

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

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Abstract. $C_{39}H_{32}N_2O.C_7H_{13}NO_3$, $M_r = 703.88$, orthorhombic, $P2_12_12_1$, $a = 9.730$ (4), $b = 18.714$ (6), $c = 21.362$ (6) Å, $V = 3889.8$ Å³, $Z = 4$, $D_x = 1.202$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.7107$ Å, $\mu = 0.72$ cm⁻¹, $F(000) = 1496$, $T = 128$ K, final $R = 0.047$ and $wR = 0.046$ for 3305 observed unique reflections. The results illustrate that functional coplanar amide groups of the host and guest complement each other excellently 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 $0.5 \times 0.2 \times 0.2$ mm, cell dimensions from setting angles of 15 reflections in the range $9 < \theta < 13^\circ$, space group by systematic absences. Data measured at low temperature on an upgraded Picker diffractometer with graphite-monochromated Mo $K\alpha$ radiation, $\theta_{\text{max}} = 27^\circ$ ($\sin\theta/\lambda < 0.64$ Å⁻¹), ω - 2θ scans, constant scan speed 3° min^{-1} , $h = 0$ to 12, $k = 0$ to 23, $l = 0$ to 27. Three reflections monitored periodically during data collection indicated no crystal deterioration. 4336 unique measured reflections (out of a total of 4763 reflections measured) of which 3305 with $I > 3\sigma(I)$ used in refinement. No corrections for absorption or secondary extinction.

The structure was solved by direct methods (*MULTAN80*; Main *et al.*, 1980), and refined by least squares (*SHELX76*; Sheldrick, 1976) 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 at calculated positions, the methyls being treated as rigid groups. Minimization of $w(\Delta F)^2$, with $w = 2.16/[\sigma^2(F) + 0.002F^2]$, converged at $R = 0.047$, $wR = 0.046$, $S = 2.06$; 487 parameters refined. Final $\Delta/\sigma < 0.3$, residual densities in Fourier maps ranging from -0.08 to 0.11 e Å⁻³. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV).

Table 1. Atomic coordinates and equivalent isotropic displacement coefficients (Å²)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
O(1)	0.9867 (2)	0.0676 (1)	0.7695 (1)	0.0209 (7)
C(2)	1.0205 (3)	0.1305 (2)	0.7620 (1)	0.0181 (10)
N(3)	1.0277 (3)	0.1791 (1)	0.8099 (1)	0.0177 (8)
N(4)	1.0607 (3)	0.1585 (1)	0.7056 (1)	0.0187 (8)
C(5)	0.9679 (3)	0.1641 (2)	0.8720 (1)	0.0174 (9)
C(6)	1.0561 (4)	0.1095 (2)	0.9082 (2)	0.0202 (10)
C(7)	1.1795 (4)	0.0839 (2)	0.8848 (2)	0.0221 (10)
C(8)	1.2637 (4)	0.0397 (2)	0.9212 (2)	0.0293 (12)
C(9)	1.2258 (4)	0.0222 (2)	0.9814 (2)	0.0331 (14)
C(10)	1.1038 (4)	0.0480 (2)	1.0054 (2)	0.0336 (14)
C(11)	1.0182 (4)	0.0910 (2)	0.9695 (2)	0.0284 (11)
C(12)	0.8179 (3)	0.1405 (2)	0.8617 (1)	0.0198 (11)
C(13)	0.7715 (4)	0.0712 (2)	0.8723 (2)	0.0257 (11)
C(14)	0.6380 (4)	0.0522 (2)	0.8556 (2)	0.0337 (12)
C(15)	0.5494 (4)	0.1009 (2)	0.8290 (2)	0.0360 (13)
C(16)	0.5938 (4)	0.1707 (2)	0.8190 (2)	0.0380 (13)
C(17)	0.7282 (4)	0.1902 (2)	0.8348 (2)	0.0299 (12)
C(18)	0.9787 (4)	0.2335 (2)	0.9110 (1)	0.0202 (10)
C(19)	1.1018 (4)	0.2731 (2)	0.9085 (2)	0.0223 (10)
C(20)	1.1180 (4)	0.3328 (2)	0.9473 (2)	0.0267 (12)
C(21)	1.0153 (4)	0.3520 (2)	0.9893 (2)	0.0322 (11)
C(22)	0.8956 (4)	0.3125 (2)	0.9918 (2)	0.0334 (13)
C(23)	0.8770 (4)	0.2534 (2)	0.9527 (2)	0.0272 (11)
C(24)	1.0637 (3)	0.1169 (2)	0.6475 (1)	0.0188 (10)
C(25)	0.9174 (3)	0.1038 (2)	0.6214 (2)	0.0192 (9)
C(26)	0.8012 (4)	0.1290 (2)	0.6524 (2)	0.0265 (12)
C(27)	0.6697 (4)	0.1188 (2)	0.6273 (2)	0.0329 (13)
C(28)	0.6538 (4)	0.0822 (2)	0.5716 (2)	0.0360 (13)
C(29)	0.7680 (4)	0.0586 (2)	0.5390 (2)	0.0323 (12)
C(30)	0.9009 (4)	0.0704 (2)	0.5632 (2)	0.0228 (11)
C(31)	1.1454 (3)	0.0475 (2)	0.6604 (1)	0.0195 (10)
C(32)	1.1029 (4)	-0.0209 (2)	0.6418 (2)	0.0219 (10)
C(33)	1.1842 (4)	-0.0807 (2)	0.6540 (2)	0.0268 (12)
C(34)	1.3075 (4)	-0.0737 (2)	0.6853 (2)	0.0346 (12)
C(35)	1.3519 (4)	-0.0059 (2)	0.7039 (2)	0.0349 (13)
C(36)	1.2716 (4)	0.0537 (2)	0.6915 (2)	0.0249 (11)
C(37)	1.1395 (3)	0.1640 (2)	0.5988 (1)	0.0198 (10)
C(38)	1.0895 (4)	0.2333 (2)	0.5877 (2)	0.0240 (11)
C(39)	1.1536 (4)	0.2776 (2)	0.5446 (2)	0.0279 (11)
C(40)	1.2652 (4)	0.2529 (2)	0.5100 (2)	0.0320 (13)
C(41)	1.3130 (4)	0.1838 (2)	0.5200 (2)	0.0318 (13)
C(42)	1.2511 (4)	0.1405 (2)	0.5650 (2)	0.0264 (10)
C(43)	1.1069 (4)	-0.0941 (2)	0.8311 (2)	0.0379 (13)
C(44)	0.9998 (4)	-0.1270 (2)	0.7890 (2)	0.0250 (11)
O(45)	0.9979 (3)	-0.1919 (1)	0.7772 (1)	0.0321 (8)
N(46)	0.9046 (3)	-0.0826 (1)	0.7663 (1)	0.0235 (9)
C(47)	0.8028 (4)	-0.1105 (2)	0.7225 (2)	0.0245 (10)
C(48)	0.7190 (4)	-0.0498 (2)	0.6945 (2)	0.0355 (13)
C(49)	0.7082 (4)	-0.1655 (2)	0.7527 (2)	0.0274 (10)
O(50)	0.6747 (3)	-0.2200 (2)	0.7272 (1)	0.0487 (11)
O(51)	0.6606 (3)	-0.1443 (1)	0.8085 (1)	0.0314 (8)
C(52)	0.5642 (4)	-0.1926 (2)	0.8386 (2)	0.0370 (14)
C(53)	0.4981 (5)	-0.1518 (2)	0.8918 (2)	0.0458 (14)

Atomic parameters are listed in Table 1; bond distances and bond angles (*PARST*; Nardelli, 1983) are presented in Table 2. The molecular structure

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

O(1)—C(2)	1.233 (4)	C(25)—C(26)	1.393 (5)
C(2)—N(3)	1.371 (3)	C(25)—C(30)	1.401 (5)
C(2)—N(4)	1.371 (3)	C(26)—C(27)	1.400 (5)
N(3)—C(5)	1.476 (3)	C(27)—C(28)	1.382 (5)
N(4)—C(24)	1.465 (3)	C(28)—C(29)	1.384 (5)
C(5)—C(6)	1.542 (5)	C(29)—C(30)	1.410 (5)
C(5)—C(12)	1.541 (4)	C(31)—C(32)	1.403 (5)
C(5)—C(18)	1.547 (4)	C(31)—C(36)	1.401 (4)
C(6)—C(7)	1.386 (5)	C(32)—C(33)	1.395 (5)
C(6)—C(11)	1.404 (6)	C(33)—C(34)	1.380 (5)
C(7)—C(8)	1.400 (5)	C(34)—C(35)	1.398 (5)
C(8)—C(9)	1.377 (6)	C(35)—C(36)	1.387 (5)
C(9)—C(10)	1.380 (5)	C(37)—C(38)	1.405 (5)
C(10)—C(11)	1.389 (5)	C(37)—C(42)	1.376 (4)
C(12)—C(13)	1.392 (5)	C(38)—C(39)	1.387 (5)
C(12)—C(17)	1.399 (5)	C(39)—C(40)	1.393 (5)
C(13)—C(14)	1.393 (5)	C(40)—C(41)	1.391 (5)
C(14)—C(15)	1.377 (5)	C(41)—C(42)	1.394 (5)
C(15)—C(16)	1.392 (5)	C(43)—C(44)	1.508 (5)
C(16)—C(17)	1.399 (5)	C(44)—O(45)	1.241 (4)
C(18)—C(19)	1.409 (5)	C(44)—N(46)	1.336 (4)
C(18)—C(23)	1.383 (5)	N(46)—C(47)	1.459 (4)
C(19)—C(20)	1.400 (5)	C(47)—C(48)	1.521 (5)
C(20)—C(21)	1.390 (5)	C(47)—C(49)	1.524 (5)
C(21)—C(22)	1.380 (5)	C(49)—O(50)	1.201 (5)
C(22)—C(23)	1.398 (5)	C(49)—O(51)	1.339 (4)
C(24)—C(25)	1.548 (4)	O(51)—C(52)	1.453 (4)
C(24)—C(31)	1.547 (5)	C(52)—C(53)	1.513 (5)
C(24)—C(37)	1.550 (4)		
O(1)—C(2)—N(4)	123.7 (3)	C(25)—C(24)—C(37)	106.6 (2)
O(1)—C(2)—N(3)	123.4 (3)	C(25)—C(24)—C(31)	113.8 (3)
N(3)—C(2)—N(4)	112.8 (3)	C(24)—C(25)—C(30)	119.7 (3)
C(2)—N(3)—C(5)	121.6 (2)	C(24)—C(25)—C(26)	121.4 (3)
C(2)—N(4)—C(24)	123.2 (3)	C(26)—C(25)—C(30)	118.7 (3)
N(3)—C(5)—C(18)	107.3 (2)	C(25)—C(26)—C(27)	120.9 (4)
N(3)—C(5)—C(12)	107.4 (2)	C(26)—C(27)—C(28)	120.0 (4)
N(3)—C(5)—C(6)	110.9 (2)	C(27)—C(28)—C(29)	120.1 (4)
C(12)—C(5)—C(18)	112.5 (3)	C(28)—C(29)—C(30)	120.1 (4)
C(6)—C(5)—C(18)	104.4 (2)	C(25)—C(30)—C(29)	120.0 (4)
C(6)—C(5)—C(12)	114.1 (3)	C(24)—C(31)—C(36)	117.7 (3)
C(5)—C(6)—C(11)	119.0 (3)	C(24)—C(31)—C(32)	124.3 (3)
C(5)—C(6)—C(7)	122.0 (3)	C(32)—C(31)—C(36)	117.9 (4)
C(7)—C(6)—C(11)	118.6 (4)	C(31)—C(32)—C(33)	120.8 (4)
C(6)—C(7)—C(8)	120.7 (4)	C(32)—C(33)—C(34)	120.5 (4)
C(7)—C(8)—C(9)	120.2 (4)	C(33)—C(34)—C(35)	119.5 (4)
C(8)—C(9)—C(10)	119.6 (4)	C(34)—C(35)—C(36)	120.1 (4)
C(9)—C(10)—C(11)	120.9 (4)	C(31)—C(36)—C(35)	121.2 (4)
C(6)—C(11)—C(10)	120.0 (4)	C(24)—C(37)—C(42)	123.1 (3)
C(5)—C(12)—C(17)	117.3 (3)	C(24)—C(37)—C(38)	118.2 (3)
C(5)—C(12)—C(13)	123.4 (3)	C(38)—C(37)—C(42)	118.7 (3)
C(13)—C(12)—C(17)	118.9 (3)	C(37)—C(38)—C(39)	120.5 (3)
C(12)—C(13)—C(14)	114.1 (3)	C(38)—C(39)—C(40)	120.3 (4)
C(13)—C(14)—C(15)	121.4 (4)	C(39)—C(40)—C(41)	119.2 (4)
C(14)—C(15)—C(16)	119.3 (4)	C(40)—C(41)—C(42)	120.1 (4)
C(15)—C(16)—C(17)	119.9 (4)	C(37)—C(42)—C(41)	121.1 (4)
C(12)—C(17)—C(16)	120.6 (3)	C(43)—C(44)—N(46)	116.2 (3)
C(5)—C(18)—C(23)	121.6 (3)	C(43)—C(44)—O(45)	122.1 (4)
C(5)—C(18)—C(19)	118.6 (3)	O(45)—C(44)—N(46)	121.7 (4)
C(19)—C(18)—C(23)	119.4 (3)	C(44)—N(46)—C(47)	118.8 (3)
C(18)—C(19)—C(20)	119.5 (3)	N(46)—C(47)—C(49)	112.3 (3)
C(19)—C(20)—C(21)	120.5 (4)	N(46)—C(47)—C(48)	110.4 (3)
C(20)—C(21)—C(22)	119.5 (4)	C(48)—C(47)—C(49)	110.3 (3)
C(21)—C(22)—C(23)	120.7 (4)	C(47)—C(49)—O(50)	112.7 (3)
C(18)—C(23)—C(22)	120.4 (4)	C(47)—C(49)—O(51)	123.0 (4)
N(4)—C(24)—C(37)	106.0 (2)	O(50)—C(49)—O(51)	124.2 (4)
N(4)—C(24)—C(31)	107.8 (2)	C(49)—O(51)—C(52)	115.7 (3)
N(4)—C(24)—C(25)	111.8 (2)	O(51)—C(52)—C(53)	107.0 (3)
C(31)—C(24)—C(37)	110.6 (3)		

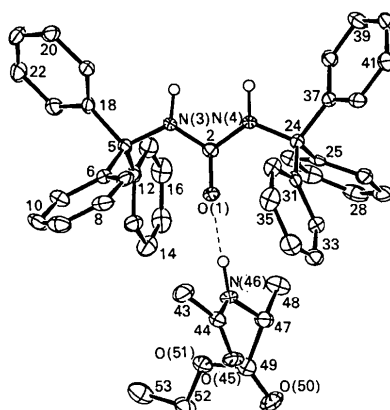


Fig. 1. Molecular structure of the title compound, showing the atom labelling and 50%-probability thermal ellipsoids. H atoms are of arbitrary size. The hydrogen-bonding interactions in this structure are: NH(3)⋯O(45) = 2.881 (3), NH(4)⋯O(45) = 3.058 (3) and NH(46)⋯O(1) = 2.923 (3) Å.

(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, 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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References

- ETTER, M. R., URBANCZYK-LIPKOWSKA, Z., ZIA-EBRAHIMI, M. & PANUNTO, T. W. (1990). *J. Am. Chem. Soc.* **112**, 8415–8426.
- GOLDBERG, I. (1988). *Top. Curr. Chem.* **149**, 1–44.
- GOLDBERG, I. (1991). In *Inclusion Compounds*, Vol. 5, edited by J. L. ATWOOD, J. E. D. DAVIES & D. D. MACNICOL. Oxford Univ. Press.
- GOLDBERG, I., LIN, L.-T. W. & HART, H. (1985). *J. Inclusion Phenom. Mol. Recognit. Chem.* **2**, 377–389.
- HART, H., LIN, L.-T. W. & GOLDBERG, I. (1986). *Mol. Cryst. Liq. Cryst.* **137**, 277–286.
- HART, H., LIN, L.-T. W., NG, K.-K. D., WARD, D. L., GOLDBERG, I. & TODA, F. (1986). Presented at the 4th International Symposium on Molecular Inclusion Phenomena, Lancaster, England.
- JOHNSON, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
- NARDELLI, M. (1983). *Comput. Chem.* **7**, 95–98.
- SHELDRIK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

and atom-labelling scheme are shown in Fig. 1 (*ORTEPII*; Johnson, 1976).*

Related literature. Structural features of the title compound have already been described elsewhere

* 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 54846 (16 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.