Acta Crystallographica Section C
Crystal Structure Communications
ISSN 0108-2701

Bis(2-chloro-*N*-methylpyridinium) iodide triiodide

Heigel, Bock and Seibel

Electronic paper
This paper is published electronically. It meets the data-validation criteria for publication in Acta Crystallographica Section C. The submission has been checked by a Section C Co-editor though the text in the 'Comments' section is the responsibility of the authors.

Acta Crystallographica Section C
Crystal Structure Communications
ISSN 0108-2701

Bis(2-chloro-*N*-methylpyridinium) iodide triiodide

Erik Heigel,* Hans Bock and Andreas Seibel

Chemische Institute der Universität Frankfurt, Marie-Curie-Straße 11, 60439 Frankfurt/Main, Germany
Correspondence e-mail: erik.heigel@gmx.de

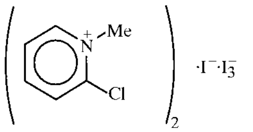
Received 5 November 2000
Accepted 13 November 2000

Data validation number: IUC0000328

The title compound, $2C_6H_7ClN^+\cdot I^-\cdot I_3^-$, crystallizes with undulating layers of chains containing alternate iodide and triiodide anions formed from iodine and the heterocyclic iodide salt.

Comment

Pyridine–iodine compounds are often an interesting starting point for studies of donor–acceptor complexes and salts (Zingaro *et al.*, 1951; Kleinberg *et al.*, 1953; Reid & Mulliken, 1954). *N*-Alkyl-substituted pyridines lose the possibility to coordinate through the nitrogen centre and, therefore, the reaction of 2-chloro-*N*-methylpyridinium iodide with iodine leads to a new iodide salt of a pyridine. The resulting product is a mixed iodide triiodide, (I).



(I)

Many crystal structures of pyridinium as well as triiodide salts are registered in the Crystal Structural Database (Allen & Kennard, 1993), but only 24 structures containing iodide triiodide are known previously. The structure of (I) exhibits a nearly linear triiodide with similar distances I—I of 2.921 (1) and 2.945 (1) Å, and an angle I—I—I of 177.06 (1)°. The I4 atoms with distances $I4\cdots I3^{i} = 4.260$ (1) Å as well as $I4\cdots I2^{ii} =$ 4.103 (1) Å and angles $I4\cdots I3^{i}—I1^{i} = 161.68$ (2)° as well as $I4\cdots I2^{ii}—I1^{ii} = 162.56$ (2)° are arranged in undulating chains of alternate iodide and triiodide anions along the *b* axis, which are generated by a 2_1 screw axis [symmetry codes: (i) $1 + x, \frac{1}{2} - y, -\frac{1}{2} + z$; (ii) $-x, -y, -1 - z$]. These parallel chains form layers separated by 2-chloro-*N*-methylpyridinium cations exhibiting bonds and angles similar to those in the salts *N*-methylpyridinium iodide (Lalancette *et al.*, 1978) and *N*-ethylpyridinium triiodide (Christie *et al.*, 1991). The chlorine substituent in (I) shows an additional weak coordination to the triiodide anion with distances $Cl2\cdots I1 = 3.889$ (2) and $Cl2\cdots I2 = 3.637$ (2) Å.

Experimental

A solution of 2-chloro-*N*-methylpyridinium iodide (0.128 g, 0.5 mmol) in 50 ml trichloromethane was covered with a filter and a solution of iodide (0.5 m*M*) in *n*-pentane added from above. After 10 d, dark-red prisms had grown.

Crystal data

$2C_6H_7ClN^+\cdot I^-\cdot I_3^-$
$M_r = 764.75$
Monoclinic, $P2_1/c$
$a = 7.2902$ (7) Å
$b = 23.644$ (2) Å
$c = 12.0207$ (9) Å
$\beta = 106.448$ (6)°
$V = 1987.2$ (3) Å^3
$Z = 4$
$D_x = 2.556$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 84 reflections
$\theta = 10.0$–15.5°
$\mu = 6.534$ mm^{-1}
$T = 150$ (2) K
Prism, red
0.35 × 0.20 × 0.15 mm

Data collection

Siemens *P*4 four-circle diffractometer
ω scans
Absorption correction: numerical (*SHELXTL*/*PC*; Sheldrick, 1997)
$T_{min} = 0.300$, $T_{max} = 0.474$
7177 measured reflections
4780 independent reflections
4004 reflections with $I > 2\sigma(I)$
$R_{int} = 0.030$
$\theta_{max} = 28°$
$h = -2 \rightarrow 9$
$k = -2 \rightarrow 31$
$l = -15 \rightarrow 15$
4 standard reflections every 100 reflections intensity decay: <5%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.085$
$S = 1.095$
4780 reflections
184 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0342P)^2 + 9.6004P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.87$ e Å^{-3}
$\Delta\rho_{min} = -1.41$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.00156 (11)

Table 1
Selected geometric parameters (Å, °).

I1—I2	2.9213 (6)	N1—C7	1.501 (7)
I1—I3	2.9446 (6)	Cl2—C11	1.704 (6)
Cl1—C1	1.704 (6)	N2—C15	1.347 (7)
N1—C1	1.334 (8)	N2—C11	1.347 (8)
N1—C5	1.356 (8)	N2—C17	1.477 (7)
I2—I1—I3	177.055 (18)	C2—C1—Cl1	119.1 (5)
C1—N1—C5	119.3 (5)	C15—N2—C17	118.9 (5)
C1—N1—C7	122.6 (5)	C11—N2—C17	121.5 (5)
C5—N1—C7	118.1 (5)	N2—C11—Cl2	117.8 (4)
N1—C1—Cl1	118.8 (5)	C12—C11—Cl2	120.8 (5)

All H atom were located from the difference map and placed in idealized positions using a riding model with distances $C—H_{phenyl} =$ 0.95 Å and $C—H_{methyl} = 0.98$ Å. The torsion of the methyl H atoms and the isotropic displacement parameters of all H atoms were refined.

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); software used to prepare material for publication: *SHELXL*97.

The project has been supported by the A. Messer Foundation, the Deutsche Forschungsgemeinschaft, the State of Hesse and the Fonds der Chemischen Industrie.

References

Allen, F. H. & Kennard, O. (1993). *Chem. Des. Autom. News*, **8**, 1, 31–37.

Christie, S., Dubois, R. H., Rogers, R. D., White, P. S. & Zaworotko, M. J. (1991). *J. Incl. Phenom. Mol. Recognit. Chem.* **11**, 103–114.

Kleinberg, J., Colton, E., Sattizahn, J. & van der Werf, C. A. (1953). *J. Am. Chem. Soc.* **75**, 442–445.

Lalancette, R. A., Furey, W., Constanzo, J. N., Hemmes, P. R. & Jordan, F. (1978). *Acta Cryst.* B**34**, 2950–2953.

Reid, C. & Mulliken, R. S. (1954). *J. Am. Chem. Soc.* **76**, 3869–3874.

Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.

Sheldrick, G. M. (1997). *SHELXL*97 and *SHELXTL/PC*. University of Göttingen, Germany.

Siemens (1994). *XSCANS*. Version 2.10b. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Zingaro, R., van der Werf, C. A. & Kleinberg, J. (1951). *J. Am. Chem. Soc.* **73**, 88–90.