

Acta Crystallographica Section E

## Structure Reports

Online

ISSN 1600-5368

## Diisopropylammonium nitrite

Ying-Chun Wang\* and Xu Jie

College of Chemistry and Chemical Engineering, Southeast University, Nanjing 210096, People's Republic of China

Correspondence e-mail: wangyc33@yahoo.com.cn

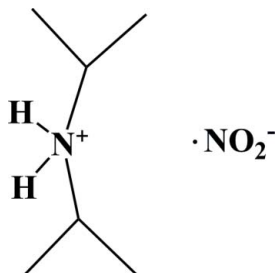
Received 25 February 2012; accepted 26 February 2012

Key indicators: single-crystal X-ray study;  $T = 298$  K; mean  $\sigma(\text{C}-\text{C}) = 0.005$  Å;  $R$  factor = 0.084;  $wR$  factor = 0.285; data-to-parameter ratio = 22.4.

In the title molecular salt,  $\text{C}_6\text{H}_{16}\text{N}^+\cdot\text{NO}_2^-$ , the cation forms two  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bonds to nearby nitrite anions which link the ionic units into chains propagating along the  $b$ -axis direction.

## Related literature

For a related structure, see: Xu (2012). For background to molecular ferroelectric compounds, see: Fu *et al.* (2011).



## Experimental

## Crystal data

 $\text{C}_6\text{H}_{16}\text{N}^+\cdot\text{NO}_2^-$  $M_r = 148.21$ Monoclinic,  $P2_1/n$  $a = 8.2314$  (16) Å $b = 7.7466$  (15) Å $c = 14.583$  (3) Å
 $\beta = 94.16$  (3)°  
 $V = 927.5$  (3) Å<sup>3</sup>  
 $Z = 4$   
 Mo  $K\alpha$  radiation

 $\mu = 0.08$  mm<sup>-1</sup>  
 $T = 298$  K  
 $0.10 \times 0.03 \times 0.03$  mm

## Data collection

 Rigaku Mercury2 CCD diffractometer  
 Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)  
 $T_{\min} = 0.910$ ,  $T_{\max} = 1.000$ 

 9189 measured reflections  
 2127 independent reflections  
 1130 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.075$ 

## Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.084$   
 $wR(F^2) = 0.285$   
 $S = 1.07$   
 2127 reflections  
 95 parameters

 15 restraints  
 H-atom parameters constrained  
 $\Delta\rho_{\max} = 0.50$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.32$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1E}\cdots\text{O1}$	0.90	1.90	2.800 (3)	174
$\text{N1}-\text{H1D}\cdots\text{O1}^{\dagger}$	0.90	2.00	2.869 (3)	161

Symmetry code: (i)  $-x + \frac{3}{2}, y - \frac{1}{2}, -z + \frac{1}{2}$ .

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; 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*.

This work was supported by the Doctoral Foundation of Southeast University, China.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6657).

## References

- Fu, D.-W., Zhang, W., Cai, H.-L., Zhang, Y., Ge, J.-Z., Xiong, R.-G., Huang, S. P. D. & Nakamura, T. (2011). *Angew. Chem. Int. Ed.* **50**, 11947–11951.  
 Rigaku (2005). *CrystalClear*. Rigaku Corporation, Tokyo, Japan.  
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.  
 Xu, J. (2012). *Acta Cryst.* **E68**, o894.

## supporting information

*Acta Cryst.* (2012). E68, o986 [https://doi.org/10.1107/S1600536812008574]

## Diisopropylammonium nitrite

Ying-Chun Wang and Xu Jie

### S1. Comment

Simple organic salts containing amino cations have attracted an attention as materials which display ferroelectric-paraelectric phase transitions (Fu *et al.*, 2011). As part of our ongoing studies in this area, (Xu, 2012), various amines have been studied and a series of new materials with these organic molecules have been elaborated. Herein we present the crystal structure of the title compound, di-isopropylammonium nitrite.

The asymmetric unit of the title compound contains one di-isopropylammonium cation and one NO<sub>2</sub><sup>-</sup> anion (Fig. 1). The amino N atom was protonated. The O-N-O bond angle of NO<sub>2</sub><sup>-</sup> anion is 116.4 (4)°. And the other geometric parameters of the title compound are in the normal range.

In the crystal structure, all the ammonium H atoms are involved in intermolecular N—H<sup>+</sup>⋯O H-bonding interactions with both the O atoms of the NO<sub>2</sub><sup>-</sup> anion (with N<sup>+</sup>⋯O distances of 2.800 (3) Å and 2.869 (3) Å, respectively). These hydrogen bonds link the ionic units into a one-dimensional chain along the *b*-axis (Table 1 and Fig. 2).

### S2. Experimental

A mixture of di-isopropylamine (0.8 mmol), HCl (0.8 mmol) and NaNO<sub>2</sub> (0.8 mmol) were dissolved in EtOH/distilled water (1:1 v/v) solvent. The solution was slowly evaporated in air affording colourless block-shaped crystals of the title compound.

### S3. Refinement

All H atoms attached to C atoms were fixed geometrically and treated as riding with C—H = 0.98 Å (C methine) and C—H = 0.96 Å (C methyl) with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C methine})$  and  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C methyl})$ . The positional parameters of the H atoms (N) were refined freely. And in the last stage of the refinement, they were constrained with N—H = 0.90 Å, and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{N})$ .

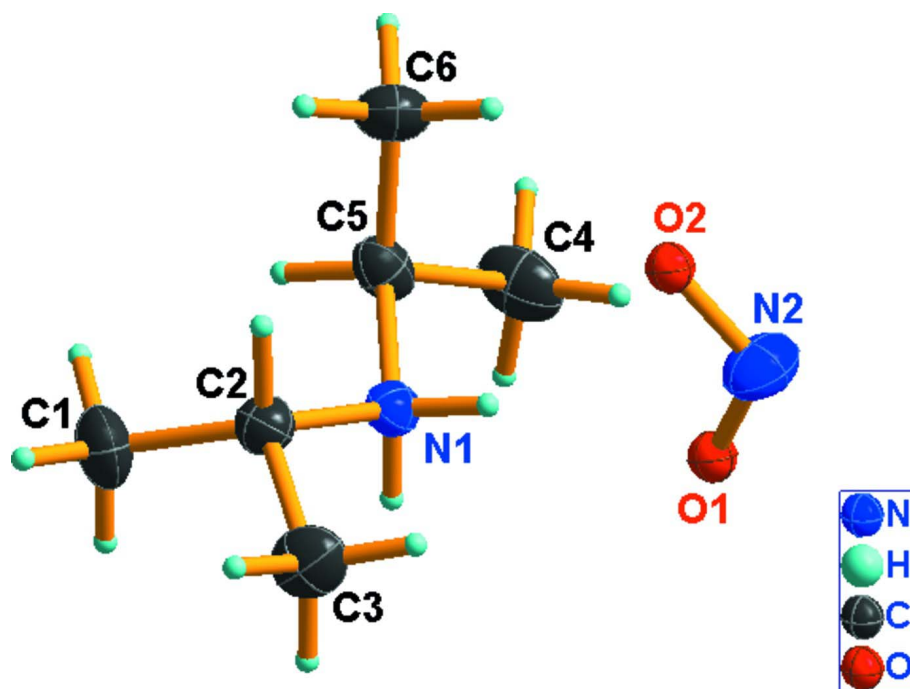


Figure 1

A view of the title compound with displacement ellipsoids drawn at the 30% probability level.

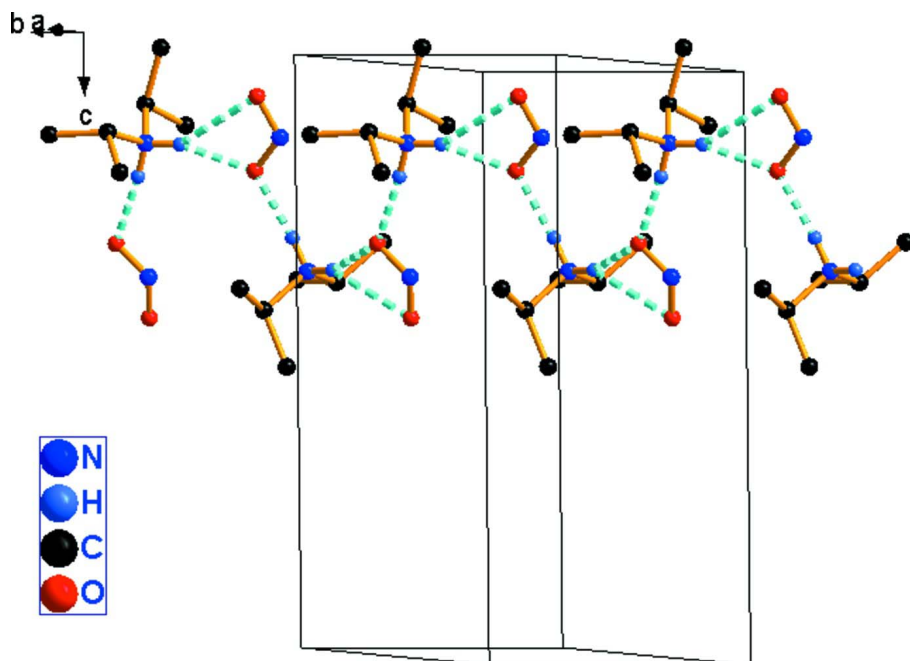


Figure 2

The crystal packing of the title compound showing the one-dimensional hydrogen bonding chain along the *b* axis (dashed line). H atoms not involved in hydrogen bonding (dashed line) have been omitted for clarity.

## Diisopropylammonium nitrite

## Crystal data

C<sub>6</sub>H<sub>16</sub>N<sup>+</sup>·NO<sub>2</sub><sup>-</sup> $M_r = 148.21$ Monoclinic,  $P2_1/n$ 

Hall symbol: -P 2yn

 $a = 8.2314 (16) \text{ \AA}$  $b = 7.7466 (15) \text{ \AA}$  $c = 14.583 (3) \text{ \AA}$  $\beta = 94.16 (3)^\circ$  $V = 927.5 (3) \text{ \AA}^3$  $Z = 4$  $F(000) = 328$  $D_x = 1.061 \text{ Mg m}^{-3}$ Mo  $K\alpha$  radiation,  $\lambda = 0.71073 \text{ \AA}$ 

Cell parameters from 2127 reflections

 $\theta = 3.6\text{--}27.5^\circ$  $\mu = 0.08 \text{ mm}^{-1}$  $T = 298 \text{ K}$ 

Block, colourless

 $0.10 \times 0.03 \times 0.03 \text{ mm}$ 

## Data collection

Rigaku Mercury2 CCD

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: 13.6612 pixels  $\text{mm}^{-1}$ 

CCD profile fitting scans

Absorption correction: multi-scan

(CrystalClear; Rigaku, 2005)

 $T_{\min} = 0.910$ ,  $T_{\max} = 1.000$ 

9189 measured reflections

2127 independent reflections

1130 reflections with  $I > 2\sigma(I)$  $R_{\text{int}} = 0.075$  $\theta_{\max} = 27.5^\circ$ ,  $\theta_{\min} = 3.6^\circ$  $h = -10 \rightarrow 10$  $k = -10 \rightarrow 10$  $l = -18 \rightarrow 18$ 

## Refinement

Refinement on  $F^2$ 

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.084$  $wR(F^2) = 0.285$  $S = 1.07$ 

2127 reflections

95 parameters

15 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

 $w = 1/[\sigma^2(F_o^2) + (0.1379P)^2 + 0.0979P]$ where  $P = (F_o^2 + 2F_c^2)/3$  $(\Delta/\sigma)_{\max} < 0.001$  $\Delta\rho_{\max} = 0.50 \text{ e \AA}^{-3}$  $\Delta\rho_{\min} = -0.32 \text{ e \AA}^{-3}$ 

## Special details

**Geometry.** All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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  $F^2 > 2\sigma(F^2)$  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 ( $\text{\AA}^2$ )

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.6713 (3)	0.1958 (3)	0.35567 (15)	0.0543 (7)
H1D	0.6950	0.0825	0.3543	0.065*
H1E	0.6890	0.2393	0.3000	0.065*
C2	0.4930 (4)	0.2132 (4)	0.3690 (2)	0.0624 (9)

H2A	0.4733	0.1702	0.4304	0.075*
O1	0.7263 (3)	0.3533 (3)	0.18791 (17)	0.0931 (9)
C5	0.7889 (4)	0.2792 (4)	0.4247 (2)	0.0686 (9)
H5A	0.7676	0.4037	0.4255	0.082*
N2	0.7438 (4)	0.2425 (4)	0.1320 (2)	0.0925 (9)
O2	0.8050 (4)	0.2847 (5)	0.0648 (2)	0.1177 (10)
C6	0.7724 (5)	0.2066 (6)	0.5196 (2)	0.0918 (12)
H6A	0.6642	0.2277	0.5376	0.138*
H6B	0.8500	0.2614	0.5626	0.138*
H6C	0.7925	0.0846	0.5192	0.138*
C3	0.3999 (4)	0.1012 (6)	0.2986 (3)	0.0909 (12)
H3A	0.4409	-0.0148	0.3032	0.136*
H3B	0.4133	0.1453	0.2381	0.136*
H3C	0.2865	0.1017	0.3099	0.136*
C4	0.9579 (5)	0.2488 (7)	0.3938 (3)	0.1037 (14)
H4A	0.9700	0.3099	0.3374	0.156*
H4B	0.9736	0.1275	0.3841	0.156*
H4C	1.0374	0.2895	0.4402	0.156*
C1	0.4425 (5)	0.3998 (5)	0.3631 (3)	0.0941 (13)
H1A	0.5025	0.4643	0.4104	0.141*
H1B	0.3280	0.4089	0.3710	0.141*
H1C	0.4646	0.4452	0.3040	0.141*

*Atomic displacement parameters (Å<sup>2</sup>)*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
N1	0.0532 (15)	0.0552 (13)	0.0547 (13)	0.0026 (10)	0.0053 (10)	0.0051 (10)
C2	0.0558 (19)	0.0711 (19)	0.0611 (17)	0.0005 (14)	0.0101 (14)	0.0032 (14)
O1	0.1178 (19)	0.0879 (17)	0.0762 (14)	0.0013 (13)	0.0258 (13)	0.0032 (11)
C5	0.063 (2)	0.0582 (17)	0.082 (2)	-0.0024 (14)	-0.0121 (16)	0.0038 (15)
N2	0.116 (2)	0.0955 (18)	0.0689 (15)	0.0076 (14)	0.0293 (14)	-0.0001 (13)
O2	0.145 (2)	0.126 (2)	0.0854 (16)	0.0130 (16)	0.0308 (15)	0.0006 (14)
C6	0.102 (3)	0.110 (3)	0.061 (2)	0.003 (2)	-0.0109 (18)	-0.005 (2)
C3	0.061 (2)	0.114 (3)	0.097 (3)	-0.0143 (19)	0.0022 (19)	-0.014 (2)
C4	0.056 (2)	0.142 (4)	0.111 (3)	-0.011 (2)	-0.003 (2)	0.025 (3)
C1	0.068 (2)	0.089 (3)	0.125 (3)	0.0240 (19)	0.006 (2)	0.003 (2)

*Geometric parameters (Å, °)*

N1—C5	1.492 (4)	C6—H6A	0.9600
N1—C2	1.500 (4)	C6—H6B	0.9600
N1—H1D	0.9000	C6—H6C	0.9600
N1—H1E	0.9000	C3—H3A	0.9600
C2—C1	1.505 (5)	C3—H3B	0.9600
C2—C3	1.509 (5)	C3—H3C	0.9600
C2—H2A	0.9800	C4—H4A	0.9600
O1—N2	1.200 (4)	C4—H4B	0.9600
C5—C6	1.510 (5)	C4—H4C	0.9600

C5—C4	1.512 (5)	C1—H1A	0.9600
C5—H5A	0.9800	C1—H1B	0.9600
N2—O2	1.181 (4)	C1—H1C	0.9600
C5—N1—C2	117.8 (2)	C5—C6—H6C	109.5
C5—N1—H1D	107.9	H6A—C6—H6C	109.5
C2—N1—H1D	107.9	H6B—C6—H6C	109.5
C5—N1—H1E	107.9	C2—C3—H3A	109.5
C2—N1—H1E	107.9	C2—C3—H3B	109.5
H1D—N1—H1E	107.2	H3A—C3—H3B	109.5
N1—C2—C1	110.4 (3)	C2—C3—H3C	109.5
N1—C2—C3	108.2 (2)	H3A—C3—H3C	109.5
C1—C2—C3	112.9 (3)	H3B—C3—H3C	109.5
N1—C2—H2A	108.4	C5—C4—H4A	109.5
C1—C2—H2A	108.4	C5—C4—H4B	109.5
C3—C2—H2A	108.4	H4A—C4—H4B	109.5
N1—C5—C6	111.1 (3)	C5—C4—H4C	109.5
N1—C5—C4	107.3 (3)	H4A—C4—H4C	109.5
C6—C5—C4	111.2 (3)	H4B—C4—H4C	109.5
N1—C5—H5A	109.1	C2—C1—H1A	109.5
C6—C5—H5A	109.1	C2—C1—H1B	109.5
C4—C5—H5A	109.1	H1A—C1—H1B	109.5
O2—N2—O1	116.4 (4)	C2—C1—H1C	109.5
C5—C6—H6A	109.5	H1A—C1—H1C	109.5
C5—C6—H6B	109.5	H1B—C1—H1C	109.5
H6A—C6—H6B	109.5		
C5—N1—C2—C1	63.0 (3)	C2—N1—C5—C6	60.4 (3)
C5—N1—C2—C3	-173.0 (3)	C2—N1—C5—C4	-177.8 (3)

*Hydrogen-bond geometry (Å, °)*

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
N1—H1E $\cdots$ O1	0.90	1.90	2.800 (3)	174
N1—H1D $\cdots$ O1 <sup>i</sup>	0.90	2.00	2.869 (3)	161

Symmetry code: (i)  $-x+3/2, y-1/2, -z+1/2$ .