The two different crystal forms of rhodochlorin XV dimethyl ester described here present an example of homoeotypes of a chlorin. Other cases of multiple crystal forms of tetrapyrroles include Ni^{II} octaethylporphyrin. This porphyrin has been found in two triclinic and one tetragonal form (Meyer, 1972; Cullen & Meyer, 1974; Brennan, Scheidt & Shelnutt, 1988). An example of a chlorin with two crystal forms is 8-isobutyl-12-ethyl methyl bacteriopheophorbide d, which has been described by Barkigia, Chantranupong, Smith & Fajer (1988). The observation of two crystalline phases for chlorins points to the conformational flexibility inherent to this type of molecule. It also shows that a given conformation, observed in the crystal, might not necessarily be the only one possible, and can be influenced by packing effects.

Financial support by the Deutsche Forschungsgemeinschaft (Se543/1-1) and the National Science Foundation (NSF CHE-90-01381) is gratefully acknowledged.

References

Barkigia, K. M., Chantranupong, L., Smith, K. M. & Fajer, J. (1988). *J. Am. Chem. Soc.* **110**, 7599–7582.

Brennan, T. D., Scheidt, W. R. & Shelnutt, J. A. (1988). *J. Am. Chem. Soc.* **110**, 3919–3924.

Cullen, D. L. & Meyer, E. F. Jr (1974). *J. Am. Chem. Soc.* **96**, 2095–2102.

Deisenhofer, J. & Michel, H. (1988). *Angew. Chem. Int. Ed. Engl.* **28**, 829–847.

Fischer, M. S., Templeton, D. H., Zalkin, A. & Calvin, M. (1972). *J. Am. Chem. Soc.* **94**, 3613–3619.

Hope, H. & Moezzi, B. (1987). *XABS*. Univ. of California, Davis, USA.

Hoppe, W., Will, G., Gassmann, J. & Weichselgartner, H. (1969). *Z. Kristallogr.* **128**, 18–35.

Hynninen, P. H. (1991). *Chlorophylls*, edited by H. Scheer, pp. 145–209. Boca Raton: CRC Press.

Kratky, C. & Dunitz, J. D. (1977). *J. Mol. Biol.* **113**, 431–442.

Meyer, E. F. Jr (1972). *Acta Cryst.* **B28**, 2162–2167.

Scheidt, W. R. & Lee, Y. J. (1987). *Struct. Bonding*, **64**, 1–70.

Senge, M. O. & Smith, K. M. (1991). *Photochem. Photobiol.* **54**, 841–846.

Senge, M. O. & Smith, K. M. (1992). *Z. Kristallogr.* **199**, 239–248.

Sheldrick, G. M. (1990). *SHELXTL-Plus*. Program for crystal structure solution. Univ. of Göttingen, Germany.

Smith, K. M. (1991). *Chlorophylls*, edited by H. Scheer, pp. 115–143. Boca Raton: CRC Press.

Smith, K. M., Goff, D. A., Fajer, J. & Barkigia, K. M. (1982). *J. Am. Chem. Soc.* **104**, 3747–3749.

Smith, K. M., Goff, D. A. & Simpson, D. J. (1985). *J. Am. Chem. Soc.* **107**, 4946–4954.

Smith, K. M. & Lewis, W. M. (1981). *Tetrahedron*, **37** (Suppl. 1), 399–403.

Tronrud, D. E., Schmid, M. F. & Matthews, B. W. (1985). *J. Mol. Biol.* **188**, 443–545.

Acta Cryst. (1992). C**48**, 1815–1818

Structure of *sp*-9-Hydroxy-9-pivaloylfluorene, Product of Base-Catalyzed Autoxidation of *ap*-9-Pivaloylfluorene

By Cal Y. Meyers* and Jennifer L. Tunnell

Department of Chemistry, Southern Illinois University, Carbondale, Illinois 62901, *USA*

Paul D. Robinson*

Department of Geology, Southern Illinois University, Carbondale, Illinois 62901, *USA*

and Duy H. Hua and Shankar Saha

Department of Chemistry, Kansas State University, Manhattan, Kansas 66506, *USA*

(*Received* 2 *October* 1991; *accepted* 11 *February* 1992)

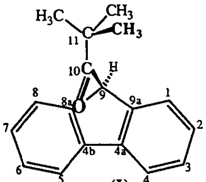
Abstract. 1-(9-Hydroxyfluoren-9-yl)-2,2-dimethyl-1-propanone, $C_{18}H_{18}O_2$, $M_r = 266.34$, orthorhombic, *Pbcn*, $a = 18.917\,(10)$, $b = 11.843\,(8)$, $c = 13.177\,(7)$ Å, $V = 2952\,(5)$ Å^3, $Z = 8$, $D_x = 1.198$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 0.72$ cm^{-1}, $F(000) = 1136$, $T = 296$ K, $R = 0.044$ for 1042 unique observed reflections. Conversion of *ap*-9-pivaloylfluorene, *ap*-(I), into lithiated (I)-9-anion followed by the addition of MeOH, then H_2O, led to unexpected hydroxylation to provide 20–40% of *sp*-9-hydroxy-9-pivaloylfluorene, *sp*-(II), and 60–80% recovery of *ap*-(I). The singular *sp* conformation of (II) in solution suggested by NMR was confirmed in the crystalline state by X-ray diffraction which showed the O(1)—C(9)—C(10)—O(2) torsion angle

---

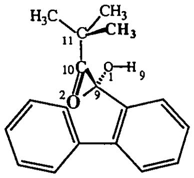
* Correspondence relating to the crystallography should be sent to PDR; that relating to the chemistry, to CYM.

~ 0° and the C(10)=O(2)···H(9)—O(1) non-bonding distance 1.95 (4) Å, suggesting strong intramolecular hydrogen bonding in this conformation.

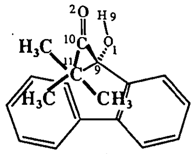
Introduction. We recently reported that treatment of lithiated 9-fluorenyl anion with pivaloyl chloride leads to the formation of *ap*-9-pivaloylfluorene, *ap*-(I), as the exclusive rotamer in solution as well as in the crystalline state (Meyers, Wahner, Manohar, Carr, Chan-Yu-King & Robinson, 1991).* Quenching an ether solution of lithiated (I)-9-anion under argon at 273 K with D_2O provided *ap*-(I)-9-*d* exclusively (Meyers & Manohar, 1987; Manohar, 1986). Desiring a larger amount of *ap*-(I)-9-*d* we repeated the reaction but quenched it with MeOD. While *ap*-(I)-9-*d* was obtained, ~ 30% of an unexpected new crystalline compound was also isolated whose structure was suggested by ^1H and ^{13}C NMR to be 9-hydroxy-9-pivaloylfluorene, (II). Likewise, (II) was formed in similar yield, along with *ap*-(I), when MeOH was used instead of MeOD. As illustrated, two rotameric structures may be presented for (II).



ap-(I)



ap-(II)



sp-(II)

A priori, 9-hydroxylation of *ap*-(I) through its lithiated 9-anion might be expected to provide the *ap* rotamer of (II): 9-protonation and 9-deuteration of the same lithiated anion were rotamer specific in providing only *ap*-(I) and *ap*-(I)-9-*d*, respectively; molecular mechanics calculations indicated that *ap*-(I) and *ap*-(II) are significantly favored over their *sp* counterparts in which the large steric interaction between the *tert*-butyl group and the fluorene-ring π electrons imparts very much higher energies; and experimental support for the calculated results was provided by the report that 9-hydroxy-9-(2′-*tert*-butylphenyl)fluorene, which is functionally and sterically similar to (II), was prepared and identified as the thermodynamically favored rotamer conformationally equivalent to *ap*-(II) (Nakamura, Nakamura & Ōki, 1977*a*,*b*).

Solution ^1H NMR data, however, implied the opposite. Compared with the sharp lower field signal (δ 1.28) of the deshielded *tert*-butyl protons of *ap*-(I)(Meyers, Wahner, Manohar, Carr, Chan-Yu-King & Robinson, 1991), the much higher field sharp signal (δ 0.78) of the *tert*-butyl protons of (II) correlated well with the enforced fluorene-ring shielding which would be expected of the *sp* rotamer of (II) (*viz.* Nakamura, Nakamura & Ōki, 1977*a*,*b*). Unequivocal molecular and conformational structure determination required X-ray crystal analysis.

Experimental. Preparation of *sp*-9-hydroxy-9-pivaloylfluorene, *sp*-(II): to a stirred solution of *ap*-(I) (Meyers, Wahner, Manohar, Carr, Chan-Yu-King & Robinson, 1991) (25 mg, 0.10 mmol) in 1 ml of ether maintained at 273 K under argon, *n*-butyllithium (0.11 mmol, 0.098 ml of a 1.12 *M* solution in hexane) was added; the solution turned yellow immediately and stirring was continued for 25 min; methanol (reagent grade; 0.1 ml, 3 mmol) was added and the color faded; the solution was stirred at 273 K for 30 min, diluted with 50 ml of ether, washed with water then brine, dried (anhydrous $MgSO_4$) and rotary vacuum evaporated leaving a crystalline residue, 25 mg. TLC: two well-defined spots, the one of higher R_f value being identical to that from *ap*-(I). ^1H NMR: mixture, composed of *ca* 70% of *ap*-(I) and 30% of a compound subsequently identified by X-ray analysis as *sp*-(II). Column chromatography afforded recovery of *ap*-(I) (19 mg, 76%) followed by isolation of *sp*-(II); white crystals [6 mg, 23% conversion or quantitative yield based on unrecovered *ap*-(I)], m.p. 377.5–378.5 K. IR(conc., $CDCl_3$), ν: 3428 (sharp, OH str.); 1685 cm^{-1} (sharp, strong, C=O str.). ^1H NMR (Bruker WM-400, 400 MHz, $CDCl_3$), δ: 7.69 (*dd*, $J_{43,56} = 7.5$, $J_{42,57} = 1.2$ Hz, 2 H, H-4,5); 7.42 (*ddd*, $J_{32,67} = 7.5$, $J_{34,65} = 7.5$, $J_{31,68} = 1.2$ Hz, 2 H, H-3,6); 7.29 (*ddd*, $J_{23,76} = 7.5$, $J_{21,78} = 7.5$, $J_{24,75} = 1.2$ Hz, 2 H, H-2,7); 7.24 (*dd*, $J_{12,87} = 7.5$, $J_{13,86} = 1.2$ Hz, 2 H, H-1,8); 5.59 (*s*, 1 H, OH); 0.78 (*s*, 9 H, CH_3). ^{13}C NMR (Bruker WM-400, 100 MHz for ^{13}C, $CDCl_3$), δ: 214.98 (C=O); 144.75 (C-4a,4b or 8a,9a); 142.04 (C-8a,9a or 4a,4b); 129.75 (C-3,6); 128.18 (C-2,7); 124.42 (C-1,8); 120.61 (C-4,5); 87.2 (C-9); 44.5 [$\underline{C}(CH_3)_3$]; 28.05 [$C(\underline{C}H_3)_3$]. A DEPT experiment resulted in no signal at δ 87.2, indicating that it represented a quaternary C atom in support of its assignment to C-9 and the proposed structure. A comparison DEPT experiment on *ap*-(I) resulted in a positive signal at δ 57.1 assigned to C-9, which is monoprotonated.

Similar results were obtained when CH_3OD was used instead of CH_3OH in the quenching step, except that much of the recovered starting material contained 9-*d*. Yields of *sp*-(II) from several runs varied but were similar.

The crystal used for data collection was a colorless fragment with dimensions 0.27 × 0.30 × 0.35 mm. Data were collected with a Rigaku AFC-5S diffrac-

---

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

Table 1. *Fractional atomic coordinates and equivalent isotropic temperature factors* (Å^2)

$B_{eq} = (8\pi^2/3)$ trace **U**. Atom H(9) was refined isotropically.

	x	y	z	B_{eq}
O(1)	0.3783 (2)	0.1186 (3)	−0.1410 (2)	5.6 (2)
O(2)	0.5093 (2)	0.1250 (3)	−0.1089 (3)	7.5 (2)
C(1)	0.3942 (2)	0.3781 (4)	−0.1846 (3)	5.0 (2)
C(2)	0.3603 (2)	0.4800 (4)	−0.2041 (4)	5.6 (3)
C(3)	0.3083 (2)	0.5195 (4)	−0.1405 (4)	5.3 (3)
C(4)	0.2885 (2)	0.4586 (4)	−0.0552 (4)	4.6 (2)
C(4a)	0.3221 (2)	0.3565 (3)	−0.0360 (3)	3.6 (2)
C(4b)	0.3130 (2)	0.2755 (3)	0.0468 (3)	3.6 (2)
C(5)	0.2714 (2)	0.2796 (4)	0.1336 (3)	4.5 (2)
C(6)	0.2761 (2)	0.1917 (5)	0.2026 (3)	5.4 (3)
C(7)	0.3204 (2)	0.1008 (4)	0.1856 (4)	5.1 (3)
C(8)	0.3618 (2)	0.0961 (3)	0.0990 (4)	4.7 (2)
C(8a)	0.3580 (2)	0.1841 (3)	0.0305 (3)	3.6 (2)
C(9)	0.3988 (2)	0.2003 (3)	−0.0675 (3)	3.8 (2)
C(9a)	0.3744 (2)	0.3164 (3)	−0.1008 (3)	3.7 (2)
C(10)	0.4796 (2)	0.1917 (4)	−0.0533 (3)	4.3 (2)
C(11)	0.5232 (2)	0.2604 (3)	0.0213 (3)	3.9 (2)
C(12)	0.5746 (3)	0.3296 (5)	−0.0417 (4)	10.4 (4)
C(13)	0.5637 (3)	0.1803 (4)	0.0887 (4)	9.7 (4)
C(14)	0.4827 (2)	0.3406 (4)	0.0891 (4)	6.7 (3)
H(9)	0.417 (2)	0.089 (4)	−0.164 (4)	8 (2)

Table 2. *Selected bond distances* (Å) *and bond angles* (°)

O(1)—C(9)	1.423 (4)	C(6)—C(7)	1.383 (6)
O(2)—C(10)	1.215 (4)	C(7)—C(8)	1.385 (6)
C(1)—C(2)	1.390 (6)	C(8)—C(8a)	1.381 (5)
C(1)—C(9a)	1.377 (5)	C(8a)—C(9)	1.516 (5)
C(2)—C(3)	1.374 (6)	C(9)—C(9a)	1.516 (5)
C(3)—C(4)	1.388 (6)	C(9)—C(10)	1.543 (5)
C(4)—C(4a)	1.389 (5)	C(10)—C(11)	1.519 (5)
C(4a)—C(4b)	1.463 (5)	C(11)—C(12)	1.519 (6)
C(4a)—C(9a)	1.391 (5)	C(11)—C(13)	1.509 (6)
C(4b)—C(5)	1.389 (5)	C(11)—C(14)	1.513 (6)
C(4b)—C(8a)	1.394 (5)	O(1)—H(9)	0.86 (4)
C(5)—C(6)	1.385 (6)		

C(2)—C(1)—C(9a)	119.0 (4)	O(1)—C(9)—C(9a)	109.7 (3)
C(1)—C(2)—C(3)	120.8 (4)	O(1)—C(9)—C(10)	107.9 (3)
C(2)—C(3)—C(4)	120.7 (4)	C(9a)—C(9)—C(9a)	101.9 (3)
C(3)—C(4)—C(4a)	118.5 (4)	C(8a)—C(9)—C(10)	113.2 (3)
C(4)—C(4a)—C(4b)	130.7 (4)	C(9a)—C(9)—C(10)	113.4 (3)
C(4)—C(4a)—C(9a)	120.7 (4)	C(1)—C(9a)—C(4a)	120.3 (4)
C(4b)—C(4a)—C(9a)	108.5 (3)	C(1)—C(9a)—C(9)	129.1 (4)
C(4a)—C(4b)—C(5)	131.1 (4)	C(4a)—C(9a)—C(9)	110.4 (3)
C(4a)—C(4b)—(C8a)	108.8 (3)	O(2)—C(10)—C(9)	115.4 (4)
C(5)—C(4b)—C(8a)	120.0 (4)	O(2)—C(10)—C(11)	119.2 (4)
C(4b)—C(5)—C(6)	118.5 (4)	C(9)—C(10)—C(11)	125.5 (4)
C(5)—C(6)—C(7)	121.2 (4)	C(10)—C(11)—C(12)	106.4 (4)
C(6)—C(7)—C(8)	120.4 (4)	C(10)—C(11)—C(13)	108.7 (4)
C(7)—C(8)—C(8a)	118.6 (4)	C(10)—C(11)—C(14)	116.4 (3)
C(4b)—C(8a)—C(8)	121.1 (4)	C(12)--C(11)—C(13)	109.6 (4)
C(4b)—C(8a)—C(9)	110.1 (4)	C(12)—C(11)—C(14)	108.0 (4)
C(8)—C(8a)—C(9)	128.7 (4)	C(13)—C(11)—C(14)	107.7 (4)
O(1)—C(9)—C(8a)	110.8 (3)	C(9)—O(1)—H(9)	107 (3)

tometer using graphite-monochromated Mo $K\alpha$ radiation, ω scans and a scan speed of 4° min^{-1} (in ω). Weak reflections [$I < 10.0\sigma(I)$] were rescanned (maximum of two rescans) and the counts accumulated to improve accuracy. The lattice parameters were obtained from a least-squares fit of 24 strong reflections in the 2θ range 30–39°. A total of 2603 reflections were measured [h 0 to 22, k 0 to 14, l 0 to 15, $(\sin\theta/\lambda)_{\max} = 0.60$ Å^{-1}, no redundant reflections] and 1561 reflections were considered unobserved with [$I < 3\sigma(I)$], leaving a data set comprised of 1042 unique observed reflections. Three standard reflections ($\bar{6}\bar{3}4$, $63\bar{4}$, 10,0,0) changed by −0.6, −0.4 and −1.7%, respectively; no decay correction was applied. The data were corrected for Lorentz and polarization; no absorption correction was applied. The direct-methods program *MITHRIL* (Gilmore, 1984) provided the locations of all non-H atoms. 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 $\sigma^2(F_o^2) = [S^2(C + R^2B) + (pF_o^2)^2]/\mathrm{Lp}^2$ (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 $p = 0.03$). After convergence methyl and phenyl H atoms were placed at assumed positions (C—H = 0.95 Å) and fixed. Methyl-group orientations were determined on the basis of H-atom positions obtained from a difference Fourier synthesis. The model was again refined to convergence and the hydroxyl H atomic position appeared as the largest peak on the difference Fourier map. The final stages of refinement were performed with 186 variables including all non-H positional and anisotropic thermal parameters, the hydroxyl H-atom positional and isotropic thermal parameter, one scale factor, and a secondary-extinction coefficient [0.9 (1) × 10^{-6}]. Convergence yielded $R = 0.044$, $wR = 0.045$, $S = 1.41$ and $(\Delta/\sigma)_{\max} = 0.0003$. A final difference synthesis produced $(\Delta\rho)_{\max} = 0.17$ and $(\Delta\rho)_{\min} = -0.17$ e Å^{-3}. Atomic scattering factors and anomalous-dispersion corrections were obtained from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 71, 148). All computer programs were from *TEXSAN* (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 (Fig. 1) verifies the molecular structure of (II) as 9-hydroxy-9-pivaloylfluorene and its conformation as *sp*, the same singular conformation it exhibits in solution as suggested by NMR. Fig. 2 is a stereoscopic illustration of its molecular packing in a unit cell. The torsion angle of ~0° for O(1)—C(9)—C(10)—O(2) and the small non-bonding distance of 1.95 (4) Å shown for C(10)═O(2)···H(9)—O(1) suggest substantial intramolecular hydrogen bonding, while the absence of corresponding intermolecular hydrogen bonds is evident from Fig. 2 (*cf.* Milstein, Fultz & Calabrese,

---

* Lists of observed and calculated structure factors, calculated H-atom coordinates and temperature factors, anisotropic thermal parameters for the non-H atoms, torsion angles, and intermolecular distances involving non-H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55182 (22 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR0397]

1986). Such intramolecular hydrogen bonding is possible only in the *sp* conformation of (II).

The data in Table 2 reflect these results. For example, the increased polarity of O=C in *sp*-(II) associated with hydrogen bonding is reflected by its greater bond length [O(2)—C(10), 1.215 (4) Å], compared to 1.198 (5) Å for the corresponding O=C of *ap*-(I). Likewise, formation of the hydrogen-bonded five-membered ring would be accommodated by a reduction in angle O(2)—C(10)—C(9) and increase in angle C(9)—C(10)—C(11), both of which are observed: 115.4 (4) and 125.5 (4)° in *sp*-(II) compared to 119.2 (4) and 118.8 (4)° in *ap*-(I), respectively. The strength of the intramolecular hydrogen bonding in *sp*-(II) is also indicated in the solution ^{1}H NMR spectrum in which the 9-OH proton resonates at δ 5.59, significantly *downfield* compared to the corresponding values for OH of δ 1.91 for 9-hydroxyfluorene itself (Harvey & Lee, 1986), δ 2.3 for 9-hydroxy-9-(2′-*tert*-butylphenyl)fluorene (Nakamura, Nakamura & Ōki, 1977*a*) and near δ 3.7 for simple α-hydroxy ketones (*e.g.* Pouchert & Campbell, 1974).

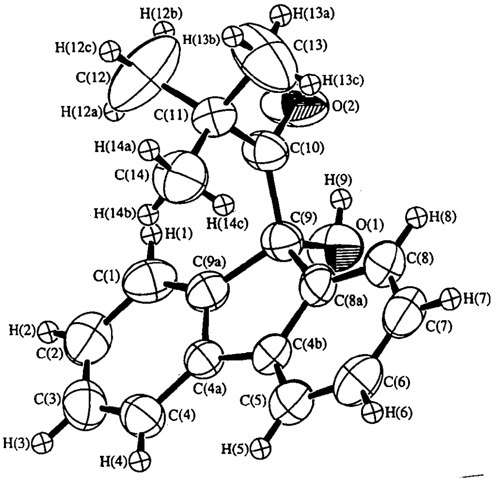


Fig. 1. Molecular configuration and IUPAC atom-numbering scheme. Thermal ellipsoids are drawn at the 50% probability level. H atoms are shown as isotropic spheres with *B* values of 1.0 Å^2.

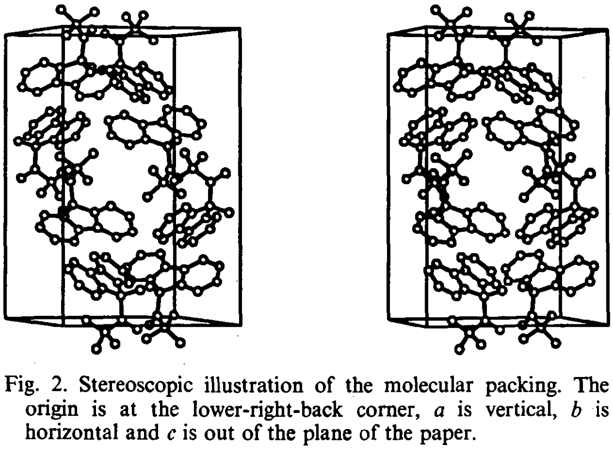


Fig. 2. Stereoscopic illustration of the molecular packing. The origin is at the lower-right-back corner, *a* is vertical, *b* is horizontal and *c* is out of the plane of the paper.

Several implications are suggested from these results. Attacks on (I)-9-anion are steric approach controlled, which leads directly (kinetic control) to products in a high-energy conformation invoking repulsion between the *tert*-butyl group and ring π electrons, as in the *sp* rotamer illustrated. In this conformation the rotational barrier is usually easily surmounted which leads to the energetically favored less sterically encumbered rotamer (thermodynamic control), *e.g.* *sp*-(I) → *ap*-(I). While (II), reasonably, is likewise formed in its *sp* conformation, intramolecular hydrogen bonding stabilizes this rotamer which enhances the rotational barrier sufficiently to allow the isolation of this rotamer. Reaction-mechanism details are currently under investigation and will be presented elsewhere.

Partial financial support of this study from University Research Foundation, Diatron Corporation, and Computer Accessories/Proxima, Inc., all of San Diego, is gratefully acknowledged. DHH expresses gratitude to the National Insitutes of Health (USA) for their financial assistance. We are grateful to Dr David Bolton, Tektronix, 393 Inverness Drive South, Englewood, Colorado 80112, USA, for carrying out the rotomer-energy calculations utilizing the Tektronix CAChe™ program Molecular Mechanics Data – *MM*2, and to Steve J. Arvanitis for his assistance in plotting these data.

References

GILMORE, C. J. (1984). *J. Appl. Cryst.* **17**, 42–46.

HARVEY, R. G. & LEE, H. (1986). In *TRC Spectral Data – ^{1}H Nuclear Magnetic Resonance*, Serial No. 79, p. 1. Thermodynamics Research Center, Texas A&M Univ., College Station, TX 77843-3111, USA.

IUPAC (1970). *J. Org. Chem.* **35**, 2849–2867.

MANOHAR, S. K. (1986). MS thesis. Southern Illinois Univ., Carbondale, IL, USA.

MEYERS, C. Y. & MANOHAR, S. K. (1987). Am Chem. Soc. National Meeting, Denver, USA, Abstracts, ORGN 260.

MEYERS, C. Y., WAHNER, A. P., MANOHAR, S. K., CARR, S. E., CHAN-YU-KING, R. & ROBINSON, P. D. (1991). *Acta Cryst.* C**47**, 1236–1239.

MILSTEIN, D., FULTZ, W. C. & CALABRESE, J. C. (1986). *J. Am. Chem. Soc.* **108**, 1336–1338.

Molecular Structure Corporation (1985). *TEXSAN. TEXRAY Structure Analysis Package.* MSC, 3200A Research Forest Drive, The Woodlands, TX 77381, USA.

NAKAMURA, M., NAKAMURA, N. & ŌKI, M. (1977*a*). *Bull. Chem. Soc. Jpn*, **50**, 1097–1101.

NAKAMURA, M., NAKAMURA, N. & ŌKI, M. (1977*b*). *Bull. Chem. Soc. Jpn*, **50**, 2986–2990.

POUCHERT, C. J. & CAMPBELL, J. R. (1974). *Aldrich Library of NMR Spectra*, Vol. II, p. 116. Aldrich Chemical Co., Inc., Milwaukee, WI 53233, USA.