

C5'	0.2887 (3)	0.2998 (3)	0.9692 (5)	0.052
C6'	0.2234 (3)	0.2232 (3)	0.8362 (6)	0.061
C7'	0.1325 (3)	0.2225 (3)	0.7364 (6)	0.063
C8'	0.1056 (3)	0.3002 (3)	0.7658 (5)	0.055
C8a'	0.1706 (2)	0.3766 (3)	0.8992 (4)	0.043
C9'	0.1548 (2)	0.4658 (3)	0.9540 (5)	0.046
C9a'	0.1747 (2)	0.4670 (3)	1.1475 (5)	0.044
C10'	0.3255 (2)	0.4660 (3)	1.1446 (4)	0.042
C10a'	0.2626 (2)	0.3766 (3)	1.0020 (4)	0.043
C11'	0.2352 (2)	0.5530 (3)	0.9455 (4)	0.045
C12'	0.3227 (2)	0.5521 (3)	1.0422 (4)	0.044
C13'	0.4264 (3)	0.4681 (3)	1.2550 (5)	0.057
C14'	0.2217 (3)	0.6326 (3)	0.8405 (5)	0.055
C15'	0.1114 (4)	0.7011 (4)	0.6689 (7)	0.097
C16'	0.4147 (3)	0.6212 (3)	1.0478 (5)	0.049
C17'	0.5472 (3)	0.7638 (4)	1.1969 (7)	0.080
O1'	0.1288 (2)	0.6207 (2)	0.7610 (4)	0.067
O2'	0.2846 (2)	0.6975 (2)	0.8298 (4)	0.072
O3'	0.4543 (2)	0.6967 (2)	1.1762 (4)	0.062
O4'	0.4500 (2)	0.6061 (2)	0.9518 (4)	0.064

Table 2. Selected bond lengths (Å) and ring-junction angles (°) with e.s.d.'s in parentheses

C=C	1.331, 1.333(5)	C=O	1.175–1.201(4)
C—C (aromatic)	1.358–1.416(6)	C—OMe	1.329–1.340(5)
C—CO <sub>2</sub> Me	1.488–1.509(6)	O—Me	1.447–1.488(6)
C—C (other)	1.510–1.550(6)		

External	125.7–127.2(3)
Internal (non-aromatic)	112.6–113.9(3)
C=C—CO <sub>2</sub> Me	120.2, 121.0(3) (at C11)
	125.8, 126.0(3) (at C12)
CH <sub>3</sub> —C—C	114.1–114.7(3)

The triclinic cell used was determined by the CAD-4 software; the transformation (001/010/101) produces the type I reduced cell (*International Tables for Crystallography*, Volume A, page 739) with  $a = 8.3073$ ,  $b = 14.747$ ,  $c = 15.038$  Å,  $\alpha = 69.29$ ,  $\beta = 82.21$ ,  $\gamma = 89.14^\circ$ .

Peaks of about  $0.8 \text{ e} \text{ \AA}^{-3}$  in the final difference map indicate minor disorder of one methoxycarbonyl group in each molecule; this disorder could not be satisfactorily modelled and is the reason for the slightly high  $R$  factors.

We thank the Natural Sciences and Engineering Research Council of Canada for financial support.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry together with details of the synthesis and stereo molecular and packing diagrams have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55869 (44 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: BR1025]

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*Acta Cryst.* (1993). **C49**, 973–975

## Structure of Dimethylammonium $N,N,N',N'$ -Tetramethylformamidinium Hexachlorotellurate(IV)

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### Abstract

The structure consists of three ionic species. The anion,  $\text{TeCl}_6^{2-}$ , is a slightly distorted octahedron with average  $\text{Te—Cl} = 2.532$  Å. One of the cations,  $(\text{CH}_3)_2\text{NH}_2^+$ , has  $\text{C—N} = 1.44$  (1) Å and  $\text{C—N—C} = 114.8$  (6)°. The other cation is the formamidinium [dimethyl(dimethylaminomethylene)ammonium] ion,  $(\text{CH}_3)_2\text{NC}(\text{H})\text{N}(\text{CH}_3)_2^+$ , with the central  $\text{C—N} = 1.30$  (1) Å,  $\text{N—C—N} = 129.8$  (8)° and the peripheral  $\text{C—N} = 1.46$  (1) Å. Planes through the terminal  $\text{C—N—C}$  atoms make angles of 14 and  $-14^\circ$ , respectively, relative to the best plane through all the non-H atoms of this ion. There are two short distances between the N atom of  $(\text{CH}_3)_2\text{NH}_2^+$  and Cl atoms of two different  $\text{TeCl}_6^{2-}$  units, directed approximately along the lines  $\text{N—H}\cdots\text{Cl}$  and with  $\text{N}\cdots\text{Cl} = 3.36, 3.44$  Å. There are also two short distances between the central C atom of  $(\text{CH}_3)_2\text{NC}(\text{H})\text{N}(\text{CH}_3)_2^+$  and Cl atoms of two different  $\text{TeCl}_6^{2-}$  units. The vector from one Cl to C atom is nearly perpendicular to the best plane through the N and C atoms of the ion,  $\text{C}\cdots\text{Cl} = 3.34$  Å. The vector from the other Cl to C is directed approximately along the line  $\text{C—H}\cdots\text{Cl}$  with  $\text{C}\cdots\text{Cl} = 3.68$  Å.

### Comment

The preparation of  $\text{C}_7\text{H}_{21}\text{Cl}_6\text{N}_3\text{Te}$  from  $N,N$ -dimethylformamide and  $\text{TeCl}_4$  was reported by Zingaro, Herrera & Meyers (1990). The structure of  $\text{TeCl}_6^{2-}$  in general positions and in the presence of unsymmetrical cations is that of a distorted octahedron (Abriel, 1987). Based on covalent radii taken from Pauling (1960), the expected  $\text{Te—Cl}$  distance is 2.51 Å, compared to the average value found here of 2.532 Å and a typical experimental value of 2.54 Å for  $(\text{NH}_4)_2\text{TeCl}_6$  (Hazell, 1966). The distortions are systematic with long and short  $\text{Te—Cl}$  distances approximately  $180^\circ$  from each other. In addition, the longest  $\text{Te—Cl}$  distances, 2.570 (2) and 2.618 (2) Å,

† Deceased.

are found between Te and those Cl atoms that are nearest to the cations. The cation  $(\text{CH}_3)_2\text{NH}_2^+$  has been observed in many structures such as that determined from the X-ray and neutron diffraction studies of dimethylammonium hydrogen oxalate at 298 K by Thomas & Pramatus (1975) and Thomas (1977). Compared to these studies the present structure shows a considerable shortening of the C—N bonds from 1.47 to 1.44 Å. This effect seems to be associated with large thermal parameters of the methyl groups. The cation  $(\text{CH}_3)_2\text{NC}(\text{H})\text{N}(\text{CH}_3)_2^+$  is more unusual and has been observed less frequently. A recent study by Benetollo, Polo, Conte & Guerrato (1987) gives structural parameters for this ion that are in excellent agreement with those obtained here.

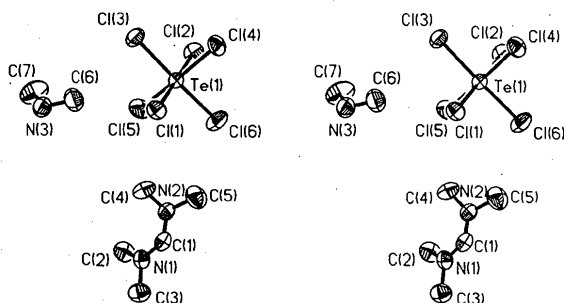
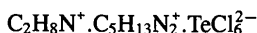


Fig. 1. Stereoview of dimethylammonium tetramethylformamidinium hexachlorotellurate(IV) with thermal ellipsoids at 0.50 probability.

## Experimental

### Crystal data



$M_r = 487.6$

Monoclinic

$P2_1/n$

$a = 13.698(4) \text{ \AA}$

$b = 8.944(4) \text{ \AA}$

$c = 16.67(1) \text{ \AA}$

$\beta = 112.02(4)^\circ$

$V = 1893(1) \text{ \AA}^3$

$Z = 4$

$D_x = 1.711 \text{ Mg m}^{-3}$

Mo radiation

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

Cell parameters from 25 reflections

$\theta = 3\text{--}10^\circ$

$\mu = 2.42 \text{ mm}^{-1}$

$T = 296 \text{ K}$

Prism  
 $0.25 \times 0.20 \times 0.15 \text{ mm}$

Yellow

Crystal source: from  $\text{TeCl}_4$  and  $\text{Me}_2\text{NCHO}$  under reflux

### Data collection

Enraf-Nonius CAD-4 diffractometer

$\theta$ - $2\theta$  scans,  $\omega$ -scan range  
 $0.7^\circ$  plus  $K\alpha$  separation, variable scan rate  
 $2\text{--}20^\circ \text{ min}^{-1}$

Absorption correction: none

4618 measured reflections

4618 independent reflections

3377 observed reflections  
 $[I \geq 0]$

$\theta_{\text{max}} = 55^\circ$

$h = 0 \rightarrow 17$

$k = 0 \rightarrow 11$

$l = -21 \rightarrow 20$

3 standard reflections

frequency: 41 min  
intensity variation: none

## Refinement

Refinement on  $F$

Final  $R = 0.0766$

$wR = 0.0423$

$S = 1.91$

3377 reflections

154 parameters

H-atom parameters not refined

$w = \sigma^{-2}$

$(\Delta/\sigma)_{\text{max}} = 0.001$

$\Delta\rho_{\text{max}} = 1.02 \text{ e \AA}^{-3}$

$\Delta\rho_{\text{min}} = -1.06 \text{ e \AA}^{-3}$

Atomic scattering factors

from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters ( $\text{\AA}^2$ )

$$U_{\text{eq}} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	$U_{\text{eq}}$
Te(1)	0.2427 (1)	0.2086 (1)	0.0212 (1)	0.041 (1)
Cl(1)	0.3629 (1)	0.4159 (2)	0.1009 (1)	0.059 (1)
Cl(2)	0.1210 (2)	-0.0072 (2)	-0.0584 (1)	0.068 (1)
Cl(3)	0.1246 (2)	0.2473 (2)	0.1050 (1)	0.069 (1)
Cl(4)	0.1246 (2)	0.3929 (2)	-0.0977 (1)	0.064 (1)
Cl(5)	0.3467 (2)	0.0277 (3)	0.1315 (1)	0.082 (1)
Cl(6)	0.3543 (2)	0.1627 (3)	-0.0662 (1)	0.083 (1)
N(1)	0.8604 (5)	0.1976 (7)	0.2308 (3)	0.053 (3)
N(2)	0.6857 (5)	0.2359 (6)	0.1332 (4)	0.052 (3)
N(3)	0.4039 (5)	0.2959 (8)	0.3979 (4)	0.073 (3)
C(1)	0.7880 (6)	0.2554 (7)	0.1633 (5)	0.051 (3)
C(2)	0.6255 (6)	0.1650 (8)	0.1776 (5)	0.072 (4)
C(3)	0.8492 (6)	0.0593 (8)	0.2731 (5)	0.076 (4)
C(4)	0.6227 (6)	0.2882 (10)	0.0464 (5)	0.089 (4)
C(5)	0.9683 (5)	0.2540 (8)	0.2584 (5)	0.075 (4)
C(6)	0.3668 (7)	0.3418 (10)	0.3087 (5)	0.092 (5)
C(7)	0.3529 (7)	0.1661 (10)	0.4143 (7)	0.115 (6)

Table 2. Geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

Te(1)—Cl(1)	2.505 (2)	Te(1)—Cl(2)	2.570 (2)
Te(1)—Cl(3)	2.527 (3)	Te(1)—Cl(4)	2.618 (2)
Te(1)—Cl(5)	2.463 (3)	Te(1)—Cl(6)	2.510 (3)
N(1)—C(1)	1.296 (8)	N(1)—C(2)	1.460 (10)
N(1)—C(3)	1.464 (9)	N(2)—C(1)	1.311 (10)
N(2)—C(4)	1.445 (11)	N(2)—C(5)	1.456 (9)
N(3)—C(6)	1.440 (10)	N(3)—C(7)	1.433 (12)
Cl(1)—Te(1)—Cl(2)	179.0 (1)	Cl(1)—Te(1)—Cl(3)	92.0 (1)
Cl(2)—Te(1)—Cl(3)	87.8 (1)	Cl(1)—Te(1)—Cl(4)	91.8 (1)
Cl(2)—Te(1)—Cl(4)	89.2 (1)	Cl(3)—Te(1)—Cl(4)	89.4 (1)
Cl(1)—Te(1)—Cl(5)	90.5 (1)	Cl(2)—Te(1)—Cl(5)	88.5 (1)
Cl(3)—Te(1)—Cl(5)	89.3 (1)	Cl(4)—Te(1)—Cl(5)	177.4 (1)
Cl(1)—Te(1)—Cl(6)	90.4 (1)	Cl(2)—Te(1)—Cl(6)	89.8 (1)
Cl(3)—Te(1)—Cl(6)	177.6 (1)	Cl(4)—Te(1)—Cl(6)	90.1 (1)
Cl(5)—Te(1)—Cl(6)	91.0 (1)	C(1)—N(1)—C(2)	124.9 (6)
C(1)—N(1)—C(3)	119.3 (7)	C(2)—N(1)—C(3)	114.6 (5)
C(1)—N(2)—C(4)	126.5 (6)	C(1)—N(2)—C(5)	119.2 (7)
C(4)—N(2)—C(5)	114.3 (6)	C(6)—N(3)—C(7)	114.8 (6)
N(1)—C(1)—N(2)	129.8 (8)		

Data collection: Preliminary examination and data collection were performed by the Molecular Structure Corporation (The Woodlands, Texas, USA), on a sample mounted in a glass capillary. The structure was solved by Patterson methods and final calculations were carried out with the *SHELXTL-Plus* programs (Sheldrick, 1990). Full-matrix least-squares refinement of coordinates and anisotropic temperature factors for non-H atoms was carried out and H atoms were assigned idealized positions at 0.96 Å from the C or N atom to which they were attached.

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assistance of Dr J. H. Reibenspies in the use of the crystallographic programs is greatly appreciated.

Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55809 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR1030]

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*Acta Cryst.* (1993). **C49**, 975–976

## Bis(3-chloro-2-pyridyl) Ditelluride

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## Abstract

The entire ditelluride molecule is planar with an average deviation of its non-H atoms of 0.016 Å. It is located on a center of symmetry at  $(\frac{1}{2}, 0, 0)$  with Te—Te<sub>A</sub> distance 2.725 (1) Å and angle C(1)—Te—Te<sub>A</sub> 85.8 (1)°. Each half of the molecule consists of a substituted pyridine ring with average C—N distance 1.334 (6) Å, C—C distance 1.38 (1) Å and angle C—N—C 117.5 (4)°. The ring is substituted in the 2-position [Te—C(1) distance 2.121 (3) Å] and in the 3-position [Cl—C(2) distance 1.736 (4) Å]. All of these values appear to be normal and there are no abnormally short intermolecular distances present in the structure.

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## Comment

The preparation of the compound is described by Junk (1988). The solid is extremely deep blue in color (as opposed to the orange-to-red colors observed for aryl ditellurides) and the structure has been examined for any structural features that might be associated with its unusual color. The pyridine ring itself has dimensions slightly distorted from those of gaseous pyridine (Bak, Hansen-Nygaard & Rastrup-Anderson, 1958). The C(2)—Cl distance of 1.736 (4) Å is only slightly longer than expected (Drendel & Sundaralingam, 1985), as is the Te—Te<sub>A</sub> distance of 2.725 (1) Å; but the angle Te<sub>A</sub>—Te—C(1) of 85.8 (1)° differs greatly from those found in alkyl ditellurides [97–102° (Van den Bossche, Spirlet, Dideberg & Dupont, 1984, and structures reviewed therein)]. It seems likely that the blue color of the present molecule is associated with its extended planar structure and the possibility of interaction between its two pyridine rings since, in the aryl ditellurides, the aromatic rings are twisted from coplanarity and the possibility of extended conjugation is greatly reduced.

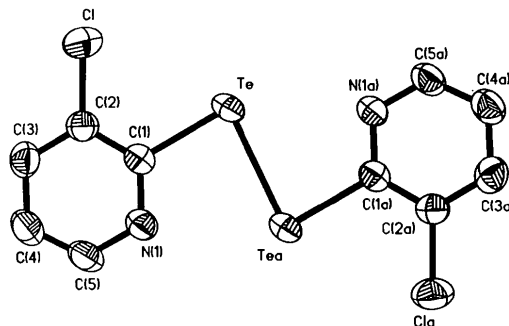


Fig. 1. Structure of bis(3-chloro-2-pyridyl) ditelluride drawn with thermal ellipsoids at 0.50 probability.

## Experimental

### Crystal data

C<sub>10</sub>H<sub>6</sub>Cl<sub>2</sub>N<sub>2</sub>Te<sub>2</sub>  
*M<sub>r</sub>* = 480.3  
 Monoclinic  
*P*2<sub>1</sub>/c  
*a* = 7.298 (2) Å  
*b* = 12.542 (3) Å  
*c* = 7.944 (2) Å  
 $\beta$  = 116.05 (2)°  
*V* = 653.3 (2) Å<sup>3</sup>  
*Z* = 2

*D<sub>x</sub>* = 2.442 Mg m<sup>-3</sup>

Mo *K* $\alpha$  radiation

$\lambda$  = 0.71073 Å

Cell parameters from 25 reflections

$\theta$  = 4–11°

$\mu$  = 4.865 mm<sup>-1</sup>

*T* = 296 K

Prism

0.30 × 0.20 × 0.20 mm

Dark blue

### Data collection

Enraf–Nonius CAD-4 diffractometer

1779 observed reflections [*I* > 0]