

– the valence angle C(10)–N(15)–N(14) is 8.4° greater than N(15)–N(14)–C(13) (Fig. 1). The replacement of the lone pair on N(15) by an N–H⁺ bond results in a diminution of repulsions and an opening of the angle C(10)–N(15)–N(14), according to the Gillespie (1972) theory.

The morpholino ring is in the chair form and the side-chain methylene groups 7 and 8 are in *synclinal* conformation, with N(4)–C(7)–C(8)–N(9): $-80.7(4)^\circ$.

Crystal cohesion is assumed by a dense network of intermolecular hydrogen bonds between two oxalate moieties, the protonated N(4) and N(15) and the exocyclic N(9) atoms of the 5-methyl analog. The hydrogen bonds are constituted by the alternation of oxalate ions ('55') and oxalic acid molecules ('66' and '77'), to form a zigzag chain (Figs. 2 and 3). The dihedral angles between the least-squares mean planes through the heavy atoms of the ion '55' and the molecules '66' and '77' are 63.3 and 74.1° respectively. The dihedral angle between planes of '66' and '77' is 16.3° . The terminal ions are linked to two 5-methyl analog molecules *via* four different N–H...O bonds, with the N(4), N(9) and N(15) atoms. N(4) participates in a bifurcated hydrogen bond: N(4)–H...O(551): $2.831(5)$ Å and N(4)–H...O(556): $2.906(6)$ Å (Fig. 3). Such a situation has been described for the dimethylammonium hydrogen oxalate (Thomas & Pramatus, 1975). As shown in Fig. 1, there are differences in the internal geometry of the oxalic acid molecule and oxalate anion. This can be explained by the sensitive response of the geometry to changes in the surrounding H atoms: the molecules '66' and '77' are planar, experimental errors excepted; the anion '55' is

slightly distorted (dihedral angle between COO[−] planes: 2.43°).

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Structure of the *N*-Tritylalanine Anhydride:Diethyl Ether (1:1) Inclusion Complex

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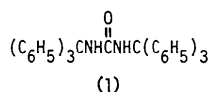
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Abstract. C₄₄H₄₀N₂O₃·C₄H₁₀O, *M_r* = 718.9, monoclinic, *P*2₁, *a* = 9.092 (3), *b* = 24.809 (4), *c* = 9.135 (3) Å, β = 102.60 (2)°, *V* = 2010.9 (9) Å³, *Z* = 2, *D_x* = 1.187 g cm^{−3}, λ(Mo Kα) = 0.7107 Å, μ = 0.70 cm^{−1}, *F*(000) = 768, room temperature. *R* = 0.090 for 1663 observed reflections. The observed

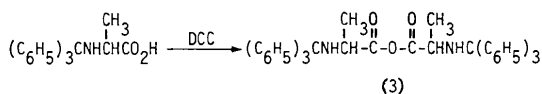
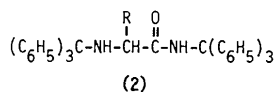
structure represents a crystalline inclusion complex of the channel type. The host lattice consists of loosely packed layers of *N*-tritylalanine anhydride molecules, and has a higher symmetry than the entire crystal. The Et₂O guest species are included between adjacent layers of the hosts. In view of the noncomplementary steric fit

and the absence of specific interactions between host and guest, the latter is partially disordered in the crystal structure.

Introduction. In a recent communication (Hart, Lin & Ward, 1984) we proposed that compounds with a long molecular axis holding large groups at each end might function as hosts in crystalline host-guest complexes. One particularly versatile host of this type is *N,N'*-ditritylurea (1) (Goldberg, Lin & Hart, 1984; Hart, Lin & Ward, 1985), which forms molecular complexes with a variety of amines, ethers, amides, alcohols, esters and small molecules with other functionality. The trityl (triphenylmethyl) group plays a significant role in these complexes, acting as a 'spacer' which tends to prevent close packing of the host molecules, thus creating substantial voids in the crystal that become occupied by the guest molecules.



During efforts to extend these ideas to *N,N'*-ditrityl derivatives of amino-acid amides (2) (Hart & Lin, unpublished results) we accidentally prepared (3), the symmetric anhydride of *N*-trityl-L-alanine. Treatment of a mixture of *N*-trityl-L-alanine (Zervas & Theodoropoulos, 1956; Kricheldorf, 1970) and *N*-tritylamine with dicyclohexylcarbodiimide (DCC) gave the symmetric anhydride (3) instead of the desired (2) ($R = \text{CH}_3$). The same anhydride was obtained in good yield when the tritylamine was omitted. Such symmetric anhydrides are well known intermediates in certain peptide syntheses (Bodanszky, Klausner & Ondetti, 1976; Chen, Kuroda & Benoiton, 1978; Wieland, Birr & Flor, 1971; Rebek & Feitler, 1974). We found that (3) forms a stable crystalline complex (1:1) with diethyl ether, and report here the structure of this complex.



Experimental. The anhydride (3) and its ether complex were prepared as follows. A solution of *N*-trityl-L-alanine (800 mg, 2.4 mmol) and DCC (600 mg, 3 mmol) in 50 ml of anhydrous methylene chloride was stirred at room temperature overnight. The solvent was removed under vacuum to give a solid which was chromatographed over silica gel with ethyl acetate:petroleum ether (1:1) as eluent, to give 660 mg (86%) of (3), m.p. 448 K (dec.). ¹H NMR (250 MHz, CDCl₃)

δ 1.20 (*d*, $J = 6$ Hz, 6 H, CH₃ groups), 2.53 (*d*, $J = 11$ Hz, 2 H, NH groups), 3.18 (*m*, $J = 6, 11$ Hz, CH groups), 7.15–7.51 (*m*, 30 H, aryl protons); ¹³C NMR (62.9 MHz, CDCl₃) δ 20.84, 53.48, 71.81, 127.70, 128.72, 129.11, 145.50, 171.34; IR (KBr) 3300 (*m*), 3020 (*w*), 2965 (*m*), 2920 (*w*), 2850 (*w*), 1795 (*s*), 1735 (*s*), 1590 cm⁻¹ (*w*). Recrystallization of (3) from diethyl ether gave the 1:1 complex, m.p. 456–457 K.

For the crystallographic analysis a crystal of size 0.1 × 0.2 × 0.4 mm was mounted within a thin capillary to prevent possible deterioration. Diffraction data out to $2\theta_{\text{max}} = 46^\circ$ measured at *ca* 291 K, Enraf-Nonius CAD-4 diffractometer, graphite monochromator, Mo *K* α radiation, ω - 2θ scans, scan range $1.2 + 0.3 \tan \theta^\circ$, varying scan rate $1-4^\circ \text{ min}^{-1}$. Unit-cell parameters determined by least squares from 2θ values of 25 reflections with $9.5 < \theta < 12^\circ$. 3213 reflections measured, range of *hkl*: $0 \leq h \leq 10$, $0 \leq k \leq 29$, $-10 \leq l \leq 10$, 2389 unique ($R_{\text{int}} = 0.035$) with a positive intensity. The crystal was stable during the measurements. Intensity data corrected for Lorentz and polarization effects, but not for absorption or secondary extinction. Final refinement calculations based on 1663 reflections with $F_o^2 > 2\sigma(F_o^2)$; atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

Crystal structure solved by a combination of direct methods and Fourier techniques, refined by least squares. During the refinement procedure it became apparent that the Et₂O guest is partially disordered within the host lattice. This was reflected in the relatively large thermal parameters obtained for the guest's atoms as well as in a distorted geometry of the molecule. Since we were not able to construct a geometrically disordered model for Et₂O which would be more consistent with the diffraction data, it has been assumed that the disorder is dynamical in nature; the refined atomic coordinates thus represent only thermally averaged positions of the corresponding disordered atoms. In order to increase the data-to-parameters ratio during the refinement, the six phenyl groups of the host were introduced into the calculations as ideal hexagons (side 1.395 Å), and subsequently treated as geometrically constrained rigid groups. The total number of refined parameters, including anisotropic thermal parameters for all the non-H atoms, was 419. The structure-factor calculations included contributions from all H atoms of the host molecule as well, which were located in theoretical positions and assigned fixed isotropic temperature factors. Least-squares computations were based on unit weights and minimized $w(\Delta F)^2$; adequacy of the weighting scheme was indicated by a nearly uniform distribution of the variance of the mean $w(\Delta F)^2$ with F_o and $\sin \theta / \lambda$. Final $R = 0.090$, $wR = 0.090$ for 1663 observations above intensity threshold. $(\Delta/\sigma)_{\text{max}} = 0.52$. Final difference map

showed no indication of incorrectly placed or missing atoms; $\Delta\rho_{\max}$ and $\Delta\rho_{\min}$ 0.32 and -0.29 e \AA^{-3} respectively. Computations and illustrations performed with the following programs: *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), an extensively modified version of *ORFLS* (Busing, Martin & Levy, 1962), *SHELX76* (Sheldrick, 1976), *PARST* (Nardelli, 1983), *ORTEPII* (Johnson, 1976).^{*} All calculations performed on CYBER 170-855 at Tel-Aviv University Computation Centre.

* Lists of structure factors, atomic parameters of the H atoms and anisotropic thermal parameters of the non-H atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42317 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Atomic coordinates and equivalent isotropic thermal parameters

U_{eq} is one third of the trace of the orthogonalized U_{ij} matrix. Atoms of the guest are C(50) through C(54).

	x	y	z	$U_{eq}(\text{\AA}^2)$
C(1)	0.7535 (15)	0.3852 (6)	0.5436 (14)	0.0324
C(2)	0.8296 (9)	0.3432 (5)	0.6522 (12)	0.0432
C(3)	0.7492 (9)	0.2986 (5)	0.6853 (12)	0.0511
C(4)	0.8240 (9)	0.2573 (5)	0.7755 (12)	0.0822
C(5)	0.9791 (9)	0.2606 (5)	0.8326 (12)	0.0779
C(6)	1.0594 (9)	0.3052 (5)	0.7995 (12)	0.0486
C(7)	0.9847 (9)	0.3465 (5)	0.7093 (12)	0.0419
C(8)	0.8183 (9)	0.3785 (4)	0.3968 (11)	0.0400
C(9)	0.7531 (9)	0.3396 (4)	0.2923 (11)	0.0528
C(10)	0.8120 (9)	0.3302 (4)	0.1658 (11)	0.0654
C(11)	0.9362 (9)	0.3596 (4)	0.1439 (11)	0.0713
C(12)	1.0014 (9)	0.3985 (4)	0.2484 (11)	0.0625
C(13)	0.9424 (9)	0.4079 (4)	0.3748 (11)	0.0457
C(14)	0.7782 (10)	0.4441 (4)	0.6039 (9)	0.0363
C(15)	0.8196 (10)	0.4539 (4)	0.7578 (9)	0.0368
C(16)	0.8272 (10)	0.5067 (4)	0.8114 (9)	0.0505
C(17)	0.7934 (10)	0.5497 (4)	0.7112 (9)	0.0490
C(18)	0.7520 (10)	0.5398 (4)	0.5573 (9)	0.0671
C(19)	0.7444 (10)	0.4870 (4)	0.5037 (9)	0.0472
N(20)	0.5886 (12)	0.3750 (5)	0.4948 (13)	0.0379
C(21)	0.5014 (16)	0.3955 (6)	0.6052 (17)	0.0417
C(22)	0.3694 (20)	0.3587 (8)	0.6039 (24)	0.0807
C(23)	0.4321 (16)	0.4495 (7)	0.5624 (20)	0.0518
O(24)	0.3755 (13)	0.4640 (5)	0.4363 (12)	0.0662
O(25)	0.4298 (12)	0.4803 (5)	0.6846 (11)	0.0584
C(26)	0.3028 (20)	0.5099 (8)	0.6900 (18)	0.0590
O(27)	0.1817 (13)	0.4949 (5)	0.6359 (13)	0.0656
C(28)	0.3407 (18)	0.5671 (6)	0.7538 (16)	0.0485
C(29)	0.3398 (22)	0.6057 (7)	0.6197 (17)	0.0654
N(30)	0.2328 (14)	0.5842 (5)	0.8421 (13)	0.0476
C(31)	0.2771 (16)	0.5722 (6)	1.0069 (14)	0.0379
C(32)	0.3470 (8)	0.5157 (4)	1.0336 (10)	0.0405
C(33)	0.2473 (8)	0.4723 (4)	1.0060 (10)	0.0477
C(34)	0.3027 (8)	0.4197 (4)	1.0156 (10)	0.0584
C(35)	0.4577 (8)	0.4105 (4)	1.0528 (10)	0.0603
C(36)	0.5574 (8)	0.4539 (4)	1.0804 (10)	0.0651
C(37)	0.5021 (8)	0.5065 (4)	1.0708 (10)	0.0471
C(38)	0.3896 (11)	0.6182 (4)	1.0876 (10)	0.0412
C(39)	0.4544 (11)	0.6135 (4)	1.2402 (10)	0.0448
C(40)	0.5446 (11)	0.6549 (4)	1.3146 (10)	0.0541
C(41)	0.5701 (11)	0.7010 (4)	1.2364 (10)	0.0572
C(42)	0.5053 (11)	0.7057 (4)	1.0837 (10)	0.0769
C(43)	0.4151 (11)	0.6643 (4)	1.0094 (10)	0.0584
C(44)	0.1415 (10)	0.5806 (4)	1.0761 (10)	0.0408
C(45)	0.1282 (10)	0.5518 (4)	1.2038 (10)	0.0482
C(46)	0.0013 (10)	0.5587 (4)	1.2645 (10)	0.0572
C(47)	-0.1123 (10)	0.5942 (4)	1.1974 (10)	0.0644
C(48)	-0.0991 (10)	0.6230 (4)	1.0697 (10)	0.0628
C(49)	0.0278 (10)	0.6162 (4)	1.0090 (10)	0.0452
C(50)	0.3472 (32)	0.2605 (13)	0.2507 (30)	0.1537
C(51)	0.2078 (37)	0.2449 (15)	0.2598 (40)	0.2140
O(52)	0.2126 (24)	0.2088 (10)	0.3594 (25)	0.1839
C(53)	0.0860 (38)	0.2248 (17)	0.3785 (47)	0.2239
C(54)	-0.0091 (39)	0.1954 (13)	0.4505 (36)	0.1677

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

C(1)-C(2)	1.50 (2)	O(25)-C(26)	1.38 (2)
C(1)-C(8)	1.59 (2)	C(26)-O(27)	1.17 (2)
C(1)-C(14)	1.56 (2)	C(26)-C(28)	1.54 (2)
C(1)-N(20)	1.49 (2)	C(28)-C(29)	1.55 (2)
N(20)-C(21)	1.50 (2)	C(28)-N(30)	1.46 (2)
C(21)-C(22)	1.51 (2)	N(30)-C(31)	1.50 (2)
C(21)-C(23)	1.50 (2)	C(31)-C(32)	1.54 (2)
C(23)-O(24)	1.21 (2)	C(31)-C(38)	1.60 (2)
C(23)-O(25)	1.36 (2)	C(31)-C(44)	1.52 (2)
C(2)-C(1)-C(8)	106 (1)	O(25)-C(26)-O(27)	122 (2)
C(2)-C(1)-C(14)	114 (1)	O(25)-C(26)-C(28)	112 (1)
C(2)-C(1)-N(20)	112 (1)	O(27)-C(26)-C(28)	124 (2)
C(8)-C(1)-C(14)	110 (1)	C(26)-C(28)-C(29)	108 (1)
C(8)-C(1)-N(20)	105 (1)	C(26)-C(28)-N(30)	111 (1)
C(14)-C(1)-N(20)	109 (1)	C(29)-C(28)-N(30)	112 (1)
C(1)-N(20)-C(21)	113 (1)	C(28)-N(30)-C(31)	116 (1)
N(20)-C(21)-C(22)	109 (1)	N(30)-C(31)-C(32)	111 (1)
N(20)-C(21)-C(23)	112 (1)	N(30)-C(31)-C(38)	109 (1)
C(22)-C(21)-C(23)	105 (1)	N(30)-C(31)-C(44)	109 (1)
C(21)-C(23)-O(24)	126 (2)	C(32)-C(31)-C(38)	112 (1)
C(21)-C(23)-O(25)	112 (1)	C(32)-C(31)-C(44)	114 (1)
O(24)-C(23)-O(25)	122 (2)	C(38)-C(31)-C(44)	102 (1)
C(23)-O(25)-C(26)	120 (1)		

Within the aromatic rings C-C bond lengths and C-C-C bond angles were constrained to 1.395 \AA and 120.0 $^\circ$ respectively.

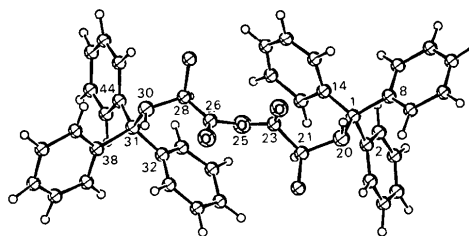


Fig. 1. Molecular structure of *N*-tritylalanine anhydride showing the atom-numbering scheme. H atoms of the methyl groups are excluded.

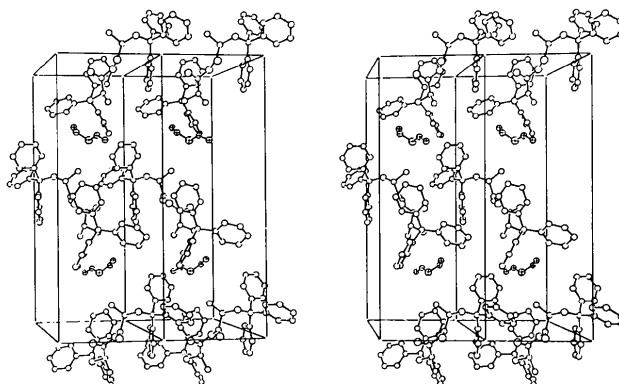


Fig. 2. Stereoview of the inclusion structure formed by *N*-tritylalanine anhydride with diethyl ether, approximately down *a*.

Discussion. Final positional parameters of the non-H atoms are given in Table 1; the intramolecular bond distances and angles are listed in Table 2. Fig. 1 illustrates the molecular structure of the host compound together with the numbering scheme used. Although the precision of the crystallographic results is relatively low, the least-squares refinement of the experimental data converged well and we believe that the structural model of the crystalline inclusion is correct. The crystal structure of the 1:1 adduct between *N*-tritylalanine anhydride and diethyl ether is illustrated in Fig. 2.

The inclusion-type structure can be best described as consisting of layers of host molecules interspaced by pseudo-channels occupied by the guest species. The channel axis is parallel to **c**. There is no structural evidence for any significant interaction between host and guest other than through dispersion forces, all intermolecular distances between non-H atoms being larger than 3.5 Å. The host molecules tend to align along their long molecular axis in *ac* planes of the crystal. Packing of these layers along **b** is inefficient due to the steric incompatibility of the large trityl end groups. This is associated with the formation of voids in the crystal, and consequently with the inclusion of smaller guest species. The above results confirm previous indications (Hart *et al.*, 1984) that the trityl groups, even when attached to a relatively flexible aliphatic fragment of atoms, can act as spacers to prevent close packing of the molecules which then become suitable hosts for crystalline inclusion of various guests. The present structure represents lattice inclusion which is not assisted by any specific coordination between host and guest (lack of complementary steric and functional relationships between the two components is probably the cause of the guest disorder in the crystal), as that observed in the inclusion complexes of *N,N'*-ditritylurea (Goldberg *et al.*, 1984).

Interestingly, the structure of the host lattice itself is characterized by a higher symmetry than that of the entire crystal. The packing arrangement of the *N*-tritylalanine anhydride molecules is close to an orthorhombic *B*-centered lattice with a unit cell of $a = 11.396$, $b = 24.809$, and $c = 14.225$ Å. Their molecular framework shows an approximate C_2 symmetry with the two carbonyl groups turning toward the same side of the host. Other details of the molecular conformation include torsion angles along the central aliphatic chain and dihedral angles between the three phenyl rings in each trityl group. The torsions are: C(1)–N(20)–C(21)–C(23) = 97.9 (14); N(20)–C(21)–C(23)–O(25) = –145.5 (13); C(21)–C(23)–O(25)–C(26) = –137.3 (15); C(23)–O(25)–C(26)–C(28) = –138.0 (15); O(25)–C(26)–C(28)–N(30) = –144.9 (13); C(26)–C(28)–N(30)–C(31) = 92.3 (15)°. They represent a stretched conformation of

the central part of the molecule [from N(20) through N(30)]. The lower values of torsion angles about the N–C(Tr) bonds illustrate a significant folding at both ends of the aliphatic fragment as a result of which two of the phenyl rings converge on the carbonyl groups [Fig. 1; the relevant shortest nonbonding distances are O(24)···C(19) = 3.35 (1) Å and O(27)···C(33) = 3.32 (1) Å]. In this manner the space around the long molecular axis appears to be occupied more efficiently. The calculated angles between normals to the phenyl rings attached to the same carbon characterize the intrinsic conformation of the trityl substituent; the corresponding values are: 63.3 (3), 68.1 (3) and 84.6 (3)° in one trityl group, and 63.0 (3), 65.4 (3) and 81.6 (3)° in the other substituent.

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