

group [O(1)...C(1) = 3.219 (3), O(1)...H(C1) = 2.20 (3) Å]. The short distance of 2.20 Å is due to the relatively long H(C1)—C(1) bond of 1.24 (1) Å. If an ideal value of 1.10 Å is adopted as the H(C1)—C(1) bond, the relevant distance is 2.36 Å.

There is an intramolecular hydrogen bond between the O(3) and O(5) atoms [O(3)...O(5) = 2.618 (3) Å, O(3)—H(O3) = 1.04 (2) Å, H(O3)...O(5) = 2.00 (2) Å, O(3)—H(O3)...O(5) = 115 (2)°].

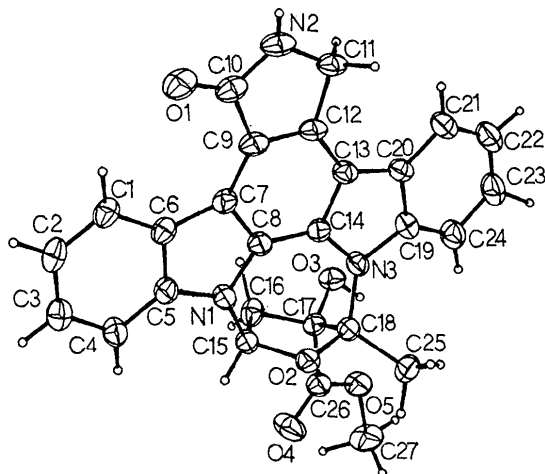


Fig. 1. View of the title compound, K-252a, showing atom numbering.

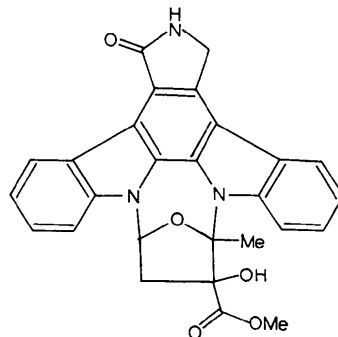


Fig. 2. Chemical structure of the molecule.

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### Structure of 6-Fluoro-10,10'-diphenyl-9-phenanthrone (1), 7-Chloro-10,10'-diphenyl-9-phenanthrone (2) and 3-Methoxy-10,10'-diphenyl-9-phenanthrone (3)

BY CARL KRÜGER AND MARIA J. ROMÃO\*

*Max-Planck-Institut für Kohlenforschung, Lembeckstraße 5, D-4330 Mülheim a.d. Ruhr, Federal Republic of Germany*

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**Abstract.** (1): C<sub>26</sub>H<sub>17</sub>FO, *M<sub>r</sub>* = 364.4, orthorhombic, *Iba*2, *a* = 17.274 (3), *b* = 13.748 (1), *c* = 15.877 (2) Å, *V* = 3770 Å<sup>3</sup>, *Z* = 8, *D<sub>x</sub>* = 1.28 g cm<sup>-3</sup>, λ(Mo *Kα*) = 0.71069 Å (graphite-crystal monochromator), μ = 0.8 cm<sup>-1</sup>, *F*(000) = 1520, *T* = 293 K, final *R* = 0.036 for 1900 unique observed reflections. (2): C<sub>26</sub>H<sub>17</sub>ClO, *M<sub>r</sub>* = 380.9, orthorhombic, *P2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>*, *a* = 9.027 (2), *b* = 12.517 (2), *c* = 16.874 (2) Å, *V* = 1907 Å<sup>3</sup>, *Z* = 4, *D<sub>x</sub>* = 1.33 g cm<sup>-3</sup>, λ(Mo *Kα*) =

0.71069 Å (graphite-crystal monochromator), μ = 2.1 cm<sup>-1</sup>, *F*(000) = 792, *T* = 293 K, final *R* = 0.046 for 2280 unique observed reflections. (3): C<sub>27</sub>H<sub>20</sub>O<sub>2</sub>, *M<sub>r</sub>* = 376.5, monoclinic, *P2<sub>1</sub>/n*, *a* = 14.647 (1), *b* = 9.666 (1), *c* = 15.102 (1) Å, β = 112.950 (3)°, *V* = 1969 Å<sup>3</sup>, *Z* = 4, *D<sub>x</sub>* = 1.27 g cm<sup>-3</sup>, λ(Cu *Kα*) = 1.54178 Å (Ni filter), μ = 5.8 cm<sup>-1</sup>, *F*(000) = 792, *T* = 293 K, final *R* = 0.039 for 3324 unique observed reflections. The molecular structures of three chemically related compounds are compared: in the three compounds there are no significant differences between comparable bond lengths and angles. The *sp*<sup>3</sup> C atom

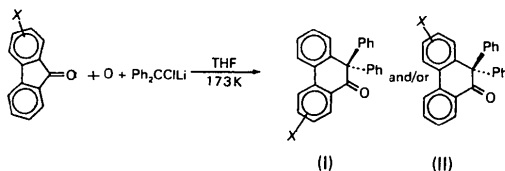
\* Present address: Laboratório de Química Orgânica, Instituto Superior Técnico, Av. Rovisco Pais, 1096 Lisboa Codex, Portugal.

Table 1. *Experimental data*

	(1)	(2)	(3)
Crystal size (mm)	0.4 × 0.4 × 0.3	0.3 × 0.5 × 0.4	0.2 × 0.6 × 0.4
$\theta$ range of reflections used for measuring lattice parameters ( $^\circ$ )	9.2–22.1	11.0–20.8	15.1–48.0
$\sin \theta/\lambda_{\max}$ ( $\text{\AA}^{-1}$ )	0.6465	0.7426	0.6284
Range of $hkl$	$\pm 21, 0 \rightarrow 23, 0 \rightarrow 18$	$0 \rightarrow 14, 0 \rightarrow 19, 0 \rightarrow 26$	$\pm 19, \pm 13, 0 \rightarrow 19$
Measured reflections	9497	3692	5077
Unique reflections	2217	3664	4083
$R_{\text{int}}$	0.013	—	0.007
Observed reflections [ $I \geq 2\sigma(I)$ ]	1900	2280	3324
Refined parameters	320	321	342
$R$	0.036	0.046	0.039
$wR w = 1/\sigma^2(F_o) $	0.040	0.048	0.057
Error of fit, $S$	2.0	2.3	3.1
Final max. shift/e.s.d.	0.05	0.1	0.032
Residual electron density ( $e \text{\AA}^{-3}$ )	0.15	0.25	0.17

C(10) leads to non-planarity of the fused-ring system. The bond lengths indicate little interaction between the two phenanthrenic benzene rings across the central ring or between those rings and the carbonyl group.

**Introduction.** The title compounds were prepared, along with other substituted phenanthrones, as part of the mechanistic study of the ring-expansion rearrangement observed in the reaction of a carbenoid with mono-substituted 9-fluorenones which gives rise to different relative amounts of isomers (I) and/or (II), depending on the nature of  $X$  (Empis, Franco, Herold & Queiroga, 1975; Herold & Romão, 1986).



Their unambiguous structural assignment is essential for a clarification of the reaction mechanism, and the X-ray crystallographic analyses of the title compounds were undertaken for that purpose. The description of the structure also gives information on the degree of bending of the molecules and allows a comparative study of the three compounds.

Independent structural assignment from  $^{13}\text{C}$  NMR data (Mynott, Herold & Romão, 1985) was confirmed, supporting the identification of the other mono-substituted phenanthrones from  $^{13}\text{C}$  NMR data alone.

**Experimental.** For all compounds, suitable crystals obtained from *n*-hexane and mounted in glass capillaries. Intensity data measured on an Enraf–Nonius CAD-4 diffractometer and corrected for Lorentz and polarization effects but not for absorption. Cell constants obtained from least-squares fits to setting angles of 75 automatically centered reflections. Three standard reflections showed no significant intensity variation

throughout experiment.  $\omega$ – $2\theta$  scan technique. Further experimental details are given in Table 1.

Table 2. *Atomic coordinates with e.s.d.'s in parentheses and equivalent isotropic thermal parameters ( $\text{\AA}^2$ ) of non-H atoms*

$U_{\text{eq}} = (U_1U_2U_3)^{1/3}$ , where  $U_i$  are the eigenvalues of the  $U_{ij}$  matrix.

	$x$	$y$	$z$	$U_{\text{eq}}$
<b>(a) Compound (1)</b>				
F	0.5259 (1)	–0.1242 (1)	0.7129 (1)	0.071
O	0.6862 (1)	–0.0033 (2)	0.3731 (2)	0.054
C(1)	0.8476 (2)	0.1662 (2)	0.5597 (2)	0.048
C(2)	0.8837 (2)	0.1637 (2)	0.6382 (2)	0.053
C(3)	0.8518 (2)	0.1113 (2)	0.7032 (2)	0.051
C(4)	0.7841 (2)	0.0605 (2)	0.6904 (2)	0.046
C(5)	0.6319 (2)	–0.0342 (2)	0.6652 (2)	0.047
C(6)	0.5678 (2)	–0.0889 (2)	0.6473 (2)	0.050
C(7)	0.5434 (2)	–0.1101 (2)	0.5670 (2)	0.051
C(8)	0.5859 (2)	–0.0731 (2)	0.5011 (2)	0.046
C(9)	0.6917 (1)	0.0303 (2)	0.4437 (2)	0.040
C(10)	0.7360 (1)	0.1245 (2)	0.4621 (2)	0.036
C(11)	0.7787 (1)	0.1157 (1)	0.5464 (2)	0.036
C(12)	0.7466 (1)	0.0620 (2)	0.6125 (2)	0.036
C(13)	0.6746 (1)	0.0052 (2)	0.5985 (2)	0.038
C(14)	0.6501 (1)	–0.0141 (2)	0.5161 (2)	0.039
C(15)	0.6719 (1)	0.2050 (2)	0.4694 (2)	0.039
C(16)	0.6752 (2)	0.2774 (2)	0.5295 (2)	0.052
C(17)	0.6187 (2)	0.3504 (2)	0.5318 (3)	0.064
C(18)	0.5586 (2)	0.3492 (3)	0.4757 (3)	0.064
C(19)	0.5543 (2)	0.2763 (3)	0.4159 (3)	0.063
C(20)	0.6105 (2)	0.2051 (2)	0.4123 (2)	0.052
C(21)	0.7920 (1)	0.1478 (2)	0.3896 (2)	0.040
C(22)	0.8487 (2)	0.0811 (2)	0.3685 (2)	0.050
C(23)	0.9015 (2)	0.0992 (2)	0.3054 (2)	0.057
C(24)	0.8987 (2)	0.1864 (3)	0.2618 (2)	0.062
C(25)	0.8443 (2)	0.2537 (3)	0.2826 (2)	0.067
C(26)	0.7910 (2)	0.2354 (2)	0.3461 (2)	0.056
<b>(b) Compound (2)</b>				
Cl	0.5222 (1)	0.1011 (1)	0.6947 (1)	0.065
O	0.6598 (3)	0.4944 (2)	0.5922 (1)	0.054
C(1)	0.9734 (4)	0.6566 (2)	0.7833 (2)	0.040
C(2)	0.9979 (4)	0.6720 (3)	0.8645 (2)	0.046
C(3)	0.9383 (4)	0.6024 (3)	0.9180 (2)	0.046
C(4)	0.8593 (4)	0.5137 (3)	0.8924 (2)	0.043
C(5)	0.7279 (4)	0.3107 (3)	0.8344 (2)	0.043
C(6)	0.6555 (4)	0.2209 (3)	0.8071 (2)	0.047
C(7)	0.6121 (3)	0.2148 (2)	0.7283 (2)	0.043
C(8)	0.6373 (3)	0.2997 (3)	0.6774 (2)	0.040
C(9)	0.7383 (4)	0.4802 (3)	0.6495 (2)	0.040
C(10)	0.8760 (3)	0.5504 (2)	0.6663 (1)	0.033
C(11)	0.8939 (3)	0.5683 (2)	0.7557 (2)	0.032
C(12)	0.8377 (3)	0.4942 (2)	0.8121 (1)	0.031
C(13)	0.7596 (3)	0.3965 (2)	0.7847 (1)	0.033
C(14)	0.7119 (3)	0.3901 (2)	0.7048 (2)	0.034
C(15)	1.0079 (3)	0.4831 (2)	0.6339 (2)	0.037
C(16)	1.1312 (4)	0.4589 (3)	0.6811 (2)	0.049
C(17)	1.2484 (4)	0.4014 (4)	0.6489 (3)	0.062
C(18)	1.2465 (5)	0.3664 (3)	0.5715 (3)	0.063
C(19)	1.1253 (5)	0.3878 (3)	0.5259 (2)	0.055
C(20)	1.0054 (5)	0.4455 (3)	0.5562 (2)	0.047
C(21)	0.8591 (3)	0.6581 (2)	0.6239 (2)	0.037
C(22)	0.7415 (4)	0.7244 (3)	0.6448 (2)	0.054
C(23)	0.7215 (5)	0.8222 (3)	0.6091 (3)	0.063
C(24)	0.8182 (5)	0.8579 (3)	0.5520 (3)	0.061
C(25)	0.9359 (5)	0.7935 (3)	0.5297 (2)	0.056
C(26)	0.9559 (4)	0.6944 (3)	0.5646 (2)	0.046
<b>(c) Compound (3)</b>				
C(1)	0.5501 (1)	0.3013 (2)	0.3870 (1)	0.045
C(2)	0.5914 (1)	0.4096 (2)	0.4498 (1)	0.047
C(3)	0.5862 (1)	0.5421 (2)	0.4132 (1)	0.045
C(4)	0.5377 (1)	0.5640 (2)	0.3152 (1)	0.044
C(5)	0.4490 (1)	0.6111 (2)	0.1059 (1)	0.055
C(6)	0.3964 (2)	0.6349 (2)	0.0096 (1)	0.065
C(7)	0.3406 (2)	0.5318 (2)	–0.0500 (1)	0.063
C(8)	0.3380 (1)	0.4024 (2)	–0.0121 (1)	0.052
C(9)	0.3880 (1)	0.2354 (1)	0.1226 (1)	0.039
C(10)	0.4657 (1)	0.1944 (1)	0.2217 (1)	0.036
C(11)	0.5032 (1)	0.3200 (1)	0.2879 (1)	0.036
C(12)	0.4971 (1)	0.4551 (1)	0.2510 (1)	0.037
C(13)	0.4476 (1)	0.4810 (1)	0.1469 (1)	0.040

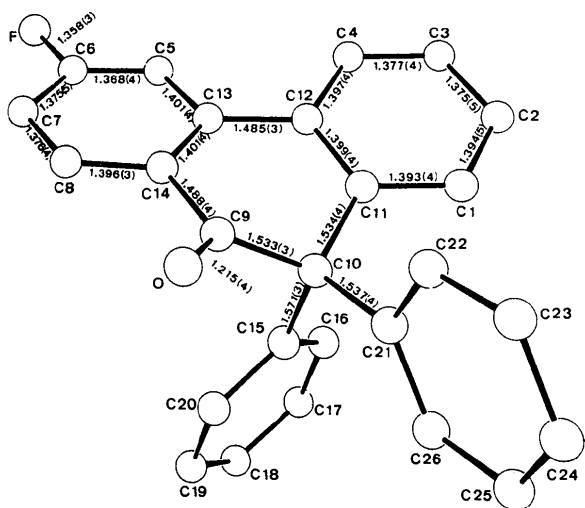
Table 2 (cont.)

	x	y	z	U <sub>eq</sub>
C(14)	0.3922 (1)	0.3762 (1)	0.0853 (1)	0.040
C(15)	0.5501 (1)	0.1310 (1)	0.1967 (1)	0.038
C(16)	0.6438 (1)	0.1897 (2)	0.2262 (1)	0.045
C(17)	0.7161 (1)	0.1305 (2)	0.1997 (1)	0.053
C(18)	0.6963 (1)	0.0129 (2)	0.1442 (1)	0.052
C(19)	0.6035 (1)	-0.0462 (2)	0.1144 (1)	0.055
C(20)	0.5309 (1)	0.0119 (2)	0.1400 (1)	0.049
C(21)	0.4222 (1)	0.0877 (1)	0.2701 (1)	0.037
C(22)	0.4775 (1)	-0.0212 (2)	0.3245 (1)	0.047
C(23)	0.4396 (1)	-0.1067 (2)	0.3763 (1)	0.054
C(24)	0.3451 (1)	-0.0862 (2)	0.3723 (1)	0.053
C(25)	0.2884 (1)	0.0205 (2)	0.3171 (1)	0.051
C(26)	0.3268 (1)	0.1075 (2)	0.2671 (1)	0.045
C(27)	0.6865 (2)	0.6366 (3)	0.5663 (2)	0.068
O(1)	0.3278 (1)	0.1512 (1)	0.0730 (1)	0.052
O(2)	0.6265 (1)	0.6567 (1)	0.4669 (1)	0.060

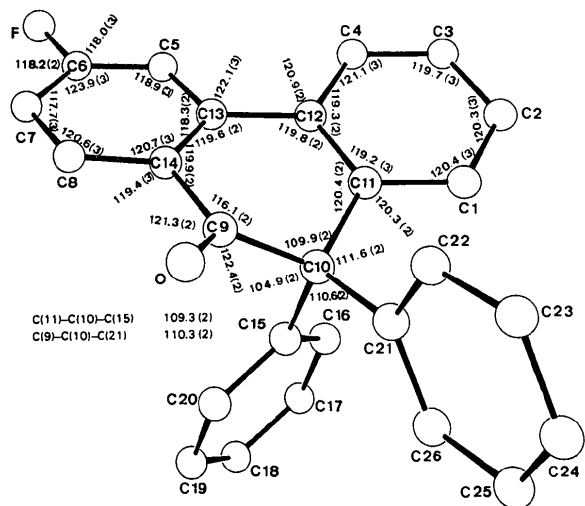
Structures solved by direct methods using *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) and refined initially using *SHELX76* (Sheldrick, 1976). After full-matrix least-squares refinement with non-H atoms treated anisotropically, all H-atom positions could be located from difference Fourier syntheses, and included isotropically in final anisotropic refinement cycles;  $\sum w(|F_o| - |F_c|)^2$  minimized. Scattering factors of Cromer & Waber (1965) for non-H atoms and best spherical scattering factors for H from Stewart, Davidson & Simpson (1965).

**Discussion.** Atomic parameters are given in Table 2.\* *ORTEP* plots (Johnson, 1965) of the molecular

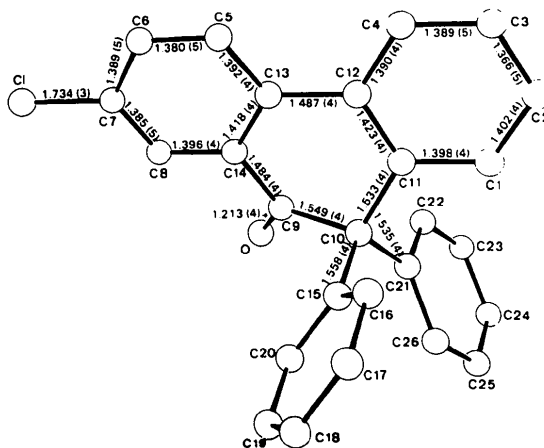
\* Lists of structure factors, anisotropic thermal parameters for the non-H atoms and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 43023 (56 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.



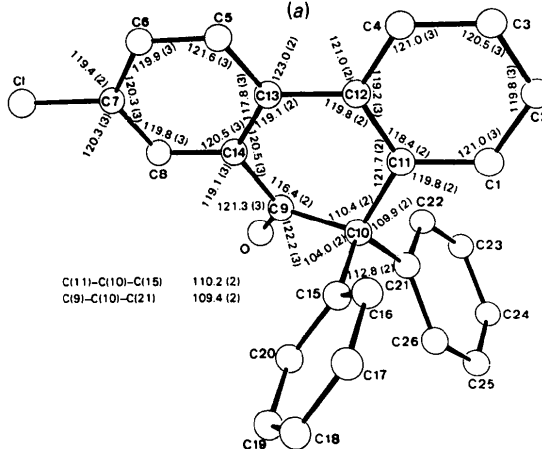
(a)



(b)



(a)



(b)

Fig. 1. Molecular structure of 6-fluoro-10,10'-diphenyl-9-phenanthrone (1), showing (a) bond distances (Å) and (b) angles (°) with e.s.d.'s in parentheses.

Fig. 2. Molecular structure of 7-chloro-10,10'-diphenyl-9-phenanthrone (2), showing (a) bond distances (Å) and (b) angles (°) with e.s.d.'s in parentheses.

structures of the title compounds (1, 2 and 3) are shown in Figs. 1, 2 and 3, respectively, together with the atomic-numbering scheme and selected bond lengths and angles.

There are no significant bond-length differences between comparable bonds in the three molecular structures. Ring *A* [C(1), C(2), C(3), C(4), C(12), C(13)] and ring *B* [C(5), C(6), C(7), C(8), C(14), C(13)] in all three molecules are planar to within  $\pm 0.02$  Å, but are twisted by different amounts relative to one another about the C(12)–C(13) bond (molecule 1: 16.2; molecule 2: 13.8; molecule 3: 9.9°). This has no significant effect on the C(12)–C(13) bond length [av. 1.482 (7) Å], which is comparable with the value observed for a single  $Csp^2$ – $Csp^2$  bond (1.48 Å) (Lide,

1962; Allmann & Hellner, 1968). Bond-length values for the biphenyl linkage range from 1.475 (9-fluorenone) to 1.519 (7) Å (2,4,7-dinitro-9-fluorenone) and are not significantly shorter in the essentially planar phenanthrene (Kay, Okaya & Cox, 1971) (1.464 Å) and its derivative 9-trimethylsilylphenanthrene (Lu, Hseu & Lee, 1977) [1.45 (1) Å].

The C(9)–C(14) bond length [av. 1.485 (2) Å] is similarly long and thus there appears to be no conjugation in the central six-membered ring (*C*) in agreement with crystallographic results for 9-fluorenone (Luss & Smith, 1972), 2-bromo-9-fluorenone (Griffiths & Hine, 1970), 2,7-diamino-9-fluorenone (Iball & Scrimgeour, 1977), 2,4,7-trinitro-9-fluorenone (Dorset, Hybl & Ammon, 1972) and 2,7-dinitro-9-fluorenone (Baughman, 1982).

The average carbonyl bond length for the three molecules [1.216 (3) Å] is comparable with the values reported for the above-mentioned substituted fluorenones. The bond lengths C(7)–Cl (molecule 2) [1.734 (3) Å] and C(6)–F (molecule 1) [1.358 (3) Å] agree with reported values within aromatic systems (Marsh & Williams, 1981; Rawas & Sutherland, 1981).

In all three molecules, intramolecular contacts between the two H atoms H(4) and H(5) [2.18 (4), 2.05 (5) and 2.03 (3) Å for molecules 1, 2 and 3, respectively] are shorter than the normal sum of their van der Waals radii (2.4 Å). No intermolecular contact shorter than 3.0 Å is found between non-H atoms.

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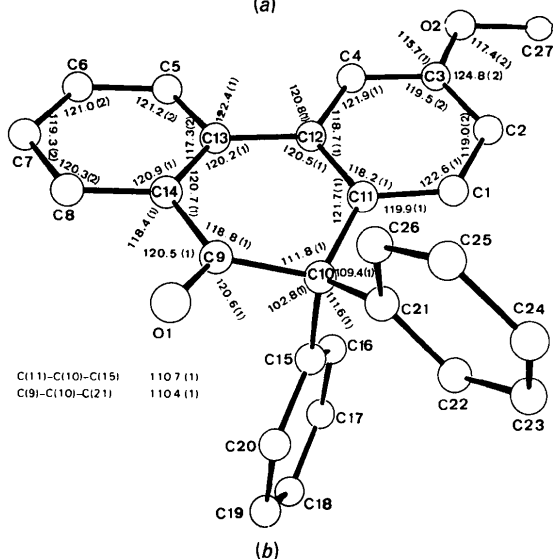
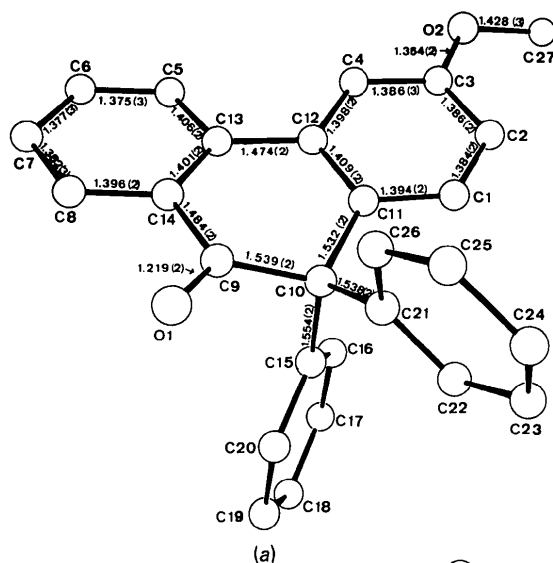


Fig. 3. Molecular structure of 3-methoxy-10,10'-diphenyl-9-phenanthrone (3), showing (a) bond distances (Å) and (b) angles (°) with e.s.d.'s in parentheses.

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*Acta Cryst.* (1986). C42, 1408–1411

## Hydrogen-Bonding Patterns in 8-Hydroxyquinoline Derivatives: (I) Structure of 5-Chloro-8-hydroxyquinoline and (II) Refinement of the Structure of 8-Hydroxyquinoline

BY T. BANERJEE AND N. N. SAHA

*Crystallography and Molecular Biology Division, Saha Institute of Nuclear Physics, Sector I, Block 'AF', Bidhannagar, Calcutta-700 064, India*

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**Abstract.** (I): C<sub>9</sub>H<sub>6</sub>ClNO,  $M_r = 179.6$ , orthorhombic, space group *Fdd2*,  $a = 29.807$  (9),  $b = 27.495$  (4),  $c = 3.814$  (4) Å,  $V = 3126$  (3) Å<sup>3</sup>,  $Z = 16$ ,  $D_m = 1.55$ ,  $D_x = 1.53$  Mg m<sup>-3</sup>,  $\lambda(\text{Cu } K\alpha) = 1.5418$  Å,  $\mu = 3.92$  mm<sup>-1</sup>,  $F(000) = 1472$ ,  $T = 297$  K,  $R = 0.049$  for 469 'observed' reflections. (II): C<sub>9</sub>H<sub>7</sub>NO,  $M_r = 145.2$ , orthorhombic, space group *Fdd2*,  $a = 28.770$  (7),  $b = 25.055$  (12),  $c = 3.857$  (5) Å,  $V = 2780$  (4) Å<sup>3</sup>,  $Z = 16$ ,  $D_m = 1.41$ ,  $D_x = 1.39$  Mg m<sup>-3</sup>,  $\lambda(\text{Cu } K\alpha) = 1.5418$  Å,  $\mu = 0.703$  mm<sup>-1</sup>,  $F(000) = 1216$ ,  $T = 297$  K,  $R = 0.056$  for 474 'observed' reflections. In both structures the hydroxyl hydrogens are involved in bifurcated O–H...N hydrogen bonds, simultaneously leading to the formation of five-membered hydrogen-bonded chelate rings and the dimerization of the molecules. The stronger intramolecular hydrogen bond in the 5-chloro derivative is consistent with IR spectroscopic studies in solution.

**Introduction.** 8-Hydroxyquinoline and its derivatives are well known for their antifungal, antibacterial and antiameobic activities (Bambury, 1979). The biological activities of 8-hydroxyquinoline derivatives have been correlated with their capacity to chelate metal ions (Martell & Calvin, 1959). Structural analyses of 5-chloro-8-hydroxyquinoline (I) and 8-hydroxyquinoline (II) were undertaken in continuation of our programme of studying the hydrogen-bonding patterns in crystal structures of 8-hydroxyquinoline derivatives (Banerjee, Basak, Mazumdar & Chaudhuri, 1984). The structure of 8-hydroxyquinoline, reported earlier (Roychowdhury, Das & Basak, 1978), was based on photographic data; it has been refined by us with diffractometric data in order to locate the hydrogen atoms and to make more quantitative comparisons with other similar structures.

**Experimental.** Both compounds crystallized as colourless needles (elongated along the  $c$  axis) from benzene; density by flotation (KI solution); preliminary cell parameters and symmetry information for (I) from oscillation and Weissenberg photographs (systematic absences:  $hkl$ :  $h + k$ ,  $k + l \neq 2n$ ,  $0kl$ :  $k + l \neq 4n$ ,  $h0l$ :  $h + l \neq 4n$ ), for (II) from published data; crystal size: (I) 1.00 × 0.08 × 0.05 mm, (II) 0.65 × 0.45 × 0.15 mm; both crystals sealed in Lindemann-glass capillaries to avoid deterioration; accurate cell parameters from 25 selected reflections [in ranges (I)  $25 \leq \theta \leq 30^\circ$  and (II)  $23 \leq \theta \leq 30^\circ$ ] centred on an Enraf–Nonius CAD-4 diffractometer, graphite-monochromatized Cu  $K\alpha$  radiation; 'needle option', with crystal needle axes normal to incident beam, adopted to minimize absorption; three 'orientation control' reflections monitored after every 50 reflections and three 'intensity control' reflections monitored after every hour of X-ray exposure; number of unique reflections measured: 599 ( $0 \leq h \leq 28$ ,  $0 \leq k \leq 28$ ,  $0 \leq l \leq 4$ ,  $2 \leq \theta \leq 55^\circ$ ) for (I) and 539 ( $0 \leq h \leq 30$ ,  $0 \leq k \leq 26$ ,  $0 \leq l \leq 3$ ,  $2 \leq \theta \leq 55^\circ$ ) for (II), number of unobserved reflections [ $I < 3\sigma(I)$ ]: 130 for (I) and 65 for (II); data corrected for  $L_p$  and for variations in intensity ( $ca$  3%) of control reflections, absorption corrections ignored; structure (I) solved by *MULTAN* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978); initial coordinates for structure (II) from published data (Roychowdhury, Das & Basak, 1978); H from  $\Delta F$  synthesis; anisotropic full-matrix least-squares refinement based on  $F$  (*ORFLS*, Busing, Martin & Levy, 1962); 133 parameters refined for (I) and 128 for (II);  $R = 0.049$  (I),  $0.056$  (II),  $wR = 0.057$  (I),  $0.068$  (II),  $S = 1.42$  (I),  $1.37$  (II),  $\Delta/\sigma < 0.05$  (I),  $< 0.03$  (II); residual  $\Delta\rho = -0.21$  to  $0.29$  e Å<sup>-3</sup> (I),  $-0.24$  to  $0.28$  e Å<sup>-3</sup> (II), weights  $w = 1/\sigma^2(|F_o|)$ ;