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Tribromo(3,5-dimethyl-2-nitrophenyl- κ^2C^1,O)tellurium(IV), bromo(3,5-dimethyl-2-nitrophenyl- κ^2C^1,O)tellurium(II) and bromo(3,5-dimethyl-2-nitrosophenyl- κ^2C^1,O)tellurium(II)

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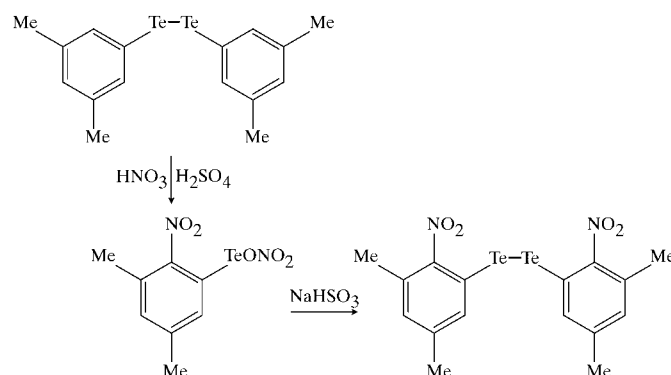
All three title compounds, prepared from bis(3,5-dimethyl-2-nitrophenyl)ditellurium, exhibit high degrees of intramolecular Te—O coordination. Their Te—O distances increase in the order $C_8H_8BrNOTe < C_8H_8BrNO_2Te < C_8H_8Br_3NO_2Te$, with distances of 2.165 (3), 2.306 (1) and 2.423 (6) Å, respectively, indicating that $C_8H_8BrNOTe$ may be more aptly described as 1-bromo-4,6-dimethyl-2,1,3-benzoxatellurazole.

Comment

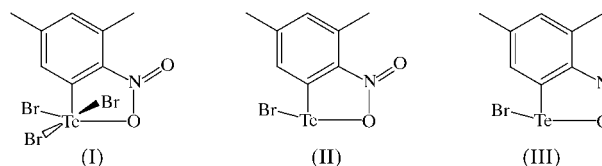
Intramolecularly coordinated organotellurium compounds commonly exhibit properties that are significantly different from those of their non-coordinated counterparts. Moieties through which a Lewis basic atom intramolecularly coordinates to tellurium include azo, imino, carbonyl and amino functionalities (Detty & O'Regan, 1994). This coordination can stabilize otherwise unstable low-valent organotellurium halides and pseudohalides (Cobbledick *et al.*, 1979; Detty *et al.*, 1989; Sadekov *et al.*, 1990; Menon *et al.*, 1996), as well as telluronium cations (Fujihara *et al.*, 1995). Intramolecular coordination by nitroso and nitro moieties has not been documented in much detail because of limited access to these classes of organotellurium compounds. Thus, the only published procedure for the preparation of an *ortho*-nitro-substituted diphenylditellurium compound (Wiriyachitra *et al.*, 1979) was found to be challenging. The few reported examples of NO and NO₂ coordination include dioxatellurapentalenes (Perrier & Vialle, 1971) and 1-nitroso-2-naphthyltellurium monohalides found in the patent literature (Gunther & Lok, 1986; Przyklek-Elling *et al.*, 1987). It appears highly probable that bromo(2-nitrophenyl)tellurium(II) and tribromo(2-

nitrophenyl)tellurium(IV) (Wiriyachitra *et al.*, 1979) also feature strong intramolecular Te—O coordination, but no structural information has been made available for these compounds to date. Compounds carrying *ortho* nitro, nitroso and amino functionalities are of interest as potential precursors to compounds featuring coordinatively stabilized telluronium cations and as intermediates to organotellurium heterocycles (Junk & Irgolic, 1988).

Recent advances in the synthesis of substituted diarylditelluriums by selective *ortho* nitration, as well as from boronic acid precursors (Clark *et al.*, 2002), have provided easier access to compounds such as bis(3,5-dimethyl-2-nitrophenyl)ditellurium, which is employed in this study. Bis(3,5-dimethyl-2-nitrophenyl)ditellurium was prepared by nitration of bis(3,5-dimethylphenyl)ditellurium to 3,5-dimethyl-2-nitrobenzenetellurinic acid nitrate, followed by reduction (see first Scheme below).



Treatment of bis(3,5-dimethyl-2-nitrophenyl)ditellurium with stoichiometric amounts of bromine produced tribromo(3,5-dimethyl-2-nitrophenyl- κ^2C,O)tellurium(IV), (I), bromo(3,5-dimethyl-2-nitrophenyl- κ^2C,O)tellurium(II), (II), and bromo(3,5-dimethyl-2-nitrosophenyl- κ^2C,O)tellurium(II), (III), which may be described as heterocyclic (a substituted benzoxatellurazole).



There are two molecules of (I), of nearly identical geometry, in the asymmetric unit (Fig. 1). There appears to be a non-crystallographic center of symmetry between the two independent molecules (at about $x = 0.105$, $y = 0.248$ and $z = \frac{1}{2}$). Similar centers appear in more than 65% of $Pca2_1$ structures in which Z is greater than 4 (Marsh *et al.*, 1998) and often result in unusual correlations between corresponding atoms in the two molecules. Since the average values of equivalent bond distances are apparently unaffected by this pseudo-symmetry (Marsh *et al.*, 1998), we discuss the average case. The geometry about the Te atom is square pyramidal, with the basal plane defined by three Br atoms and an O atom. A C atom is in the axial position, and a stereochemical lone pair is

located *trans* to this C atom. The Te atom sits 'below' the basal plane, which is consistent with the principles of VSEPR (valence-shell electron-pair repulsion) theory. The Te—Br distance for the Br atom *trans* to atom O1 is significantly shorter than the other Te—Br distances [2.5047 (12) Å *versus* 2.6540 (11) and 2.6754 (11) Å; Table 1]. The Te1—C1 bond distance [2.132 (7) Å] is in good agreement with those reported for non-coordinated aryl—Te bonds, *e.g.* in *catena-μ*-bromo-dibromo(phenyl)tellurium(IV) [2.140 (8) Å; Alcock & Harrison, 1982*a*]. The O1—Te1—C1 angle is constrained to 71.8 (2)° by the five-membered ring. These structural features are similar to those reported for trichloro(2-phenylazophenyl-*C,N'*)tellurium(IV), in which there is coordination of tellurium by the N atom of the azo group (Ahmed *et al.*, 1985*a*). There is a short intermolecular contact in (I) between atoms O2*A* and N1*B*, which is reminiscent of the perpendicular motif found for carbonyl—carbonyl interactions (Allen *et al.*, 1998).

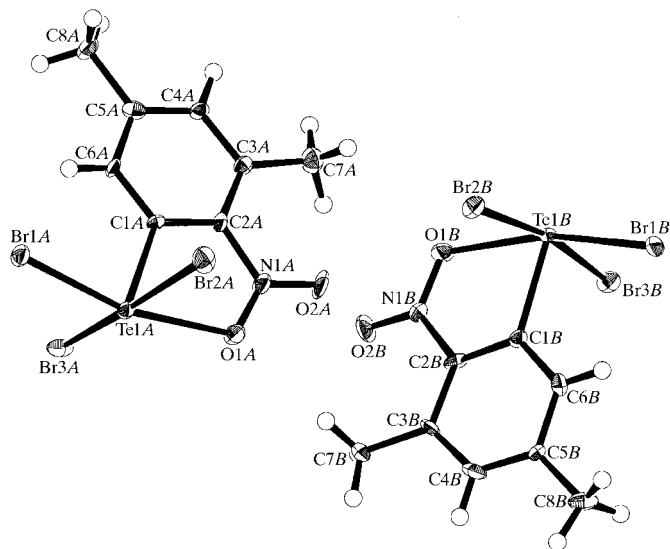


Figure 1
The molecular structure of (I), with displacement ellipsoids drawn at the 50% probability level.

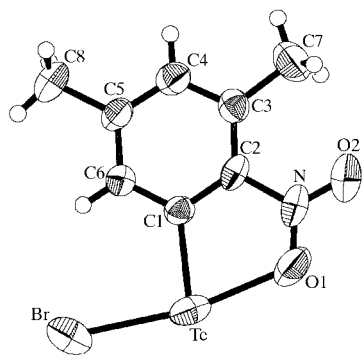


Figure 2
The molecular structure of (II), with displacement ellipsoids drawn at the 50% probability level.

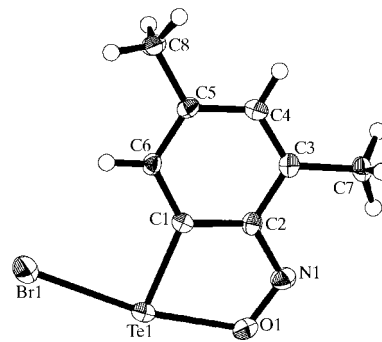


Figure 3
The molecular structure of (III), with displacement ellipsoids drawn at the 50% probability level.

The geometries of (II) and (III) are 'T-shaped' about the Te atom, with the O—Te—Br angles being distinctly non-linear [168.80 (14) and 169.57 (9)°, respectively; Tables 2 and 3, and Figs. 2 and 3] and the bromide ion displaced towards the phenyl ring, which geometry is again consistent with VSEPR theory and the presence of two stereochemical lone pairs of electrons on the Te^{II} centers. The N—O distance in (III) [1.315 (5) Å] is significantly longer than the analogous distance in (II) [1.268 (8) Å], which is in turn significantly longer than the other (non-coordinated) N—O distance of 1.208 (8) Å. The Te—C distance of 2.098 (6) Å in (II) is the same as that found in bromo(2-phenylazophenyl-*C,N'*)tellurium(II) [2.092 (8) Å; Majeed *et al.*, 1997], while the Te—C distance is shortened to 2.038 (4) Å in (III).

The Te—O bond distances of 2.307 (6) Å in (II) and 2.165 (3) Å in (III) are shorter than the Te—O distance of 2.423 (6) Å for (I) and are comparable to Te—O distances observed for amides [*e.g.* 2.237 (8) Å for bromo(2-amidophenyl-*C,O*)tellurium(II); Dupont *et al.*, 1979], for carboxylates [*e.g.* 2.167 (4) Å for acetato(2-phenylazophenyl-*C,N'*)tellurium(II); Ahmed *et al.*, 1985*b*] and for nitrates [*e.g.* 2.171 (3) Å for diphenyltellurium(IV) dinitrate; Alcock & Harrison, 1982*b*].

The relative ease with which (I) was reduced to (III) is indicative of the high stability of *ortho*-nitroso-stabilized aryltellurium subhalides. In practice, exposure of the tellurium(IV) compound to a variety of ketones and alcohols resulted in gradual color changes indicative of reduction.

Experimental

All of the title compounds were prepared from bis(3,5-dimethylphenyl)diteLLurium, accessible *via* the Haller–Irgolic method (Haller & Irgolic, 1972). This compound was converted to bis(3,5-dimethyl-2-nitrophenyl)diteLLurium by nitration and subsequent reduction (Nair, 2002). For the preparation of (I), bis(3,5-dimethyl-2-nitrophenyl)diteLLurium (1.0 g, 1.8 mmol) was dissolved in carbon tetrachloride (30 ml). A solution of bromine (10% *w/w*) in carbon tetrachloride was added slowly, and a gradual darkening to magenta was observed, followed by a sharp color change to yellow. Addition of

bromine was discontinued and the product was crystallized by open-air evaporation. In contrast to non-coordinated aryltellurium trihalides, this compound was found to be insensitive to moisture (yield quantitative; m.p. 467–469 K). ^1H NMR (CDCl_3 , p.p.m.): 2.58 (3H), 2.80 (3H), 7.51 (1H), 8.41 (1H); ^{13}C NMR (CDCl_3 , p.p.m.): 22.5, 22.7, 129.5, 136.0, 137.5, 138.1, 140.8, 151.4.

For the preparation of (II), bis(3,5-dimethyl-2-nitrophenyl)-ditellurium (1.0 g, 1.8 mmol) was dissolved in carbon tetrachloride (30 ml). One third of the previously prepared ditellurium solution was converted to a solution of the tribromide by addition of bromine as above. Subsequently, the tribromide solution was combined with the remaining two parts of the bis(3,5-dimethyl-2-nitrophenyl)-ditellurium solution. Equilibration to the desired monobromide occurred rapidly. The crude product was collected by open-air evaporation of the solvent and recrystallized from cyclohexane (brown-red needles; yield 1.06 g, 82%; m.p. 380–381 K). ^1H NMR (CDCl_3 , p.p.m.): 2.43 (3H), 2.79 (3H), 7.19 (1H), 8.15 (1H); ^{13}C NMR (CDCl_3 , p.p.m.): 21.9, 23.0, 131.8, 133.2, 134.1, 140.5, 144.3, 146.8. Well formed crystals were obtained by slow open-air evaporation of a solution in dichloromethane.

For the preparation of (III), a stirred solution of (I) (0.2 g, 0.4 mmol) in acetone (20 ml) and 2-propanol (20 ml) was heated to reflux for 24 h. A color change from yellow to blue was noticed. The blue solid remaining after open-air evaporation under the hood was taken up in carbon tetrachloride, and the well formed purple crystals remaining after open-air evaporation were collected (yield 42 mg, 32%; m.p. 430–431 K). ^1H NMR (CDCl_3 , p.p.m.): 2.36 (3H), 2.99 (3H), 7.26 (1H), 8.35 (1H); ^{13}C NMR (CDCl_3 , p.p.m.): 18.9, 23.3, 132.4, 133.7, 138.1, 146.2, 149.1, 154.41.

Compound (I)

Crystal data

$\text{C}_8\text{H}_8\text{Br}_3\text{NO}_2\text{Te}$
 $M_r = 517.48$
 Orthorhombic, $Pca2_1$
 $a = 7.371$ (2) Å
 $b = 12.139$ (5) Å
 $c = 28.274$ (11) Å
 $V = 2529.9$ (16) Å³
 $Z = 8$
 $D_x = 2.717$ Mg m⁻³

Mo $K\alpha$ radiation
 Cell parameters from 28 416 reflections
 $\theta = 2.5$ – 32.0°
 $\mu = 11.81$ mm⁻¹
 $T = 120$ K
 Prism, yellow
 $0.20 \times 0.17 \times 0.15$ mm

Data collection

Nonius KappaCCD diffractometer (with an Oxford Cryosystems Cryostream cooler)
 ω scans with κ offsets
 Absorption correction: multi-scan (*HKL SCALEPACK*; Otwinowski & Minor, 1997)
 $T_{\min} = 0.108$, $T_{\max} = 0.170$
 28 416 measured reflections

4303 independent reflections
 3800 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.032$
 $\theta_{\text{max}} = 32.0^\circ$
 $h = -10 \rightarrow 10$
 $k = -18 \rightarrow 18$
 $l = -41 \rightarrow 41$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.035$
 $wR(F^2) = 0.087$
 $S = 1.01$
 4303 reflections
 272 parameters
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0533P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 2.77$ e Å⁻³
 $\Delta\rho_{\text{min}} = -2.44$ e Å⁻³
 Extinction correction: *SHELXL97*
 Extinction coefficient: 0.00162 (17)

Table 1

Selected geometric parameters (Å, °) for (I).

Te1A—C1A	2.138 (7)	Te1B—C1B	2.127 (7)
Te1A—O1A	2.448 (6)	Te1B—O1B	2.398 (6)
Te1A—Br1A	2.5165 (12)	Te1B—Br1B	2.4929 (12)
Te1A—Br2A	2.6495 (11)	Te1B—Br3B	2.6584 (10)
Te1A—Br3A	2.6731 (11)	Te1B—Br2B	2.6776 (11)
O1A—N1A	1.236 (8)	O1B—N1B	1.271 (8)
O2A—N1A	1.212 (8)	O2B—N1B	1.218 (9)
N1A—C2A	1.466 (9)	N1B—C2B	1.464 (9)
C1A—Te1A—O1A	71.3 (2)	C1B—Te1B—O1B	72.2 (2)
C1A—Te1A—Br1A	94.7 (2)	C1B—Te1B—Br1B	94.3 (2)
O1A—Te1A—Br1A	165.75 (12)	O1B—Te1B—Br1B	166.60 (12)
C1A—Te1A—Br2A	85.6 (2)	C1B—Te1B—Br3B	84.41 (19)
O1A—Te1A—Br2A	84.59 (17)	O1B—Te1B—Br3B	85.96 (15)
Br1A—Te1A—Br2A	92.08 (4)	Br1B—Te1B—Br3B	92.86 (4)
C1A—Te1A—Br3A	85.4 (2)	C1B—Te1B—Br2B	86.66 (19)
O1A—Te1A—Br3A	89.08 (17)	O1B—Te1B—Br2B	87.63 (15)
Br1A—Te1A—Br3A	92.27 (4)	Br1B—Te1B—Br2B	91.68 (4)
Br2A—Te1A—Br3A	170.27 (3)	Br3B—Te1B—Br2B	170.26 (3)
N1A—O1A—Te1A	114.1 (5)	N1B—O1B—Te1B	114.0 (4)
C6A—C1A—Te1A	121.1 (6)	C6B—C1B—Te1B	121.7 (5)
C2A—C1A—Te1A	118.9 (5)	C2B—C1B—Te1B	119.6 (5)

Compound (II)

Crystal data

$\text{C}_8\text{H}_8\text{BrNO}_2\text{Te}$
 $M_r = 357.66$
 Monoclinic, $I2/a$
 $a = 14.175$ (4) Å
 $b = 9.582$ (2) Å
 $c = 15.433$ (4) Å
 $\beta = 95.70$ (2)°
 $V = 2085.8$ (9) Å³
 $Z = 8$

$D_x = 2.278$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 25 reflections
 $\theta = 10.6$ – 18.8°
 $\mu = 6.65$ mm⁻¹
 $T = 298$ K
 Parallelepiped, brown-red
 $0.35 \times 0.22 \times 0.20$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer
 $\theta/2\theta$ scans
 Absorption correction: ψ scan (North *et al.*, 1968)
 $T_{\min} = 0.202$, $T_{\max} = 0.266$
 7028 measured reflections
 2393 independent reflections
 1325 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.043$
 $\theta_{\text{max}} = 27.5^\circ$
 $h = -18 \rightarrow 18$
 $k = -11 \rightarrow 12$
 $l = 0 \rightarrow 20$
 3 standard reflections
 frequency: 120 min
 intensity decay: 0.8%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.043$
 $wR(F^2) = 0.104$
 $S = 1.10$
 2393 reflections
 121 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0117P)^2 + 3.15P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.003$
 $\Delta\rho_{\text{max}} = 0.95$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.81$ e Å⁻³
 Extinction correction: *SHELXL97*
 Extinction coefficient: 0.00020 (7)

Table 2

Selected geometric parameters (Å, °) for (II).

Te—C1	2.098 (6)	O1—N	1.268 (8)
Te—O1	2.307 (6)	O2—N	1.208 (8)
Te—Br	2.5584 (12)	N—C2	1.445 (9)
C1—Te—O1	73.4 (2)	O1—N—C2	117.1 (6)
C1—Te—Br	95.38 (18)	C6—C1—C2	118.4 (6)
O1—Te—Br	168.80 (14)	C6—C1—Te	123.5 (5)
N—O1—Te	115.1 (4)	C2—C1—Te	118.1 (5)
O2—N—O1	120.6 (7)	C1—C2—N	116.3 (7)
O2—N—C2	122.4 (7)	C3—C2—N	120.4 (6)

Table 3
Selected geometric parameters (Å, °) for (III).

Te1—C1	2.038 (4)	O1—N1	1.315 (5)
Te1—O1	2.165 (3)	N1—C2	1.348 (5)
Te1—Br1	2.6401 (16)		
C1—Te1—O1	76.81 (14)	C6—C1—C2	118.6 (3)
C1—Te1—Br1	92.76 (11)	C6—C1—Te1	129.2 (3)
O1—Te1—Br1	169.57 (9)	C2—C1—Te1	112.2 (3)
N1—O1—Te1	116.6 (3)	N1—C2—C3	118.0 (4)
O1—N1—C2	113.5 (4)	N1—C2—C1	120.8 (4)

Compound (III)

Crystal data

$C_8H_8BrNO_2Te$	$D_x = 2.376 \text{ Mg m}^{-3}$
$M_r = 341.66$	Mo $K\alpha$ radiation
Monoclinic, $C2/c$	Cell parameters from 4719 reflections
$a = 11.291 (6) \text{ \AA}$	$\theta = 2.5\text{--}32.0^\circ$
$b = 12.788 (5) \text{ \AA}$	$\mu = 7.25 \text{ mm}^{-1}$
$c = 14.332 (7) \text{ \AA}$	$T = 120 \text{ K}$
$\beta = 112.62 (3)^\circ$	Prism, dark red
$V = 1910.2 (16) \text{ \AA}^3$	$0.20 \times 0.15 \times 0.15 \text{ mm}$
$Z = 8$	

Data collection

Nonius KappaCCD diffractometer (with an Oxford Cryosystems Cryostream cooler)	4540 measured reflections
ω scans with κ offsets	3272 independent reflections
Absorption correction: multi-scan (HKL SCALEPACK; Otwinowski & Minor, 1997)	2511 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.268$, $T_{\max} = 0.337$	$R_{\text{int}} = 0.032$
	$\theta_{\text{max}} = 32.0^\circ$
	$h = -16 \rightarrow 16$
	$k = -18 \rightarrow 13$
	$l = -20 \rightarrow 21$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0702P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.040$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.123$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 1.09$	$\Delta\rho_{\text{max}} = 1.30 \text{ e \AA}^{-3}$
3272 reflections	$\Delta\rho_{\text{min}} = -2.03 \text{ e \AA}^{-3}$
112 parameters	Extinction correction: SHELXL97
H-atom parameters constrained	Extinction coefficient: 0.00086 (19)

H atoms were treated as riding in idealized positions, with C—H distances of 0.93–0.98 Å, depending on atom type and temperature. A torsional parameter was refined for each methyl group. Displacement parameters for H atoms were assigned as $1.2U_{\text{eq}}$ of the attached atom (1.5 for methyl groups). Friedel pairs were averaged for (I), since refinement of the Flack (1983) parameter led to a value of 0.509 (8), indicative of a racemic twin. The largest residual peak was 1.45 Å from atom Br3B for (I), 0.83 Å from the Te atom for (II) and 0.55 Å from atom Te1 for (III).

For compounds (I) and (III), data collection: COLLECT (Nonius, 2000); cell refinement: HKL DENZO and SCALEPACK (Otwinowski & Minor, 1997); data reduction: HKL DENZO and SCALEPACK. For compound (II), data collection: CAD-4 EXPRESS (Enraf–Nonius, 1994); cell refinement: CAD-4 EXPRESS; data reduction: maXus (Mackay *et al.*, 1999). Program(s) used to solve structure: SIR97 (Altomare *et al.*, 1999) for (I) and (II), and SIR92 (Altomare *et al.*, 1994) for (III). For all three compounds, program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEPII (Johnson, 1976).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SQ1028). Services for accessing these data are described at the back of the journal.

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