II   V   M = Pd, Pt

CO in acetonitrile solution only on reflux, and the dicarbonyl species [(Cu⊂NPPh$_2$)$_2$Rh(CO)$_2$Cl](BF$_4$)$_2$ (13; ν_{CO} bands at 2075 and 1987 cm^{-1}) may be isolated as a yellow solid.[13] Surprisingly, if the dicationic complex 6 is refluxed in acetonitrile solution under an argon atmosphere for ca. 1 h, a disproportionation reaction occurs which can be observed by IR but is more easily investigated by ^{31}P NMR spectroscopy. The spectrum of the dication 6 in CH$_3$CN-d_3 (δ P 31.74, J_{Rh-P} 123 Hz) is transformed after a 1-h reflux (argon atmosphere) into a spectrum containing two 1:1 doublets of approximately equal intensities. One of these resonances (δ P 60.52, J_{Rh-P} 96 Hz) corresponds very well with the ^{31}P NMR spectrum of the dicarbonyl species 13. The other (δ P 74.54, J_{Rh-P} 118 Hz) is presumably due to a "carbonyl-free" Rh(I) complex, 14, (i.e., reaction 2). When CO is bubbled

$$2[(Cu \subset NPPh_2)_2Rh(CO)Cl](BF_4)_2 \xrightarrow[CH_3CN]{\Delta}$$

$$13 \quad + \quad [(Cu \subset NPPh_2)_2RhCl]_n(BF_4)_{2n} \quad (2)$$

$$14$$

$$\Delta, CO$$
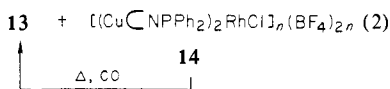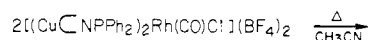
through a refluxing solution of 13 and 14, the resonances due to 14 disappear and those of 13 grow in intensity; i.e., 14 reacts with CO to give 13. The disproportionation (reaction 2) is, so far, unique to the dication 6. Neither the dications 8 and 9 nor the "Cu$^+$-free" complexes 3, 4, and 5 are affected by prolonged reflux in acetonitrile under argon. Although the exact nature of the species 14 is still under investigation, a plausible structure is IV. Support for P,S-bidentate complexation comes from the reaction 2a with PdCl$_2$(PhCN)$_2$, [PtCl$_2$(C$_2$H$_4$)]$_2$, and [(COD)RhCl]$_2$/2AgBF$_4$ (COD = cyclooctadiene) to give 15 (M = Pd, Pt, structure V) and [(COD)RhPPh$_2$-N⊃S](BF$_4$), 16.[14]

IV
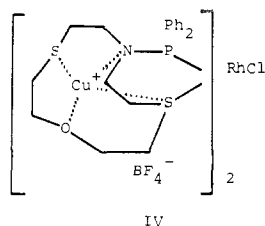
While the postulated structures II–IV are, at best, tenuous (and should be considered so until crystallographic data become available), the above results clearly indicate that phosphine functionalization of macrocyclic ligands is a viable route to new types of multimetallic systems. We are currently synthesising metal carbonyl complexes of 2a,b and their phosphine-derivatized aza-crown ether analogues with a view to promoting CO activation in a way analogous to previous studies involving chelating bis-phosphinite complexes with crown ether properties.[15]

(13) Anal. Calcd for C$_{42}$H$_{52}$B$_2$ClCu$_2$F$_8$N$_2$O$_3$P$_2$RhS$_4$: C, 39.54; H, 4.01; N, 2.14; Cl, 2.71. Found: C, 39.09; H, 4.59; N, 2.37; Cl, 2.76.

(14) Anal. Calcd for C$_{20}$H$_{26}$Cl$_2$NOPPdS$_2$ (15, M = Pd): C, 42.23; H, 4.61. Found: C, 42.48; H, 4.72. IR data (ν_{Pd-Cl} 305 and 274 cm^{-1}) support a *cis*-"PdCl$_2$" configuration. Anal. Calcd for C$_{20}$H$_{26}$Cl$_2$NOPPtS$_2$·0.5CHCl$_3$ (15, M = Pt): C, 34.64; H, 3.27; N, 1.96; Cl, 17.63. Found: C, 34.33; H, 3.72; N, 1.95; Cl, 17.30. IR ν_{Pt-Cl} 320 and 283 cm^{-1}. Complexes 15 are too insoluble for NMR or molecular weight studies. However, 15 (M = Pt) reacts with Tl(acac) to give S⊂N-Ph$_2$PPtCl(γ-acac), characterized by IR and ^{1}H NMR analysis. Anal. Calcd for C$_{28}$H$_{38}$BF$_4$NOPRhS$_2$ (16): C, 48.77; H, 5.50; N, 2.03. Found: C, 49.22, H, 5.73; N, 1.98.

(15) Powell, J.; Kuksis, A.; May, C. J.; Nyburg, S. C.; Smith, S. J. *J. Am. Chem. Soc.* 1981, *103*, 5941.

Characterization of the Vapor Species of Dilithiomethane by Flash Vaporization Mass Spectroscopy and the Ziegler Synthesis of Dilithiomethane

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The composition of the vapor species of the "electron-deficient" class of compounds, the lithiocarbons (C$_n$Li$_m$), is of widespread current interest. We report the first example of direct characterization and observation of the rather complex vapor species of polylithium organic compounds.

The vapor species of dilithiomethane, (CH$_2$Li$_2$)$_n$, are of particular interest in view of the possibility proposed by the work of Schleyer, Pople, and co-workers[1] of the existence of fluctional cis, tetrahedral, and possibly trans gas-phase forms. Curiously, the first report of a polylithium organic compound[2] (more than one lithium per carbon atom), the synthesis of (CH$_2$Li$_2$)$_n$ in 1955, went largely unnoticed until the early 1970s, when the field of polylithium organic compounds was firmly established by synthetic work in the laboratories of Lagow,[3] West,[4] and others.[5] This important Ziegler report went unrecognized, partly because it was 20 years ahead of its time and because it had gained a reputation for poor reproducibility in spite of a few reports of its derivative chemistry.[6]

Currently dilithiomethane, which may also be prepared by the reaction of lithium vapor with methylene chloride,[7] is best prepared

(1) J. B. Collins, J. D. Dill, E. D. Jemmis, Y. Apeloig, P. v. R. Schleyer, R. Seeger, and J. A. Pople, *J. Am. Chem. Soc.*, **98**, 5419 (1976).

(2) K. Ziegler, K. Nagel, and M. Patheiger, *Z. Anorg. Allgem. Chem.*, **282**, 345 (1955).

(3) (a) C. Chung and R. J. Lagow, *J. Chem. Soc., Chem. Commun.*, 1970 (1972). (b) L. A. Shimp and R. J. Lagow, *J. Am. Chem. Soc.*, **95**, 1343 (1973). (c) L. G. Sneddon and R. J. Lagow, *J. Chem. Soc., Chem. Commun.*, 302 (1975). (d) J. A. Morrison, C. Chung, and R. J. Lagow, *J. Am. Chem. Soc.*, **97**, 5015 (1975). (e) J. A. Morrison and R. J. Lagow, *Inorg. Chem.*, **16**, 2972 (1977). (f) L. A. Shimp, C. Chung, and R. J. Lagow, *Inorg. Chim. Acta*, **29**, 77–81 (1978). (g) K. M. Abraham and R. J. Lagow, *Tetrahedron Lett.*, 3, 211 (1979). (h) L. A. Shimp and R. J. Lagow, *J. Am. Chem. Soc.*, **101**, 2214 (1979). (i) L. A. Shimp and R. J. Lagow, *J. Org. Chem.*, **44**, 2231 (1979).

(4) (a) R. West and P. C. Jones, *J. Am. Chem. Soc.*, **91**, 6155 (1969). (b) G. A. Gornowicz and R. West, *ibid.*, **93**, 1720 (1971). (c) R. West, P. A. Carney, and I. C. Mimco, *ibid.*, **81**, 3788 (1965). (d) G. A. Gornowicz and R. West, *ibid.*, **93**, 1714 (1971). (e) W. Priester, R. West, and T. L. Chwang, *ibid.*, **98**, 8413 (1976).

(5) (a) A. F. Halasa and D. P. Tate, *J. Organomet. Chem.*, **24**, 796 (1970). (b) A. F. Halasa and D. P. Tate, *ibid.*, **31**, 369 (1971). (c) M. D. Raush and D. J. Ciappenelli, *ibid.*, **10**, 127 (1967). (d) J. Klein and A. Medlik-Balan, *J. Am. Chem. Soc.*, **99**, 1473 (1979). (e) J. Klein and A. Medlick-Balan, *Tetrahedron Lett.*, 279 (1978).
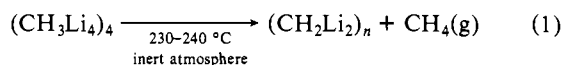
(6) P. Krohmer and J. Goubeau, *Chem. Ber.*, **104**, 1347 (1971).

(7) F. J. Landro, J. W. Chinn, Jr., J. A. Gurak, and R. J. Lagow, *J. Am. Chem. Soc.*, in press.

Table I. Composition of Hydrolysis Product (%)

CH_2D_2	95.1	C_2D_2	1.3
CH_3D	3.0	C_3D_4	0.6

by the Ziegler pyrolysis of methyllithium between 230 and 240 °C (eq 1).

$$(CH_3Li_4)_4 \xrightarrow[\substack{\text{inert atmosphere}}]{\text{230–240 °C}} (CH_2Li_2)_n + CH_4(g) \quad (1)$$

The reason for failure of the Ziegler synthesis to produce $(CH_2Li_2)_n$ in most modern laboratories has been the use of commercially available methyllithium $(CH_3Li)_{4-n}(LiX)_n$ (containing an equimolar amount of lithium halide) as a starting material rather than halide-free methyllithium prepared by the reaction of lithium and dimethylmercury.[8] Pyrolysis of such methyllithium proceeds very slowly under the Ziegler conditions and, at higher temperatures, reaction at a practical rate yields primarily lithium carbide, C_2Li_2, and occasionally small amounts of the West compound, $(C_3Li_4)_n$.[9] (eq 2).

$$(CH_3Li)_{4-n}(LiBr)_n \rightarrow \underset{95-100\%}{(C_2Li_2)_n} + \underset{0-5\%}{(C_3Li_4)_n} \quad (2)$$
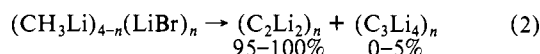
Hydrolysis of dilithiomethane synthesized by the Ziegler procedure was established solid-product yields of $(CH_2Li_2)_n$ (82%), C_2Li_2 (6%), and lithium (12%) plus lithium halide. Modification of Ziegler's synthesis has resulted in higher yields of $(CH_2Li_2)_n$ and less byproduct (eq 3).

$$(CH_3Li)_4 \xrightarrow[\substack{\text{vacuum, rapid, stirring}}]{\text{223–226 °C}}$$
$$\underset{95-96\%}{(CH_2Li_2)_n} + \underset{1-2\%}{(C_2Li_2)_n} + \underset{0.5-1\%}{(C_3Li_4)_n} + \underset{2-3\%}{(CH_3Li_4)_4} \quad (3)$$
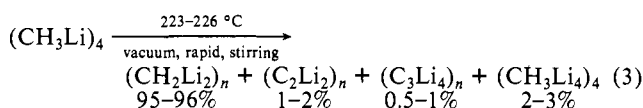
These yields were determined by hydrolyzing with D_2O vapor and analyzing the deuterated products with a temperature-programmed gas chromatograph incorporating a phenyl isocyanate/porasil C column and high-resolution mass spectroscopy. The results are listed in Table I.   No evidence for partially deuterated acetylene or propyne was observed.

In addition to the nature of the $(CH_3Li)_4$ precursor, the pyrolysis temperature and use of a very rapid stirring procedure are crucial factors in this modified synthesis. The use of vacuum conditions during the pyrolysis is also necessary for higher yields.

Halide-free methyllithium was prepared by literature methods.[8] The methyllithium prepared was pyrolyzed under vacuum within the temperature range 223–226 °C with continuous mixing. A high-temperature silicone-oil bath equipped with a magnetic stirring bar was employed for heating purposes. A second magnetic stirring bar was used to provide uniform mixing of the reacting materials. The pyrolysis was complete when the line pressure dropped below 1 μm (usually 8–10 h).

Care must be taken to ensure that neither stirring bar stops during the pyrolysis. This is especially critical for the temperature bath, as temperatures in excess of 250 °C have been observed when mixing ceased.

The results of this work and earlier studies have demonstrated that, like other lithiocarbons,[10] dilithiomethane is thermally stable only to 200–225 °C, at which point it begins to rearrange to species such as C_2Li_2 and C_3Li_4. Unfortunately, polylithium organic compounds have no observable vapor pressure below 650 °C, even in high vacuum (<10⁻⁶ torr or 10⁻⁴ Pa). Thus, they decompose or rearrange completely long before they vaporize, giving lithium carbide as the principle lithiocarbon product. It would appear, then, that unless a polylithium compound could be synthesized in the gas phase, the possibility of observing the gas-phase species predicted by Schleyer and others would be remote.

(8) An adaptation of the Ziegler procedure with $(CH_3Li)_{4-n}(LiI)_n$ can be made to produce some $(CH_2Li_2)_n$. These details will follow later: J. A. Gurak and R. J. Lagow, to be submitted for publication.

(9) R. West and P. C. Jones, *J. Am. Chem. Soc.*, **91**, 6155 (1969).

(10) L. A. Shimp, J. A. Morrison, J. A. Gurak, J. W. Chinn, Jr., and R. J. Lagow, *J. Am. Chem. Soc.*, **103**, 5951 (1981).
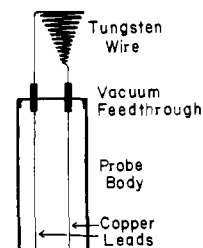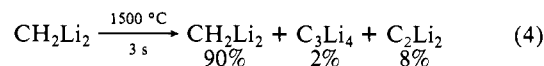
Figure 1. Probe tip of flash-vaporization apparatus.

However, we have recently observed that if flash heated (from room temperature to about 1500 °C in less than 3 s), CH_2Li_2 can be transported over a distance of 10 cm with less than 10% decomposition[10] (eq 4). It was this latter experiment that suggested

$$CH_2Li_2 \xrightarrow[\substack{3 s}]{1500 °C} \underset{90\%}{CH_2Li_2} + \underset{2\%}{C_3Li_4} + \underset{8\%}{C_2Li_2} \quad (4)$$
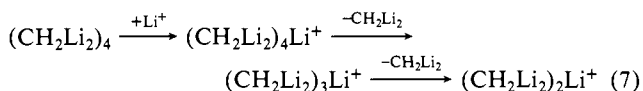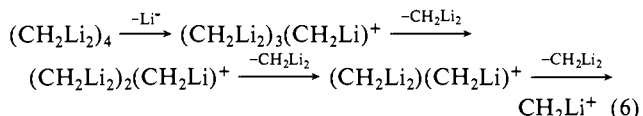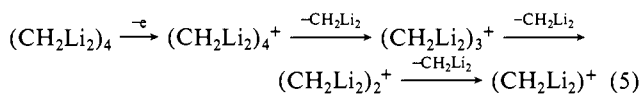
to us the possibility of producing vapor species in a mass spectrometer by flash vaporization.

A sample probe was constructed with a conically wound tungsten-wire tip (Figure 1), onto which a finely powdered sample was dusted. The extreme sensitivity of lithiocarbons to air and moisture made it necessary to use an isolation valve to transport the sample intact from our drybox to the spectrometer inlet. A Huntington all-metal, bakeable, high-vacuum valve was used for this purpose.

The sample probe was introduced into the spectrometer, a Finnigan Model 4023 automated GC–mass spectrometer with a quadrupole analyzer and Incos data system, to within a centimeter of the ionizing source, and the tungsten-wire tip was resistively heated with a variable transformer. The wire could be heated from room temperature to incandescence (1500–2000 °C) in less than 2 s. The ionizing energy (for EI mode) was maintained at 70.0 eV for this study, and the spectrometer was set to scan the full mass range of interest (usually 19–200 amu) in 1 s. Samples from several preparations were mass analyzed on separate days to remove any errors associated with the background of the instrument. Individual spectra were background substracted to eliminate any contaminant peaks associated with the vacuum system.

Table II lists common peaks found in dilithiomethane. Not only are the monomer through tetramer clusters present but also those ions plus and minus a lithium atom. These schemes are diagrammed in eq 5–7. Since the pyrolysis of methyllithium is

$$(CH_2Li_2)_4 \xrightarrow{-e} (CH_2Li_2)_4^+ \xrightarrow{-CH_2Li_2} (CH_2Li_2)_3^+ \xrightarrow{-CH_2Li_2}$$
$$(CH_2Li_2)_2^+ \xrightarrow{-CH_2Li_2} (CH_2Li_2)^+ \quad (5)$$

$$(CH_2Li_2)_4 \xrightarrow{-Li^-} (CH_2Li_2)_3(CH_2Li)^+ \xrightarrow{-CH_2Li_2}$$
$$(CH_2Li_2)_2(CH_2Li)^+ \xrightarrow{-CH_2Li_2} (CH_2Li_2)(CH_2Li)^+ \xrightarrow{-CH_2Li_2}$$
$$CH_2Li^+ \quad (6)$$

$$(CH_2Li_2)_4 \xrightarrow{+Li^+} (CH_2Li_2)_4Li^+ \xrightarrow{-CH_2Li_2}$$
$$(CH_2Li_2)_3Li^+ \xrightarrow{-CH_2Li_2} (CH_2Li_2)_2Li^+ \quad (7)$$

never complete, these spectra also contain a background of ions from that species. As indicated earlier, continued heating of dilithiomethane above 225 °C causes further rearrangements to C_3Li_4 and C_2Li_2. Thus, there will be a low-intensity background of ions from these compounds present.

Schleyer and co-workers have lately reported calculations on dimer[11] and trimer[12] species of dilithiomethane. These studies suggest that the lithium substituents are tightly bound in bridging positions leaving the hydrogens exposed to attack. If these structures are representative of the compound, then it is not

(11) E. D. Jemmis, P. v. R. Schleyer, and J. A. Pople, *J. Organomet. Chem.*, **154**, 327 (1978).

(12) P. v. R. Schleyer, private communication.

Table II. Selected Ions from the Flash-Vaporization Mass Spectrum of Dilithiomethane

possible ions	m/e	typical rel intensity, %
$CHLi_2$	27	42.1
N_2, CH_2Li_2	28	95.0
CH_3Li_2	29	37.4
CH_4Li_2	30	5.7
$(CH_3Li)CH_3$	37	3.1
C_2Li_2	38	6.9
C_2HLi_2	39	47.9
Ar, C_2Li_2	40	4.0
$C_2H_3Li_2$	41	100
$C_2H_4Li_2$	42	40.5
$C_2H_5Li_2$	43	5.3
CO_2, $(CH_3Li)_2$	44	15.2
C_2Li_3, $C_2H_7Li_2$	45	12.1
$(CH_2Li_2)(CH_2Li)$	49	2.9
C_3Li_2, $(CH_2Li_2)(CH_3Li)$	50	22.8
C_3HLi_2	51	8.3
$C_3H_2Li_2$, C_2Li_4	52	7.0
$C_3H_3Li_2$, C_2HLi_4	53	15.0
$C_3H_4Li_2$, $C_2H_2Li_4$	54	10.0
$C_3H_5Li_2$, $C_2H_3Li_4$	55	91.1
$C_3H_6Li_2$, $(CH_2Li_2)_2$	56	79.6
$C_3H_7Li_2$, $C_2H_5Li_4$, C_3Li_3	57	39.2
$C_3H_6Li_3$	63	2.1
C_3HLi_4	65	4.6
$C_3H_2Li_4$, C_2Li_6	66	3.1
$C_3H_3Li_4$, C_2HLi_6	67	17.0
$C_3H_4Li_4$, $C_2H_2Li_6$	68	9.5
$C_3H_5Li_4$	69	27.6
$C_3H_6Li_4$	70	48.2
C_3Li_5	71	62.3
C_3HLi_5	72	6.4
$C_3H_2Li_5$	73	4.4
$C_3H_3Li_5$	74	7.2
$C_3H_4Li_5$	75	5.9
$C_3H_5Li_5$	76	30.6
$C_3H_6Li_5$	77	9.2
$C_3H_7Li_5$, C_3Li_6	78	11.4
$C_3H_8Li_5$, C_3HLi_6	79	6.0
$C_3H_2Li_6$	80	2.5
$C_3H_3Li_6$	81	7.8
$C_3H_4Li_6$	82	8.0
$C_3H_5Li_6$	83	18.0
$C_3H_6Li_6 = (CH_2Li_2)_3$	84	9.5
$C_3H_7Li_6$	85	5.3
$C_3H_6Li_7$	91	3.7
$C_4H_3Li_6$	93	2.8
$C_4H_4Li_6$	94	3.3
$C_4H_5Li_6$	95	11.5
$C_4H_6Li_6$	96	3.4
$C_4H_7Li_6$	97	7.6
$(CH_2Li_2)_3(CH_2)$	98	5.2
$(CH_2Li_2)_3(CHLi)$	104	15.6
$(CH_2Li_2)_3(CH_2Li)$	105	2.9
$(CH_2Li_2)_3(CLi_2)$	110	3.6
$(CH_2Li_2)_3(CHLi_2)$	111	2.4
$(CH_2Li_2)_4$	112	7.7
$(CH_2Li_2)_5$	140	<1.0
$(CH_2Li_2)_6$	168	<1.0

surprising that there are many fragment progressions in which subsequent hydrogens are lost.

These results suggest the possibility of condensing dilithiomethane into inert low-temperature matrices to seek evidence for unusual structural features (such as the proposed cis and trans isomers) by vibrational spectroscopy and other methods. It also opens the door to the possibility of using other types of gas-phase techniques for characterizing these interesting vapor species.

Electron Transfer from Nitrogen in Microsomal Oxidation of Amine and Amide. Simulation of Microsomal Oxidation by Anodic Oxidation[1]

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In enzymatic reactions catalyzed by the cytochrome *P*-450 monooxygenase system, secondary and tertiary amines and amides are oxidized to give N-dealkylated amines and amides.[2] In a number of studies on the mechanism of reaction of the *P*-450, an intermediate containing an iron–oxygen species has been proposed to be involved in some step following the initial formation of a complex of *P*-450–substrate–O_2.[3] However, the mechanism of the cytochrome *P*-450 catalyzed N-dealkylation reaction, especially that of the oxidation step of amines and amides, still remains vague. The initial formation of aminium cation radicals from amines by one-electron transfer from nitrogen to oxidizing agents has been described as one of the possibilities.[4,5]

We have reported previously that anodic oxidation is an effective method to simulate the microsomal oxidation of amines and to prepare N-dealkylated metabolites from unstable drugs under mild conditions.[6]

Since the reaction mechanism of anodic oxidation of amines and amides has been rather clearly established,[7] we compared the selectivity of liver microsomal N-dealkylation of *N,N*-dialkyl amines and amides with that of anodic oxidation to get an insight into the cytochrome *P*-450 catalyzed N-dealkylation. The substrates used in the present study were N-substituted and deuterated derivatives of imipramine, an antidepressant drug, and of hydrocinnamide.[8] The microsomal oxidation was carried out with

(1) Electroorganic Chemistry. 57.

(2) (a) McMahon, R. E. *J. Pharm. Sci.* **1966**, *55*, 457. (b) Fish, M. S.; Johson, N. M.; Horning, E. C. *J. Am. Chem. Soc.* **1956**, *78*, 3668. (c) McMahon, R. E.; Culp, H. W.; Occolowitz, J. C. *Ibid.* **1969**, *91*, 3389.

(3) (a) Hrycay, E. G.; Gustafsson, J. A.; Sundberg, M. I.; Ernster, L. *Biochem. Biophys. Res. Commun.* **1975**, *66*, 209. (b) Lichtenberger, F.; Nastajnczyk, W.; Ullrich, V. *Ibid.* **1976**, *70*, 939. (c) Nordblom, G. D.; White, R. E.; Coon, M. J. *Arch. Biochem. Biophys.* **1976**, *175*, 524. (d) Groves, J. T.; Nemo, T. E.; Myers, R. S. *J. Am. Chem. Soc.* **1979**, *101*, 1032. (e) Chang, C. K.; Kuo, M. S. *Ibid.* **1979**, *101*, 3413.

(4) In the oxidation of amines catalyzed by mitochondrial monoamine oxidase, two one-electron transfers from the substrate to the flavin have been proposed.[5c]

(5) (a) Griffin, B. W.; Ting, P. L. *Biochemistry* **1978**, *17*, 2206. (b) Shannon, P.; Bruice, T. C. *J. Am. Chem. Soc.* **1981**, *103*, 4580. (c) Silverman, R. B.; Hoffman, S. J.; Williams, B. C. *Ibid.* **1980**, *102*, 7126.

(6) Shono, T.; Toda, T.; Oshino, N. *Drug Metab. Dispos.* **1981**, *9*, 481.

(7) In the anodic oxidation of amines and amides in methanol, the first products are the methoxylated compounds at the position α to nitrogen. The α-methoxylated products are, however, easily hydrolyzed to the N-dealkylated compounds in the solution or in the working up with acid. Thus, the selectivity of N-dealkylation in anodic oxidation is just the same as that of anodic α-methoxylation.

(8) Imipramine, demethylimipramine, and dedimethylimipramine were supplied from Schering AG (Berlin, GFR). The substrates **1**, **2**, and **3** were prepared from corresponding alkyl halides according to the general method.[9] Compound **7** was prepared by $LiAlD_4$ reduction of carbamate of demethylimipramine, obtained from the treatment of the amine with ethyl chloroformate.[10,11] NMR (CDCl₃) δ 7.30–6.70 (m, 8, aromatic), 3.75 (t, 2, iminodibenzyl N–CH₂), 3.15 (br s, 4, iminodibenzyl (CH₂)₂), 2.50–1.95 (m, 2, α-CH₂), 2.15 (s, 3, N–CH₃), 2.05–1.40 (m, 2, β-CH₂). The substrates **4–6** and **8** were prepared from the reaction of acyl chloride with corresponding amines.[12] NMR (CDCl₃) δ 7.40–7.05 (m, 5, phenyl), 3.15–2.85 (m, 2, benzyl CH₂), 2.95 (s, 2, N–CH₃), 2.75–2.45 (m, 2, α-CH₂).

(9) Deno, N. C.; Fruit, R. E. *J. Am. Chem. Soc.* **1968**, *90*, 3506.

(10) Lindeke, B.; Anderson, E.; Jenden, D. J. *Biomed. Mass. Spectrom.* **1976**, *3*, 257.

(11) Marshall, F. J.; McMahon, R. E. *J. Labelled Compd. Radiopharm.* **1970**, 261.

(12) Marvel, C. S.; Lazier, W. A. *Org. Synth. Collect.* **1941**, *1*, 99.