

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.046$
 $wR(F^2) = 0.118$
 $S = 1.018$
 4968 reflections
 345 parameters
 H-atom parameters
 constrained
 $w = 1/[\sigma^2(F_o^2) + (0.052P)^2 + 0.6603P]$
 where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.015$
 $\Delta\rho_{\max} = 0.24 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.18 \text{ e } \text{Å}^{-3}$
 Extinction correction: none
 Scattering factors from
*International Tables for
 Crystallography* (Vol. C)

Table 1. Selected geometric parameters (Å , $^\circ$)

S1—C1	1.663 (2)	S1'—C1'	1.660 (3)
N1—C2	1.376 (3)	N1'—C2'	1.378 (3)
N1—C1	1.389 (3)	N1'—C1'	1.382 (3)
N1—C3	1.441 (3)	N1'—C3'	1.437 (3)
N2—C2	1.295 (3)	N2'—C2'	1.300 (3)
N2—N3	1.367 (3)	N2'—N3'	1.377 (3)
N3—C1	1.333 (3)	N3'—C1'	1.339 (3)
C2—C9	1.474 (3)	C2'—C9'	1.480 (3)
C2—N1—C1	107.00 (18)	C2'—N1'—C1'	107.43 (19)
C2—N1—C3	128.52 (18)	C2'—N1'—C3'	128.79 (19)
C1—N1—C3	123.69 (19)	C1'—N1'—C3'	123.3 (2)
C2—N2—N3	103.78 (19)	C2'—N2'—N3'	103.5 (2)
C1—N3—N2	114.20 (18)	C1'—N3'—N2'	113.97 (19)
C11—N4—C12	116.5 (2)	C11'—N4'—C12'	116.9 (2)
N3—C1—N1	103.19 (19)	N3'—C1'—N1'	103.3 (2)
N3—C1—S1	127.46 (17)	N3'—C1'—S1'	129.05 (18)
N1—C1—S1	129.33 (18)	N1'—C1'—S1'	127.7 (2)
N2—C2—N1	111.83 (19)	N2'—C2'—N1'	111.8 (2)
N2—C2—C9	121.2 (2)	N2'—C2'—C9'	124.2 (2)
N1—C2—C9	126.9 (2)	N1'—C2'—C9'	123.9 (2)
C1—N1—C3—C4	-106.7 (3)	C1'—N1'—C3'—C4'	-98.8 (3)
N1—C2—C9—C10	-147.0 (2)	N1'—C2'—C9'—C10'	-144.3 (3)

Table 2. Hydrogen-bonding geometry (Å , $^\circ$)

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
N3—H3 \cdots N4 ⁱⁱ	0.86	1.96	2.803 (3)	166
N3'—H3' \cdots N4 ⁱⁱ	0.86	2.02	2.852 (3)	164

Symmetry codes: (i) $1 - x, 1 - y, 1 - z$; (ii) $-1 - x, y - \frac{1}{2}, \frac{1}{2} - z$.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *CAD-4 Software*. Program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997a). Program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997b). Molecular graphics: *ZORTEP* (Zsolnai, 1997). Software used to prepare material for publication: *PARST* (Nardelli, 1995).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1553). Services for accessing these data are described at the back of the journal.

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2-Methyltelluro-*N*-phenylbenzamide

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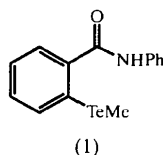
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Abstract

The title tellurium compound, C₁₄H₁₃NOTe, is isostructural with the selenium analogue, 2-methylseleno-*N*-phenylbenzamide [Fong, Gable & Schiesser (1996). *Acta Cryst.* **C52**, 1886–1889], having a Te \cdots O separation of 2.835 (2) Å. Steric repulsions between the *ortho*-H atom on the *N*-phenyl ring and the amide-O atom result in asymmetry in the bond angles around the junction C atom of the *N*-phenyl ring, while the angles around the N and amide C are influenced by the Te \cdots O interaction. The amide-N atom is involved in hydrogen bonding with the O atom of an adjacent molecule, forming zigzag chains that lie along the *a* axis.

Comment

We have recently been interested in the use of free-radical homolytic substitution chemistry for the preparation of higher heterocycles. During the course of this work, we had cause to prepare 2-methyltelluro-*N*-phenylbenzamide, (1), and to explore similar ring-closure chemistry to that reported by us for the analogous selenium-containing compounds (Fong & Schiesser, 1997). To our surprise, (1) failed to react with benzoyl peroxide under standard radical conditions. In order to provide some insight into possible conformational effects that may be responsible for the unusual lack of reactivity, the crystal structure of (1) was determined.



The title compound is isostructural with the Se analogue, 2-methylseleno-*N*-phenylbenzamide (Fong *et al.*, 1996), with only minor differences being observed in the geometric parameters. The Te—C distances (Table 1) are about 0.2 Å longer than the corresponding Se—C1 and Se—C14 distances of 1.920 (2) and 1.935 (3) Å, respectively. The Te···O separation of 2.835 (2) Å is similar to the corresponding Se···O separation of 2.829 (2) Å, which, when compared with the sum of the relevant van der Waals radii (Bondi, 1964) of about 3.6 and 3.4 Å, for Te and O, and Se and O, respectively, indicates that the Te···O interaction in (1) is slightly stronger than the Se···O interaction observed in the Se analogue. This, however, is still a weak interaction, similar to that observed in acetato(2-phenylazophenyl-*C,N'*)tellurium(II) [Te···O 2.953 (4) Å; Ahmed *et al.*, 1985].

The asymmetry of the bond angles around C8 is also observed in both 2-methylseleno-*N*-phenylbenzamide (Fong *et al.*, 1996) and 2-(acetyl)seleno-benzanilide (Dupont *et al.*, 1990), as well as in similar compounds without X···O interactions, such as *trans*-2-methylbenzanilide (Azumaya *et al.*, 1994), *p,p'*-dinitrobenzanilide (Hariharan & Srinivasan, 1990) and 4-amino-*N*-phenylbenzamide (Duke & Codding, 1992). In all of these compounds the *ortho*-H atom on the *N*-phenyl ring is in close contact with the amide O, with separations of 2.25–2.49 Å [for (1) H9···O = 2.33 (3) Å], somewhat less than the sum of the van der Waals radii of 2.72 Å (Bondi, 1964), so that the asymmetry around C8 can be ascribed to steric repulsions between these atoms. It is notable that in *N*-methylbenzanilide (Azumaya *et al.*, 1994), where the amide adopts a *cis* conformation and there is no close contact between the amide O and the H atoms on the

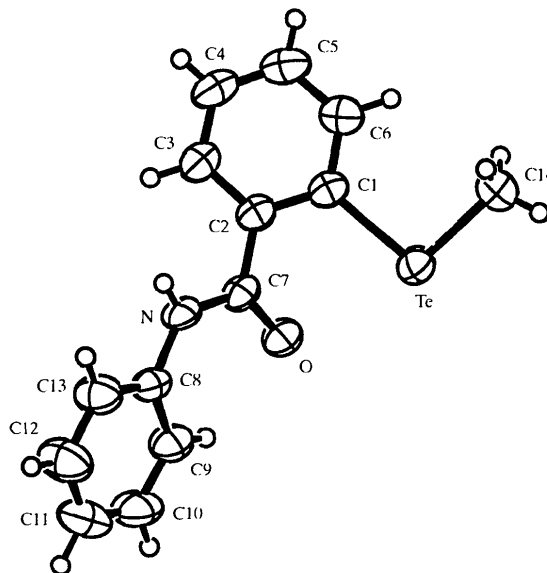


Fig. 1. An ORTEP (Johnson, 1976) drawing of compound (1) with displacement ellipsoids drawn at the 50% probability level. H atoms are shown as circles of an arbitrary radius.

N-phenyl group, the corresponding bond angles are much less asymmetric. The bond angles around C7 and N, however, do show a variation, with those compounds with X···O interactions having smaller O—C7—C2 angles [120.1–120.7° versus 120.9–122.3°], larger N—C7—C2 angles [116.5–117.5° versus 114.1–115.4°] and larger C7—N—C8 angles [126.3–129.0° versus 122.7–127.3°], compared with those compounds where X···O interactions are absent. The O—C7—C2 angle for (1) is slightly less than that for the Se analogue [120.7 (2)°], consistent with a stronger Te···O interaction.

As in 2-methylseleno-*N*-phenylbenzamide, the amide N of (1) is involved in hydrogen bonding with the O atom of an adjacent molecule [N···Oⁱ 2.924 (3), N—HN 0.72 (3) and HN···Oⁱ 2.24 (3) Å, and N—HN···Oⁱ 161 (3)°; symmetry code: (i) $\frac{1}{2} + x, y, \frac{1}{2} - z$], forming zigzag chains which lie along the *a* axis.

Although the Te···O interaction in (1) is only slightly stronger than the Se···O interaction in 2-methylseleno-*N*-phenylbenzamide, it is presumably sufficient to lock the radical derived from (1) in a conformation not conducive to ring closure.

Experimental

Compound (1) was prepared as described by Engman *et al.* (1999) and crystallized from ethanol/petrol.

Crystal data

C₁₄H₁₃NOTe
M_r = 338.85

Mo Kα radiation
λ = 0.71073 Å

Orthorhombic

Pbca

$a = 9.714 (2) \text{ \AA}$
 $b = 16.742 (2) \text{ \AA}$
 $c = 16.540 (2) \text{ \AA}$
 $V = 2689.9 (7) \text{ \AA}^3$
 $Z = 8$
 $D_x = 1.673 \text{ Mg m}^{-3}$
 D_m not measured

Cell parameters from 25 reflections

$\theta = 10.4\text{--}15.1^\circ$
 $\mu = 2.195 \text{ mm}^{-1}$
 $T = 293 (1) \text{ K}$
 Plate
 $0.70 \times 0.50 \times 0.06 \text{ mm}$
 Light brown

Data collection

Enraf–Nonius CAD-4-MACHS diffractometer
 $\omega/2\theta$ scans
 Absorption correction: Gaussian (Sheldrick, 1976)
 $T_{\min} = 0.343$, $T_{\max} = 0.876$
 3371 measured reflections
 2636 independent reflections
 2158 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.016$
 $\theta_{\text{max}} = 25.98^\circ$
 $h = -1 \rightarrow 11$
 $k = -1 \rightarrow 20$
 $l = -1 \rightarrow 20$
 3 standard reflections
 frequency: 160 min
 intensity decay: none

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.027$
 $wR(F^2) = 0.074$
 $S = 1.031$
 2636 reflections
 180 parameters
 H atoms: see below
 $w = 1/[\sigma^2(F_o^2) + (0.0408P)^2 + 1.7223P]$
 where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.62 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.90 \text{ e \AA}^{-3}$
 Extinction correction: none
 Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. Selected geometric parameters (\AA , $^\circ$)

Te—C14	2.120 (4)	N—C8	1.416 (4)
Te—C1	2.122 (3)	C2—C7	1.488 (4)
O—C7	1.230 (3)	C8—C13	1.376 (5)
N—C7	1.341 (4)	C8—C9	1.392 (4)
C14—Te—C1	96.66 (16)	O—C7—C2	120.1 (3)
C7—N—C8	129.0 (3)	N—C7—C2	117.5 (2)
C3—C2—C1	119.6 (3)	C13—C8—C9	119.1 (3)
C3—C2—C7	121.1 (3)	C13—C8—N	117.8 (3)
C1—C2—C7	119.3 (2)	C9—C8—N	123.0 (3)
O—C7—N	122.4 (3)		
C14—Te—C1—C6	19.2 (3)	C1—C2—C7—O	-32.0 (4)
C14—Te—C1—C2	-162.1 (3)	C3—C2—C7—N	-34.4 (4)
C8—N—C7—O	-5.8 (5)	C1—C2—C7—N	147.7 (3)
C8—N—C7—C2	174.5 (3)	C7—N—C8—C13	167.2 (3)
C3—C2—C7—O	145.9 (3)	C7—N—C8—C9	-15.4 (5)

The coordinates of the H atom attached to N were refined [$\text{N—H} = 0.72 (3) \text{ \AA}$]; other H atoms were treated as riding. U_{iso} values of all H atoms were refined.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *PROCESS DATA* (Gable *et al.*, 1993). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997). Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *SHELXL97*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: JA1000). Services for accessing these data are described at the back of the journal.

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The biologically active iron chelators 2-pyridylcarboxaldehyde isonicotinoyl-hydrazone, 2-pyridylcarboxaldehyde benzoylhydrazone monohydrate and 2-furaldehyde isonicotinoylhydrazone

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Abstract

In the crystal structures of the respective title compounds, C₁₂H₁₀N₄O, C₁₃H₁₁N₃O·H₂O and C₁₁H₉N₃O₂, variations in the torsion angles of the aromatic pyridyl