

e.s.d.'s in the positional and thermal parameters. The ester group adopts a *cis* bisected conformation with torsion angle $X(1)-C(1)-C(4)-O(1)$ of $1(1)^\circ$; $X(1)$ is the midpoint of $C(2)-C(3)$, distal to $C(1)$ as defined by Allen (1980). This conformation allows optimum conjugation between the $C=O$ of the ester group and the cyclopropane ring. The phenyl substituent adopts the bisected conformation with a torsion angle $X(2)-C(2)-C(7)-C(8)$ of $-2(1)^\circ$; $X(2)$ is the midpoint of $C(1)-C(3)$, distal to $C(2)$. Sterically similar derivatives such as *trans*-2-(*p*-tolyl)cyclopropanoic acid (Ramírez *et al.*, 1990) and *trans*-(*p*-nitrophenyl)cyclopropyl methyl ketone (Bordner *et al.*, 1972) also exhibit this conformation (the torsion angle is 5.1 and 4.8° , respectively). Thus, both phenyl and ester groups withdraw electron density from the cyclopropane ring. These electronic interactions should induce an asymmetric pattern in the cyclopropane ring (Hoffmann, 1970; Allen, 1980). However, this cannot be detected within the limits of experimental error here: the cyclopropane ring is an equilateral triangle (Table 2).

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Structure of 9-Pivaloylfluorene

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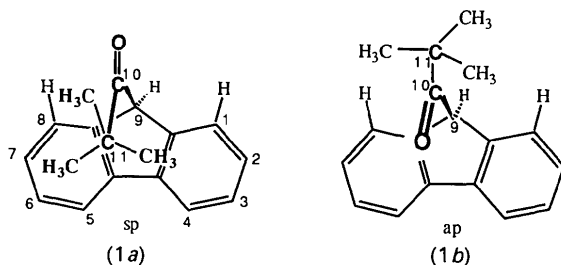
Abstract. $C_{18}H_{18}O$, $M_r = 250.34$, orthorhombic, $P2_12_12_1$, $a = 14.051(2)$, $b = 15.769(3)$, $c = 6.398(4)$ Å, $V = 1417.6(8)$ Å³, $Z = 4$, $D_x = 1.17$ g cm⁻³, $\lambda(Mo K\alpha) = 0.71069$ Å, $\mu = 0.66$ cm⁻¹, $F(000) = 536$, $T = 296$ K, $R = 0.046$ for 896 unique reflections. Rotationally hindered 9-pivaloylfluorene (I) was prepared in good yield from the reaction of fluorene with *n*-butyllithium followed by pivaloyl chloride. ¹H NMR spectra of all fractions of chromatographed (I) were identical and suggested a single rotamer in solution, the same one shown in the

crystal structure: $H(9)-C(9)-C(10)-O(1)$ torsion angle $\sim 180^\circ$, the antiperiplanar rotamer, (Ib).

Introduction. During our initial investigation comparing the reactions of 9-methyl-9-keto-substituted fluorenes with *n*-butyllithium versus Grignard reagents (Meyers, Arnold & Wahner, 1975; Arnold, Meyers & Wahner, 1975), previously unreported 9-pivaloylfluorene (I) was prepared but set aside. Darling Molecular Models[®], equipped with space-filling H atoms and *p* lobes, suggested that the significant rotational restriction around the $C(9)-C(10)$ bond of (I) might allow it to exist in solution at ambient temperatures as two distinct rotamers, (Ia) (*sp*) and

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(1*b*) (*ap*)* or as only one of them if there were a large difference in their respective rotational barriers. Ōki *et al.* have succeeded in separating, identifying and studying the reactivities of the *ap* and *sp* rotamers of several 9-monoarylfluorenes (Sonoda, Tsukahara, Nakamura & Ōki, 1989, and earlier references cited therein); Nishida *et al.* have investigated rotational isomerism in various 9-substituted fluorenes (Nishida, Takeshita, Fujisaki & Kajjgaeshi, 1988, and earlier references cited therein); Ōki has reviewed this area (Ōki, 1985). However, little attention, if any, along these lines has been given to rotationally hindered 9-acyl-substituted fluorenes like (I).



In our continuing study (Manohar, 1986; Meyers & Manohar, 1987) a sample of (I) was used in the attempted preparation of 9-methyl-9-pivaloylfluorene [(I) $\xrightarrow{t\text{-BuLi}}$ (I)-9-Li $\xrightarrow{\text{CH}_3\text{I}}$]. The expected simple methylation failed, although it was demonstrated that (I)-9-Li had indeed been formed: quenching a portion of the reaction mixture with H_2O led to recovery of (I), while quenching another portion with D_2O provided (I)-9*d*. Melting points, mixed melting points and ^1H NMR (CDCl_3) spectra indicated that substrate (I), and (I) and (I)-9*d* obtained from the quenching of (I)-9-Li were structurally identical. The NMR spectra showed a single or time-averaged conformation in solution. Failure of methylation was attributed to steric hindrance of reagent approach, but it is more likely associated with a high-energy conformation required of the transition state.

With respect to the conformational structure of (I), these data allowed useful but limited conclusions. Formation or equilibration of two stable rotamers under ambient conditions was ruled out by the solution NMR data. Thus, two reasonable possibilities remained: (1) rotamer interconversion in solution is rapid and crystallization affords a single rotamer; or (2) there is a large difference in the respective barriers to rotation which enables one rotamer to predominate exclusively under ambient conditions. While a detailed NMR study would serve to elimi-

nate one of these two possibilities, X-ray crystallography is required to identify the single rotamer in the crystalline state.

Experimental. Preparation of 9-pivaloylfluorene (I). To a stirred solution of fluorene (12.05 mmol, 2.00 g, dried *in vacuo*; m.p. 388–389 K) in anhydrous oxygen-free tetrahydrofuran* (12 ml) in a septum-sealed flask maintained under argon at 210 K (ethanol/liquid-nitrogen bath), *n*-butyllithium (14.57 mmol, 9.4 ml of a 1.55 M solution in hexane) was added *via* syringe. The resulting brown suspension was stirred for 20 min after which time a solution (prepared under argon) of pivaloyl chloride (14.57 mmol, 1.8 ml) in anhydrous oxygen-free THF (6 ml) was added in a single portion *via* syringe. The solution became bright yellow immediately and solidified within 20 min (210 K). The cooling bath was removed and the mixture, which liquefied at *ca* 243 K, was stirred for 90 min. A sample withdrawn *via* syringe and directly assayed by TLC (9:1 hexane-ether) contained (I) (single spot), 9-fluorenone and residual fluorene. The solution was diluted with water (the yellow color faded) and extracted with ether. The extract (water-washed, dried and vacuum-evaporated at room temperature) provided 2.78 g of yellow crystals which were flash chromatographed (column, silica gel 60, 200–400 mesh). Initial elution with petroleum ether provided only fluorene (0.63 g, 32% recovery). Subsequent elution with 19:1 petroleum ether-ether provided the remaining fractions, all shown by TLC to contain (I) almost exclusively (a single spot), with traces of 9-fluorenone [yellow spot just below (I)]. Overnight evaporation of these fractions provided white microneedles, isolated by filtration. The filtrates, allowed to evaporate even more slowly, afforded long thick colorless needles, used for the X-ray crystallographic study. Total yield of (I), 1.93 g (64%; or 94% based on unrecovered fluorene), m.p. 440.5–441.5 K, uncorr. ^1H NMR (Varian VXR 500 MHz, CDCl_3): δ 7.764 [*d*, H-4 (H-5), $J_{43(56)} = 7.5$ Hz, 2 H]; 7.382 [*dd*, H-3 (H-6) $J_{32(67)} = 7.5$ Hz, $J_{34(65)} = 7.5$ Hz, 2 H]; 7.367 [*d*, H-1 (H-8), $J_{12(87)} = 7.0$ Hz, 2H]; 7.267 [split *dd*, H-2 (H-7), $J_{23(76)} = 7.5$ Hz, $J_{21(78)} = 7.0$ Hz, $J_{24(75)} = 1.5$ Hz, 2 H]; 5.38 (*s*, H-9, 1 H) and 1.28 (*s*, CH_3 , 9 H). The identical melting point and spectrum were exhibited by (I) isolated from the various chromatographic fractions. Analysis (MicAnal) calc. for $\text{C}_{18}\text{H}_{18}\text{O}$: C, 86.36; H, 7.25%; found: C, 86.17; H, 7.05%.

Crystal used for data collection $0.20 \times 0.20 \times 0.22$ mm, clear colorless prism cut from larger crystal. Rigaku AFC5S diffractometer, graphite-monochromated $\text{Mo K}\alpha$ radiation, ω - 2θ scans, scan

* The designations *sp* (synperiplanar) and *ap* (antiperiplanar) for these conformations are in accord with Rule E-6.6, *IUPAC Tentative Rules* (IUPAC, 1970).

* THF refluxed with Na/benzophenone and distilled from the deep-blue solution.

speed 4° min^{-1} , maximum of three scan repetitions to obtain $\sigma F/F < 0.10$. Lattice parameters from least-squares fit of 24 strong reflections in 2θ range $31\text{--}39^\circ$. A total of 1467 reflections measured (h 0 to 16, k 0 to 18, l 0 to 7), 571 reflections considered unobserved with $[I < 2\sigma(I)]$, data set comprised of 896 unique reflections, $(\sin\theta/\lambda)_{\text{max}} = 0.60 \text{ \AA}^{-1}$. Three standard reflections (011 , 121 , $12\bar{1}$) changed by -0.2 , 1.4 and -0.1% , respectively; no decay correction applied. Data corrected for Lorentz and polarization (no absorption correction). Direct methods provided the locations of all non-H atomic positions. Full-matrix least-squares refinement was performed to minimize $\sum w(|F_o| - |F_c|)^2$ where $w = 4F_o^2/\sigma^2(F_o^2)$ and $\sigma^2(F_o^2) = [S^2(C + R^2B) + (pF_o^2)^2]/Lp^2$ (S = scan rate, C = total integrated peak count, R = ratio of scan time to background counting time, B = total background count, Lp = Lorentz-polarization factor, and $p = 0.05$). Ring H atoms placed in geometrically assumed positions ($C\text{--}H = 0.95 \text{ \AA}$) and fixed, methyl H-atom sites located from difference Fourier synthesis, mathematically optimized (tetrahedral geometry, $CH\text{--}H = 0.95 \text{ \AA}$) and fixed. The H(9) atomic site was obtained from a difference Fourier synthesis. Final stage of refinement performed with 177 variables including all non-H positional and anisotropic thermal parameters, one isotropic H atom [H(9)], a secondary-extinction coefficient (0.41676×10^{-5}) and one scale factor. Convergence yielded $R = 0.046$, $wR = 0.056$, $S = 1.36$ and $(\Delta/\sigma)_{\text{max}} = 0.00$. Final difference synthesis produced $(\Delta\rho)_{\text{max}} = 0.15$ and $(\Delta\rho)_{\text{min}} = -0.15 \text{ e \AA}^{-3}$. Atomic scattering factors and anomalous-dispersion corrections from Cromer & Waber (1974). All computer programs from the TEXSAN crystal structure analysis package (Molecular Structure Corporation, 1985).

Discussion. Table 1 lists atomic coordinates and equivalent isotropic temperature factors. Table 2 contains a compilation of selected bond distances and angles.* The crystal structure shows a single rotamer (Fig. 1) in which the *tert*-butyl methyl groups are in close proximity to H(1), H(8) and H(9), and whose H(9)—C(9)—C(10)—O(1) torsion angle is $\sim 180^\circ$, *i.e.* the *ap* rotamer, (Ib). In this structure the methyl hydrogens can approach H(9) within a distance less than the sum of van der Waal's radii of two H atoms, but they are considerably more distant from

* Lists of observed and calculated structure factors, calculated H-atom coordinates and temperature factors, anisotropic thermal parameters for the non-H atoms, torsion or conformation angles, selected least-squares planes, and intermolecular distances involving non-H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53593 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Fractional atomic coordinates and equivalent isotropic temperature factors

	$B_{\text{eq}} = (8\pi^2/3) \text{ trace } U.$			
	x	y	z	$B_{\text{eq}}(\text{\AA}^2)$
O(1)	0.2081 (3)	0.6784 (2)	0.4482 (5)	4.5 (2)
C(1)	0.0277 (4)	0.8441 (3)	0.381 (1)	5.0 (3)
C(2)	-0.0359 (4)	0.8822 (4)	0.514 (1)	5.8 (4)
C(3)	-0.0027 (5)	0.9274 (4)	0.686 (1)	5.9 (4)
C(4)	0.0913 (4)	0.9352 (3)	0.730 (1)	4.9 (3)
C(4a)	0.1551 (3)	0.8949 (3)	0.597 (1)	3.6 (3)
C(4b)	0.2596 (4)	0.8946 (3)	0.601 (1)	4.0 (3)
C(5)	0.3225 (4)	0.9300 (3)	0.740 (1)	5.2 (3)
C(6)	0.4203 (4)	0.9208 (4)	0.707 (1)	5.5 (3)
C(7)	0.4526 (5)	0.8746 (4)	0.536 (1)	5.6 (4)
C(8)	0.3879 (4)	0.8385 (3)	0.396 (1)	4.2 (3)
C(8a)	0.2947 (4)	0.8487 (3)	0.4267 (8)	3.7 (2)
C(9)	0.2081 (4)	0.8160 (3)	0.3004 (7)	3.5 (2)
C(9a)	0.1261 (4)	0.8505 (3)	0.423 (1)	3.6 (2)
C(10)	0.2088 (4)	0.7185 (3)	0.2892 (7)	3.2 (2)
C(11)	0.2090 (4)	0.6758 (3)	0.0745 (6)	4.0 (2)
C(12)	0.1195 (5)	0.7049 (4)	-0.0453 (9)	5.9 (3)
C(13)	0.2064 (5)	0.5796 (3)	0.1030 (9)	6.2 (3)
C(14)	0.2977 (5)	0.7029 (4)	-0.0444 (8)	6.0 (3)
H(9)	0.214 (3)	0.841 (2)	0.160 (6)	3.2 (8)*

* Isotropic B .

Table 2. Selected bond distances (\AA), bond angles ($^\circ$) and their e.s.d.'s

O(1)—C(10)	1.198 (5)	C(6)—C(7)	1.390 (8)
C(1)—C(2)	1.372 (8)	C(7)—C(8)	1.399 (8)
C(1)—C(9a)	1.411 (8)	C(8)—C(8a)	1.334 (8)
C(2)—C(3)	1.390 (8)	C(8a)—C(9)	1.549 (7)
C(3)—C(4)	1.355 (7)	C(9)—C(9a)	1.496 (7)
C(4)—C(4a)	1.387 (7)	C(9)—C(10)	1.539 (6)
C(4a)—C(4b)	1.469 (6)	C(10)—C(11)	1.530 (6)
C(4a)—C(9a)	1.378 (8)	C(11)—C(12)	1.543 (8)
C(4b)—C(5)	1.375 (7)	C(11)—C(13)	1.527 (7)
C(4b)—C(8a)	1.416 (8)	C(11)—C(14)	1.522 (8)
C(5)—C(6)	1.398 (7)	C(9)—H(9)	0.99 (4)
C(2)—C(1)—C(9a)	119.2 (6)	C(8a)—C(9)—C(10)	110.6 (4)
C(1)—C(2)—C(3)	119.7 (5)	C(9a)—C(9)—C(10)	113.2 (4)
C(2)—C(3)—C(4)	122.5 (6)	C(8a)—C(9)—H(9)	106 (3)
C(3)—C(4)—C(4a)	117.5 (6)	C(9a)—C(9)—H(9)	114 (3)
C(4)—C(4a)—C(4b)	129.7 (6)	C(10)—C(9)—H(9)	111 (2)
C(4)—C(4a)—C(9a)	122.4 (5)	C(1)—C(9a)—C(4a)	118.7 (5)
C(4b)—C(4a)—C(9a)	107.8 (6)	C(1)—C(9a)—C(9)	128.9 (5)
C(4a)—C(4b)—C(5)	130.6 (6)	C(4a)—C(9a)—C(9)	112.4 (5)
C(4a)—C(4b)—C(8a)	109.8 (6)	O(1)—C(10)—C(9)	119.2 (4)
C(5)—C(4b)—C(8a)	119.5 (5)	O(1)—C(10)—C(11)	122.0 (4)
C(4b)—C(5)—C(6)	119.6 (6)	C(9)—C(10)—C(11)	118.8 (4)
C(5)—C(6)—C(7)	119.5 (6)	C(10)—C(11)—C(12)	108.3 (5)
C(6)—C(7)—C(8)	120.4 (6)	C(10)—C(11)—C(13)	109.3 (4)
C(7)—C(8)—C(8a)	119.6 (5)	C(10)—C(11)—C(14)	109.1 (5)
C(4b)—C(8a)—C(8)	121.3 (5)	C(12)—C(11)—C(13)	109.6 (5)
C(4b)—C(8a)—C(9)	107.8 (5)	C(12)—C(11)—C(14)	109.6 (4)
C(8)—C(8a)—C(9)	130.9 (5)	C(13)—C(11)—C(14)	111.0 (5)
C(8a)—C(9)—C(9a)	102.1 (3)		

H(1) and H(8) (Table 3) which enhances the stability of this conformation.

The 500 MHz ^1H NMR spectra in CDCl_3 (298 K) of all the fractions of chromatographed (I) were identical and show a single structure which could represent either one rotamer or the rotationally time-averaged structure. The chemical shifts of the respective aromatic protons of (I) and those assigned

to fluorene itself (Pretsch, Seibl, Simon & Clerc, 1983) are very similar, with a single exception: H(1), H(8) of (I) at $\delta = 7.37$ are substantially *shielded* compared with the same protons of fluorene at $\delta = 7.55$ (also in CDCl_3). At the same time the *tert*-butyl protons, a singlet at $\delta = 1.28$, are substantially *deshielded* compared with those of other *tert*-butyl ketones, *e.g.* $\delta = 1.15$ for *tert*-butyl methyl ketone (in CDCl_3). We have found (Manohar, 1986) that 9-methyl-9-fluorenyl *n*-alkyl ketones exhibit the 9-methyl-proton singlet at exceptionally low field because these protons reside in the *deshielding* zone of the fluorene plane, while the β , γ and δ protons of the alkyl group resonate at exceptionally high fields because they have a significant residence time in the π -*shielding* zone of the fluorene plane (the *sp* conformation) presumably imposed by the steric effect of the 9-methyl group. These data suggest that at ambient temperature (I) exists in solution as the single *ap* rotamer (Ib), the same one shown by the X-ray crystal structure.

Rotational constraint around C(9)—C(10) of (I) anion $[\text{C}(9)^-]$ is also apparent and does not permit the geometry required for the enolate stabilization largely responsible for the much lower $\text{p}K_a$ values of other 9-keto-substituted fluorenes.* The fact that (I) is still more acidic than fluorene itself by more than 8 $\text{p}K_a$ units suggests that much of the strain energy in

* The authors are grateful to F. G. Bordwell and J. A. Harrelson for determining the $\text{p}K_a$ of (I) under their standard conditions (DMSO, 298 K) for comparison with their related fluorenes: fluorene (Fl), 22.6; 9-NH₂C(O)Fl, 11.9; 9-MeOC(O)Fl, 10.35; 9-PhC(O)Fl, 10.0; 9-CNFl, 8.3. This work will be described in another paper.

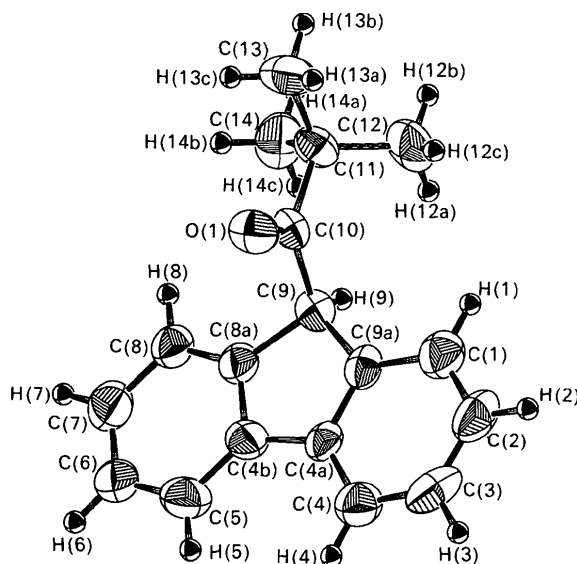


Fig. 1. Molecular configuration and IUPAC atom-numbering scheme, thermal ellipsoids at the 50% probability level. H atoms are shown as isotropic spheres with B 's of 1.0 \AA^2 .

Table 3. Selected intramolecular nonbonding distances between H atoms (\AA)

H(1)···H(12c)	2.59	H(9)···H(14c)	2.21
H(1)···H(12a)	2.74	H(9)···H(12a)	2.27
H(8)···H(14b)	2.61	H(9)···H(14b)	3.23
H(8)···H(14c)	2.78	H(9)···H(12c)	3.30

(I) is released by the removal of H(9). Residual steric strain in the (I) anion is reflected by the exceptional ease of autoxidative cleavage of (I) into strain-free 9-fluorenone in dissociating solvents (*e.g.* *tert*-butyl alcohol), which is almost immediate in the presence of base. Sterically encumbered α -sulfonyl carbanions perform similarly (Chan-Yu-King, 1986; Meyers, Chan-Yu-King, Likibi & McCollum, 1985; Meyers, Likibi, Chan-Yu-King & McCollum, 1985).

Dynamic ^1H and ^{13}C NMR studies of (I), (I)-9-Li and related structures are now underway.

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