

Mechanistic information about the formation of **4c** and **4t** was obtained by mixing **2** and **3** at  $-35\text{ }^{\circ}\text{C}$  in  $\text{CD}_2\text{Cl}_2$ : a single new product (**7**) was obtained, along with the byproducts **1**, **5**, and **6**.<sup>10</sup> Warming **7** to  $0\text{ }^{\circ}\text{C}$  resulted in slow conversion to **4c** and **4t**. A structure for **7** consistent with the spectroscopic data<sup>4</sup> is shown in Scheme I. The lack of any splitting of the CpMo and CpFe signals in the  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra down to  $-70\text{ }^{\circ}\text{C}$  is consistent with the presence of an unbridged Mo–Fe bond, particularly since the isomers **4c** and **4t** exhibit substantial cyclopentadienyl chemical shift differences (0.50 and 0.10 ppm in the  $^1\text{H}$  NMR and both 1.8 ppm in the  $^{13}\text{C}$  NMR for CpMo and CpFe, respectively). The acyl resonance at 265.0 ppm is consistent with both  $\eta^1$ -coordination<sup>5b</sup> and  $\eta^2$ -coordination,<sup>11</sup> but the latter is more likely based on the effective atomic number rule and the absence of other coordinating ligands.<sup>12</sup> Finally, the  $^{13}\text{C}$  peaks at 220.3 and 218.2 ppm are consistent with the presence of diastereotopic carbonyl ligands on iron.<sup>5</sup> In order to confirm this point we combined  $\text{Cp}(^{13}\text{CO})_2\text{Fe}(\text{THF})^+\text{BF}_4^-$  ( $3\text{-}^{13}\text{C}_2$ )<sup>13</sup> with **2** to give **7** in which the only labeled carbons were the diastereotopic carbonyl ligands. However, an unexpected singlet was observed at the center of each doublet, the intensity of which indicated the presence of a 0.75:1 ratio of  $7\text{-}(^{13}\text{CO})(^{12}\text{CO})/7\text{-}(^{13}\text{CO})_2$ . The  $^{13}\text{C}$ – $^{13}\text{C}$  coupling constant in  $7\text{-}(^{13}\text{CO})_2$  was 11.4 Hz; evidence that this is indeed a geminal coupling constant is its similarity to the value of 10.2 Hz observed in  $\text{Cp}(^{13}\text{CO})_2\text{Fe}(\text{Ph}(\text{CH}_3)\text{NH})^+\text{BF}_4^-$  ( $8\text{-}^{13}\text{C}_2$ ), prepared by reaction of  $\text{Ph}(\text{CH}_3)\text{NH}$  with  $3\text{-}^{13}\text{C}_2$ .<sup>4</sup>

A mechanism for the formation of **4c** and **4t** consistent with the above data is presented in Scheme I. Initial reaction of **2** and **3** could occur to directly give the dinuclear intermediate **9**. The  $^{13}\text{C}$ O-labeling experiment requires rapid exchange of CO between the metal centers in order to account for the partial<sup>14</sup> loss of  $^{13}\text{C}$ O label in **7**, and we propose that such a process occurs in **9**.<sup>15</sup> Formation of **7** from **9** is proposed to occur by dissociation of CO from molybdenum, since the 16-electron center that would remain should be particularly stable both due to the possibility of  $\eta^2$ -coordination of the acyl and by analogy to the stable 16-electron compounds  $\text{Cp}(\text{NO})\text{MoR}_2$  ( $\text{R} = \text{CH}_2\text{SiMe}_3, \text{SPh}$ ).<sup>16</sup> Finally, conversion of **7** to **4c** and **4t** can readily occur by acyl carbon migration to iron with concomitant formation of the  $\mu$ -CO interaction.

In conclusion, the synthesis of **4c** and **4t** represents a novel and potentially general synthesis of heterodinuclear  $\mu$ -acyls. The acyl migration may be related to those previously observed during syntheses of other  $\mu$ -acyls,<sup>7h,8b,c,e,i,m,17</sup> but in only one other case has a metal–metal-bonded<sup>18</sup> intermediate—actually a stable isomer—been isolated.<sup>6b</sup> Based on that example, Kaesz suggested that reversible acyl flipping might be analogous to that of fluxional  $\mu$ - $\sigma$ , $\pi$ -vinyl complexes.<sup>19</sup> Based on the observed conversion of

**7** to **4c** and **4t**, however, it is tempting to suggest that acyl migrations may also involve *stepwise* interconversion of bridging and nonbridging  $\eta^2$ -acyls. Further work on the synthetic and mechanistic chemistry of the acyls described herein is in progress.

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**Supplementary Material Available:** Spectroscopic data for **2**, **4c**, **4t**, **5**, **7**, **8**, and **10** and tables of crystallographic data for **4c** and **4t** and details of the solution (23 pages); tables of observed and calculated structure factors for **4c** and **4t** (26 pages). Ordering information is given on any current masthead page.

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## Length of the Ethane Bond in Hexaphenylethane and Its Derivatives

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The length of the central carbon–carbon bond ( $r_c$ ) in hexaphenylethane and its derivatives has been the subject of some dispute. It was originally predicted, by the use of empirical force field calculations, that  $r_c$  in hexaphenylethane is  $1.64\text{ \AA}$ .<sup>1</sup> This prediction was challenged by an announcement of the X-ray structure of hexakis(2,6-di-*tert*-butyl-4-biphenyl)ethane (**1a**), in which it was claimed that far from being lengthened relative to the standard value of  $1.54\text{ \AA}$ ,  $r_c$  is appreciably shortened, to  $1.47(2)\text{ \AA}$ .<sup>2</sup> A subsequent and more elaborate computational study<sup>3</sup> not only confirmed the original conclusions<sup>1</sup> in all essential details but predicted  $r_c = 1.64\text{ \AA}$  (MM2) for **1a** as well,<sup>4</sup> thus discounting the possibility that the substituents on the benzene

(10) Intermediate **7** can be isolated in 35–45% yield by carrying out the reaction at  $-78\text{ }^{\circ}\text{C}$  and collecting the resultant orange precipitate.

(11) (a) Curtis, M. D.; Shiu, K.-B.; Butler, W. M. *J. Am. Chem. Soc.* **1986**, *108*, 1550–1561. (b) Rusik, C. A.; Tonker, T. L.; Templeton, J. L. *Ibid.* **1986**, *108*, 4652–4653.

(12) Solutions of **7** free of THF can be prepared by using  $\text{Cp}(\text{CO})_2\text{Fe-FBF}_3$ ; the isolable reagent is described in: Mattson, B. M.; Graham, W. A. *G. Inorg. Chem.* **1981**, *20*, 3186–3189.

(13) (a) Jonas, K.; Schieferstein, L. *Angew. Chem., Int. Ed. Engl.* **1979**, *18*, 549–550. (b) Reger, D. L.; Coleman, C. *J. Organomet. Chem.* **1977**, *131*, 153–162.

(14) Complete scrambling prior to CO loss would give a 2:1 ratio of  $7\text{-}(^{13}\text{CO})(^{12}\text{CO})/7\text{-}(^{13}\text{CO})_2$ , so the scrambling is less than 40% complete.

(15) We observe fast exchange (on the NMR time scale) of CO in the model compounds  $\text{Cp}(\text{CO})_2\text{Fe-M}(\text{CO})_3\text{Cp}$  ( $\text{M} = \text{Mo}, \text{W}$ ) down to  $-90\text{ }^{\circ}\text{C}$ .

(16) (a) Legzdins, P.; Rettig, S. J.; Sanchez, L.; Bursten, B. E.; Gatter, M. G. *J. Am. Chem. Soc.* **1985**, *107*, 1411–1413. (b) Ashby, M. T.; Enemark, J. H. *Ibid.* **1986**, *108*, 730–733.

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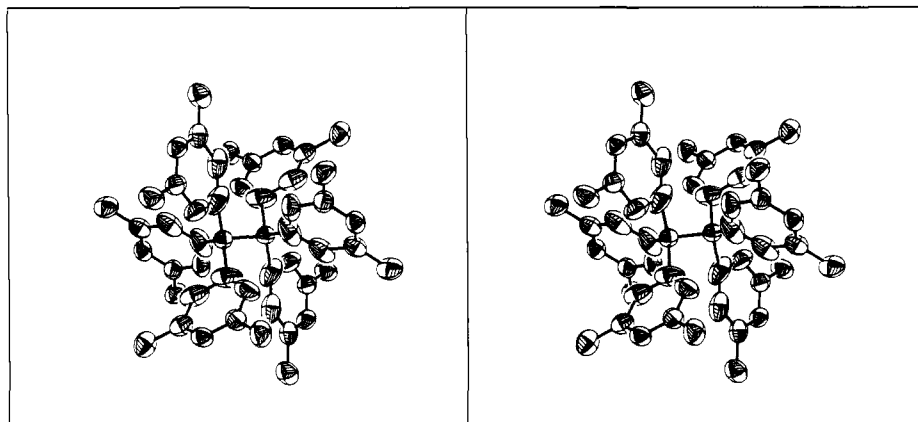
(18) Non-metal–metal bonded acyls have been observed to rearrange to  $\mu$ -acyls,<sup>8e,17</sup> and reversible carbonylation with concomitant acyl flipping and metal–metal bond cleavage has been reported;<sup>8n</sup> see also: Martin, B. D.; Matchett, S. A.; Norton, J. R.; Anderson, O. P. *J. Am. Chem. Soc.* **1985**, *107*, 7952–7959.

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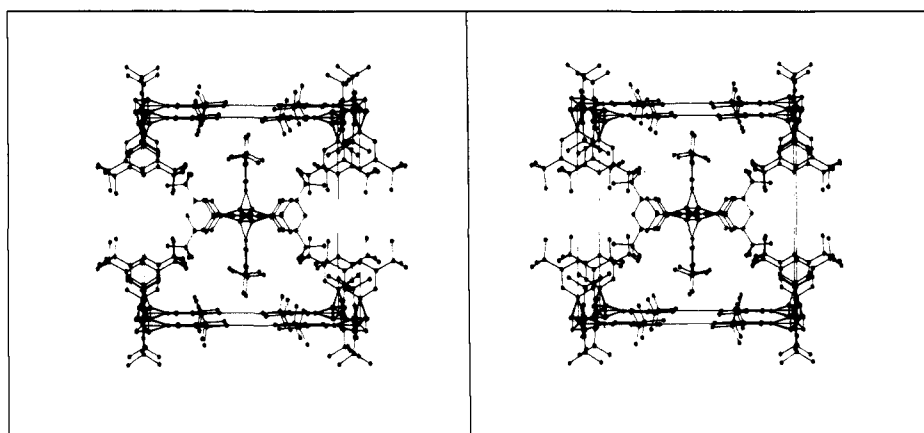
(2) Stein, M.; Winter, W.; Rieker, A. *Angew. Chem.* **1978**, *90*, 737; *Angew. Chem., Int. Ed. Engl.* **1978**, *17*, 692. See also: Winter, W. *Fresenius' Z. Anal. Chem.* **1980**, *304*, 279. Winter, W.; Butters, T.; Rieker, A.; Butsugan, Y. *Z. Naturforsch.* **1982**, *37b*, 855. Winter, W.; Moosmayer, A.; Rieker, A. *Ibid.* **1982**, *37b*, 1623.

(3) Osawa, E.; Onuki, Y.; Mislow, K. *J. Am. Chem. Soc.* **1981**, *103*, 7475.

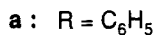
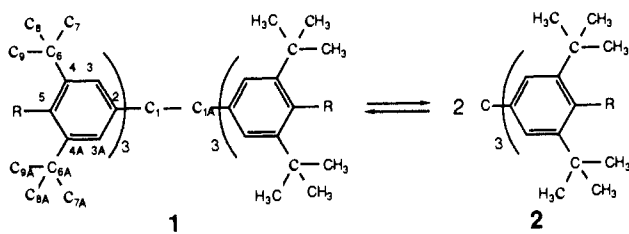
(4) Force field calculations are remarkably successful in predicting substantial elongations of bonds between tetracoordinate carbon atoms in highly strained molecules. For a recent example, see: Flamm-ter Meer, M. A.; Beckhaus, H.-D.; Peters, K.; von Schnering, H.; Rüdhardt, C. *Chem. Ber.* **1985**, *118*, 4665. See also: Rüdhardt, C.; Beckhaus, H.-D. *Top. Curr. Chem.* **1986**, *130*, 1 and references therein. Rüdhardt, C.; Beckhaus, H.-D. *Angew. Chem.* **1985**, *97*, 538; *Angew. Chem., Int. Ed. Engl.* **1985**, *24*, 529 and references therein.



**Figure 1.** Stereoview of the X-ray structure of hexakis(3,5-di-*tert*-butylphenyl)ethane (**1b**). Methyl carbons and all hydrogen atoms have been deleted for clarity.



**Figure 2.** Stereoview of the unit cell of **1b**, showing the crystallographic superposition of the four ethane orientations. The small cube centered at  $(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$  defines the volume enclosed by the ethane carbons. The six averaged phenyl rings are perpendicular to the faces of the cubic cell with mutually perpendicular rings directed toward the (1,0,0), (0,1,0), and (0,0,1) faces. Thus, a pair of phenyl rings lying horizontally is pointing directly in and out of the plane of the paper. Line segments are drawn connecting the ethane carbons to each phenyl ring to which it may be bonded. The disorder of the *tert*-butyl groups about the mirror planes is not shown, and the included cyclohexane molecules and all hydrogen atoms have been deleted for clarity.



ring have a significant effect on the calculated value of  $r_c$ . We now report the X-ray structure of hexakis(3,5-di-*tert*-butylphenyl)ethane (**1b**) and the salient finding that  $r_c = 1.67$  (3) Å,<sup>5</sup> in agreement with our calculated value of 1.65 Å (MM2).

Benzene and toluene solutions of tris(3,5-di-*tert*-butylphenyl)methyl (**2b**) yielded red crystals which were monomeric

by ESR<sup>6</sup> and X-ray diffraction.<sup>7</sup> As previously observed by Stein et al.,<sup>2</sup> solutions of **2b** in cyclohexane deposit colorless crystals of the dimer **1b** upon evaporation. Crystals (mp 214 °C, dec) of **1b** suitable for X-ray analysis were grown by allowing cyclohexane solutions of **2b** to stand for 2 mo at room temperature. The crystals, which include cyclohexane and effloresce at room temperature, were found to belong to the space group  $Pm\bar{3}n$ .<sup>8</sup> The high symmetry,  $T_h$  ( $m\bar{3}$ ) at Wyckoff position *a*, is a manifestation of fourfold disorder: each individual molecule of **1b** has  $S_6$  (3)

(6) Schreiner, K.; Berndt, A.; Baer, F. *Mol. Phys.* **1973**, *26*, 929.

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(8) Crystals of **1b** are cubic, space group  $Pm\bar{3}n$  (No. 223), with  $a = b = c = 16.673$  (4) Å,  $V = 4635$  (3) Å<sup>3</sup>, and  $d_{\text{calcd}} = 1.01$  g cm<sup>-3</sup> for  $Z = 2$  (C<sub>86</sub>H<sub>126</sub>·3C<sub>6</sub>H<sub>12</sub>,  $M = 1412.6$ ). The single crystal used for data collection measured 0.25 × 0.32 × 0.46 mm and was mounted on a glass fiber. Intensity data were measured on a Nicolet R3m diffractometer with 3° < 2θ < 114° using graphite-monochromated Cu Kα radiation (λ = 1.541 78 Å) at 175 K. Of 594 unique reflections, 508 were considered to be observed [|F<sub>o</sub>| > 3σ(F<sub>o</sub>)] after applying Lorentz, polarization, and absorption corrections. The structure was solved in the lower symmetry rhombohedral space group  $R\bar{3}c$  using the SHELXTL direct methods software and was refined in  $Pm\bar{3}n$ . All carbons in the ethane molecule were refined anisotropically while those in the included cyclohexane were refined isotropically. Final atomic parameters for **1b** are listed in Table I. Hydrogen atoms were included at standard positions (C-H 0.96 Å, C-C-H 120° or 109.5°). Phenyl hydrogens were varied by using a riding model and the methyl hydrogens were fixed. Refinement with 79 parameters converged at  $R = 0.093$  and  $wR = 0.079$ . A final difference map was featureless with a maximum peak of 0.23 e Å<sup>-3</sup> in the area of the cyclohexane molecule. Further details are available in the supplementary material.

(5) There are numerous examples in the literature of bonds between tetra-coordinate carbon atoms that equal or exceed 1.6 Å in length. See ref 1 (footnote 30), ref 3 (footnote 7), and ref 4. To our knowledge, the longest acyclic C-C bond length on record is 1.671 (4) Å, reported for  $r_c$  in 2,3-bis(trifluoromethyl)-2,3-bis(*p*-fluorophenyl)hexafluorobutane. See: Polishchuk, V. R.; Antipin, M. Yu.; Bakhumtov, V. I.; Bubnov, N. N.; Solodovnikov, S. P.; Timofeeva, T. V.; Struchkov, T. Yu.; Tumanskii, B. L.; Knunyants, I. L. *Dokl. Acad. Nauk SSSR* **1979**, *249*, 1125; *Dokl. Chem. (Engl. Transl.)* **1979**, *249*, 547.

**Table I.** Atomic Coordinates ( $\times 10^4$ ) and Isotropic Thermal Parameters ( $\text{\AA}^2 \times 10^3$ ) for Hexakis(3,5-di-*tert*-butylphenyl)ethane (**1b**)

	x	y	z	$U^a$
C(1)	-290 (5)	-290 (5)	-290 (5)	49 (2)
C(2)	0	-1204 (4)	0	100 (2)
C(3)	700 (3)	-1645 (3)	0	83 (2)
C(4)	729 (3)	-2478 (3)	0	62 (1)
C(5)	0	-2877 (4)	0	59 (2)
C(6)	1528 (3)	-2921 (3)	0	78 (2)
C(7)	1983 (9)	-2778 (10)	762 (6)	145 (3)
C(8)	2011 (9)	-2625 (11)	-728 (10)	169 (3)
C(9)	1459 (3)	-3817 (4)	-68 (16)	127 (2)

<sup>a</sup> Equivalent isotropic  $U$  defined as one third of the trace of the orthogonalized  $U_{ij}$  tensor.

symmetry (Figure 1), but the molecular axis, which includes the central carbon-carbon bond, is oriented along the four body diagonals of the cubic unit cell with equal probability (Figure 2).<sup>9</sup> The phenyl rings are represented as averages perpendicular to the faces of the cube. The disorder is apparent from the large anisotropic thermal parameters tangential to the plane of the benzene ring (e.g., C(2):  $U_{11} = 183$  (5),  $U_{22} = 39$  (3),  $U_{33} = 77$  (4)  $\text{\AA}^2 \times 10^3$ ). The C(1)-C(2) benzylic bond length of 1.670 (7)  $\text{\AA}$  as well as the C(1)-C(2)-C(3) and C(1)-C(2)-C(3A) angles of 137.0 (4) $^\circ$  and 103.9 (4) $^\circ$  are artifacts of averaging the four phenyl orientations. The solvent molecules are located at the intersections of the three mirror planes (Wyckoff position *b*) and are highly disordered.

The ethane carbon atoms, by contrast with the phenyl carbons, are not represented at average positions. Furthermore, these atoms are well defined, with relatively small thermal parameters ( $U_{11} = U_{22} = U_{33} = 49$  (4)  $\text{\AA}^2 \times 10^3$ ). The estimated standard deviation associated with the central carbon-carbon bond length, 1.67 (3)  $\text{\AA}$ , is significantly higher than that for other bond lengths in the molecule. Among the contributing reasons are the following. First, the ethane carbon lies on a special position and hence has an inherent indeterminacy associated with this position.<sup>10</sup> Second, the ethane carbons are related by a center of symmetry; this correlation leads to an error in bond length equal to  $2\sigma(x)$ . Third, there is a greater error in the position of the ethane carbon due to the reduced occupancy; this follows from the proportionality of  $\sigma(x)$  and  $\sigma(dp/dx)$ .<sup>10,11</sup>

In light of the present findings and of the previous computational studies,<sup>1,3</sup> the claim for bond shortening of  $r_c$  in **1a**<sup>2</sup> may now be safely dismissed.<sup>12</sup>

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**Supplementary Material Available:** Bond lengths, bond angles, with standard deviations for **1b** (Tables II and III), final anisotropic thermal parameters (Table IV), and atomic parameters for hydrogen (Table V) and for cyclohexane (Table VI) (2 pages); listing of observed and calculated structure factors (3 pages). Ordering information is given on any current masthead page.

(9) It is not uncommon for "globular" ethanes to exhibit a cubic solid phase in which the ethane bond is disordered among the four threefold axes. See: (a) Hexamethylethane: Koide, T. *Bull. Chem. Soc. Jpn.* **1967**, *40*, 2026. (b) Hexachloroethane: Atoji, M.; Oda, T.; Watanabe, T. *Acta Crystallogr.* **1953**, *6*, 868. (c) Hexabromoethane: Koide, T.; Tsujino, M.; Sawada, K.; Oda, T. *Bull. Chem. Soc. Jpn.* **1974**, *47*, 2998.

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## High Yields of Formal CH Insertion Products in the Reactions of Polyfluorinated Aromatic Nitrenes

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Aryl azides continue to be widely used as photoaffinity labeling (PAL) reagents for biomolecules.<sup>2</sup> Unfortunately, photolysis of phenyl azide or its simple derivatives in a model solvent such as toluene leads only to trace amounts of aniline and azobenzene and large quantities of intractable tars.<sup>3</sup> An adduct of phenylnitrene and toluene, the useful product of PAL studies, is not formed in detectable quantities. We recently reported the formation of modest yields ( $\sim 30\%$ ) of a formal insertion adduct in the reaction of triplet (*m*-nitrophenyl)nitrene with frozen polycrystalline toluene at the benzylic position.<sup>4</sup> Adduct formation did not occur upon direct photolysis or in only very low yield on triplet-sensitized photolysis of *m*-nitrophenyl azide in toluene solution. In this paper we report the effect of temperature and phase on the photochemistry of pentafluorophenyl and 2,6-difluorophenyl azides. Our interest in fluorine-substituted aryl azides was motivated by Reiser and Leyshon's report that electron-withdrawing substituents enhance the reactivity of triplet aryl nitrenes.<sup>5</sup> Fluorine is also an ideal substituent for PAL as it is nearly isosteric with hydrogen<sup>6</sup> and thus should not interfere with substrate recognition.

Photolysis of **1a** and **1b** in toluene at 25  $^\circ\text{C}$  gives small amounts of tar, the fluorinated anilines **3a** and **3b**, azo compound **5b**, and the adducts **4a** and **4b** and their isomers (Table I and II).<sup>7</sup> The ratio of isomers formed by photolysis of **1a** in toluene at 25  $^\circ\text{C}$  was in agreement with the reports of Abramovitch, Challand, and Scriven<sup>8</sup> who deoxygenated pentafluoronitrosobenzene in toluene. At -78  $^\circ\text{C}$ , some tar is still formed on photolysis of **1a** and **1b**, there is an increase in yields of the azo compounds **5a,b**, but there is a decrease in the yields of the nitrene-toluene adducts. Simply lowering the temperature of a solution-phase photolysis does not lead to an increased yield of the desired adducts. Sensitized photolysis of **1a** and **1b** at 25  $^\circ\text{C}$  led to a decrease in the amount of tar produced, high yields of anilines **3a** and **3b**, low yields of insertion adducts **4a**, **4a'**, and isomerically pure **4b**. The generation of triplet nitrenes in solution by sensitized photolysis does not lead to appreciable yields of the adducts desired in a PAL experiment. However, photolysis of frozen polycrystalline toluene containing **1a** or **1b** gave almost quantitative yields of insertion adducts **4a** and **4b** which were essentially isomerically pure. Tar was not present in the low-temperature photolysis reaction mixture. Thus the combination of fluorine substitution and low-temperature irradiation appears to be an ideal strategy for efficient PAL (Scheme I).

The origin of the enhanced yields of adducts formed in matrices was revealed by EPR analysis. Photolysis of **1a** and **1b** in polycrystalline toluene at -196  $^\circ\text{C}$  leads to triplet nitrenes **2a** and **2b** which are indefinitely stable in the dark at this temperature.

(1) Camille and Henry Dreyfuss Teacher-Scholar.

(2) Bailey, H. *Photogenerated Reagents in Biochemistry and Molecular Biology*; Elsevier: New York, 1983.

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(4) Torres, M. J.; Zayas, J.; Platz, M. S. *Tetrahedron Lett.* **1986**, *27*, 791.

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(6) The atomic radii in covalent bonds for H and F are 0.37 and 0.72  $\text{\AA}$ , respectively: Gordon, A. J.; Ford, R. A. *Chemist's Companion*; Wiley: New York, 1972; pp 82-85.

(7) The benzylic insertion adducts **4a** and **4b** were differentiated from their aromatic insertion isomers by GC-MS analysis and by independent synthesis. Compounds were analyzed for by GC; products were identified by coinjection with authentic samples.

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