Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)*

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
O1	0.13782 (7)	0.3792 (2)	−0.5006 (2)	0.0510 (6)
N1	0.37017 (10)	0.4032 (2)	−0.3021 (3)	0.0553 (7)
C1	0.09437 (11)	0.3439 (2)	−0.4023 (3)	0.0501 (8)
C2	0.11662 (13)	0.2425 (3)	−0.3133 (3)	0.0566 (8)
C3	0.17315 (13)	0.2287 (3)	−0.2759 (3)	0.0529 (8)
C4	0.21455 (11)	0.3095 (2)	−0.3308 (3)	0.0395 (7)
C5	0.19473 (10)	0.3808 (2)	−0.4437 (3)	0.0386 (7)
C6	0.23234 (12)	0.4556 (2)	−0.5143 (3)	0.0446 (7)
C7	0.28952 (12)	0.4602 (2)	−0.4667 (3)	0.0448 (7)
C8	0.31239 (11)	0.3916 (2)	−0.3474 (3)	0.0415 (7)
C9	0.27493 (11)	0.3148 (2)	−0.2792 (3)	0.0408 (7)
C10	0.30027 (14)	0.2458 (3)	−0.1635 (3)	0.0549 (8)
C11	0.3584 (2)	0.2592 (3)	−0.1203 (4)	0.0652 (10)
C12	0.39126 (15)	0.3392 (3)	−0.1903 (3)	0.0607 (9)
C13	0.0828 (2)	0.4479 (3)	−0.3066 (5)	0.0695 (10)
C14	0.04104 (15)	0.3123 (4)	−0.5049 (5)	0.0681 (10)
O*W*	0.46867 (12)	0.5390 (3)	−0.3869 (3)	0.0764 (8)

Table 2. *Geometric parameters (Å, °)*

O1—C5	1.373 (3)	C4—C5	1.371 (4)
O1—C1	1.468 (3)	C4—C9	1.436 (4)
N1—C12	1.323 (4)	C5—C6	1.420 (4)
N1—C8	1.371 (3)	C6—C7	1.357 (4)
C1—C2	1.490 (4)	C7—C8	1.417 (4)
C1—C13	1.523 (4)	C8—C9	1.421 (4)
C1—C14	1.530 (4)	C9—C10	1.414 (4)
C2—C3	1.332 (4)	C10—C11	1.378 (4)
C3—C4	1.455 (4)	C11—C12	1.387 (5)
C5—O1—C1	116.9 (2)	C4—C5—C6	122.1 (2)
C12—N1—C8	117.8 (3)	O1—C5—C6	116.0 (2)
O1—C1—C2	109.5 (2)	C7—C6—C5	119.6 (3)
O1—C1—C13	107.8 (2)	C6—C7—C8	121.0 (2)
C2—C1—C13	111.3 (3)	N1—C8—C7	118.0 (2)
O1—C1—C14	104.0 (2)	N1—C8—C9	122.8 (2)
C2—C1—C14	112.0 (3)	C7—C8—C9	119.2 (2)
C13—C1—C14	111.8 (3)	C10—C9—C8	117.0 (3)
C3—C2—C1	121.2 (3)	C10—C9—C4	123.5 (2)
C2—C3—C4	119.7 (3)	C8—C9—C4	119.5 (2)
C5—C4—C9	118.4 (2)	C11—C10—C9	119.0 (3)
C5—C4—C3	117.3 (2)	C10—C11—C12	120.0 (3)
C9—C4—C3	124.2 (2)	N1—C12—C11	123.4 (3)
C4—C5—O1	121.8 (2)		

Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELX*76 (Sheldrick, 1976). Molecular graphics: *ORTEP*II (Johnson, 1976). Material was prepared for publication using an IBM PC AT 486 .

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: PA1202). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Comparison of the Geometries of 3,7,10-Tribenzyl-3,7,10-triaza[3.3.3]propellane, (1), 7,10-Dibenzyl-3-*p*-tolyl-3,7,10-triaza[3.3.3]propellane, (2), and 7,10-Dibenzyl-3-oxa-7,10-diaza[3.3.3]propellane, (3)

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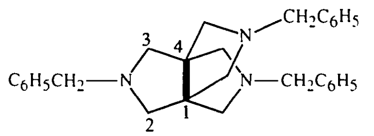
Abstract

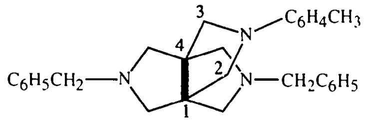
Room-temperature crystal structures are reported for three analogous [3.3.3]propellanes. Compound (1), $C_{29}H_{33}N_3$, is trigonal, (2), $C_{29}H_{33}N_3$, triclinic and (3), $C_{22}H_{26}N_2O$, orthorhombic. All three molecules have eclipsed conformations about the central conjoining bond and the five-membered rings all have envelope conformations (C_s-m symmetry).

Comment

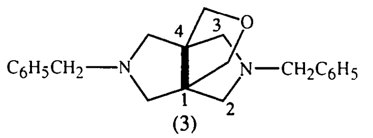
A [p,q,r]propellane is a tricyclic compound whose (p + 2),(q + 2),(r + 2)-membered alicyclic or heterocyclic rings share a common (conjoining) C—C bond; for an overview refer to Ginsburg (1975) and subsequent updates. Using the crystal structures of eighteen dithia[n.3.3]propellanes (n = 1, 2, 3) and dithia[4.3.3]-propellenes (in various forms) as a basis (Herbstein *et al.*, 1986), we proposed two generalizations for the conformations of propellane-type molecules: (*a*) propellane molecules are either eclipsed or staggered about the conjoining bond, intermediate situations not being found, *i.e.* τ(C2—C1—C4—C3) $\simeq$ 0 or 60° (atom numbering as in scheme below, in which the conjoining bonds are emphasized); (*b*) it follows from (*a*) that the three rings of a propellane molecule all have the same conformation and symmetry, *e.g.* considering only five-membered rings (n = 3), all three have envelope conformations (C_s-m symmetry; $\tau \simeq 0°$) or all three have twist conformations (C_2-2 symmetry; $\tau \simeq 60°$).



(1)



(2)



(3)

We now report the structures (at 298 K) of three substituted [3.3.3]propellanes that conform to these generalizations.

3,7,10-Tribenzyl-3,7,10-triaza[3.3.3]propellane, (1) (Kapon, Kaftory, Knowles, Weinberg & Ginsburg, 1987; Weinberg, Knowles & Ginsburg, 1985), has crystallographic C_3-3 symmetry and thus the flaps of the three pyrrolidine rings all point in the same direction (Fig. 1). The benzyl groups are linked equatorially to the ring N atoms and are twisted about the N—CH_2 bonds such that the molecule as a whole has the shape of a three-legged footstool with all three phenyl groups pointing up from the plane perpendicularly bisecting the conjoining bond. The Cremer & Pople (1975) puckering parameters (Table 4) describe quantitatively the conformations of the five-membered rings (*cf.* Dunitz, 1978).

In 7,10-dibenzyl-3-*p*-tolyl-3,7,10-triaza[3.3.3]propellane, (2) (Kapon *et al.*, 1987; Weinberg *et al.*, 1985; Fig. 2), the triazapropellane moiety has approximate C_3 symmetry as in (1) and all three substituents are linked equatorially to the ring N atoms. However, the two benzyl groups are differently disposed with respect to the propellane moiety, one pointing upwards from the mean plane of the propellane moiety and the other downwards; the axis of the *p*-tolyl group lies parallel to the perpendicular bisector of the conjoining bond. Thus, the overall shapes of (1) and (2) are rather different. The five-membered rings have C_s-m symmetry.

For 7,10-dibenzyl-3-oxa-7,10-diaza[3.3.3]propellane, (3) (Kapon *et al.*, 1987; Weinberg *et al.*, 1985; Fig. 3), the intensities of the reflections fell off steeply with increasing θ. The propellane ring system has pseudo-threefold symmetry, with the ring flaps of the three envelopes all pointing in the same direction. The two benzyl groups are linked equatorially to the ring N atoms but are disposed above and below the plane that perpendicularly bisects the conjoining bond. Thus, there is a resemblance to (2). The five-membered rings have envelope conformations. The O atom and linked C atoms of the oxo ring have relatively high displacement parameters, but there is no evidence suggesting that the five-membered ring flaps between two envelope conformations.

The dimensions of the three molecules are as expected, with larger displacements for the phenyl rings. There is a small but systematic lengthening, compared with the standard C—C single bond length, of the conjoining bond to 1.564 (9), 1.566 (8) and 1.554 (7) Å in (1), (2) and (3), respectively. All three crystals have only van der Waals interactions between molecules and the packing does not show any unusual or special features. A full listing of geometric parameters has been deposited.

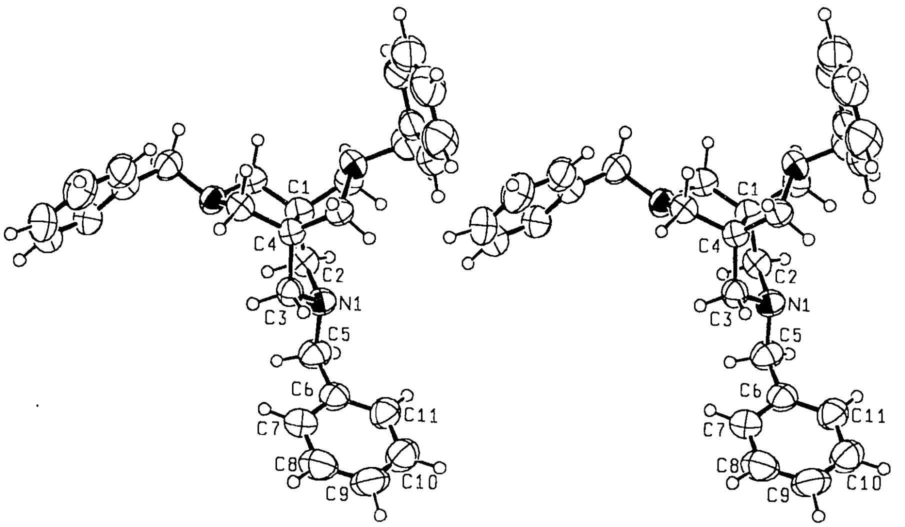


Fig. 1. Stereodiagram of (1) (50% probability ellipsoids).

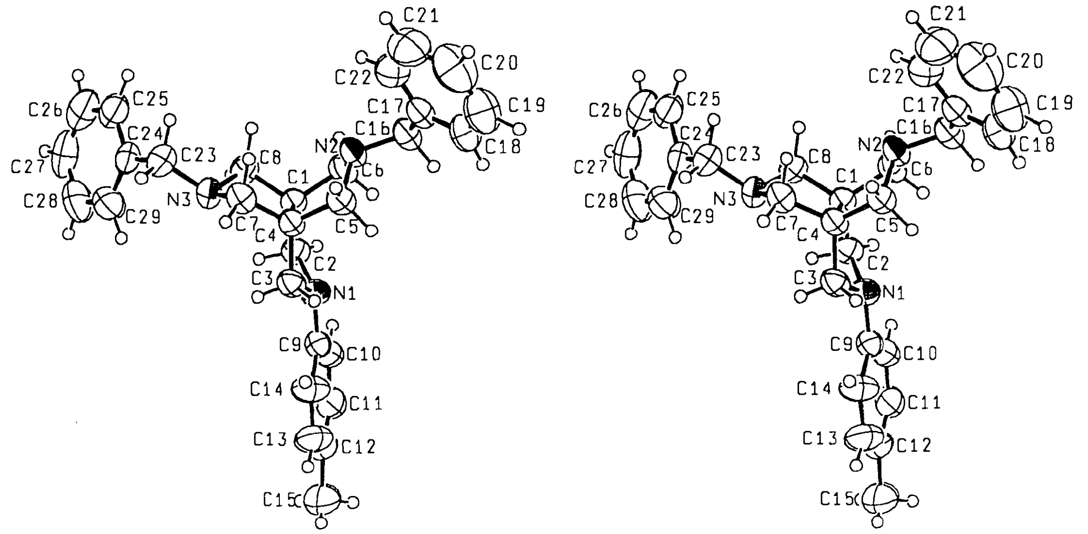


Fig. 2. Stereodiagram of (2) (50% probability ellipsoids).

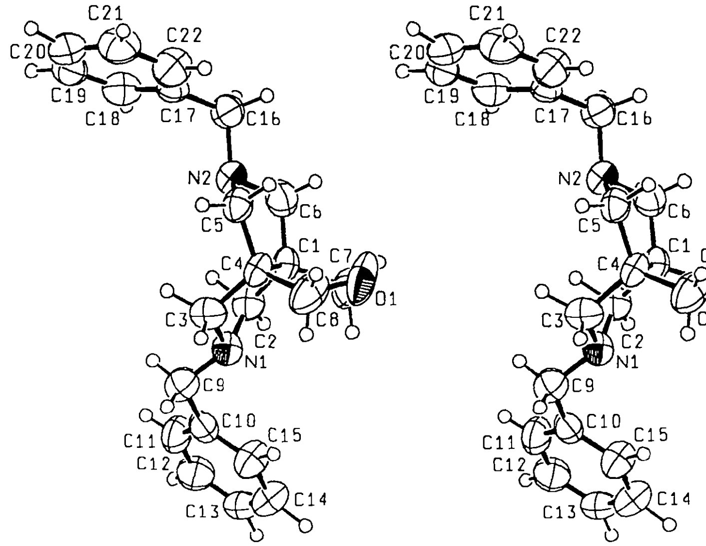


Fig. 3. Stereodiagram of (3) (50% probability ellipsoids).

Experimental

All three compounds were prepared according to Weinberg, Knowles & Ginsburg (1985), and crystallized from diethyl ether.

Compound (1)

Crystal data

$C_{29}H_{33}N_3$
$M_r = 423.58$
Trigonal
$R\bar{3}$
$a = 11.354\,(5)$ Å
$\alpha = 107.21\,(5)°$
$V = 1211.9\,(9)$ Å^3
$Z = 2$
$D_x = 1.161$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 3.0$–$10.4°$
$\mu = 0.068$ mm^{-1}
$T = 293\,(2)$ K
Prism
$0.3 \times 0.2 \times 0.2$ mm
Colourless

Data collection

Philips PW1100 computer-controlled diffractometer
ω–2θ scans
Absorption correction: none
683 measured reflections
683 independent reflections
659 observed reflections [$I > 2\sigma(I)$]

$\theta_{\max} = 22.50°$
$h = -9 \rightarrow 6$
$k = -6 \rightarrow 6$
$l = 1 \rightarrow 12$
3 standard reflections
frequency: 120 min
intensity decay: insignificant

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0544$
$wR(F^2) = 0.1357$
$S = 1.131$

$(\Delta/\sigma)_{\max} = 0.15$
$\Delta\rho_{\max} = 0.10$ e Å^{-3}
$\Delta\rho_{\min} = -0.13$ e Å^{-3}
Extinction correction: none

659 reflections
141 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.052P)^2]$
where $P = [\max(F_o^2,0) + 2F_c^2]/3$

Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²) for (1)*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
C1	0.1900 (5)	0.1900 (5)	0.1900 (5)	0.052 (2)
C2	0.2519 (4)	0.0971 (4)	0.1331 (4)	0.0590 (12)
C3	0.4339 (4)	0.2781 (4)	0.3212 (4)	0.0529 (12)
C4	0.3145 (5)	0.3145 (5)	0.3145 (5)	0.046 (2)
C5	0.4771 (5)	0.1125 (5)	0.1635 (6)	0.0682 (14)
C6	0.6230 (4)	0.2027 (4)	0.2052 (5)	0.0616 (12)
C7	0.7201 (5)	0.2474 (5)	0.3356 (6)	0.0783 (15)
C8	0.8543 (6)	0.3277 (6)	0.3730 (6)	0.091 (2)
C9	0.8952 (6)	0.3627 (6)	0.2807 (7)	0.093 (2)
C10	0.8009 (6)	0.3173 (5)	0.1519 (7)	0.084 (2)
C11	0.6662 (5)	0.2397 (5)	0.1141 (6)	0.0703 (14)
N1	0.3947 (3)	0.1886 (3)	0.1796 (3)	0.0541 (11)

Compound (2)

Crystal data

$C_{29}H_{33}N_3$
$M_r = 423.58$
Triclinic
$P\overline{1}$
$a = 13.283$ (7) Å
$b = 11.748$ (6) Å
$c = 8.533$ (4) Å
$\alpha = 103.94$ (5)°
$\beta = 96.87$ (5)°
$\gamma = 104.87$ (5)°
$V = 1225.5$ (11) Å^3
$Z = 2$
$D_x = 1.148$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 2.5$–$7.9°$
$\mu = 0.067$ mm^{-1}
$T = 293$ (2) K
Prism
0.3 × 0.2 × 0.15 mm
Colourless

Data collection

Philips PW1100 computer-controlled diffractometer
ω–2θ scans
Absorption correction: none
2994 measured reflections
2994 independent reflections
1953 observed reflections $[I > 2\sigma(I)]$

$\theta_{\rm max} = 22.02°$
$h = -13 \rightarrow 13$
$k = -12 \rightarrow 12$
$l = 0 \rightarrow 8$
3 standard reflections
frequency: 120 min
intensity decay: 5.4%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0610$
$wR(F^2) = 0.1365$
$S = 1.081$
2994 reflections
421 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0084P)^2 + 0.5631P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} = 0.13$
$\Delta\rho_{\rm max} = 0.15$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.14$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 2. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²) for (2)*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
C1	0.8079 (2)	−0.0456 (3)	0.4148 (4)	0.0460 (9)
C2	0.8458 (3)	0.0801 (4)	0.5432 (5)	0.0527 (10)
C3	0.6670 (3)	−0.0003 (4)	0.5548 (5)	0.0558 (10)
C4	0.6880 (2)	−0.0979 (3)	0.4209 (4)	0.0492 (9)
C5	0.6294 (3)	−0.1136 (4)	0.2487 (4)	0.0555 (10)
C6	0.8062 (3)	−0.0375 (4)	0.2384 (4)	0.0571 (10)
C7	0.6818 (3)	−0.2196 (4)	0.4608 (5)	0.0573 (10)
C8	0.8585 (3)	−0.1401 (4)	0.4586 (5)	0.0545 (10)
C9	0.7550 (3)	0.2216 (4)	0.6840 (4)	0.0543 (10)
C10	0.8436 (4)	0.3234 (4)	0.7237 (5)	0.0671 (12)
C11	0.8516 (4)	0.4304 (4)	0.8413 (5)	0.0748 (13)
C12	0.7719 (4)	0.4410 (4)	0.9278 (5)	0.0723 (12)
C13	0.6839 (4)	0.3415 (5)	0.8879 (6)	0.0840 (15)
C14	0.6737 (3)	0.2330 (4)	0.7689 (5)	0.0737 (13)
C15	0.7850 (8)	0.5555 (7)	1.0672 (9)	0.106 (2)
C16	0.6772 (3)	−0.1077 (4)	−0.0162 (5)	0.0635 (11)
C17	0.5794 (3)	−0.2013 (4)	−0.1252 (4)	0.0603 (10)
C18	0.4979 (3)	−0.1644 (5)	−0.1928 (5)	0.0721 (12)
C19	0.4110 (4)	−0.2459 (7)	−0.3016 (7)	0.098 (2)
C20	0.4033 (5)	−0.3657 (8)	−0.3447 (8)	0.119 (2)
C21	0.4802 (6)	−0.4092 (6)	−0.2804 (8)	0.110 (2)
C22	0.5699 (4)	−0.3253 (5)	−0.1675 (6)	0.0829 (14)
C23	0.8065 (4)	−0.3102 (4)	0.5803 (6)	0.0690 (12)
C24	0.9142 (3)	−0.2855 (4)	0.6863 (5)	0.0632 (11)
C25	0.9847 (4)	−0.3445 (4)	0.6291 (7)	0.0758 (13)
C26	1.0811 (5)	−0.3253 (6)	0.7289 (10)	0.097 (2)
C27	1.1060 (5)	−0.2496 (6)	0.8833 (10)	0.101 (2)
C28	1.0383 (5)	−0.1885 (6)	0.9416 (7)	0.104 (2)
C29	0.9422 (4)	−0.2073 (5)	0.8429 (6)	0.0863 (14)
N1	0.7477 (2)	0.1134 (3)	0.5636 (3)	0.0529 (8)
N2	0.7067 (2)	−0.1289 (3)	0.1427 (3)	0.0545 (8)
N3	0.7880 (2)	−0.1973 (3)	0.5561 (3)	0.0547 (8)

Compound (3)

Crystal data

$C_{22}H_{26}N_2O$
$M_r = 334.45$
Orthorhombic
$Pbca$
$a = 35.656$ (20) Å
$b = 9.652$ (5) Å
$c = 11.018$ (6) Å
$V = 3791.9$ (36) Å^3
$Z = 8$
$D_x = 1.172$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 2.8$–$10.9°$
$\mu = 0.072$ mm^{-1}
$T = 293$ (2) K
Transparent plate
0.3 × 0.23 × 0.1 mm
Colourless

Data collection

Philips PW1100 computer-controlled diffractometer
ω–2θ scans
Absorption correction: none
2475 measured reflections
2475 independent reflections
1096 observed reflections $[I > 2\sigma(I)]$

$\theta_{\rm max} = 22.50°$
$h = 0 \rightarrow 38$
$k = 0 \rightarrow 10$
$l = 0 \rightarrow 11$
3 standard reflections
frequency: 120 min
intensity decay: 2.2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0702$
$wR(F^2) = 0.1691$
$S = 0.982$

$(\Delta/\sigma)_{\rm max} = 0.074$
$\Delta\rho_{\rm max} = 0.17$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.22$ e Å^{-3}
Extinction correction: none

2475 reflections
330 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0324P)^2 + 2.48P]$
where $P = (F_o^2 + 2F_c^2)/3$

Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 3. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²) for (3)*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
C1	0.11402 (14)	0.0429 (5)	0.2715 (4)	0.0511 (14)
C2	0.0749 (2)	0.0127 (6)	0.3187 (6)	0.057 (2)
C3	0.1050 (2)	−0.1946 (7)	0.3330 (7)	0.061 (2)
C4	0.13447 (15)	−0.0986 (5)	0.2833 (4)	0.0514 (14)
C5	0.1663 (2)	−0.0702 (7)	0.3723 (5)	0.055 (2)
C6	0.1367 (2)	0.1418 (7)	0.3513 (7)	0.070 (2)
C7	0.1154 (2)	0.0757 (10)	0.1361 (6)	0.079 (2)
C8	0.1476 (2)	−0.1284 (10)	0.1538 (6)	0.084 (2)
C9	0.0367 (2)	−0.1941 (8)	0.3478 (7)	0.068 (2)
C10	0.0004 (2)	−0.1492 (6)	0.2890 (5)	0.057 (2)
C11	−0.0305 (2)	−0.1103 (7)	0.3562 (6)	0.071 (2)
C12	−0.0638 (2)	−0.0721 (7)	0.3028 (7)	0.074 (2)
C13	−0.0671 (2)	−0.0712 (6)	0.1796 (6)	0.064 (2)
C14	−0.0366 (2)	−0.1086 (7)	0.1107 (6)	0.074 (2)
C15	−0.0031 (2)	−0.1472 (7)	0.1647 (6)	0.069 (2)
C16	0.1840 (2)	0.1173 (7)	0.5102 (6)	0.063 (2)
C17	0.19892 (13)	0.0272 (6)	0.6096 (5)	0.0476 (14)
C18	0.1801 (2)	0.0156 (7)	0.7172 (6)	0.066 (2)
C19	0.1929 (2)	−0.0675 (8)	0.8099 (6)	0.078 (2)
C20	0.2252 (2)	−0.1417 (8)	0.7965 (7)	0.083 (2)
C21	0.2457 (2)	−0.1280 (7)	0.6909 (7)	0.079 (2)
C22	0.2318 (2)	−0.0442 (8)	0.5972 (7)	0.076 (2)
N1	0.06974 (12)	−0.1341 (5)	0.2907 (4)	0.0552 (12)
N2	0.15356 (12)	0.0494 (4)	0.4413 (4)	0.0535 (12)
O1	0.14755 (13)	0.0023 (6)	0.0922 (4)	0.103 (2)

Table 4. *Cremer & Pople ring-puckering parameters (Cremer & Pople, 1975; Cremer, 1979) for the C_4N rings in (1) and (2), and the two C_4N rings and C_4O ring in (3)*

The τ values are the torsion angles about the conjoining bond in the three rings: calculations by *PARST* (Nardelli, 1983).

	Q (Å)	φ (°)	τ (°)	Comments
(1)	0.420 (5)	104.6 (7)	−2.7 (5)	C_3 symmetry
(2)	0.368 (4)	105.8 (6)	−1.5 (4)	N1 ring (tolyl)
	0.402 (4)	107.4 (6)	−0.5 (4)	N2 ring
	0.443 (3)	105.1 (5)	−2.2 (4)	N3 ring
(3)	0.448 (6)	109.6 (7)	1.1 (6)	Aza ring (N1)
	0.406 (6)	110.8 (7)	1.9 (6)	Aza ring (N2)
	0.361 (6)	113 (1)	3.3 (6)	Furan ring

For all compounds, program(s) used to solve structures: *SHELXS*86 (Sheldrick, 1990); program(s) used to refine structures: *SHELXL*93 (Sheldrick, 1993); molecular graphics: *ORTEP* (Johnson, 1965); software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1166). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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1,1-Bis(3′,4,4′-trimethyltetrathiafulvalen-3-yl)ethanol Ethanol Hemisolvate; a New Bifunctional π-Donor Molecule

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Abstract

The title compound {1,1-bis[2-(4,5-dimethyl-1,3-dithiol-2-ylidene)-5-methyl-1,3-dithiol-4-yl]ethanol ethanol hemisolvate, $C_{20}H_{22}OS_8.0.5C_2H_5OH$} contains bridged tetrathiafulvalene (TTF) molecules. The two TTF moieties of the bifunctional donor are quasi-orthogonal. Donor layers and hydrogen-bonded solvent layers alternate along the c axis.

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