Fig. 1. General view of the molecule.

equal. Other bond lengths in this ligand almost match the corresponding values in the other metal complexes. The phenyl ring is planar with an average C—C distance of 1·381 (4) Å. The angle O(2)—C(7)—C(6) is = 117·8 (2)° [119·5 (3), 120·0 (4)°] whereas other O—C—C angles involving O(4) and O(6) are 109·8 (2)° [114·3 (3), 117·0 (4)°] and 112·5 (2)° [117·1 (3), 117·3 (4)°] respectively. The interplanar angle between the phenyl ring and the carboxylate group is 3·2 (3)° [29·6 (2), 18·9 (3)°]. The corresponding angle in anthranilic acid is 1·71 (5)°. The torsion angle C(1)—C(6)—C(7)—O(2) is = 5·9 (4)° [29·9 (3), 27·7 (6)°]. These values indicate that the carboxylate group is almost coplanar with the phenyl ring in free cpida. The torsion angles

N—C(8)—C(9)—O(4) and N—C(10)—C(11)—O(6) with values −2·7 (2) [16·3 (3), 22·9 (6)] and −3·6 (2)° [−17·6 (5), −24·1 (6)°] indicate that the glycinato arms are almost planar. In general, the strain in the glycinato arms increases when they are coordinated. The N—H(11) group is involved in intramolecular hydrogen bonding with O(2). The distance N···O(2) is 2·564 (3) Å. The hydroxyl groups are involved in intermolecular hydrogen bonding with values O(4)···O(2^{iii}) 2·610 (4) and O(6)···O(1^{iv}) 2·548 (3) Å where iii, iv represent the symmetry codes −x + 1, −y + 1, −z + 1 and x, −y, z + ½ − 1, respectively.

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Structure of the
3-(Methoxycarbonylamino)phenyl Ester of *N*-(3-Methylphenyl)carbamate

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Abstract. Methyl *N*-{3-[*N*-(3-methylphenyl)carbamoyloxy]phenyl}carbamate, $C_{16}H_{16}N_2O_4$, M_r = 300·3, triclinic, $P\bar{1}$, a = 11·528 (1), b = 12·885 (2), c = 5·086 (2) Å, α = 90·47 (3), β = 101·62 (3), γ = 94·97 (1)°, V = 736·93 (7) Å^3, Z = 2, D_x = 1·352 g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, μ = 7·26 cm^{-1}, $F(000)$ = 316, T = 293 K, final R = 0·077 for 2611 observed independent diffractometer-measured intensity data. Conformations around the carbamate groups and benzene rings are similar to those found

$$C_{16}H_{16}N_2O_4$$

in other carbamates. The structure consists of sheets of molecules linked by two N—H⋯O hydrogen bonds oriented approximately in the c direction.

Introduction. Bussacchini, Pouyet & Meallier (1985) have observed different photochemical behaviour of the bis(phenylcarbamate) family. In order to correlate this feature with the conformation, we studied the title compound. It is a selective weedkiller, used for treatment of beet and strawberry plants, acting on photosynthesis at very small concentrations.

The structure is reported here to ascertain its molecular geometry particularly with reference to the relative positions of the carbamate groups.

Experimental. X-ray diffraction study on a single crystal at room temperature. Needle of approximately $0.3 \times 0.3 \times 1$ mm used for data collection with an automatic Enraf–Nonius CAD-4 four-circle diffractometer, Cu $K\alpha$ radiation; cell dimensions obtained by least squares from setting angles of 25 reflections in the range $10 < \theta < 40°$, ω–2θ scan technique. Number of reflections measured 5981 (range of hkl: $h - 14 \rightarrow 14$, $k - 6 \rightarrow 6$, $l - 15 \rightarrow 15$). Number of unique reflections 2869, $R_{int} = 0.02$, 2611 with $I > 2.5\sigma(I)$ considered observed. One standard reflection measured every 60 min to control the intensity and the same measured at 100-reflection intervals to control the orientation of the crystal. Lorentz and polarization corrections, no absorption correction. Structure determined by direct methods and refined on F by SHELX76 (Sheldrick, 1976). Number of independent parameters 247. An E map showed clearly all non-H atoms of the molecule. H-atom positions determined by a difference Fourier synthesis. Non-H atoms refined isotropically, then anisotropically; H atoms refined, first with fixed, then with isotropic U's. C—H distances for methyl groups were constrained at 1.08 Å while the H—C—H angles were kept constant at $109°$. $(\Delta/\sigma)_{max} = 0.079$ for z of N(2), $(\Delta/\sigma)_{av} = 0.016$. Residual electron density in final difference Fourier synthesis within ± 0.18 e Å^{-3}. Unit weights used. Final $R = 0.077$, $wR = 0.077$, $S = 0.940$. Atomic scattering factors from SHELX76.
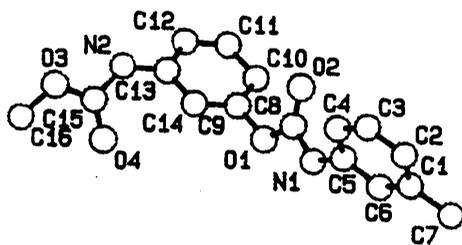
Discussion. Fig. 1 shows the molecule and the atom-numbering scheme, and Fig. 2 shows a stereoscopic view of the structure. The final atomic coordinates with their e.s.d.'s and equivalent isotropic thermal parameters are given in Table 1. The bond distances and angles are listed in Table 2.* In both benzene rings, two bonds are significantly shorter than the expected value: C(3)—C(4) = 1.362 (7) and C(1)—C(6) = 1.374 (7), C(11)—C(12) = 1.365 (7) and C(9)—C(14) = 1.360 (6) Å. If we adopt the labelling defined by Domenicano, Vaciago & Coulson (1975) to describe ring deformations in monosubstituted benzene derivatives, and consider the carbon substituted by the N—H group, the short bonds are in the 'b' position. Systematic angular distortion of the benzene ring has been observed in compounds containing C_6H_5—NH—X molecular fragments; it has been related to the interactions of the nitrogen lone pair with the π-electron system of the ring, but such conclusions and interpretations require great care (Domenicano & Murray-Rust, 1979).

Two angles differ significantly from the expected value in one benzene ring: C(10)—C(9)—C(14) = 124.3 (4) and C(9)—C(10)—C(11) = 116.2 (5)°. They may be correlated with the withdrawing effect of the substituent of C(9) (Domenicano & Murray-Rust, 1979). As can be seen from the relevant bond lengths and interbond angles, the two carbamate groups have practically the same geometry. All C—N and C—O distances are in agreement with those found in similar products: 2-propylpentyl carbamate (A) and 4-propylheptyl N-phenylcarbamate (B) (Cohen-Addad, D'Assenza, Taillandier & Benoit-Guyod,

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, torsion angles and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52795 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
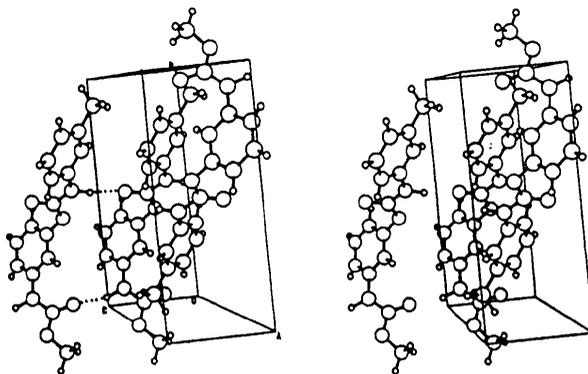

Fig. 1. View of a molecule with numbering scheme.

Fig. 2. Stereoscopic view of the structure along [100] drawn by PLUTO (Motherwell & Clegg, 1978).

Table 1. *Fractional coordinates* ($\times 10^4$) *and equivalent isotropic temperature factors* ($\text{Å}^2 \times 10^3$) *with e.s.d.'s in parentheses*

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

	x	y	z	U_{eq}
O(1)	1662 (3)	4255 (2)	4830 (5)	47 (1)
O(2)	2728 (4)	5181 (3)	8456 (5)	78 (2)
O(3)	1918 (3)	− 635 (2)	7931 (6)	51 (1)
O(4)	1494 (4)	483 (3)	4551 (5)	64 (1)
N(1)	3017 (4)	5508 (3)	4214 (6)	45 (1)
N(2)	1360 (4)	900 (3)	8845 (6)	46 (1)
C(1)	4894 (5)	7944 (4)	3787 (9)	51 (2)
C(2)	5814 (5)	7865 (5)	5995 (11)	61 (2)
C(3)	5808 (5)	6988 (5)	7572 (10)	67 (2)
C(4)	4904 (5)	6215 (5)	7034 (9)	56 (2)
C(5)	3970 (4)	6290 (3)	4872 (7)	41 (1)
C(6)	3991 (5)	7155 (4)	3243 (8)	46 (2)
C(7)	4889 (6)	8900 (5)	2068 (12)	75 (3)
C(8)	2496 (4)	4992 (4)	6028 (7)	45 (2)
C(9)	1140 (4)	3580 (3)	6545 (7)	42 (1)
C(10)	278 (5)	3935 (4)	7800 (9)	49 (2)
C(11)	− 202 (5)	3228 (4)	9477 (9)	51 (2)
C(12)	161 (4)	2248 (4)	9797 (8)	46 (2)
C(13)	1003 (4)	1906 (3)	8447 (7)	40 (1)
C(14)	1503 (4)	2603 (3)	6779 (7)	40 (1)
C(15)	1585 (4)	261 (4)	6934 (8)	44 (1)
C(16)	2186 (6)	− 1406 (4)	6089 (11)	67 (2)

Table 2. *Bond distances* (Å) *and bond angles* (°) *with e.s.d.'s in parentheses*

C(8)—O(1)	1·340 (5)	C(9)—O(1)	1·418 (5)
C(8)—O(2)	1·227 (5)	C(15)—O(3)	1·322 (5)
C(16)—O(3)	1·454 (6)	C(15)—O(4)	1·233 (5)
C(5)—N(1)	1·410 (5)	C(8)—N(1)	1·348 (5)
C(13)—N(2)	1·398 (6)	C(15)—N(2)	1·347 (5)
C(2)—C(1)	1·392 (7)	C(6)—C(1)	1·374 (7)
C(7)—C(1)	1·516 (7)	C(3)—C(2)	1·391 (8)
C(4)—C(3)	1·362 (7)	C(5)—C(4)	1·386 (6)
C(9)—C(14)	1·360 (6)	C(5)—C(6)	1·396 (6)
C(9)—C(10)	1·390 (6)	C(11)—C(10)	1·407 (6)
C(12)—C(11)	1·365 (7)	C(13)—C(12)	1·395 (6)
C(13)—C(14)	1·406 (5)		
C(9)—O(1)—C(8)	116·5 (3)	C(16)—O(3)—C(15)	117·6 (3)
C(8)—N(1)—C(5)	124·4 (3)	C(15)—N(2)—C(13)	125·6 (3)
C(6)—C(1)—C(2)	118·8 (5)	C(7)—C(1)—C(2)	119·9 (5)
C(7)—C(1)—C(6)	121·3 (5)	C(3)—C(2)—C(1)	119·5 (5)
C(4)—C(3)—C(2)	121·3 (5)	C(5)—C(4)—C(3)	120·0 (5)
C(4)—C(5)—N(1)	122·3 (4)	C(6)—C(5)—N(1)	118·9 (4)
C(6)—C(5)—C(4)	118·8 (4)	C(5)—C(6)—C(1)	121·6 (4)
O(2)—C(8)—O(1)	123·8 (4)	N(1)—C(8)—O(1)	111·1 (3)
N(1)—C(8)—O(2)	125·0 (4)	C(10)—C(9)—O(1)	119·1 (4)
C(14)—C(9)—O(1)	116·6 (4)	C(14)—C(9)—C(10)	124·3 (4)
C(12)—C(11)—C(10)	120·9 (5)	C(13)—C(12)—C(11)	121·4 (4)
C(12)—C(13)—N(2)	119·7 (4)	C(14)—C(13)—N(2)	121·6 (4)
C(14)—C(13)—C(12)	118·7 (4)	C(13)—C(14)—C(9)	118·5 (4)
O(4)—C(15)—O(3)	124·0 (4)	N(2)—C(15)—O(3)	111·5 (4)
N(2)—C(15)—O(4)	124·4 (4)	C(11)—C(10)—C(9)	116·2 (5)

1977); two isomers of isopropyl *N*-(methylfuroxan)-carbamate (*C* and *D*) (Calleri, Chiari, Chiesi Villa, Gaetani Manfredotti, Guastini & Viterbo, 1977); *p*-(dimethylamino)phenyl *N*-methyl-*N*-(*p*-nitro-phenylsulfonylmethyl)carbamate (*E*) (Visser, Vos & Engherts 1977); and ammonium carbamate (*F*) (Adams & Small, 1973).

Four least-squares planes can be defined: plane I through atoms C(1) to C(6) (first benzene ring); plane II through atoms C(9) to C(14) (second benzene ring); plane III through atoms N(1), C(8), O(2) and O(1) (first carbamate group) and plane IV through atoms N(2), C(15), O(3) and O(4) (second carbamate group).

Angles between planes I and III, II and IV are 42·3 (2) and 40·5 (2)°, respectively. These values can be compared with the angle 35·0° found for (*A*). The angle between planes II and III is 79·0 (2)°. It can be compared with the angle 88·4° found for (*E*). The carbamate group and the benzene ring are nearly perpendicular to each other when they are connected by the O atom, while they make a much smaller angle when they are connected by the N atom. The angles between the two benzene rings and the two carbamate groups are 77·4 (2) and 63·7 (2)°, respectively.

There are two intermolecular H bonds along the *z* direction. The N(1)···O(2)(*x*, *y*, *z* − 1) and N(2)···O(4) (*x*, *y*, *z* + 1) distances are 2·903 (4) and 2·932 (4) Å, respectively. The N—H distances are 1·066 (3) and 1·091 (3) Å, and the N—H···O bond angles are 158·1 (4) and 161·8 (2)°, respectively.

These bonds are roughly parallel to the *c* axis and they lead to the formation of sheets. The same

bonding scheme has been observed in (*A*), where N···O bonds are 2·884, 2·995 (4) and 2·952 (4) Å. For (*C*) two values are given, 3·169 (2) and 2·928 (2) Å, while for isomer (*D*) N···O = 2·813 (4) Å. In ammonium carbamate (*F*), *N*···O is 2·982 (5) Å. It has been pointed out (Cohen-Addad *et al.*, 1977) that this arrangement of chains parallel to a crystallographic axis can be correlated with the value of the corresponding cell parameter which is about 5 Å [5·086 (2) Å in the title compound].

Between two sheets only van der Waals contacts are observed.

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