

ether as prisms: mp 170–171.5 °C; yield 156 mg (80%).

Anal. Calcd for $C_{13}H_{17}NO_6S$: C, 49.51; H, 5.44; N, 4.44; S, 10.17. Found: C, 49.79; H, 5.59; N, 4.18; S, 10.10.

Methyl *trans*-3-Methylpyroglutamate (4). A suspension of 7 (1.00 g, 6.99 mmol) in methanol (10 mL) was treated with an excess of ethereal diazomethane. After evaporation the residual solid was recrystallized from ethyl acetate/petroleum ether as plates: mp 70–71 °C; yield 1.05 g (95%); NMR, see Table I.

Anal. Calcd for $C_7H_{11}NO_3$: C, 53.49; H, 7.06; N, 8.91. Found: C, 53.31; H, 6.98; N, 8.76.

***N*-(*p*-Toluenesulfonyl)-*cis*-3-methylprolinol (5).** A solution of 2 (855 mg, 5.00 mmol) in dry tetrahydrofuran (10 mL) was gradually added to a stirred suspension of powdered lithium aluminum hydride (532 mg, 14 mmol) in dry tetrahydrofuran (15 mL) in a stream of nitrogen. After being heated under reflux for 18 h, the mixture was cooled and treated with acetone (5 mL) for 1 h. Water (20 mL) and sodium bicarbonate (1.0 g) were added, and the mixture was stirred during addition of *p*-toluenesulfonyl chloride (955 mg, 5.01 mmol) in acetone (15 mL). After 2 h, 1 N hydrochloric acid (75 mL) was added and the solution was extracted with ethyl acetate. The extracts were washed with aqueous sodium bicarbonate and sodium chloride and dried (Na_2SO_4). Evaporation gave a gum which was chromatographed on a column (17 × 2 cm) of silica gel with 2:1 chloroform/ethyl acetate. The residue from the appropriate fractions crystallized from ethyl acetate/petroleum ether as prisms: mp 74–75 °C; yield 848 mg (63%); NMR ($CDCl_3$, internal Me_4Si) δ 0.98 (d, $J = 6.3$ Hz, 3- CH_3 , 3), 2.44 (s, $ArCH_3$, 3), 7.34 (d, $Ar H$, 2), 7.75 (d, $Ar H$, 2).

Anal. Calcd for $C_{13}H_{19}NO_3S$: C, 57.96; H, 7.11; N, 5.20; S, 11.91. Found: C, 57.79; H, 7.33; N, 5.28; S, 12.18.

***N*-(*p*-Toluenesulfonyl)-*trans*-3-methylprolinol (8)** was prepared from 4 (785 mg, 5.00 mmol) by the same procedure as the above (2 → 5) and crystallized from ethyl acetate/petroleum ether at -15 °C as needles: mp 61–62 °C; yield 873 mg (65%); NMR ($CDCl_3$, internal Me_4Si) δ 0.67 (d, $J = 6.9$ Hz, 3- CH_3 , 3), 1.01 (dd, $J = 7.6$ and 12.4 Hz, 4 α -H, 1), 1.86 (dd, $J = 6.2$ and 12.4 Hz, 4 β -H, 1), 2.05 (m, 3-H, 1), 2.45 (s, $ArCH_3$, 3), 7.35 (d, $Ar H$, 2), 7.75 (d, $Ar H$, 2).

Anal. Calcd for $C_{13}H_{19}NO_3S$: C, 57.96; H, 7.11; N, 5.20; S, 11.91. Found: C, 57.72; H, 7.13; N, 5.06; S, 11.52.

Oxidation of 5 to *N*-(*p*-Toluenesulfonyl)-*cis*-3-methylproline (9). A solution of 5 (641 mg, 2.38 mmol) in acetone (40

mL) was mixed with a solution of chromium trioxide (1.20 g, 12.0 mmol) in water (5 mL) containing sulfuric acid (1 mL). After 1.5 h, methanol (10 mL) was added, and after a further 15 min, the mixture was poured into aqueous sodium chloride (200 mL) and extracted with ethyl acetate. The extracts were washed with aqueous sodium chloride and dried (Na_2SO_4). A small aliquot was treated with ethereal diazomethane and analyzed by GC on column B at 230 °C. The product was 95% *cis* and 5% *trans* when compared with the authentic diastereoisomers of *p*-toluenesulfonyl-3-methylproline methyl ester (retention times 15.7 and 13.6 min, respectively). The bulk of the extract was evaporated and the residual solid recrystallized from ethyl acetate as needles, mp 181.5–183 °C, alone or mixed with an authentic sample (mp 181.5–183 °C) prepared from *cis*-3-methylproline,^{10,14} yield 573 mg (84%).

Oxidation of 8 to *N*-(*p*-Toluenesulfonyl)-*trans*-3-methylproline (12). A solution of 8 (369 mg, 1.37 mmol) in acetone (23 mL) was mixed with a solution of chromium trioxide (583 mg, 5.83 mmol) in water (2.5 mL) containing sulfuric acid (0.5 mL). After 1.5 h, the reaction was worked up as for 9 and similar GC analysis indicated that the crude product was 99% *trans*. After recrystallization from ethyl acetate/petroleum ether, 12 formed needles, mp 123–124 °C, alone or mixed with an authentic sample (mp 123–124 °C) prepared from *trans*-3-methylproline,^{10,14} yield 361 mg (93%).

Acknowledgment. I thank Dr. E. Becker (National Institute of Arthritis, Metabolism and Digestive Diseases, NIH) for the use of an NMR spectrometer and Dr. D. F. Johnson (same Institute) for analytical services. This investigation was supported by U.S. Public Health Service Grant CA-11627 from the National Cancer Institute.

Registry No. 1, 2446-12-0; 2, 76318-70-2; 3, 76318-71-3; 4, 76251-46-2; 5, 76251-47-3; 6, 76318-72-4; 7, 76318-73-5; 8, 76251-48-4; 9, 76318-74-6; 10, 63088-04-0; 11, 76318-75-7; 12, 76318-76-8; 13, 76251-49-5; 14, 76251-50-8; methyl *cis*-3-methylpyroglutamate, 76251-51-9; methyl *N*-(trifluoroacetyl)-*erythro*- β -methylglutamate, 76251-52-0; methyl *N*-(trifluoroacetyl)-*threo*- β -methylglutamate, 76251-53-1; glutamic acid, 617-65-2.

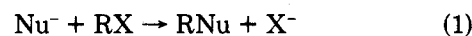
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Communications

Correlation between the Basicity of Carbanions and Their Ability to Transfer an Electron

Summary: In Me_2SO solution electron transfer from 9-arylfuorenyl carbanions, 9- $ArFl^-$, to electron acceptors including $PhSO_2CH_2Br$, $PhSO_2CH_2I$, or $R_2C(NO_2)_2$ follows second-order kinetics and results in the formation of dimers, $(9-ArFl^-)_2$, in high yields. Brønsted-type correlations of $\log k_2$ vs. pK_a (Table I) show that these electron-transfer processes have a much higher sensitivity to changes in 9- $ArFl^-$ ion basicity ($\beta_{Nu} > 1.0$) than do S_N2 reactions of RX with these carbanions ($\beta_{Nu} = 0.3-0.5$).

Sir: Carbanions and other nucleophiles are known to react in dipolar nonhydroxylic ("aprotic") solvents with electron acceptors, RX , of the type $p-NO_2C_6H_4CR_2X$ ($X = Cl$ or NO_2) and NO_2CR_2X ($X = Cl$ or Br) by substitution reactions (eq 1) involving electron-transfer chain mechanisms.^{1,2}



A number of other electron-transfer reactions are known in which the nucleophile is dimerized;^{3,4} for example, a few 9-substituted fluorenyl anions, 9- $G-Fl^-$, have been shown to react with nitrobenzene to form $(9-G-Fl^-)_2$ dimers.³ We have observed similar dimerizations of 9- $ArFl^-$ anions in reactions with excess $PhSO_2CH_2Br$, $PhSO_2CH_2I$, $Me_2C(NO_2)_2$, and 1,1-dinitrocyclohexane ($c-C_6H_{10}(NO_2)_2$) as electron acceptors. The major products (>85% material

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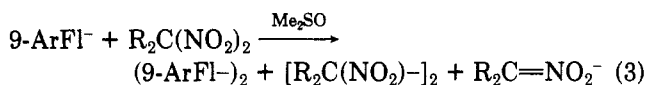
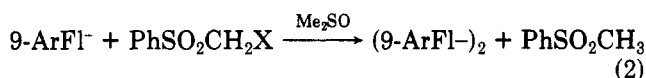
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Table I. Reactions of $\text{PhSO}_2\text{CH}_2\text{X}$ and $\text{R}_2\text{C}(\text{NO}_2)_2$ with 9-Substituted Fluorenyl Carbanions, 9-G- Fl^- , in Dimethyl Sulfoxide at 25 °C

electron acceptor ^a	G ^b	pK_a^c	$10k_2, \text{M}^{-1} \text{s}^{-1}{}^d$
$\text{PhSO}_2\text{CH}_2\text{Cl}$ (45-62)	CH_3	22.3	0.14 ± 0.01
$\text{PhSO}_2\text{CH}_2\text{Br}$ (15-33)	<i>m</i> - ClC_6H_4	16.8	0.158 ± 0.008
$\text{PhSO}_2\text{CH}_2\text{Br}$ (7-10)	C_6H_5	17.9	3.03 ± 0.12
$\text{PhSO}_2\text{CH}_2\text{Br}$ (8-10)	<i>p</i> - $\text{CH}_3\text{C}_6\text{H}_4$	18.3	8.14 ± 0.34
$\text{PhSO}_2\text{CH}_2\text{I}$ (8-16)	<i>m</i> - ClC_6H_4	16.8	0.113 ± 0.007
$\text{PhSO}_2\text{CH}_2\text{I}$ (7-12)	C_6H_5	17.9	2.81 ± 0.25
$\text{PhSO}_2\text{CH}_2\text{I}$ (8-22)	<i>p</i> - $\text{CH}_3\text{C}_6\text{H}_4$	18.3	8.87 ± 0.32
$\text{Me}_2\text{C}(\text{NO}_2)_2$ (16-26)	<i>m</i> - ClC_6H_4	16.8	2.45 ± 0.91
$\text{Me}_2\text{C}(\text{NO}_2)_2$ (5-12)	C_6H_5	17.9	66.0 ± 8.2
$\text{Me}_2\text{C}(\text{NO}_2)_2$ (6-10)	<i>p</i> - $\text{CH}_3\text{C}_6\text{H}_4$	18.3	140 ± 10
<i>c</i> - $\text{C}_6\text{H}_{10}(\text{NO}_2)_2$ (7-44)	<i>m</i> - ClC_6H_4	16.8	6.07 ± 0.78
<i>c</i> - $\text{C}_6\text{H}_{10}(\text{NO}_2)_2$ (2-4)	C_6H_5	17.9	175 ± 23
<i>c</i> - $\text{C}_6\text{H}_{10}(\text{NO}_2)_2$	<i>p</i> - $\text{CH}_3\text{C}_6\text{H}_4$	18.3	too fast

^a Molar excess in parentheses. ^b G in 9-G- Fl^- . ^c pK_a of 9-G- FlH in Me_2SO . ^d These are averages of three or four runs carried out to 1-2 half-lives with R^2 values of 0.999 (R^2 is the least-squares fit of the first-order plot).

balance) are given in eq 2 and 3. The reactions are believed to occur by single electron transfer.⁵



Pseudo-first-order kinetics were observed in these reactions for 1-3 half-lives. Variation of the excess electron-acceptor concentrations showed the reactions to be first order in the electron acceptor as well as in the electron donor (Table I). Brønsted-type plots of $\log k_2$ vs. pK_a drawn by using the data shown in Table I were found to be linear ($R^2 \geq 0.99$) with slopes of 1.15, 1.27, 1.20, and 1.32 for $\text{PhSO}_2\text{CH}_2\text{Br}$, $\text{PhSO}_2\text{CH}_2\text{I}$, $\text{Me}_2\text{C}(\text{NO}_2)_2$, and *c*- $\text{C}_6\text{H}_{10}(\text{NO}_2)_2$, respectively. These appear to be the first Brønsted-type correlations recorded for an electron-transfer reaction. The high β_{Nu} values (>1) are noteworthy. We associate this unusually high sensitivity of rates to changes in basicity with a rate-limiting electron transfer (eq 4).

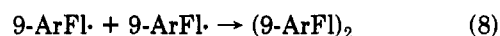
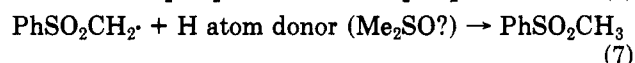
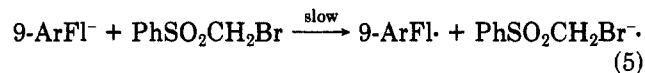


This interpretation is supported strongly by the observation that oxidation potentials for these 9- ArFl^- anions in Me_2SO are also highly sensitive to changes in their basicities (ΔpK , kcal/mol); i.e., $\Delta V/\Delta\text{pK} = 1.08$ ($R^2 = 0.99$).⁶ Also, the reduction potentials in Me_2SO for the electron acceptors show a correlation with the rates: $\text{PhSO}_2\text{CH}_2\text{Cl}$ (-1.5 V), $\text{PhSO}_2\text{CH}_2\text{Br}$ (-0.6 V), $\text{PhSO}_2\text{CH}_2\text{I}$ (-0.5 V), $\text{Me}_2\text{C}(\text{NO}_2)_2$ (-0.5 V). Note that the latter three have comparable reduction potentials and comparable rates with 9- ArFl^- , whereas $\text{PhSO}_2\text{CH}_2\text{Cl}$, which has a 0.9-1.0-V more negative reduction potential, fails to react with these anions and reacts only slowly with 9- MeFl^- which is 4.0-5.5 pK units more basic (Table I). The dimer (9- MeFl)₂ is formed in the latter reaction.

(5) Hua, D. H.; Kolb, V. M.; Meyers, C. Y. Northeast Regional Meeting of the American Chemical Society, Potsdam, NY, June 29-July 3, 1980. These authors have suggested that reductions of α -bromo sulfones by Zn-MeOH or $\text{Na}_2\text{SO}_3\text{-MeOH-H}_2\text{O}$ occur by electron-transfer mechanisms.

(6) The electrochemical experiments were carried out in collaboration with John Begemann and Professor Donald E. Smith. Measurements with glassy carbon, platinum, and hanging mercury drop electrodes gave comparable results.

These reactions are believed to be nonchain or short-chain electron transfers of the type shown in eq 5-9 since second-order kinetics are observed for as much as 2 or 3 half-lives.



Rates of reactions of these electron acceptors with 9-G- Fl^- anions generated from 9-G- FlH compounds with pK_a 's of 6-15 are too slow to measure by our spectrophotometric method at 25 °C, whereas those anions derived from compounds with pK_a 's above 19 are too fast to measure. This is a consequence of the high sensitivity of these rates to changes in basicity ($\beta_{\text{Nu}} = 1.15\text{-}1.32$). This behavior contrasts sharply with that of the same anions undergoing $\text{S}_{\text{N}}2$ -type reactions with PhCH_2Cl ⁷ or many other types of alkyl halides (BuCl , BuBr , BuI , $\text{CH}_2=\text{CHCH}_2\text{Cl}$, $\text{HC}\equiv\text{C-CH}_2\text{Cl}$, *c*- $\text{C}_6\text{H}_{11}\text{Br}$, ClCH_2CN , BrCH_2CN , etc.) where β_{Nu} values usually fall in the range 0.3-0.5.⁸ For example, with PhCH_2Cl , β_{Nu} is sufficiently low (~ 0.3) for us to measure rates at 25 °C for anions derived from 9-G- FlH compounds with pK_a 's varying from 6 to 22.⁷ These results lead us to expect $\text{S}_{\text{N}}2$ -type mechanisms to take precedence over electron-transfer mechanisms for most alkyl halides reacting in the dark with carbanions in this pK region.⁹ The emergence of the electron-transfer mechanism for $\text{PhSO}_2\text{CH}_2\text{X}$ halides can be attributed to (a) retardation of $\text{S}_{\text{N}}2$ -type mechanisms for "steric" reasons^{10,11} and (b) acceleration of electron transfer due to the presence of the powerfully electron-withdrawing PhSO_2 group.¹¹

Factors recognized previously as favoring an electron transfer from a nucleophile, Nu^- , to an electrophile, RX , over a (formal) two-electron transfer (e.g., as in $\text{S}_{\text{N}}2$ reactions) include (a) a poor leaving group, X, (b) steric hindrance in RX , and (c) a strong electron acceptor in R .¹⁴ Our results suggest that the basicity of the anion is another important factor—the more strongly basic the carbanion the greater the likelihood that it will react by an electron-transfer pathway rather than an $\text{S}_{\text{N}}2$ pathway.^{15,16}

(7) Bordwell, F. G.; Hughes, D. L. *J. Org. Chem.* 1980, 45, 3314-3320.

(8) Bausch, M. J.; Clemens, A. H.; Hughes, D. L.; Romberg, S. D., unpublished results.

(9) It seems likely that this will hold also for nitranions and for oxanions. We are investigating the behavior of these anions and of thianions.

(10) Bordwell, F. G.; Jarvis, B. B. *J. Org. Chem.* 1968, 33, 1182-1185. We use the term "steric" in its broadest sense to include all types of rate retardations caused by proximity effects such as repulsions between bonds, repulsions between lone pairs, restrictions of bond rotations, etc.

(11) The powerful electron-withdrawing nature of the PhSO_2 group is demonstrated by its ability to increase the acidity in Me_2SO of the C-H bond in PhSO_2CH_3 relative to that in CH_4 by at least 25 pK_a units.¹²

(12) A conservative estimate places the pK_a of methane at about 55 in a dipolar, nonhydroxylic solvent like Me_2SO .¹³

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(14) Kornblum, N. *Angew. Chem., Int. Ed. Engl.* 1975, 14, 734-745.

(15) Meyers, C. Y.; Hsu, Mao-Lin, "Abstracts of Papers", 170th National Meeting of the American Chemical Society, Chicago, IL, Aug 1975; American Chemical Society: Washington, DC, 1975; ORGN-45. These authors have presented results concerning the reactions of ArSNa nucleophiles with Ph_3CCl in various solvents which they interpret as indicating that an electron-transfer pathway is more sensitive to changes in basicity of the nucleophile than is a nucleophilic pathway.

(16) The products reported herein have been identified by melting point and NMR and mass spectral analysis.

Acknowledgment. We are grateful to the donors of the Petroleum Research Fund, administered by the American Chemical Society, for support.

Registry No. PhSO₂CH₂Cl, 7205-98-3; PhSO₂CH₂Br, 19169-90-5; PhSO₂CH₂I, 65492-21-9; Me₂C(NO₂)₂, 595-49-3; *c*-C₆H₁₀(NO₂)₂, 4028-15-3; 9-CH₃-Fl⁻, 31468-21-0; 9-*m*-ClC₆H₄-Fl⁻, 73872-45-4; 9-C₆H₅-Fl⁻, 31468-22-1; 9-*p*-CH₃C₆H₄-Fl⁻, 42730-14-3.

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Synthesis of Quaternary Carbon Compounds¹

Summary: A general synthesis of quaternary carbon compounds RR'R''CCH₂NO₂ which gives excellent yields of pure products is described. Of special interest is the preparation of quaternary aldehydes by permanganate oxidation of these nitro compounds.

Sir: A number of simple and effective procedures are now available for the synthesis of tertiary nitroparaffins.²⁻¹² Most of them are carbon-carbon bond forming processes, and they enable one to prepare highly branched compounds which are virtually unobtainable by other means. Another attractive feature of these reactions is their capability for providing tertiary nitro compounds in which other functional groups are present, e.g., keto, ester, and cyano.

Of the common functional groups, a tertiary nitro group is the most susceptible to electron-transfer substitution, and this has enabled us to devise a general procedure for converting tertiary nitro compounds into quaternary carbon compounds.^{13,14} Equation 1 is illustrative, and

(1) This is paper 25 in the series "Substitution Reactions Which Proceed via Radical Anion Intermediates". For the preceding paper see N. Kornblum, P. Ackermann and R. T. Swiger, *J. Org. Chem.*, 45, 5294 (1980).

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(11) N. Kornblum, S. C. Carlson, J. Widmer, M. J. Fifolt, B. N. Newton, and R. G. Smith, *J. Org. Chem.*, 43, 1394 (1978).

(12) G. A. Russell, J. Hershberger, and K. Owens, *J. Am. Chem. Soc.*, 101, 1312 (1979).

(13) Previously the conversion of tertiary nitro compounds to quaternary carbon compounds has only been possible with systems in which the intermediate free radical of eq 3 and 4 is resonance stabilized, e.g., with Me₂C(NO₂)COOEt. Now, by employment of NaH in Me₂SO, it is possible to achieve this transformation even with compounds such as *tert*-nitrobutane and *tert*-nitrooctane (Table I).

(14) A recent review by S. F. Martin [*Tetrahedron*, 36, 419 (1980)] describes the problems associated with the construction of quaternary carbon centers.

Table I. Replacement of NO₂ by CH₂NO₂^a

tertiary nitro compd	reaction time, h	primary nitro compd	yield, ^b %
+NO_2	4	$\text{+CH}_2\text{NO}_2$	60
$\text{+CH}_2\text{+NO}_2$	4	$\text{+CH}_2\text{+CH}_2\text{NO}_2$	60
	4		75
EtOOC+NO_2	16 ^c	$\text{EtOOC+CH}_2\text{NO}_2$	88 ^d
+O-C(=O)-+NO_2	5	$\text{+O-C(=O)-+CH}_2\text{NO}_2$	95
NC+NO_2	2	$\text{NC+CH}_2\text{NO}_2$	91
	3		81
	2		91
	2		89
	5		85
	18 ^e		84
	18 ^e		87
	18 ^e		95
	140 ^e		83
	4		89
	3		76
	42 ^e		60 ^f

^a All reactions were carried out at 25 °C in Me₂SO with exposure to two 20-W fluorescent lights. Unless otherwise noted the molar ratio of tertiary nitro compound to nitromethane to NaH is 1:4:8. ^b Pure, isolated product. ^c No free NaH present. ^d This experiment first carried out by Dr. R. Boss. ^e Less than the standard amount of NaH was employed (25% to 100% excess relative to the tertiary nitro compound). ^f This product contains ca. 5% *p*-NCC₆H₄C(Me)₂CH₂NO₂ which derives from fragmentation of the β-nitroaryl radical anion [cf. eq 18 of N. Kornblum, J. Widmer, and S. C. Carlson, *J. Am. Chem. Soc.*, 101, 661 (1979)].

Tables I and II summarize our results; yields refer to pure, isolated products.

