Universidad de Chile. The initial collaboration was made possible by the University of Chile–University of California Cooperative Program.

Registry No. **1a**, 16751-01-2; **1b**, 16751-02-3; **1c**, 20536-36-1; **1** (X = OH), 106-25-2; **1** (X = OMe), 2565-83-5; **2a**, 16750-99-5; **2b**, 763-10-0; **2c**, 5389-87-7; **2** (X = OH), 106-24-1; **2** (X = OMe), 2565-82-4; **3** (X = OH), 78-70-6; **3** (X = OMe), 60763-44-2; **4c**, 39864-10-3; **4** (X = OH), 98-55-5; **4** (X = OMe), 14576-08-0; **7**, 138-86-3; **8**, 586-62-9; **9**, 123-35-3; **10**, 3779-61-1; β-terpinyl chloride, 70682-36-9.

Molecular Rearrangements. 14. Photolysis and Thermolysis of Phenylpropionanilides

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Received December 19, 1978

Photolysis and thermolysis of phenylpropionanilide and (*o*-methylphenyl)- and (*p*-methylphenyl)propionanilides gave the corresponding arylamine, 1,4-diphenylbutane, together with ortho and para (α-phenethyl)arylamine. The first step in such a process is the homolysis of the acyl–N bond into arylamino and phenylpropionyl free radicals. The latter undergo decarbonylation into β-phenethyl radicals which rearrange to their α isomers. Photolysis was found to be an intramolecular process, while thermolysis was found to be mainly intermolecular as shown by cross-over experiments. On the other hand, alkyl derivatives of the solvent were isolated when the anilides were pyrolyzed in the presence of β-naphthol or isoquinoline as aromatic solvents.

Acetanilide, propionanilide, butyranilide, and benzanilide were reported[1-3] to rearrange on irradiation forming the corresponding *o*- and *p*-aminophenyl alkyl ketones through an intramolecular mechanism where the acyl–N bond undergoes cleavage. Partial transfer of the acyl group from acetanilide to a foreign molecule was also observed.[2]

Recently,[4] photolysis of benzanilide in ethanol as a solvent was found to produce a mixture of *o*- and *p*-aminobenzophenones together with ethyl benzoate, benzamide, azobenzene, and phenanthridone suggesting a free-radical intermolecular mechanism. Far less is known about the behavior of anilides on thermolysis.

The present work describes a study on the rearrangement pathway of β-phenylpropionanilide, (*o*-methyl-β-phenyl)propionanilide, and (*p*-methyl-β-phenyl)propionanilide on photolysis and thermolysis. The acetone-initiated ultraviolet irradiation of β-phenylpropionanilide for 30 h at room temperature (25 °C) gave rise to ethylbenzene, 1,4-diphenylbutane, and 2,3-diphenylbutane as neutral products together with aniline and a mixture of *o*- and *p*-(α-phenethyl)aniline.

Similar results were also obtained on photolysis of (*o*-methyl-β-phenyl)propionanilide under the same conditions whereby the above-mentioned neutral products were obtained in addition to *o*-toluidine and 4-(α-phenethyl)-*o*-toluidine as amine products. Also, the photolysis of (*p*-methyl-β-phenyl)propionanilide gave rise to *p*-toluidine and 2-(α-phenethyl)-*p*-toluidine in addition to the above-mentioned neutral products as shown in Table I.

Thermolysis of β-phenylpropionanilide either by reflux or by heating in a sealed tube at about 350 °C for 7 days gave rise to aniline, ethylbenzene, 2,3-diphenylbutane, and a mixture of *o*- and *p*-(α-phenethyl)aniline in addition to evolution of carbon monoxide.

In isoquinoline as a solvent, the same products were obtained together with 1-(α-phenethyl)isoquinoline.

In β-naphthol as a solvent, the previous products were formed in addition to 2,2′-dinaphthol and a nitrogenous byproduct, mp 112 °C, which was not further identified.

Table I. Percentage Composition of β-Phenylpropionanilide Photolysates

	β-phenyl-propion-anilide	(*o*-methyl-β-phenyl)-propion-anilide	(*p*-methyl-β-phenyl)-propion-anilide
ethyl-benzene	14.6	14.70	2.1
1,4-diphenyl-butane	11.9	22.40	3.3
2,3-diphenyl-butane	3.8	2.45	0.6
arylamine	9.5[a]	6.25[b]	20.5[c]
alkylaryl-amine	40.4[d]	32.25[e]	4.2[f]
unchanged anilide	14.9	20.75	69.0

[a] Aniline. [b] *o*-Toluidine. [c] *p*-Toluidine. [d] *o*-(α-Phenethyl)aniline and its para isomer in the ratio 1:5. [e] 4-(α-Phenethyl)-*o*-toluidine. [f] 2-(α-Phenethyl)-*p*-toluidine.

Similar results were also obtained from thermolysis of (*o*-methyl-β-phenyl)propionanilide and its para isomer where *o*- or *p*-toluidine were obtained in addition to substitution products of the α-phenethyl group on the corresponding toluidine moiety as shown in Table II.

The photorearrangement of such anilides was found to be an intra- rather than intermolecular process since irradiation of a mixture of α-phenylacetanilide and (*o*-methyl-β-phenyl)propionanilide in acetone solution gave products indicating that each anilide rearranges independently. Thus the amine products identified were only aniline, *N*-benzylaniline, (*o*-aminophenyl)phenylmethane, (*p*-aminophenyl)phenylmethane, *o*-toluidine, and 4-(α-phenethyl)-*o*-toluidine. No substitution of the alkyl group into the ring of the other compound took place. Had the rearrangement proceeded through an intermolecular pathway, cross-bred products such as *o*- and *p*-(α-phenethyl)aniline or (4-amino-3-methylphenyl)phenylmethane should be obtained, yet they are not detected among the products by GLC or TLC analyses of the amine products as compared with reference samples. Hence, it can be

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0022-3263/79/1944-3244$01.00/0

Table II. Products of β-Phenylpropionanilide Thermolysis (g)

	β-phenylpropionanilide				(o-methyl-β-phenyl)-propion-anilide	(p-methyl-β-phenyl)-propion-anilide
expt no.[k]	1	2	3	4	5	6
anilide, consumed/used	15/20	18/20	20/20	20/20	15/20	14/20
solvent, consumed/used			6/10	7/10		
products						
ethylbenzene[a]	2.5	2.6	2.5	2.25	2.6	2.25
arylamine	4.0	4.6	4.0	2.5[b]	3.25[c]	2.1[d]
C-alkylarylamine	3.0	3.6	3.5	3.2[e]	3.35[f]	2.8[g]
alkyl deriv of solvent			6.8[h]			
2,3-diphenylbutane[i]	1.5	2.3	1.2	2.4	2.15	2.2
other products				4.5[j]		
residue	3.0	3.25	4.0	3.5	3.0	3.2

[a] n^{20}_D 1.4953, bp 136 °C (760 mmHg), on oxidation with potassium permanganate gave benzoic acid, mp 122 °C. [b] Aniline, bp 184 °C (760 mmHg), n^{20}_D 1.5857; acetyl derivative mp and mmp 113–114 °C. [c] o-Toluidine, bp 200 °C (760 mmHg), n^{20}_D 1.5679; benzoyl derivative mp and mmp 144–145 °C. [d] p-Toluidine, bp 200 °C (760 mmHg), mp 46–47 °C; benzoyl derivative mp and mmp 158–159 °C. [e] Bp 180–190 °C (13 mmHg) found by TLC to contain o-(α-phenethyl)-aniline, R_f 0.47, and its para isomer, R_f 0.37. [f] 4-(α-Phenethyl)-o-toluidine, mp 55–6 °C; picrate mp and mmp 174–6 °C. [g] 2-(α-Phenethyl)-p-toluidine, bp 150–170 °C (13 mmHg), R_f 0.46; picrate mp and mmp 166–7 °C. [h] 1-(α-Phenethyl)isoquinoline, bp 230–5 °C (13 mmHg), R_f 0.75, n^{20}_D 1.6428; its IR spectrum is coincident with that of authentic sample. [i] Mp and mmp 123–4 °C. [j] 2,2-Dinaphthol mp and mmp 212 °C; dibenzoate mp and mmp 160 °C. [k] Experiment no.: (1) by reflux; (2, 5, and 6) in sealed tubes under nitrogen atmosphere; (3) with isoquinoline as solvent; (4) with β-naphthol as solvent.

concluded that the photolysis occurs through homolysis into a free-radical pair held together within the solvent cage.

Although it was reported[2-4] that the photorearrangement of acetanilide and benzanilide in ethanol solution to the corresponding amino ketones was intermolecular, our results show that the photolyses of the present anilides in acetone are intramolecular. No escape of the acyl radicals out of the solvent cage was observed. Such difference in behavior cannot be attributed to solvent nature since acetone is known to be of lower viscosity and dielectric constant than ethanol. However, it can be ascribed to the large volumes of the phenylpropionyl and phenylacetyl radicals obtained from photodissociation of the anilides under investigation as compared with acetyl radicals.

The main feature of phenylpropionanilide behavior on photolysis or thermolysis is the homolytic fission of the acyl–N bond into anilino (or toluidino) and β-phenylpropionyl free radicals. The latter undergoes preferential decarbonylation[5] forming carbon monoxide and β-phenethyl free radical which may subsequently abstract hydrogen forming ethylbenzene or undergo dimerization to 1,4-diphenylbutane.

However, ring substitution products of β-phenethyl radicals were not detected among the products by GLC analysis as compared with authentic samples. It may be expected that β-phenethyl radicals rearrange[6] to their α isomers on the basis of stability of the secondary radicals as compared with the primary ones.

$$Ph\text{–}CH_2\text{–}\dot{C}H_2 \rightarrow Ph\text{–}\dot{C}H\text{–}CH_3$$

Yet, such rearrangement that involves 1,2-hydrogen shift is unknown.[7] Hence it can be assumed that the formation of α-phenethyl radicals is due to selective hydrogen abstraction of the secondary α-hydrogen rather than the primary β-hydrogen of ethylbenzene, since removal of such hydrogen is favored by resonance stabilization of the secondary free radical formed. The anilino (or toluidino) and α-phenethyl free radicals subsequently contribute to the formation of the identified products. Thus, hydrogen abstraction by the arylamino radicals formed leads to formation of the corresponding arylamines, namely, aniline and o- or p-toluidines, while the α-phenethyl radicals dimerize into 2,3-diphenylbutane or couples with the arylamino radicals forming the corresponding (α-phenethyl)arylamines as shown in Scheme I.

Formation of 1-(α-phenethyl)isoquinoline and the absence of biisoquinolinyl among the products when the rearrangement was carried out in the presence of isoquinoline may give favor to the assumption that direct substitution by the radical on the aromatic nucleus could be one of the pathways involved in the formation of substitution products. On the other hand, the high reactivity of the formed isoquinolinyl free radicals should selectively act as effective scavengers for α-phenethyl free radicals rather than undergoing dimerization.

Formation of 2,2′-dinaphthol when the thermolysis was affected in the presence of β-naphthol may be attributed to dimerization of β-naphthyloxy radicals formed by hydrogen abstraction from β-naphthol as compared with that of the isoquinolyl radicals.

The formation of alkyl derivatives of solvents used on pyrolysis may suggest an intermolecular nature for the process. Furthermore, convincing evidence for the intermolecularity of the pyrolysis is that when a mixture of α-phenylacetanilide and (o-methyl-β-phenyl)propionanilide in β-naphthol as a high-boiling solvent was subjected to pyrolysis, (4-amino-3-methylphenyl)phenylmethane and a mixture of o- and p-(α-phenethyl)anilines were identified among the amine products through their melting points and mixture melting point determinations together with GLC, TLC, and mass spectral analyses.

Experimental Section

All melting points are uncorrected. The IR spectroscopic analyses were carried out on a Pye-Unicam I.R., Spectrophotometer, Model SP 200G. The UV absorptions were carried out on a Pye-Unicam UV spectrophotometer, Model SP 8000, using 95% ethanol as solvent. GLC was carried out on a Pye-Unicam gas chromatograph, "Series 104", Dual flame ionization detector, Model 24. The products were characterized by comparison of GLC retention times with those of authentic samples on two columns with different separation characteristics. The columns used are 4 ft × 5 mm packed with 20% SE 30 on Chromosorb

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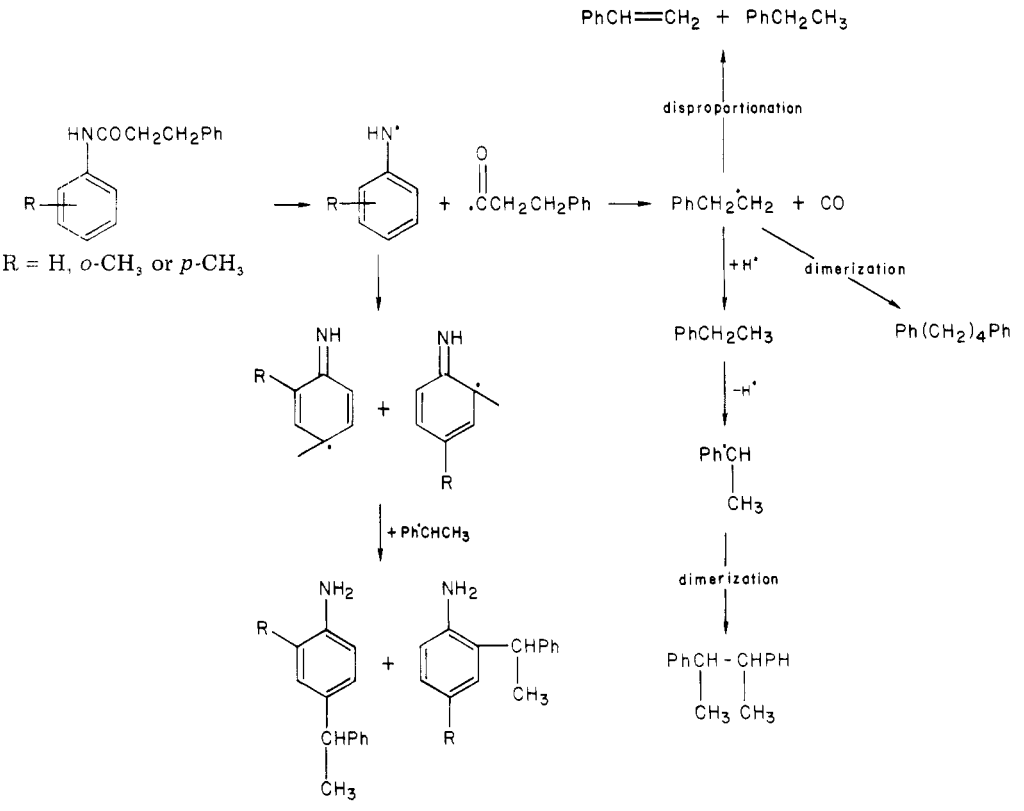
Scheme I



W (35–80 mesh) or 10% SE 30 on Celite (60–80 mesh). Quantitative GLC was accomplished by determination of the peak areas of the products. TLC was carried out on plates covered with silica gel (25–40 mesh) and eluted with benzene/cyclohexane (3:1 v/v) or ether/petroleum ether (40–60 °C) (1:7 v/v). Molecular weight determination of some reaction products was carried out by mass spectrophotometer Model A.E.I.M.S. 902. Ultraviolet irradiation was carried out using a Mallinkrodt 150 W mercury discharge lamp, and the solutions were contained in open-topped Pyrex beakers. Solvents used were of Analar grade and were used without further purification.

Photolysis of Anilides. The anilide (1 g) in acetone (100 mL) was irradiated at room temperature (25 °C) for 30 h. The photolysate was separated as indicated in a previous work[9] into amine and neutral products and analyzed by GLC and TLC as compared with authentic samples. The results are summarized in Table I.

Cross-over Photolysis of Anilides. A solution of α-phenylacetanilide (0.5 g) and (*o*-methyl-β-phenyl)propionanilide (0.5 g) in acetone (100 mL) was irradiated for 30 h, and the amine fraction was analyzed by TLC as compared with that of authentic samples.

Thermolysis of Anilides. The anilide (20 g) was heated under reflux in air or in sealed tubes in a nitrogen atmosphere at about 350 °C for 4 days either alone or in the presence of aromatic solvents such as β-naphthol or isoquinoline. The carbon monoxide evolved was detected by platinum chloride test paper and absorbed in a trap containing ammoniacal cuprous chloride. The products were separated as usual[9] into amine and neutral products. Each type of products was subjected to fractional distillation under reduced pressure. Further separation of each fraction was accomplished by means of column chromatography into its pure constituents and identified by elemental analysis, TLC, GLC, UV, and IR spectroscopy, or mixture melting points with authentic samples whenever possible as summarized in Table II.

Cross-Over Thermolysis of Anilides. α-Phenylacetanilide (5 g), (*o*-methyl-β-phenyl)propionanilide (5 g) and β-naphthol (20 g) were heated together in a sealed tube under nitrogen atmosphere for 4 days. The amine portion of the reaction product (4.3 g) was subjected to fractional distillation under reduced pressure, and the following products were obtained; a mixture of aniline and *o*-toluidine (0.62 g), bp 50–62 °C (2 mm), identified by TLC (silica gel 25–40 mesh, ether/petroleum ether 40–60 °C, 1:7 v/v) R_f aniline 0.85, R_f *o*-toluidine 0.78; *o*-(α-phenethyl)aniline and its para isomer (0.43 g), bp 140–5 °C (2 mm), separated from each other by trituration with petroleum ether (40–60 °C); [ortho isomer mp and mmp 61–2 °C, whereas the para isomer was isolated from the mother liquor through picrate formation, mp and mmp 111–3 °C]; (4-amino-3-methylphenyl)phenylmethane (0.35 g), bp 160–6 °C (2 mm), identified by mp and mmp 62–4 °C [picrate mp and mmp 181–2 °C, and its mass spectrum shows a molecular ion at *m/e* 197].

Preparation of Reference Compounds. β-Phenylpropionanilide[10] crystallization from ethanol, mp 98 °C. (*o*-Methyl-β-phenyl)propionanilide[10] crystallization from ethanol, mp 127–8 °C. (*p*-Methyl-β-phenyl)propionanilide[10] crystallization from ethanol, mp 129–130 °C. *o*-(α-Phenethyl)aniline[11] crystallization from petroleum ether (40–60 °C), mp 62 °C. *p*-(α-Phenethyl)aniline[11] colorless oil, bp 170–180 °C (20 mmHg); picrate mp 112–3 °C. (4-Amino-3-methylphenyl)phenylmethane:[12] crystallization from petroleum ether (40–60 °C), mp 63 °C; picrate mp 181–2 °C. 2,2′-Dinaphthol:[13] crystallization from toluene, mp 216–8 °C; dibenzoate mp 160 °C. 1-(α-Phenethyl)isoquinoline:[14] bp 160–170 °C (10 mmHg); n^{20}_D 1.6428. 1-(α-Phenethyl)isoquinoline: prepared from α-phenethyl bromide and isoquinoline by a procedure similar to that of 1-benzylisoquinoline:[14] bp 160–178 °C (10 mmHg); n^{20}_D 1.6428; molecular ion at *m/e* 233. Anal. Calcd for $C_{17}H_{15}N$: C, 87.55; H, 6.43; N, 6.00. Found: C, 87.30; H, 6.54; N, 6.12. Moreover, its UV absorption spectrum shows a hyperchromic shift as compared with that of isoquinoline or 1-benzylisoquinoline.

Isoquinoline: λ_{max} (Å) 2620 (ϵ_{max} 3700), 3710 (ϵ_{max} 3500). 1-Benzylisoquinoline: λ_{max} (Å) 2620 (ϵ_{max} 3800), 3170 (ϵ_{max} 3680). 1-(α-Phenethyl)isoquinoline λ_{max} (Å) 2620 (ϵ_{max} 4020), 3170 (ϵ_{max} 3900).

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2,3-Diphenylbutane:[15] crystallization from dilute ethanol, mp 124 °C. 1,4-Diphenylbutane:[16] bp 140–2 °C (15 mmHg). 2-(α-Phenethyl)-p-toluidine:[11] yellow oil, bp 178–181 °C (15 mmHg); picrate from benzene, mp 165–7 °C; acetyl derivative, mp 141–2 °C. 4-(α-Phenethyl)-o-toluidine: prepared as described for 2-(α-phenethyl)-p-toluidine;[11] crystallization from petroleum ether (40–60 °C) as colorless needles, mp 55–6 °C;picrate from ethanol, mp 174–6 °C. Anal. Calcd for $C_{15}H_{17}N$: C, 85.30; H, 8.06; N, 6.64. Found: C, 85.59; H, 7.90; N, 6.86. Its IR absorption spectrum shows two sharp bands due to NH_2 stretching vibrations at 3522 and 3435 cm^{-1} and NH bending vibration at 1620 cm^{-1}. Its mass spectrum shows a molecular ion at m/e 211.

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Registry No. β-Phenylpropionanilide, 19341-03-8; (o-methyl-β-phenyl)propionanilide, 70660-35-4; (p-methyl-β-phenyl)propionanilide, 70660-36-5; ethylbenzene, 100-41-4; 1,4-diphenylbutane, 1083-56-3; 2,3-diphenylbutane, 5789-35-5; aniline, 62-53-3; o-toluidine, 95-53-4; p-toluidine, 106-49-0; o-(α-phenethyl)aniline, 28534-06-7; p-(α-phenethyl)aniline, 7476-71-3; 4-(α-phenethyl)-o-toluidine, 70660-37-6; 2-(α-phenethyl)-p-toluidine, 70660-38-7; α-phenylacetanilide, 621-06-7; β-naphthol, 135-19-3; p-(α-phenethyl)aniline picrate, 70692-41-0; (4-amino-3-methylphenyl)phenylmethane, 62441-37-6; (4-amino-3-methylphenyl)phenylmethane picrate, 70660-39-8; 2,2′-dinaphthol, 602-09-5; 2,2′-dinaphthol dibenzoate, 53545-44-1; 1-(α-phenethyl)isoquinoline, 70660-40-1; α-phenethyl bromide, 585-71-7; isoquinoline, 119-65-3; 1-benzylisoquinoline, 6907-59-1; 2-(α-phenethyl)-p-toluidine picrate, 70660-41-2; acetyl 2-(α-phenethyl)-p-toluidine, 70660-42-3; 4-(α-phenethyl)-o-toluidine picrate, 70660-43-4; benzoic acid, 65-85-0; acetylaniline, 103-84-4; benzoyl o-toluidine, 584-70-3; benzoyl p-toluidine, 582-78-5.

Molecular Associations of Acetylcholine with Aromatic Molecules in Water. Nuclear Magnetic Resonance Spectral Evidence

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Received January 3, 1979

When the sodium salt of an aromatic carboxylate ion is added to a dilute D_2O solution of acetylcholine chloride, the signals of the *N*-methyl and *N*-methylene protons of acetylcholine are shifted upfield relative to the other signals, indicating preferential interaction between the quaternary ammonium head group of acetylcholine and the aromatic ring of the anion. These spectral changes can be related to apparent association equilibrium constants for 1:1 complexation and association constants for acetylcholine complexes with several aromatic anions and phenols are reported. The equilibrium constants are largest for aromatic anions with hydrophobic or electron-donating groups on the ring, especially methoxy groups. Multiring aromatic anions like indole-3-acetate cause very large spectral changes whereas aromatic anions with low electron density in the ring, e.g. *p*-nitrobenzoate ion, do not cause an upfield shift. These observations suggest that electrostatic, water structure, and dispersion forces are all involved in stabilizing the complex, with dispersion (i.e., van der Waals') forces between the aromatic π system and the protons of the quaternary ammonium group predominating in many cases. The effect of added electrolytes or urea is consistent with this view. Complexation with arenesulfonate ions reduces the rate of hydrolysis of acetylcholine in alkaline solution.

There have been many suggestions that aromatic compounds interact strongly with both cationic micelles and unmicellized tetraalkylammonium ions.[2-6] Larsen[7] has demonstrated that the enthalpies of transfer of aromatic compounds from water to hexadecyltrimethylammonium bromide (CTABr) micelles are larger than those for similar size aliphatic compounds and Bunton[8,9] has shown that arenesulfonate and similar anions interact strongly with the cationic head groups in CTABr micelles. Bunton[9] also demonstrated that aromatic anions associate with nonmicellized *n*-butyltrimethylammonium ion in aqueous solution.

The paucity of detailed studies of weak intermolecular associations involving small molecules where both the geometry and strength of association have been determined is surprising, especially since the existence of such complexes is taken for granted and the forces involved are invoked to explain a wide variety of phenomena. It is to fulfill the need for an example of how the geometry and strength of association of such a complex depend on systematic changes in the structure of the aromatic component that we examined the weak association complexes between a wide variety of aromatic compounds and acetylcholine cation by NMR spectroscopy. We chose to study acetylcholine because hydrophobic forces are known to play a role[10] in its association with acetylcholinesterase and because it does not self-associate[11,12] at

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