All the acetoxy groups are planar and their geometry is similar to that reported earlier (*e.g.* Foces-Foces, Cano & García-Blanco, 1980). The OAc groups bonded to the benzene ring at C(16) and C(18) form dihedral angles of 75·6 and 63·8°, respectively, with the least-squares plane of the ring. The displacements of O(3) and O(5) from the benzene-ring plane are 0·175 (2) and 0·142 (2) Å, respectively. The OAc group bonded to anthracene forms a dihedral angle of 81·8° with the anthracene least-squares plane.

The geometry of the anthracene ring system is, in general, very similar to that of 9-acetoxy-10-(2-acetoxy-5-bromophenyl)anthracene (Roszak & Skrzat, 1985).

There are no unusually short contacts in this structure.

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# Structure of 2-(4-Chlorophenylhydrazono)-*N*-methylacetoacetamide

BY SAFIA MEHDI, Y. S. SADANANDAM, MEERA M. SHETTY AND B. RAMA RAO

*Regional Research Laboratory, Hyderabad-500 007, India*

**Abstract.** $C_{11}H_{12}ClN_3O_2$, $M_r = 253·7$, $P2_1/a$, $a = 11·476$ (1), $b = 13·918$ (3), $c = 7·565$ (5) Å, $\beta = 97·9$ (1)°, $V = 1196·8$ Å$^3$, $Z = 4$, $D_x = 1·41$, $D_m = 1·39$ g cm$^{-3}$, $\lambda(\text{Mo } K\alpha) = 0·71069$ Å, $\mu = 2·65$ cm$^{-1}$, $F(000) = 528$, room temperature, $R = 0·0558$ with 1002 independent non-zero reflections. Intermolecular bonding is entirely by van der Waals interactions. However, the molecule is stabilized by intramolecular hydrogen bonds. Except for one, all the bond lengths agree with those reported for azo pigments. The

C(O)–N bond in the acetoacetamide group [length 1·315 (8) Å] has partial double-bond character suggesting some electron delocalization.

**Introduction.** The structure of the title compound has been characterized by TLC, IR, NMR and mass spectrometry. In the NMR spectra, peaks for aryl protons ($\delta = 7\cdot3$) and highly deshielded protons ($\delta = 9\cdot23$) were observed, no signal in the range $\delta = 5$–6. In the IR spectra characteristic absorption bands at 3280, 1580, 1650 and 800 cm$^{-1}$ were observed. These have been assigned to NH, CN, CO and Cl, respectively. Diazonium rearrangement occurs during the synthesis. Peaks in the far down-field region which are ascribed to NH suggest that the protons are involved in strong intramolecular hydrogen bonding to the carbonyl group. No evidence of a second tautomeric form was obtained. The X-ray investigation was undertaken to determine the structure and ascertain the hydrogen bonding.

**Experimental.** The title compound was synthesized by the reaction of 4-chlorobenzenediazonium chloride (0·1 mol) with *N*-methylacetoacetamide (0·1 mol) in the presence of sodium acetate and ethanol.

Crystals pale yellow and rectangular in shape, 0·2 $\times$ 0·4 $\times$ 0·5 mm; $D_m$ by flotation in zinc bromide solution. Intensity data collected on Enraf–Nonius CAD-4 diffractometer. Lattice parameters determined from 15 high-angle reflections ($7 < \theta < 17°$). Systematic absences $h0l$ $h = 2n+1$, $0k0$ $k = 2n+1$ indicated uniquely the space group $P2_1/a$.

Intensities collected using monochromated Mo $K\alpha$ radiation. Two standard reflections chosen did not vary more than 8% in intensity; 2063 reflections measured, sin $\theta \le 0\cdot39$ Å$^{-1}$, 1962 unique, $R_{int} = 0\cdot036$, 1002 reflections at $4\sigma(F)$ significance level used in refinement. Lorentz and polarization corrections but none for absorption.

Structure solved by direct methods using *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). All non-H atoms readily located. $R = 0\cdot33$. H atoms calculated using a riding model and rigid-group and bond-length constraints. Refinement of H atoms isotropically, all other atoms anisotropically converged to $R = 0\cdot0557$ for 1002 reflections. Unit weights used throughout refinement (on $F$), index range: $h$ 0 to 12, $k$ 0 to 15, $l$ −8 to 8. The final coordinates and equivalent isotropic temperature factors for non-H atoms are given in Table 1.* Maximum least-squares shift/e.s.d. 0·053; $\Delta\rho$ in final

difference synthesis within $\pm 0\cdot12$ e Å$^{-3}$. Refinements performed with *SHELX*76 (Sheldrick, 1976); atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

Table 1. *Positional parameters* ($\times 10^4$) *and equivalent isotropic thermal parameters* (Å$^2$ $\times 10^3$), *with e.s.d.'s in parentheses*

$$U_{eq} = \tfrac{1}{3}(U_{11} + U_{22} + U_{23}).$$

|        | x          | y         | z          | $U_{eq}$ |
|--------|------------|-----------|------------|----------|
| Cl     | 8489 (2)   | 3 (1)     | 812 (3)    | 74 (1)   |
| C(2)   | 8372 (6)   | 2857 (5)  | 934 (10)   | 41 (7)   |
| C(3)   | 8063 (6)   | 1909 (4)  | 598 (10)   | 42 (4)   |
| C(4)   | 8862 (6)   | 1207 (4)  | 1191 (9)   | 52 (1)   |
| C(5)   | 9950 (6)   | 1428 (5)  | 2123 (10)  | 51 (4)   |
| C(6)   | 10252 (6)  | 2372 (5)  | 2432 (9)   | 39 (2)   |
| C(7)   | 9463 (5)   | 3097 (4)  | 1881 (9)   | 34 (5)   |
| N(1)   | 9724 (5)   | 4065 (4)  | 2169 (8)   | 37 (8)   |
| N(2)   | 10706 (4)  | 4299 (4)  | 3167 (7)   | 35 (6)   |
| C(10)  | 10970 (5)  | 5208 (4)  | 3498 (9)   | 39 (2)   |
| C(11)  | 10198 (6)  | 6026 (5)  | 2810 (9)   | 46 (8)   |
| C(12)  | 12099 (6)  | 5333 (5)  | 4700 (10)  | 42 (6)   |
| O(1)   | 9272 (4)   | 5858 (3)  | 1787 (7)   | 57 (8)   |
| O(2)   | 12465 (4)  | 6137 (4)  | 5158 (8)   | 52 (8)   |
| N(3)   | 10501 (6)  | 6901 (4)  | 3339 (8)   | 54 (2)   |
| C(16)  | 12786 (8)  | 4471 (7)  | 5335 (15)  | 43 (4)   |
| C(17)  | 9796 (10)  | 7730 (6)  | 2793 (15)  | 89 (6)   |

Table 2. *Bond angles* (°) *with e.s.d.'s*

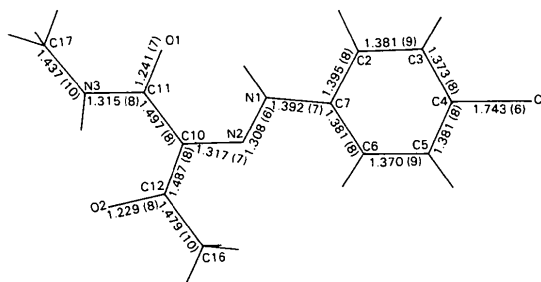| | | | |
|---|---|---|---|
| Cl–C(4)–C(3)   | 119·5 (5) | N(1)–N(2)–C(10)   | 120·4 (5) |
| Cl–C(4)–C(5)   | 118·9 (5) | N(2)–C(10)–C(11)  | 123·6 (5) |
| C(3)–C(4)–C(5) | 121·6 (6) | N(2)–C(10)–C(12)  | 112·8 (5) |
| C(4)–C(5)–C(6) | 119·4 (6) | C(11)–C(10)–C(12) | 123·6 (5) |
| C(5)–C(6)–C(7) | 120·6 (6) | C(10)–C(11)–O(1)  | 119·3 (6) |
| C(6)–C(7)–C(2) | 119·0 (6) | C(10)–C(11)–N(3)  | 118·2 (6) |
| C(7)–C(2)–C(3) | 120·8 (6) | O(1)–C(11)–N(3)   | 122·4 (7) |
| C(2)–C(3)–C(4) | 118·5 (6) | C(10)–C(12)–O(2)  | 120·9 (6) |
| C(2)–C(7)–N(1) | 118·1 (6) | C(16)–C(12)–O(2)  | 120·1 (7) |
| C(6)–C(7)–N(1) | 122·8 (6) | C(10)–C(12)–C(16) | 119·0 (6) |
| C(7)–N(1)–N(2) | 118·8 (5) | C(11)–N(3)–C(17)  | 122·5 (7) |



Fig. 1. Diagram showing atomic numbering and bond lengths (Å) in the molecule.
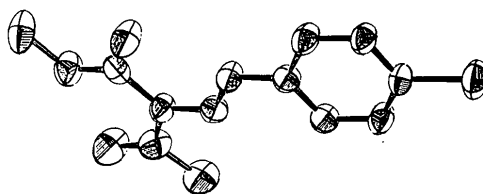


Fig. 2. *ORTEP* drawing of the molecule with H atoms omitted.

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42604 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

**Discussion.** The atomic numbering and the bond lengths are shown in Fig. 1. Bond angles are given in Table 2. The structure consists of independent molecules which are almost planar. Except for the C(11)–N(3) bond all other bond lengths have values expected for such structures. The C(11)–N(3) bond has partial double-bond character suggesting some form of delocalization in the structure. This effect has been observed in some azo pigments (Goliński, Reck & Kutschabsky, 1982; Whitaker, 1977a,b, 1983a,b, 1984). Further, the interatomic distances N(1)–O(1) = 2·557 (7) Å and N(3)–O(2) = 2·692 (8) Å suggest that both these contacts involve hydrogen bonds. These values compare well with 2·591 and 2·666 Å, the hydrogen-bond lengths found by Whitaker (1983b). This hydrogen bonding inhibits any rotation and will tend to cause the molecule to be planar. This is confirmed by the values of the torsion angles, the maximum deviation from 0 or 180° being 7·5 (9)°, indicating only a very minor deviation from planarity. The intermolecular interactions are all van der Waals. This structure confirms the results of the physico-chemical studies.

An *ORTEP* (Johnson, 1965) diagram of the molecule is shown in Fig. 2.

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# Structure of Leuconolam, an Alkaloid Containing an Unusual Nine-Membered Ring

BY CHEN WEI,* A. RAZAK MOHD. ALI AND S. H. GOH

*Department of Chemistry, University of Malaya, Kuala Lumpur, Malaysia*

EKKEHARD SINN

*Department of Chemistry, University of Virginia, Charlottesville, VA 22901, USA*

AND R. J. BUTCHER

*Department of Chemistry, Howard University, Washington, DC 20059, USA*

**Abstract.** $C_{19}H_{22}N_2O_3.C_2H_6O$, $M_r = 372·51$, $P2_12_12_1$, $a = 8·073$ (5), $b = 11·377$ (5), $c = 20·461$ (9) Å, $V = 1879$ (2) Å$^3$, $Z = 4$, $D_x = 1·32$ g cm$^{-3}$, $\lambda(Mo\,K\alpha_{1,2}) = 0·70926$, $0·713554$ Å, $\mu = 0·98$ cm$^{-1}$, $F(000) = 800$, $T = 297$ K, $R = 3·7\%$, 2046 unique reflections. The oxygen atoms are involved in hydrogen bonding, with the ethanolic oxygen atom acting both as a donor and as an acceptor intermolecularly. In the conformation adopted by the molecule the two conjugated groups make angles of 54·5 (2) and 60·4 (3)° with the plane of the benzene ring.

**Introduction.** X-ray analysis was undertaken to determine the structure and stereochemistry of leuconolam, an uncommon alkaloid isolated in fairly good quantities from *Leuconotis griffithii* (Apocynaceae). Spectral (NMR, MS, IR and UV) data obtained were too ambiguous for structural assignment (Goh, Chen Wei & A. Razak Mohd. Ali, 1984). The MS and NMR spectral data indicated considerable unsaturation in the molecule but this was not reflected in the UV spectrum, which appears to exclude 'expected' indole or acylated *o*-substituted aniline derivatives. The data suggest a new or unusual structural formula for this alkaloid and, since spectral comparisons with structurally related

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* To whom all correspondence should be addressed.