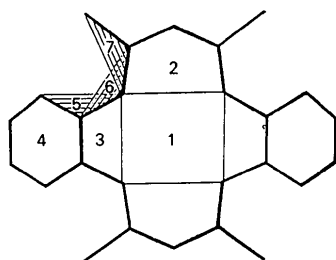


Table 3. Comparison of selected dihedral angles ($^{\circ}$) for $M(\text{tmtaa})$ compounds

Planes	H_2tmtaa	$[\text{Fe}(\text{tmtaa})]$	$[\text{Pd}(\text{tmtaa})]$	$[\text{Cu}(\text{tmtaa})]$
1-2	34.3	24.5	14.7	26.0
1-3	20.1	17.5	1.4	16.2
1-4	24.6	23.7	13.0	21.0
5-6	45.6	31.5	25.0	34.1
6-7	3.6	9.27	5.2	5

the metal center comes as close as it can to achieving a square-planar coordination with tmtaa without forcing the centrosymmetric geometry upon the ligand.

Although only a handful of $[M(\text{tmtaa})]$ -type complexes have been structurally characterized to date, it is worthwhile to compare some of their features. Table 3 presents the dihedral angles for these compounds. These dihedral angles are good indicators of the degree of deviation of the macrocyclic ligand from planarity. The free ligand as well as the ligands in the complexes with iron and copper have similar saddle-shape conformations. For $[\text{Pd}(\text{tmtaa})]$, on the

other hand, deviations from planarity are much smaller. Table 3 also shows that the shapes of the $[\text{Cu}(\text{tmtaa})]$ and $[\text{Fe}(\text{tmtaa})]$ molecules are very similar, despite a smaller displacement of the Cu center out of the N_4 plane.

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Structure of 3,5-Dimethyl-1,2-tellurazole

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Abstract. 3,5-Dimethyl-1,2-tellurazole, $\text{C}_5\text{H}_7\text{NTe}$, $M_r = 208.7$, monoclinic, $P2_1/c$, $a = 13.592$ (3), $b = 11.460$ (3), $c = 25.751$ (9) Å, $\beta = 101.08$ (2) $^{\circ}$, $V = 3936$ (2) Å 3 , $Z = 24$, $D_x = 2.113$ g cm $^{-3}$, $\lambda(\text{Mo K}\alpha) = 0.71069$ Å, $\mu = 44.3$ cm $^{-1}$, $F(000) = 2304$, $T = 298$ K, final $R = 0.045$ and $wR = 0.057$ for 5298 reflections with $I > 3\sigma(I)$. The title compound is monomeric in the gaseous state and in solution, and forms $\text{Te}\cdots\text{N}$ intermolecular bonds in the solid state.

Introduction. Monocyclic derivatives of 1,2-tellurazole were synthesized for the first time in 1983 (Lucchesini & Bertini, 1983) by reacting α -acetylenic

oxime-*O*-sulfonic acids with potassium telluride in aqueous medium. They are rather stable solids, which can be crystallized from the usual organic solvents and from the melt without decomposition. Their melting point is considerably higher than that of the selenium-containing isologues, which are liquid at room temperature (Lucchesini, Bertini & De Munno, 1984); it is also higher than that of the tellurophene analogues (Mack, 1966). Low solubility and high melting point were also found for 1,2,5-telluradiazole, with respect to the selenium homologue (Bertini, Dapporto, Lucchesini, Sega & De Munno, 1984).

Table 1. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement coefficients ($\text{\AA}^2 \times 10^3$)Equivalent isotropic U is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Te(1)	4418 (1)	10699 (1)	992 (1)	59 (1)
N(1)	3774 (6)	9290 (6)	586 (3)	63 (3)
C(11)	4159 (8)	11417 (8)	250 (4)	67 (3)
C(12)	3711 (7)	10673 (9)	-99 (4)	68 (3)
C(13)	3526 (7)	9544 (9)	76 (3)	63 (3)
C(14)	4398 (11)	12671 (10)	125 (5)	90 (5)
C(15)	3004 (10)	8646 (11)	-296 (4)	87 (5)
Te(2)	3580 (1)	7396 (1)	1141 (1)	60 (1)
N(2)	3137 (6)	6083 (6)	1596 (3)	61 (3)
C(21)	2177 (7)	8048 (8)	1174 (4)	57 (3)
C(22)	1735 (7)	7300 (8)	1467 (4)	60 (3)
C(23)	2244 (7)	6249 (8)	1662 (3)	54 (3)
C(24)	1714 (8)	9109 (8)	923 (4)	69 (3)
C(25)	1743 (7)	5364 (9)	1951 (4)	68 (3)
Te(3)	4114 (1)	4071 (1)	1491 (1)	59 (1)
N(3)	5156 (6)	2784 (6)	1458 (3)	63 (3)
C(31)	5210 (7)	4602 (8)	2125 (3)	58 (3)
C(32)	5967 (8)	3900 (9)	2176 (4)	69 (4)
C(33)	5913 (7)	2913 (8)	1811 (4)	60 (3)
C(34)	5088 (9)	5647 (9)	2458 (4)	75 (4)
C(35)	6815 (8)	2121 (9)	1850 (5)	75 (4)
Te(4)	8508 (1)	6544 (1)	1066 (1)	63 (1)
N(4)	8058 (6)	7886 (6)	1489 (3)	61 (3)
C(41)	7111 (7)	5900 (8)	1098 (4)	57 (3)
C(42)	6631 (7)	6673 (9)	1355 (4)	65 (3)
C(43)	7160 (7)	7720 (7)	1562 (3)	55 (3)
C(44)	6659 (8)	4816 (8)	845 (5)	75 (4)
C(45)	6628 (1)	8597 (1)	1857 (1)	73 (4)
Te(5)	9452 (1)	3245 (1)	1045 (1)	56 (1)
N(5)	8787 (1)	4565 (1)	575 (1)	60 (3)
C(51)	9768 (1)	2622 (1)	340 (1)	56 (3)
C(52)	9359 (1)	3360 (1)	-60 (1)	62 (3)
C(53)	8820 (7)	4384 (8)	82 (4)	60 (3)
C(54)	10334 (9)	1561 (9)	269 (4)	80 (4)
C(55)	8312 (9)	5178 (9)	-334 (4)	71 (4)
Te(6)	9125 (1)	-84 (1)	1479 (1)	58 (1)
N(6)	10208 (6)	1200 (6)	1511 (3)	59 (3)
C(61)	10169 (7)	-727 (8)	2108 (3)	60 (3)
C(62)	10964 (8)	-49 (8)	2188 (4)	67 (3)
C(63)	10953 (7)	957 (8)	1862 (4)	64 (3)
C(64)	10015 (10)	-1809 (9)	2419 (4)	82 (4)
C(65)	11861 (9)	1740 (11)	1945 (5)	91 (5)

Table 2. Bond lengths (\AA) and angles ($^\circ$) (average values)

Te(1)—N(1)	2.028 (7)	Te(1)—C(11)	2.047 (10)
N(1)—C(13)	1.322 (11)	C(11)—C(12)	1.303 (13)
C(11)—C(14)	1.521 (15)	C(12)—C(13)	1.409 (14)
C(13)—C(15)	1.491 (15)	Te(2)—N(2)	2.068 (8)
Te(2)—C(21)	2.066 (10)	N(2)—C(23)	1.271 (12)
C(21)—C(22)	1.356 (14)	C(21)—C(24)	1.462 (13)
C(22)—C(23)	1.431 (12)	C(23)—C(25)	1.496 (14)
Te(3)—N(3)	2.058 (8)	Te(3)—C(31)	2.079 (8)
N(3)—C(33)	1.245 (11)	C(31)—C(32)	1.294 (14)
C(31)—C(34)	1.500 (14)	C(32)—C(33)	1.464 (14)
C(33)—C(35)	1.512 (14)	Te(4)—N(4)	2.045 (8)
Te(4)—C(41)	2.054 (9)	N(4)—C(43)	1.285 (13)
C(41)—C(42)	1.347 (14)	C(41)—C(44)	1.480 (13)
C(42)—C(43)	1.446 (13)	C(43)—C(45)	1.523 (9)
Te(5)—N(5)	2.038 (1)	Te(5)—C(51)	2.073 (1)
N(5)—C(53)	1.297 (11)	C(51)—C(52)	1.365 (1)
C(51)—C(54)	1.469 (11)	C(52)—C(53)	1.465 (9)
C(53)—C(55)	1.471 (13)	Te(6)—N(6)	2.071 (7)
N(6)—C(61)	2.073 (9)	N(6)—C(63)	1.251 (11)
C(61)—C(62)	1.315 (14)	C(61)—C(64)	1.513 (14)
C(62)—C(63)	1.425 (14)	C(63)—C(65)	1.509 (15)
N—Te—C1	82.1 (3)	Te—N—C3	110.8 (6)
Te—C1—C2	109.2 (7)	Te—C1—C4	125.3 (7)
C2—C1—C4	125.5 (9)	C1—C2—C3	118.2 (9)
N—C3—C2	119.7 (8)	N—C3—C5	120.6 (9)
C2—C3—C5	119.7 (8)		

Table 3. Intermolecular distances (\AA) and angles ($^\circ$)

Te(1)⋯N(3 ^b)	2.774 (7)	Te(4)⋯N(5)	2.658 (8)
Te(2)⋯N(1)	2.640 (8)	Te(5)⋯N(6)	2.741 (7)
Te(3)⋯N(2)	2.700 (8)	Te(6)⋯N(4 ^a)	2.744 (8)
N(3 ^b)—Te(1)—N(1)	173.3 (3)	N(4)—Te(4)—N(5)	168.3 (3)
N(3 ^b)—Te(1)—C(11)	92.4 (3)	C(41)—Te(4)—N(5)	86.0 (3)
N(1)—Te(2)—N(2)	167.4 (3)	N(5)—Te(5)—N(6)	168.9 (3)
N(1)—Te(2)—C(21)	85.1 (3)	C(51)—Te(5)—N(6)	88.1 (3)
N(2)—Te(3)—N(3)	166.4 (3)	N(6)—Te(6)—N(4 ^a)	167.0 (3)
N(2)—Te(3)—C(31)	87.9 (3)	C(61)—Te(6)—N(4 ^a)	88.6 (3)
Te(1)—N(1)—Te(2)	117.0 (3)	Te(6 ^b)—N(4)—Te(4)	114.8 (3)
C(13)—N(1)—Te(2)	133.3 (6)	Te(6 ^b)—N(4)—C(43)	130.2 (6)
Te(4)—N(2)—Te(3)	111.2 (3)	Te(4)—N(5)—Te(5)	116.0 (3)
C(23)—N(2)—Te(3)	129.9 (7)	Te(4)—N(5)—C(53)	129.6 (6)
Te(3)—N(3)—Te(1)	116.2 (3)	Te(5)—N(6)—Te(6)	112.7 (3)
C(33)—N(3)—Te(1)	126.8 (6)	Te(5)—N(6)—C(63)	133.6 (6)

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

Because of the high melting point of 3,5-dimethyl-1,2-tellurazole (383–384 K) it has been possible to carry out the crystal structure analysis at room temperature and identify the intermolecular bonds which are responsible for this property.

Experimental. Colourless crystals were obtained by slow crystallization from acetone in the dark. To avoid the slow sublimation of the compound, a $0.58 \times 0.29 \times 0.22$ mm crystal was covered with fast-setting acrylic glue and used for intensity-data collection. Accurate unit-cell dimensions and crystal-orientation matrix were obtained from least-squares refinement of 35 strong reflections in the range $14 < 2\theta < 30^\circ$; $R3m/V$ Siemens four-circle diffractometer, ω -scan technique, graphite-monochromated Mo $K\alpha$ radiation, scan range $2\theta = 3\text{--}50^\circ$ ($0 \leq h \leq 16$, $0 \leq k \leq 13$, $-30 \leq l \leq 30$). No systematic loss of intensity of two standard reflections ($41\bar{8}$, $71\bar{3}$) was observed during data collection. 7891 reflections were measured (6978 unique, $R_{int} = 0.025$), and Lorentz and polarization corrections were applied to the intensity data. Effects of absorption were determined by

ψ -scan method (absorption correction maximum and minimum transmission factors 0.193 and 0.163 respectively), and extinction corrections ignored. The structure was solved by Patterson and Fourier techniques. 5298 reflections having $I > 3\sigma(I)$ were used to refine 370 parameters by a full-matrix least-squares procedure; $\sum w(|F_o| - |F_c|)^2$ minimized. The weighting scheme used in the last refinement cycles was $w = 1.0000/[\sigma^2(F_o) + q(F_o)^2]$, with $q = 0.008879$. Final $R = 0.045$, $wR = 0.057$, $S = 0.891$, maximum $\Delta/\sigma = 0.009$, maximum and minimum $\Delta\rho$ excursions in ΔF synthesis 0.94 and 0.41 e \AA^{-3} , respectively.

All non-H atoms were refined anisotropically, while H atoms, in calculated positions, were included with a common thermal parameter. All calculations were performed with *SHELXTL-Plus* (Sheldrick, 1989) and *PARST* (Nardelli, 1983). Scattering factors for non-H atoms from *International Tables for X-ray Crystallography* (1974, Vol. IV, p. 99), and for H atoms from Stewart, Davidson & Simpson (1965).

Discussion. Final atomic coordinates and equivalent isotropic temperature factors are listed in Table 1,* bond lengths and angles in Table 2, and intermolecular bond lengths and angles in Table 3.

The asymmetric unit is formed by as many as six molecules of 3,5-dimethyl-1,2-tellurazole, which are held together, three by three, by weak Te...N intermolecular bonds (see Fig. 1). Other weak Te...N bonds are found between neighbouring molecules along the *y* direction and rows along which the compound grows may be identified (see Fig. 2). These rows are held together only by weak van der Waals interactions. The intermolecular Te...N distances fall within the range 2.640–2.774 Å; they are longer than the intramolecular distances (2.020–2.071 Å) and are comparable with those found in similar compounds, where the Te and N atoms are

* Lists of calculated and observed structure factors, anisotropic thermal parameters, least-squares planes and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54977 (30 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: GE0290]

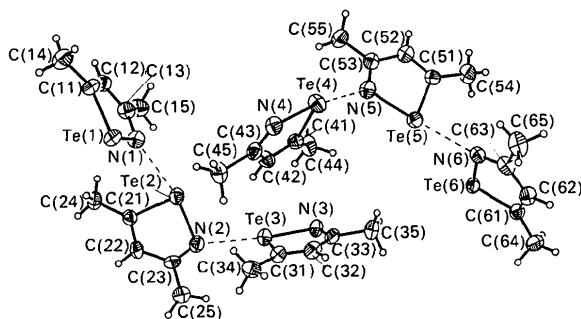


Fig. 1. ORTEP (Johnson, 1965) view of the complex C_5H_7NTe , showing the asymmetric unit with the numbering scheme.

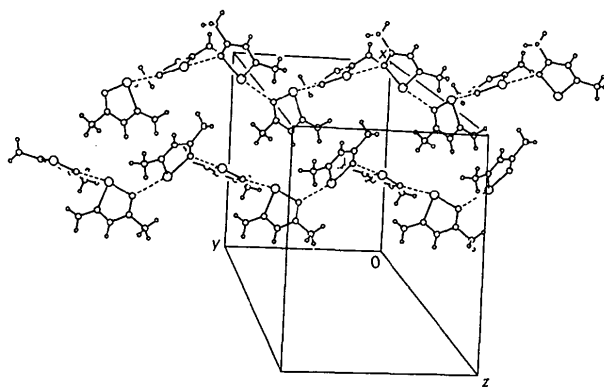


Fig. 2. View of the compound showing the intermolecular bonds along the *y* axis.

involved both in strong and in weak bonds, as well as with those found in other compounds with Te and N atoms not directly linked but only involved in weak bonds. Indeed in 1,2,5-telluradiazole the Te...N intermolecular distance is 2.764 (6) Å, with an intramolecular distance of 2.023 (6) Å (Bertini, Dapporto, Lucchesini, Segà & De Munno, 1984), and in bis[2-(2-pyridyl)phenyl] tritelluride the distance between non-bonded Te and N atoms is 2.55 (7) Å (Hamor, Al-Salim, West & McWinnie, 1986). All six Te atoms show a trigonal coordination (*t*-shaped coordination) with angles N—Te—N within 166.4–173.3° and N—Te—C within 85.1–92.4°. Several examples of trigonal coordination of Te are found in the literature (Hauge & Vikane, 1985; Detty, Murray, Smith & Zumbulyadis, 1983; Ahmed, McWinnie & Hamor, 1985).

The C—C and C—N bonds show some irregularities but it is not possible to identify a trend similar for all six molecules. Single C—C bonds are a little longer, whereas the double ones are a little shorter than those reported by Fanfani, Nunzi, Zanazzi, Zanzari & Pellinghelli (1972) for the analogous distances in the tellurophenic ring (1.412 and 1.357–1.384 Å, respectively).

The rings of the six independent molecules are all practically planar. The maximum deviation observed is 0.030 Å.

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