organic compounds

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

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The crystal structure of the title compound, $C_9H_{12}ClNTe$, contains isolated molecules with no close Te···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···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).
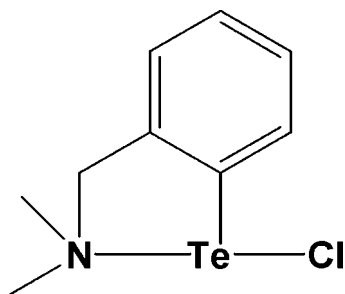
Experimental

Crystal data

C₉H₁₂ClNTe
$M_r = 297.25$
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—C1	2.116 (3)	Te—Cl	2.5657 (11)
Te—N	2.355 (3)		

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

D—H···A	D—H	H···A	D···A	D—H···A
C9—H9C···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*.

References

Engman, L., Wojton, A., Oleksyn, B. J. & Sliwinski, J. (2004). *Phosphorus Sulfur Silicon Relat. Elem.* **179**, 285–292.

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

Oxford Diffraction (2007). *CrysAlis PRO*. Oxford Diffraction Ltd, Abingdon, England.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Singh, H. B., Sudha, N., West, A. A. & Hamor, T. A. (1990). *J. Chem. Soc. Dalton Trans.* pp. 907–913.

supporting information

Chlorido{2-[(dimethylamino)methyl]phenyl-*κ²N,C¹*}tellurium

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S1. Comment

Attempts to synthesize protonated bis[2-(dimethylaminomethyl)phenyl]ditelluride resulted in the formation of 2-(*N,N*-di-methylaminomethyl)phenyl)tellurenyl chloride, $C_9H_{12}ClNTe$, **(1)** a compound which had been previously prepared *via* a different method (Singh *et al.*, 1990). The structure of $C_9H_{12}ClNTe$ 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 arelinked into dimers by C—H···Cl interactions.

S2. 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 $C_9H_{12}ClTe$: C, 36.37; H, 4.07; N, 4.37. Found C, 36.44; H, 4.04; N, 4.66.

S3. 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 [$U_{iso}(H) = 1.2U_{eq}(CH, CH_2)$ [$U_{iso}(H) = 1.5U_{eq}(CH_3)$].
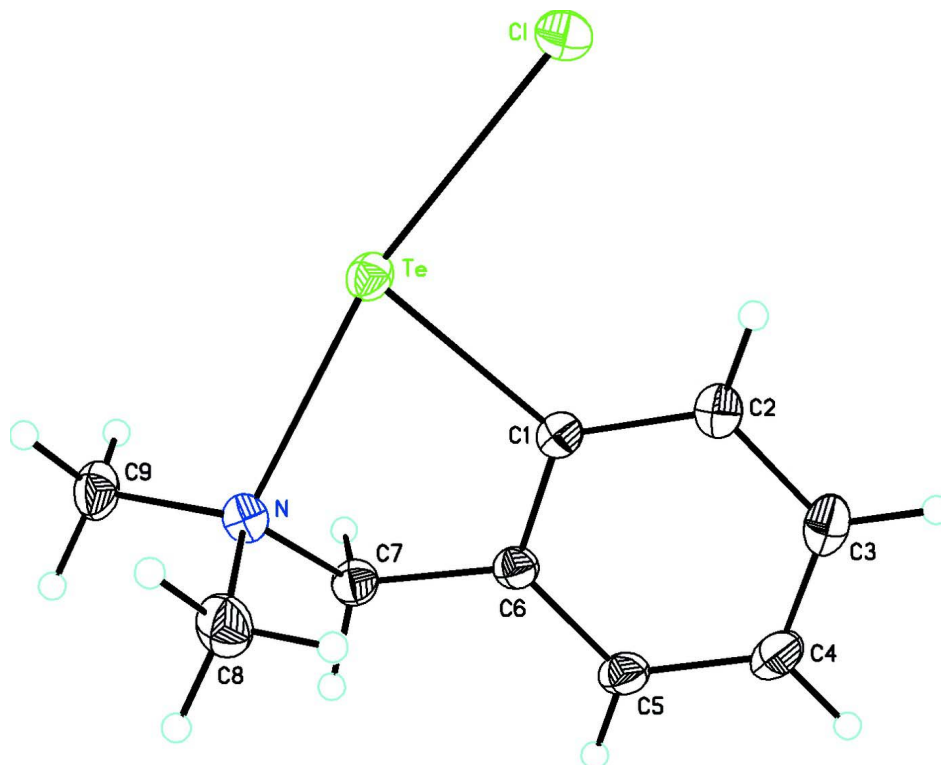
Figure 1

Diagram of the contents of the asymmetric unit of $C_9H_{12}ClNTe$. Atomic displacement ellipsoids are drawn at the 30% level.
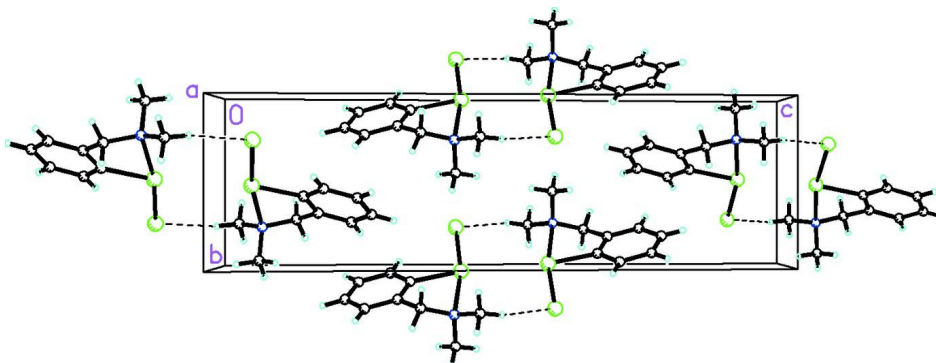
Figure 2

The molecular packing for $C_9H_{12}ClNTe$ viewed down the *c* axis. C—H⋯Cl secondary interactions are shown by dashed lines.

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

Crystal data

$C_9H_{12}ClNTe$	$b = 7.0287\ (7)$ Å
$M_r = 297.25$	$c = 23.847\ (2)$ Å
Monoclinic, $P2_1/n$	$\beta = 95.967\ (9)°$
Hall symbol: -P 2yn	$V = 1075.49\ (17)$ Å³
$a = 6.4514\ (6)$ Å	$Z = 4$

$F(000) = 568$
$D_x = 1.836$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3931 reflections
$\theta = 5.1–32.6°$

$\mu = 2.96$ mm^{-1}
$T = 295$ K
Irregular plate, pale yellow
$0.45 \times 0.36 \times 0.12$ mm

Data collection

Oxford Diffraction Xcalibur Ruby Gemini
   diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 10.5081 pixels mm^{-1}
ω scans
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 $I > 2\sigma(I)$
$R_{int} = 0.023$
$\theta_{max} = 32.7°$, $\theta_{min} = 5.1°$
$h = -9 \rightarrow 9$
$k = -6 \rightarrow 10$
$l = -35 \rightarrow 34$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.096$
$S = 1.20$
3587 reflections
111 parameters
0 restraints
Primary atom site location: structure-invariant
   direct methods

Secondary atom site location: difference Fourier
   map
Hydrogen site location: inferred from
   neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0327P)^2 + 1.2847P]$
   where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 2.25$ e Å^{-3}
$\Delta\rho_{min} = -0.98$ e Å^{-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 ($Å^2$)

	x	y	z	U_{iso}*/U_{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 (Å^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)
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, º)

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, º)

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
C9—H9*C*⋯Cl[i]	0.96	2.89	3.822 (5)	163

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