

Diisopropylammonium perchlorate at 263 K

Sara Bajorat and Guido J. Reiss*

Institut für Anorganische Chemie und Strukturchemie, Lehrstuhl für Material- und Strukturforschung, Heinrich-Heine-Universität Düsseldorf, Universitätsstrasse 1, 40225 Düsseldorf, Germany

Correspondence e-mail: reissg@uni-duesseldorf.de

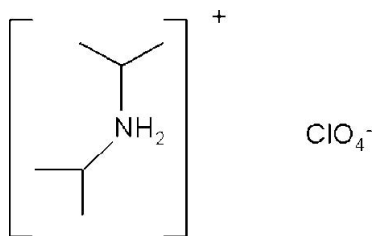
Received 27 April 2007; accepted 6 June 2007

 Key indicators: single-crystal X-ray study; $T = 263$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.044; wR factor = 0.090; data-to-parameter ratio = 16.0.

Diisopropylammonium perchlorate, $\text{C}_6\text{H}_{16}\text{N}^+\cdot\text{ClO}_4^-$, crystallizes in the monoclinic space group $P2_1/c$ in the temperature range from 213 to 291 K. The asymmetric unit contains two diisopropylammonium cations and two perchlorate anions in general positions. Each cation acts as double hydrogen-bond donor and one O atom of each perchlorate anion accepts two hydrogen bonds from chains that are classified as $C(4)$.

Related literature

For related literature, see: Blume *et al.* (2000); Etter *et al.* (1990); Kitaigorodski (1961); Reiß (1998, 2000, 2001, 2002); Reiß & Engel (2004); Reiß & Koppelhuber-Bitschnau (2002).



Experimental

Crystal data

$\text{C}_6\text{H}_{16}\text{N}^+\cdot\text{ClO}_4^-$
 $M_r = 201.65$
 Monoclinic, $P2_1/c$
 $a = 17.7018$ (18) Å
 $b = 8.2468$ (7) Å
 $c = 16.0601$ (16) Å
 $\beta = 116.334$ (11)°

$V = 2101.2$ (3) Å³
 $Z = 8$
 Mo $K\alpha$ radiation
 $\mu = 0.35$ mm⁻¹
 $T = 263$ (2) K
 $0.3 \times 0.15 \times 0.15$ mm

Data collection

Stoe IPDS diffractometer
 Absorption correction: none
 26389 measured reflections

3673 independent reflections
 1435 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.055$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.044$
 $wR(F^2) = 0.090$
 $S = 0.96$
 3673 reflections
 229 parameters
 24 restraints

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.34$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.19$ e Å⁻³

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1A}\cdots\text{O11}^{\text{i}}$	0.82 (3)	2.34 (3)	3.115 (4)	157 (3)
$\text{N1}-\text{H1B}\cdots\text{O11}$	0.86 (3)	2.27 (3)	3.119 (4)	169 (3)
$\text{N2}-\text{H2A}\cdots\text{O21}$	0.86 (3)	2.30 (3)	3.124 (4)	160 (3)
$\text{N2}-\text{H2B}\cdots\text{O21}^{\text{ii}}$	0.82 (3)	2.42 (3)	3.219 (4)	166 (3)

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

Data collection: *IPDS* (Stoe & Cie, 2000); cell refinement: *IPDS*; data reduction: *IPDS*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 2001); software used to prepare material for publication: *SHELXL97*.

The authors thank P. Roloff for the DSC measurements.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BC3050).

References

- Blume, A., Förster, G. & Meister, A. (2000). *Phys. Chem. Chem. Phys.* **2**, 4503–4508.
 Brandenburg, K. (2001). *DIAMOND*. Version 3.1. Crystal Impact GbR, Bonn, Germany.
 Etter, M. C., MacDonald, J. C. & Bernstein, J. (1990). *Acta Cryst.* **B46**, 256–262.
 Kitaigorodski, A. I. (1961). *Organic Chemical Crystallography*. New York: Academic Press.
 Reiß, G. J. (1998). *Acta Cryst.* **C54**, 1489–1491.
 Reiß, G. J. (2000). *Z. Kristallogr. Suppl.* **17**, 184.
 Reiß, G. J. (2001). *Acta Cryst.* **C57**, 994–995.
 Reiß, G. J. (2002). *Acta Cryst.* **E58**, m47–m50.
 Reiß, G. J. & Engel, J. S. (2004). *Acta Cryst.* **E60**, o985–o987.
 Reiß, G. J. & Koppelhuber-Bitschnau, B. (2002). *Acta Cryst.* **E58**, o1309–o1311.
 Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
 Stoe & Cie (2000). *IPDS*. Version 2.93. Stoe & Cie GmbH, Darmstadt, Germany.

supplementary materials

Acta Cryst. (2007). E63, o3144 [doi:10.1107/S1600536807027894]

Diisopropylammonium perchlorate at 263 K

S. Bajorat and G. J. Reiss

Comment

In recent years a series of diisopropylammonium (dip) salts were synthesized and structurally characterized. Their structures are dominated by hydrogen-bonding patterns. These pattern can be classified as quasi molecular ($\text{dip}_2[\text{SiF}_6]$) (Reiß, 1998), one dimensional polymeric (dipHF_2 , dipCl , dipBr , dipI , dipNO_3) (Reiß, 2001; Reiß & Koppelhuber-Bitschnau, 2002), two dimensional networks ($\text{dip}_2[\text{SO}_4]$) (Reiß, 2000) and three dimensional networks ($\text{dip}[\text{IrCl}_6]_2$) (Reiß, 2004). This class of compounds is an ideal model system for the investigation of anion-dependent formation of one- or higher-dimensional hydrogen-bonded polymers. Here we present a further example of a dip salt that shows a chain- type structure for an intermediate phase.

The asymmetric unit of the the title compound, (I), consists of two perchlorate anions and two diisopropylammonium cations, both located in general positions (Fig. 1). The C—C and N—C bond lengths in the cation are as expected, and both perchlorate anions show almost tetrahedral geometry (Table 1). One oxygen atom of each perchlorate anion (O11 and O21) accepts two hydrogen bonds from neighbouring ammonium groups, while each NH_2 group donates two hydrogen bonds. In the asymmetric unit, two crystallographically independent chains are found with very similar geometries. According to Etter's nomenclature, both chains can be classified as C(4). Fig. 2 illustrates the packing of the compound along the *b* axis. Chains are stacked to form the well known herring-bone motif (Blume *et al.*, 2000; Kitaigorodski, 1961; Reiß, 2002).

Thermal analysis (heating rate of 5°C/min) and temperature-dependent powder diffraction of the title compound in the temperature range from -150°C to $+20^\circ\text{C}$ reveal two first-order phase transformations at -60°C and $+18^\circ\text{C}$. In addition to crystals of (I) (the phase stable from -60°C to 18°C), crystals were also obtained at room temperature forming extremely weak diffracting platelets were always non-merohedrally twinned. A partial dataset adequate to solve the structure (after discarding all overlapping reflections) was collected but the structure could not be refined in detail. Nevertheless, it can be concluded that this phase is composed of quasi-molecular ring-shaped, dimeric $\text{dip}_2[\text{ClO}_4]_2$ units. The space group P1 [$a = 8.180(5) \text{ \AA}$, $b = 8.530(5) \text{ \AA}$, $c = 8.730(5) \text{ \AA}$, $\alpha = 83.040(5)^\circ$, $\beta = 64.837(5)^\circ$, $\gamma = 80.078(5)^\circ$] is a plausible choice as no center of symmetry is present in the $\text{dip}_2[\text{ClO}_4]$ dimer and no higher metrical symmetry of the lattice is found.

Experimental

At room temperature, 14.3 mmol diisopropylamine were added to (70 percent) perchloric acid (2.5 ml, 38.5 mmol) producing a colourless solution. From this solution, small thin platelets were obtained within a few days at room temperature. The crystals were investigated under ambient conditions. At -20°C , colourless needles grew from the solution in a few days. One crystal was mounted on a Stoe image plate diffractometer while keeping the temperature below the transition temperature of $+18^\circ\text{C}$. Cooling the crystals below -60°C resulted in twinning as revealed by split reflection profiles.

Refinement

All H atoms were located in successive Fourier synthesis. In the final stages of refinement, all H atoms of the CH₃ and CH groups were refined using a riding model with U_{iso} set to 1.5 and 1.2 times U_{eq} of the corresponding C atoms. The four H atoms of the two ammonium groups were refined freely with their U_{iso} set at 1.2 times U_{eq} of the N atoms. As a consequence of the low intensity, correlations appear for some displacement parameters. These effects were reduced by introducing anti-bump restraints for N1, C11, N2 and C24.

Figures

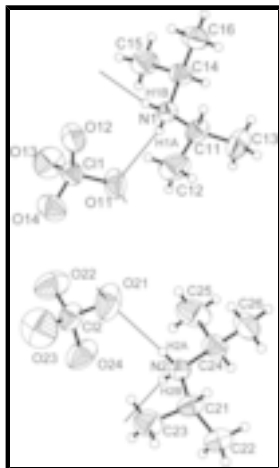


Fig. 1. The asymmetric unit of the title compound (displacement ellipsoids at the 40% probability level, H atoms drawn with arbitrary radius).

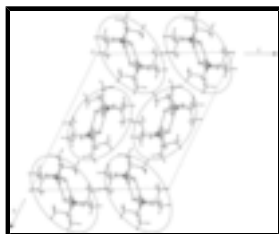


Fig. 2. Crystal packing seen along the b direction.

Diisopropylammonium perchlorate

Crystal data

$\text{C}_6\text{H}_{16}\text{N}^+\cdot\text{ClO}_4^-$

$M_r = 201.65$

Monoclinic, $P2_1/c$

$a = 17.7018 (18) \text{ \AA}$

$b = 8.2468 (7) \text{ \AA}$

$c = 16.0601 (16) \text{ \AA}$

$\beta = 116.334 (11)^\circ$

$V = 2101.2 (3) \text{ \AA}^3$

$Z = 8$

$F_{000} = 864$

$D_x = 1.275 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 7606 reflections

$\theta = 4.2\text{--}25.0^\circ$

$\mu = 0.35 \text{ mm}^{-1}$

$T = 263 (2) \text{ K}$

Needle, colourless

$0.3 \times 0.15 \times 0.15 \text{ mm}$

Data collection

Stoe IPDS diffractometer	3673 independent reflections
Radiation source: fine-focus sealed tube	1435 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.055$
Detector resolution: 50 pixels mm^{-1}	$\theta_{\text{max}} = 25.0^\circ$
$T = 263(2)$ K	$\theta_{\text{min}} = 4.2^\circ$
ω scans	$h = -21 \rightarrow 21$
Absorption correction: none	$k = -9 \rightarrow 9$
26389 measured reflections	$l = -19 \rightarrow 19$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.044$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.090$	$w = 1/[\sigma^2(F_o^2) + (0.026P)^2]$
$S = 0.96$	where $P = (F_o^2 + 2F_c^2)/3$
3673 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
229 parameters	$\Delta\rho_{\text{max}} = 0.34 \text{ e } \text{\AA}^{-3}$
24 restraints	$\Delta\rho_{\text{min}} = -0.19 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 $F^2 > 2\sigma(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl2	0.60921 (5)	0.20439 (13)	0.26584 (7)	0.0772 (3)
O21	0.57452 (19)	0.0819 (4)	0.2987 (3)	0.1385 (13)
O22	0.68890 (17)	0.2498 (4)	0.3304 (2)	0.1210 (10)
O23	0.6161 (2)	0.1435 (5)	0.1871 (2)	0.1564 (14)

supplementary materials

O24	0.55388 (16)	0.3388 (3)	0.2396 (2)	0.1166 (11)
Cl1	0.10780 (5)	0.28299 (12)	0.34991 (6)	0.0651 (3)
O11	0.07405 (15)	0.4100 (3)	0.28279 (17)	0.0988 (9)
O12	0.05430 (15)	0.1454 (3)	0.31697 (19)	0.1012 (9)
O13	0.11082 (16)	0.3366 (4)	0.43558 (18)	0.1087 (9)
O14	0.19014 (15)	0.2426 (3)	0.36572 (19)	0.0996 (8)
N1	0.09458 (16)	0.7607 (4)	0.36280 (19)	0.0585 (8)
H1A	0.0584 (19)	0.824 (4)	0.329 (2)	0.070*
H1B	0.0812 (19)	0.665 (4)	0.338 (2)	0.070*
C11	0.0929 (2)	0.7492 (4)	0.4552 (2)	0.0667 (9)
H11	0.1313	0.6622	0.4910	0.080*
C12	0.0049 (2)	0.7030 (5)	0.4375 (3)	0.1037 (13)
H12A	-0.0114	0.6055	0.4012	0.145*
H12B	-0.0333	0.7889	0.4044	0.145*
H12C	0.0032	0.6852	0.4958	0.145*
C13	0.1219 (3)	0.9042 (5)	0.5087 (2)	0.0944 (12)
H13A	0.1778	0.9291	0.5172	0.132*
H13B	0.1220	0.8922	0.5683	0.132*
H13C	0.0844	0.9904	0.4750	0.132*
C14	0.17520 (18)	0.8031 (4)	0.3579 (2)	0.0646 (9)
H14	0.1918	0.9127	0.3831	0.078*
C15	0.1570 (2)	0.8060 (5)	0.2562 (2)	0.0911 (12)
H15A	0.1167	0.8897	0.2246	0.127*
H15B	0.1345	0.7030	0.2284	0.127*
H15C	0.2082	0.8269	0.2513	0.127*
C16	0.2454 (2)	0.6886 (5)	0.4154 (3)	0.0871 (12)
H16A	0.2541	0.6916	0.4788	0.122*
H16B	0.2963	0.7207	0.4124	0.122*
H16C	0.2306	0.5804	0.3916	0.122*
N2	0.40327 (16)	0.2296 (4)	0.27392 (19)	0.0630 (8)
H2A	0.4432 (19)	0.167 (4)	0.276 (2)	0.076*
H2B	0.418 (2)	0.319 (4)	0.264 (2)	0.076*
C21	0.32467 (18)	0.1813 (4)	0.1909 (2)	0.0699 (10)
H21	0.3116	0.0693	0.2004	0.084*
C22	0.2512 (2)	0.2844 (5)	0.1790 (3)	0.1014 (13)
H22A	0.2428	0.2783	0.2340	0.142*
H22B	0.2015	0.2464	0.1266	0.142*
H22C	0.2620	0.3948	0.1686	0.142*
C23	0.3428 (2)	0.1828 (5)	0.1076 (2)	0.0957 (13)
H23A	0.3920	0.1186	0.1205	0.134*
H23B	0.3524	0.2923	0.0943	0.134*
H23C	0.2955	0.1385	0.0549	0.134*
C24	0.4043 (2)	0.2392 (4)	0.3673 (2)	0.0736 (10)
H24	0.3630	0.3208	0.3644	0.088*
C25	0.4913 (3)	0.2970 (6)	0.4363 (2)	0.1139 (14)
H25A	0.5042	0.3970	0.4148	0.159*
H25B	0.5325	0.2167	0.4418	0.159*
H25C	0.4920	0.3139	0.4958	0.159*
C26	0.3802 (2)	0.0803 (5)	0.3943 (3)	0.0959 (12)

H26A	0.3231	0.0546	0.3520	0.134*
H26B	0.3853	0.0881	0.4562	0.134*
H26C	0.4170	-0.0034	0.3921	0.134*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl2	0.0682 (6)	0.0601 (7)	0.1082 (7)	-0.0049 (5)	0.0436 (6)	-0.0054 (6)
O21	0.131 (2)	0.077 (2)	0.256 (4)	0.0094 (17)	0.130 (3)	0.044 (2)
O22	0.0790 (16)	0.117 (3)	0.129 (2)	-0.0064 (16)	0.0116 (16)	-0.001 (2)
O23	0.198 (4)	0.146 (3)	0.141 (3)	0.005 (2)	0.089 (3)	-0.046 (2)
O24	0.0857 (18)	0.070 (2)	0.195 (3)	0.0166 (15)	0.0628 (19)	0.0217 (18)
Cl1	0.0609 (5)	0.0534 (6)	0.0699 (6)	0.0020 (5)	0.0190 (4)	0.0013 (5)
O11	0.0980 (18)	0.0623 (19)	0.0930 (19)	0.0013 (14)	0.0033 (14)	0.0214 (15)
O12	0.0899 (18)	0.0562 (18)	0.110 (2)	-0.0123 (14)	0.0010 (15)	0.0083 (14)
O13	0.133 (2)	0.114 (3)	0.093 (2)	0.0004 (16)	0.0633 (18)	-0.0212 (17)
O14	0.0694 (14)	0.102 (2)	0.126 (2)	0.0190 (14)	0.0431 (14)	0.0024 (17)
N1	0.0544 (16)	0.059 (2)	0.0609 (17)	0.0007 (13)	0.0249 (13)	-0.0054 (14)
C11	0.087 (2)	0.065 (3)	0.0551 (19)	0.0092 (17)	0.0378 (17)	0.0068 (16)
C12	0.117 (3)	0.109 (3)	0.121 (3)	-0.010 (2)	0.087 (3)	-0.004 (3)
C13	0.129 (3)	0.089 (3)	0.065 (2)	0.003 (2)	0.042 (2)	-0.013 (2)
C14	0.0551 (19)	0.060 (2)	0.081 (2)	-0.0053 (17)	0.0320 (17)	-0.0027 (19)
C15	0.087 (3)	0.115 (4)	0.088 (3)	0.002 (2)	0.054 (2)	0.002 (3)
C16	0.064 (2)	0.082 (3)	0.112 (3)	0.009 (2)	0.036 (2)	0.004 (2)
N2	0.0567 (16)	0.066 (2)	0.0618 (15)	0.0019 (15)	0.0226 (13)	0.0093 (16)
C21	0.054 (2)	0.069 (3)	0.071 (2)	0.0000 (17)	0.0140 (18)	0.0006 (19)
C22	0.066 (2)	0.132 (4)	0.092 (3)	0.020 (2)	0.023 (2)	0.025 (3)
C23	0.095 (3)	0.110 (4)	0.070 (2)	-0.002 (2)	0.026 (2)	-0.004 (2)
C24	0.087 (2)	0.078 (3)	0.059 (2)	0.0179 (18)	0.0353 (17)	0.0066 (19)
C25	0.134 (3)	0.115 (4)	0.063 (2)	-0.016 (3)	0.017 (2)	-0.009 (2)
C26	0.123 (3)	0.094 (3)	0.084 (3)	0.006 (2)	0.058 (2)	0.018 (2)

Geometric parameters (\AA , $^\circ$)

Cl2—O22	1.381 (3)	C15—H15C	0.9600
Cl2—O21	1.402 (3)	C16—H16A	0.9600
Cl2—O24	1.414 (3)	C16—H16B	0.9600
Cl2—O23	1.416 (3)	C16—H16C	0.9600
Cl1—O14	1.404 (2)	N2—C21	1.492 (4)
Cl1—O12	1.421 (2)	N2—C24	1.493 (4)
Cl1—O13	1.423 (3)	N2—H2A	0.86 (3)
Cl1—O11	1.430 (2)	N2—H2B	0.82 (3)
N1—C11	1.500 (4)	C21—C22	1.494 (5)
N1—C14	1.505 (4)	C21—C23	1.509 (5)
N1—H1A	0.82 (3)	C21—H21	0.9800
N1—H1B	0.86 (3)	C22—H22A	0.9600
C11—C13	1.499 (5)	C22—H22B	0.9600
C11—C12	1.504 (5)	C22—H22C	0.9600
C11—H11	0.9800	C23—H23A	0.9600

supplementary materials

C12—H12A	0.9600	C23—H23B	0.9600
C12—H12B	0.9600	C23—H23C	0.9600
C12—H12C	0.9600	C24—C26	1.500 (5)
C13—H13A	0.9600	C24—C25	1.521 (5)
C13—H13B	0.9600	C24—H24	0.9800
C13—H13C	0.9600	C25—H25A	0.9600
C14—C16	1.507 (4)	C25—H25B	0.9600
C14—C15	1.518 (4)	C25—H25C	0.9600
C14—H14	0.9800	C26—H26A	0.9600
C15—H15A	0.9600	C26—H26B	0.9600
C15—H15B	0.9600	C26—H26C	0.9600
O22—C12—O21	112.6 (2)	C14—C16—H16A	109.5
O22—C12—O24	110.68 (19)	C14—C16—H16B	109.5
O21—C12—O24	108.44 (17)	H16A—C16—H16B	109.5
O22—C12—O23	107.6 (2)	C14—C16—H16C	109.5
O21—C12—O23	107.9 (2)	H16A—C16—H16C	109.5
O24—C12—O23	109.5 (2)	H16B—C16—H16C	109.5
O14—C11—O12	110.17 (17)	C21—N2—C24	120.2 (3)
O14—C11—O13	107.94 (16)	C21—N2—H2A	107 (2)
O12—C11—O13	109.93 (18)	C24—N2—H2A	111 (2)
O14—C11—O11	111.29 (16)	C21—N2—H2B	108 (2)
O12—C11—O11	108.52 (15)	C24—N2—H2B	107 (3)
O13—C11—O11	108.97 (18)	H2A—N2—H2B	103 (3)
C11—N1—C14	120.1 (2)	C22—C21—N2	112.1 (3)
C11—N1—H1A	110 (2)	C22—C21—C23	113.4 (3)
C14—N1—H1A	107 (2)	N2—C21—C23	108.0 (3)
C11—N1—H1B	106 (2)	C22—C21—H21	107.7
C14—N1—H1B	105 (2)	N2—C21—H21	107.7
H1A—N1—H1B	108 (3)	C23—C21—H21	107.7
C13—C11—N1	111.2 (3)	C21—C22—H22A	109.5
C13—C11—C12	112.7 (3)	C21—C22—H22B	109.5
N1—C11—C12	107.7 (3)	H22A—C22—H22B	109.5
C13—C11—H11	108.4	C21—C22—H22C	109.5
N1—C11—H11	108.4	H22A—C22—H22C	109.5
C12—C11—H11	108.4	H22B—C22—H22C	109.5
C11—C12—H12A	109.5	C21—C23—H23A	109.5
C11—C12—H12B	109.5	C21—C23—H23B	109.5
H12A—C12—H12B	109.5	H23A—C23—H23B	109.5
C11—C12—H12C	109.5	C21—C23—H23C	109.5
H12A—C12—H12C	109.5	H23A—C23—H23C	109.5
H12B—C12—H12C	109.5	H23B—C23—H23C	109.5
C11—C13—H13A	109.5	N2—C24—C26	111.5 (3)
C11—C13—H13B	109.5	N2—C24—C25	107.8 (3)
H13A—C13—H13B	109.5	C26—C24—C25	112.9 (3)
C11—C13—H13C	109.5	N2—C24—H24	108.2
H13A—C13—H13C	109.5	C26—C24—H24	108.2
H13B—C13—H13C	109.5	C25—C24—H24	108.2
N1—C14—C16	111.5 (3)	C24—C25—H25A	109.5
N1—C14—C15	107.8 (3)	C24—C25—H25B	109.5

C16—C14—C15	113.1 (3)	H25A—C25—H25B	109.5
N1—C14—H14	108.1	C24—C25—H25C	109.5
C16—C14—H14	108.1	H25A—C25—H25C	109.5
C15—C14—H14	108.1	H25B—C25—H25C	109.5
C14—C15—H15A	109.5	C24—C26—H26A	109.5
C14—C15—H15B	109.5	C24—C26—H26B	109.5
H15A—C15—H15B	109.5	H26A—C26—H26B	109.5
C14—C15—H15C	109.5	C24—C26—H26C	109.5
H15A—C15—H15C	109.5	H26A—C26—H26C	109.5
H15B—C15—H15C	109.5	H26B—C26—H26C	109.5

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1A \cdots O11 ⁱ	0.82 (3)	2.34 (3)	3.115 (4)	157 (3)
N1—H1B \cdots O11	0.86 (3)	2.27 (3)	3.119 (4)	169 (3)
N2—H2A \cdots O21	0.86 (3)	2.30 (3)	3.124 (4)	160 (3)
N2—H2B \cdots O21 ⁱⁱ	0.82 (3)	2.42 (3)	3.219 (4)	166 (3)

Symmetry codes: (i) $-x, y+1/2, -z+1/2$; (ii) $-x+1, y+1/2, -z+1/2$.

Fig. 1

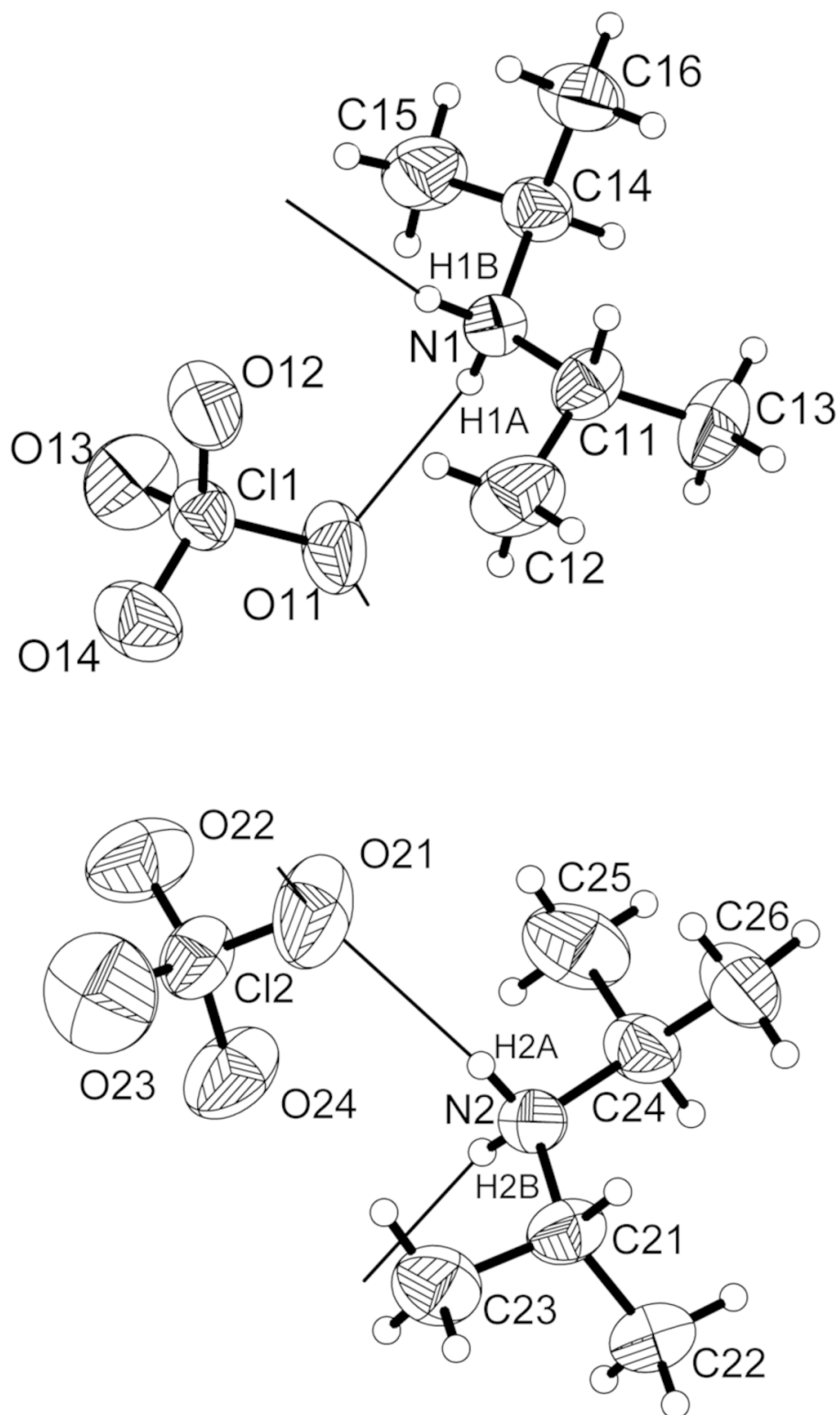


Fig. 2

