2100

 $[\text{Re}_4\text{Cl}_2(\text{PPh}_2)_4\text{PPh}(\text{CO})_8]$ AND $[\text{Re}_4\text{H}_2(\text{PPh}_2)_4\text{PPh}(\text{CO})_8].0.5\text{CH}_2\text{Cl}_2$

References

- FLÖRKE, U., WOYCECHOWSKI, M. & HAUPT, H.-J. (1988). Acta Cryst. C44, 2101–2104.
- HAUPT, H.-J., BALSAA, P. & FLÖRKE, U. (1987). Z. Anorg. Allg. Chem. 548, 151–160.
- HAUPT, H.-J., BALSAA, P. & FLÖRKE, U. (1988a). Angew. Chem. 100, 280–281.
- HAUPT, H.-J., BALSAA, P. & FLÖRKE, U. (1988b). Inorg. Chem. 27, 280–286.

HAUPT, H.-J., WOYCIECHOWSKI, M. & FLÖRKE, U. (1990). Z. Anorg. Allg. Chem. Submitted.

- NICHOLSON, R. S. & SHAIN, I. (1964). Anal. Chem. 36, 706-723.
- OWEN, S. M. (1988). Polyhedron, 7, 253-283.
- SHELDRICK, G. M. (1988). SHELXTL-Plus Structure Determination Software Programs. Nicolet Instrument Corporation, Madison, Wisconsin, USA.
- WOYCIECHOWSKI, M. (1989). Dissertation, Univ. of Paderborn, Federal Republic of Germany.

Acta Cryst. (1990). C46, 2100-2102

Structure of Tetrakis(diisopropyldithiocarbamato)tellurium(IV)

BY V. KUMAR AND G. ARAVAMUDAN*

Department of Chemistry, Indian Institute of Technology, Madras 600 036, India

M. SESHASAYEE

Department of Physics, Indian Institute of Technology, Madras 600 036, India

AND P. SELVAM AND K. YVON

Laboratoire de Cristallographie aux Rayons X, Université de Genève, CH-1211, Genève-4, Switzerland

(Received 19 December 1989; accepted 5 March 1990)

Abstract. [Te(C₇H₁₄NS₂)₄], $M_r = 832.86$, orthorhombic, $P2_12_12_1$, a = 13.946 (1), b = 14.833 (1), c = 20.213 (3) Å, V = 4181.54 Å³, Z = 4, $D_m = 1.35$, $D_x = 1.32$ Mg m⁻³, λ (Mo $K\alpha$) = 0.71069 Å, $\mu = 1.119$ mm⁻¹, F(000) = 1728, T = 293 K, final R(F) = 0.037, S = 0.83 for 2920 unique reflections. The central Te atom is eight coordinated so that the S atoms form a slightly distorted dodecahedral arrangement around the metal. The lone pair of electrons on Te^{1V} is found to be stereochemically inert. Interligand S···S short contacts of 3.097 (3) and 3.246 (2) Å are observed.

Introduction. Dithiocarbamates are known to undergo redox complexation reactions with metal ions such as Se^{IV} and Te^{IV}, forming divalent selenium and tellurium complexes and the corresponding thiuram disulfides as the oxidation products (Fabiani, Spagna, Vaciago & Zambonelli, 1971; Schnabel, Deuten & Klar, 1980; Kumar, Aravamudan & Seshasayee, 1990). The general reaction between tellurium(IV) and dithiocarbamates (L)can be given as: $Te^{IV} + 4L^- \rightleftharpoons Te^{IV}L_4 \rightleftharpoons Te^{II}L_2 +$ L-L, where L-L is the thiuram disulfide. In various solutions such as dichloromethane, carbon tetrachloride, benzene and acetonitrile, $Te^{IV}LA$ is found to undergo an autoredox reaction leading to the formation of the corresponding $Te^{II}L_2$ species and the thiuram disulfide, L-L.

The structure determination of the title compound was carried out to study whether any structural feature in the solid state can account for its facile autoredox reaction in solutions, to examine whether the lone pair of electrons on tellurium has any stereochemical significance, and also to study the effect of the bulkiness of the ligand on the stereochemistry. Other structures of TeL_4 complexes which have been reported are those of tetrakis(diethyldithiocarbamato)tellurium(IV) (Husebye & Svaeren, 1973): tetrakis(4-morpholinecarbodithioato)tellurium(IV) (Esperas & Husebye, 1975); tetrakis[N-(2hydroxyethyl)-N-methyldithiocarbamatoltellurium-(IV) (Husebye, 1979) and tetrakis[bis(2-hydroxyethyl)dithiocarbamato]tellurium(IV) (Rout, Seshasayee, Aravamudan & Radha, 1984).

Experimental. The title compound was prepared as a yellow solid on addition of 50 ml of 2M acetic acid to 30 ml of 2M sodium hydroxide containing 4 mmol of sodium tellurite and 100 mmol of the sodium salt of diisopropyldithiocarbamic acid. The precipitate was washed with water and air dried. 1 mmol

© 1990 International Union of Crystallography

^{*} To whom correspondence should be addressed.

Table 1. Positional parameters and U_{eq} for non-H Table 2. Bond lengths (Å) and angles (°) of non-H atoms with e.s.d.'s in parentheses

atoms with e.s.d.'s in parentheses

$ \begin{array}{cccccccccccccccccccccccccccccccccccc$			$U_{\rm eq} = 1/3 \sum_{i=1}^{3}$	U _{ii} .		Te—S(1) Te—S(2) T=_S(2)		C(11)—C(3) C(20)—C(3)	1·51 (2) 1·45 (2)
$ \begin{array}{c c c c c c c c c c c c c c c c c c c $			1-1			T_{α} $S(4)$	2.812 (2)	C(24) - C(5)	1.57(1)
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$		r	ν	7	$U(\dot{\Delta}^2)$	T_{α} S(5)	2.012 (2)	C(2+) - C(3)	1.35 (1)
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	π.	0.25240 (2)	y 0.11592.(2)	0.252(7.(2))		Te-S(6)	2.646 (2)	C(15) = N(3)	1.50 (1)
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	le	0.25349 (3)	0.11382 (2)	0.35267(2)	0.0307(2)	T_{α} S(7)	2,727 (2)	C(25) = N(3)	1.46 (1)
$ \begin{array}{c} S(2) & 0.4335 (1) & 0.0997 (1) & 0.2292 (1) & 0.0467 (10) & C(7) = S(2) & 1.725 (7) & C(9) = 1.44 (17) \\ S(4) & 0.3630 (1) & -0.0140 (1) & 0.4290 (1) & 0.0438 (10) & C(1) = S(2) & 1.743 (7) & C(1) = N(4) & 1.49 (17) \\ S(5) & 0.1525 (1) & 0.02579 (1) & 0.4000 (1) & 0.0449 (10) & C(1) = S(3) & 1.743 (7) & C(1) = C(1) \\ S(6) & 0.1683 (1) & 0.0393 (2) & 0.4553 (1) & 0.0481 (12) & C(1) = S(6) & 1.727 (7) & C(2) = C(12) & 1.51 (2) \\ S(7) & 0.2290 (1) & 0.2202 (1) & 0.2499 (1) & 0.0497 (10) & C(2) = S(6) & 1.727 (7) & C(2) = C(12) & 1.51 (2) \\ S(8) & 0.2072 (1) & -0.033 (1) & 0.2496 (1) & 0.0477 (10) & C(2) = S(6) & 1.727 (7) & C(2) = C(1) & 1.48 (2) \\ N(1) & 0.2523 (4) & 0.3673 (3) & 0.4780 (3) & 0.0426 (3) & C(1) = N(1) & 1.51 (1) & C(2) = C(1) & 1.48 (2) \\ N(2) & 0.2477 (4) & 0.2577 (3) & 0.2573 (3) & 0.0549 (38) & C(3) = N(1) & 1.51 (1) & C(2) = C(1) & 1.48 (2) \\ N(3) & 0.2678 (4) & -0.0506 (4) & 0.2217 (3) & 0.0583 (38) & C(3) = N(1) & 1.51 (1) & C(19) = C(1)8 & 1.59 (2) \\ N(4) & 0.0166 (4) & -0.0509 (4) & 0.4201 (4) & 0.0468 (38) & C(4) = N(2) & 1.347 (9) & C(2) = C(1)8 & 1.59 (2) \\ C(2) & 0.2571 (5) & -0.0270 (4) & 0.4714 (4) & 0.0488 (38) & C(1) = N-N(2) & 1.49 (1) & C(16) = C(25) & 1.55 (2) \\ C(3) & 0.3363 (6) & 0.3994 (6) & 0.5184 (5) & 0.0666 (58) & S(1) = Te = S(6) & 64.9 (1) & N(2) = C(4) = S(7) & 1.13 (1) \\ C(5) & 0.1603 (6) & 0.4718 (6) & 0.4997 (5) & 0.0665 (58) & S(1) = Te = S(6) & 64.9 (1) & N(2) = C(4) = S(7) & 1.12 1 (5) \\ C(6) & 0.418 (5) & 0.0214 (5) & 0.2256 (4) & 0.0990 (3) & S(1) = Te = S(6) & 64.9 (1) & N(2) = C(4) = S(7) & 1.12 1 (2) (5) \\ C(6) & 0.418 (6) & 0.0394 (6) & 0.5184 (5) & 0.0666 (56) & S(2) = TE = S(7) & 63.7 (1) & N(2) = C(4) = S(7) & 1.12 1 (2) (5) \\ C(6) & 0.418 (6) & 0.0320 (6) & 0.1307 (6) & 0.0907 (7) & S(3) = TE = S(6) & 64.9 (1) & N(2) = C(4) = S(7) & 1.12 1 (6) \\ C(6) & 0.418 (6) & 0.0320 (6) & 0.1307 (6) & 0.0907 (7) & S(3) = TE = S(6) & 64.9 (1) & N(2) = C(4) = S(7) & 1.12 1 (6) \\ C(6) & 0.149 (7) & 0.3408 (8) & 0.1907 (7) & 0.0665 (53) & S(1) = TE = S(6) & 64.$	S(1)	0.0733(1)	0.1139 (10)	0.3034(1)	0.0445 (10)	T_{α} S(8)	2.735 (2)	C(23) = N(3)	1.332 (0)
	S(2)	0.4335 (1)	0.0997 (1)	0.2920 (1)	0·0467 (10)	C(7) = S(1)	1.722 (7)	C(9) = N(4)	1.48 (1)
$ \begin{array}{c} S(4) & 0.3630 (1) & -0.0140 (1) & 0.4203 (1) & 0.0480 (10) & C(1) - S(3) & 1.743 (7) & C(10) - C(3) & 1.56 (2) \\ S(5) & 0.1525 (1) & 0.02579 (1) & 0.4000 (1) & 0.0449 (10) & C(2) - S(4) & 1.699 (7) & C(10) - C(10) & 1.56 (2) \\ S(7) & 0.2890 (1) & 0.2302 (1) & 0.2490 (1) & 0.0497 (10) & C(2) - S(6) & 1.721 (7) & C(2) - C(12) & 1.47 (2) \\ S(7) & 0.2890 (1) & -0.0339 (1) & 0.2805 (1) & 0.0571 (12) & C(4) - S(7) & 1.720 (7) & C(2) - C(13) & 1.47 (2) \\ S(8) & 0.277 (1) & -0.0339 (1) & 0.2805 (1) & 0.0570 (12) & C(4) - S(7) & 1.720 (7) & C(2) - C(13) & 1.53 (2) \\ N(1) & 0.2523 (4) & 0.3673 (3) & 0.4780 (3) & 0.0426 (30) & C(1) - N(1) & 1.322 (8) & C(2) - C(17) & 1.49 (2) \\ N(3) & 0.2678 (4) & -0.0666 (4) & 0.2219 (3) & 0.0583 (38) & C(5) - N(1) & 1.51 (1) & C(2) - C(17) & 1.49 (2) \\ N(3) & 0.2678 (4) & -0.0666 (4) & 0.2219 (3) & 0.0583 (38) & C(5) - N(1) & 1.51 (1) & C(2) - C(17) & 1.49 (2) \\ N(4) & 0.0166 (4) & -0.0409 (4) & 0.0407 (40) & C(12 - N(2) & 1.347 (9) & C(2) - C(18) & 1.51 (2) \\ C(1) & 0.2521 (5) & -0.2399 (4) & 0.4409 (3) & 0.4007 (40) & C(12 - N(2) & 1.347 (9) & C(2) - C(25) & 1.55 (2) \\ C(2) & 0.2673 (5) & -0.0270 (4) & 0.4714 (4) & 0.0418 (38) & C(18 - N(2) & 1.307 (9) & C(2) - C(25) & 1.51 (2) \\ C(3) & 0.3366 (6) & 0.3994 (6) & 0.5184 (5) & 0.0666 (5) & S(2) - TS(6) & 649 (1) & S(7) - C(4) - S(2) & 1167 (4) \\ C(4) & 0.4081 (5) & 0.2014 (5) & 0.2561 (4) & 0.0399 (38) & S(2) - T - S(6) & 649 (1) & N(2) - C(4) - S(2) & 1157 (2) \\ C(5) & 0.1603 (6) & 0.4178 (6) & 0.4897 (5) & 0.0665 (5) & S(2) - T S(6) & 649 (1) & N(2) - C(4) - S(2) & 1157 (2) \\ C(5) & 0.1603 (6) & 0.0177 (6) & 0.0997 (8) & 0.0997 (7) & C(3) C(3) & 649 (1) & N(2) - C(4) - S(2) & 113 (6) \\ C(6) & 0.4130 (7) & 0.3248 (8) & 0.4544 (7) & 0.0104 (59) & C(1) S(6) & 649 (1) & N(2) - C(3) - N(1) & 1114 (8) \\ C(7) & -0.0857 (9) & 0.0238 (6) & 0.0107 (8) & C(1) 116 & 877 (2) & C(2) - N(3) - C(2) & -N(3) - C(2) & -N(3) - C(2) & -N(3) - C(3) & -N(1) & -1134 (6) \\ C(10) & -0.486 (10) & -0.2318 (6) & 0.0386 (6) & 0.1077 (8)$	S(3)	0.3552 (1)	0.2292 (1)	0.4299 (1)	0.0534 (10)	C(4) = S(2)	1.711(7)	C(17) - N(4)	1.40(1)
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	S(4)	0.3630(1)	-0.0140(1)	0.4203(1)	0.0480 (10)	C(1) = S(3)	1.743 (7)	$C(10) \rightarrow C(9)$	1.56 (2)
$ \begin{array}{c} \hat{s}(6) \\ 0 & -(63) (1) \\ 0 & -2302 (1) \\ 0 & -2373 (3) \\ 0 & -2477 (4) \\ 0 & -2573 (4) \\ 0 & -2573 (4) \\ 0 & -2573 (4) \\ 0 & -2573 (4) \\ 0 & -2573 (4) \\ 0 & -2573 (4) \\ 0 & -2573 (4) \\ 0 & -2573 (4) \\ 0 & -2573 (4) \\ 0 & -2573 (4) \\ 0 & -2573 (4) \\ 0 & -2573 (4) \\ 0 & -2573 (4) \\ 0 & -2573 (4) \\ 0 & -2573 (4) \\ 0 & -2573 (4) \\ 0 & -2573 (4) \\ 0 & -2521 (5) \\ 0 & -2399 (4) \\ 0 & -4409 (3) \\ 0 & -0449 (3) \\ 0 & -0449 (3) \\ 0 & -0440 (4) \\ 0 & -16(3) \\ 0 & -$	S(S)	0.1525 (1)	0·2579 (1)	0·4000 (1)	0.0449 (10)	C(2) - S(4)	1.699 (7)	C(13) - C(19)	1.56 (2)
$ \begin{array}{c} 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 $	S(6)	0.1683(1)	0.0393 (2)	0.4553 (1)	0.0581 (12)	$C(1) \rightarrow S(5)$	1.703 (7)	C(6) - C(12)	1.51 (2)
$ \begin{array}{c} 363 \\ 363 \\ 364 \\ 367 \\ 368 \\ 367 \\ 368 \\ 367 \\ 368 \\ 367 \\ 368 \\ 367 \\ 368 \\ 367 \\ 368 \\ 367 \\ 368 \\ 367 \\ 368 $	S(7)	0.2890 (1)	0.2302(1)	0.2499(1)	0.0497(10)	C(2) - S(6)	1.727 (7)	C(23) - C(12)	1.47(2)
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	S(7)	0.2070 (1)	-0.0220(1)	0.2905(1)	0.0570(10)	C(4) - S(7)	1.720 (7)	C(14) - C(15)	1.50 (2)
$ \begin{array}{c} N(1) & 0.232 \ (4) & 0.337 \ (5) & 0.4760 \ (5) & 0.4426 \ (30) & C(1)-N(1) & 1.322 \ (6) & C(21)-C(1) & 1.48 \ (2) \\ N(3) & 0.2678 \ (4) & -0.2579 \ (4) & 0.2577 \ (3) & 0.0583 \ (38) & C(5)-N(1) & 1.51 \ (1) & C(19)-C(18) & 1.59 \ (2) \\ N(4) & 0.0166 \ (4) & -0.0509 \ (4) & 0.2671 \ (4) & 0.0566 \ (38) & C(5)-N(1) & 1.51 \ (1) & C(19)-C(18) & 1.59 \ (2) \\ C(1) & 0.2521 \ (5) & 0.2939 \ (4) & 0.4409 \ (3) & 0.0407 \ (40) & C(12)-N(2) & 1.347 \ (9) & C(22)-C(18) & 1.51 \ (2) \\ C(2) & 0.2673 \ (5) & -0.0270 \ (4) & 0.4714 \ (4) & 0.0418 \ (38) & C(18)-N(2) & 1.50 \ (1) & C(16)-C(25) & 1.55 \ (2) \\ C(3) & 0.3363 \ (6) & 0.3994 \ (6) & 0.5184 \ (5) & 0.0660 \ (56) \\ C(4) & 0.4081 \ (5) & 0.2014 \ (5) & 0.2561 \ (4) & 0.0399 \ (38) & S(1)-Te-S(8) & 649 \ (1) & N(2)-C(4)-S(2) & 1167 \ (4) \\ C(5) & 0.1603 \ (6) & 0.4178 \ (6) & 0.4997 \ (5) & 0.0666 \ (54) & S(3)-Te-S(5) & 654 \ (1) & N(2)-C(4)-S(2) & 1167 \ (4) \\ C(6) & 0.4130 \ (7) & 0.3640 \ (8) & 0.1507 \ (6) & 0.0992 \ (22) \ S(4)-Te-S(5) & 654 \ (1) & C(2)-C(4)-S(2) & 1170 \ (2) \\ C(6) & 0.1159 \ (6) & 0.3928 \ (8) & 0.5544 \ (6) & 0.0997 \ (2) \ C(2)-Te-S(5) & 654 \ (1) & C(2)-C(4)-S(2) & 1170 \ (2) \\ C(8) & 0.1159 \ (8) & 0.3328 \ (8) & 0.5544 \ (6) & 0.0997 \ (2) \ C(2)-Te-S(5) & 654 \ (1) & C(2)-C(4)-TE \ (2) \ (2) \ C(1)-N(3)-C(2) & 214 \ (7) \\ C(10) & -0.0433 \ (8) & 0.4744 \ (7) & 0.1078 \ (9) \ (C)(2)-S(4)-Te-S(5) \ (2) \ (2)-N(3)-C(2) & 214 \ (7) \\ C(11) & -0.143 \ (8) & 0.4744 \ (7) & 0.1014 \ (8) \ C(1)-C(5)-Te-S(5) \ (2) \ (2)-C(3)-N(4)-C(7) \ (235 \ (6) \ (1) \ (-3)39 \ (2) \ (2)-N(4)-C(7) \ (235 \ $	3(0) N(1)	0.2072(1)	-0.0339(1)	0.2003(1) 0.4780(2)	0.0370(12)	C(7)S(8)	1.720 (7)	C(28)-C(15)	1.58 (2)
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	N(1)	0.2323 (4)	0.3073 (3)	0.4780 (3)	0.0420 (30)	C(1)—N(1)	1.322 (8)	C(21)-C(17)	1.48 (2)
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	N(2)	0.4///(4)	0.25/9(4)	0.2357(3)	0.0549 (38)	C(3)—N(1)	1.51 (1)	C(26)—C(17)	1.49 (2)
$ \begin{array}{llllllllllllllllllllllllllllllllllll$	N(3)	0.2678 (4)	<i>−</i> 0·0866 (4)	0.5219 (3)	0.0283 (38)	C(5)—N(1)	1.51 (1)	C(19)-C(18)	1.59 (2)
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	N(4)	0.0166 (4)	<i>−</i> 0·0509 (4)	0.2671 (4)	0.0566 (38)	C(4)—N(2)	1.347 (9)	C(22)—C(18)	1.51 (2)
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	C(1)	0.2521 (5)	0.2939 (4)	0.4409 (3)	0.0407 (40)	C(12)—N(2)	1.49 (1)	C(16)—C(25)	1.55 (2)
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	C(2)	0.2673 (5)	-0.0270(4)	0.4714 (4)	0.0418 (38)	C(18)—N(2)	1.20 (1)	C(27)—C(25)	1.51 (2)
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	Cisi	0.3363 (6)	0.3994 (6)	0.5184 (5)	0.0660 (56)				
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	Ciá	0.4081 (5)	0.2014 (5)	0.2561(4)	0.0399(38)	S(1)—Te— $S(8)$	64-9 (1)	S(7)C(4)S(2)	116.7 (4)
$ \begin{array}{c} C(5) & 0 & 1032 & (6) & 0 & 1017 & (6) & 0 & 1007 & (6) & 0 & 0002 & (72) & S(3)-1(S(5)) & 654 & (1) & N(2)-C(4)-S(7) & [21-3 (5) \\ (7) & 0 & 0003 & (5) & 0 & 0029 & (4) & 0 & 2813 & (3) & 0 & 0 & 0017 & (36) & C(7)-S(1)-Te & 88.7 & (2) & C(24)-C(5)-N(1) & 110e & (7) \\ C(8) & 0 & 1159 & (8) & 0 & 3928 & (8) & 0 & 5544 & (6) & 0 & 0947 & (79) & C(4)-S(2)-Te & 85.7 & (2) & C(24)-C(5)-N(3) & 110e & (7) \\ C(9) & -0 & 0817 & (6) & -0 & 0137 & (7) & 0 & 2723 & (5) & 0 & 0740 & (59) & C(1)-S(3)-Te & 89.0 & (2) & C(15)-N(3)-C(2) & 1209 & (7) \\ C(10) & -0 & 1403 & (8) & -0 & 0635 & (10) & 0 & 3269 & (6) & 0 & 1078 & (90) & C(2)-S(4)-Te & 86.9 & (2) & C(25)-N(3)-C(12) & 1209 & (7) \\ C(11) & 0 & 4139 & (7) & 0 & 4368 & (8) & 0 & 4744 & (7) & 0 & 1014 & (83) & C(2)-S(6)-Te & 91.9 & (2) & C(25)-N(3)-C(15) & 114.9 & (8) \\ C(12) & 0 & 4626 & (7) & 0 & 3539 & (5) & 0 & 2164 & (5) & 0 & 0623 & (51) & C(4)-S(7)-Te & 88.0 & (2) & C(17)-N(4)-C(7) & 125.5 & (6) \\ C(14) & 0 & -1586 & (11) & -0 & 0151 & (11) & 0 & 6054 & (5) & 0 & 134 & (12) & C(3)-N(1)-C(1) & 124.8 & (6) & S(8)-C(7)-S(1) & 115.9 & (4) \\ C(15) & 0 & 1860 & (7) & -0 & 0997 & (9) & 0 & 5829 & (6) & 0 & 0978 & (80) & C(5)-N(1)-C(1) & 124.8 & (6) & S(8)-C(7)-S(1) & 115.9 & (4) \\ C(16) & 0 & 3286 & (10) & -0 & 2410 & (9) & 0 & 5329 & (3) & 0 & 134 & (12) & C(3)-N(1)-C(1) & 119.8 & (6) & N(4)-C(7)-S(1) & 125.4 & (5) \\ C(16) & 0 & -3286 & (10) & -0 & 2410 & (9) & 0 & 5329 & (3) & 0 & 106 & (3) & N(1)-C(1) & 119.8 & (6) & N(4)-C(7)-S(1) & 125.9 & (22) & 0.532 & (7) & 0 & 2380 & (6) & 0 & 0377 & (60) & S(5)-C(1)-S(3) & 116.0 & (4) & C(10)-C(9)-N(4) & 112.0 & (8) & C(2)-N(2) & 113.0 & (7) & C(2)-N(2) & 113.0 & (7) & C(2)-N(2) & 113.0 & (7) & C(2)-C(2) & (13) & C(1)-C(2)-N(2) & 113.0 & (7) & C(2)-C(2)-N(3) & 114.9 & (6) & N(4)-C(7)-S(1) & 122.4 & (5) & C(13)-C(9)-N(4) & 112.2 & (8) & C(2)-C(13)-N(1) & 116.8 & (8) & C(2)-C(1)-N(2) & 113.0 & (7) & C(2)-C(2)-N(3) & 113.0 & (7) & $	C	0.1603 (6)	0.4178(6)	0.4897(5)	0.0665 (54)	S(2) - Te - S(7)	63.7 (1)	N(2) - C(4) - S(2)	121.9 (5)
$ \begin{array}{c} C(7) & 0.913 & (7) & 0.944 & (6) & 0.120 & (1) & 0.902 & (12) & (3) & (1-6)(5) & (4-1)(5) & (10) & (10) & (11)(11)(10) & (11$	C(6)	0.4130(7)	0.3640 (8)	0.1507 (6)	0.0002(34)	S(3) - 1e - S(5)	65.4 (1)	N(2) - C(4) - S(7)	121.3 (5)
$ \begin{array}{c} C(1) & 0.0905 (5) & 0.0029 (4) & 0.2815 (5) & 0.0401 (5) & C(1) = C(1) = C & 88^{-1} (2) & C(24) = C(5) = C(8) & 1170 (9) \\ C(8) & 0.1159 (8) & 0.3928 (8) & 0.5544 (6) & 0.0947 (79) & C(1) = S(3) = Te & 85^{-1} (2) & C(24) = C(5) = C(8) & 1170 (9) \\ C(9) & -0.0817 (6) & -0.0137 (7) & 0.2723 (5) & 0.0740 (59) & C(1) = S(3) = Te & 89^{-0} (2) & C(25) = N(3) = C(2) & 124^{-1} (7) \\ C(10) & -0.1403 (8) & -0.0655 (10) & 0.3269 (6) & 0.1078 (90) & C(2) = S(4) = Te & 86^{-9} (2) & C(25) = N(3) = C(2) & 114^{-9} (8) \\ C(11) & 0.4139 (7) & 0.4368 (8) & 0.4744 (7) & 0.1014 (83) & C(1) = S(5) = Te & 89^{-5} (2) & C(25) = N(3) = C(1) & 1149 (8) \\ C(12) & 0.4626 (7) & 0.3539 (5) & 0.2164 (5) & 0.0623 (51) & C(4) = S(7) = Te & 88^{-7} (2) & C(17) = N(4) = C(7) & 118^{+} (7) \\ C(13) & -0.1327 (8) & -0.0085 (9) & 0.2038 (6) & 0.1027 (84) & C(7) = S(8) = Te & 87^{-7} (2) & C(17) = N(4) = C(7) & 118^{+} (7) \\ C(14) & 0.1586 (11) & -0.0151 (11) & 0.0554 (5) & 0.134 (12) & C(3) = N(1) = C(1) & 1198 (6) & N(4) = C(7) = S(1) & 1159 (4) \\ C(16) & 0.3286 (10) & -0.2410 (9) & 0.5529 (8) & 0.134 (12) & C(3) = N(1) = C(1) & 1198 (6) & N(4) = C(7) = S(8) & 1226 (5) \\ C(17) & 0.0238 (6) & -0.1475 (6) & 0.2484 (6) & 0.0737 (60) & S(5) = C(1) = S(3) & 1160 (4) & C(10) = C(9) = N(4) & 1122 (8) \\ C(18) & 0.5805 (6) & 0.2278 (7) & 0.2380 (6) & 0.0988 (73) & N(1) = C(1) = S(3) & 1160 (4) & C(10) = C(9) = N(4) & 1122 (8) \\ C(19) & 0.6328 (9) & 0.2367 (8) & 0.5690 (5) & 0.0997 (81) & N(3) = C(2) = S(4) & 1226 (5) & C(13) = C(9) = N(4) & 1122 (8) \\ C(21) & 0.0721 (8) & -0.1581 (8) & 0.1838 (6) & 0.0950 (77) & N(3) = C(2) = S(4) & 1226 (5) & C(23) = C(12) = N(3) & 1104 (10) \\ C(24) & 0.1751 (9) & 0.5210 (6) & 0.4758 (6) & 0.1027 (81) & C(13) = N(2) = C(4) & 119^{-6} (7) & C(28) = C(15) = N(3) & 1104 (10) \\ C(25) & 0.3534 (7) & -0.1390 (8) & 0.5372 (5) & 0.0890 (73) & C(12) = N(2) & 115^{-7} (9) & C(23) = C(15) = N(3) & 1104 (10) \\ C(24) & 0.1751 (9) & 0.5210 (6) & 0.4758 (6) & 0.1027 (81) & C(13) = N(2) = (12^{-7} (13) = N(4) & 1139 (9) \\ C($	C(0)	0 0002 (5)	0.0000 (4)	0 1007 (0)	0.0401(72)	S(4) - 1e - S(6)	64.5 (1)	C(8) - C(5) - N(1)	111.6 (8)
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	$C(\eta)$	0.0903(3)	0.0029 (4)	0.2813(3)	0.0401(30)	C(7) = S(1) = 1e	88.7 (2)	C(24) - C(5) - N(1)	110.2 (7)
$ \begin{array}{c} C(9) & -0.0817\ (6) & -0.0137\ (7) & 0.2723\ (5) & 0.0740\ (59) & C(1) = 363 - 1e & 3970\ (2) & C(13) = N(3) = C(2) & 1241\ (7) \\ C(10) & -0.1403\ (8) & -0.0635\ (10) & 0.3269\ (6) & 0.1078\ (90) & C(2) = S(4) = Te & 869\ (2) & C(25) = N(3) = C(15) & 1149\ (8) \\ C(1) & 0.4139\ (7) & 0.4368\ (8) & 0.4744\ (7) & 0.1014\ (83) & C(2) = S(6) = Te & 91.9\ (2) & C(9) = N(4) = C(7) & 1184\ (7) \\ C(12) & 0.4626\ (7) & 0.3539\ (5) & 0.2164\ (5) & 0.0623\ (51) & C(2) = S(6) = Te & 91.9\ (2) & C(9) = N(4) = C(7) & 1184\ (7) \\ C(13) & -0.1327\ (8) & -0.0085\ (9) & 0.2038\ (6) & 0.1027\ (84) & C(7) = Fe & 87.7\ (2) & C(17) = N(4) = C(7) & 1164\ (6) \\ C(14) & 0.1586\ (11) & -0.0151\ (11) & 0.6054\ (5) & 0.134\ (12) & C(3) = N(1) = C(1) & 1248\ (6) & S(8) = C(7) = S(1) & 1159\ (4) \\ C(16) & 0.3286\ (10) & -0.2410\ (9) & 0.5329\ (8) & 0.134\ (12) & C(5) = N(1) = C(3) & 1149\ (6) & N(4) = C(7) = S(8) & 1226\ (5) \\ C(17) & 0.0238\ (6) & -0.2484\ (6) & 0.0737\ (60) & S(5) = C(1) = S(3) & 1160\ (4) & C(10) = C(9) = N(4) & 1110\ (8) \\ C(18) & 0.5805\ (6) & 0.2278\ (7) & 0.2380\ (6) & 0.0888\ (73) & N(1) = C(1) = S(3) & 124\ (6) & C(13) = C(9) = N(4) & 1110\ (8) \\ C(20) & 0.3680\ (9) & 0.3367\ (8) & 0.5690\ (5) & 0.0997\ (81) & N(3) = C(2) = S(4) & 1166\ (4) & C(6) = C(12) = N(2) & 1130\ (7) \\ C(22) & 0.3680\ (9) & 0.3367\ (8) & 0.5690\ (5) & 0.0997\ (81) & N(3) = C(2) = S(4) & 1166\ (4) & C(6) = C(12) = N(2) & 1130\ (7) \\ C(22) & 0.6303\ (7) & 0.2375\ (9) & 0.1723\ (6) & 0.1062\ (87)\ C(12) = N(2) = S(6) & 1213\ (5) & C(23) = C(12) = N(3) & 1134\ (10) \\ C(24) & 0.1751\ (9) & 0.5210\ (6) & 0.4758\ (6) & 0.1027\ (81)\ C(18) = N(2) = C(4) & 1194\ (6) & C(21) = C(16) = N(3) & 1134\ (10) \\ C(24) & 0.1751\ (9) & 0.5210\ (6) & 0.372\ (6) & 0.127\ (81)\ C(18) = N(2) = C(14) & 1194\ (6) & C(21) = C(15) = N(3) & 1134\ (10) \\ C(25) & 0.3534\ (7) & -0.1380\ (8) & 0.377\ (8) & 0.1218\ (98)\ C(20) = C(3) = N(1) & 1148\ (8)\ C(21) = C(18) = N(2) = C(14) & 1144\ (8)\ C(22) = C(16) = N(3) & 1134\ (10) \\ C(25) & 0.303\ (7) & 0.2375\ (9) & 0.1$	C(8)	0.1159 (8)	0.3928 (8)	0.5544 (6)	0.0947 (79)	C(4) = S(2) = 1e	85.7 (2)	C(24) - C(3) - C(8)	117.0 (9)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(9)	-0.0817 (6)	-0.0137(7)	0.2723 (5)	0.0740 (59)	C(1) = S(3) = 1e $C(2) = S(4) = T_{2}$	89.0 (2)	C(15) = N(3) = C(2) C(25) = N(3) = C(2)	124.1 (7)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(10)	-0·1403 (8)	-0·0635 (10)	0.3269 (6)	0·1078 (90)	C(2) = 3(4) = 10 $C(1) = S(5) = T_0$	80.5 (2)	C(25) = N(2) = C(15)	120.9 (7)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(11)	0.4139 (7)	0.4368 (8)	0.4744 (7)	0.1014 (83)	C(1) = S(5) = 1c C(2) = S(6) = Tc	91.9(2)	C(2) - N(3) - C(1)	118.4 (7)
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	C(12)	0.4626 (7)	0.3539 (5)	0.2164 (5)	0.0623 (51)	C(4) = S(7) = Te	88.0 (2)	C(17) - N(4) - C(7)	125.5 (6)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(13)	-0.1327 (8)	-0.0085 (9)	0.2038 (6)	0.1027 (84)	$C(7) \rightarrow S(8) \rightarrow Te$	87.7 (2)	C(17) - N(4) - C(9)	116-1 (6)
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	C(14)	0.1586 (11)	-0.0151 (11)	0.6054 (5)	0.134 (12)	C(3) - N(1) - C(1)	124.8 (6)	S(8) - C(7) - S(1)	115.9 (4)
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	CUS	0·1860 (7)	-0.0997 (9)	0.5689 (6)	0.0978 (80)	C(5) - N(1) - C(1)	119.8 (6)	N(4) - C(7) - S(1)	121.4 (5)
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	CUG	0.3286(10)	-0.2410(9)	0.5329(8)	0.134(12)	C(5) - N(1) - C(3)	114.9 (6)	N(4)-C(7)-S(8)	122.6 (5)
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	C(17)	0.0238 (6)	-0.1475(6)	0.2484 (6)	0.0737(12)	S(5) - C(1) - S(3)	116.0 (4)	C(10) - C(9) - N(4)	111.0 (8)
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	C(17)	0.5205 (6)	0.2279(7)	0.2290 (6)	0.0999 (72)	N(1) - C(1) - S(3)	121.6 (5)	C(13)-C(9)-N(4)	112.2 (8)
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	C(10)	0.6228 (0)	0.2276(7)	0.2380(0)	0.0000(73)	N(1)—C(1)—S(5)	122-4 (5)	C(13)-C(9)-C(10)	114-4 (8)
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	C(19)	0.0328 (9)	0.2800 (13)	0.2932 (7)	0.199(10)	S(6)—C(2)—S(4)	116.6 (4)	C(6)—C(12)—N(2)	113.0 (7)
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	C(20)	0.3680(9)	0.3367(8)	0.2690 (2)	0.0997 (81)	N(3)—C(2)—S(4)	122.0 (5)	C(23)—C(12)—N(2)	112.9 (8)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(21)	0.0721 (8)	-0.1281(8)	0.1838(6)	0.0950 (77)	N(3)—C(2)—S(6)	121-3 (5)	C(23) - C(12) - C(6)	114-2 (9)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(22)	0.6303 (7)	0.2375 (9)	0.1723 (6)	0·1062 (87)	C(12) - N(2) - C(4)	124.9 (6)	C(14) - C(15) - N(3)	113.4 (10)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(23)	0.4207 (9)	0.4085 (6)	0.2697 (7)	0.1007 (81)	C(18) - N(2) - C(4)	119-6 (7)	C(28) - C(15) - N(3)	110.1 (10)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(24)	0.1751 (9)	0.5210 (6)	0.4758 (6)	0.1027 (81)	C(18) - N(2) - C(12)	115-2 (7)	C(28) - C(15) - C(14)	113.0 (10)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(25)	0.3534 (7)	-0.1390 (8)	0.5372 (5)	0.0890 (73)	$C(11) \rightarrow C(3) \rightarrow N(1)$	110-8 (8)	C(21) - C(17) - N(4)	111-2 (8)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(26)	0.0635 (10)	- 0.2063 (8)	0.3017 (8)	0.1218 (98)	C(20) = C(3) = N(1)	114.0 (8)	C(20) - C(17) - N(4)	113.9 (9)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(27)	0.4013(9)	-0.1113(12)	0.6011(7)	0.136 (11)	$C(20) \rightarrow C(3) \rightarrow C(11)$	115.7 (9)	(126) - ((17) - ((21))	113.9 (9)
$\begin{array}{cccccc} C(25) & 0.000 (0) & 0.1451 (10) & 0.0521 (2) & 0.142 (12) & 0.142 (12) & 0.001 (2) & 0.014 (13) & 0.001 (2) & 0.014 (13) & $	C(28)	0.0986 (8)	-0.1451(10)	0.5321(9)	0.142(12)	C(22) = C(18) = IN(2) C(22) = C(18) = C(10)	112.7 (9)	C(10) = C(12) = C(10)	107.1 (0)
	C(20)	0 0 0 0 0 (0)	0 1451 (10)	0.0021 (7)	5 172 (12)	C(16) - C(15) - N(3)	109.0 (9)	C(17)-C(10)(2)	107-1 (9)
						C(27) - C(25) - N(3)	113.5 (10)		

(0.831 g) of TeL₄ was dissolved in 10 ml of dichloromethane and 5 ml of acetonitrile in the presence of 1 mmol (0.325 g) of the thiuram disulfide and allowed to evaporate slowly at room temperature giving orange red crystals. Any adhering disulfide was removed by washing with methanol. A suitable single crystal of dimensions $0.72 \times 0.37 \times 0.25$ mm chosen. D_m by flotation in acetone-carbon tetrachloride mixture, $P2_12_12_1$ from systematic absences; Enraf-Nonius CAD-4 diffractometer; cell parameters by least squares from setting angles of 40 reflections with $22 \le 2\theta \le 28^\circ$; 3359 reflections with 4 $\leq 2\theta \leq 46^{\circ}$ and $0 \leq h \leq 15$, $0 \leq k \leq 16$, $0 \leq l \leq 22$ $(R_{\rm int} = 0.03)$ were collected using $\omega - 2\theta$ scans; three standard reflections monitored every hour showed no significant change; correction for Lorentz and polarization effects; no absorption correction made; 2920 reflections (N) with $I > 3\sigma(I)$ used for structure

determination, 455 parameters (P), N/P = 6.5; structure solved by SHELX76 (Sheldrick, 1976); Te position from Patterson map; successive difference Fourier maps based on Te position gave the locations of all non-H atoms; of the 56 H atoms, 21 appeared in the final difference Fourier map and were refined isotropically; positions of the remaining 35 H atoms geometrically fixed; non-H atoms refined anisotropically and H atoms with isotropic temperature factors; atomic scattering factors for non-H atoms from Cromer & Mann (1968); maximum and minimum electron density in the final difference Fourier map 0.25 and $-0.67 \text{ e} \text{ Å}^{-3}$, $(\Delta/\sigma)_{\text{max}} = 0.03$, $w = 1/[\sigma^2(F_o) +$ R(F) = 0.037, wR = 0.045, $0.0039 |F_o|^2$, S = 0.83.

Discussion. Table 1 contains the atomic coordinates and the thermal parameters of all non-H atoms. Bond distances and angles are given in Table 2.* Fig. 1 shows the *PLUTO* (Motherwell & Clegg, 1978) plot of the molecule. Fig. 2 shows the packing of the molecules in the unit cell.

In the title compound the four dithiocarbamate ligands are asymmetrically bound to the central Te

* Lists of calculated and observed structure factors, anisotropic thermal parameters of non-H atoms and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53112 (18 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

O C24



Fig. 1. PLUTO plot of the molecule.



Fig. 2. Packing of the molecules in the unit cell.

atom. The TeS₈ core is made up of two planar TeS₄ trapezoids, namely Te, S(1), S(8), S(3), S(5) and Te, S(2), S(4), S(6), S(7) interlocking at the Te position at an angle of $87 \cdot 6^{\circ}$ to give a slightly distorted dodecahedral arrangement. The TeS₈ core is similar to that found in the other reported TeL₄ complexes. The ligands are nearly planar and the average C—S and C—N bond lengths of $1 \cdot 72$ (2) and $1 \cdot 44$ (4) Å respectively are normal.

According to Esperas & Husebye (1975), the S...S interligand distance is of importance in the stability of a TeL₄ complex with respect to its transformation to TeL_2 and L-L. Thus, the greater this S...S distance, the more stable the complex should be. Of all the Te L_4 complexes, we find that the present compound, namely the isopropyl derivative, undergoes the most facile changeover to TeL_2 and L-L. In the present compound in one of the TeS₄ trapezoids, namely, Te, S(1), S(8), S(3), S(5), an interligand S.S. short contact is observed with the S(1)...S(5) distance being 3.097(3) Å {cf. 3.17 Å in [Te(Et₂NCS₂)₄]; 3.39 Å in [Te(OC₄H₈NCS₂)₄], 3.25 Å in Te-[(OHC₂H₄)₂NCS₂]₄ and 3.16 Å in Te[HOCH₂- $CH_2(CH_3)NCS_2]_4$. This short separation is consistent with the ease of the autoredox reaction. The proximity of the two S atoms for an intramolecular electron transfer may be the predominant reason for this behaviour, but other factors such as the reducing ability of the dithiocarbamate ligand have also to be taken into consideration.

One of the authors, V. Kumar, thanks the Council of Scientific and Industrial Research, New Delhi, India, for the award of a fellowship. The facilities offered at the Department of Physics, Indian Institute of Science, Bangalore, for the *PLUTO* plots, is gratefully acknowledged.

References

- CROMER, D. T. & MANN, J. B. (1968). Acta Cryst. A24, 321–324.
 ESPERAS, S. & HUSEBYE, S. (1975). Acta Chem. Scand. Ser. A, 29, 185–194.
- FABIANI, C., SPAGNA, R., VACIAGO, A. & ZAMBONELLI, L. (1971). Acta Cryst. B27, 1499–1504.
- HUSEBYE, S. (1979). Acta Chem. Scand. Ser. A, 33, 485-490.
- HUSEBYE, S. & SVAEREN, S. E. (1973). Acta Chem. Scand. 27, 763–778.
- KUMAR, V., ARAVAMUDAN, G. & SESHASAYEE, M. (1990). Acta Cryst. C46, 674-676.
- MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.
- ROUT, G. C., SESHASAYEE, M., ARAVAMUDAN, G. & RADHA, K. (1984). Acta Cryst. C40, 1142–1145.
- SCHNABEL, W., DEUTEN, K. V. & KLAR, G. (1980). Phosphorus Sulfur, 9, 93–98.
- SHELDRICK, G. M. (1976). SHELX76. Program for crystal structure determination. Univ. of Cambridge, England.

2102