9-*tert*-Butyl-9,10-dihydroacridine

By Shirley S. C. Chu and Robert D. Rosenstein

School of Engineering and Applied Science, Southern Methodist University, Dallas, Texas 75275, USA

Abstract. $C_{17}H_{19}N$, orthorhombic, $P2_12_12_1$, $Z = 4$, $M_r = 237.33$, $a = 11.119$ (1), $b = 5.823$ (1), $c = 20.325$ (2) Å, $V = 1316.05$ (38) Å³, $D_x = 1.198$ Mg m⁻³, λ(Cu $K\alpha$) = 1.5418 Å, μ(Cu $K\alpha$) = 0.531 mm⁻¹. Final $R = 0.057$. The *tert*-butyl group is in a boat-axial conformation with respect to the central acridine ring.

Introduction. Single crystals of the title compound (I) were obtained through the courtesy of Dr C. T. Taylor of the Chemistry Department of the University of Sheffield, Sheffield, England. The crystals are transparent prisms. The unit-cell parameters were obtained by measuring the 2θ values of 15 reflections. The space group, $P2_12_12_1$, was deduced from systematic absences ($h00$ absent with h odd, $0k0$ absent with k odd, and $00l$ absent with l odd). The intensity data were collected on a Syntex $P2_1$ automatic diffractometer with a crystal approximately $0.12 \times 0.59 \times 0.09$ mm in size. A $\theta/2\theta$ scanning mode with graphite-monochromated Cu $K\alpha$ radiation was used to measure 1326 independent reflections with 2θ values below 130°, of which 766 reflections were considered as observed by the criterion $I > 3.0\sigma(I)$, where $\sigma(I)$ was determined from counting statistics. The intensity data were reduced to structure amplitudes by the application of Lorentz and polarization factors, and no absorption corrections were applied.

(I)
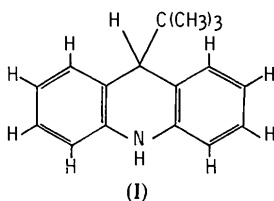
The structure was determined by the application of direct methods with the weighted multisolution tangent-refinement technique, *MULTAN* (Germain, Main & Woolfson, 1971). The E map showed the positions of all non-hydrogen atoms. The refinement was carried out by the full-matrix least-squares method (Busing, Martin & Levy, 1962) with isotropic temperature factors and the block-diagonal least-squares method (Shiono, 1971) with anisotropic temperature factors. Some H positions were located on a difference Fourier

0567-7408/79/020480-03$01.00

synthesis, and the rest were calculated with reasonable bond lengths and bond angles with respect to the atoms to which they are bonded. The isotropic temperature factors were used for the H atoms in the refinement. The weight of the reflection was assigned as $1/[\sigma(F)]^2$, where $\sigma(F)$ was calculated from counting statistics. The quantity $\sum w\{||F_o| - |F_c||\}^2$ was minimized. The final R index ($\sum ||F_o| - |F_c||/\sum |F_o|$) was 0.057.* The magnitude, $[\sum w(F_o - F_c)^2/(m - n)]^{1/2}$, where m is the number of reflections and n is the number of parameters refined, was 1.72. The atomic scattering factors used for N and C atoms were those from *International Tables for X-ray Crystallography* (1962). For H, the values given by Stewart, Davidson & Simpson (1965) were used. The final positional parameters are given in Table 1.

Discussion. The crystal structure of 9-*tert*-butyl-9,10-dihydroacridine is one of a series of structures of tricyclic compounds under study in this laboratory. The identification of the atoms and the configuration of the molecule are shown in Fig. 1. The bond lengths and bond angles with their standard deviations are shown in Fig. 2. The mean value of the C–N bond length is 1.414 (6) Å, and the C–N–C bond angle is 120.0 (4)°; these values are similar to those found in

---
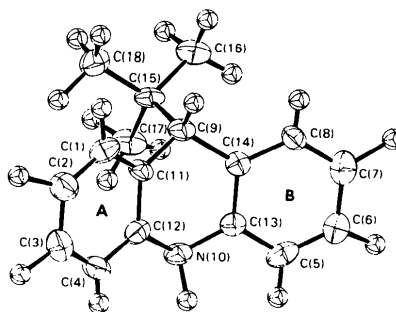
Fig. 1. *ORTEP* drawing (Johnson, 1965) of one molecule of 9-*tert*-butyl-9,10-dihydroacridine.

Table 1. *Fractional atomic coordinates* (×10⁴) *for non-hydrogen atoms and fractional coordinates* (×10³) *and thermal parameters for* H *atoms*

The estimated standard deviations are given in parentheses and refer to the last positions of respective values.

	x	y	z		x	y	z	B (Å²)
N(10)	3709 (4)	1133 (6)	1725 (2)	H(N)	367 (4)	28 (8)	219 (2)	8·7 (16)
C(1)	1813 (4)	6061 (9)	1271 (2)	H(1)	188 (3)	721 (6)	94 (2)	2·7 (10)
C(2)	740 (5)	5513 (9)	1561 (2)	H(2)	−18 (4)	610 (9)	146 (2)	8·6 (17)
C(3)	630 (5)	3468 (10)	1890 (2)	H(3)	−30 (4)	295 (7)	208 (2)	5·3 (13)
C(4)	1603 (4)	1977 (9)	1940 (2)	H(4)	154 (3)	60 (7)	216 (2)	4·1 (11)
C(5)	5847 (5)	1103 (9)	1972 (2)	H(5)	566 (3)	−14 (6)	226 (2)	2·2 (9)
C(6)	6952 (5)	2052 (10)	1954 (2)	H(6)	784 (4)	141 (9)	207 (2)	7·2 (14)
C(7)	7127 (5)	4125 (10)	1605 (2)	H(7)	805 (5)	486 (10)	153 (2)	9·1 (18)
C(8)	6137 (5)	5123 (8)	1307 (2)	H(8)	623 (5)	640 (9)	107 (2)	5·2 (15)
C(9)	3955 (5)	5059 (7)	912 (2)	H(9)	411 (4)	674 (7)	92 (2)	4·8 (10)
C(11)	2803 (4)	4596 (8)	1282 (2)	H(16)1	586 (4)	439 (8)	11 (2)	6·0 (14)
C(12)	2677 (4)	2539 (8)	1656 (2)	H(16)2	521 (5)	664 (11)	−20 (3)	11·0 (20)
C(13)	4867 (4)	2100 (8)	1664 (2)	H(16)3	510 (7)	436 (15)	−63 (3)	15·0 (31)
C(14)	5020 (4)	4108 (8)	1302 (2)	H(17)1	459 (4)	95 (9)	15 (2)	6·0 (14)
C(15)	3927 (5)	4238 (7)	178 (2)	H(17)2	354 (4)	109 (9)	−37 (2)	4·9 (15)
C(16)	5103 (5)	4994 (8)	−171 (2)	H(17)3	334 (4)	98 (7)	49 (2)	2·9 (11)
C(17)	3835 (6)	1593 (7)	126 (2)	H(18)1	210 (4)	467 (9)	1 (2)	5·8 (16)
C(18)	2855 (5)	5295 (9)	−180 (2)	H(18)2	292 (4)	494 (7)	−66 (2)	2·6 (11)
				H(18)3	288 (5)	700 (12)	−12 (3)	9·4 (20)
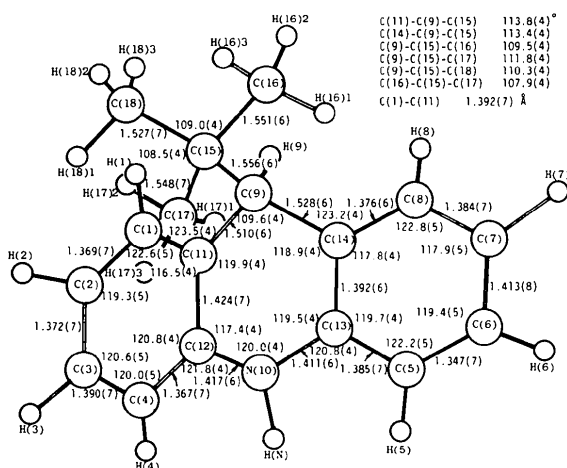
Fig. 2. Bond lengths (Å) and bond angles (°) of 9-*tert*-butyl-9,10-dihydroacridine with e.s.d.'s in parentheses.

the central ring of phenothiazine derivatives (Chu & van der Helm, 1976, 1977). In aromatic acridines, the C—N bond length has been found to be in the range 1·339 to 1·364 Å, and the C—N—C bond angle in the range 116·9 to 123·3° (Phillips, Ahmed & Barnes, 1960; Talacki, Carrell & Glusker, 1974; Zacharias & Glusker, 1974; Stallings & Glusker, 1977). The C(9)—C(15) bond length is 1·556 (6) Å which is significantly longer than the C—C single-bond length. This lengthening was also observed in 9-*tert*-butyl derivatives of 9,10-dihydroanthracene (Brennan, Putkey & Sundaralingam, 1971) and thioxanthene (Chu & Kou, 1978). The non-bonded intramolecular distances are 2·50, 2·54, 2·61, and 2·63 Å between C(8)—H(16)1,

N(10)—H(17)3, C(14)—H(16)1, and C(12)—H(17)3, respectively. The C—H bond lengths range from 0·89 to 1·14 Å (mean 1·01 Å) with a r.m.s. standard deviation of 0·05 Å. The C—C—H bond angles involving benzene rings range from 106 to 133° (mean 119°), and the C—C—H and H—C—H bond angles involving tetrahedral C atoms range from 102 to 115° (mean 109°). The standard deviation of these bond angles is 3°. The N—H bond length is 1·07 (5) Å and the mean value of the two C—N—H bond angles is 108 (3)°.

The equations of the least-squares planes of the two benzene rings are $0.2988x + 0.4565y + 0.8381z = 4.364$ and $-0.2123x + 0.5178y + 0.8288z = 2.273$ (where x, y, and z are in Å) for rings A and B (Fig. 1), respectively. The deviations of atoms from the planes range from 0·001 to 0·027 Å. The folding angle between the planes of the two benzene rings is 150·2°, compared with 139·7° in 9-*tert*-butylthioxanthene (Chu & Kou, 1978), 147° in 9-*tert*-butyl-9,10-dihydroanthracene (Brennan, Putkey & Sundaralingam, 1971), and 144° in 10,10'-dimethyl-9,9',10,10'-tetrahydro-9,9'-biacridinyl (Preuss, Zanker & Gieren, 1977). These differences can be attributed to the different bonding characteristics of the *meso* atoms of the tricyclic ring system. The magnitude of the folding angle is correlated to the size of the bond angle involving *meso* atoms. This is supported by the fact that the C—N—C bond angles of 120·0 and 116·9° in 9-*tert*-butyl-9,10-dihydroacridine and 10,10'-dimethyl-9,9',10,10'-tetrahydro-9,9'-biacridinyl, respectively, are larger than the C—S—C bond angle of 100·9° in 9-*tert*-butylthioxanthene.

The central ring is intermediate between a boat and a half-boat conformation as shown by the ring torsion angles given in Table 2. The Cremer & Pople (1975)

Table 2. *Torsion angles* (°) *in* 9-*tert-butyl*-9,10-*dihydroacridine*

Central ring

C(14)–C(9)–C(11)–C(12)	−34·7
C(9)–C(11)–C(12)–N(10)	7·4
C(11)–C(12)–N(10)–C(13)	24·3
C(12)–N(10)–C(13)–C(14)	−24·5
N(10)–C(13)–C(14)–C(9)	−7·1
C(13)–C(14)–C(9)–C(11)	34·6

tert-Butyl group

C(11)–C(9)–C(15)–C(16)	174·8
C(11)–C(9)–C(15)–C(17)	−65·7
C(11)–C(9)–C(15)–C(18)	55·0
C(14)–C(9)–C(15)–C(16)	−59·0
C(14)–C(9)–C(15)–C(17)	60·4
C(14)–C(9)–C(15)–C(18)	−178·9
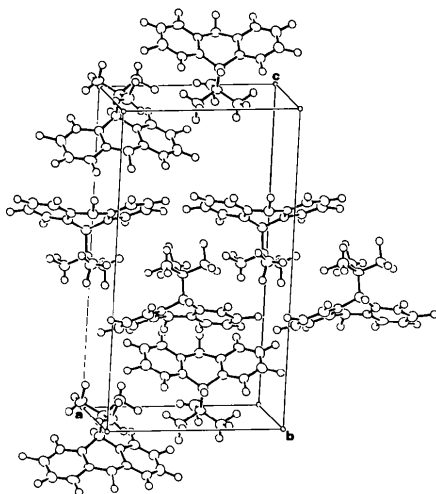
Fig. 3. The molecular packing of 9-*tert*-butyl-9,10-dihydroacridine in a unit cell.

puckering parameters for the central ring are $q_2 = 0.396$, $q_3 = −0.072$, $Q = 0.402$ Å, $\varphi_2 = 359.7$ and $\theta = 100.3°$. The *tert*-butyl group is in a 'boat-axial' conformation, in agreement with the result predicted by NMR (Taylor & Procter, 1971). The torsion angles about the C(9)–C(15) bond are also shown in Table 2. The *tert*-butyl group in the title compound has the same conformation as that in 9-*tert*-butylthioxanthene. The

N—H bond is in a quasi-equatorial conformation with respect to the central ring. The packing of the molecules in the crystal is shown in Fig. 3. There are no intermolecular contacts less than van der Waals distances.

References

BRENNAN, T., PUTKEY, E. F. & SUNDARALINGAM, M. (1971). *J. Chem. Soc. D*, pp. 1490–1491.

BUSING, W. R., MARTIN, K. O. & LEVY, H. A. (1962). *ORFLS*. Report ORNL-TM-305. Oak Ridge National Laboratory, Tennessee.

CHU, S. S. C. & VAN DER HELM, D. (1976). *Acta Cryst.* B**32**, 1012–1016.

CHU, S. S. C. & VAN DER HELM, D. (1977). *Acta Cryst.* B**33**, 873–876.

CHU, S. S. C. & KOU, W. (1978). *Acta Cryst.* B**34**, 2348–2350.

CREMER, D. & POPLE, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.

GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1971). *Acta Cryst.* A**27**, 368–376.

International Tables for X-ray Crystallography (1962). Vol. III, pp. 201–207. Birmingham: Kynoch Press.

JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.

PHILLIPS, D. C., AHMED, F. R. & BARNES, W. H. (1960). *Acta Cryst.* **13**, 365–377.

PREUSS, V. J., ZANKER, V. & GIEREN, A. (1977). *Acta Cryst.* B**33**, 2317–2319.

SHIONO, R. (1971). Tech. Rep. 49, Crystallography Department, Univ. of Pittsburgh.

STALLINGS, W. C. & GLUSKER, J. P. (1977). *Acta Cryst.* B**33**, 1927–1934.

STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

TALACKI, R., CARRELL, H. L. & GLUSKER, J. P. (1974). *Acta Cryst.* B**30**, 1044–1047.

TAYLOR, G. A. & PROCTER, S. A. (1971). *J. Chem. Soc. C*, pp. 2537–2538.

ZACHARIAS, D. E. & GLUSKER, J. P. (1974). *Acta Cryst.* B**30**, 2046–2049.