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## Tetragonal polymorphic modification of tetrakis(*N,N*-diethyldithiocarbamate-*S,S'*)tellurium(IV)

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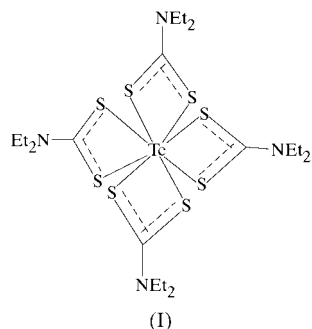
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During an investigation of the W/Te/Br<sub>2</sub> system, we prepared crystals of the title compound, C<sub>20</sub>H<sub>40</sub>N<sub>4</sub>S<sub>8</sub>Te, from acetonitrile solution by slow addition of diethyl ether. It appeared to be a new tetragonal modification. The calculated density of the tetragonal modification (1.513 Mg m<sup>-3</sup>) is slightly greater than found for the known orthorhombic modification (1.46 Mg m<sup>-3</sup>). The bond distances and angles are practically the same.

### Comment

Previously, the crystal structure of the title compound, (I), was investigated (Husebye & Svaeren, 1973) and an orthorhombic unit cell with space group *Pn*2<sub>1</sub>*a* was found. Orthorhombic crystals were prepared by crystallization from benzene solution.



During an investigation of the W/Te/Br<sub>2</sub> system, we prepared the title compound. Crystals were obtained from acetonitrile solution by slow addition of diethyl ether. Chemical composition and molecular structure are the same as previously, but the crystal structure appeared to be tetragonal, space group *P*4<sub>3</sub>2<sub>1</sub>2. This means that we have found a new tetragonal modification of Te(S<sub>2</sub>CNEt<sub>2</sub>)<sub>4</sub>.

In the previously known orthorhombic modification, there are two crystallographically independent molecules both in general positions. In our case, we have only one molecule lying on a twofold axis. The bond lengths and angles are practically the same. As previously, the Te atom is coordinated by four bidentate dithiocarbamate ligands. The coordination polyhedron around Te is a flattened square prism.

The possible reason for formation of two different modifications is the nature of the solvent used for preparation of the crystals: non-polar (C<sub>6</sub>H<sub>6</sub>) for orthorhombic modification and polar (CH<sub>3</sub>CN + Et<sub>2</sub>O) for tetragonal one.

The calculated density of the tetragonal modification (1.513 Mg m<sup>-3</sup>) is slightly greater than found for the orthorhombic crystals (1.46 Mg m<sup>-3</sup>).

### Experimental

The title compound was prepared during the investigation of a high-temperature reaction between W, Te and Br<sub>2</sub>. The product was heated with NaS<sub>2</sub>CNEt<sub>2</sub> at 453 K. The product was recrystallized from acetonitrile solution and after slow addition of diethyl ether, octahedral-shaped orange crystals were obtained.

#### Crystal data

C<sub>20</sub>H<sub>40</sub>N<sub>4</sub>S<sub>8</sub>Te  
*M*<sub>r</sub> = 720.64  
Tetragonal, *P*4<sub>3</sub>2<sub>1</sub>2  
*a* = 10.3970 (15) Å  
*c* = 29.262 (6) Å  
*V* = 3163.2 (9) Å<sup>3</sup>  
*Z* = 4  
*D*<sub>x</sub> = 1.513 Mg m<sup>-3</sup>

Mo *K*α radiation  
Cell parameters from 32 reflections  
θ = 11.2–14.4°  
μ = 1.486 mm<sup>-1</sup>  
*T* = 212 (2) K  
Octahedron, orange  
0.33 × 0.30 × 0.28 mm

#### Data collection

Stoe Stadi-4 diffractometer  
ω scans  
Absorption correction: empirical (*X-RED*; Stoe & Cie, 1998)  
*T*<sub>min</sub> = 0.596, *T*<sub>max</sub> = 0.660  
3161 measured reflections  
2791 independent reflections  
2527 reflections with *I* > 2σ(*I*)

*R*<sub>int</sub> = 0.021  
θ<sub>max</sub> = 25°  
*h* = 0 → 12  
*k* = 0 → 12  
*l* = 0 → 34  
3 standard reflections  
frequency: 60 min  
intensity decay: none

#### Refinement

Refinement on *F*<sup>2</sup>  
*R* [*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.029  
*wR*(*F*<sup>2</sup>) = 0.057  
*S* = 1.125  
2791 reflections  
150 parameters  
H-atom parameters constrained

*w* = 1/[σ<sup>2</sup>(*F*<sub>o</sub><sup>2</sup>) + (0.0079*P*)<sup>2</sup> + 5.2275*P*]  
where *P* = (*F*<sub>o</sub><sup>2</sup> + 2*F*<sub>c</sub><sup>2</sup>)/3  
(Δ/σ)<sub>max</sub> = 0.001  
Δρ<sub>max</sub> = 0.29 e Å<sup>-3</sup>  
Δρ<sub>min</sub> = -0.28 e Å<sup>-3</sup>  
Absolute structure: Flack (1983),  
1094 Friedel pairs  
Flack parameter = -0.06 (3)

**Table 1**

Selected geometric parameters (Å, °).

Te1–S11	2.8088 (11)	N1–C11	1.462 (7)
Te1–S12	2.7758 (13)	N1–C13	1.479 (6)
Te1–S21	2.7377 (12)	C11–C12	1.498 (7)
Te1–S22	2.6584 (13)	C13–C14	1.501 (7)
S11–C1	1.718 (4)	C2–N2	1.325 (5)
S12–C1	1.723 (5)	N2–C21	1.479 (6)
S21–C2	1.723 (5)	N2–C23	1.466 (6)
S22–C2	1.730 (5)	C21–C22	1.517 (8)
C1–N1	1.341 (5)	C23–C24	1.503 (7)

S11 <sup>i</sup> —Te1—S11	132.28 (5)	S21—Te1—S21 <sup>i</sup>	132.01 (5)
S12—Te1—S11 <sup>i</sup>	83.60 (4)	S22—Te1—S11 <sup>i</sup>	81.30 (4)
S12 <sup>i</sup> —Te1—S11	83.60 (4)	S22—Te1—S11	136.80 (4)
S12—Te1—S11	63.58 (3)	S22 <sup>i</sup> —Te1—S11	81.30 (4)
S12 <sup>i</sup> —Te1—S12	93.10 (5)	S22—Te1—S12 <sup>i</sup>	92.64 (4)
S21—Te1—S11 <sup>i</sup>	131.50 (3)	S22—Te1—S12	159.41 (4)
S21—Te1—S11	70.51 (3)	S22 <sup>i</sup> —Te1—S12	92.64 (4)
S21 <sup>i</sup> —Te1—S11	131.50 (3)	S22—Te1—S21	66.35 (4)
S21—Te1—S12 <sup>i</sup>	82.16 (4)	S22 <sup>i</sup> —Te1—S21	79.65 (4)
S21—Te1—S12	134.09 (3)	S22—Te1—S21 <sup>i</sup>	79.65 (4)
S21 <sup>i</sup> —Te1—S12	82.16 (4)	S22—Te1—S22 <sup>i</sup>	88.86 (6)

Symmetry code: (i)  $1 - y, 1 - x, \frac{3}{2} - z$ .

Data collection: *STADIA* (Stoe, 1998); cell refinement: *STADIA*;  
data reduction: *X-RED* (Stoe, 1998); program(s) used to solve

structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: local programs; software used to prepare material for publication: local programs.

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