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Methyl Carbamate

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Abstract. $C_2H_5NO_2$, $M_r = 75{\cdot}1$, triclinic, $P\bar{1}$, $a = 5{\cdot}146\,(4)$, $b = 5{\cdot}357\,(4)$, $c = 7{\cdot}147\,(6)$ Å, $\alpha = 103{\cdot}86\,(6)$, $\beta = 104{\cdot}39\,(5)$, $\gamma = 100{\cdot}65\,(8)°$, $V = 178{\cdot}9$ Å^3, $Z = 2$, $D_x = 1{\cdot}394$ Mg m^{-3}, $\lambda(\mathrm{Mo}\,K\alpha) = 0{\cdot}71069$ Å, $\mu = 0{\cdot}133$ mm^{-1}, $F(000) = 80$, $T = 123$ K, $R = 0{\cdot}073$ for 1603 unique observed reflections. The molecule has the expected conformation with the methyl group *trans* to the amino group with respect to the central C—O bond. The non-H atoms are planar with the exception of the methyl C which is 0·015 (2) Å out of the plane. The molecules are hydrogen-bonded into planar layers parallel to $(01\bar{2})$ by NH···O=C and $\mathrm{CH}\cdots\mathrm{O}\leqslant$ hydrogen bonds.

Introduction. The crystal structure of ethyl carbamate was reported by Braeker & Small (1967). There are no reports of crystal-structural work on the methyl compound. The structure is of interest since it contains C=O and NH_2 groups and is small enough to permit a comparison with accurate theoretical *ab initio* quantum mechanical calculations of the molecular geometry of the isolated molecule (Jeffrey, 1984, 1985). Crystals of neutron diffraction size can be obtained.

Experimental. Suitable crystals were obtained by fast evaporation of an ether solution. Since they sublime at room temperature, a crystal 0·25 × 0·3 × 0·2 mm was enclosed in a glass capillary. The intensity data were measured at 123 K by $\omega/2\theta$ scans on a CAD-4 diffractometer using Nb-filtered Mo $K\alpha$ radiation, for $-10 \le h \le 10$, $0 \le k \le 10$, $0 \le l \le 10$ to $\sin\theta/\lambda$ of 0·977 Å^{-1}. Three standard reflections $40\bar{4}$, $\bar{2}4\bar{2}$ and 214 showed a maximum deviation of $3{\cdot}9\sigma$. The crystals were soft, very mosaic, and diffracted poorly; this led to a higher-than-usual value for R; of the 3158 reflections measured up to $\theta_{\max} = 45°$, only 1603 had $F_o > 3\sigma$. The unit-cell dimensions were obtained from a least-squares fit of $\sin\theta$ values for 25 reflections with $0{\cdot}27 < \sin\theta < 0{\cdot}40$. The structure was solved using *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) on 717 reflections of which 150 had $E > 1{\cdot}29$. Atomic scattering factors for C, O and N were those of Cromer & Waber (1965), and for H those of Stewart, Davidson & Simpson (1965). The H positions were observed on difference maps. Refinement was of $\sum w(F_o - kF_c)^2$, $w^{-1} = \sigma^2(F_o)$, with anisotropic thermal parameters for the non-H atoms and isotropic parameters for the H atoms. There were no shifts greater than $0{\cdot}01\sigma$ in any of the 66 parameters. The maximum peak height in the final difference Fourier map was 0·48 e Å^{-3}. The final agreement factors were $R = 0{\cdot}073$, $wR = 0{\cdot}071$, $S = 2{\cdot}22$. The atomic parameters are given in Table 1, the atomic notation and thermal ellipsoids are shown in Fig. 1.* The bond lengths and principal torsion and valence angles are given in Table 2.

Discussion. The O.C=O.N group is planar within 0·003 (2) Å; the methyl C atom is 0·015 (2) Å out of this plane. The methyl group is oriented with one C—H bond almost in the molecular plane. The refined positions of the methyl H atoms suggest that there is a small twist of ~10° from the *m* symmetry orientation, but this needs confirmation by neutron diffraction.

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43262 (30 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 NH_2 group is planar within the experimental errors. The molecules are hydrogen-bonded and form layers, as illustrated in Fig. 2. The hydrogen-bond lengths and angles are shown in Table 3. The principal hydrogen bonding is from the $\mathrm{NH}\cdots\mathrm{O{=}C}$ bonds which form a cycle of four hydrogen bonds linking the molecules into a planar tetramer. There is a secondary centrosymmetrically related pair of weak $\mathrm{C{-}H}\cdots\mathrm{O}$ hydrogen bonds which forms hydrogen-bonded dimers and is responsible for the formation of planar layers of tetramers. The normalized $\mathrm{NH}\cdots\mathrm{O{=}C}$ bond lengths correspond closely to the mean value of 1·929 (4) Å for bonds from the survey by Taylor, Kennard & Versichel (1984). The $\mathrm{CH}\cdots\mathrm{O}\mathord{<}$ hydrogen-bond length of 2·60 Å is longer than the arbitrary van der Waals cut-off* used by Taylor & Kennard (1982) in their survey of $\mathrm{C{-}H}\cdots\mathrm{O}$ bonds.

Table 1. *Atomic parameters and equivalent isotropic temperature factors for methyl carbamate*

Fractional coordinates $\times 10^4$ for non-H atoms, $\times 10^3$ for H atoms. $B_{eq} \times 10^2$ (in Å^2) for non-H atoms is defined by $B_{eq} = \frac{4}{3}\sum_i\sum_j B_{ij} a_i^2$. E.s.d.'s given in parentheses refer to the least-significant digit.

	x	y	z	B_{eq}
O(1)	−3192 (2)	3657 (3)	1927 (2)	197 (3)
O(2)	652 (2)	7055 (3)	3529 (2)	189 (3)
N	−3553 (3)	7487 (3)	3779 (3)	207 (4)
C(1)	−1457 (4)	2098 (4)	1184 (3)	199 (4)
C(2)	−1859 (3)	6137 (3)	3119 (3)	137 (3)
H(1)	−9 (5)	193 (5)	228 (3)	
H(2)	−81 (5)	257 (6)	22 (4)	
H(3)	−262 (5)	25 (6)	51 (4)	
H(4)	−523 (5)	690 (5)	352 (4)	
H(5)	−282 (4)	905 (5)	456 (3)	

Table 2. *Molecular dimensions of methyl carbamate*

Distances are in Å, angles in °. E.s.d.'s given in parentheses refer to the least-significant digit.

Bond lengths			
C(1)—O(1)	1·440 (3)	C(1)—H(2)	0·90 (2)
O(1)—C(2)	1·338 (2)	C(1)—H(3)	0·99 (3)
C(2)—O(2)	1·227 (2)	N—H(4)	0·82 (3)
C(2)—N	1·334 (2)	N—H(5)	0·84 (2)
C(1)—H(1)	0·95 (2)		
Valence angles			
C(1)—O(1)—C(2)	115·2 (1)	H(1)—C(1)—H(2)	115 (2)
O(1)—C(2)—O(2)	122·9 (2)	H(1)—C(1)—H(3)	103 (2)
O(1)—C(2)—N	112·8 (1)	H(2)—C(1)—H(3)	105 (2)
O(2)—C(2)—N	124·2 (2)	H(4)—N—C(2)	125 (2)
H(1)—C(1)—O(1)	110 (1)	H(5)—N—C(2)	117 (2)
H(2)—C(1)—O(1)	115 (2)	H(4)—N—H(5)	118 (2)
H(3)—C(1)—O(1)	107 (2)		
Torsion angles			
H(1)—C(1)—O(1)—C(2)	60 (2)	H(3)—C(1)—O(1)—C(2)	171 (2)
H(2)—C(1)—O(1)—C(2)	−73 (2)		

The largest deviation of the C and O atoms from the least-squares plane of the hydrogen-bonded layers is 0·05 Å for O(1). The plane of the layers makes an angle of 0·9° with the crystallographic (01$\bar{2}$) planes and the mean layer separation is 3·26 Å.

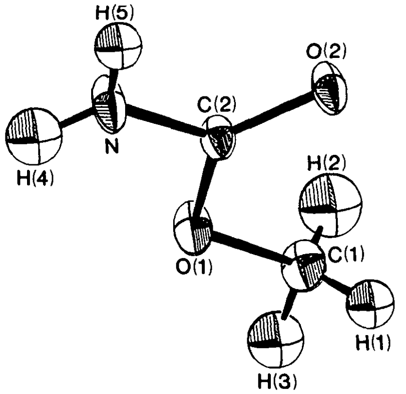

Fig. 1. Atomic notation and thermal ellipsoids of methyl carbamate, at 50% probability.

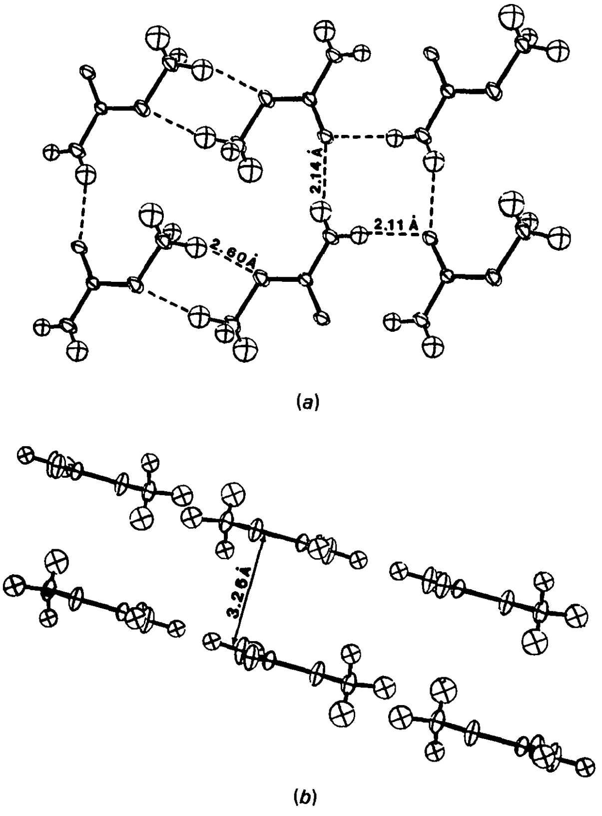

Fig. 2. (*a*) View of the crystal structure perpendicular to the (01$\bar{2}$) plane. (*b*) View of the crystal structure parallel to the (01$\bar{2}$) plane.

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* Such a cut-off is inappropriate, since at this distance the attractive component of the hydrogen bond is primarily electrostatic and attenuates at r^{-1}, whereas the attractive component of the van der Waals forces attenuates at $\sim r^{-6}$.

Table 3. *Hydrogen-bond lengths and angles in the crystal structure of methyl carbamate*

Distances in Å, angles in °. H···O^r normalized values with N—H bond lengths extended to 1·03 Å, and C—H bond lengths extended to 1·09 Å.

Donor Acceptor	H···O	H···O^r	N—H···O	Symmetry of acceptor molecule
NH(4)···O=C	2·14 (2)	1·95	155 (2)	$1 + x, y, z$
NH(5)···O=C	2·11 (2)	1·93	173 (2)	$-x, 2 - y, 1 - z$
C(1)H(3)···O(1)	2·60 (2)	2·51	158 (2)	$-1 - x, -y, -z$

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Structure of 9-Acetoxy-10-(4-acetoxyphenyl)anthracene at 95 K*

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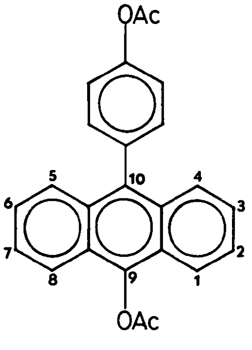
Abstract. $C_{24}H_{18}O_4$, $M_r = 370·4$, monoclinic, $P2_1/n$, $a = 11·021$ (2), $b = 18·518$ (4), $c = 9·125$ (2) Å, $\beta = 102·99$ (2)°, $V = 1814·6$ (7) Å^3, $Z = 4$, $D_m = 1·31$ (2), $D_x = 1·356$ (1) Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0·53$ cm^{-1}, $F(000) = 776$, $T = 95$ K. Final $R = 0·049$ for 4727 unique observed reflections. The anthracene and phenyl rings exhibit deformations consistent with the σ-electron characteristics of their substituents. The dihedral angle between the phenyl and the anthracene ring system [84·4 (1)°] is sensitive to phenyl ring substitution and may have a bearing on the fluorescent properties of compounds in this series.

Introduction. The structure of the title compound (I) was determined as a part of investigations of 9-acetoxy-10-arylanthracenes, which exhibit marked fluorescence in organic solvents (Heldt, Heldt & Gronowska, 1975; Gronowska, Aleksandrzak & Heldt, 1981) and are used as active media for dye lasers (Heldt, 1983). Knowledge of the three-dimensional structures of these compounds could contribute to an improved understanding of the molecular basis for the influence of phenyl ring substitution on laser activity (Heldt, 1983).



(I)

Experimental. Light-brown rhombic crystals grown from ethanol solution by slow evaporation, m.p. 470·0–471·0 K; space group and approximate cell dimensions determined from Weissenberg and oscillation photographs; density measured by flotation in aqueous KI solution; crystal size: 0·20 × 0·28 × 0·60 mm; Syntex $P3$ diffractometer with a low-

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* IUPAC name: 10-(4-acetoxyphenyl)-9-anthryl acetate.