Table II. ^{13}C Chemical Shifts of the Tritopic Adamantanes 7–11 and 13[a]

C	7	8	9	10	11	13
1	44.6	45.4	54.1	53.4	54.3	45.1
2	208.1	207.9	207.7	207.7	208.5	210.9
3	62.2	62.0	54.1	54.1	54.3	61.8
4	55.9	56.7	59.2	59.1	56.0	74.4
5	34.8	34.1	34.1	34.8	42.3	34.2[b]
6	31.8	36.7	37.7	31.5	34.6	30.3
7	34.1	34.1	34.1	33.8	25.8	33.6[b]
8	36.2	34.2	59.2	55.5	32.2	35.7
9	34.1	34.2	35.6	28.7	52.3	33.1
10	56.3	56.7	35.6	37.4	41.5	55.2

[a] In ppm, relative to internal tetramethylsilane, solvent $CDCl_3$. [b] May be interchanged.

from COSY45-2D NMR (homonuclear) experiments[8] so that unequivocal ^1H signal assignments were possible. Heteronuclear two-dimensional ^{13}C–^1H correlated spectra[8] afforded unambiguous ^{13}C signal assignments as well.

The structure determination of **13** was made by a comparison of its ^{13}C chemical shifts with those of the related dibromoadamantanones **7** and **8**. The CH_2 (C^6) signal at δ 30.3 is consistent only with formula **13**, which is structurally related to **7**; the diaxial[9] isomer can be excluded since no CH_2 signal below $\delta \sim 34$ is expected for that structure. The 4[e]-hydroxy 10[a]-bromo isomer can be excluded also because its C^8 chemical shift must be in the range of $\delta \sim 39$–40.[10]

For a number of tritopic adamantanes[4,10] we were able to show that interaction effects of pairs of substituents on the ^{13}C chemical shifts are additive to a large extent so that their ^{13}C chemical shifts can easily be calculated if those of the corresponding mono- and ditopic adamantanes are known. This rule can be applied to the dibromoadamantanones as well as is demonstrated for C^4 of **7**:

$\delta(C^4)$ in adamantanone	39.2
α subst effect of Br^4	25.9
γ-gauche subst effect of Br^{10}	−6.1
interactn effect (C=O, Br^4)	−5.0
interactn effect (Br^4, Br^{10})	0.4
calcd ^{13}C chem shift	54.4
exptl ^{13}C chem shift	55.9

Substantial deviations (>|2| ppm) between calculated and experimental chemical shifts are occasionally found only for carbonyl and brominated carbon atoms. Thus, the structural assignment may be carried out unequivocally also by such increment rules.

Experimental Section

Melting points were determined on a Büchi-Tottoli melting point apparatus and are uncorrected. Infrared spectra were recorded on a Shimadzu IR-400 spectrophotometer; ^1H NMR spectra were obtained on Varian T-60 and Bruker WP-80 and AM-400 and ^{13}C NMR spectra on Bruker WH-90, WM-250, and AM-400 spectrometers. For the measurement of the two-dimensional NMR spectra[8] Bruker software was employed. ^1H and ^{13}C chemical shifts are referenced to internal tetramethylsilane. Mass spectra and high-resolution mass spectra (HRMS) were recorded on Varian MAT CH-5, CH-7, and 731 spectrometers.

All compounds were purified by column chromatography using Lobar columns (Merck) under medium pressure with various ligroin–acetone mixtures as eluants and were >98% pure. Compounds already known (**1**-4[11], **5**[10], **6**,[10] and **12**[1]) were identified by comparing their spectra. In most cases authentic samples were available for comparison by thin-layer chromatography. All yields refer to isolated material after purification and are uncorrected.

General Synthetic Procedure. The bromo lactones **1**–**4**, respectively (1.0 g, 4.08 mmol), were refluxed in 40–50 mL of 48% hydrobromic acid for 24 h. After cooling, the mixture was extracted with methylene chloride, washed with sodium bicarbonate and water, and dried over anhydrous magnesium sulfate. After evaporation of the solvent the crude brown oil was subjected to column chromatography.

4[a],10[e]-Dibromoadamantan-2-one (7):[9] Yield 6% as a white solid; mp 132–134 °C; IR ($CHCl_3$) 2940, 1740, 1725 cm^{-1}; mass spectrum *m/e* (relative intensity) 310/308/306 (3/7/3, M^+), 229/227 (78/78), 201/199 (33/33), 119 (100), 93 (20), 91 (70), 79 (88); molecular weight by HRMS for $C_{10}H_{12}Br_2$ (calcd) 309.9211/307.9231/305.9251, (found) 309.9185/307.9202/305.9229.

4[a],10[a]-Dibromoadamantan-2-one (8):[9] yield 9%; IR ($CHCl_3$) 2920, 1730, 1715 cm^{-1}; mass spectrum, *m/e* (relative intensity) 310/308/306 (5/10/5), 229/227 (74/74), 201/199 (35/39), 119 (100), 93 (17), 91 (98), 79 (64); molecular weight by HRMS for $C_{10}H_{12}Br_2$, (calcd) 309.9211/307.9231/305.9251, (found) 309.9193/307.9192/305.9218.

4[a],8[a]-Dibromoadamantan-2-one (9):[9] yield 4% (from **2**), 8% from **3** and **10**% from **4** as a white solid; mp 233–235 °C; IR ($CHCl_3$) 2935, 1735, 1730 cm^{-1}; mass spectrum, *m/e* (relative intensity) 310/308/306 (7/13/6), 229/227 (96/100), 201/199 (18/18), 119 (90), 91 (61), 79 (47); molecular weight by HRMS for $C_{10}H_{12}Br_2$, (calcd) 309.9211/307.9231/305.9251, (found) 309.9178/307.9222/305.9279.

4[a],8[e]-Dibromoadamantan-2-one (10):[9] yield 4% as a white solid; mp 104 °C; IR ($CHCl_3$) 2910, 1720 cm^{-1}; mass spectrum, *m/e* (relative intensity) 310/308/306 (4/8/4), 229/227 (100/99), 201/199 (17/17), 119 (85), 91 (58), 79 (45); molecular weight by HRMS for $C_{10}H_{12}Br_2$, (calcd) 309.9211/307.9231/305.9251, (found) 309.9198/307.9208/305.9225.

4[a],9[e]-Dibromoadamantan-2-one (11):[9] yield 4% as a white solid; mp 99–101 °C; IR ($CHCl_3$) 2910, 1715 cm^{-1}; mass spectrum, *m/e* (relative intensity) 310/308/306 (4/7/4), 229/227 (94/98), 201/199 (18/21), 119 (89), 91 (62), 85 (73), 83 (100), 79 (65); molecular weight by HRMS for $C_{10}H_{12}Br_2$, (calcd) 309.9211/307.9231/305.9251, (found) 309.9215/307.9231/305.9244.

4[a]-Hydroxy-10[e]-bromoadamantan-2-one (13):[9] yield 1%; IR ($CHCl_3$) 3600–3100 (br), 2925, 1740, 1720 cm^{-1}; ^1H NMR (80 MHz) δ ($CDCl_3$) 4.77 (1 H, m), 4.46 (1 H, m), 2.94 (1 H, m), 2.87–1.42 (9 H, complex); mass spectrum, *m/e* (relative intensity) 246/244 (4/4; M^+), 165 (76), 147 (16), 135 (27), 121 (27), 119 (65), 91 (49), 85 (65), 83 (100), 79 (60).

Acknowledgment. The authors gratefully acknowledge valuable discussions with Prof. L. Vodička and Dr. J. Třiska, Prague, and technical assistance from Dr. W. Dietrich and Dr. M. Kaiser, Bochum. This work and supported by the Deutsche Forschungsgemeinschaft and the Fonds der Chemischen Industrie.

Registry No. **1**, 85114-85-8; **2**, 85114-87-0; **3**, 85069-88-1; **4**, 85114-86-9; **5**, 32456-49-8; **6**, 32456-48-7; **7**, 99232-54-9; **8**, 99295-19-9; **9**, 94239-95-9; **10**, 94293-63-7; **11**, 99232-55-0; **12**, 19214-00-7; **13**, 99232-56-1.

(9) The designations "a" (axial) and "e" (equatorial) refer to the stereochemical positions of the substituents with respect to the substituent-bearing cyclohexanone subunit in the adamantane molecule.
(10) Duddeck, H. *Tetrahedron* **1983**, *39*, 1365 and references therein.
(11) Duddeck, H.; Kaiser, M. *Z. Naturforsch. B: Anorg. Chem., Org. Chem.* **1982**, *37b*, 1672.

Improved Procedure for Introducing the α-Fluoroacetyl Group via the Directed Aldol Reaction

John T. Welch* and Seetha Eswarakrishnan

Department of Chemistry, State University of New York at Albany, Albany, New York 12222

Received March 22, 1985

Recently we have reported several examples of the directed aldol reaction with the lithium enolate of ethyl fluoroacetate[1] and 1-fluoro-3,3-dimethyl-2-butanone.[2]

Table I. Products of the Directed Aldol Reaction of Fluoroacetamide Enolates

$CH_2FCONR_2 + LDA \rightarrow LiCHFCONR_2$

$LiCHFCONR_2 + R'C(O)R'' \rightarrow R'R''COHCHFCONR_2$

entry	R	R′	R″	yield, %	diastereoselectivity[e]	mp,[f] °C
1	CH_3	Ph	H	90[a]	2.5:1	106–108
2	CH_3	$(CH_3)_3C$	H	63[a]	1.2:1	88–89.5
3	CH_3	CH_3CH_2	H	63[b]	1:1	
4	CH_3	$(CH_3)_2CH$	H	67[b]	1.6:1	
5	CH_3	Ph	CH_3	89[c]	1.9:1	49–51
6	CH_3	Ph	Ph	98[a]	–	104–105
7	CH_3	adamantanone		99[a]	–	110–113
8	CH_3	3,3-dimethyl-2,4-dioxol-1-yl		86[a]	1:1	–
9	$(CH_2)_4$	Ph	H	75[a]	1.6:1	98–100
10	$(CH_2)_4$	$(CH_3)_3C$	H	95[a]	1.6:1	79–80
11	$(CH_2)_4$	CH_3CH_2	H	89[a]	1:1	
12	$(CH_2)_4$	$(CH_3)_2CH$	H	50[b]	1.1:1	72–74
13	$(CH_2)_4$	Ph	Ph	97[a]	–	157–158
14	$CH(CH_3)_2$	Ph	H	81[d]	1.6:1	107–112
15	$CH(CH_3)_2$	$(CH_3)_3C$	H	68[c]	4.0:1	70–73
16	$CH(CH_3)_2$	$CH_3CH_2CH_2$	H	42[d]	1.3:1	
17	$CH(CH_3)_2$	$(CH_3)_2CH$	H	86[d]	1.2:1	
18	$CH(CH_3)_2$	Ph	CH_3	74[b]	1.2:1	
19	$CH(CH_3)_2$	norbornanone		80[c]	1.7:1	79–80
20	$CH(CH_3)_2$	Ph	Ph	78[d]	–	122–125
21	$CH(CH_3)_2$	adamantanone		88[d]	–	96–105

[a] Crude yield. [b] Yield after chromatography on silica gel. [c] Yield after recrystallization. [d] Yield based upon NMR analysis of products. [e] As determined by ^{13}C NMR. [f] mp of mixture of diastereomers as physical characteristic of the isolated product, not as a criteria of purity.

Prior to this work the directed aldol reaction of *tert*-butyl fluoroacetate was also reported.[3] These reagents each have associated with them the considerable toxicity of fluoroacetate derivatives. To facilitate our exploration of the stereoselectivity possible with such reagents complementary to that reported in the literature for a wide variety of compounds,[4] we sought to prepare less volatile fluorinated starting materials.

Results and Discussion

We have found that the *N*-(2-fluoroacetyl)pyrrolidine, *N*,*N*-diisopropylfluoroacetamide, and *N*,*N*-dimethylfluoroacetamide may be easily prepared from the corresponding amines and fluoroacetyl chloride. Addition of

$$CH_2FCOCl + HNR_2 \rightarrow CH_2FCONR_2$$

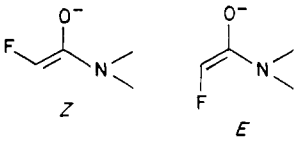
these amides to lithium diisopropylamide (LDA) in THF at temperatures between −75 °C and −85 °C generated the lithium enolates. These readily prepared enolates have been utilized in the directed aldol reaction (Table I). The

$$CH_2FCONR_2 + LDA \rightarrow LiCHFCONR_2$$

$$LiCHFCONR_2 + R'R''CO \rightarrow R'R''C(OH)CHFCONR_2$$

availability of compounds containing the fluoroacetate residue useful as enzyme inhibitors such as γ-fluoroglutamic acid or fluorocitric acid or a masked fluoroacyl unit such as 2-deoxy-2-fluororibose, required for preparation of novel nucleosides, has been limited by the difficulty of constructing specifically fluorinated molecules. The use of fluoroacetamide enolates is potentially more convenient than generation of the lithium enolate of commercially available ethyl fluoroacetate for preparing fluoro carbohydrates. In the case of *N*,*N*-diisopropylfluoroacetamide, the yield of aldol product is good, the amide starting material is a highly crystalline compound, and the frequently solid products were easily purified by recrystallization.

The failure of the enolates to react with greater diastereoselectivity results from a lack of stereoselectivity in the deprotonation of the amides. Attempted O-silylation of the enolate prepared from *N*,*N*-dimethylfluoroacetamide with chlorotrimethylsilane or *tert*-butyldimethylsilyl triflate was not successful. However, ^{19}F NMR studies of this enolate at −85 °C clearly indicated two distinct fluorine resonances in a 1:1 ratio at δ −213.15 (d, $J_{F,H}$ = 85.4 Hz) and δ −214.13 (d, $J_{F,H}$ = 71.7 Hz) attributable to a mixture of enolates. The resonance observed at higher field (δ −214.13) was broadened relative to the resonance at δ −213.14. The failure of the enolates to form with greater



stereoselectivity was disappointing, but similar results with simple amide enolates have been reported.[5] We are anxious to explore the effect of intramolecular chelation on fluorinated amide and imide enolates as significant improvements in selectivity have been observed in nonfluorinated molecules.[6]

Experimental Section

Aldehydes and ketones were purified by fractional distillation from calcium sulfate. THF and diethyl ether were purified by distillation from sodium benzophenone ketyl. Diisopropylamine

(1) (a) Welch, J. T.; Seper, K.; Eswarakrishnan, S.; Samartino, J. *J. Org. Chem.* **1984**, *49*, 4720–4721. (b) Welch, J. T.; Eswarakrishnan, S. *J. Chem. Soc., Chem. Commun.* **1985**, 186–188.

(2) Welch, J. T.; Seper, K. *Tetrahedron Lett.* **1984**, *25*, 5247–5250.

(3) Molines, H.; Massoudi, M. H.; Cantacuzene, D.; Wakselman, C. *Synthesis* **1983**, 322–324.

(4) (a) Mukaiyama, T. *Org. React.* (*N.Y.*) **1982**, *28*, 203–331. (b) Heathcock, C. H. In "Comprehensive Carbanion Chemistry"; Buncel, E., Durst, T., ed.; Elsevier: Amsterdam, 1984; Part B, Chapter 4.

(5) (a) Heathcock, C. H.; Buse, C. T.; Kleschick, W. A.; Pirrung, M. C.; Sohn, J. E.; Lampe, J. *J. Org. Chem.* **1980**, *45*, 1066–1081. (b) Takacs, J. M. Ph.D. Thesis, California Institute of Technology, Pasadena, 1981. (c) Heathcock, C. H. In "Asymmetric Synthesis"; Morrison, J. D., Ed.; Academic: Orlando, 1984; Vol. III, Chapter 2.

(6) (a) Evans, D. A. In "Asymmetric Synthesis"; Morrison, J. D., Ed.; Academic: Orlando, 1984; Vol. III, Chapter 1. (b) Evans, D. A.; Nelson, J. V.; Taber, T. R. *Top. Stereochem.* **1982**, *13*, 1–115.

(7) Hudlicky, M. "Chemistry of Organic Fluorine Compounds"; Ellis Horwood: Chichester, 1976; p 699.

was purified by distillation from calcium hydride. Infrared spectra were recorded on a Perkin-Elmer Model 710B infrared spectrometer. ^1H magnetic resonance spectra were determined on a Varian EM-360A, XL-300, or Bruker WH 90D spectrometer and are reported relative to tetramethylsilane. ^{13}C magnetic resonance spectra were determined on a Bruker WH 90D spectrometer and are reported relative to tetramethylsilane. ^{19}F magnetic resonance spectra were measured on a Varian XL-300 spectrometer and are reported relative to CCl_3F. Analytical samples were prepared by column chromatography on silical gel 200-425 (Davisil). Combustion analyses were performed by MicAnal (Tucson, AZ).

***N,N*-Dimethylfluoroacetamide.** To 30 mL of anhydrous diethyl ether at 0 °C containing 3.6 g (0.08 mol) of dimethylamine (Matheson) and 10.0 g of anhydrous potassium carbonate was dropwise added slowly 3.84 g (0.04 mol) of fluoroacetyl chloride (prepared by treatment of sodium fluoroacetate (Sigma) with phthaloyl chloride,[7] bp 65–71 °C) dissolved in 20 mL of anhydrous ether. After stirring at room temperature overnight, the product was filtered and concentrated in vacuo. The crude product, a colorless oil, isolated quantitatively, was used without further purification: ^1H NMR ($CDCl_3$) δ 4.72 (d, $J_{F,H}$ = 48 Hz, 2 H, CH_2F), 2.68 (d, $J_{F,H}$ = 2 Hz, 6 H, CH_3); ^{13}C NMR ($CDCl_3$) δ 166.1 (d, $J_{F,C}$ = 19.53 Hz, C=O), 78.6 (d, $J_{F,C}$ = 176.3 Hz, CH_2F), 34.91 (d, $J_{C,F}$ = 4.1 Hz, CH_3), 34.64 (CH_3); ^{19}F NMR ($CDCl_3$) δ −227.54 (t, $J_{F,H}$ = 45.8 Hz).

***N*-(Fluoroacetyl)pyrrolidine** was prepared as above. Crude product was a yellow solid which was purified by recrystallization from hexanes: mp 43–44 °C; ^1H NMR ($CDCl_3$) δ 4.85 (d, $J_{F,H}$ = 47 Hz, 2 H, CH_2F), 3.47 (t, 6.8 Hz, 2 H), 3.37 (t, 6.8, 2 H), 1.93 (p, 6.4, 2 H), 1.81 (p, 6.4, 2 H); ^{13}C NMR ($CDCl_3$) δ 165.3 (d, $J_{F,C}$ = 19.1 Hz, C=O), 79.8 (d, $J_{F,C}$ = 179.3 Hz, CFH_2), 46.0, 45.1 (d, $J_{F,C}$ = 5.0 Hz), 26.0, 23.50; ^{19}F NMR ($CDCl_3$) δ −227.75 (t, $J_{F,H}$ = 45.8 Hz).

Anal. Calcd for $C_6H_{10}FNO$: C, 54.95; H, 7.69. Found: C, 54.54; H, 7.73.

***N,N*-Diisopropylfluoroacetamide** was prepared as described and purified by recrystallization from hexanes or pentane: mp 61–62 °C; ^1H NMR ($CDCl_3$) δ 4.8 (d, $J_{F,H}$ = 47.4 Hz, 2 H, CH_2F), 3.67 (m, 1 H, CH), 4.3 (m, 1 H, CH), 1.37 (d, 6 H, $J_{H,H}$ = 5.86 Hz, $(CH_3)_2$), 1.17 (d, 6 H, $J_{H,H}$ = 5.37 Hz, $(CH_3)_2$); ^{13}C NMR ($CDCl_3$) δ 165.2 (d, $J_{F,C}$ = 18.1 Hz, C=O), 80.24 (d, $J_{F,C}$ = 177.3 Hz, CH_2F), 47.57 (CH), 45.85 (CH), 20.59 (CH_3), 20.11 (CH_3); ^{19}F NMR ($CDCl_3$) δ −224.67 (t, $J_{F,H}$ = 45.8).

Anal. Calcd for $C_8H_{16}FNO$: C, 59.60; H, 10.00. Found: C, 59.70; H, 10.08.

Typical Procedure for Enolate Formation and Aldol Reaction with *N,N*-Dimethylfluoroacetamide and *N*-(Fluoroacetyl)pyrrolidine. To a stirred round-bottomed flask containing 20 mL of anhydrous THF and 0.31 mL (0.0022 mol) of diisopropyl amine at 0 °C was added 1.4 mL (0.0022 mol) of a 1.5 M solution of methyllithium in diethyl ether. After 10 min of stirring at 0 °C and cooling to −85 °C, 0.002 mol of either *N,N*-dimethylfluoroacetamide or *N*-(fluoroacetyl)pyrrolidine dissolved in 2 mL of THF was added dropwise. After 5 additional min, 0.001 mol of the carbonyl compound dissolved in 2 mL of THF was rapidly added. The mixture was stirred for 5 min longer and then was quenched with 10 mL of saturated ammonium chloride. On warming to room temperature the mixture was diluted with 20 mL of distilled hexanes and was separated and the aqueous phase was extracted with three 10-mL portions of diethyl ether. The combined organic phases were washed twice with 20 mL of water, were dried over anhydrous magnesium sulfate, and were concentrated in vacuo to yield the crude product.

Typical Procedure for Enolate Formation and Directed Aldol Reaction with *N,N*-Diisopropylfluoroacetamide. LDA (0.015 mol) was prepared as described above in 20 mL of THF. After cooling to −75 °C, 0.24 g (0.0015 mol) of *N,N*-diisopropylfluoroacetamide dissolved in 2 mL of THF was added. After an additional hour at −75 °C, the substrate carbonyl compound (0.001 mol) dissolved in 2 mL of THF was rapidly added and was allowed to stir for 30 min. The reaction mixture was quenched and the products were isolated as described above.

Acknowledgment. We thank the Research Corporation and the donors of the Petroleum Research Fund, administered by the American Chemical Society, for support of this research.

Supplementary Material Available: Complete spectral and analytical data for all new compounds are available (11 pages). Ordering information is given on any current masthead page.

Synthesis of 4-, 5-, and 6-Methyl-2,2′-bipyridinyls[1]

Kevin T. Potts* and Paul A. Winslow

Department of Chemistry, Rensselaer Polytechnic Institute, Troy, New York 12181

Received April 23, 1985

2,2′-Bipyridinyls continue to attract appreciable attention with practical applications ranging from effective herbicides[2a] to important ligands of great interest for chelation of transition metals, the ruthenium complexes being useful for photochemical generation of hydrogen from water and in chemically modified electrode studies.[2b] 2,2′-Bipyridinyls are prepared by four principal routes: (a) from the reaction of 2,2′-bipyridnyl with alkyllithium reagents[3] that leads to isomeric products; (b) from an α,β-unsaturated ketone and an appropriately substituted acylpyridinium salt in the presence of hot ammonium acetate/acetic acid[4] (the Kröhnke procedure); (c) by coupling reactions of pyridine *N*-oxides with pyridine,[5] or nickel–phosphine complex mediated homocoupling of halopyridines;[6] (d) by radical substitution of 2,2′-bipyridinyl complexes of iron(III), ruthenium(III), or osmium(III), the methyl radicals being generated[7] by thermolysis of acetyl peroxide or by oxidative cleavage of alkyl metals such as $(CH_3)_4Sn$, $(CH_3)_4Pb$, or $(CH_3)_2Hg$. These methods lead to symmetrically substituted 2,2′-bipyridinyls or result in complex mixtures of isomers and fail to provide a general method for the convenient synthesis of a monoalkyl-2,2′-bipyridinyl.

We now describe unambiguous syntheses of 4-, 5- and 6-methyl-2,2′-bipyridinyls from the appropriate α-oxoketene dithioacetals and the enolate of a suitable carbonyl compound. Two choices exist for the synthesis of a 2,2′-bipyridinyl by this route: the reaction of 2-acetylpyridine with an appropriately functionalized α-oxoketene dithioacetal or, conversely, the reaction of an α-oxoketene dithioacetal derived from 2-acetylpyridine with the enolate of an appropriate carbonyl compound.

The syntheses of 6- and 5-methyl-2,2′-bipyridinyls were the most straightforward of the isomeric series (Scheme I). Thus, reaction of equimolar quantities of 3,3-bis(methylthio)-1-(2-pyridinyl)-2-propen-1-one (1) with the

(1) Abstracted from the Ph.D. Dissertation of P. A. W., RPI, 1984.

(2) For recent reviews see: (a) Summers, L. A. "The Bipyridinium Herbicides"; Academic Press: New York, 1980. (b) Skorobogaty, A.; Smith, T. D. *Coord. Chem. Rev.* **1984**, *53*, 55. See also: Abruña, H. D.; Denisevich, P.; Umana, M.; Meyer, T. J.; Murray, R. W. *J. Am. Chem. Soc.* **1981**, *103*, 1. Leidner, C. R.; Murray, R. W. *J. Am. Chem. Soc.* **1985**, *107*, 551. Ghosh, P. K.; Spiro, T. G. *J. Am. Chem. Soc.* **1980**, *102*, 5543. For other references relating to 2,2′-bipyridinyl chemistry see: Newkome, G. R.; Puckett, W. E.; Kiefer, G. E.; Gupta, V. K.; Xia, Y.; Coreil, M.; Hackney, M. A. *J. Org. Chem.* **1982**, *47*, 4116. Newkome, G. R.; Pappalardo, S.; Gupta, V. K.; Fronczek, F. R. *J. Org. Chem.* **1983**, *48*, 4848. Majestic, V. K.; Newkome, G. R. *Top. Curr. Chem.* **1982**, *105*, 79.

(3) Kauffmann, T.; Konig, J.; Woltermann, A. *Chem. Ber.* **1976**, *109*, 3864.

(4) Kröhnke, F. *Angew. Chem., Int. Ed. Engl.* **1963**, *2*, 225, 380.

(5) Cislak, F. E. U.S. Patent 2 992 224, 1961; *Chem. Abstr.* **1962**, *56*, 461.

(6) Tielcco, M.; Testaferri, L.; Tingoli, M.; Chianelli, D.; Montanucci, M. *Synthesis* **1984**, 736 and references listed therein. See also: Ogawa, S.; Kishii, N.; Shiraishi, S. *J. Chem. Soc., Perkin Trans. 1* **1984**, 2023.

(7) Rollick, L.; Kochi, J. K. *J. Org. Chem.* **1982**, *47*, 435.

0022-3263/85/1950-5405$01.50/0