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(μ-Diazenediyldiphenyl-$\kappa^2C^2,N^2:\kappa^2C^{2'},N^1$)-bis[(3,5-dimethylphenyl)tellurium(II)]

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Key indicators

Single-crystal X-ray study
T = 105 K
Mean σ(C–C) = 0.003 Å
R factor = 0.026
wR factor = 0.062
Data-to-parameter ratio = 25.6

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $C_{28}H_{26}N_2Te_2$, prepared by reduction of (3,5-dimethylphenyl)(2-nitrophenyl)tellurium(II), is the first structurally characterized example of an azo group bridging two Te^{II} centers. The compound is centrosymmetric and the Te—N distance [2.6916 (19) Å] is longer than in non-bridging azo compounds.

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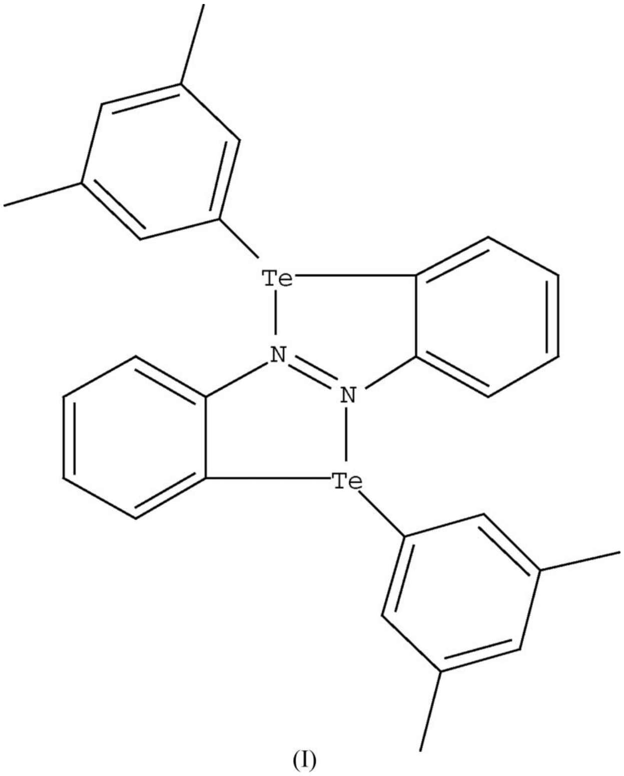
Comment

Intramolecularly coordinated organotellurium compounds commonly exhibit properties significantly different from those of their non-coordinated counterparts. Groups in which a Lewis basic atom coordinates to Te include nitro, nitroso, azo, imino, carbonyl, alkoxy and amino (Detty & O'Regan, 1994). A representative covalent Te—N distance of 2.023 Å has been reported for 1,2,5-telluradiazole (Bertini *et al.*, 1984). While X-ray crystallographic structure determinations have been reported for compounds with Te—azo coordination (Cobbledick *et al.*, 1979; Ahmed *et al.*, 1985*a*,*b*; Majeed *et al.*, 1997), there are no examples reported to date of azo compounds bridging two Te centers.



(I)

The title compound, (I), is centrosymmetric, with the center of symmetry located at the center of the N═N bond; the 2-

phenylazophenyl azo unit is planar and bridges two Te centers. Each azo N atom coordinates to a Te atom. Each Te atom is also coordinated by a benzene C atom of the 2-phenylazophenyl unit. Thus, the 2-phenylazophenyl unit functions as a bridging tetradentate ligand. The Te coordination is completed by a C atom of the dimethylphenyl unit, which is rotated by 84.22 (2)° with respect to the 2-phenylazophenyl plane.

The geometry of each Te center is 'T-shaped', in accord with the presence of two stereochemical lone pairs and the principles of valence-shell electron-pair repulsion (VSEPR) theory, and is typical for Te^{II} centers (see, for example, Mallikaratchy *et al.*, 2003). The N1′—Te—C7 angle is distinctly non-linear [162.41 (7)°], typical of this geometry (Mallikaratchy *et al.*, 2003), with atom C7 displaced toward atom C2. Thus, the sum of the C2—Te—C7 and C2—Te—N1′ angles is 163.18 (8)°. The Te—C distances are typical [2.104 (2) and 2.144 (2) Å; see Mallikaratchy *et al.*, 2003]. The Te—N distance [2.6916 (19) Å] is long compared with other Te–azo compounds. In (2-phenylazophenyl-*C*,*N*′)tellurium(IV) trichloride, the distance is 2.417 (4) Å (Ahmed *et al.*, 1985*a*); in acetato(2-phenylazophenyl-*C*,*N*′)tellurium(II), the distance is 2.260 (4) Å (Ahmed *et al.*, 1985*b*); in (2-phenylazophenyl-*C*,*N*′)thiocyanatotellurium(II), the distance is 2.243 (3) Å (Ahmed *et al.*, 1985*b*); in chloro(2-phenylazophenyl-*C*,*N*′)tellurium(II), the distance is 2.210 (7) Å (Majeed *et al.*, 1997); in bromo(2-phenylazophenyl-*C*,*N*′)tellurium(II), the distance is 2.219 (8) Å (Majeed *et al.*, 1997); and in iodo(2-phenylazophenyl-*C*,*N*′)tellurium(II), the distance is 2.252 (8) Å. The longer Te—N distance in the title compound is accompanied by a typical N═N distance [1.263 (4) Å].

Experimental

During the attempted reduction of (3,5-dimethylphenyl-*C*)(2-nitrophenyl-*C*,*O*)tellurium(II) (prepared according to the method of Clark *et al.*, 2002) to (3,5-dimethylphenyl-*C*)(2-aminophenyl-*C*,*N*)tellurium(II), it was noted that the reaction mixture turned intensely red as the title compound, (I), was generated instead of the expected amine. Furthermore, evidence was found that any (3,5-dimethylphenyl-*C*)(2-aminophenyl-*C*,*N*)tellurium(II) prepared under more rigorous conditions than those employed here is oxidized back to the title compound upon exposure to air. The preparation is as follows. (3,5-Dimethylphenyl-*C*)(2-nitrophenyl-*C*)tellurium(II) (100 mg, 0.28 mmol) and sodium borohydride (90 mg, 2.1 mmol) were placed in propan-2-ol (10 ml). The mixture was heated to reflux for 3 h, then allowed to cool to room temperature and stirred overnight with the condenser removed to ensure re-oxidation of any (3,5-dimethylphenyl-*C*)(2-aminophenyl-*C*,*N*)tellurium(II) formed. The product mixture was subsequently transferred into an open beaker, the solvent allowed to evaporate and the residue extracted with dichloromethane (DCM). Column chromatography (DCM, alumina, 200 mesh, Brockman, neutral) furnished an intensely red–orange crystalline solid. This was subsequently dissolved in DCM (10 ml), followed by the addition of cyclohexane (10 ml). X-ray quality dark red-orange crystals of (I) were obtained by slow open-air evaporation of the solvents (yield 33 mg, 36.3%; m.p. 483 K, with partial decomposition above 473 K).

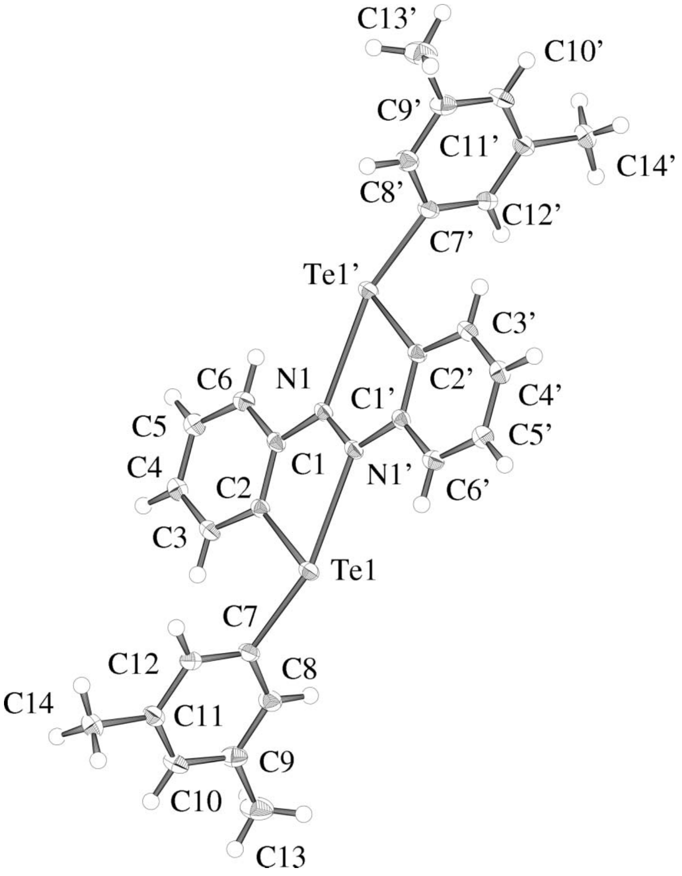


Figure 1
A drawing of (I), with displacement ellipsoids drawn at the 50% probability level.

Crystal data

$C_{28}H_{26}N_2Te_2$
$M_r = 645.71$
Triclinic, $P\overline{1}$
$a = 5.8988$ (10) Å
$b = 7.935$ (2) Å
$c = 13.903$ (4) Å
$\alpha = 97.762$ (10)°
$\beta = 91.487$ (10)°
$\gamma = 106.146$ (15)°
$V = 618.0$ (3) Å^3

$Z = 1$
$D_x = 1.735$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3604 reflections
$\theta = 2.5$–30.5°
$\mu = 2.38$ mm^{-1}
$T = 105$ K
Lath fragment, red-orange
0.22 × 0.17 × 0.05 mm

Data collection

Nonius KappaCCD diffractometer (with Oxford Cryosystems Cryostream cooler)
ω scans, with κ offsets
Absorption correction: multi-scan (*SCALEPACK*; Otwinowski & Minor, 1997)
$T_{\min} = 0.669$, $T_{\max} = 0.890$

18111 measured reflections
3761 independent reflections
3378 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.021$
$\theta_{\max} = 30.5°$
$h = -8 \rightarrow 8$
$k = -11 \rightarrow 11$
$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.026$
$wR(F^2) = 0.062$
$S = 1.03$
3761 reflections
147 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0299P)^2 + 0.3859P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 1.19$ e Å^{-3}
$\Delta\rho_{\min} = -0.90$ e Å^{-3}

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Table 1
Selected geometric parameters (Å, °).

Te1—C2	2.104 (2)	C5—C6	1.386 (3)
Te1—C7	2.144 (2)	C7—C12	1.392 (3)
Te1—N1^{i}	2.6916 (19)	C7—C8	1.397 (3)
N1—N1^{i}	1.263 (4)	C8—C9	1.399 (3)
N1—C1	1.412 (3)	C9—C10	1.397 (4)
C1—C6	1.396 (3)	C9—C13	1.509 (3)
C1—C2	1.414 (3)	C10—C11	1.398 (3)
C2—C3	1.403 (3)	C11—C12	1.393 (3)
C3—C4	1.381 (3)	C11—C14	1.511 (3)
C4—C5	1.391 (3)		
C2—Te1—C7	94.87 (8)	C5—C6—C1	120.7 (2)
C2—Te1—N1^{i}	68.31 (7)	C12—C7—C8	119.3 (2)
C7—Te1—N1^{i}	162.41 (7)	C12—C7—Te1	119.98 (16)
N1^{i}—N1—C1	114.9 (2)	C8—C7—Te1	120.44 (17)
C6—C1—N1	115.29 (19)	C7—C8—C9	120.7 (2)
C6—C1—C2	120.7 (2)	C10—C9—C8	118.4 (2)
N1—C1—C2	123.98 (19)	C10—C9—C13	121.1 (2)
C3—C2—C1	117.4 (2)	C8—C9—C13	120.5 (2)
C3—C2—Te1	121.12 (15)	C9—C10—C11	122.0 (2)
C1—C2—Te1	121.49 (15)	C12—C11—C10	118.1 (2)
C4—C3—C2	121.3 (2)	C12—C11—C14	120.8 (2)
C3—C4—C5	121.0 (2)	C10—C11—C14	121.1 (2)
C6—C5—C4	119.0 (2)	C7—C12—C11	121.5 (2)

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

H atoms were treated as riding in idealized positions, with C—H distances in the range 0.95–0.98 Å, depending on atom type. A torsional parameter was refined for each methyl group. Displacement parameters for H were assigned as $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}$ of the attached atom (1.5 for methyl groups). The largest residual peak was 1.19 Å from Te1.

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* and *DENZO* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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