Table 1. *Selected geometric parameters (Å, °)*

S1*A*—C7*A*	1.7421 (17)	S1*B*—C7*B*	1.7404 (18)
S1*A*—C1*A*	1.7651 (15)	S1*B*—C1*B*	1.7672 (16)
N1*A*—C7*A*	1.369 (2)	N1*B*—C7*B*	1.3711 (19)
N1*A*—C8*A*	1.3779 (19)	N1*B*—C8*B*	1.3778 (19)
N1*A*—C6*A*	1.4076 (18)	N1*B*—C6*B*	1.4098 (18)
N2*A*—C7*A*	1.308 (2)	N2*B*—C7*B*	1.306 (2)
N2*A*—N3*A*	1.412 (2)	N2*B*—N3*B*	1.410 (2)
N3*A*—C8*A*	1.317 (2)	N3*B*—C8*B*	1.318 (2)
C8*A*—C9*A*	1.469 (2)	C8*B*—C9*B*	1.466 (2)
C9*A*—C10*A*	1.489 (2)	C9*B*—C10*B*	1.478 (2)
C9*A*—C11*A*	1.487 (3)	C9*B*—C11*B*	1.483 (3)
C7*A*—S1*A*—C1*A*	89.22 (7)	C7*B*—S1*B*—C1*B*	89.26 (7)
C7*A*—N1*A*—C8*A*	105.51 (13)	C7*B*—N1*B*—C8*B*	105.48 (13)
C7*A*—N1*A*—C6*A*	114.83 (12)	C8*B*—N1*B*—C6*B*	139.87 (13)
C8*A*—N1*A*—C6*A*	139.63 (13)	C7*B*—N2*B*—N3*B*	105.44 (13)
C7*A*—N2*A*—N3*A*	105.30 (14)	C2*B*—C1*B*—S1*B*	126.40 (12)
C5*A*—C6*A*—N1*A*	128.64 (13)	C5*B*—C6*B*—N1*B*	128.66 (13)
N2*A*—C7*A*—S1*A*	135.70 (13)	N2*B*—C7*B*—S1*B*	135.63 (13)
N3*A*—C8*A*—C9*A*	127.96 (15)	N3*B*—C8*B*—C9*B*	127.05 (15)
N1*A*—C8*A*—C9*A*	123.53 (14)	N1*B*—C8*B*—C9*B*	124.49 (14)

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H2O1···N3*B*	0.88 (4)	2.16 (4)	3.011 (3)	163 (3)
O2*W*—H2O2···N3*A*^{i}	0.85 (4)	2.26 (4)	3.067 (3)	157 (3)
O2*W*—H1O2···N2*A*^{ii}	0.84 (3)	2.14 (3)	2.980 (2)	177 (3)
O1*W*—H1O1···N2*B*^{iii}	0.80 (3)	2.22 (3)	3.006 (2)	167 (3)

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

H atoms on O1*W* and O2*W* were located by difference Fourier synthesis, while others were placed in calculated positions using a riding model.

Data collection: *SMART* (Siemens, 1996). Cell refinement: *SAINT* (Siemens, 1996). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997). Molecular graphics: *ZORTEP* (Zsolnai, 1997). Software used to prepare material for publication: *SHELXL*97 and *PARST* (Nardelli, 1983, 1995).

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Acta Cryst. (1999). C**55**, 1121–1123

Tetraethylammonium (*N*,*N*-diethyldithiocarbamato-*S*,*S'*)tetraiodotellurate(IV)

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Abstract

The title compound, $[(C_2H_5)_4N][TeI_4L]$ (*L* is diethyldithiocarbamate, $C_5H_{10}NS_2$) or $C_8H_{20}N^+\cdot C_5H_{10}I_4NS_2Te^-$, was prepared by the addition of tetraethylammonium

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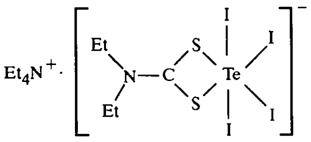
iodide to TeLI_3. The anionic complex is monomeric, with the geometry around the central Te atom being distorted octahedral. The four Te—I distances are 2.921 (1), 3.100 (1), 2.933 (1) and 2.950 (1) Å.

Comment

Complexes of tellurium with sulfur ligands possess interesting stereochemical features and supramolecular associations. Our interest revolves around the synthesis and structural studies of mixed halide–dithiocarbamate complexes of tellurium in its +II and +IV oxidation states (Krishna Kumar *et al.*, 1993*a*,*b*, 1996). In the solid state, all three tellurium(IV) complexes of the type Te$L_{4-n}I_n$ (n = 1, 2 or 3) exhibit distorted pentagonal-bipyramidal geometry around the Te atom. While TeL_3I is monomeric (Krishna Kumar, 1995), TeL_2I_2 is an iodide-bridged dimer (Krishna Kumar *et al.*, 1993*b*) and TeLI_3 is polymeric with iodide bridges (Krishna Kumar *et al.*, 1996). The title compound, (I), is the first example of an anionic mixed iodide–dithiocarbamate complex of tellurium(IV).



(I)

Structural studies show that the anionic moiety is monomeric. The S1 and S2 atoms of the dithiocarbamate anion are bound to the tellurium at distances of 2.581 (1) and 2.528 (1) Å, respectively. The average distance is shorter than the average Te—S distances observed in TeL_3I and TeL_2I_2, but longer than that observed in TeLI_3. However, in the present instance, the dithiocarbamate ligand is symmetrically bound to tellurium, compared with TeLI_3. All four Te—I distances are longer than the sum of the covalent radii of Te and I (2.70 Å), but are of the same order as observed (2.94 Å) for $[TeI_6]^{2-}$ compounds (Abriel, 1982; Kiriyama *et al.*, 1986). The I1 atom is *trans* to I3, and atoms I2 and I4 are *trans* to S2 and S1, respectively. The longer Te—I2 bond [3.100 (1) Å], compared with the other Te—I bond distances [2.921 (1)–2.950 (1) Å], is attributed to the *trans* influence of S2, which has the shortest Te—S bond.

The geometry around the central Te atom can thus be described as distorted octahedral (Table 1). The distortion arises due to the presence of two large I atoms and the small 'bite' of the chelating dithiocarbamate ligand. It is interesting to note that the lone pair of electrons on the Te^{IV} atom is stereochemically inert.

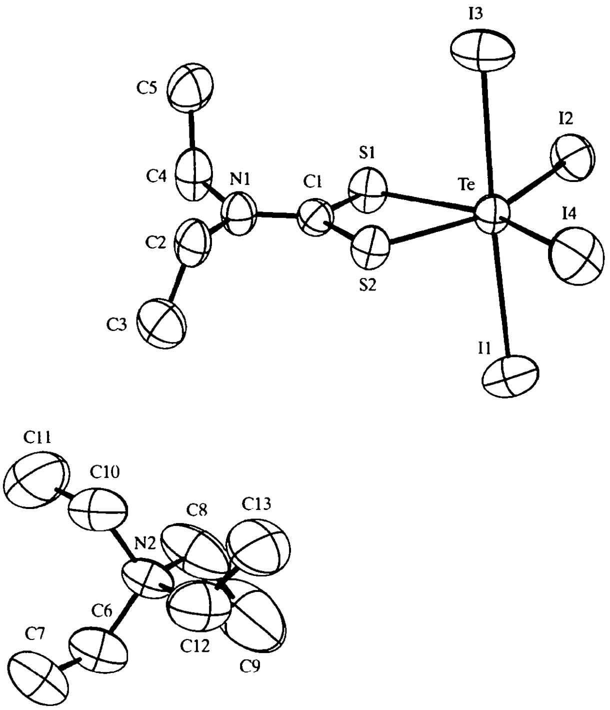


Fig. 1. The molecular structure of (I), shown with 50% probability ellipsoids. H atoms have been omitted for clarity.

Experimental

Tetraethylammonium iodide (0.4 mmol, 0.103 g) was added to a suspension of TeLI_3 (0.2 mmol, 0.131 g) in dichloromethane (30 ml), resulting in a clear dark-red solution. The solvent was evaporated at room temperature and the remaining gummy residue was redissolved in acetonitrile, from which crystals suitable for analysis were obtained. Analysis found: C 17.1, H 3.3, N 3.0, I 55.7, Te 14.1%; calculated for $C_{13}H_{30}I_4N_2S_2Te$: C 17.3, H 3.4, N 2.9, I 55.6, Te 14.0%.

Crystal data

$(C_8H_{20}N)[TeI_4(C_5H_{10}NS_2)]$
M_r = 913.71
Monoclinic
$P2_1/n$
a = 11.568 (1) Å
b = 14.356 (2) Å
c = 16.243 (1) Å
β = 98.30 (1)°
V = 2669.2 (5) Å^3
Z = 4
D_x = 2.274 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 36 reflections
θ = 8–25°
μ = 5.897 mm^{-1}
T = 293 (2) K
Needle
0.56 × 0.26 × 0.22 mm
Black

Data collection

Siemens $P4$ diffractometer
θ–2θ scans

R_{int} = 0.024
θ_{max} = 27.5°

Absorption correction:
 ψ scan (*XSCANS*; Siemens, 1994)
 $T_{\min} = 0.172$, $T_{\max} = 0.273$
7512 measured reflections
6067 independent reflections
4258 reflections with $I > 2\sigma(I)$

$h = -1 \rightarrow 15$
$k = -1 \rightarrow 18$
$l = -21 \rightarrow 21$
3 standard reflections every 97 reflections
 intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.096$
$S = 0.934$
6067 reflections
199 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0503P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.960$ e Å^{-3}
$\Delta\rho_{\min} = -0.943$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

Te—S2	2.528 (1)	Te—I4	2.950 (1)
Te—S1	2.581 (1)	Te—I2	3.100 (1)
Te—I1	2.921 (1)	S1—C1	1.735 (5)
Te—I3	2.933 (1)	S2—C1	1.734 (5)
S2—Te—S1	70.52 (4)	I3—Tc—I4	89.68 (2)
S2—Te—I1	91.09 (4)	S2—Te—I2	151.15 (3)
S1—Te—I1	93.94 (4)	S1—Te—I2	80.63 (3)
S2—Te—I3	90.55 (4)	I1—Te—I2	90.48 (2)
S1—Te—I3	89.16 (4)	I3—Te—I2	89.38 (2)
I1—Te—I3	176.84 (2)	I4—Te—I2	125.34 (2)
S2—Te—I4	83.51 (3)	C1—S1—Te	85.6 (2)
S1—Te—I4	153.99 (3)	C1—S2—Te	87.3 (2)
I1—Te—I4	87.82 (2)		

Data collection: *XSCANS* (Siemens, 1994). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*97.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: GD1007). Services for accessing these data are described at the back of the journal.

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2,2-Bis(2-methoxyphenyl)-4-methyl-2*H*-benzo[*h*]chromene

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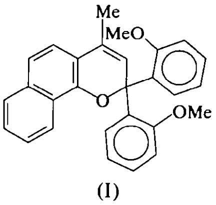
Abstract

In the title compound, $C_{28}H_{24}O_3$, the pyran ring adopts a half-chair conformation. The dihedral angle between the pyran and the naphthalene ring system is 8.50 (6)°. The phenyl rings are nearly orthogonal to each other and they form dihedral angles of 84.21 (7) and 49.89 (8)° with the pyran ring.

Comment

The title compound is a newly added member of the chromane family. Generally, chromane derivatives are very useful in the treatment of inflammation, atherosclerosis, restenosis and immune disorders such as arthritis and transplant rejection (Trivedi, 1998). Furthermore, it has been very recently reported that 3,4-disubstituted chromane derivatives are useful in the prevention and treatment of estrogen-related diseases or syndromes (Jacobson *et al.*, 1998). The structure determination of the title compound, (I), was carried out in order to elucidate the molecular conformation.



(I)

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