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Molecular Recognition in Amides. Structure of *N,N'*-Di(triphenylmethyl)urea–*N*-Acetylmethionine Ethyl Ester (1/1)

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Abstract. $C_{39}H_{32}N_2O.C_9H_{17}NO_3S$, $M_r = 763.99$, orthorhombic, $P2_12_12_1$, $a = 17.108$ (4), $b = 19.415$ (5), $c = 24.066$ (5) Å, $V = 7993.6$ Å^3, $Z = 8$, $D_x = 1.270$ g cm^{-3}, Mo $K\alpha$ radiation, $\lambda = 0.7107$ Å, $\mu = 1.23$ cm^{-1}, $F(000) = 3248$, $T = 128$ K, final $R = 0.054$ and $wR = 0.059$ for 5563 observed unique reflections. The results illustrate the excellent functional complementarity between coplanar amide groups of host and guest for an effective interaction and favourable guest inclusion.

Experimental. The title compound was prepared by H. Hart and co-workers (Michigan State University) by dissolving the host and guest components in hot ethyl acetate, and subsequent crystallization by gradual cooling. Crystal size $0.4 \times 0.3 \times 0.2$ mm; cell dimensions determined from setting angles of 15 reflections in the range $10 < \theta < 14°$; space group derived by systematic absences. Data measured at low temperature on an upgraded Picker diffractometer with graphite-monochromated Mo $K\alpha$ radiation, $\theta_{max} = 27°$ ($\sin\theta/\lambda < 0.64$ Å^{-1}), ω–2θ scans, constant scan speed 3° min^{-1}, $h = 0$ to 21, $k = 0$ to 24, $l = 0$ to 30. Three reflections monitored periodically during data collection indicated no crystal deterioration. 8111 unique reflections were measured of which 5563 with $I > 3\sigma(I)$ were used in refinement. No corrections for absorption or secondary extinction were applied.

The structure was solved by direct methods (*MULTAN*80; Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), and refined by block-diagonal least squares [using an extensively modified version of *ORFLS* (Busing, Martin & Levy, 1962)] on F, including positional and anisotropic thermal parameters of all the non-H atoms. H atoms involved in hydrogen bonds were located in difference Fourier maps. The remaining H atoms were introduced in calculated positions, the methyls being treated as rigid groups. Minimization of $w(\Delta F)^2$, with $w = 1/[\sigma^2(F) + 0.002F^2]$, converged at $R = 0.056$, $wR = 0.059$, $S = 1.56$. Final $\Delta/\sigma < 0.36$, residual densities in Fourier maps ranging from -0.29 to 0.26 e Å^{-3}. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV).

Atomic parameters are listed in Table 1; bond distances and bond angles (Nardelli, 1983) are presented in Table 2. The molecular structure and atom-labelling scheme are shown in Fig. 1 (Johnson, 1976).*

Related literature. Structural features of the title compound have already been described elsewhere (Goldberg, 1988, 1991). The hydrogen-bond-directed molecular-recognition features of *N*-tritylurea and *N,N*-ditritylurea (Goldberg, Lin & Hart, 1985; Hart, Lin & Goldberg, 1986; Hart, Lin, Ng, Ward,

* Lists of anisotropic thermal parameters, H-atom coordinates and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55019 (29 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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

Primed and unprimed atoms represent the two crystallographically independent molecular entities in the asymmetric unit. U_{eq} is defined as one third of the trace of the U_{ij} tensor.

	x	y	z	U_{eq}
O(1)	0.1859 (2)	0.3180 (1)	0.0224 (1)	0.0236 (12)
C(2)	0.1984 (2)	0.2560 (2)	0.0184 (2)	0.0202 (16)
N(3)	0.2194 (2)	0.2140 (2)	0.0613 (1)	0.0216 (13)
N(4)	0.1967 (2)	0.2196 (2)	−0.0308 (1)	0.0202 (13)
C(5)	0.2248 (3)	0.2303 (2)	0.1208 (2)	0.0203 (16)
C(6)	0.2805 (3)	0.1736 (2)	0.1448 (2)	0.0208 (16)
C(7)	0.3456 (3)	0.1896 (2)	0.1764 (2)	0.0256 (17)
C(8)	0.3915 (3)	0.1377 (3)	0.1987 (2)	0.0315 (20)
C(9)	0.3742 (3)	0.0691 (3)	0.1884 (2)	0.0371 (22)
C(10)	0.3095 (3)	0.0531 (2)	0.1565 (2)	0.0364 (23)
C(11)	0.2620 (3)	0.1047 (2)	0.1354 (2)	0.0281 (17)
C(12)	0.1444 (3)	0.2225 (2)	0.1495 (2)	0.0198 (17)
C(13)	0.0757 (3)	0.2395 (2)	0.1222 (2)	0.0257 (18)
C(14)	0.0042 (3)	0.2308 (2)	0.1479 (2)	0.0314 (19)
C(15)	−0.0003 (3)	0.2071 (2)	0.2023 (2)	0.0315 (19)
C(16)	0.0679 (3)	0.1911 (2)	0.2300 (2)	0.0305 (19)
C(17)	0.1403 (3)	0.1983 (2)	0.2039 (2)	0.0273 (19)
C(18)	0.2602 (3)	0.3020 (2)	0.1305 (2)	0.0198 (16)
C(19)	0.3261 (3)	0.3211 (2)	0.0997 (2)	0.0268 (18)
C(20)	0.3656 (3)	0.3809 (2)	0.1125 (2)	0.0305 (19)
C(21)	0.3401 (3)	0.4226 (2)	0.1561 (2)	0.0298 (17)
C(22)	0.2745 (3)	0.4049 (2)	0.1857 (2)	0.0280 (19)
C(23)	0.2346 (3)	0.3441 (2)	0.1726 (2)	0.0246 (18)
C(24)	0.1536 (3)	0.2433 (2)	−0.0807 (2)	0.0214 (17)
C(25)	0.0727 (3)	0.2707 (2)	−0.0620 (2)	0.0220 (16)
C(26)	0.0423 (3)	0.3339 (2)	−0.0793 (2)	0.0303 (20)
C(27)	−0.0304 (3)	0.3555 (3)	−0.0599 (2)	0.0364 (21)
C(28)	−0.0730 (3)	0.3159 (3)	−0.0232 (2)	0.0359 (20)
C(29)	−0.0420 (3)	0.2529 (3)	−0.0057 (2)	0.0342 (20)
C(30)	0.0288 (3)	0.2297 (2)	−0.0255 (2)	0.0264 (18)
C(31)	0.2025 (3)	0.2941 (2)	−0.1153 (2)	0.0245 (18)
C(32)	0.2723 (3)	0.3206 (2)	−0.0962 (2)	0.0296 (19)
C(33)	0.3209 (3)	0.3597 (2)	−0.1309 (2)	0.0337 (22)
C(34)	0.2988 (3)	0.3718 (2)	−0.1856 (2)	0.0422 (22)
C(35)	0.2278 (4)	0.3475 (3)	−0.2043 (2)	0.0443 (24)
C(36)	0.1805 (3)	0.3089 (2)	−0.1697 (2)	0.0360 (22)
C(37)	0.1451 (3)	0.1787 (2)	−0.1174 (2)	0.0202 (16)
C(38)	0.0750 (3)	0.1595 (2)	−0.1417 (2)	0.0264 (18)
C(39)	0.0719 (3)	0.1015 (2)	−0.1760 (2)	0.0313 (18)
C(40)	0.1383 (3)	0.0630 (2)	−0.1853 (2)	0.0311 (20)
C(41)	0.2094 (3)	0.0826 (2)	−0.1619 (2)	0.0271 (19)
C(42)	0.2118 (3)	0.1408 (2)	−0.1282 (2)	0.0263 (17)
C(43)	0.5892 (3)	0.1069 (3)	0.3636 (2)	0.0431 (23)
S(44)	0.5002 (1)	0.0729 (1)	0.3346 (1)	0.0340 (5)
C(45)	0.4392 (3)	0.0718 (2)	0.3960 (2)	0.0301 (18)
C(46)	0.4593 (3)	0.0162 (2)	0.4381 (2)	0.0288 (19)
C(47)	0.4244 (3)	0.0323 (2)	0.4949 (2)	0.0245 (16)
N(48)	0.3393 (2)	0.0366 (2)	0.4936 (1)	0.0241 (15)
C(49)	0.2924 (3)	−0.0177 (2)	0.4986 (2)	0.0286 (17)
O(50)	0.3206 (2)	−0.0765 (1)	0.5057 (1)	0.0286 (13)
C(51)	0.2069 (3)	−0.0064 (3)	0.4961 (2)	0.0380 (21)
C(52)	0.4540 (3)	−0.0159 (2)	0.5396 (2)	0.0288 (20)
O(53)	0.5050 (2)	−0.0575 (1)	0.5334 (1)	0.0343 (13)
O(54)	0.4205 (2)	−0.0006 (1)	0.5890 (1)	0.0297 (13)
C(55)	0.4501 (3)	−0.0394 (2)	0.6363 (2)	0.0331 (19)
C(56)	0.4161 (4)	−0.0067 (3)	0.6874 (2)	0.0434 (22)
O(1′)	0.8334 (2)	0.3128 (1)	0.5254 (1)	0.0222 (11)
C(2′)	0.8153 (2)	0.2515 (2)	0.5204 (2)	0.0210 (16)
N(3′)	0.8167 (2)	0.2160 (2)	0.4714 (1)	0.0225 (15)
N(4′)	0.7877 (2)	0.2114 (1)	0.5626 (1)	0.0220 (14)
C(5′)	0.8589 (3)	0.2393 (2)	0.4209 (2)	0.0221 (18)
C(6′)	0.9392 (3)	0.2696 (2)	0.4371 (2)	0.0232 (16)
C(7′)	0.9682 (3)	0.3297 (2)	0.4151 (2)	0.0291 (18)
C(8′)	1.0404 (3)	0.3553 (3)	0.4332 (2)	0.0371 (20)
C(9′)	1.0823 (3)	0.3214 (3)	0.4734 (2)	0.0380 (21)
C(10′)	1.0551 (3)	0.2601 (3)	0.4950 (2)	0.0349 (20)
C(11′)	0.9839 (3)	0.2339 (2)	0.4763 (2)	0.0283 (18)
C(12′)	0.8075 (3)	0.2889 (2)	0.3864 (2)	0.0268 (18)
C(13′)	0.7425 (3)	0.3207 (2)	0.4094 (2)	0.0297 (21)
C(14′)	0.6942 (3)	0.3608 (3)	0.3758 (2)	0.0384 (22)
C(15′)	0.7105 (3)	0.3694 (2)	0.3194 (2)	0.0409 (23)
C(16′)	0.7758 (4)	0.3383 (3)	0.2966 (2)	0.0437 (23)
C(17′)	0.8239 (3)	0.2984 (2)	0.3301 (2)	0.0369 (22)
C(18′)	0.8685 (3)	0.1729 (2)	0.3856 (2)	0.0220 (16)
C(19′)	0.9387 (3)	0.1534 (2)	0.3623 (2)	0.0248 (17)
C(20′)	0.9429 (3)	0.0935 (2)	0.3297 (2)	0.0290 (19)
C(21′)	0.8770 (3)	0.0526 (2)	0.3219 (2)	0.0301 (18)
C(22′)	0.8064 (3)	0.0735 (2)	0.3445 (2)	0.0277 (17)
C(23′)	0.8016 (3)	0.1336 (2)	0.3755 (2)	0.0258 (17)

Table 1 (*cont.*)

	x	y	z	U_{eq}
C(24′)	0.7826 (2)	0.2281 (2)	0.6220 (2)	0.0183 (14)
C(25′)	0.7263 (3)	0.1740 (2)	0.6477 (2)	0.0221 (17)
C(26′)	0.6604 (3)	0.1915 (2)	0.6777 (2)	0.0267 (18)
C(27′)	0.6119 (3)	0.1411 (3)	0.6998 (2)	0.0332 (21)
C(28′)	0.6294 (3)	0.0722 (3)	0.6916 (2)	0.0320 (20)
C(29′)	0.6949 (3)	0.0542 (2)	0.6609 (2)	0.0341 (20)
C(30′)	0.7436 (3)	0.1046 (2)	0.6393 (2)	0.0304 (19)
C(31′)	0.8627 (3)	0.2204 (2)	0.6505 (2)	0.0221 (17)
C(32′)	0.8670 (3)	0.1997 (2)	0.7059 (2)	0.0256 (18)
C(33′)	0.9392 (3)	0.1911 (2)	0.7324 (2)	0.0319 (21)
C(34′)	1.0074 (3)	0.2021 (2)	0.7034 (2)	0.0321 (21)
C(35′)	1.0037 (3)	0.2234 (2)	0.6488 (2)	0.0311 (19)
C(36′)	0.9319 (3)	0.2325 (2)	0.6228 (2)	0.0270 (17)
C(37′)	0.7480 (3)	0.3002 (2)	0.6304 (2)	0.0206 (15)
C(38′)	0.7719 (3)	0.3435 (2)	0.6738 (2)	0.0227 (16)
C(39′)	0.7330 (3)	0.4050 (2)	0.6844 (2)	0.0273 (17)
C(40′)	0.6700 (3)	0.4244 (2)	0.6519 (2)	0.0318 (20)
C(41′)	0.6461 (3)	0.3825 (2)	0.6080 (2)	0.0291 (19)
C(42′)	0.6845 (3)	0.3210 (2)	0.5981 (2)	0.0249 (17)
C(43′)	0.4799 (3)	0.0993 (3)	−0.1791 (2)	0.0440 (21)
S(44′)	0.4272 (1)	0.1027 (1)	−0.1144 (1)	0.0335 (5)
C(45′)	0.4792 (3)	0.0388 (3)	−0.0746 (2)	0.0297 (19)
C(46′)	0.5599 (3)	0.0621 (2)	−0.0557 (2)	0.0248 (18)
C(47′)	0.6049 (3)	0.0044 (2)	−0.0252 (2)	0.0263 (18)
N(48′)	0.6816 (2)	0.0296 (2)	−0.0118 (1)	0.0267 (14)
C(49′)	0.7394 (3)	−0.0145 (2)	0.0019 (2)	0.0256 (17)
O(50′)	0.7279 (2)	−0.0776 (1)	0.0049 (1)	0.0282 (13)
C(51′)	0.8178 (3)	0.0166 (3)	0.0127 (2)	0.0327 (21)
C(52′)	0.5603 (3)	−0.0247 (2)	0.0250 (2)	0.0281 (17)
O(53′)	0.5061 (2)	−0.0629 (2)	0.0188 (1)	0.0414 (15)
O(54′)	0.5867 (2)	−0.0031 (1)	0.0737 (1)	0.0285 (12)
C(55′)	0.5453 (3)	−0.0312 (2)	0.1216 (2)	0.0364 (22)
C(56′)	0.5784 (3)	0.0014 (3)	0.1732 (2)	0.0409 (23)

Table 2. *Bond lengths* (Å) *and bond angles* (°)

O(1)—C(2)	1.226 (5)	O(1′)—C(2′)	1.235 (6)
C(2)—N(3)	1.364 (6)	C(2′)—N(3′)	1.366 (6)
C(2)—N(4)	1.380 (6)	C(2′)—N(4′)	1.362 (6)
N(3)—C(5)	1.469 (6)	N(3′)—C(5′)	1.484 (6)
N(4)—C(24)	1.483 (6)	N(4′)—C(24′)	1.469 (6)
C(5)—C(6)	1.566 (7)	C(5′)—C(6′)	1.544 (7)
C(5)—C(12)	1.548 (7)	C(5′)—C(12′)	1.546 (7)
C(5)—C(18)	1.535 (6)	C(5′)—C(18′)	1.553 (7)
C(6)—C(7)	1.384 (7)	C(6′)—C(7′)	1.375 (7)
C(6)—C(11)	1.392 (7)	C(6′)—C(11′)	1.397 (7)
C(7)—C(8)	1.386 (7)	C(7′)—C(8′)	1.401 (8)
C(8)—C(9)	1.386 (8)	C(8′)—C(9′)	1.372 (8)
C(9)—C(10)	1.383 (8)	C(9′)—C(10′)	1.380 (8)
C(10)—C(11)	1.386 (8)	C(10′)—C(11′)	1.395 (8)
C(12)—C(13)	1.386 (7)	C(12′)—C(13′)	1.387 (7)
C(12)—C(17)	1.393 (7)	C(12′)—C(17′)	1.396 (8)
C(13)—C(14)	1.382 (8)	C(13′)—C(14′)	1.393 (8)
C(14)—C(15)	1.389 (8)	C(14′)—C(15′)	1.394 (9)
C(15)—C(16)	1.380 (8)	C(15′)—C(16′)	1.383 (9)
C(16)—C(17)	1.396 (8)	C(16′)—C(17′)	1.388 (8)
C(18)—C(19)	1.399 (7)	C(18′)—C(19′)	1.379 (7)
C(18)—C(23)	1.375 (7)	C(18′)—C(23′)	1.396 (7)
C(19)—C(20)	1.378 (7)	C(19′)—C(20′)	1.405 (7)
C(20)—C(21)	1.396 (7)	C(20′)—C(21′)	1.390 (8)
C(21)—C(22)	1.373 (8)	C(21′)—C(22′)	1.386 (8)
C(22)—C(23)	1.401 (7)	C(22′)—C(23′)	1.388 (7)
C(24)—C(25)	1.549 (7)	C(24′)—C(25′)	1.553 (7)
C(24)—C(31)	1.538 (7)	C(24′)—C(31′)	1.540 (7)
C(34)—C(37)	1.540 (7)	C(24′)—C(37′)	1.534 (6)
C(25)—C(26)	1.396 (7)	C(25′)—C(26′)	1.382 (7)
C(25)—C(30)	1.404 (7)	C(25′)—C(30′)	1.394 (7)
C(26)—C(27)	1.393 (8)	C(26′)—C(27′)	1.388 (8)
C(27)—C(28)	1.379 (8)	C(27′)—C(28′)	1.385 (8)
C(28)—C(29)	1.397 (8)	C(28′)—C(29′)	1.387 (8)
C(29)—C(30)	1.378 (8)	C(29′)—C(30′)	1.387 (8)
C(31)—C(32)	1.378 (7)	C(31′)—C(32′)	1.394 (7)
C(31)—C(36)	1.393 (8)	C(31′)—C(36′)	1.379 (7)
C(32)—C(33)	1.402 (8)	C(32′)—C(33′)	1.400 (8)
C(33)—C(34)	1.389 (8)	C(33′)—C(34′)	1.376 (8)
C(34)—C(35)	1.379 (9)	C(34′)—C(35′)	1.379 (8)
C(35)—C(36)	1.381 (8)	C(35′)—C(36′)	1.389 (8)
C(37)—C(38)	1.386 (7)	C(37′)—C(38′)	1.402 (7)
C(37)—C(42)	1.381 (7)	C(37′)—C(42′)	1.395 (7)
C(38)—C(39)	1.399 (7)	C(38′)—C(39′)	1.391 (7)
C(39)—C(40)	1.378 (8)	C(39′)—C(40′)	1.383 (8)
C(40)—C(41)	1.393 (8)	C(40′)—C(41′)	1.395 (8)
C(41)—C(42)	1.392 (7)	C(41′)—C(42′)	1.385 (7)
C(43)—S(44)	1.800 (6)	C(43′)—S(44′)	1.802 (6)

Table 2 (*cont.*)

S(44)—C(45)	1.810 (5)	S(44′)—C(45′)	1.803 (5)
C(45)—C(46)	1.521 (7)	C(45′)—C(46′)	1.521 (7)
C(46)—C(47)	1.523 (7)	C(46′)—C(47′)	1.545 (7)
C(47)—N(48)	1.460 (6)	C(47′)—N(48′)	1.438 (7)
C(47)—C(52)	1.514 (7)	C(47′)—C(52′)	1.537 (7)
N(48)—C(49)	1.330 (7)	N(48′)—C(49′)	1.349 (7)
C(49)—O(50)	1.251 (6)	C(49′)—O(50′)	1.244 (6)
C(49)—C(51)	1.480 (8)	C(49′)—C(51′)	1.493 (8)
C(52)—O(53)	1.198 (6)	C(52′)—O(53′)	1.209 (6)
C(52)—O(54)	1.354 (6)	C(52′)—O(54′)	1.325 (6)
O(54)—C(55)	1.457 (6)	O(54′)—C(55′)	1.458 (7)
C(55)—C(56)	1.501 (8)	C(55′)—C(56′)	1.504 (8)
O(1)—C(2)—N(4)	124.5 (5)	O(1′)—C(2′)—N(4′)	124.3 (5)
O(1)—C(2)—N(3)	125.0 (5)	O(1′)—C(2′)—N(3′)	124.4 (5)
N(3)—C(2)—N(4)	110.4 (4)	N(3′)—C(2′)—N(4′)	111.2 (4)
C(2)—N(3)—C(5)	128.7 (4)	C(2′)—N(3′)—C(5′)	124.2 (4)
C(2)—N(4)—C(24)	123.1 (4)	C(2′)—N(4′)—C(24′)	128.3 (4)
N(3)—C(5)—C(18)	111.6 (4)	N(3′)—C(5′)—C(18′)	104.3 (4)
N(3)—C(5)—C(12)	111.0 (4)	N(3′)—C(5′)—C(12′)	110.6 (4)
N(3)—C(5)—C(6)	104.3 (4)	N(3′)—C(5′)—C(6′)	110.0 (4)
C(12)—C(5)—C(18)	111.8 (4)	C(12′)—C(5′)—C(18′)	106.5 (4)
C(6)—C(5)—C(18)	110.0 (4)	C(6′)—C(5′)—C(18′)	111.1 (4)
C(6)—C(5)—C(12)	107.9 (4)	C(6′)—C(5′)—C(12′)	113.8 (4)
C(5)—C(6)—C(11)	118.6 (5)	C(5′)—C(6′)—C(11′)	117.9 (5)
C(5)—C(6)—C(7)	122.3 (4)	C(5′)—C(6′)—C(7′)	123.2 (5)
C(7)—C(6)—C(11)	119.1 (5)	C(7′)—C(6′)—C(11′)	118.8 (5)
C(6)—C(7)—C(8)	120.4 (5)	C(6′)—C(7′)—C(8′)	120.0 (5)
C(7)—C(8)—C(9)	120.5 (5)	C(7′)—C(8′)—C(9′)	120.6 (6)
C(8)—C(9)—C(10)	119.1 (6)	C(8′)—C(9′)—C(10′)	120.3 (6)
C(9)—C(10)—C(11)	120.6 (5)	C(9′)—C(10′)—C(11′)	119.1 (6)
C(6)—C(11)—C(10)	120.2 (5)	C(6′)—C(11′)—C(10′)	121.1 (5)
C(5)—C(12)—C(17)	119.9 (5)	C(5′)—C(12′)—C(17′)	119.3 (5)
C(5)—C(12)—C(13)	121.2 (5)	C(5′)—C(12′)—C(13′)	121.3 (5)
C(13)—C(12)—C(17)	119.0 (5)	C(13′)—C(12′)—C(17′)	119.3 (5)
C(12)—C(13)—C(14)	120.5 (5)	C(12′)—C(13′)—C(14′)	119.5 (5)
C(13)—C(14)—C(15)	120.8 (5)	C(13′)—C(14′)—C(15′)	120.8 (6)
C(14)—C(15)—C(16)	118.9 (5)	C(14′)—C(15′)—C(16′)	119.7 (6)
C(15)—C(16)—C(17)	120.6 (5)	C(15′)—C(16′)—C(17′)	119.4 (6)
C(12)—C(17)—C(16)	120.1 (5)	C(12′)—C(17′)—C(16′)	121.2 (6)
C(5)—C(18)—C(23)	121.7 (5)	C(5′)—C(18′)—C(23′)	117.4 (5)
C(5)—C(18)—C(19)	118.6 (4)	C(5′)—C(18′)—C(19′)	122.9 (5)
C(19)—C(18)—C(23)	119.3 (5)	C(19′)—C(18′)—C(23′)	119.6 (5)
C(18)—C(19)—C(20)	120.1 (5)	C(18′)—C(19′)—C(20′)	119.9 (5)
C(19)—C(20)—C(21)	120.1 (5)	C(19′)—C(20′)—C(21′)	120.4 (5)
C(20)—C(21)—C(22)	120.1 (5)	C(20′)—C(21′)—C(22′)	119.1 (5)
C(21)—C(22)—C(23)	119.5 (5)	C(21′)—C(22′)—C(23′)	120.6 (5)
C(18)—C(23)—C(22)	120.8 (5)	C(18′)—C(23′)—C(22′)	120.2 (5)
N(4)—C(24)—C(37)	104.9 (4)	N(4′)—C(24′)—C(37′)	110.6 (4)
N(4)—C(24)—C(31)	111.5 (4)	N(4′)—C(24′)—C(31′)	111.1 (4)
N(4)—C(24)—C(25)	108.4 (4)	N(4′)—C(24′)—C(25′)	106.0 (4)
C(31)—C(24)—C(37)	105.3 (4)	C(31′)—C(24′)—C(37′)	111.9 (4)
C(25)—C(24)—C(37)	111.2 (4)	C(25′)—C(24′)—C(37′)	109.0 (4)
C(25)—C(24)—C(31)	115.1 (4)	C(25′)—C(24′)—C(31′)	108.0 (4)
C(24)—C(25)—C(30)	117.7 (5)	C(24′)—C(25′)—C(30′)	117.6 (5)
C(24)—C(25)—C(26)	123.3 (5)	C(24′)—C(25′)—C(26′)	123.3 (4)
C(26)—C(25)—C(30)	119.0 (5)	C(26′)—C(25′)—C(30′)	119.1 (5)
C(25)—C(26)—C(27)	119.9 (5)	C(25′)—C(26′)—C(27′)	121.0 (5)
C(26)—C(27)—C(28)	121.2 (6)	C(26′)—C(27′)—C(28′)	119.8 (5)
C(27)—C(28)—C(29)	118.7 (5)	C(27′)—C(28′)—C(29′)	119.6 (5)
C(28)—C(29)—C(30)	121.0 (6)	C(28′)—C(29′)—C(30′)	120.5 (5)
C(25)—C(30)—C(29)	120.1 (5)	C(25′)—C(30′)—C(29′)	120.0 (5)
C(24)—C(31)—C(36)	119.6 (5)	C(24′)—C(31′)—C(36′)	122.1 (5)
C(24)—C(31)—C(32)	122.0 (5)	C(24′)—C(31′)—C(32′)	120.0 (5)
C(32)—C(31)—C(36)	118.1 (5)	C(32′)—C(31′)—C(36′)	117.9 (5)
C(31)—C(32)—C(33)	121.1 (5)	C(31′)—C(32′)—C(33′)	121.0 (5)
C(32)—C(33)—C(34)	119.7 (5)	C(32′)—C(33′)—C(34′)	120.0 (5)
C(33)—C(34)—C(35)	119.4 (6)	C(33′)—C(34′)—C(35′)	119.3 (5)
C(34)—C(35)—C(36)	120.4 (6)	C(34′)—C(24′)—C(36′)	120.6 (5)
C(31)—C(36)—C(35)	121.3 (6)	C(31′)—C(36′)—C(35′)	121.2 (5)
C(24)—C(37)—C(42)	117.7 (5)	C(24′)—C(37′)—C(42′)	119.4 (5)
C(24)—C(37)—C(38)	122.8 (5)	C(24′)—C(37′)—C(38′)	122.1 (5)
C(38)—C(37)—C(42)	119.4 (5)	C(38′)—C(37′)—C(42′)	118.1 (5)
C(37)—C(38)—C(39)	119.9 (5)	C(37′)—C(38′)—C(39′)	120.8 (5)
C(38)—C(39)—C(40)	120.1 (5)	C(38′)—C(39′)—C(40′)	120.2 (5)
C(39)—C(40)—C(41)	120.4 (5)	C(39′)—C(40′)—C(41′)	119.9 (5)
C(40)—C(41)—C(42)	118.9 (5)	C(40′)—C(41′)—C(42′)	119.6 (5)
C(37)—C(42)—C(41)	121.2 (5)	C(37′)—C(42′)—C(41′)	121.5 (5)
C(43)—S(44)—C(45)	100.1 (3)	C(43′)—S(44′)—C(45′)	100.8 (3)
S(44)—C(45)—C(46)	115.0 (4)	S(44′)—C(45′)—C(46′)	113.7 (4)
C(45)—C(46)—C(47)	111.3 (5)	C(45′)—C(46′)—C(47′)	112.2 (4)
C(46)—C(47)—C(52)	112.3 (4)	C(46′)—C(47′)—C(52′)	113.1 (4)
C(46)—C(47)—N(48)	112.5 (4)	C(46′)—C(47′)—N(48′)	108.3 (4)
N(48)—C(47)—C(52)	112.6 (4)	N(48′)—C(47′)—C(52′)	113.7 (5)
C(47)—N(48)—C(49)	123.6 (4)	C(47′)—C(48′)—C(49′)	120.5 (4)
C(48)—C(49)—C(51)	118.3 (5)	N(48′)—C(49′)—C(51′)	116.4 (5)
N(48)—C(49)—O(50)	120.3 (5)	N(48′)—C(49′)—O(50′)	121.6 (5)

Table 2 (*cont.*)

O(50)—C(49)—C(51)	121.4 (5)	O(50′)—C(49′)—C(51′)	122.1 (5)
C(47)—C(52)—O(54)	110.3 (4)	C(47′)—C(52′)—O(54′)	114.2 (5)
C(47)—C(52)—C(53)	124.9 (5)	C(47′)—C(52′)—O(53′)	121.2 (5)
O(53)—C(52)—O(54)	124.6 (5)	O(53′)—C(52′)—O(54′)	124.6 (5)
C(52)—O(54)—C(55)	115.2 (4)	C(52′)—O(54′)—C(55′)	114.6 (4)
O(54)—C(55)—C(56)	106.7 (5)	O(54′)—C(55′)—C(56′)	108.2 (5)

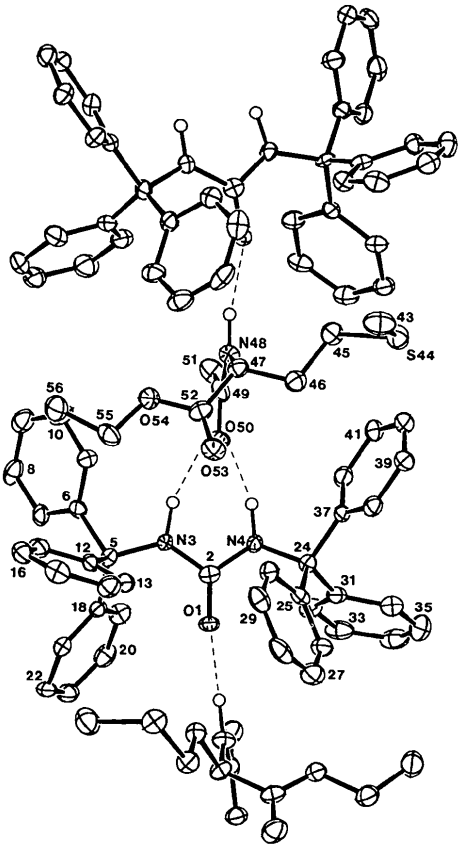

Fig. 1. Molecular structure of the asymmetric unit, showing the atom-labelling pattern and 50% probability ellipsoids. The hydrogen-bonding interactions in this structure are: NH(3)···O(50) = 3.064 (5), NH(4)···O(50) = 2.929 (5), NH(48)···O(1′) = 2.962 (5), NH(48′)···O(1) = 2.970 (5) [denoted by broken lines], NH(3′)···O(50′) = 2.907 (5) and NH(4′)···O(50′) = 2.957 (5) Å.

Goldberg & Toda, 1986), and of a series of diarylurea compounds (Etter, Urbanczyk-Lipkowska, Zia-Ebrahimi & Panunto, 1990), in closely related host–guest systems have previously been discussed; present data are perfectly consistent with the results reported therein.

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Structure of [20-CH_3],[20-CD_3]-Methylpregnene-3,20-diol Methanolate from Neutron Diffraction at 123 K

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Abstract. $C_{22}H_{33}D_3O_2.CH_3OH$, $M_r = 367.59$, monoclinic, $P2_1$, $a = 13.345\ (3)$, $b = 7.729\ (2)$, $c = 10.471\ (2)$ Å, $\beta = 101.18\ (2)°$, $V = 1059.5\ (4)$ Å^3, $Z = 2$, $D_n = 1.152$ g cm^{-3}, λ_n(Be,002) = 1.0508 Å, $\mu_n = 2.794$ cm^{-1}, $F(000) = 51.93$ fm, $T = 123$ K, $R(F^2) = 0.0390$, $wR(F^2) = 0.0408$, $S = 1.32$ for 3826 unique reflections and 601 variable parameters, including D/H scattering lengths at six sites. The statistical distribution of D between two methyl C20 groups, derived from b_{obs} (e.s.d.'s < 2%), shows the chirality at C20 to be 84.0% *S*, 16.0% *R* within ±0.6 and ±1.0% as estimated from respective averages of three values in each methyl group. The C—H bond lengths of tetrahedral C atoms (uncorrected for thermal motion) have average values and σ values from observed distributions of 1.098 (4) Å for 16 methylene C—H bonds, 1.090 (4) Å for 12 methyl C—H(D) bonds, and 1.105 (6) Å for five methine C—H bonds. The average H(D)—C—H(D) angles are 106.5 (4) and 107.8 (10)° for eight methylene and 15 methyl angles, respectively. The average e.s.d.'s are 0.003 (C—H and C—D) and 0.002 Å (C—C and C—O) for bond lengths and 0.3 [H(D)—C—H(D)], 0.2 [C—C—H(D)] and 0.09° (C—C—C) for bond angles. The H···O hydrogen-bond lengths and O—H···O angles are 1.693 (3) Å, 175.7 (3)°; 1.756 (3) Å, 171.6 (3)°; 1.771 (3) Å, 171.7 (3)°. Together, the three O—H···O bonds form an infinite helical chain about the 2_1 axis at $\frac{1}{2}$, 0. There are five distinct intermolecular H···H distances less than 2.3 Å, one being exceptionally short at 2.020 Å.

Experimental. Deuterated 20-methylpregnenediol was synthesized by the Grignard addition of a deuterated methyl group to pregnenolone and crystallized by slow cooling from a methanol–chloroform solution. The diffraction data from a crystal of dimensions 3.5 × 0.8 × 1.4 mm, 3.9 mm^3, were measured with the four-circle diffractometer at port H6M of the Brookhaven High Flux Beam Reactor. The neutron beam monochromated by reflection from Be(002) planes was of wavelength 1.0504 (1) Å as determined by calibration with a KBr crystal ($a_o = 6.6000$ Å at 295 K). The crystal was maintained at 123.0 (5) K inside a double-stage DISPLEX® helium cryostat. Lattice parameters were determined by a least-squares fit of $\sin^2\theta$ values for 29 reflections within the range $42 < 2\theta < 51°$. 5023 reflections [h, $-k$, $\pm l$; $h \le 20$, $|k| \le 10$, $|l| \le 15$; $\sin\theta/\lambda \le 0.77$ Å^{-1}] were measured by the $\theta/2\theta$ step-scan

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