best planes formed by C1, C2 and the atoms bonded directly to them [*cf.* 2,3:5,6-di-*O*-isopropylidene-α-D-mannofuranose (Sheldrick, Mackie & Akrigg, 1985)].

Variations of the O···O distances in this and other structures with only one hydrogen bond per molecule seem to indicate that the energy of the hydrogen bond is not sufficient to dominate the crystal packing.

We wish to thank Andrea Whitehead for taking a series of X-ray photographs and the University of Leeds Computing Service for the provision of computing facilities.

References

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

Motherwell, W. D. S. & Clegg, W. (1978). *PLUTO78*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.

Sheldrick, B., Mackie, W. & Akrigg, D. (1985). *Acta Cryst.* C41, 431–433.

Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

Sheldrick, G. M. (1985). *SHELXS86*. *Crystallographic Computing* 3, edited by G. M. Sheldrick, C. Krüger & R. Goddard, pp. 175–189. Oxford Univ. Press.

Acta Cryst. (1988). C**44**, 579–581

Structure of Dimethyl(picrato)(trifluoromethyl)tellurium

By Hans Preut, Jürgen Fischer and Dieter Naumann

Fachbereich Chemie der Universität Dortmund, Postfach 500500, *D*-4600 *Dortmund* 50, *Federal Republic of Germany*

(*Received* 29 *July* 1987; *accepted* 10 *November* 1987)

Abstract. $[Te(C_6H_2N_3O_7)(CF_3)(CH_3)_2]$, $M_r = 454{\cdot}78$, monoclinic, $P2_1/c$, $a = 12{\cdot}468\,(5)$, $b = 7{\cdot}239\,(9)$, $c = 16{\cdot}346\,(9)$ Å, $\beta = 103{\cdot}31\,(5)°$, $V = 1436\,(2)$ Å^3, $Z = 4$, $D_x = 2{\cdot}104$ Mg m^{-3}, $\lambda(\mathrm{Mo}\,K\alpha) = 0{\cdot}71069$ Å, $\mu = 2{\cdot}1$ mm^{-1}, $F(000) = 872$, $T = 291\,(1)$ K, final $R = 0{\cdot}033$ for 2255 unique observed $[F \geq 3{\cdot}0\sigma(F)]$ diffractometer data and 221 variables. In the crystalline state $[(CH_3)_2Te(CF_3)][C_6H_2O(NO_2)_3]$ molecules are linked *via* weak intermolecular Te···O contacts [3·134 (4), 3·138 (3) Å] such that if the free electron pair is taken into account a distorted pseudopentagonal bipyramid around Te is formed with C (trifluoromethyl) [Te—C 2·192 (5) Å] and O (picrate) [Te—O 2·496 (3) Å] in the apical positions and two C (methyl) [Te—C 2·115 (5), 2·111 (5) Å], two O (NO_2) atoms of two neighbouring molecules and the free electron pair in the equatorial positions. In the picrate group the planes through the NO_2 groups form dihedral angles of 46·0 (1), 7·8 (1) and 14·4 (1)° with the plane through the C atoms of the ring. Aromatic C—C bond lengths in the picrate ring range between 1·366 (6) and 1·445 (6) Å and intra-ring C—C—C angles between 111·5 (4) and 126·1 (4)°.

Experimental. Dimethyl(trifluoromethyl)tellurium iodide (Wilkes, 1984) reacts with picric acid to give the hitherto unknown title compound. Yellow single crystals could be obtained by slow evaporation of an ethanol–water solution at room temperature. Crystal size ~0·13 × 0·29 × 0·19 mm, $\omega/2\theta$ scan, scan speed 2·0–5·0° min^{-1} in θ, Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$; lattice parameters from least-squares fit with 25 reflections up to $2\theta = 26{\cdot}2°$ equally distributed in reciprocal space; six standard reflections recorded every 2·5 h, only random

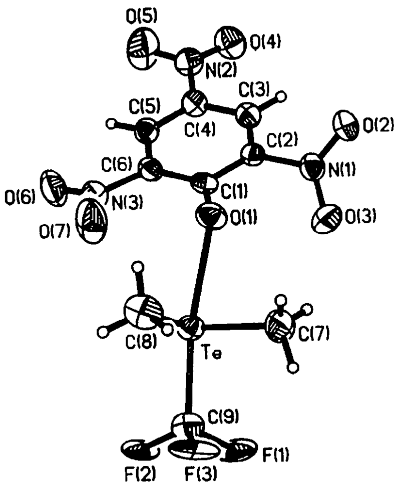

Fig. 1. View (*SHELXTL-Plus* graphic) of the molecule, showing the atom-numbering scheme. The F atoms are disordered and for each F atom two positions have been refined. Only one of these two positions is shown in the figure.

0108-2701/88/030579-03$03.00

Table 1. *Atomic coordinates and equivalent isotropic or isotropic thermal parameters* (Å^2 × 10^3)

$$U_{\rm eq} = \tfrac{1}{3}\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}/U$
Te	0·2161 (1)	0·2999 (1)	0·6477 (1)	34 (1)
F(1)	0·0514 (7)	0·5825 (10)	0·6589 (5)	72 (3)
F(2)	0·1351 (5)	0·4900 (16)	0·7818 (4)	77 (3)
F(3)	0·0049 (5)	0·3340 (9)	0·7042 (5)	63 (3)
F(1′)	0·1294 (16)	0·6059 (30)	0·7308 (13)	178 (6)†
F(2′)	0·0974 (14)	0·3777 (25)	0·7810 (11)	157 (6)†
F(3′)	0·0108 (13)	0·4859 (24)	0·6625 (10)	147 (5)†
O(1)	0·3294 (3)	0·1473 (5)	0·5595 (2)	43 (1)
O(2)	0·3512 (3)	0·1990 (5)	0·3352 (2)	56 (1)
O(3)	0·2708 (3)	0·3592 (6)	0·4130 (2)	58 (1)
O(4)	0·7526 (3)	0·3181 (5)	0·4340 (3)	57 (1)
O(5)	0·8241 (3)	0·2316 (8)	0·5597 (3)	81 (2)
O(6)	0·6081 (3)	0·1544 (7)	0·7567 (2)	65 (2)
O(7)	0·4406 (3)	0·0668 (8)	0·7168 (3)	83 (2)
N(1)	0·3480 (3)	0·2691 (5)	0·4016 (2)	38 (1)
N(2)	0·7451 (3)	0·2698 (6)	0·5032 (3)	43 (1)
N(3)	0·5237 (3)	0·1339 (6)	0·7021 (2)	45 (1)
C(1)	0·4243 (4)	0·1906 (5)	0·5518 (3)	32 (1)
C(2)	0·4442 (3)	0·2474 (6)	0·4718 (3)	31 (1)
C(3)	0·5451 (4)	0·2727 (6)	0·4541 (3)	34 (1)
C(4)	0·6373 (4)	0·2534 (6)	0·5200 (3)	35 (1)
C(5)	0·6272 (4)	0·2130 (6)	0·6006 (3)	36 (1)
C(6)	0·5247 (4)	0·1849 (6)	0·6160 (3)	32 (1)
C(7)	0·0980 (4)	0·2796 (9)	0·5323 (3)	57 (2)
C(8)	0·1838 (4)	0·0354 (7)	0·6909 (3)	54 (2)
C(9)	0·0925 (4)	0·4299 (9)	0·7051 (3)	56 (2)

† Isotropically refined.

Table 2. *Bond distances* (Å), *angles* (°), *short intramolecular and intermolecular contacts* (Å,°)

Te—O(1)	2·496 (3)	O(4)—N(2)	1·208 (6)
Te—C(7)	2·115 (5)	O(5)—N(2)	1·217 (6)
Te—C(8)	2·111 (5)	O(6)—N(3)	1·222 (5)
Te—C(9)	2·192 (5)	O(7)—N(3)	1·218 (6)
F(1)—C(9)	1·369 (8)*	N(1)—C(2)	1·465 (5)
F(2)—C(9)	1·318 (8)*	N(2)—C(4)	1·436 (6)
F(3)—C(9)	1·291 (8)*	N(3)—C(6)	1·457 (5)
F(1′)—C(9)	1·387 (21)*	C(1)—C(2)	1·445 (6)
F(2′)—C(9)	1·285 (16)*	C(1)—C(6)	1·437 (6)
F(3′)—C(9)	1·166 (15)*	C(2)—C(3)	1·366 (6)
O(1)—C(1)	1·259 (6)	C(3)—C(4)	1·391 (6)
O(2)—N(1)	1·208 (5)	C(4)—C(5)	1·384 (6)
O(3)—N(1)	1·213 (5)	C(5)—C(6)	1·373 (6)
C(7)—Te—O(1)	79·9 (2)	C(3)—C(2)—C(1)	126·1 (4)
C(8)—Te—O(1)	88·2 (2)	C(4)—C(3)—C(2)	117·3 (4)
C(8)—Te—C(7)	95·1 (2)	C(3)—C(4)—N(2)	119·1 (4)
C(9)—Te—O(1)	169·7 (2)	C(5)—C(4)—N(2)	119·6 (4)
C(9)—Te—C(7)	89·9 (2)	C(5)—C(4)—C(3)	121·2 (4)
C(9)—Te—C(8)	92·3 (2)	C(6)—C(5)—C(4)	119·9 (4)
C(1)—O(1)—Te	128·2 (3)	C(1)—C(6)—N(3)	120·9 (4)
O(3)—N(1)—O(2)	123·8 (4)	C(5)—C(6)—N(3)	115·4 (4)
C(2)—N(1)—O(2)	118·0 (4)	C(5)—C(6)—C(1)	123·5 (4)
C(2)—N(1)—O(3)	118·2 (4)	F(1)—C(9)—Te	108·7 (4)*
O(5)—N(2)—O(4)	123·5 (4)	F(2)—C(9)—Te	112·3 (4)*
C(4)—N(2)—O(4)	118·7 (4)	F(2)—C(9)—F(1)	106·0 (7)*
C(4)—N(2)—O(5)	117·8 (4)	F(3)—C(9)—Te	116·1 (4)*
O(7)—N(3)—O(6)	122·2 (4)	F(3)—C(9)—F(1)	102·7 (6)*
C(6)—N(3)—O(6)	118·5 (4)	F(3)—C(9)—F(2)	110·0 (7)*
C(6)—N(3)—O(7)	119·3 (4)	F(1′)—C(9)—Te	107·9 (8)*
C(2)—C(1)—O(1)	121·6 (4)	F(2′)—C(9)—Te	113·9 (8)*
C(6)—C(1)—O(1)	126·9 (4)	F(2′)—C(9)—F(1′)	92·2 (11)*
C(6)—C(1)—C(2)	111·5 (4)	F(3′)—C(9)—Te	119·8 (9)*
C(1)—C(2)—N(1)	117·3 (4)	F(3′)—C(9)—F(1′)	92·6 (12)*
C(3)—C(2)—N(1)	116·5 (4)	F(3′)—C(9)—F(2′)	121·3 (12)*
O(1)···O(3)	2·796 (6)	O(5)···C(5)	2·693 (6)
O(1)···O(7)	2·689 (6)	O(6)···C(5)	2·651 (6)
O(2)···C(3)	2·782 (6)	O(7)···C(1)	2·805 (6)
O(4)···C(3)	2·702 (6)		
Te···O(2$^{\rm i}$)	3·138 (3)	C(9)—Te···O(2$^{\rm i}$)	81·6 (2)
Te···O(4$^{\rm ii}$)	3·134 (4)	O(1)—Te···O(4$^{\rm ii}$)	90·1 (1)
		C(7)—Te···O(4$^{\rm ii}$)	79·1 (2)
O(1)—Te···O(2$^{\rm i}$)	108·5 (1)	C(8)—Te···O(4$^{\rm ii}$)	174·1 (2)
C(7)—Te···O(2$^{\rm i}$)	168·1 (1)	C(9)—Te···O(4$^{\rm ii}$)	88·4 (2)
C(8)—Te···O(2$^{\rm i}$)	77·1 (2)	O(2$^{\rm i}$)···Te···O(4$^{\rm ii}$)	108·8 (1)

Symmetry operation: (i) x, $0·5-y$, $0·5+z$; (ii) $1-x$, $1-y$, $1-z$.

* These values belong to disordered positions.

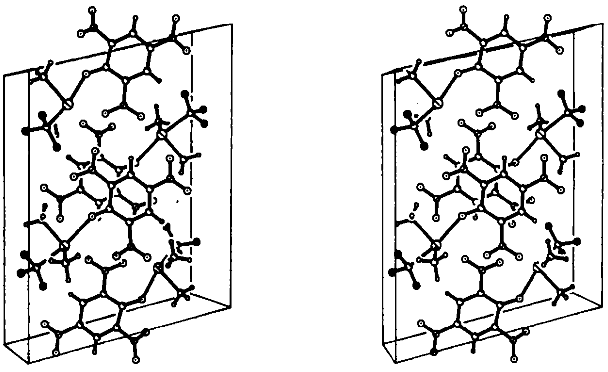

Fig. 2. Stereoscopic view (*SHELXTL-Plus* graphic) of the unit cell (*c* vertical, *a* nearly horizontal).

deviations; 5623 reflections measured, $1·5 \leq \theta \leq 25·0°$, $-14 \leq h \leq 14$, $0 \leq k \leq 8$, $-19 \leq l \leq 19$; after averaging ($R_{\rm int} = 0·012$): 2525 unique reflections, 2255 with $F \geq 3·0\sigma(F)$; Lorentz–polarization correction and absorption correction *via* ψ scans; max./min. transmission 1·00/0·83; systematic absences ($h0l$) $l = 2n + 1$, ($0k0$) $k = 2n + 1$ conform to space group $P2_1/c$; structure solution *via* Patterson function, ΔF syntheses and full-matrix least-squares refinement with anisotropic temperature factors for all non-H atoms except three disordered positions for the F atoms which were isotropically refined, H atoms were placed in calculated positions (C—H 0·95 Å) and a common isotropic temperature factor was refined for these atoms; the F atoms are disordered, for each F atom two positions F and F′ have been refined with the occupancy factor 0·5 and anisotropic temperature factors for F and isotropic temperature factors for F′; refinement on F with 2255 reflections and 221 refined parameters; $w = 1·0/[\sigma^2(F) + 0·0005F^2]$; $S = 1·26$, $R = 0·033$, $wR = 0·036$, $(\Delta/\sigma)_{\rm max} = 0·8$ for the disordered F positions and 0·1 for the remaining parameters; no extinction correction; largest peak in final ΔF map ±0·8 (2) e Å^{-3}; complex neutral-atom scattering factors from Cromer & Mann (1968) and Cromer & Liberman (1970); programs: *SHELXTL-Plus* (Sheldrick, 1987) for the structure solution, refinement and plots, Enraf–Nonius *SDP-Plus* (Frenz, 1985) for data reduction.

The molecule and the numbering scheme are shown in Fig. 1 and a stereoscopic view of the unit cell in Fig. 2. Positional parameters and the isotropic temperature factors or the equivalent values of the anisotropic temperature factors for the non-H atoms are given in Table 1.* Bond lengths and angles are given in Table 2.

Related literature. A distorted pseudopentagonal bipyramid around Te is formed as in dimethyltellurium dichloride (Ziolo & Troup, 1983). As in dimethylammonium picrate (Walkinshaw, 1986) the geometry of the picrate ring shows significant distortions from the standard benzene ring with C—C 1·395 Å and C—C—C 120°. Lattice-energy calculations with the program *PCK*83 (Williams, 1983) using the potential curves given by Mirsky (1978) show that the lattice energies for the crystal structure with the conformation F(1), F(2) and F(3) and the crystal structure with the conformation F(1′), F(2′) and F(3′) differ only by 1·0 kJ mol^{-1}.

---

* Lists of H-atom coordinates, anisotropic thermal parameters, structure-factor amplitudes and least-squares planes and dihedral angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44524 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Cromer, D. T. & Liberman, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.

Cromer, D. T. & Mann, J. B. (1968). *Acta Cryst.* A**24**, 321–324.

Frenz, B. A. (1985). *Enraf–Nonius SDP-Plus Structure Determination Package*, version 3·0. Enraf–Nonius, Delft, The Netherlands.

Mirsky, K. (1978). In *Computing in Crystallography*. Delft Univ. Press.

Sheldrick, G. M. (1987). *SHELXTL-Plus*, release 2. *An Integrated System for Solving, Refining and Displaying Crystal Structures from Diffraction Data*. For Nicolet *R*3*m*/V. Univ. of Göttingen, Federal Republic of Germany.

Walkinshaw, M. D. (1986). *Acta Cryst.* C**42**, 246–249.

Wilkes, B. (1984). Dissertation, Univ. of Dortmund, Federal Republic of Germany.

Williams, D. E. (1983). *QCPE Bull.* No. 481.

Ziolo, R. F. & Troup, J. M. (1983). *J. Am. Chem. Soc.* **105**, 229–235.

Acta Cryst. (1988). C**44**, 581–583

Diméthylamino-3 Diméthyl-5,8 Carbazolecarbaldéhyde-4

Par B. Viossat

Laboratoire de Chimie minérale, UFR de Médecine et de Pharmacie, 34 *rue du Jardin des Plantes,*
86034 *Poitiers CEDEX, France*

N. Rodier

Laboratoire de Chimie minérale, Faculté des Sciences pharmaceutiques et biologiques, rue J.-B. Clément,
92296 *Châtenay-Malabry CEDEX, France*

et C. Gansser et C. Viel

Laboratoire de Chimie thérapeutique UA 496 *CNRS, Faculté des Sciences pharmaceutiques et biologiques,*
rue J.-B. Clément, 92296 *Châtenay-Malabry CEDEX, France*

(*Reçu le* 29 *avril* 1987, *accepté le* 2 *novembre* 1987)

Abstract. $C_{17}H_{18}N_2O$, $M_r = 266{\cdot}3$, monoclinic, $P2_1/c$, $a = 8{\cdot}397\,(1)$, $b = 13{\cdot}506\,(2)$, $c = 13{\cdot}656\,(3)$ Å, $\beta = 117{\cdot}01\,(1)^\circ$, $V = 1379{\cdot}8\,(8)$ Å^3, $Z = 4$, $D_m = 1{\cdot}24$, $D_x = 1{\cdot}282$ Mg m^{-3}, $\lambda(\text{Mo }K\alpha) = 0{\cdot}7107$ Å, $\mu = 0{\cdot}075$ mm^{-1}, $F(000) = 568$, room temperature. $R = 0{\cdot}035$ for 1641 independent reflections $[I > 3\sigma(I)]$. The Vilsmeier–Haack formylation of 3-dimethylamino-5,8-dimethylcarbazole leads to several carbaldehydes depending on the pH of the medium. One of them, the title compound, has been analysed by X-ray crystallography to determine with certainty the position of the formyl group. The usual planar geometry of the carbazole group is distorted in this molecule with the largest deviation from the mean plane being 0·186 (2) Å. The molecules are linked together by N—H···O hydrogen bonds and form layers which spread out along the $y = \pm\frac{1}{4}$ planes.

Partie expérimentale. Masse volumique par flottaison. Cristal prismatique: 0,25 × 0,30 × 0,40 mm. Dimensions de la maille déterminées sur monocristal avec 25 réflexions telles que $7{,}79 \le \theta \le 19{,}38^\circ$. Diffractomètre Enraf–Nonius CAD-4. $0{,}039 \le (\sin\theta)/\lambda \le 0{,}595$ Å^{-1}; $0 \le h \le 9$, $0 \le k \le 16$, $-16 \le l \le 14$. Réflexions de

0108-2701/88/030581-03$03.00