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The Structures of Dimethyl *N,N'*-[1,1'-*p*-Phenylenebis(1-methylethyl)]dicarbamate (I) and Diethyl *N,N'*-[1,1'-*p*-Phenylenebis(1-methylethyl)]dicarbamate (II)

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Abstract. (I) $C_{16}H_{24}N_2O_4$, $M_r = 308\cdot4$, monoclinic, $C2/c$, $a = 22\cdot791$ (5), $b = 7\cdot735$ (2), $c = 9\cdot741$ (3) Å, $\beta = 96\cdot32$ (2)°, $V = 1706\cdot9$ (8) Å^3, $Z = 4$, $D_x = 1\cdot20$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1\cdot54184$ Å, $\mu = 6\cdot7$ cm^{-1}, $F(000) = 664$, room temperature. $R = 0\cdot041$, 1343 independent observed reflections. (II) $C_{18}H_{28}N_2O_4$, $M_r = 336\cdot4$, monoclinic, $C2/c$, $a = 18\cdot653$ (5), $b = 11\cdot063$ (4), $c = 9\cdot697$ (3) Å, $\beta = 95\cdot39$ (2)°, $V = 1992$ (1) Å^3, $Z = 4$, $D_x = 1\cdot122$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0\cdot71073$ Å, $\mu = 0\cdot739$ cm^{-1}, $F(000) = 728$, room temperature. $R = 0\cdot056$, 782 independent observed reflections. The angle between the least-squares planes for the phenyl and carbamate groups is 86·3° in (I) and 82·5 (5)° in (II). In each structure one half of the molecule is related to the other half by a crystallographic inversion center in the middle of the phenyl ring at $\frac{1}{2},0,\frac{1}{2}$ (I) and $\frac{1}{2},0,0$ (II) in the unit cell. The phenyl groups tend to stack in the ab plane nearly perpendicular to the c axis in both cases.

Experimental. Clear colorless single crystals of each type were grown by slow evaporation of a mixture of 5 g of *p*-tetramethylxylene diisocyanate (*p*-TMXDI) (Zentner, 1987) after being refluxed in 75 ml of absolute methanol or ethanol, respectively, in the presence of triethylamine which was used as a catalyst. Rectangular parallelepiped crystals with dimensions (I) 0·28 × 0·30 × 0·90 mm and (II) 0·20 × 0·30 × 0·90 mm were each mounted on a glass fiber [(I) 0·20; (II) 0·15 mm] with the needle axis nearly parallel to the φ axis of a computer-controlled Nicolet $P\bar{1}$ autodiffractometer with a normal-focus X-ray tube and 4° take-off angle. (I): 1550 independent reflections, nickel-filtered Cu $K\alpha$ radiation; 2θ in shell 1 (3·0–105·1°) at 6° min^{-1}, shell 2 (105·1–136·3°) at 3° min^{-1}; background counts (each lasting one-fourth of the total scan time) at both ends of the scan range; index ranges $0 \le h \le 28$, $0 \le k \le 9$, $-11 \le l \le 11$. (II): 1363 independent reflections, Mo $K\alpha$ radiation, graphite monochromator; 2θ in shell 1 (3·0–39·7°) at 6° min^{-1}, shell 2 (39·7–45·8°) at 3° min^{-1}; ratio of background counting time to net scanning time 1·00; total number of steps in ω scan 15; 11 steps used in intensity calculation; index ranges $-19 \le h \le 19$, $0 \le k \le 12$, $0 \le l \le 10$. In both molecules: cell constants refined from $\pm 2\theta$ values of 15 reflections in the range 24–30°; ψ scans for several intense reflections denoted absence of variable absorption; six standard reflections measured every 300 reflections; scan between 2θ settings 1·00 (I) and 0·50° (II) above and below calculated $K\alpha$ doublet values for each reflection; ratio of background counting time to net scanning time 0·50 (I) and 1·00 (II). Structure solution and refinement for each molecule were carried out by different programs and methods.

In (I) structure solved by direct methods using a modified version of *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), *SDP-Plus* (B. A. Frenz & Associates Inc., 1984) and *TEXRAY* 234 (Molecular Structure Corporation, 1984); O, N and C atoms anisotropic, hydrogen atoms

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Table 1. *Atomic coordinates* [$\times 10^4$; $\times 10^5$ *for x coordinate in* (I)] *and equivalent isotropic thermal parameters* (Å$^2 \times 10$) *in* (I) *and* (II) *with e.s.d.'s in parentheses*

	x	y	z	U_{eq}*
(I) $C_{16}H_{24}N_2O_4$				
O(1)	22907 (5)	−416 (2)	5462 (1)	52·8 (2)
O(2)	16478 (5)	660 (2)	6850 (1)	47·5 (2)
N(1)	15515 (5)	1187 (2)	4532 (1)	40·1 (2)
C(1)	10016 (6)	2196 (2)	4447 (1)	39·4 (3)
C(2)	4857 (6)	1061 (2)	4778 (1)	36·5 (2)
C(3)	4265 (6)	−603 (2)	4234 (2)	43·5 (3)
C(4)	482 (7)	1635 (2)	5544 (2)	42·8 (3)
C(5)	8898 (8)	2798 (2)	2936 (2)	54·2 (3)
C(6)	10963 (8)	3780 (2)	5384 (2)	50·9 (3)
C(7)	18093 (6)	492 (2)	5710 (1)	38·0 (3)
C(8)	26026 (9)	−1254 (3)	6637 (2)	60·6 (4)
(II) $C_{18}H_{28}N_2O_4$				
O(1)	2222 (1)	−670 (3)	299 (3)	84 (1)
O(2)	2895 (2)	−107 (4)	−1410 (3)	92 (1)
N(1)	3103 (1)	646 (3)	784 (3)	49 (1)
C(1)	3721 (2)	1420 (4)	536 (4)	51 (1)
C(2)	4377 (2)	669 (4)	222 (4)	45 (1)
C(3)	4889 (2)	1107 (4)	−565 (4)	62 (1)
C(4)	5500 (2)	451 (4)	−784 (4)	62 (1)
C(5)	3901 (2)	2090 (4)	1898 (4)	70 (2)
C(6)	3505 (3)	2335 (5)	−624 (6)	86 (2)
C(7)	2753 (3)	−49 (5)	−229 (5)	62 (2)
C(8)	1762 (3)	−1429 (6)	−625 (7)	119 (3)
C(9)	1101 (3)	−931 (7)	−957 (9)	147 (4)

* $U_{eq} = \frac{1}{3}$ trace U_{ij}.

Table 2. *Selected bond lengths* (Å) *and bond angles* (°) *for* (I) *and* (II)

	(I) $C_{16}H_{24}N_2O_4$	(II) $C_{18}H_{28}N_2O_4$
C(1)—C(2)	1·530 (1)	1·532 (6)
C(1)—C(5)	1·538 (1)	1·524 (6)
C(1)—C(6)	1·529 (1)	1·539 (7)
C(2)—C(3)	1·393 (1)	1·376 (6)
C(2)—C(4′)	1·383 (1)	1·365 (6)
C(8)—C(9)	—	1·361 (9)
C(1)—N(1)	1·471 (1)	1·467 (5)
C(7)—N(1)	1·343 (1)	1·341 (6)
C(7)—O(1)	1·347 (1)	1·345 (6)
C(7)—O(2)	1·215 (1)	1·201 (5)
C(8)—O(1)	1·434 (1)	1·449 (7)
C(2)—C(1)—C(5)	108·98 (7)	108·8 (3)
C(2)—C(1)—C(6)	113·26 (8)	111·8 (4)
C(5)—C(1)—C(6)	109·07 (9)	109·6 (4)
C(1)—C(2)—C(3)	119·64 (8)	122·1 (4)
C(1)—C(2)—C(4)	123·10 (8)	121·4 (4)
C(3)—C(2)—C(4)	117·17 (9)	116·4 (4)
N(1)—C(1)—C(2)	110·68 (7)	111·4 (3)
N(1)—C(1)—C(5)	105·36 (8)	105·9 (3)
N(1)—C(1)—C(6)	109·18 (8)	109·3 (3)
O(1)—C(7)—O(2)	123·65 (8)	124·9 (4)
O(1)—C(7)—N(1)	110·11 (7)	110·6 (4)
O(2)—C(7)—N(1)	126·23 (9)	124·6 (5)
O(1)—C(8)—C(9)	—	112·5 (6)
C(7)—O(1)—C(8)	115·77 (9)	118·4 (4)
C(1)—N(1)—C(7)	123·37 (7)	125·0 (3)

from $\Delta\rho$ map refined isotropically for three cycles and subsequently held fixed. Model converged with R(148 variables) = 0·041 and wR = 0·059 for 1343 independent reflections, S = 0·20, $(\Delta/\sigma)_{max}$ = 0·03.

In (II) intensities reduced without an absorption correction to relative square amplitudes, $|F_o|^2$, by means of standard Lorentz and polarization corrections; structure solved by direct methods with Nicolet *SHELXTL* (Sheldrick, 1981) software; three methyl groups [C(5), C(6), C(9) and their hydrogens from $\Delta\rho$ map] refined as rigid groups (C—H 0·96 Å); final orientation for each methyl group determined by three rotational parameters; remaining H atoms except H(N1) from $\Delta\rho$ map and refined isotropically [B_{eq} = 5 (1)(B) Å^2], included using riding model with C—H 0·96 Å; B(H) = 1·2 × B_{eq}(C); anisotropic refinement converged to R(123 variables) = 0·056, wR = 0·056 (GOF = 2·48) for 782 reflections; S = 1·86; blocked-cascade least-squares techniques (with blocking varying from 96 to 102 parameters per cycle in the final cycles) used for all refinement cycles; $(\Delta/\sigma)_{max}$ = 0·20.

For both structures: $\sum w(|F_o| - |F_c|)^2$ minimized; $w = 1/\sigma_F^2$, where $\sigma_F = \{[\sigma(F_o)]^2 + (p|F_o|)^2\}^{-1/2}$ and p = 0·03 (I) or 0·01 (II) with $I > 3\sigma(I)$; atomic scattering factors from *International Tables for X-ray Crystallography* (1974); no absorption corrections applied; features in final $\Delta\rho$ map ±0·20 e Å^{-3}. Final fractional coordinates for (I) and (II) are given in Table 1 and selected bond data for each single molecule are

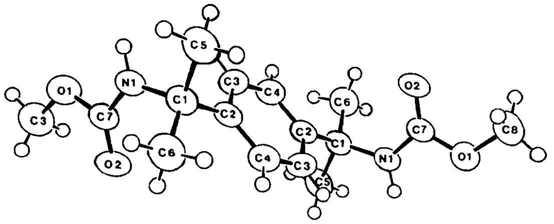

Fig. 1. Molecular drawing of $C_{16}H_{24}N_2O_4$.

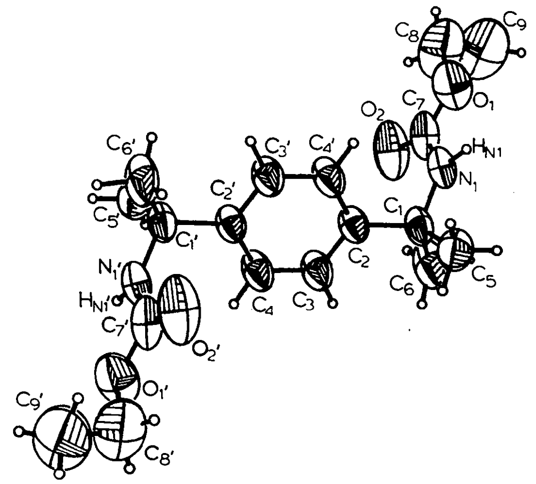

Fig. 2. Molecular drawing of $C_{18}H_{28}N_2O_4$ with nonhydrogen atoms represented by thermal ellipsoids depicted at the 50% probability level. H atoms are represented by arbitrarily small spheres. Atoms labeled with a prime (′) are related to nonprimed atoms by the crystallographic inversion center at ($\frac{1}{2}$,0,0) in the unit cell.

shown in Table 2 in accordance with the atom-numbering scheme shown in Fig. 1 (I) and Fig. 2 (II).*

Related literature. The inclination angles between the least-squares planes of the carbamate and phenyl groups, 86·3 (I) and 82·5° (II), differ from related molecules including dimethyl 4,4′-methylenebis(phenylcarbamate) (Gardner & Blackwell, 1980) and bis(4-hydroxybutyl)-4,4′-methylenebis(phenyl carbamate) (Forcier & Blackwell, 1981). A structure reported by Born, Hocker, Paulus & Wolfel, (1981) shows a number of similarities.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters and packing diagrams have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44737 (22 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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The Structures of Dimethyl *N,N′-trans*-1,4-Cyclohexanedicarbamate (I) and Diethyl *N,N′-trans*-1,4-Cyclohexanedicarbamate (II)

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Abstract. (I) $C_{10}H_{18}N_2O_4$, $M_r = 230{\cdot}27$, monoclinic, $P2_1/c$, $a = 10{\cdot}563\,(2)$, $b = 7{\cdot}265\,(1)$, $c = 8{\cdot}375\,(1)$ Å, $\beta = 106{\cdot}87\,(1)°$, $V = 615{\cdot}0$ Å^3, $Z = 2$, $D_x = 1{\cdot}243$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71073$ Å, $\mu = 0{\cdot}899$ cm^{-1}, $F(000) = 248$, room temperature. $R = 0{\cdot}058$, 1041 independent observed reflections. (II) $C_{12}H_{22}N_2O_4$, $M_r = 258{\cdot}32$, triclinic, $P\bar{1}$, $a = 8{\cdot}251\,(5)$, $b = 9{\cdot}284\,(5)$, $c = 12{\cdot}479\,(7)$ Å, $\alpha = 69{\cdot}30\,(1)$, $\beta = 108{\cdot}18\,(1)$, $\gamma = 68{\cdot}78\,(1)°$, $V = 719{\cdot}7$ Å^3, $Z = 2$, $D_x = 1{\cdot}192$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71073$ Å, $\mu =$

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