

Fig. 2. A stereoview packing diagram of the unit cell of BPDT-TTF. Hydrogen atoms have been omitted for clarity.

Related literature. The X-ray crystal structures and conductivity properties of (BPDT-TTF)₂I₃ (Kobayashi, Takahashi, Kato, Kobayashi & Sasaki, 1984) and (BPDT-TTF)₃(PF₆)₂ (Kato, Mori, Kobayashi, Sasaki & Kobayashi, 1984) have been reported. The subject of tetrathiafulvalene-based organic synthetic metals has been reviewed (Williams, Beno, Wang, Leung, Emge, Geiser & Carlson, 1985).

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Structural Chirality of (–)-N-(3,4,4-Trimethyl-5-hexenoyl)camphorsultam*

BY G. BERNARDINELLI

Laboratoire de Cristallographie aux Rayons X, Université de Genève, 24 quai Ernest Ansermet, CH-1211 Genève 4, Switzerland

AND W. OPPOLZER AND P. SCHNEIDER

Département de Chimie Organique, Université de Genève, 30 quai Ernest Ansermet, CH-1211 Genève 4, Switzerland

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Abstract. The title compound (I) was obtained by BF₃OEt₂-mediated asymmetric 1,4-addition of PBU₃-stabilized methylcopper to the camphorsultam derivative (*E*)-10,10-dimethyl-4-(4,4-dimethyl-2,5-hexadienoyl)-3-thia-4-azatricyclo[5.2.1.0^{1,5}]decane 3,3-dioxide (II). (I): C₁₉H₃₁NO₃S, *M_r* = 353.5, orthorhombic, *P*2₁2₁2, *a* = 11.628 (2), *b* = 21.310 (7), *c* = 8.0574 (12) Å, *V* = 1996.6 (8) Å³, *Z* = 4, *D_x* = 1.176 g cm⁻³, λ(Mo *Kα*) = 0.71069 Å, μ = 1.692 cm⁻¹, *F*(000) = 768, m.p. 405–406 K, [α]_D^{20°C} = –47.0° (*c* = 1.14% in CHCl₃), room temperature, *R* = 0.049 for 1334 observed reflections [*|F_o*| ≥ 3σ(*F_o*)

and *|F_o*| ≥ 8.0]. The structural chirality of enantiomerically pure (I) was confirmed from the known absolute configuration of the camphorsultam moiety as well as by least-squares refinement of the absolute-structure parameter *x* = –0.02 (16) [Bernardinelli & Flack (1985). *Acta Cryst.* **A41**, 500–511].

Experimental. Single crystals of (I) were grown from an ethanol solution at room temperature. Colourless crystal of average dimensions 0.24 × 0.28 × 0.30 mm; Philips PW 1100 diffractometer, graphite-monochromated Mo *Kα*; cell dimensions from 21 reflections (2θ = 24–36°); data collection: sinθ/λ ≤ 0.505 Å⁻¹, *h* 0–10, *k* 0–20, *l* 0–8 and all antireflections of these; ω/2θ scans, ω-scan angle 1.2°; two standard

* (1*S*,5*R*)-10,10-Dimethyl-4-[(3*R*)-3,4,4-trimethyl-5-hexenoyl]-3-thia-4-azatricyclo[5.2.1.0^{1,5}]decane 3,3-dioxide.

reflections varied by a maximum of $2.3\sigma(I)$; 2558 measured reflections; 1334 observed with $|F| \geq 3\sigma(F)$ and $|F| \geq 8$; Lorentz-polarization; no absorption correction; systematic absences: $h00: h = 2n + 1, 0k0: k = 2n + 1$; structure solved by *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Full-matrix least squares using $|F|$ values. 218 parameters refined; no secondary-extinction correction; all coordinates of the H atoms were calculated. $R = 4.9\%$, $wR = 6.8\%$, $w(F) = \exp[18(\sin\theta/\lambda)^2]$; max. and av. ratio of $|\text{shift}|$ to e.s.d.: 0.045 and 0.005 respectively; max. and min. heights in the final

Table 1. Fractional coordinates and equivalent isotropic temperature factors, U_{eq} ($\text{\AA}^2 \times 10^3$), with e.s.d.'s in parentheses

U_{eq} is the average of the eigenvalues of U.				
	x	y	z	U_{eq}
S	0.03537 (10)	0.39612 (6)	0.00596 (19)	47.5 (3)
O(1)	0.1036 (3)	0.45188 (20)	0.0051 (7)	65.9 (14)
O(2)	0.0957 (4)	0.33902 (21)	-0.0230 (6)	70.4 (15)
O(3)	-0.0579 (4)	0.4092 (3)	0.4547 (5)	86.6 (22)
N	-0.0406 (4)	0.38980 (22)	0.1841 (5)	47.0 (14)
C(1)	-0.1909 (5)	0.38527 (24)	-0.0279 (7)	46.0 (17)
C(2)	-0.1651 (5)	0.3974 (3)	0.1581 (6)	44.4 (15)
C(3)	-0.2432 (6)	0.3494 (3)	0.2487 (7)	62.8 (21)
C(4)	-0.3205 (6)	0.3257 (3)	0.1069 (7)	66.7 (22)
C(5)	-0.3910 (6)	0.3809 (4)	0.0459 (9)	79 (3)
C(6)	-0.3036 (6)	0.4204 (3)	-0.0590 (9)	68.6 (22)
C(7)	-0.2318 (6)	0.3148 (3)	-0.0323 (8)	65.7 (22)
C(8)	-0.1409 (7)	0.2654 (3)	0.0046 (11)	87 (3)
C(9)	-0.2849 (8)	0.2969 (4)	-0.2007 (10)	96 (3)
C(10)	-0.0855 (5)	0.4038 (3)	-0.1270 (6)	53.7 (19)
C(11)	0.0061 (4)	0.3996 (4)	0.3380 (7)	57.9 (19)
C(12)	0.1347 (5)	0.4003 (4)	0.3520 (7)	64.9 (22)
C(13)	0.1788 (5)	0.3878 (3)	0.5269 (7)	54.6 (17)
C(14)	0.3082 (5)	0.4070 (3)	0.5455 (7)	65.8 (22)
C(15)	0.3816 (6)	0.3711 (5)	0.4284 (11)	85 (3)
C(16)	0.4535 (13)	0.3308 (7)	0.4643 (18)	155 (6)
C(17)	0.1571 (7)	0.3194 (4)	0.5708 (8)	76.7 (25)
C(18)	0.3195 (6)	0.4780 (4)	0.5036 (14)	99 (3)
C(19)	0.3449 (6)	0.3998 (6)	0.7263 (9)	102 (3)

Table 2. Interatomic distances (\AA) and selected torsional angles ($^\circ$) with e.s.d.'s in parentheses

S—O(1)	1.429 (4)	C(4)—C(5)	1.517 (10)
S—O(2)	1.424 (5)	C(4)—C(7)	1.541 (9)
S—N	1.691 (4)	C(5)—C(6)	1.567 (10)
S—C(10)	1.775 (6)	C(7)—C(8)	1.522 (10)
O(3)—C(11)	1.217 (7)	C(7)—C(9)	1.539 (11)
N—C(2)	1.471 (7)	C(11)—C(12)	1.499 (7)
N—C(11)	1.370 (7)	C(12)—C(13)	1.523 (8)
C(1)—C(2)	1.550 (7)	C(13)—C(14)	1.568 (8)
C(1)—C(6)	1.530 (9)	C(13)—C(17)	1.520 (10)
C(1)—C(7)	1.575 (8)	C(14)—C(15)	1.485 (11)
C(1)—C(10)	1.515 (8)	C(14)—C(18)	1.556 (11)
C(2)—C(3)	1.551 (9)	C(14)—C(19)	1.526 (9)
C(3)—C(4)	1.539 (9)	C(15)—C(16)	1.234 (17)
C(10)—S—N—C(2)	-4.0 (4)	O(3)—C(11)—C(12)—C(13)	23.4 (11)
C(10)—S—N—C(11)	-161.6 (5)	C(11)—C(12)—C(13)—C(14)	-164.3 (6)
N—S—C(10)—C(1)	-13.6 (4)	C(11)—C(12)—C(13)—C(17)	70.4 (8)
S—N—C(2)—C(1)	20.0 (6)	C(12)—C(13)—C(14)—C(15)	-60.1 (7)
S—N—C(11)—O(3)	162.3 (6)	C(12)—C(13)—C(14)—C(18)	58.7 (7)
S—N—C(11)—C(12)	-15.4 (9)	C(12)—C(13)—C(14)—C(19)	174.4 (7)
C(10)—C(1)—C(2)—N	-29.4 (6)	C(13)—C(14)—C(15)—C(16)	-111.8 (12)
C(2)—C(1)—C(10)—S	26.3 (5)	C(18)—C(14)—C(15)—C(16)	129.4 (12)
N—C(11)—C(12)—C(13)	-159.1 (6)		

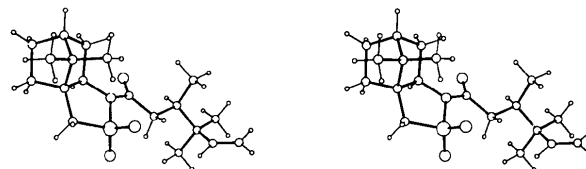
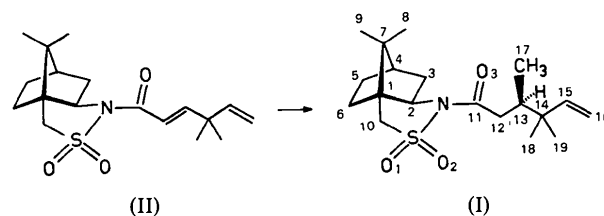


Fig. 1. Stereopair showing the configuration of the molecule.

difference electron density map of 0.40 and -0.39 e \AA^{-3} ; atomic scattering factors and anomalous-dispersion terms for S, N and O atoms from *International Tables for X-ray Crystallography* (1974); all calculations performed with a local version of *XRAY76* (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976) and *ORTEPII* (Johnson, 1976).*



Atomic coordinates are given in Table 1, bond lengths and torsional angles in Table 2 and a stereoview of the molecule in Fig. 1.

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* Lists of structure factors, atomic positional and thermal parameters for all atoms and other information in the printed form of the Standard Crystallographic File Structure of Brown (1985) have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43537 (40 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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