

Chlorido{2-[(dimethylamino)methyl]-phenyl- κ^2N,C^1 }tellurium

Prakul Rakesh,^a Harkesh B. Singh^a and Ray J. Butcher^{b*}

^aDepartment of Chemistry, Indian Institute of Technology Bombay, Powai, Mumbai 400 076, India, and ^bDepartment of Chemistry, Howard University, 525 College Street NW, Washington, DC 20059, USA

Correspondence e-mail: rbutcher99@yahoo.com

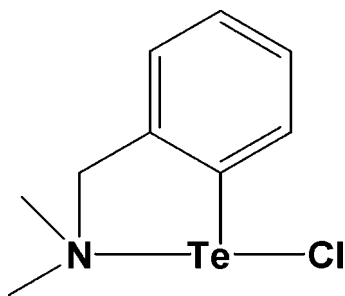
Received 7 December 2011; accepted 17 December 2011

Key indicators: single-crystal X-ray study; $T = 295$ K; mean $\sigma(C-C) = 0.006$ Å; R factor = 0.043; wR factor = 0.096; data-to-parameter ratio = 32.3.

The crystal structure of the title compound, $C_9H_{12}ClNTe$, contains isolated molecules with no close $Te \cdots Cl$ intermolecular contacts and has the same composition as a previously published structure [Engman *et al.* (2004). *Phosphorus Sulfur Silicon Relat. Elem.* **179**, 285–292]. However, in this case, the compound has crystallized in a centrosymmetric space group, unlike the previously published structure which contained enantiomerically pure chiral molecules. In all other aspects, the metrical parameters are similar. The molecules with a T-shaped coordination environment about the Te atom are linked into dimers by $C-H \cdots Cl$ interactions.

Related literature

For a related structure, see: Engman *et al.* (2004). For related syntheses, see: Singh *et al.* (1990); Kaur *et al.* (1995).



Experimental

Crystal data

$C_9H_{12}ClNTe$
 $M_r = 297.25$

Monoclinic, $P2_1/n$
 $a = 6.4514$ (6) Å

$b = 7.0287$ (7) Å
 $c = 23.847$ (2) Å
 $\beta = 95.967$ (9)°
 $V = 1075.49$ (17) Å³
 $Z = 4$

Mo $K\alpha$ radiation
 $\mu = 2.96$ mm⁻¹
 $T = 295$ K
 $0.45 \times 0.36 \times 0.12$ mm

Data collection

Oxford Diffraction Xcalibur Ruby Gemini diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2007)
 $T_{min} = 0.504$, $T_{max} = 1.000$

7778 measured reflections
3587 independent reflections
2998 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.023$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.043$
 $wR(F^2) = 0.096$
 $S = 1.20$
3587 reflections

111 parameters
H-atom parameters constrained
 $\Delta\rho_{max} = 2.25$ e Å⁻³
 $\Delta\rho_{min} = -0.98$ e Å⁻³

Table 1

Selected bond lengths (Å).

Te—Cl	2.116 (3)	Te—Cl	2.5657 (11)
Te—N	2.355 (3)		

Table 2

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$C9-H9C \cdots Cl^i$	0.96	2.89	3.822 (5)	163

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

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2007); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

RJB acknowledges the NSF–MRI program (grant CHE-0619278) for funds to purchase the diffractometer. HBS acknowledges the DST (New Delhi) for funding. PR acknowledges the CSIR for a fellowship.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PK2376).

References

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Kaur, R., Singh, H. B. & Butcher, R. J. (1995). *Organometallics*, **14**, 4755–4763.
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supplementary materials

Acta Cryst. (2012). E68, o214 [doi:10.1107/S1600536811054328]

Chlorido{2-[(dimethylamino)methyl]phenyl- κ^2N,C^1 }tellurium

P. Rakesh, H. B. Singh and R. J. Butcher

Comment

Attempts to synthesize protonated bis[2-(dimethylaminomethyl)phenyl]ditelluride resulted in the formation of 2-(*N,N*-dimethylaminomethyl)phenyltellurenyl chloride, C₉H₁₂ClN₂Te, (**1**) a compound which had been previously prepared *via* a different method (Singh *et al.*, 1990). The structure of C₉H₁₂ClN₂Te contains isolated molecules with no close Te...Cl intermolecular contacts, and is chemically related to a previously published structure (Engman, *et al.*, 2004), even though it had been prepared by the same method as the title compound. However, in this case the compound has crystallized in a centrosymmetric space group, unlike the previously published structure (Engman, *et al.*, 2004) which contained enantiomerically pure, chiral molecules. In all other aspects the metrical parameters are similar. The molecules are linked into dimers by C—H...Cl interactions.

Experimental

A stirred solution of bis[2-(dimethylaminomethyl)phenyl]ditelluride (Kaur *et al.*, 1995) (0.5 g, 0.94 mmol) in diethylether (10 ml) was treated with HCl (3 ml in 20 ml distilled water). The reaction mixture was further stirred for 10 min. The resulting reaction mixture was evaporated to one third of its original volume and ethanol (5 ml) was added to get a yellow solid. It was redissolved in ethanol and stored in the refrigerator to get yellow needles of X-ray quality crystals. Yield 0.2 g, 35% mp 121 °C (lit value 121 °C). Anal. Calcd for C₉H₁₂ClN₂Te: C, 36.37; H, 4.07; N, 4.37. Found C, 36.44; H, 4.04; N, 4.66.

Refinement

H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms with C—H distances of 0.95 - 0.97 Å [$U_{iso}(H) = 1.2U_{eq}(CH, CH_2)$] [$U_{iso}(H) = 1.5U_{eq}(CH_3)$].

Figures

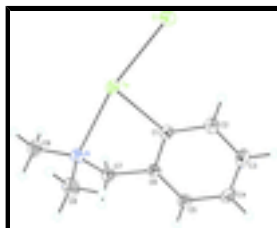


Fig. 1. Diagram of the contents of the asymmetric unit of C₉H₁₂ClN₂Te. Atomic displacement ellipsoids are drawn at the 30% level.



Fig. 2. The molecular packing for C₉H₁₂ClN₂Te viewed down the *c* axis. C—H...Cl secondary interactions are shown by dashed lines.

Chlorido{2-[(dimethylamino)methyl]phenyl- κ^2 N,C¹}tellurium

Crystal data

C ₉ H ₁₂ ClNTe	$F(000) = 568$
$M_r = 297.25$	$D_x = 1.836 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
Hall symbol: -P 2yn	Cell parameters from 3931 reflections
$a = 6.4514 (6) \text{ \AA}$	$\theta = 5.1\text{--}32.6^\circ$
$b = 7.0287 (7) \text{ \AA}$	$\mu = 2.96 \text{ mm}^{-1}$
$c = 23.847 (2) \text{ \AA}$	$T = 295 \text{ K}$
$\beta = 95.967 (9)^\circ$	Irregular plate, pale yellow
$V = 1075.49 (17) \text{ \AA}^3$	$0.45 \times 0.36 \times 0.12 \text{ mm}$
$Z = 4$	

Data collection

Oxford Diffraction Xcalibur Ruby Gemini diffractometer	3587 independent reflections
Radiation source: fine-focus sealed tube graphite	2998 reflections with $I > 2\sigma(I)$
Detector resolution: $10.5081 \text{ pixels mm}^{-1}$	$R_{\text{int}} = 0.023$
ω scans	$\theta_{\text{max}} = 32.7^\circ$, $\theta_{\text{min}} = 5.1^\circ$
Absorption correction: multi-scan (<i>Crys.Alis PRO</i> ; Oxford Diffraction, 2007)	$h = -9 \rightarrow 9$
$T_{\text{min}} = 0.504$, $T_{\text{max}} = 1.000$	$k = -6 \rightarrow 10$
7778 measured reflections	$l = -35 \rightarrow 34$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.043$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.096$	H-atom parameters constrained
$S = 1.20$	$w = 1/[\sigma^2(F_o^2) + (0.0327P)^2 + 1.2847P]$
3587 reflections	where $P = (F_o^2 + 2F_c^2)/3$
111 parameters	$(\Delta/\sigma)_{\text{max}} = 0.001$
0 restraints	$\Delta\rho_{\text{max}} = 2.25 \text{ e \AA}^{-3}$
	$\Delta\rho_{\text{min}} = -0.98 \text{ e \AA}^{-3}$

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 $F^2 > \sigma(F^2)$ 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	$U_{\text{iso}}^*/U_{\text{eq}}$
Te	0.58681 (4)	0.01772 (4)	0.423777 (10)	0.03896 (9)
Cl	0.29601 (17)	-0.23269 (17)	0.41307 (5)	0.0537 (3)
N	0.8374 (5)	0.2560 (4)	0.41417 (13)	0.0372 (6)
C1	0.5512 (6)	0.0787 (5)	0.33640 (14)	0.0347 (7)
C2	0.3769 (6)	0.0346 (6)	0.29976 (16)	0.0413 (8)
H2A	0.2649	-0.0291	0.3127	0.050*
C3	0.3712 (7)	0.0865 (7)	0.24370 (18)	0.0505 (10)
H3A	0.2543	0.0571	0.2190	0.061*
C4	0.5367 (8)	0.1816 (6)	0.22353 (17)	0.0515 (10)
H4A	0.5311	0.2154	0.1857	0.062*
C5	0.7099 (7)	0.2255 (5)	0.26028 (16)	0.0456 (9)
H5A	0.8212	0.2899	0.2471	0.055*
C6	0.7191 (6)	0.1741 (5)	0.31665 (15)	0.0366 (7)
C7	0.9058 (6)	0.2144 (6)	0.35786 (15)	0.0416 (8)
H7A	0.9982	0.1052	0.3604	0.050*
H7B	0.9816	0.3225	0.3450	0.050*
C8	0.7367 (8)	0.4460 (6)	0.4157 (2)	0.0588 (12)
H8A	0.8373	0.5433	0.4104	0.088*
H8B	0.6837	0.4636	0.4515	0.088*
H8C	0.6240	0.4539	0.3861	0.088*
C9	1.0127 (6)	0.2409 (7)	0.45859 (18)	0.0538 (11)
H9A	1.1119	0.3397	0.4537	0.081*
H9B	1.0786	0.1191	0.4562	0.081*
H9C	0.9620	0.2539	0.4948	0.081*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Te	0.03675 (13)	0.04765 (15)	0.03311 (13)	0.00064 (11)	0.00659 (8)	0.00140 (11)
Cl	0.0484 (5)	0.0601 (6)	0.0536 (6)	-0.0112 (5)	0.0102 (4)	0.0036 (5)
N	0.0340 (14)	0.0375 (15)	0.0403 (16)	0.0027 (12)	0.0043 (12)	-0.0043 (13)
C1	0.0397 (17)	0.0331 (15)	0.0320 (16)	0.0061 (14)	0.0069 (13)	-0.0021 (13)
C2	0.0409 (18)	0.0428 (19)	0.0399 (19)	0.0057 (16)	0.0039 (14)	-0.0059 (16)
C3	0.057 (2)	0.050 (2)	0.042 (2)	0.014 (2)	-0.0078 (18)	-0.0079 (18)
C4	0.079 (3)	0.039 (2)	0.0348 (19)	0.010 (2)	0.0016 (19)	0.0035 (16)
C5	0.066 (3)	0.0367 (18)	0.0357 (19)	-0.0026 (18)	0.0112 (17)	0.0009 (15)
C6	0.0438 (18)	0.0320 (16)	0.0348 (17)	-0.0002 (14)	0.0071 (14)	-0.0014 (14)

supplementary materials

C7	0.0404 (18)	0.049 (2)	0.0364 (18)	-0.0033 (16)	0.0097 (14)	-0.0003 (16)
C8	0.071 (3)	0.043 (2)	0.061 (3)	0.007 (2)	0.000 (2)	-0.012 (2)
C9	0.042 (2)	0.074 (3)	0.043 (2)	-0.007 (2)	-0.0038 (17)	-0.004 (2)

Geometric parameters (Å, °)

Te—C1	2.116 (3)	C4—H4A	0.9300
Te—N	2.355 (3)	C5—C6	1.387 (5)
Te—Cl	2.5657 (11)	C5—H5A	0.9300
N—C9	1.471 (5)	C6—C7	1.500 (5)
N—C7	1.486 (5)	C7—H7A	0.9700
N—C8	1.487 (5)	C7—H7B	0.9700
C1—C2	1.385 (5)	C8—H8A	0.9600
C1—C6	1.397 (5)	C8—H8B	0.9600
C2—C3	1.383 (6)	C8—H8C	0.9600
C2—H2A	0.9300	C9—H9A	0.9600
C3—C4	1.387 (7)	C9—H9B	0.9600
C3—H3A	0.9300	C9—H9C	0.9600
C4—C5	1.382 (6)		
C1—Te—N	76.45 (13)	C6—C5—H5A	119.8
C1—Te—Cl	92.14 (10)	C5—C6—C1	119.7 (4)
N—Te—Cl	168.59 (8)	C5—C6—C7	122.4 (3)
C9—N—C7	111.0 (3)	C1—C6—C7	117.9 (3)
C9—N—C8	110.7 (3)	N—C7—C6	109.6 (3)
C7—N—C8	111.7 (3)	N—C7—H7A	109.7
C9—N—Te	111.3 (3)	C6—C7—H7A	109.7
C7—N—Te	102.7 (2)	N—C7—H7B	109.7
C8—N—Te	109.3 (3)	C6—C7—H7B	109.7
C2—C1—C6	120.1 (3)	H7A—C7—H7B	108.2
C2—C1—Te	125.0 (3)	N—C8—H8A	109.5
C6—C1—Te	114.9 (3)	N—C8—H8B	109.5
C3—C2—C1	119.3 (4)	H8A—C8—H8B	109.5
C3—C2—H2A	120.4	N—C8—H8C	109.5
C1—C2—H2A	120.4	H8A—C8—H8C	109.5
C2—C3—C4	121.2 (4)	H8B—C8—H8C	109.5
C2—C3—H3A	119.4	N—C9—H9A	109.5
C4—C3—H3A	119.4	N—C9—H9B	109.5
C5—C4—C3	119.3 (4)	H9A—C9—H9B	109.5
C5—C4—H4A	120.4	N—C9—H9C	109.5
C3—C4—H4A	120.4	H9A—C9—H9C	109.5
C4—C5—C6	120.5 (4)	H9B—C9—H9C	109.5
C4—C5—H5A	119.8		
C1—Te—N—C9	-152.7 (3)	C2—C3—C4—C5	-0.2 (6)
Cl—Te—N—C9	-154.3 (3)	C3—C4—C5—C6	0.4 (6)
C1—Te—N—C7	-33.9 (2)	C4—C5—C6—C1	-0.5 (6)
Cl—Te—N—C7	-35.5 (5)	C4—C5—C6—C7	178.5 (4)
C1—Te—N—C8	84.8 (3)	C2—C1—C6—C5	0.3 (5)
Cl—Te—N—C8	83.2 (5)	Te—C1—C6—C5	-178.4 (3)
N—Te—C1—C2	-160.8 (3)	C2—C1—C6—C7	-178.7 (3)

Cl—Te—C1—C2	18.9 (3)	Te—C1—C6—C7	2.6 (4)
N—Te—C1—C6	17.8 (2)	C9—N—C7—C6	163.4 (3)
Cl—Te—C1—C6	-162.5 (3)	C8—N—C7—C6	-72.6 (4)
C6—C1—C2—C3	-0.1 (5)	Te—N—C7—C6	44.4 (3)
Te—C1—C2—C3	178.4 (3)	C5—C6—C7—N	145.9 (3)
C1—C2—C3—C4	0.0 (6)	C1—C6—C7—N	-35.1 (5)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C9—H9C...Cl ⁱ	0.96	2.89	3.822 (5)	163.

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

Fig. 1

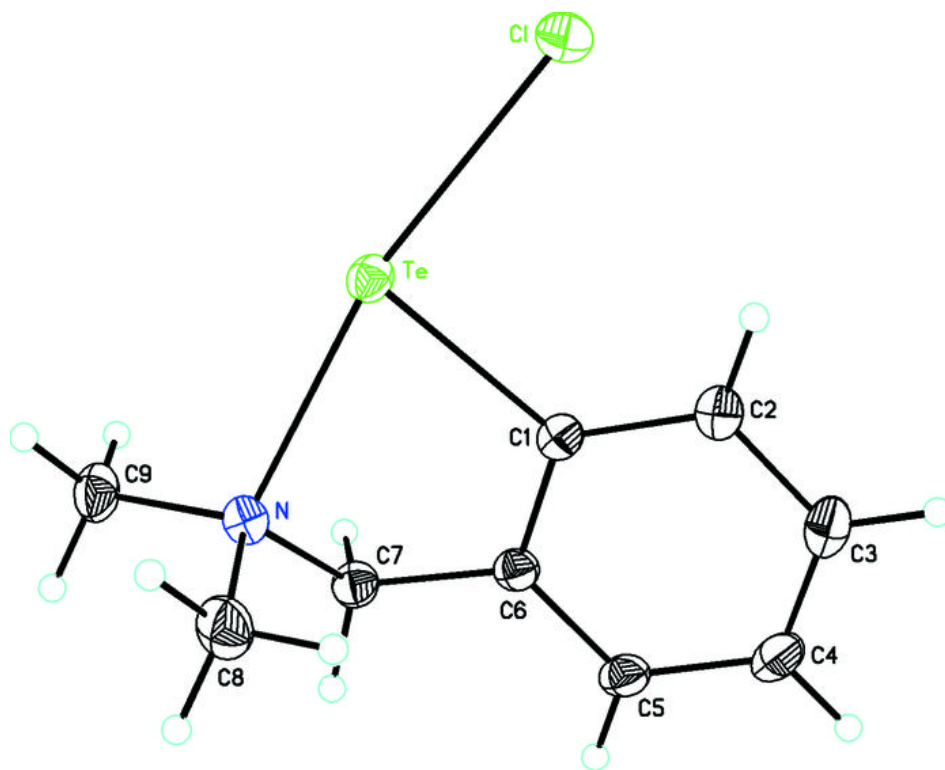


Fig. 2

