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Crystal and molecular structure of phenanthridone.

By Deb Kumar Sen, *X-ray Laboratory, Presidency College, Calcutta, India.*

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Phenanthridone, $C_{13}H_9NO$, has space group $P2_12_12_1$, with $a=12{\cdot}512$, $b=16{\cdot}080$, $c=4{\cdot}676$ Å with four molecules per unit cell. There is a hydrogen bond between the nitrogen atom of one molecule and the oxygen atom of a neighbouring one.

In the course of study on the structures closely related to the phenanthrene molecule the study of phenanthridone was undertaken. Crystals of phenanthridone, $C_{13}H_9NO$, are orthorhombic, space group $P2_12_12_1$, $a=12{\cdot}512$, $b=16{\cdot}080$, $c=4{\cdot}676$ Å with four molecules per unit cell. Approximate positional parameters were obtained by a combination of a weighted reciprocal-lattice plot for $hk0$ reflexions and the trial-and-error method. A least-squares refinement using the computer program *ORFLS*, written by Martin, Busing & Levy (1962), led to the complete solution of the structure. Starting from an initial value of 49·14% the final residual value obtained is 10·15% with 781 reflexions collected along the c axis and scaled on a common base. Visual methods of intensity measurement were adopted. Refinement was based on F. Because of the program limitation during the final round of refinement with anisotropic temperature factors and hydrogen atoms the parameters were adjusted into three separate groups. Electron-density sections along the c axis have been computed and a difference synthesis without hydrogen atoms has also been computed to confirm the location of the hydrogen atoms. The conventional structural formula is shown in the Fig. 1. The final values of the atomic parameters are listed in Tables 1, 2 and 3. Bond lengths and angles are given in Tables 4 and 5. The striking feature of the structure is the closest approach of the nitrogen atom of the original molecule to the oxygen atom of the second molecule. The value of the distance is 2·85(7) Å which reveals hydrogen bonding between those two atoms. Some of the approach distances are shown in Table 6. The bond distances C(3)–H(17) and C(10)–H(22), and angles involving H(17) and H(23) are found to be significantly longer than the normal value which is most likely to be due to erroneous shift given in

Table 1. *Positional parameters of non-hydrogen atoms and their estimated standard deviations*

	x/a ($\times 10^4$)	y/b ($\times 10^4$)	z/c ($\times 10^4$)
C(1)	5996 (6)	3248 (4)	2780 (22)
C(2)	6325 (7)	2685 (5)	758 (23)
C(3)	5700 (7)	1998 (5)	−50 (27)
C(4)	4689 (7)	1901 (5)	1286 (24)
C(5)	4352 (7)	2454 (5)	3305 (23)
C(6)	4971 (6)	3164 (5)	4099 (21)
C(7)	4606 (6)	3782 (5)	6199 (20)
C(8)	3587 (6)	3771 (5)	7544 (22)
C(9)	3333 (7)	4362 (6)	9502 (25)
C(10)	4012 (7)	5025 (5)	232 (23)
C(11)	5035 (7)	5046 (5)	8868 (22)
C(12)	5304 (6)	4443 (5)	6853 (23)
C(13)	6684 (7)	3959 (5)	3469 (23)
N(14)	6324 (5)	4487 (4)	5529 (18)
O(15)	7569 (4)	4056 (3)	2394 (17)

Table 2. *Thermal parameters of non-hydrogen atoms and their estimated standard deviations*

β is defined by

$$T=\exp\left[-(\beta_{11}h^2+\beta_{22}k^2+\beta_{33}l^2+2\beta_{12}hk+2\beta_{13}hl+2\beta_{23}kl)\right].$$

	β_{11} ($\times 10^4$)	β_{22} ($\times 10^4$)	β_{33} ($\times 10^4$)	β_{12} ($\times 10^4$)	β_{13} ($\times 10^4$)	β_{23} ($\times 10^4$)
C(1)	43 (5)	26 (3)	315 (63)	3 (3)	−30 (15)	11 (12)
C(2)	64 (6)	27 (3)	468 (73)	8 (4)	−10 (18)	30 (13)
C(3)	85 (7)	25 (3)	544 (77)	3 (4)	−33 (20)	−18 (14)
C(4)	82 (7)	32 (3)	469 (73)	−6 (4)	−63 (19)	−13 (14)
C(5)	66 (6)	36 (3)	461 (70)	−15 (4)	−19 (18)	10 (14)
C(6)	57 (5)	27 (3)	255 (59)	−4 (3)	−23 (15)	5 (12)
C(7)	51 (5)	32 (3)	248 (61)	−3 (3)	−6 (14)	9 (11)
C(8)	49 (5)	42 (4)	304 (65)	−4 (4)	−7 (16)	9 (14)
C(9)	54 (6)	49 (4)	489 (75)	−2 (4)	17 (18)	42 (16)
C(10)	67 (6)	42 (4)	366 (69)	9 (4)	12 (18)	30 (14)
C(11)	66 (6)	29 (3)	348 (69)	0 (4)	−22 (18)	−32 (13)
C(12)	54 (5)	29 (3)	380 (66)	−2 (4)	−25 (17)	20 (13)
C(13)	61 (6)	30 (3)	308 (69)	1 (4)	−23 (17)	10 (12)
N(14)	38 (4)	30 (2)	400 (52)	−10 (3)	25 (12)	−2 (10)
O(15)	49 (4)	41 (2)	573 (49)	−6 (3)	37 (13)	12 (10)

Table 3. *Parameters of hydrogen atoms and their estimated standard deviations*

	x/a ($\times 10^4$)	y/b ($\times 10^4$)	z/c ($\times 10^4$)	β_{11} ($\times 10^4$)	β_{22} ($\times 10^4$)	β_{33} ($\times 10^4$)
H(16)	6991 (65)	2754 (49)	−497 (243)	55	33	392
H(17)	6119 (64)	1722 (50)	−2203 (234)	54	33	389
H(18)	4209 (67)	1498 (51)	124 (251)	60	36	428
H(19)	3640 (70)	2490 (54)	3634 (254)	63	38	452
H(20)	3020 (65)	3350 (51)	7125 (246)	57	34	406

Table 3 (*cont.*)

	x/a ($\times 10^4$)	y/b ($\times 10^4$)	z/c ($\times 10^4$)	β_{11} ($\times 10^4$)	β_{22} ($\times 10^4$)	β_{33} ($\times 10^4$)
H(21)	2677 (72)	4264 (53)	889 (242)	69	42	497
H(22)	3905 (69)	5321 (54)	2562 (251)	69	42	491
H(23)	5200 (66)	5460 (51)	197 (227)	57	34	406
H(24)	6769 (65)	4829 (50)	5501 (238)	52	32	374

Table 4. *Bond lengths*

C(1)—C(2)	1·372 Å	C(8)—C(9)	1·357 Å
C(2)—C(3)	1·407	C(9)—C(10)	1·405
C(3)—C(4)	1·418	C(10)–C(11)	1·431
C(4)—C(5)	1·363	C(11)–C(12)	1·394
C(5)—C(6)	1·428	C(2)—H(16)	1·025
C(6)—C(1)	1·429	C(3)—H(17)	1·219
C(1)—C(13)	1·467	C(4)—H(18)	1·038
C(13)–N(14)	1·361	C(5)—H(19)	0·906
N(14)–C(12)	1·420	C(8)—H(20)	1·001
C(12)–C(7)	1·408	C(9)—H(21)	1·058
C(7)—C(6)	1·470	C(10)–H(22)	1·196
C(7)—C(8)	1·422	C(11)–H(23)	0·933
C(13)–O(15)	1·227	N(14)–H(24)	0·782

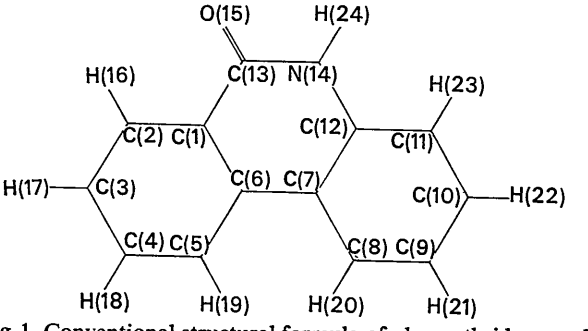

Fig. 1. Conventional structural formula of phenanthridone and the numbering of the different atoms.

Table 5. *Bond angles*

C(2)—C(1)—C(6)	120·3°	C(13)–N(14)–C(12)	125·0°
C(2)—C(1)—C(13)	119·2	C(1)—C(13)–O(15)	122·5
C(6)—C(1)—C(13)	120·3	C(1)—C(13)–N(14)	116·9
C(1)—C(2)—C(3)	122·4	H(16)–C(2)—C(1)	124·6
C(2)—C(3)—C(4)	117·6	H(16)–C(2)—C(3)	112·5
C(5)—C(4)—C(3)	120·6	H(17)–C(3)—C(2)	105·5
C(4)—C(5)—C(6)	122·2	H(17)–C(3)—C(4)	135·0
C(5)—C(6)—C(1)	116·7	H(18)–C(4)—C(5)	126·2
C(5)—C(6)—C(7)	123·1	H(18)–C(4)—C(3)	110·7
C(1)—C(6)—C(7)	120·2	H(19)–C(5)—C(4)	117·6
C(12)–C(7)—C(8)	118·0	H(19)–C(5)—C(6)	115·9
C(12)–C(7)—C(6)	117·6	H(20)–C(8)—C(9)	116·1
C(8)—C(7)—C(6)	124·4	H(20)–C(8)—C(7)	123·9
C(9)—C(8)—C(7)	119·9	H(21)–C(9)—C(8)	119·4
C(8)—C(9)—C(10)	123·6	H(21)–C(9)—C(10)	115·7
C(9)—C(10)–C(11)	116·8	H(22)–C(10)–C(9)	117·1
C(12)–C(11)–C(10)	120·0	H(22)–C(10)–C(11)	119·7
C(7)—C(12)–N(14)	119·9	H(23)–C(11)–C(12)	153·3
C(11)–C(12)–C(7)	121·6	H(23)–C(11)–C(10)	85·3
C(11)–C(12)–N(14)	118·4	H(24)–N(14)–C(13)	101·0
O(15)–C(13)–N(14)	120·6	H(24)–N(14)–C(12)	133·0

Table 6. *Some of the molecular approach distances*

The symbols I, II, III and IV indicate the symmetry positions as listed in *International Tables for X-ray Crystallography* (1969).

C(2, I)–C(8, III)	3·76 Å	C(9, I)—C(10, II)	3·68 Å
C(3, I)–C(11, IV)	3·72	C(9, I)—C(9, II)	3·74
C(3, I)–C(10, IV)	3·91	C(11, I)–O(15, II)	3·40
C(3, I)–C(9, III)	3·96	C(12, I)–O(15, II)	3·60
C(3, I)–C(8, III)	3·99	C(13, I)–N(14, II)	3·79
C(4, I)–O(15, III)	3·52	C(13, I)–O(15, II)	3·80
C(4, I)–C(11, IV)	3·76	N(14, I)–O(15, II)	2·86
C(4, I)–C(10, IV)	3·79	O(15, I)–O(15, II)	3·83
C(5, I)–O(15, III)	3·86	O(15, I)–O(15, III)	3·86
C(8, I)–C(10, II)	3·93		

the least-squares refinement and hence they are more likely to be found from difference synthesis.

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