Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Redetermination of hydrogenhydrazinium dichloride

Rafal Kruszynski* and Agata Trzesowska

Institute of General and Ecological Chemistry, Technical University of Łódź, ul. Żeromskiego 116, 90-924 Łódź, Poland
Correspondence e-mail: rafal.kruszynski@p.lodz.pl

Received 28 August 2007; accepted 30 August 2007

Key indicators: single-crystal X-ray study; T = 291 K; mean σ(N–N) = 0.001 Å; R factor = 0.017; wR factor = 0.044; data-to-parameter ratio = 12.2.

The crystal structure of the title compound, $N_2H_6^{2+}\cdot 2Cl^-$, previously determined by photographic methods [Wyckoff (1923). *Am. J. Sci.* **5**, 15–22; Donohue & Lipscomb (1947). *J. Chem. Phys.* **15**, 115–119], has been redetermined from single-crystal data. The N and Cl atoms are located on threefold rotation axes, whereas the H atom lies in a general position. The $[H_6N_2]^{2+}$ cations and the Cl^- anions are connected *via* N—H···Cl hydrogen bonds forming a three-dimensional net, whereby $R_6^3(14)$ rings are generated.

Related literature

For previously reported determinations of the title compound, see: Wyckoff (1923); Donohue & Lipscomb (1947). For the crystal structures of hydrazinium chloride, see: Sakurai & Tomiie (1952); Chekhlov & Martynov (1988). For the crystal structure of hydrazine, see: Collin & Lipscomb (1951). For patterns in hydrogen bonding, see: Bernstein *et al.* (1995).

Experimental

Crystal data

$N_2H_6^{2+}\cdot 2Cl^-$
M_r = 104.97
Cubic, $Pa\bar{3}$
a = 7.8731 (1) Å
V = 488.02 (1) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.15 mm^{-1}
T = 291.0 (3) K
0.37 × 0.11 × 0.11 mm

Data collection

Kuma KM4 CCD diffractometer
Absorption correction: numerical (*X-RED*; Stoe & Cie, 1999)
T_{min} = 0.837, T_{max} = 0.880
4076 measured reflections
146 independent reflections
146 reflections with $I > 2\sigma(I)$
R_{int} = 0.015

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.017
$wR(F^2)$ = 0.044
S = 1.34
146 reflections
12 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.42 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—N1^{i}	1.4302 (10)	N1—H1	0.862 (13)
N1^{i}—N1—H1	107.5 (9)		

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

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···Cl1	0.862 (13)	2.242 (13)	3.0944 (3)	170.0 (12)

Data collection: *CrysAlis CCD* (UNIL IC & Kuma, 2000); cell refinement: *CrysAlis RED* (UNIL IC & Kuma, 2000); data reduction: *CrysAlis RED* (UNIL IC & Kuma, 2000); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 1990) and *ORTEP-3* (Farrugia 1997); software used to prepare material for publication: *SHELXL97* (Sheldrick, 1997) and *PLATON* (Spek, 2003).

This work was supported by funds allocated by the Ministry of Science and Higher Education to the Institute of General and Ecological Chemistry, Technical University of Łódź, Poland.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WM2141).

References

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Chekhlov, A. N. & Martynov, I. V. (1988). *Kristallografiya*, **33**, 1010–1011.
Collin, R. L. & Lipscomb, W. N. (1951). *Acta Cryst.* **4**, 10–14.
Donohue, J. & Lipscomb, W. N. (1947). *J. Chem. Phys.* **15**, 115–119.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Sakurai, K. & Tomiie, Y. (1952). *Acta Cryst.* **5**, 293–294.
Sheldrick, G. M. (1990). *SHELXTL*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Stoe & Cie (1999). *X-RED*. Version 1.18. Stoe & Cie GmbH, Darmstadt, Germany.
UNIL IC & Kuma (2000). *CrysAlis CCD* and *CrysAlis RED*. Versions 1.163. Kuma Diffraction Instruments GmbH, Wrocław, Poland.
Wyckoff, R. W. G. (1923). *Am. J. Sci.* **5**, 15–22.

 doi:10.1107/S1600536807042523

supplementary materials

Acta Cryst. (2007). E**63**, i179 [**doi:10.1107/S1600536807042523**]

Redetermination of hydrogenhydrazinium dichloride

R. Kruszynski and A. Trzesowska

Comment

Hydrogenhydrazinium dichloride, or commonly hydrazine dihydrochloride, is the most excellent source of dry hydrazine for organic syntheses. It is commonly used as a reducing agent for the recovery of precious metals or in soldering fluxes for aluminium and magnesium alloys. It is also used as a polymerization catalyst and a chain extender, and is a source of a large number of derivatives, for example used in agrochemicals, pharmaceuticals, photographics, heat stabilizers, polymerization catalysts, flame-retardants, blowing agents for plastics, explosives, and dyes.

The crystal structure of the title compound has been previously determined by photographic methods (Wyckoff, 1923; Donohue & Lipscomb, 1947). The results of the present re-determination show a much higher precision and accuracy. The nitrogen and chlorine atoms are located on threefold rotation axis, whereas the hydrogen atom lies in a general position. The $[H_6N_2]^{2+}$ cations and Cl^- anions are connected *via* N—H···Cl hydrogen bonds (Table) to a 3-D net, generating $R_6^3(14)$ rings (Bernstein *et al.*, 1995). The bond lengths in the title compound (Table) are comparable to those observed for hydrazinium chloride (Sakurai & Tomiie, 1952; Chekhlov & Martynov, 1988) and hydrazine (Collin & Lipscomb, 1951), respectively.

Experimental

Commercially available hydrogenhydrazinium dichloride (pure, Merck, CAS 5341–61-7) was recrystallized from a saturated solution of 0.2 mol/dm^3 hydrochloric acid (about 2.7 g of the title compound in 1 cm^3 of acid).

Refinement

The H atom was found from a difference Fourier synthesis after four cycles of anisotropic refinement for the N and Cl atoms, and was refined freely.

Figures

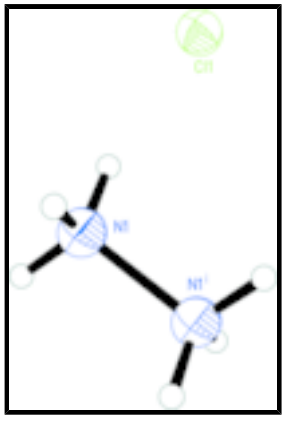

Fig. 1. The molecular structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level. [Symmetry code: (i) −*x* + 1, −*y* + 1, −*z*.]

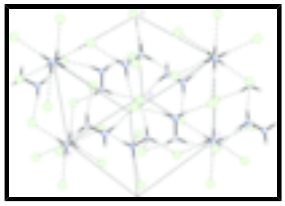

Fig. 2. The packing of the structure units in a view approximately along [111]. Hydrogen bonds are indicated by dashed lines.

Hydrogenhydrazinium dichloride

Crystal data

$N_2H_6^{2+}\cdot 2Cl^-$	$Z = 4$
$M_r = 104.97$	$F_{000} = 216$
	$D_x = 1.429$ Mg m^{-3}
Cubic, $Pa\bar{3}$	$D_m = 1.43$ Mg m^{-3}
	D_m measured by Berman density torsion balance
	Mo $K\alpha$ radiation
Hall symbol: -P 2ac 2ab	$\lambda = 0.71073$ Å
$a = 7.8731$ (1) Å	Cell parameters from 1003 reflections
$b = 7.8731$ (1) Å	$\theta = 4.0$–25.0º
$c = 7.8731$ (1) Å	$\mu = 1.15$ mm^{-1}
$\alpha = 90$º	$T = 291.0$ (3) K
$\beta = 90$º	Needle, colourless
$\gamma = 90$º	0.37 × 0.11 × 0.11 mm
$V = 488.020$ (11) Å^3	

Data collection

Kuma KM4 CCD diffractometer	146 independent reflections
Radiation source: fine-focus sealed tube	146 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.015$
Detector resolution: 1048576 pixels mm^{-1}	$\theta_{max} = 25.1$º
$T = 291.0(3)$ K	$\theta_{min} = 4.5$º
ω scans	$h = -9\rightarrow 9$
Absorption correction: numerical (X-RED; Stoe & Cie, 1999)	$k = -9\rightarrow 9$
$T_{min} = 0.837$, $T_{max} = 0.880$	$l = -9\rightarrow 9$
4076 measured reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	All H-atom parameters refined
$R[F^2 > 2\sigma(F^2)] = 0.017$	$w = 1/[\sigma^2(F_o^2) + (0.0276P)^2 + 0.0263P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.044$	$(\Delta/\sigma)_{max} < 0.001$

S = 1.34	$\Delta\rho_{max}$ = 0.16 e Å^{-3}
146 reflections	$\Delta\rho_{min}$ = −0.42 e Å^{-3}
12 parameters	Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.46 (3)
Secondary atom site location: difference Fourier map	

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}
Cl1	0.77910 (3)	0.27910 (3)	0.22090 (3)	0.0283 (4)
N1	0.55244 (9)	0.44756 (9)	−0.05244 (9)	0.0254 (4)
H1	0.6227 (17)	0.3953 (15)	0.0133 (16)	0.042 (3)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0283 (4)	0.0283 (4)	0.0283 (4)	0.00117 (8)	−0.00117 (8)	−0.00117 (8)
N1	0.0254 (4)	0.0254 (4)	0.0254 (4)	0.0005 (3)	0.0005 (3)	−0.0005 (3)

Geometric parameters (Å, °)

N1—N1^{i}	1.4302 (10)	N1—H1	0.862 (13)
N1^{i}—N1—H1	107.5 (9)		

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···Cl1	0.862 (13)	2.242 (13)	3.0944 (3)	170.0 (12)

Fig. 1

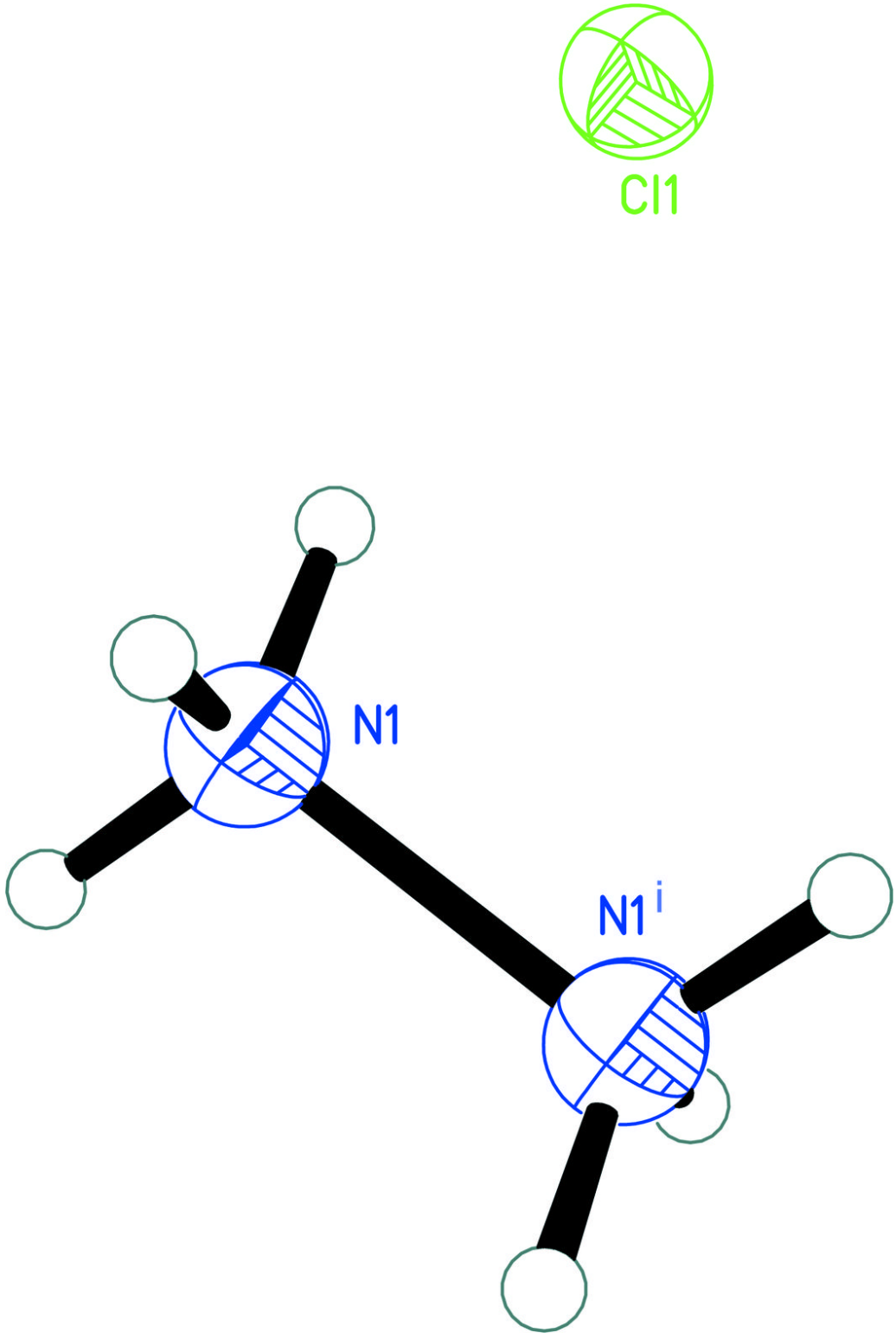

Fig. 2