$S = 0.910$
4152 reflections
305 parameters
All H-atom parameters refined
Calculated weights $w = 1/[\sigma^2(F_o^2) + (0.0572P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
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 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

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

	x	y	z	U_{eq}
C1*A*	−0.0716 (2)	0.55378 (9)	0.74258 (10)	0.0395 (3)
C2*A*	−0.1922 (2)	0.51035 (9)	0.66521 (11)	0.0424 (4)
C3*A*	−0.3378 (2)	0.51229 (11)	0.67073 (14)	0.0524 (4)
C4*A*	−0.3687 (2)	0.55541 (12)	0.75322 (15)	0.0579 (5)
C5*A*	−0.2542 (2)	0.59798 (13)	0.83043 (15)	0.0605 (5)
C6*A*	−0.1087 (2)	0.59697 (11)	0.82466 (13)	0.0522 (4)
C7*A*	0.0854 (2)	0.55290 (9)	0.73940 (10)	0.0407 (4)
C8*A*	0.2110 (2)	0.60172 (12)	0.82110 (13)	0.0507 (4)
C9*A*	0.2879 (3)	0.5422 (2)	0.9151 (2)	0.0776 (7)
N1*A*	0.10925 (15)	0.50514 (9)	0.66502 (9)	0.0465 (3)
O1*A*	0.26130 (13)	0.50491 (9)	0.66789 (10)	0.0610 (3)
O2*A*	−0.16895 (14)	0.46549 (8)	0.58157 (8)	0.0542 (3)
C1*B*	0.2082 (2)	0.74457 (9)	1.07936 (11)	0.0423 (4)
C2*B*	0.2675 (2)	0.79876 (10)	1.01492 (11)	0.0443 (4)
C3*B*	0.4197 (2)	0.79548 (12)	1.02358 (14)	0.0547 (4)
C4*B*	0.5176 (2)	0.74053 (13)	1.09804 (15)	0.0606 (5)
C5*B*	0.4639 (2)	0.68842 (13)	1.16442 (15)	0.0629 (5)
C6*B*	0.3118 (2)	0.69006 (12)	1.15399 (13)	0.0551 (4)
C7*B*	0.0454 (2)	0.74464 (9)	1.06989 (11)	0.0431 (4)
C8*B*	−0.0204 (2)	0.67514 (11)	1.12598 (14)	0.0519 (4)
C9*B*	−0.0252 (3)	0.7087 (2)	1.2325 (2)	0.0677 (6)
N1*B*	−0.03563 (15)	0.80753 (8)	1.01210 (10)	0.0473 (3)
O1*B*	−0.18876 (14)	0.80397 (8)	1.00698 (10)	0.0622 (4)
O2*B*	0.1770 (2)	0.85614 (8)	0.94125 (9)	0.0578 (3)

Table 2. *Selected geometric parameters* (Å, °)

C1*A*—C6*A*	1.397 (2)	C1*B*—C6*B*	1.397 (2)
C1*A*—C2*A*	1.412 (2)	C1*B*—C2*B*	1.404 (2)
C1*A*—C7*A*	1.470 (2)	C1*B*—C7*B*	1.475 (2)
C2*A*—O2*A*	1.368 (2)	C2*B*—O2*B*	1.364 (2)
C2*A*—C3*A*	1.376 (2)	C2*B*—C3*B*	1.380 (2)
C3*A*—C4*A*	1.374 (2)	C3*B*—C4*B*	1.376 (3)
C4*A*—C5*A*	1.374 (3)	C4*B*—C5*B*	1.376 (3)
C5*A*—C6*A*	1.376 (3)	C5*B*—C6*B*	1.373 (3)
C7*A*—N1*A*	1.289 (2)	C7*B*—N1*B*	1.285 (2)
C7*A*—C8*A*	1.502 (2)	C7*B*—C8*B*	1.506 (2)
C8*A*—C9*A*	1.509 (3)	C8*B*—C9*B*	1.513 (3)
N1*A*—O1*A*	1.399 (2)	N1*B*—O1*B*	1.401 (2)
O1*A*—H1*AO*	0.86 (2)	O1*B*—H1*BO*	0.91 (2)
O2*A*—H2*AO*	0.96 (2)	O2*B*—H2*BO*	0.98 (2)
C6*A*—C1*A*—C2*A*	116.21 (15)	C6*B*—C1*B*—C2*B*	116.6 (2)
C6*A*—C1*A*—C7*A*	121.14 (14)	C6*B*—C1*B*—C7*B*	121.12 (14)
C2*A*—C1*A*—C7*A*	122.64 (13)	C2*B*—C1*B*—C7*B*	122.32 (13)
O2*A*—C2*A*—C3*A*	117.54 (14)	O2*B*—C2*B*—C3*B*	117.56 (15)
O2*A*—C2*A*—C1*A*	121.32 (14)	O2*B*—C2*B*—C1*B*	121.38 (15)
C3*A*—C2*A*—C1*A*	121.15 (15)	C3*B*—C2*B*—C1*B*	121.06 (15)
C4*A*—C3*A*—C2*A*	120.5 (2)	C4*B*—C3*B*—C2*B*	120.4 (2)
C5*A*—C4*A*—C3*A*	120.1 (2)	C3*B*—C4*B*—C5*B*	120.0 (2)
C4*A*—C5*A*—C6*A*	119.6 (2)	C6*B*—C5*B*—C4*B*	119.6 (2)
C5*A*—C6*A*—C1*A*	122.4 (2)	C5*B*—C6*B*—C1*B*	122.4 (2)
N1*A*—C7*A*—C1*A*	115.98 (13)	N1*B*—C7*B*—C1*B*	116.20 (13)
N1*A*—C7*A*—C8*A*	122.37 (15)	N1*B*—C7*B*—C8*B*	122.4 (2)
C1*A*—C7*A*—C8*A*	121.60 (14)	C1*B*—C7*B*—C8*B*	121.42 (13)
C7*A*—C8*A*—C9*A*	112.0 (2)	C7*B*—C8*B*—C9*B*	112.13 (15)
C7*A*—N1*A*—O1*A*	113.32 (12)	C7*B*—N1*B*—O1*B*	113.25 (12)

Table 3. *Selected intra- and intermolecular hydrogen-bond distances* (Å) *and angles* (°)

D	H	*A*	*D*···*A*	*D*—H···*A*
O2*A*	H2*AO*	N1*A*	2.534 (2)	146 (2)
O2*B*	H2*BO*	N1*B*	2.542 (2)	151 (2)
O1*B*	H1*BO*	O2*A*^{i}	2.797 (2)	166 (2)
O1*A*	H1*AO*	O2*B*^{ii}	2.788 (2)	154 (2)

Symmetry codes: (i) $-\frac{1}{2} - x, \frac{1}{2} + y, \frac{3}{2} - z$; (ii) $\frac{1}{2} - x, -\frac{1}{2} + y, \frac{3}{2} - z$.

Data collection, cell refinement and data reduction: Enraf–Nonius software. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL* (Sheldrick, 1994). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and a stereoview of the crystal packing have been deposited with the IUCr (Reference: AB1142). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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(1*S*,2*S*,5*R*,6*S*)-(+)-6-Carbanilino-1,5-dimethyltricyclo[3.2.0.0^{2,6}]heptane

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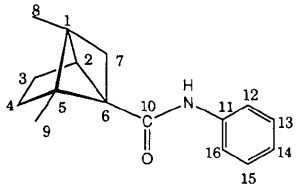
Abstract

The asymmetric unit of the title compound, *N*-phenyl-6-(1,5-dimethyltricyclo[3.2.0.0^{2,6}]heptane)carboxamide, $C_{16}H_{19}NO$, contains three independent molecules. There are intermolecular hydrogen bonds of the N—H···O type.

Comment

The synthesis of the title compound (1) was accomplished by the Favorskii rearrangement of (1*R*,4*S*)-(+)-3,3,8-tribromocamphor, initiated by potassium anilide (Lu, Liu & Wang, 1994). The compound was purified by flash column chromatography (silica gel, 70~230 mesh, ethyl acetate/hexane from 1/50 to 1/20) and recrystallized from *n*-hexane.



(1)

The structure determination of the title compound was undertaken in order to establish the identity of this hitherto unknown compound. Fig. 1 shows displacement ellipsoid plots of the three independent molecules in the asymmetric unit and Fig. 2 depicts the packing within the unit cell along with the hydrogen-bonding scheme.

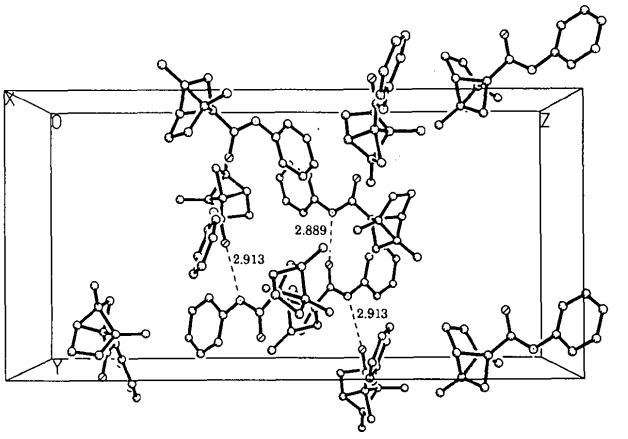


Fig. 2. Packing of the unit cell with dotted lines indicating the hydrogen bonds. Distances are in Å.

For additional information on the related structure 1,2,5,6-tetraphenyltricyclo[3.3.0.0^{2,6}]octane, see Hasegawa & Mukai (1989). The structural parameters of the title compound provide important information about related highly strained skeletons.

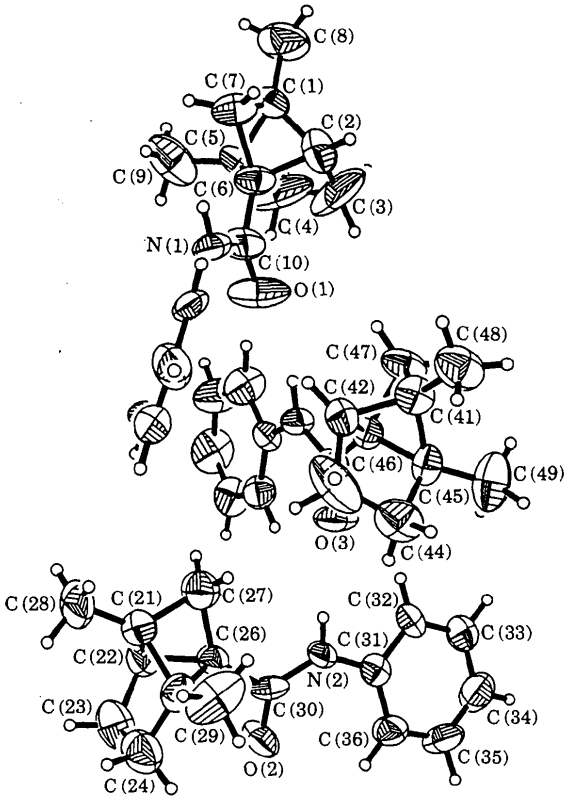


Fig. 1. View of the three independent molecules in the asymmetric unit of the title compound plotted with 40% probability ellipsoids. In the numbering scheme, atoms C1, C2, *etc.* correspond to atoms C21, C22 and C41, C42, *etc.*, respectively, for the three molecules.

Experimental

Crystal data

$C_{16}H_{19}NO$
M_r = 241.34
Orthorhombic
$P2_12_12_1$
a = 12.454 (3) Å
b = 13.106 (4) Å
c = 26.755 (8) Å
V = 4367 (2) Å^3
Z = 12
D_x = 1.101 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 45 reflections
θ = 1.5–25.0°
μ = 0.068 mm^{-1}
T = 293 K
0.6 × 0.6 × 0.5 mm
Yellow

Data collection

Siemens *R3m/V* diffractometer
$2\theta/\theta$ scans
Absorption correction: none
3273 measured reflections
3250 independent reflections
1726 observed reflections [$F > 4.0\sigma(F)$]
θ_{max} = 27.5°
$h = 0 \rightarrow 13$
$k = 0 \rightarrow 14$
$l = 0 \rightarrow 28$
2 standard reflections monitored every 100 reflections
intensity variation: none

Refinement

Refinement on F
R = 0.0634
wR = 0.0674
S = 1.92
1726 reflections
487 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F) + 0.0005F^2]$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

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

	x	y	z	U_{eq}
Molecule (I)				
O(1)	0.4645 (6)	0.0340 (6)	0.1328 (4)	0.120 (5)
N(1)	0.3364 (6)	−0.0819 (6)	0.1499 (3)	0.068 (4)
C(1)	0.6285 (11)	−0.2348 (9)	0.1362 (5)	0.076 (5)
C(2)	0.5931 (14)	−0.1663 (13)	0.0943 (6)	0.122 (8)
C(3)	0.6764 (19)	−0.0756 (17)	0.0907 (8)	0.211 (14)
C(4)	0.6988 (13)	−0.0650 (12)	0.1422 (9)	0.160 (11)
C(5)	0.6283 (11)	−0.1353 (9)	0.1679 (5)	0.077 (5)
C(6)	0.5188 (10)	−0.1371 (8)	0.1386 (5)	0.069 (5)
C(7)	0.5102 (10)	−0.2520 (8)	0.1508 (5)	0.086 (6)
C(8)	0.7195 (10)	−0.3145 (10)	0.1356 (6)	0.134 (7)
C(9)	0.6236 (12)	−0.1357 (11)	0.2230 (6)	0.140 (8)
C(10)	0.4386 (12)	−0.0561 (9)	0.1399 (5)	0.076 (6)
C(11)	0.2457 (12)	−0.0160 (9)	0.1569 (4)	0.065 (5)
C(12)	0.1452 (12)	−0.0511 (9)	0.1405 (5)	0.073 (5)
C(13)	0.0603 (12)	0.0144 (13)	0.1439 (5)	0.097 (7)
C(14)	0.0703 (17)	0.1119 (5)	0.1637 (6)	0.120 (9)
C(15)	0.1703 (18)	0.1420 (12)	0.1812 (7)	0.123 (9)
C(16)	0.2574 (13)	0.0796 (10)	0.1765 (4)	0.096 (6)
Molecule (II)				
O(2)	0.2293 (6)	0.7210 (6)	0.1191 (3)	0.091 (4)
N(2)	0.2865 (7)	0.5863 (6)	0.0722 (3)	0.056 (3)
C(21)	0.1076 (10)	0.4729 (9)	0.1937 (5)	0.075 (5)
C(22)	0.1867 (10)	0.5677 (9)	0.2061 (5)	0.080 (5)
C(23)	0.1174 (12)	0.6557 (11)	0.2219 (5)	0.105 (7)
C(24)	0.0226 (12)	0.6429 (11)	0.1846 (6)	0.109 (7)
C(25)	0.0576 (10)	0.5513 (10)	0.1577 (5)	0.073 (5)
C(26)	0.1806 (9)	0.5571 (8)	0.1475 (4)	0.061 (5)
C(27)	0.1891 (10)	0.4402 (9)	0.1552 (5)	0.090 (6)
C(28)	0.0448 (11)	0.4084 (10)	0.2305 (5)	0.117 (7)
C(29)	−0.0134 (11)	0.5134 (9)	0.1168 (5)	0.129 (7)
C(30)	0.2369 (9)	0.6284 (8)	0.1103 (5)	0.063 (5)
C(31)	0.3489 (10)	0.6401 (9)	0.0364 (4)	0.056 (5)
C(32)	0.4318 (11)	0.5876 (9)	0.0145 (5)	0.073 (5)
C(33)	0.4953 (10)	0.6362 (12)	−0.0215 (5)	0.086 (6)
C(34)	0.4765 (12)	0.7343 (13)	−0.0347 (5)	0.099 (7)
C(35)	0.3944 (13)	0.7831 (10)	−0.0134 (6)	0.092 (6)
C(36)	0.3270 (10)	0.7382 (9)	0.0233 (5)	0.074 (5)
Molecule (III)				
O(3)	0.3586 (6)	0.3774 (6)	0.0652 (3)	0.088 (4)
N(3)	0.4446 (7)	0.2474 (6)	0.1038 (3)	0.054 (3)
C(41)	0.2674 (10)	0.1201 (8)	−0.0108 (5)	0.069 (5)
C(42)	0.2284 (10)	0.1467 (8)	0.0408 (4)	0.067 (5)
C(43)	0.1324 (10)	0.2161 (13)	0.0360 (6)	0.122 (8)
C(44)	0.1696 (10)	0.2846 (10)	−0.0064 (6)	0.098 (6)
C(45)	0.2781 (9)	0.2373 (9)	−0.0177 (4)	0.066 (5)
C(46)	0.3307 (8)	0.2113 (8)	0.0335 (4)	0.056 (4)
C(47)	0.3832 (9)	0.1162 (8)	0.0090 (4)	0.079 (5)
C(48)	0.2144 (9)	0.0513 (9)	−0.0493 (5)	0.104 (6)
C(49)	0.3376 (10)	0.2851 (11)	−0.0612 (5)	0.121 (7)
C(50)	0.3787 (9)	0.2870 (8)	0.0682 (4)	0.053 (4)
C(51)	0.5073 (9)	0.3026 (9)	0.1388 (4)	0.057 (5)
C(52)	0.5796 (11)	0.2437 (10)	0.1661 (5)	0.092 (6)
C(53)	0.6469 (12)	0.2924 (11)	0.2012 (6)	0.106 (7)
C(54)	0.6433 (13)	0.3958 (13)	0.2080 (5)	0.111 (8)
C(55)	0.5732 (12)	0.4500 (11)	0.1827 (5)	0.085 (6)
C(56)	0.5046 (10)	0.4055 (9)	0.1477 (5)	0.075 (5)

Table 2. *Selected geometric parameters* (Å, °)

Molecule (I)			
O(1)—C(10)	1.239 (14)	C(5)—C(6)	1.573 (18)
N(1)—C(10)	1.345 (18)	C(5)—C(9)	1.477 (21)
N(1)—C(11)	1.434 (17)	C(6)—C(7)	1.544 (15)
C(1)—C(2)	1.502 (20)	C(6)—C(10)	1.457 (17)
C(1)—C(5)	1.555 (17)	C(11)—C(12)	1.404 (20)
C(1)—C(7)	1.541 (18)	C(11)—C(16)	1.367 (17)
C(1)—C(8)	1.541 (18)	C(12)—C(13)	1.365 (20)
C(2)—C(3)	1.580 (28)	C(13)—C(14)	1.388 (25)
C(2)—C(6)	1.552 (21)	C(14)—C(15)	1.388 (30)
C(3)—C(4)	1.413 (31)	C(15)—C(16)	1.365 (25)
C(4)—C(5)	1.445 (22)		
C(1)—C(2)—C(3)	107.6 (14)	C(5)—C(1)—C(8)	125.1 (11)
C(1)—C(2)—C(6)	75.7 (10)	C(5)—C(6)—C(7)	88.3 (9)
C(1)—C(5)—C(4)	106.0 (12)	C(5)—C(6)—C(10)	124.9 (10)
C(1)—C(5)—C(6)	73.6 (9)	C(6)—C(5)—C(9)	117.5 (11)
C(1)—C(5)—C(9)	122.8 (11)	C(7)—C(1)—C(8)	127.3 (10)
C(1)—C(7)—C(6)	74.8 (8)	C(7)—C(6)—C(10)	131.2 (11)
C(2)—C(1)—C(5)	84.5 (10)	O(1)—C(10)—N(1)	121.0 (11)
C(2)—C(1)—C(7)	89.7 (11)	O(1)—C(10)—C(6)	120.9 (12)
C(2)—C(1)—C(8)	127.8 (12)	N(1)—C(10)—C(6)	118.1 (10)
C(2)—C(3)—C(4)	98.2 (15)	N(1)—C(11)—C(12)	117.7 (10)
C(2)—C(6)—C(5)	82.3 (10)	N(1)—C(11)—C(16)	121.2 (13)
C(2)—C(6)—C(7)	87.8 (10)	C(10)—N(1)—C(11)	128.4 (9)
C(2)—C(6)—C(10)	127.2 (12)	C(11)—C(12)—C(13)	117.6 (12)
C(3)—C(2)—C(6)	104.7 (13)	C(11)—C(16)—C(15)	120.0 (15)
C(3)—C(4)—C(5)	106.3 (15)	C(12)—C(11)—C(16)	121.1 (13)
C(4)—C(5)—C(6)	107.5 (12)	C(12)—C(13)—C(14)	122.4 (15)
C(4)—C(5)—C(9)	120.1 (13)	C(13)—C(14)—C(15)	118.1 (17)
C(5)—C(1)—C(7)	89.0 (9)	C(14)—C(15)—C(16)	120.8 (15)
Molecule (II)			
O(2)—C(30)	1.241 (13)	C(25)—C(26)	1.558 (17)
N(2)—C(30)	1.312 (15)	C(25)—C(29)	1.491 (18)
N(2)—C(31)	1.422 (14)	C(26)—C(27)	1.549 (15)
C(21)—C(22)	1.619 (17)	C(26)—C(30)	1.535 (16)
C(21)—C(25)	1.540 (18)	C(31)—C(32)	1.372 (17)
C(21)—C(27)	1.509 (18)	C(31)—C(36)	1.361 (16)
C(21)—C(28)	1.514 (18)	C(32)—C(33)	1.398 (18)
C(22)—C(23)	1.502 (19)	C(33)—C(34)	1.354 (23)
C(22)—C(26)	1.575 (17)	C(34)—C(35)	1.333 (21)
C(23)—C(24)	1.556 (20)	C(35)—C(36)	1.419 (19)
C(24)—C(25)	1.466 (19)		
C(21)—C(22)—C(23)	107.3 (10)	C(25)—C(21)—C(28)	124.8 (11)
C(21)—C(22)—C(26)	72.5 (8)	C(25)—C(26)—C(27)	89.7 (8)
C(21)—C(25)—C(24)	111.0 (11)	C(25)—C(26)—C(30)	126.3 (10)
C(21)—C(25)—C(26)	75.2 (8)	C(26)—C(25)—C(29)	118.1 (11)
C(21)—C(25)—C(29)	118.5 (11)	C(27)—C(21)—C(28)	129.2 (10)
C(21)—C(27)—C(26)	76.4 (8)	C(27)—C(26)—C(30)	131.1 (10)
C(22)—C(21)—C(25)	82.1 (9)	O(2)—C(30)—N(2)	126.5 (11)
C(22)—C(21)—C(27)	87.0 (9)	O(2)—C(30)—C(26)	115.9 (10)
C(22)—C(21)—C(28)	127.6 (11)	N(2)—C(30)—C(26)	117.6 (9)
C(22)—C(23)—C(24)	99.9 (10)	N(2)—C(31)—C(32)	116.9 (10)
C(22)—C(26)—C(25)	83.0 (9)	N(2)—C(31)—C(36)	122.2 (10)
C(22)—C(26)—C(27)	87.3 (9)	C(30)—N(2)—C(31)	124.9 (9)
C(22)—C(26)—C(30)	124.8 (9)	C(31)—C(32)—C(33)	119.5 (11)
C(23)—C(22)—C(26)	108.7 (10)	C(31)—C(36)—C(35)	116.9 (11)
C(23)—C(24)—C(25)	100.3 (11)	C(32)—C(31)—C(36)	120.9 (11)
C(24)—C(25)—C(26)	109.7 (10)	C(32)—C(33)—C(34)	120.9 (12)
C(24)—C(25)—C(29)	117.1 (11)	C(33)—C(34)—C(35)	118.5 (13)
C(25)—C(21)—C(27)	91.9 (10)	C(34)—C(35)—C(36)	123.3 (13)
Molecule (III)			
O(3)—C(50)	1.212 (13)	C(45)—C(46)	1.556 (16)
N(3)—C(50)	1.361 (14)	C(45)—C(49)	1.514 (17)
N(3)—C(51)	1.417 (14)	C(46)—C(47)	1.553 (15)
C(41)—C(42)	1.503 (17)	C(46)—C(50)	1.484 (16)
C(41)—C(45)	1.553 (16)	C(51)—C(52)	1.394 (18)
C(41)—C(47)	1.538 (16)	C(51)—C(56)	1.369 (16)
C(41)—C(48)	1.521 (17)	C(52)—C(53)	1.409 (20)
C(42)—C(43)	1.508 (18)	C(53)—C(54)	1.368 (23)
C(42)—C(46)	1.542 (16)	C(54)—C(55)	1.313 (21)
C(43)—C(44)	1.519 (21)	C(55)—C(56)	1.396 (18)
C(44)—C(45)	1.518 (17)		
C(41)—C(42)—C(43)	108.5 (10)	C(45)—C(41)—C(48)	122.9 (10)
C(41)—C(42)—C(46)	75.2 (8)	C(45)—C(46)—C(47)	89.0 (8)
C(41)—C(45)—C(44)	107.7 (10)	C(45)—C(46)—C(50)	125.0 (9)
C(41)—C(45)—C(46)	73.4 (8)	C(46)—C(45)—C(49)	124.1 (10)
C(41)—C(45)—C(49)	122.9 (10)	C(47)—C(41)—C(48)	128.4 (10)
C(41)—C(47)—C(46)	73.9 (7)	C(47)—C(46)—C(50)	129.1 (9)
C(42)—C(41)—C(45)	84.7 (8)	O(3)—C(50)—N(3)	122.9 (10)
C(42)—C(41)—C(47)	89.7 (9)	O(3)—C(50)—C(46)	122.0 (10)
C(42)—C(41)—C(48)	128.3 (10)	N(3)—C(50)—C(46)	115.2 (9)
C(42)—C(43)—C(44)	100.3 (10)	N(3)—C(51)—C(52)	114.8 (10)
C(42)—C(46)—C(45)	83.3 (8)	N(3)—C(51)—C(56)	127.1 (10)
C(42)—C(46)—C(47)	87.7 (8)	C(50)—N(3)—C(51)	126.9 (9)
C(42)—C(46)—C(50)	128.4 (10)	C(51)—C(52)—C(53)	118.8 (12)

C(43)—C(42)—C(46)	108.2 (10)	C(51)—C(56)—C(55)	120.8 (11)
C(43)—C(44)—C(45)	100.3 (10)	C(52)—C(51)—C(56)	118.0 (11)
C(44)—C(45)—C(46)	106.8 (9)	C(52)—C(53)—C(54)	121.2 (13)
C(44)—C(45)—C(49)	114.7 (10)	C(53)—C(54)—C(55)	119.3 (14)
C(45)—C(41)—C(47)	89.6 (8)	C(54)—C(55)—C(56)	121.8 (13)

Data were corrected for Lorentz and polarization effects. The structure was solved by direct methods using *SHELXTL-Plus* (Sheldrick, 1989). Blocked least-squares refinements were carried out with *SHELX*76 (Sheldrick, 1976). Anisotropic refinement reduced R to 0.094. H atoms were added at idealized positions and included in the final refinement.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HL1036). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Molecules Isoelectronic with 2,2,2-Triphenylethanol: Multiple Hydrogen-Bonding Modes in the Structures of *O*-Tritylhydroxylamine, Ph_3CONH_2, and Triphenylmethanesulfenamide, Ph_3CSNH_2

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Abstract

O-Tritylhydroxylamine, $C_{19}H_{17}NO$ (IV), forms dimers in the solid state which are made up from two different molecules; these dimers exhibit three type of hydrogen bond, intermolecular N—H···N and N—H···π(arene), and intramolecular (aryl)C—H···O. Triphenylmethanesulfenamide, $C_{19}H_{17}NS$ (V), forms centrosymmetric dimers in the solid state in which N—H···S hydrogen bonds are the sole type observed.

Comment

Some general principles underlying hydrogen-bond formation in systems containing equal numbers of hydrogen-bond donors and acceptors have been formulated recently (Etter, 1990; Etter, MacDonald & Bernstein, 1990). The strongest hydrogen-bond donor will interact preferentially with the strongest acceptor, followed by the next-strongest donor and acceptor pair and so on until all the hydrogen-bond donor and acceptor capacity has been employed. Where there is a numerical mismatch between hydrogen-bond donors and acceptors, an excess of donors can often be accommodated either by formation of X—H···π(arene) hydrogen bonds or by a change of hybridization at the acceptor site (Hanton, Hunter & Purvis, 1992), while an excess of acceptors can be accommodated by formation of C—H···X hydrogen bonds involving C—H bonds on benzenoid rings as hydrogen-bond donors (Hunter, 1991).

We have recently tested these general ideas by comparing the hydrogen bonding in three isoelectronic and isosteric molecular systems, Ph_3COH (I), Ph_3CNH_2 (II) and $Ph_2C(C_5H_4N)OH$ (III). In this series, (I) contains equal numbers of hydrogen-bond donors and acceptors, (II) contains an excess of donors and (III) contains an excess of acceptors. It is found that whereas compound (I) forms tetrahedral tetrameric hydrogen-bonded aggregates (Ferguson, Gallagher, Glidewell, Low & Scrimgeour, 1992), (II) forms no hydrogen bonds at all (Glidewell & Ferguson, 1994), and of the potential hydrogen-bond acceptor sites in (III), only the N atom is used and the O atom is not involved in any hydrogen bonding at all (Glidewell & Ferguson, 1994).

As a further test, we have now compared the structures of *O*-tritylhydroxylamine, Ph_3CONH_2 (IV), and triphenylmethanesulfenamide, Ph_3CSNH_2 (V), with the isosteric 2,2,2-triphenylethanol, Ph_3CCH_2OH (VI) (Ferguson, Glidewell & Zakaria, 1994). Both (IV) and (V) contain two potential hydrogen-bond donors (in the NH_2 groups) and two potential acceptor sites (N and O or S) per molecule, but compound (VI) contains just one donor and one acceptor per molecule. Hence the numbers of hydrogen-bond donors and acceptors in each compound are matched, although their identities differ. The structure of (VI) consists of cyclic, almost planar centrosymmetric tetramers built up using only O—H···O hydrogen bonds with precise pairing of donors and acceptors (Ferguson, Glidewell & Zakaria, 1994). Compound (IV), by contrast, crystallizes as dimers in which the principal intermolecular hydrogen bonding is of the N—H···N type with the O atoms, unused in in-