—C5A | 1.4 (2) | C3A—C2A—C7A—C9A | −60.4 (2) |
| C2A—C3A—C4A—N1A | 177.89 (13) | C1A—C2A—C7A—C9A | 118.50 (17) |
| O3A—N1A—C4A—C5A | 177.35 (12) | C3A—C2A—C7A—C8A | 63.9 (2) |
| O2A—N1A—C4A—C5A | −2.18 (18) | C1A—C2A—C7A—C8A | −117.23 (18) |
| O3A—N1A—C4A—C3A | 0.8 (2) | C2A—C7A—C9A—C10A | −61.0 (2) |
| O2A—N1A—C4A—C3A | −178.72 (14) | C8A—C7A—C9A—C10A | 174.80 (18) |
| C3A—C4A—C5A—C6A | −2.7 (2) | C3C—C2C—C7C—C9C | 40.3 (6) |
| N1A—C4A—C5A—C6A | −179.14 (11) | C1C—C2C—C7C—C9C | −144.7 (5) |
| C4A—C5A—C6A—C1A | 3.3 (2) | C3C—C2C—C7C—C8C | −87.2 (7) |
| C4A—C5A—C6A—N2A | 178.96 (11) | C1C—C2C—C7C—C8C | 87.8 (7) |
| O1A—C1A—C6A—C5A | 177.91 (15) | C2C—C7C—C9C—C10C | 66.4 (11) |
| C2A—C1A—C6A—C5A | −2.5 (2) | C8C—C7C—C9C—C10C | −166.5 (11) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1*B*—H1*B*···O1*A* | 0.904 (16) | 1.914 (16) | 2.7585 (15) | 154.7 (14) |
| N1*B*—H2*B*···O1*A*$^{i}$ | 0.902 (17) | 1.868 (17) | 2.7173 (16) | 156.2 (15) |

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