

reported in Table II for series V and VI represent the average of at least two determinations. The maximum deviation from the mean of replicate pK_a values did not exceed 1.5% except for the 3-Cl compound of series VI (2.2%).

The pK_a 's of series XI (Table V) were determined in water at 25 °C either by potentiometric titration^{10b} of the sodium salt of the acid (for G = H, 3-Me, 4-Me, 4-OMe, and 4-F) or by conductimetry^{10c} (for G = 3-OMe, 3-Cl, 4-Cl, and 4-NO₂). The pK_a 's determined by potentiometry are the average of three determinations and those determined by conductimetry are the average of two determinations. The maximum deviation from the mean of replicate pK_a values did not exceed 0.9% for any of the compounds studied.

The substituent chemical shifts (SCS) in hertz for series VIII, IX, and X (Table III) and the *N*-methyl amide series listed in ref 11 were measured on a Varian T-60 spectrometer vs. Me₄Si at 37 °C either in 10% (w/v) Me₂SO-*d*₆ or in 10% (w/v) CCl₄ solutions. All of the values reported represent the average of at least three determinations. The maximum deviation from the mean of replicate SCS values did not exceed 0.7% for any of the

compounds in any of the series studied.

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Registry No. V (G = H), 16777-78-9; V (G = 4-Me), 72269-13-7; V (G = 4-Cl), 59291-79-1; V (G = 3-Cl), 59291-77-9; V (G = 4-OMe), 72269-14-8; V (G = 3-OMe), 72269-15-9; V (G = 4-NO₂), 72269-16-0; VI (G = H), 582-80-9; VI (G = 4-Me), 72269-17-1; VI (G = 3-Me), 72269-18-2; VI (G = 4-Cl), 70204-56-7; VI (G = 3-Cl), 70204-55-6; VI (G = 4-OMe), 28547-12-8; VI (G = 4-NO₂), 19717-14-7; VIII (G = NO₂), 50440-54-5; VIII (G = Cl), 72269-19-3; VIII (G = H), 72269-20-6; VIII (G = Me), 72269-21-7; VIII (G = MeO), 72269-22-8; IX (G = NO₂), 62507-48-6; IX (G = Cl), 72269-23-9; IX (G = H), 5411-13-2; IX (G = Me), 72269-24-0; IX (G = MeO), 72269-25-1; X (G = NO₂), 135-69-3; X (G = Cl), 5002-07-3; X (G = H), 92-91-1; X (G = MeO), 13021-18-6; XI (G = H), 500-72-1; XI (G = 3-Me), 17738-78-2; XI (G = 4-Me), 42868-89-3; XI (G = 3-OMe), 72269-26-2; XI (G = 4-OMe), 41374-62-3; XI (G = 4-F), 69066-43-9; XI (G = 3-Cl), 17709-99-8; XI (G = 4-Cl), 17738-71-5; XI (G = 4-NO₂), 103-94-6.

Substituent Effects in Hydrogen Abstraction from 10-Substituted-9-methylanthracenes. Correspondence with Ring Substitution¹

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Hydrogen abstraction from a series of eight 10-substituted-9-methylanthracenes at 70 °C has been examined by means of photoinduced reaction with bromotrichloromethane. An excellent Hammett correlation could be obtained with σ^+ parameters. The ρ value of -0.78 ± 0.05 showed a correlation coefficient of 0.99 and a standard regression from the mean of 0.06. This corresponds well to the value of -0.83 ± 0.04 previously determined for the trichloromethylation of 9-substituted anthracenes. These results extend to polycyclic systems the relationship recently proposed by Pryor of correspondence between ρ values for hydrogen abstraction from toluenes and substitution into benzenes.

In benzenoid systems, there exists a decided similarity in Hammett correlations between studies in which the reaction site occurs in conjugation with the ring system and those examples of attack upon the ring itself. This is especially true for electrophilic aromatic substitution at the para position where correlations with σ^+ are obtained. This observation is clarified and rationalized by a consideration of resonance structures for the respective transition states.³ Where analogous radical processes are concerned, the above should at least also be qualitatively true and most pertinent in situations where a relatively high degree of charge separation is present in the transition state. More explicitly, the anticipation is that the transition states for hydrogen abstraction from a series of substituted toluenes and ring substitution into a series of substituted benzenes by a common radical should be markedly similar.

Noting the above correspondence in transition-state models, Pryor and co-workers have postulated a quantitative relationship between the respective reactions.⁴ It

is held that the ρ value for hydrogen abstraction by a given radical from substituted toluenes is, within experimental tolerances, identical with that obtained for substitution by the same radical in corresponding benzenes. Supportive experimental data have been summarized by these workers.⁴

Some concern must be expressed about the data utilized to generate the relationship. The most compelling shortcoming arises from the fact that few radicals will both abstract hydrogen from a series of toluene substrates and also undergo substitution to the corresponding benzenes at the same temperature. None of the common, typically electrophilic radicals employed for benzylic hydrogen abstraction studies, e.g., chlorine, bromine, trichloromethyl, and *tert*-butoxy radicals, etc., will ring substitute in benzene under comparable reaction conditions. Pryor has been obliged to base his relationship on generally nonselective carbon radicals with rather small ρ values. At this extreme of nonselectivity, comparisons are more tenuous.

Polycyclic aromatic compounds undergo radical ring substitution much more readily than simple benzene de-

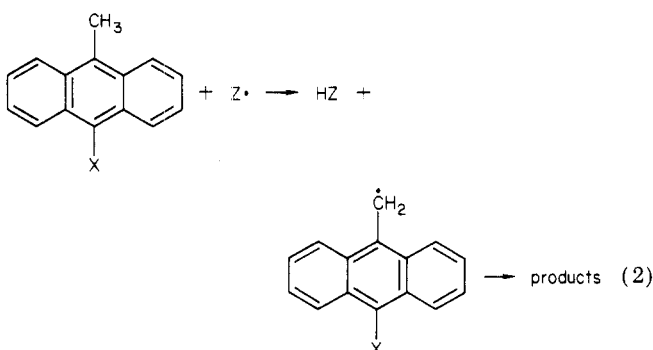
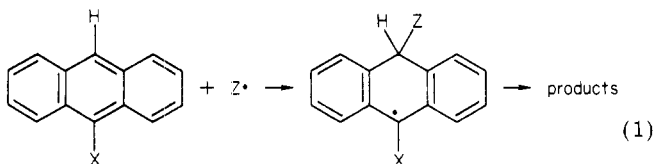
(1) Presented at the 34th Northwest Regional Meeting of the American Chemical Society, June 15, 1979.

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derivatives. This is true for both nonselective, somewhat nucleophilic, alkyl radicals⁵ and the traditionally more selective, electrophilic radicals⁶ which have been utilized in studies concerned with benzylic hydrogen abstraction.⁷ Hydrogen abstraction from the methyl homologues of these polycyclics proceed in the expected manner.⁸ Meso-substituted anthracenes, in particular, might thus prove to be excellent in providing access to conditions where ring substitution, eq 1, and benzylic-type hydrogen abstraction, eq 2, may be observed under identical conditions.



Some time ago an example of eq 1 was studied with the trichloromethyl radical as the attacking species.⁹ A ρ value of -0.83 ± 0.04 was observed.¹⁰ The view that benzylic brominations utilizing bromotrichloromethane involve hydrogen abstraction by bromine atom has been advanced.¹¹ We, however, have chosen to adopt the traditional view that in these reactions, the trichloromethyl radical is the species involved,¹² hence allowing for the desired comparison to be made.

The effect of substituents on the relative rate of hydrogen abstraction from 10-substituted-9-methylantracenes was studied by competitive kinetic techniques. Rates of hydrogen abstraction from individual anthracenes were determined relative to the corresponding abstraction from the central ring of fluorene. The course of reaction was monitored by proton nuclear magnetic resonance with the decrease in signal of the reactive protons being measured relative to that of the nonreactive aliphatic protons of *p*-di-*tert*-butylbenzene. A mixed solvent of bromobenzene-bromotrichloromethane of approximate 2:3 molar ratio ensured solubility of all compounds. All reactions were run in replicate under a nitrogen atmosphere at reduced pressure at 70 °C to allow for direct comparison with the results of the trichloromethylation of 9-substituted anthracenes. Reaction times varied from 1 to 4 h. Extents of substrate disappearance ranged from 10% to 80%. The

Table I. Relative Rates of Hydrogen Abstraction by Trichloromethyl Radical from 10-Substituted-9-methylantracenes at 70.0 °C

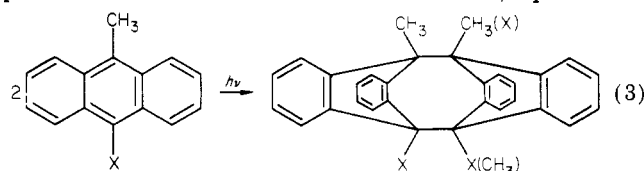
substituent	σ_p^+	σ_p	k_{rel}^a	no. of runs
OCH ₃	-0.778	-0.268	9.00 ± 0.52^b	6
CH ₃	-0.311	-0.170	4.03 ± 0.55^c	7
C ₆ H ₅	-0.179	-0.010	1.92 ± 0.11	5
H	0	0	2.75 ± 0.32^d	6
Cl	0.114	0.277	1.99 ± 0.29	5
Br	0.150	0.232	1.67 ± 0.12	6
COCH ₃	0.502	0.502	1.17 ± 0.18	6
C=N	0.659	0.660	0.57 ± 0.03	5

^a Relative to fluorene. ^b Average deviation. ^c Statistically correct. ^d Corrected for ring substitution.

photochemical initiation of all reactions was accomplished by a 275-W General Electric sunlamp.

Ring substitution at the α or β positions of the 10-substituted-9-methylantracenes could be dismissed as an important side reaction. Radical bromination of anthracene occurs almost exclusively at the 9 position.¹³ Even highly reactive phenyl radicals show little tendency to attack at these positions relative to meso attack.¹⁴ Only in the case of parent 9-methylantracene itself might ring substitution be competitive with benzylic hydrogen abstraction. However, even in this compound, benzylic hydrogen abstraction is the favored process.⁹

The use of photolytic means to initiate radical formation suggests a much more formidable problem in the possible photodimerization of anthracene substrates, eq 3.¹⁵ Al-



though rapid for anthracene itself, the introduction of substituents at the meso position greatly retards this process.¹⁵ Deliberate attempts at the photodimerization of 9,10-dimethylantracene led to only 4.5% conversion after 24 h of irradiation. This exposure is greatly in excess of those encountered in our kinetic runs. Any propensity to undergo photodimerization could be further decreased by the use of bromobenzene, an effective singlet quencher,¹⁶ as a cosolvent. No dimer could be detected in any kinetic run.

The product of the photoinitiated reaction of a 10-substituted-9-methylantracene with bromotrichloromethane should be the corresponding 9-(bromomethyl)anthracene. The signal for the aliphatic protons in this compound could be detected at ca. δ 5.5. Unfortunately the area of this peak was much less than expected. The product molecule is very labile to hydrolysis and might disappear by this route although precautions were taken to use dry glassware throughout the study. We think it is more likely that the initially formed product undergoes further hydrogen abstraction to yield di- and possibly tribrominated materials. It has been recently reported that in the radical bromination of substituted toluenes, the second successive hydrogen abstraction occurs more rapidly than the first.¹⁷ In

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(10) As reaction occurred in the meso position(s), para substituent constants were made use of. This is tantamount to regarding the system as a true dibenzologue of benzene. Optimum correlation was found with σ_p^+ parameters.

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Table II. Linear Free Energy Correlations of Hydrogen Abstraction from 10-Substituted-9-methylantracene by Trichloromethyl Radicals

σ employed	remarks	ρ	correl coeff	regression from mean
σ	all points	-1.05 ± 0.09	0.94	0.16
σ^+	all points	-0.75 ± 0.07	0.96	0.09
σ	excluding phenyl	-1.10 ± 0.08	0.97	0.12
σ^+	excluding phenyl	-0.78 ± 0.05	0.99	0.06
σ^+	all points, value for phenyl	-0.78 ± 0.05	0.98	0.06

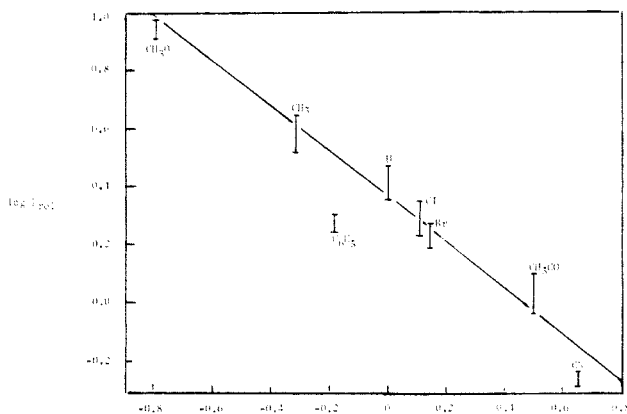


Figure 1. Logarithms of the relative rates of hydrogen abstraction from 10-substituted-9-methylantracenes vs. σ^+ constants.

our own laboratories we observed that hydrogen abstractions from 9-(halomethyl)anthracenes occur at rates comparable to those reported below. Table I presents the relative rate data determined in this study.

In terms of the correlation, all compounds were well-behaved, save 10-phenyl-9-methylantracene which is less reactive than expected. We feel this is a result of this substituent's inability to adopt a coplanar conformation with the anthracene moiety. Such coplanarity would be necessary to maximize any mesomeric stabilization of the developing radical but would be prohibited by peri interactions between the ortho hydrogens on the phenyl group and those on positions 4 and 5 of the anthracene. The two rings in 9-phenylantracene are known to form a dihedral angle of ca. 70° .¹⁸

Table II summarizes the results of several linear least-squares fittings of the data in Table I to Hammett correlations. Average deviations, correlation coefficients, and regressions from the mean uniformly agree that optimum correlation is obtained when para σ^+ parameters are utilized and the apparently misbehaving phenyl point is discarded. This is graphically shown in Figure 1. A reasonable correlation is also observed if the para σ substituent constant is assigned to phenyl while all other substituents are given σ^+ constants. Such an approach would reflect the aforementioned inhibition of mesomerism.

A comparison of the present results to those of the substitution experiment evince a fair amount of corre-

spondence. The ρ values of -0.78 ± 0.05 here and -0.83 ± 0.04 for that of Arnold et al.⁹ are equivalent within experimental error. That the optimum correlation should be with σ^+ is not surprising based upon hydrogen abstractions from several aralkyl systems.¹⁹ It should be pointed out, however, that if correlations with σ constants are used, the correspondence for hydrogen abstraction ($\rho = -1.10 \pm 0.08$) and ring substitution ($\rho = -1.12 \pm 0.09$) is still found.

The above lends apparent experimental support to Pryor's postulate. The ρ value for hydrogen abstraction from "benzylic" positions on aromatic compounds is essentially the same as that realized from the corresponding ring substitution reactions. The present findings might be seen as a more rigorous test of the above-mentioned hypothesis. The trichloromethyl radical, unlike the generally unselective radical agents originally cited by Pryor, exhibits the dual nature of being sufficiently reactive under identical conditions for both hydrogen abstraction and aromatic substitution and yet being a rather selective species.

It is also interesting that the original benzene-toluene comparison can apparently be extended to higher aromatics (i.e., anthracene and 9-methylantracene). SCF-MO calculations indicate that the energy differences associated with benzylic hydrogen abstractions and ring substitution in the mono- and tricyclic systems are similar but not identical.²⁰ It is possible, however, that solvation effects may remove this small, gas phase oriented difference in transition-state π energies.

A direct comparison of the present result with that for hydrogen abstraction from substituted toluenes by the same radical is obscured in that the latter systems were studied at an appreciably lower temperature (50°C).^{11,12} The ρ values found in those studies, -1.29 ¹¹ and -1.46 ,¹² would still probably be greater than -0.78 if corrected to 70°C . Hydrogen abstraction from the anthracenes should be expected to show a diminished sensitivity to substituent changes since the polycyclic system can much more effectively delocalize the developing radical than can a phenyl group. It has been shown that 9-methylantracene undergoes hydrogen abstraction 450 times as rapidly as toluene under the present conditions.⁸

Experimental Section

Quantitative analyses of the kinetic trials were performed on a Varian HA-100 nuclear magnetic resonance spectrometer. Reference compounds and reagents were analyzed for purity when necessary with a Varian Aerograph 202-B gas-liquid chromatograph. Melting points were ascertained with a standard Mel-Temp apparatus and are uncorrected.

Reagents. Bromotrichloromethane, bromobenzene, *p*-di-*tert*-butylbenzene, fluorene, and 9-methylantracene were commercially available. All were purified prior to use by standard techniques and were found to be in excess of 99% pure.

9,10-Dimethylantracene. A crude (90% pure) grade of anthracene from Eastman Organics was used in reaction with a mixture of *p*-formaldehyde and gaseous hydrochloric acid in glacial acetic acid.²¹ The bis(chloromethyl) product obtained after a reaction time of 24 h was (tediously) purified via successive recrystallizations from benzene. The dimethyl compound was obtained by a subsequent lithium aluminum hydride reduction of the substitution product in a dry tetrahydrofuran solution.²² Recrystallized from benzene-ethanol, the final product possessed

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a melting point of 182–183.5 °C (lit. mp 184–185 °C).²²

9-Methyl-10-methoxyanthracene. A solution of anthrone, excess potassium hydroxide, and methyl iodide were reacted together according to an original procedure of Meyer and Schlosser²³ as modified by Barnett and Cook.²⁴ Recrystallization from light petroleum ether yielded a product possessing a melting point of 138–142 °C (lit. mp 141–143 °C).²⁴

9-Methyl-10-cyanoanthracene. The method of Fieser and Jones²⁵ was used to react 9-methylanthracene-10-carboxaldehyde with hydroxylamine to give the oxime. This, in turn, was converted to the desired product by the action of acetic anhydride.²⁶ The compound obtained had a melting point of 208–210 °C (lit. mp 209–210 °C).

9-Methyl-10-acetylanthracene. A standard sequence²⁷ was followed in which 9-methylanthracene, acetyl chloride, and AlCl₃ in a benzene solution reacted to yield the desired product. The compound formed melted between 131 and 135 °C (lit. mp 133–135 °C).

9-Methyl-10-phenylanthracene. The scheme outlined for preparation of 9,10-dimethylanthracene was utilized to synthesize the present compound from 9-phenylanthracene in two steps.²² Recrystallization from benzene-ethanol yielded a product with a melting point of 105–107 °C (lit. mp 111–113 °C).

9-Methyl-10-haloanthracenes. Both the chlorine- and bromine-containing materials were synthesized via the method of Nonhebel et al.²⁹ Anhydrous cupric halides were prepared by drying the appropriate halide under reduced (aspirator) pressure and heating until all water was removed. The salt was then

immediately transferred to carbon tetrachloride solutions of methylanthracene. The materials were allowed to react at a temperature of 78 °C, that of refluxing carbon tetrachloride, for 12 h. Immediate evolution of hydrogen halide was noted by holding litmus paper over the refluxing solution. The crude products were recrystallized from light petroleum ether. The 9-methyl-10-chloroanthracene obtained melted between 177 and 179.5 °C (lit. mp 180–181 °C) and the bromo analogue between 169 and 171 °C (lit. mp 170–172 °C).

Kinetics. Solutions consisting of fluorene, the appropriate anthracene, *p*-di-*tert*-butylbenzene, bromotrichloromethane, and bromobenzene were prepared in approximate ratios of 0.5:1:0.1:30:20. The solutions were each divided among several ampules. After nitrogen flushing during several successive freeze-thaw cycles, the ampules were sealed under a reduced pressure of nitrogen. With an ampule reserved for analysis of starting material concentrations, the remaining samples were immersed in a constant temperature (70.0 ± 0.1 °C) oil bath. An ultraviolet lamp was placed at a distance of 20 cm from the surface of the oil, the ampules being just under the oil's surface. After an appropriate duration of time, ranging from 1 to 4 h depending on a given substrate's proclivity to reaction, the ampules were cooled in a dry ice-acetone bath to quench reaction. The contents were subsequently transferred into NMR tubes and small amounts of Me₄Si were added. Extents of reaction were ascertained by examining the areas of the aliphatic protons corresponding to unreacted fluorene and substituted anthracenes relative to the signal from the internal standard. These areas were converted by a computer program using standard kinetics methodology into relative rates of reaction.³⁰

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Registry No. 10-Methoxy-9-methylanthracene, 21992-33-6; 9,10-dimethylanthracene, 781-43-1; 10-phenyl-9-methylanthracene, 13425-08-6; 9-methylanthracene, 779-02-2; 10-chloro-9-methylanthracene, 19096-07-2; 10-bromo-9-methylanthracene, 23674-17-1; 10-acetyl-9-methylanthracene, 36778-18-4; 10-cyano-9-methylanthracene, 1467-01-2.

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α-Lithio-3-indolylacetate Synthons: Generation and Synthetic Utilization

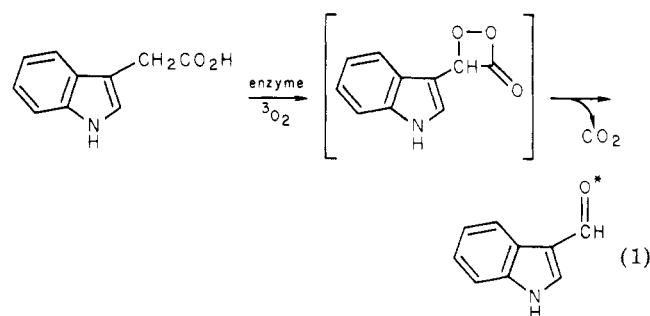
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3-Indolyl-, (*N*-methyl-3-indolyl)-, and (*N*-methyl-2-methyl-3-indolyl)acetic acids are quantitatively α-lithiated directly with *n*-BuLi, while methyl (*N*-methyl-3-indolyl)acetate is quantitatively α-lithiated with lithium diisopropylamide (LDA) at -78 °C in THF. These useful synthons react readily with electrophiles such as alkyl halides, chlorosilanes, and ketones to afford the respective α-alkyl derivatives 1, ketene silyl acetals 2, and β-hydroxy acids 3. Photosensitized oxygenation of the ketene silyl acetals 2 affords 1,2-diox-4-ene products through (2 + 4) cycloaddition with singlet oxygen.

Cilento and co-workers recently discovered² that in the peroxidase-catalyzed oxygenative decarboxylation of 3-indolylacetic acid the 3-indolecarboxaldehyde product is generated electronically excited and is presumed to be chemienergized through the corresponding α-peroxy lactone³ (eq 1). This significant discovery enticed us to venture into the synthesis of model compounds of such intriguing dioxetane derivatives. For this purpose we



(1) NIH Career Development Awardee (1975–1980).

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decided to employ the synthetic sequence shown in eq 2 which proved successful for the preparation of simple α-peroxy lactones.⁴ For the preparation of the essential