A correlation between interfacial tension and composition of ternary systems has been developed by the authors (*3*). The correlation also fits aqueous and nonaqueous interfacial tension data for binary systems with good accuracy.

ACKNOWLEDGMENT

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A Study of the Nuclear Magnetic Resonance Spectra of Aryl Nitrogen Derivatives of Some Estratriens

T. C. CHOU and HSI HU LIN
Department of Chemistry, Saint Joseph's College, Philadelphia, Pa.

The nuclear magnetic resonance chemical shifts and coupling constants are reported for seven aryl nitrogen derivatives of estratriens. This study confirmed the position of the group on the aromatic ring.

IN THE PAST, workers (*2, 4, 5, 6*) have studied the positions of substituents on the aromatic ring of estratriens. This laboratory would like to confirm these assignments, by means of their nuclear magnetic resonance spectra. The results are given in Table I.

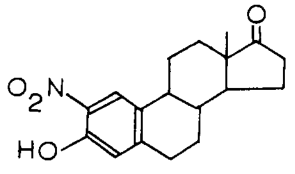
This type of problem is particularly susceptible to solution by this method since the coupling patterns of the various types of aromatic substitution are thoroughly established (*1*). This evidence is, therefore, direct and unequivocal.

The mononitroestrone, which was assigned as

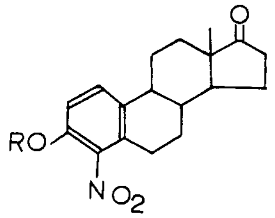
Table I Chemical Shifts, τ^a, and Coupling Constants, J^b, of Characteristic Protons in the NMR Spectra and Melting Points

Compound	Solvent	13 $-CH_3$	3 $-OCH_3$	1 & 2 or 1 & 4 Aromatic	2 or 4 $-NH_2$	M.P., ° C.
4-Nitroestrone	$CDCl_3$ & DMSO	9.09 $J = 0$		2.68 & 3.08 $J = 9$ $J = 9$		272[c]
4-Nitroestrone, 3-methyl ether	$CDCl_3$	9.09 $J = 0$	6.09 $J = 0$	2.51 & 3.02 $J = 9$ $J = 8$		253–256
4-Aminoestrone, 3-methyl ether	$CDCl_3$	9.09 $J = 0$	6.08 $J = 0$	3.18 $J = 0$	6.28[d]	188–191
4-Aminoestrone, 3-methyl ether	$CDCl_3$ + DCl	9.08 $J = 0$	6.08 $J = 0$	2.52 & 3.02 $J = 8$ $J = 8$		
2-Nitroestrone	$CDCl_3$	9.08 $J = 0$		1.99 & 3.12 $J = 0.4$ $J = 0.4$		172–175
2-Aminoestrone, 3-methyl ether	$CDCl_3$	9.10 $J = 0$	6.15 $J = 0$	3.23 & 3.40 $J = 0.5$ $J = 0.5$	6.45[d]	171–175
2-Aminoestrone, 3-methyl ether	$CDCl_3$ & DCl	9.12 $J = 0$	6.17 $J = 0$	2.42 & 3.44 $J = 0.5$ $J = 0.5$		
4-Nitroestrone, 3-benzyl ether	$CDCl_3$	9.12 $J = 0$		2.71 & 3.14 $J = 9$ $J = 9$		199–201
4-Aminoestrone, 3-benzyl ether	$CDCl_3$	9.11 $J = 0$		3.27 $J = 0$	6.28[d]	223–226
4-Aminoestrone, 3-benzyl ether	$CDCl_3$ + DCl	9.12 $J = 0$		2.67 & 3.13 $J = 9$ $J = 9$		

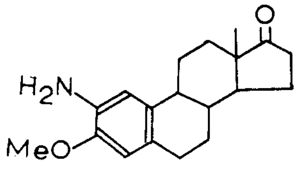
[a] Proton NMR spectra were measured at 60 Mc. on the Varian A-60 spectrometer using 5 to 10% solution in $CDCl_3$, except where noted, containing tetramethylsilane (TMS) as an internal reference standard. Chemical shifts are expressed in τ units measured downfield from the reference (τ0). [b] In c.p.s. [c] Decomposed. [d] Broad peaks and coupling constants could not be determined.



I



Compound	R
II	H
III	CH_3
IV	Benzyl



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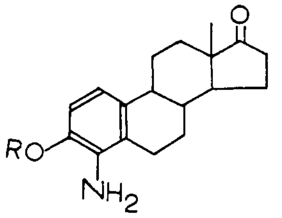

Compound	R
VI	CH_3
VII	Benzyl

2-nitroestrone (I) (*4, 6*) (m.p. 172–175° C.), has two protons in the aromatic region of the NMR spectrum centered at 2.16 and 3.06τ. These protons showed little coupling (J = 0.5 c.p.s.). Since the two protons on the aromatic ring of 2-nitroestrone are para to each other, coupling should be small. Therefore, this compound is confirmed as 2-nitroestrone (I). The 1 proton of 2-nitroestrone is deshielded by the nitro group and is shifted downfield to 2.16τ.

The mononitroestrone, which was assigned as 4-nitroestrone (II) (*4, 6*) (m.p. 272–276° C.), was found to have two protons in the aromatic region of the NMR spectrum centered at 2.60 and 3.03τ. Each of these protons show strong coupling (J = 9.0 c.p.s.). Since the two protons on the aromatic ring of 4-nitroestrone are ortho to each other, they should show strong coupling. Therefore, this compound is confirmed as 4-nitroestrone (II).

The chemical shifts of the 1 and 2 protons of 4-aminoestrone, 3-methyl ether (VI) in the NMR spectrum, appeared to be identical. As two protons with identical chemical shifts do not show any visible evidence of coupling they appear as a sharp single peak. This peak is centered at 3.18τ. This was rather unexpected, but in fact a precedent for this does exist (*7*), insofar as the four aromatic protons of *o*-amino anisole appear as a single peak.

When the hydrochloride of 4-aminoestrone, 3-methyl ether was prepared by adding DCl and D_2O to the $CDCl_3$ solution, the NMR spectrum showed the 1 and 2 protons as two doublets centered at 2.52 and 3.02τ (J = 9.0 c.p.s.). Again, the positions of these two doublets are very similar, if not identical, to those of the doublets produced by the 1 and 2 protons of 4-nitroestrone, 3-methyl ether (III). This would indicate that the electronic influence exerted by the 4-amino hydrochloride upon the aromatic ring is much the same as that of the 4-nitro group.

4-Nitroestrone, 3-benzyl ether (IV) and 4-aminoestrone, 3-benzyl ether (VII) were prepared (*3*) so that the NMR spectra of these compounds could be compared with those of the corresponding methoxyl compounds.

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Solubilities of the Cyclohexylamine Salts of the Odd and Even Normal C_{10} to C_{20} Fatty Acids in Benzene, Methanol, and Acetone

ROBERT R. MOD, JAMES A. HARRIS, and EVALD L. SKAU
Southern Regional Research Laboratory, Agricultural Research Service, U. S. Department of Agriculture, New Orleans, La.

Experimental solubility data are reported for the cyclohexylamine salts of decanoic, dodecanoic, tridecanoic, tetradecanoic, hexadecanoic, heptadecanoic, and octadecanoic acids in benzene, methanol, and acetone. These data have been used to calculate the solubilities of the cyclohexylamine salts of undecanoic, pentadecanoic, nonodecanoic, and eicosanoic acids by means of the isopleth and isotherm methods of graphical interpolation and extrapolation.

SOLUBILITY tables have been constructed for the cyclohexylamine salts of the normal fatty acids containing 10 to 20 carbon atoms in benzene, methanol, and acetone. The data are based on experimental solubility measurements of the salts of the C_{10} to C_{18} even carbon acids and of the C_{13} and C_{17} members of the odd carbon acid series. These data were used to calculate the supplementary solubility data for the series by means of the isopleth (*3*) and the isotherm (*4*) methods of graphical interpolation and extrapolation.