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4-(Dimethylamino)phenyl ethynyl telluride

Joan Farran, Angel Alvarez-Larena, Joan F. Piniella and Mario V. Capparelli*

Unitat de Cristal·lografia, Universitat Autònoma de Barcelona, 08193 Bellaterra, Spain

Correspondence e-mail: mariocapparelli@cantv.net

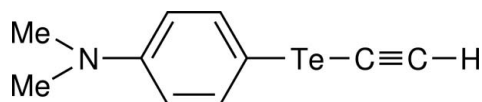
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Key indicators: single-crystal X-ray study; $T = 294$ K; mean $\sigma(\text{C}-\text{C}) = 0.006$ Å; R factor = 0.034; wR factor = 0.087; data-to-parameter ratio = 21.8.

The title compound, $\text{C}_{10}\text{H}_{11}\text{NTe}$, is the first organyl ethynyl telluride, $R-\text{Te}-\text{C}\equiv\text{C}-\text{H}$, to be structurally characterized. In the L-shaped molecule, the aryl moiety, *viz.* $\text{Me}_2\text{NC}_6\text{H}_4\text{Te}$, is almost perpendicular to the $\text{Te}-\text{C}\equiv\text{C}-\text{H}$ fragment. The $\text{Te}-\text{C}_{sp^2}$ bond [2.115 (3) Å] is significantly longer than the $\text{Te}-\text{C}_{sp}$ bond [2.041 (4) Å]. The $\text{Te}-\text{C}\equiv\text{C}$ group is approximately linear [$\text{Te}-\text{C}-\text{C} = 178.5$ (4)° and $\text{C}\equiv\text{C} = 1.161$ (5) Å], while the coordination at the Te atom is angular [$\text{C}-\text{Te}-\text{C} = 95.92$ (14)°]. In the crystal structure, there are $\text{C}_{sp}-\text{H}\cdots\text{N}$ hydrogen bonds which are perpendicular to the CNMe_2 group; the N atom displays some degree of pyramidalization. Centrosymmetrically related pairs of molecules are linked by $\text{Te}\cdots\pi(\text{aryl})$ interactions, with $\text{Te}\cdots\text{Cg} = 3.683$ (4) Å and $\text{C}_{sp}-\text{Te}\cdots\text{Cg} = 159.1$ (2)° (Cg is the centroid of the benzene ring). These interactions lead to the formation of zigzag ribbons which run along c and are approximately parallel to (110).

Related literature

For general background, see: Dabdoub *et al.* (1998); Gillespie & Hargittai (1991); Kauffmann & Ahlers (1983); Murai *et al.* (1994); Petragani (1994); Potapov & Trofimov (2005); Schulz Lang *et al.* (2006); Yoshimatsu (2005); Zukerman-Schpector & Haiduc (2001). For related structures, see: Farran *et al.* (2002). For details of the synthesis, see: Brandsma (1988); Petragani *et al.* (1975).



Experimental

Crystal data

$\text{C}_{10}\text{H}_{11}\text{NTe}$
 $M_r = 272.80$
 Triclinic, $P\bar{1}$
 $a = 7.8857$ (7) Å
 $b = 8.3851$ (8) Å
 $c = 9.3364$ (9) Å
 $\alpha = 65.788$ (2)°
 $\beta = 66.922$ (1)°
 $\gamma = 83.444$ (2)°
 $V = 517.18$ (8) Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 2.82$ mm⁻¹
 $T = 294$ K
 $0.36 \times 0.30 \times 0.10$ mm

Data collection

Bruker SMART APEX diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2001)
 $T_{\min} = 0.403$, $T_{\max} = 0.754$
 3574 measured reflections
 2401 independent reflections
 2080 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.012$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.034$
 $wR(F^2) = 0.087$
 $S = 1.04$
 2401 reflections
 110 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.75$ e Å⁻³
 $\Delta\rho_{\min} = -0.35$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C}2-\text{H}2\cdots\text{N}1^i$	0.93	2.48	3.379 (6)	163

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

Data collection: SMART (Bruker, 2002); cell refinement: SAINT (Bruker, 2001); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 (Farrugia, 1997); software used to prepare material for publication: WinGX (Farrugia, 1999) and PLATON (Spek, 2009).

The authors gratefully acknowledge the financial support of the European Union (project No. CII*.0574.ES). JF thanks the Generalitat de Catalunya for an FI grant and MVC thanks the Ministerio de Educación y Cultura of Spain for a sabbatical grant (project No. SAB95-0281). The sample studied was kindly provided by Professor L. Torres-Castellanos. The X-ray measurements were carried out at the Servei de Difracció de Raigs X (UAB).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BG2241).

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supporting information

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4-(Dimethylamino)phenyl ethynyl telluride

Joan Farran, Angel Alvarez-Larena, Joan F. Piniella and Mario V. Capparelli

S1. Comment

Although organotellurium compounds have attracted considerable interest as reagents and intermediates in organic synthesis (Petragnani, 1994), only a limited number of compounds with mono- and ditelluroethyne cores, R—Te—C≡CH and R—Te—C≡C—Te—R', have been reported, in spite of the potential reactivity of the acetylene unit towards addition reactions. We recently reported the syntheses and crystal structures of several symmetrical ($R = R'$) bis(aryl-telluro)ethynes, Ar—Te—C≡C—Te—Ar (Farran *et al.*, 2002). On the other hand, only five R—Te—C≡CH derivatives have been prepared so far, with $R = \text{Me}$, Et, ⁱPr, n-Bu and Ph (Kauffmann & Ahlers, 1983; Dabdoub *et al.*, 1998; Potapov & Trofimov, 2005; Yoshimatsu, 2005), and none has been structurally characterized (in addition, molecular orbital calculations for $R = \text{HC}\equiv\text{C}$ were carried out by Murai *et al.*, 1994). Here we describe the crystal structure of the title compound ($R = p\text{-Me}_2\text{NC}_6\text{H}_4$), the first of an organyl ethynyl telluride to be reported.

The structure analysis showed that the crystal contains discrete *L*-shaped molecules of the title compound (Figure 1), in which the aryl moiety, Me₂NC₆H₄Te, is almost perpendicular to the Te—C≡C—H fragment (*cf.* C—Te—C angle, Table 1), but bent *ca* 13° towards the C12—C13 side of the ring (*cf.* C—Te—C—C angles, Table 1), probably to optimize the C—H⋯N interaction (see below).

As expected, the Te—C(*sp*²) bond is significantly longer than the Te—C(*sp*) one. The Te—C≡C moiety is approximately linear, while the coordination at the Te atom is angular, as predicted by the valence-shell electron-pair repulsion (VSEPR) model for an AX₂E₂ molecule (Gillespie & Hargittai, 1991). The values of these geometric parameters (Table 1) are similar to the ranges observed in several bis(aryltelluro)ethynes, Ar—Te—C≡C—Te—Ar (Farran *et al.*, 2002), *viz.* Te—C(*sp*²), 2.103 (5)–2.142 (6) Å; Te—C(*sp*), 2.021 (6)–2.058 (6) Å; C≡C, 1.166 (12)–1.203 (11) Å and C—Te—C, 94.2 (3)–97.2 (2)°, which are substantially smaller than the tetrahedral value (109.5°) due to the repulsion of the lone pairs of electrons on the bonded ones.

In the crystal structure the molecules are linked by C(*sp*)—H⋯N hydrogen bonds (Table 2) which are perpendicular to the CNMe₂ group. The N atom displays some degree of pyramidalization: it is 0.123 (5) Å out of the plane of the three C atoms, towards the H atom. There are also Te⋯π(aryl) interactions, similar to those described by Zukerman-Schpector & Haiduc (2001) or Schulz Lang *et al.* (2006) for Te(IV) compounds, in which centrosymmetrically related pairs of molecules are at Te⋯Cg 3.683 (4) Å and C(*sp*)—Te⋯Cg 159.1 (2)° (Cg = centroid of the phenyl ring at 1 - *x*, -*y*, 1 - *z*). These interactions lead to the formation of zigzag ribbons, made of pairs of chains, which run along *c* and are approximately parallel to (110) (Figure 2).

S2. Experimental

Ethynyl magnesium bromide, HC≡CMgBr, was prepared according to published procedures (Brandsma, 1988). The corresponding diaryl ditelluride, (Me₂NC₆H₄Te)₂, was synthesized as reported elsewhere (Petragnani *et al.*, 1975). A dark solution of the diaryl ditelluride (2.0 mmol, 0.94 g) in 40 ml of THF was treated dropwise with bromine (2.0 mmol, 0.32

g, 0.10 ml) in 10 ml of benzene, at 0°C, in N₂ atmosphere, while efficient cooling was applied. The Grignard reagent was then added dropwise. Gradual disappearance of the dark color of the solution was observed until it finally became almost colorless when about 10% excess of the reagent was added. After stirring for 30 min at room temperature, the solution was diluted with 50 ml of low boiling point petroleum ether, treated with aqueous NH₄Cl and washed with brine. The organic layer was dried over magnesium sulfate and the solvents were evaporated. The residue was purified by flash chromatography (silica gel/hexane). Yield 51%. Crystals suitable for X-ray analysis were obtained by slow evaporation of a chloroform solution. The specimen used for data collection was air-protected with a thin coat of Loctite epoxy adhesive.

S3. Refinement

Hydrogen atoms were placed in calculated positions using a riding atom model with fixed C—H distances [0.93 Å for C(*sp*) and C(*sp*²), 0.96 Å for C(*sp*³)] and $U_{\text{iso}} = p U_{\text{eq}}(\text{parent atom})$ [$p = 1.2$ for C(*sp*) and C(*sp*²), 1.5 for C(*sp*³)].

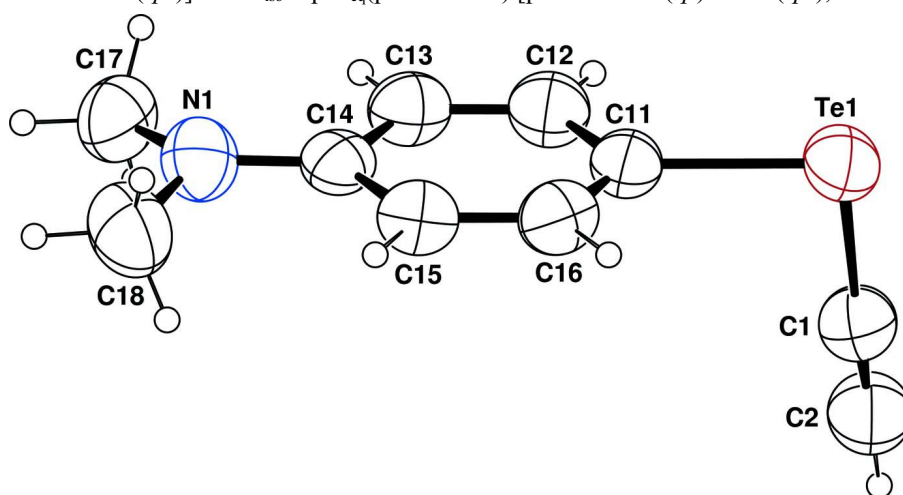


Figure 1

Molecular structure of the title compound showing the atomic numbering. Displacement ellipsoids are drawn at 50% probability level.

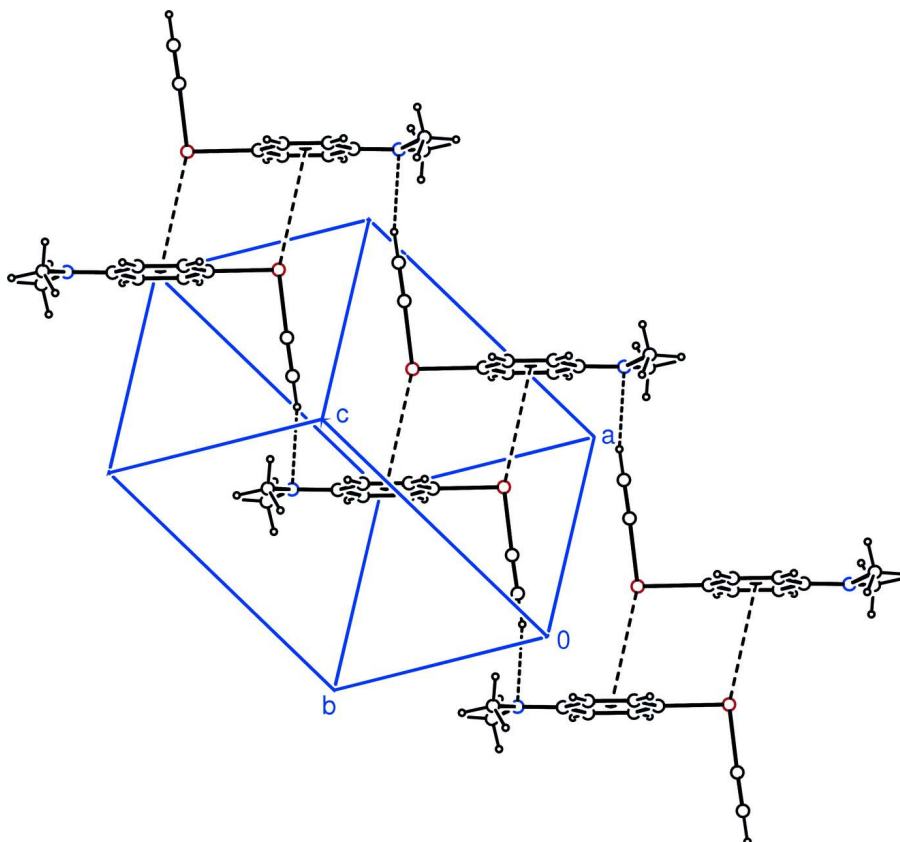


Figure 2

View of the zigzag ribbon generated by the hydrogen bonds and the Te $\cdots\pi$ (aryl) interactions (shown as dashed lines).

4-(Dimethylamino)phenyl ethynyl telluride

Crystal data

$C_{10}H_{11}NTe$

$M_r = 272.80$

Triclinic, $P\bar{1}$

Hall symbol: $-P\ 1$

$a = 7.8857$ (7) Å

$b = 8.3851$ (8) Å

$c = 9.3364$ (9) Å

$\alpha = 65.788$ (2)°

$\beta = 66.922$ (1)°

$\gamma = 83.444$ (2)°

$V = 517.18$ (8) Å³

$Z = 2$

$F(000) = 260$

$D_x = 1.752$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 1735 reflections

$\theta = 2.7\text{--}25.6^\circ$

$\mu = 2.82$ mm⁻¹

$T = 294$ K

Plate, pale brown

$0.36 \times 0.30 \times 0.10$ mm

Data collection

Brucker SMART APEX
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: 8.13 pixels mm⁻¹

φ and ω scans

Absorption correction: multi-scan
(*SADABS*; Bruker, 2001)

$T_{\min} = 0.403$, $T_{\max} = 0.754$

3574 measured reflections

2401 independent reflections

2080 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.012$

$\theta_{\max} = 28.9^\circ$, $\theta_{\min} = 2.6^\circ$
 $h = -10 \rightarrow 10$

$k = -10 \rightarrow 10$
 $l = -10 \rightarrow 12$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.034$
 $wR(F^2) = 0.087$
 $S = 1.04$
 2401 reflections
 110 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
 $w = 1/[\sigma^2(F_o^2) + (0.0487P)^2 + 0.1129P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.005$
 $\Delta\rho_{\max} = 0.75 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.35 \text{ e } \text{\AA}^{-3}$

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Te1	0.45985 (3)	0.02042 (3)	0.26991 (3)	0.06634 (13)
C1	0.3522 (5)	0.1483 (5)	0.0886 (5)	0.0612 (8)
C2	0.2942 (7)	0.2203 (6)	-0.0170 (6)	0.0747 (11)
H2	0.2478	0.2779	-0.1016	0.090*
C11	0.3173 (5)	0.1554 (5)	0.4271 (4)	0.0532 (7)
C12	0.3748 (5)	0.3243 (5)	0.3871 (5)	0.0608 (9)
H12	0.4778	0.3792	0.2894	0.073*
C13	0.2819 (5)	0.4118 (5)	0.4899 (5)	0.0595 (8)
H13	0.3219	0.5256	0.4592	0.071*
C14	0.1272 (5)	0.3316 (4)	0.6407 (4)	0.0515 (7)
C15	0.0702 (5)	0.1609 (5)	0.6793 (5)	0.0568 (8)
H15	-0.0321	0.1042	0.7771	0.068*
C16	0.1641 (5)	0.0766 (4)	0.5739 (5)	0.0566 (8)
H16	0.1236	-0.0362	0.6022	0.068*
N1	0.0368 (5)	0.4146 (5)	0.7474 (4)	0.0639 (8)
C17	0.0794 (7)	0.5988 (5)	0.6960 (6)	0.0714 (11)
H171	0.0033	0.6351	0.7852	0.107*
H172	0.0561	0.6671	0.5949	0.107*
H173	0.2071	0.6158	0.6739	0.107*
C18	-0.1341 (7)	0.3391 (7)	0.8888 (6)	0.0834 (13)
H181	-0.1834	0.4182	0.9439	0.125*
H182	-0.1121	0.2302	0.9681	0.125*
H183	-0.2209	0.3184	0.8485	0.125*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Te1	0.0694 (2)	0.0780 (2)	0.06248 (18)	0.02561 (14)	-0.03447 (14)	-0.03496 (14)
C1	0.061 (2)	0.067 (2)	0.060 (2)	0.0095 (17)	-0.0267 (17)	-0.0276 (17)
C2	0.085 (3)	0.081 (3)	0.069 (2)	0.023 (2)	-0.041 (2)	-0.033 (2)
C11	0.0526 (18)	0.061 (2)	0.0527 (18)	0.0111 (15)	-0.0256 (15)	-0.0263 (15)
C12	0.0518 (18)	0.067 (2)	0.056 (2)	-0.0026 (16)	-0.0174 (16)	-0.0194 (17)
C13	0.060 (2)	0.0532 (19)	0.064 (2)	-0.0041 (16)	-0.0241 (17)	-0.0194 (16)
C14	0.0541 (18)	0.0528 (18)	0.0510 (17)	0.0093 (14)	-0.0274 (15)	-0.0186 (14)
C15	0.0558 (19)	0.0539 (19)	0.0522 (18)	-0.0010 (15)	-0.0163 (15)	-0.0163 (15)
C16	0.064 (2)	0.0460 (17)	0.061 (2)	0.0023 (15)	-0.0293 (17)	-0.0174 (15)
N1	0.070 (2)	0.0569 (17)	0.0628 (19)	0.0078 (14)	-0.0228 (16)	-0.0254 (14)
C17	0.088 (3)	0.062 (2)	0.085 (3)	0.019 (2)	-0.048 (2)	-0.039 (2)
C18	0.076 (3)	0.095 (3)	0.073 (3)	0.013 (2)	-0.015 (2)	-0.043 (2)

Geometric parameters (Å, °)

Te1—C1	2.041 (4)	C15—C16	1.376 (5)
Te1—C11	2.115 (3)	C15—H15	0.9300
C1—C2	1.161 (5)	C16—H16	0.9300
C2—H2	0.9300	N1—C18	1.440 (6)
C11—C16	1.384 (5)	N1—C17	1.454 (6)
C11—C12	1.390 (5)	C17—H171	0.9600
C12—C13	1.378 (5)	C17—H172	0.9600
C12—H12	0.9300	C17—H173	0.9600
C13—C14	1.410 (5)	C18—H181	0.9600
C13—H13	0.9300	C18—H182	0.9600
C14—N1	1.372 (5)	C18—H183	0.9600
C14—C15	1.407 (5)		
C1—Te1—C11	95.92 (14)	C15—C16—C11	121.8 (3)
C2—C1—Te1	178.5 (4)	C15—C16—H16	119.1
C1—C2—H2	180.0	C11—C16—H16	119.1
C16—C11—C12	118.1 (3)	C14—N1—C18	120.5 (3)
C16—C11—Te1	120.6 (3)	C14—N1—C17	120.7 (4)
C12—C11—Te1	121.3 (3)	C18—N1—C17	116.6 (4)
C13—C12—C11	121.2 (3)	N1—C17—H171	109.5
C13—C12—H12	119.4	N1—C17—H172	109.5
C11—C12—H12	119.4	H171—C17—H172	109.5
C12—C13—C14	121.0 (3)	N1—C17—H173	109.5
C12—C13—H13	119.5	H171—C17—H173	109.5
C14—C13—H13	119.5	H172—C17—H173	109.5
N1—C14—C15	121.0 (3)	N1—C18—H181	109.5
N1—C14—C13	121.8 (3)	N1—C18—H182	109.5
C15—C14—C13	117.2 (3)	H181—C18—H182	109.5
C16—C15—C14	120.8 (3)	N1—C18—H183	109.5
C16—C15—H15	119.6	H181—C18—H183	109.5

C14—C15—H15	119.6	H182—C18—H183	109.5
C1—Te1—C11—C16	102.7 (3)	C13—C14—C15—C16	0.7 (5)
C1—Te1—C11—C12	-77.8 (3)	C14—C15—C16—C11	0.1 (6)
C16—C11—C12—C13	-0.4 (6)	C12—C11—C16—C15	-0.3 (5)
Te1—C11—C12—C13	-179.9 (3)	Te1—C11—C16—C15	179.3 (3)
C11—C12—C13—C14	1.2 (6)	C15—C14—N1—C18	-8.9 (6)
C12—C13—C14—N1	177.8 (4)	C13—C14—N1—C18	172.1 (4)
C12—C13—C14—C15	-1.3 (5)	C15—C14—N1—C17	-171.4 (4)
N1—C14—C15—C16	-178.4 (3)	C13—C14—N1—C17	9.6 (6)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
C2—H2 \cdots N1 ⁱ	0.93	2.48	3.379 (6)	163

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