Quantitative Aspects of Lewis Acidity. Part XVI.[1] Acidity of Covalent Metal Halides towards Substituted Anilines in Dioxan. The Validity of ΔH^0 as a Measure of Lewis Acid Strength

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$AlCl_3$, $GaCl_3$, $SnCl_4$, $PhSnCl_3$, and $ZnCl_2$ form 1 : 1 adducts with substituted anilines in dioxan. The equilibrium constant for adduct formation, K, has been determined for each acid with several anilines at, normally, several temperatures; ΔH^0 and ΔS^0 values have been derived. Whereas, for any given base, K_{25} values follow the sequence $SnSI_4 > AlCl_3 > GaCl_3 > PhSnCl_3 > ZnCl_2$ ($\gg MeSnCl_3$, Ph_2SnCl_2, $HgCl_2$), the values of $-\Delta H^0$ follow the different sequence $PhSnCl_3 > SnCl_4 > GaCl_3 \geqslant ZnCl_2$. The corresponding ΔS^0 values vary irregularly from one acid to another. For most of the acids adduct formation probably involves the replacement, by an aniline molecule, of a solvent molecule co-ordinated to the metal atom, but for $PhSnCl_3$ the reaction is probably an addition of a base molecule to the solvent–acid adduct. A comparison is made of the level of acidity exhibited by the various acids in dioxan with that exhibited in diethyl ether, and for each acid equations are given relating the pK in dioxan of unhindered anilines to the pK_a values of the anilinium ions in water. The changes in pK found on moving from one substituted aniline to another, although always regularly related to changes in pK_a, are not determined primarily by changes in either ΔH^0 or ΔS^0; both quantities are important. This fact is discussed.

PAST comparisons of Lewis acidity have often been based on the relative values of ΔH for the reactions between some reference base and a series of acids.[2] Since ΔH is closely related to bond strengths, changes in ΔH *may* reflect alterations in the strength of the bond between the acid and the base as the acid is changed, although interpretation is often complicated by the incorporation in ΔH of lattice and/or solvation energies.[3] However, as we have previously argued,[3] the best measure of thermodynamic acidity is $\Delta G^0 (= -RT\ln K)$. Only if ΔG^0 and ΔH^0 values parallel one another can ΔH^0 values be used to some extent in lieu of ΔG^0. Such parallelism requires that ΔS^0 is a constant for a given type of reaction and independent of the acid. To assume this, as has often been done in the past, seems unjustified; the few relevant data referring to covalent metal halide acids suggest that changes in ΔS^0 can be as significant as changes in ΔH^0.[3-5]

We now report results that permit a more searching test of such an assumption. We have measured the values of K for 1 : 1 adduct formation between substituted anilines and a variety of metal halides in dioxan solution over a range of temperatures, and so obtained also ΔH^0 and ΔS^0. The results comprise the largest single set of equilibrium measurements with covalent metal halides and represent a sizeable fraction of the total information available in this field.[3]

EXPERIMENTAL

Materials.—Anhydrous dioxan was prepared by methods previously used in this laboratory.[6] Karl Fischer titration showed the water level in the purified solvent to be *ca.* 3×10^{-3}mol dm^{-3}. The majority of the bases were commercial samples, which were either recrystallised to constant m.p., or redistilled immediately prior to use. All m.p.s/b.p.s agreed with literature values. 3-Methyl-4-nitroaniline was a previous sample.[7] Aluminium chloride,[5] gallium chloride,[8] stannic chloride,[9] and zinc chloride[10] were purified by established methods. Trichloro(phenyl)tin was prepared

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[1] Part XV, D. Berry, K. Bukka, and R. S. Satchell, *J.C.S. Perkin II*, 1976, 89.

[2] *E.g.* F. G. A. Stone, *Chem. Rev.*, 1958, **58**, 101; R. J. Gillespie and G. A. Olah in 'Friedel–Crafts and Related Reactions,' ed. G. A. Olah, vol. I, Interscience, New York, 1963; the class A–B and hard–soft distinctions also lean heavily on ΔH measurements, see S. Ahrland, J. Chatt, and N. R. Davies, *Quart. Rev.*, 1958, **12**, 265.

[3] D. P. N. Satchell and R. S. Satchell, *Chem. Rev.*, 1969, **69**, 251.

[4] Y. Farhangi and D. P. Graddon, *Austral. J. Chem.*, 1973, **26**, 983; 1974, **27**, 2103.

[5] R. S. Satchell, K. Bukka, and C. J. Payne, *J.C.S. Perkin II*, 1975, 541.

[6] K. Bukka and R. S. Satchell, *J.C.S. Perkin II*, 1975, 1110.

[7] D. P. N. Satchell and R. S. Satchell, *Trans. Faraday Soc.*, 1965, **61**, 1118.

[8] A. Mohammad and D. P. N. Satchell, *J. Chem. Soc.* (*B*), 1967, 403.

[9] R. M. Evans and R. S. Satchell, *J. Chem. Soc.* (*B*), 1970, 300.

[10] R. S. Satchell, *J. Chem. Soc.*, 1964, 5464.

as previously.[5] All manipulations with the purified metal halides and their solutions were performed in a dry-box.

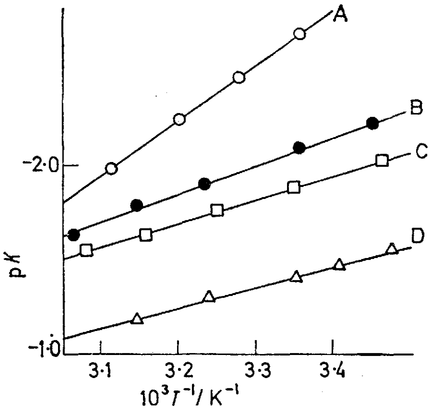
Reaction Mixtures.—Stock solutions of the metal halides were prepared as before.[6,8,10-12] These solutions were analysed for halide by conductimetric titration with silver nitrate.

Equilibrium Measurements.—The stoicheiometry of the adduct, and the equilibrium constant * $K = [\text{Adduct}]/[\text{B}][\text{MX}_n\text{S}_y]^x$ for reaction (1), or the spectroscopically indistinguishable reaction (2), were determined as before.[6]

$$\text{ArNH}_2 + x\text{MX}_n\text{S}_y \rightleftharpoons \text{ArNH}_2(\text{MX}_n\text{S}_{y-1})_x + x\text{S} \quad (1)$$

$$\text{ArNH}_2 + x\text{MX}_n\text{S}_y \rightleftharpoons \text{ArNH}_2(\text{MX}_n\text{S}_y)_x \quad (2)$$

The cell compartment of the spectrophotometer was thermostatted at ±0.1 °C. Each of the five acids was studied with 6—12 aniline bases. For all systems the u.v.–visible spectra of solutions containing a fixed overall concentration of base and a varying amount of acid showed excellent isosbestic points. Hence in each system only one adduct is formed in significant amounts.



Typical plots of p*K* *versus* $1/T$: A, PhSnCl_3–aniline; B, SnCl_4–4-chloro-3-nitroaniline; C, ZnCl_2-3-chloro-4-methylaniline: D, GaCl_3–4-chloro-3-nitroaniline

Estimation of ΔH^0 *and* ΔS^0.—Except for aluminium chloride, the equilibrium constants were determined at between four and six temperatures (usually in the range 10—50 °C). The exact temperature range was determined by the *K* values for the system, and by the solubility of the acid concerned. Values of ΔH^0 and ΔS^0 were calculated using equation (3). Plots of p*K* *versus* $1/T$ were always

$$\text{p}K = \Delta H^0/2.30RT - \Delta S^0/2.30R \quad (3)$$

rectilinear (Figure). Values of ΔH^0 and ΔS^0 were obtained by the method of least-squares fit, using a CDC 6600 computer.

Adduct formation with aluminium chloride was not examined over a range of temperatures because it proved more difficult with this acid to obtain sufficiently accurate values of *K* to make a temperature study worthwhile.

RESULTS AND DISCUSSION

Adduct Stoicheiometry and Nature of the Equilibria.—If D_0, D_e, and D_∞ represent the absorbances at some chosen wavelength in the absence of acid, at equilibrium for a given acid concentration, and for complete conversion of base to adduct respectively, then the slope of the plot of $\log(D_0 - D_e)/(D_e - D_\infty)$ against $\log[\text{MX}_n]_e$ gives the stoicheiometry of the adduct.[6] In most cases the plots were straight lines with slopes close to unity, indicating that 1 acid–1 base adducts are formed. However, for aluminium chloride the plots were curves that only approached a straight line of unit slope when $[\text{AlCl}_3]_3 > 7 \times 10^{-3}$ mol dm^{-3}. In the past [8,13] we have identified this type of effect as arising from deactivation of the acid by water remaining in the dried solvent. We have therefore used the results associated with the straight line part of the curve to estimate [6] the value of $K \{= (D_0 - D_e)/(D_e - D_\infty)[\text{AlCl}_3]_e\}$ for 1 : 1 adduct formation. Using this value of *K*, the true value of $[\text{AlCl}_3]_e = (D_0 - D_e)/(D_e - D_\infty)K$ was calculated for the mixtures corresponding to the curved region of the plot. Then $[\text{AlCl}_3]_r$ (the concentration of AlCl_3 effectively removed by water) could be estimated using equation (4). $[\text{AlCl}_3]_r$ was found to be 1—2 × 10^{-3} mol dm^{-3}, which agrees well with the value of 3 × 10^{-3} mol dm^{-3}

$$[\text{AlCl}_3]_r = [\text{AlCl}_3]_{\text{stoic}} - [\text{AlCl}_3]_e - [\text{Adduct}] = [\text{AlCl}_3]_{\text{stoic}} - \frac{(D_0 - D_e)}{(D_e - D_\infty)}\frac{1}{K} - \frac{(D_0 - D_e)}{(D_0 - D_\infty)}[\text{Base}]_{\text{stoic}} \quad (4)$$

found by Karl Fischer titration (see Experimental section). This agreement supports our interpretation of the curvature, and our assumption that aluminium chloride also forms 1 acid–1 base adducts.

Equilibrium Constants.—All our p*K* values determined at 25.0 °C are in the Table. Sample sets of p*K* values for a range of temperatures are also given for each acid with just one base. As in our earlier studies using diethyl ether as solvent [3] we find that, for a given acid, in the absence of steric complications, rectilinear relationships exist between the p*K* value in dioxan and the pK_a value of the base in water. The relevant correlation equations at 25.0 °C are (5)—(9).

ZnCl_2 $\quad \text{p}K = -0.83\,\text{p}K_a + 1.45 \quad (5)$

PhSnCl_3 $\quad \text{p}K = -0.79\,\text{p}K_a + 0.94 \quad (6)$

GaCl_3 $\quad \text{p}K = -0.93\,\text{p}K_a + 0.38 \quad (7)$

AlCl_3 $\quad \text{p}K = -1.17\,\text{p}K_a + 0.60 \quad (8)$

SnCl_4 (ref. 5) $\quad \text{p}K = -0.97\,\text{p}K_a - 0.21 \quad (9)$

p*K* Values calculated using these equations are compared in the Table with the experimental values. This comparison which clearly further confirms the essential regularity of behaviour shown by typical Lewis acids towards unhindered bases,[3] also shows that, in addition to anilines with 2-substituents, three apparently unhindered anilines consistently lead to deviant behaviour.

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* The solvent concentration is omitted conventionally [3] from the expression for *K*.

[11] R. M. Evans and R. S. Satchell, *J. Chem. Soc.* (*B*), 1970, 1667.

[12] A. Mohammad and D. P. N. Satchell, *J. Chem. Soc.* (*B*), 1968, 331.

[13] R. S. Satchell and D. P. N. Satchell, *J. Chem. Soc.* (*B*), 1967, 36.

$\mathrm{p}K$, ΔH^0, and ΔS^0 values for 1 : 1 adduct formation between metal halides and substituted anilines in dioxan

Units of K, ΔH^0, and ΔS^0 are $\mathrm{mol^{-1}\,dm^3}$, $\mathrm{kJ\,mol^{-1}}$, and $\mathrm{J\,mol^{-1}\,K^{-1}}$ respectively. Most of the quoted $\mathrm{p}K$ values refer to 25°. Sample sets of values are quoted for other temperatures. All $\mathrm{p}K$ values were reproducible to within ±0.02 units, except for $AlCl_3$ for which the reproducibility was ±0.05. $\mathrm{p}K$ Values in parentheses are calculated using the $\mathrm{p}K$–$\mathrm{p}K_\mathrm{a}$ correlation equations given in the text

$ZnCl_2$

Aniline derivative	$\mathrm{p}K_\mathrm{a}$	$\mathrm{p}K_{25.0}$	ΔH^0	ΔS^0
4-Methyl	5.10	−2.78 (−2.78)	−28.8 ± 0.8	−43.8 ± 2.9
3-Methyl	4.70	−2.48 (−2.45)	−28.0 ± 0.4	−45.9 ± 1.7
Hydrogen	4.60	−2.38 (−2.37)	−27.6 ± 0.4	−45.9 ± 1.7
3-Chloro-4-methyl	4.05	−1.89 (−1.91)	−24.2 ± 0.4	−44.3 ± 1.7
4-Bromo	3.91	−1.80 (−1.80)	−26.3 ± 2.5	−53.9 ± 7.5
3-Bromo	3.58	−1.49 (−1.52)		

3-Methylaniline $\mathrm{p}K_{15.4}$ −2.65, $\mathrm{p}K_{35.1}$ −2.33, $\mathrm{p}K_{43.7}$ −2.20, $\mathrm{p}K_{52.7}$ −2.07

$PhSnCl_3$

Aniline derivative	$\mathrm{p}K_\mathrm{a}$	$\mathrm{p}K_{25.0}$	ΔH^0	ΔS^0
3-Methyl	4.70	−2.79 (−2.77)		
Hydrogen	4.60	−2.68 (−2.69)	−54.7 ± 0.4	−135 ± 1
3-Chloro-4-methyl	4.05	−2.24 (−2.26)	−43.4 ± 2.9	−99 ± 9
4-Bromo	3.91	−2.14 (−2.15)	−49.7 ± 0.8	−126 ± 3
3-Bromo	3.58	−1.78 (−1.89)	−51.8 ± 0.8	−140 ± 3
3,4-Dichloro	2.93	−1.36 (−1.37)	−47.5 ± 0.8	−133 ± 3
4-Methyl-3-nitro	2.90	−1.44 (−1.35)	−46.8 ± 1.2	−129 ± 4
3-Nitro	2.50	−0.94 (−1.04)	−45.9 ± 0.4	−136 ± 2
3,5-Dichloro	2.37	−0.74 (−0.93)		
6-Methyl-3-nitro	2.32	−0.44 (−0.89)	−56.8 ± 0.4	−191 ± 2
4-Chloro-3-nitro	1.93	−0.54 (−0.58)	−45.5 ± 1.7	−142 ± 6
3-Methyl-4-nitro	1.45	−0.23 (−0.21)		

3-Nitroaniline: $\mathrm{p}K_{11.1}$ −1.34, $\mathrm{p}K_{18.0}$ −1.14, $\mathrm{p}K_{32.0}$ −0.75

$GaCl_3$

Aniline derivative	$\mathrm{p}K_\mathrm{a}$	$\mathrm{p}K_{25.0}$	ΔH^0	ΔS^0
3-Bromo	3.58	−2.85 (−2.95)	−27.6 ± 2.1	−38.4 ± 6.7
3,4-Dichloro	2.93	−2.33 (−2.34)	−22.5 ± 1.3	−31.3 ± 4.2
4-Methyl-3-nitro	2.90	−2.45 (−2.32)	−20.9 ± 0.4	−23.4 ± 1.2
3-Nitro	2.50	−2.01 (−1.95)	−24.6 ± 0.4	−44.3 ± 1.3
3,5-Dichloro	2.37	−1.69 (−1.82)	−21.7 ± 0.8	−40.9 ± 2.1
6-Methyl-3-nitro	2.32	−1.27 (−1.78)	−15.9 ± 0.8	−28.8 ± 2.9
2,4-Dichloro	2.00	−1.08 (−1.48)		
4-Chloro-3-nitro	1.93	−1.42 (−1.41)	−21.3 ± 0.8	−44.3 ± 2.9
2,3-Dichloro	1.83	−0.73 (−1.32)		

4-Methyl-3-nitroaniline $\mathrm{p}K_{14.5}$ −2.59, $\mathrm{p}K_{44.4}$ −2.23, $\mathrm{p}K_{52.4}$ −2.14

$SnCl_4$

Aniline derivative	$\mathrm{p}K_\mathrm{a}$	$\mathrm{p}K_{25.0}$*	ΔH^0	ΔS^0
3,4-Dichloro	2.93	−2.94 (−3.05)	−32.2 ± 0.8	−51.8 ± 2.5
4-Methyl-3-nitro	2.90	−3.06 (−3.02)	−32.2 ± 1.3	−49.3 ± 3.8
3-Nitro	2.50	−2.62 (−2.64)	−34.7 ± 0.8	−66.8 ± 2.1
3,5-Dichloro	2.37	−2.19 (−2.51)	−30.9 ± 2.5	−61.4 ± 7.9
6-Methyl-3-nitro	2.32	−2.13 (−2.46)	−29.6 ± 0.8	−58.9 ± 2.1
2,4-Dichloro	2.00	−1.76 (−2.15)	−28.8 ± 2.1	−63.5 ± 7.1
4-Chloro-3-nitro	1.93	−2.09 (−2.08)	−29.2 ± 1.3	−57.6 ± 3.8
2,3-Dichloro	1.83	−1.25 (−1.99)	−26.7 ± 0.4	−66.0 ± 1.7
3-Methyl-4-nitro	1.45	−1.96 (−1.62)	−27.6 ± 0.4	−54.7 ± 2.1
4-Nitro	0.99	−1.69 (−1.17)	−30.5 ± 0.4	−70.2 ± 1.3
2-Methyl-4-nitro	0.94	−1.18 (−1.12)	−30.5 ± 2.9	−79.8 ± 9.6

6-Methyl-3-nitroaniline $\mathrm{p}K_{16.0}$ −2.28, $\mathrm{p}K_{36.1}$ −1.94, $\mathrm{p}K_{44.2}$ −1.82, $\mathrm{p}K_{52.8}$ −1.67

$AlCl_3$

Aniline derivative	$\mathrm{p}K_\mathrm{a}$	$\mathrm{p}K_{25}$
3,4-Dichloro	2.93	−2.83 (−2.83)
4-Methyl-3-nitro	2.90	−2.88 (−2.79)
3-Nitro	2.50	−2.35 (−2.33)
6-Methyl-3-nitro	2.32	−2.26 (−2.11)
2,4-Dichloro	2.00	−2.14 (−1.74)
4-Chloro-3-nitro	1.93	−1.67 (−1.66)
3-Methyl-4-nitro	1.45	−1.82 (−1.10)
4-Nitro	0.99	−1.52 (−0.56)

* See ref. 5.

3,5-Dichloroaniline leads to lower K values than expected, and 3-methyl-4-nitro- and 4-nitro-aniline both lead to higher K values than expected. The reason for the behaviour of the dichloro-derivative is mysterious. The effect of a 4-nitro-substituent in these reactions has been discussed elsewhere.[6]

Our present results show also that similar successful correlation equations can be set up at temperatures other than 25°. We find that the slope of the plot of pK *versus* pK_a shows little dependence on temperature but that the value of the intercept (*i.e.* pK when p$K_a = 0$) increases with temperature. The increase is most marked for $PhSnCl_3$ and least for $ZnCl_2$ and $GaCl_3$.

Solvent Effects and Relative Acidities.—Dioxan (ε 2.2) and diethyl ether (ε 4.3) have similar dielectric constants and basicities; they might therefore be expected, with any given Lewis acid, to provide systems of similar acidity. Our present and earlier results [3] are in agreement with this expectation, and show that for any particular acid and reference base the pK values in the two solvents are the same within ± 0.5 units.

The full sequence of acid strengths at 25 °C now available for dioxan solutions is $ZrBr_4 \gg TeCl_4 > SnCl_4 > AlCl_3 > GaCl_3 > PhSnCl_3 > ZnCl_2 \gg MeSnCl_3$, Ph_2SnCl_2, $HgCl_2$ * (see Table and ref. 6). As expected [3] this is similar to the more limited sequence available for diethyl ether.[3,8,12,13]

An important point to emerge from the present study is that, in donor solvents at least, halides derived from the larger group IV metals are more powerful acids than those derived from group III elements. This result is in disagreement with our earlier predictions.[3,14]

ΔH^0 and ΔS^0 Values.—These are summarised in the Table.

(1) For *a given acid with a series of bases* the values of ΔH^0 and ΔS^0 fluctuate, but ΔH^0, in general, becomes more negative as the conventional (pK_a) base strength increases. Because of the significant fluctuations in both ΔH^0 and ΔS^0, even for unhindered bases, it is surprising that the correlations between pK and pK_a are so very good. This can scarcely be due entirely to chance; it is evident that similar factors must control these *fluctuations* in ΔH^0 and ΔS^0 within any one of the metal halide–dioxan–aniline systems *and* in the proton–water–aniline system. There can be no easy 'parallel changes in solvation' type of explanation. The common feature in each series is the systematic change in the aniline structure and it appears likely that whether a proton in water or a metal halide in dioxan is added to the nitrogen atom the trends in ΔH^0 and also in ΔS^0 as we pass from one ring substituent to another are usually similar. Our data go some way to support this conclusion. It is particularly significant that although for *meta*- and *para*-substituted anilines, the plot of ΔH^0 *versus* ΔS^0 for protonation in water is found to be rectilinear, *i.e.* it does not show fluctuations,[15-17] plots of our ΔH^0 values for $PhSnCl_3$ and $ZnCl_2$ *versus* ΔH^0 for protonation [17] for the same monosubstituted anilines are also reasonable straight lines. Our results with a wider range of anilines therefore make it unlikely that the straight line nature of the ΔH^0–ΔS^0 plot found for protonation of monosubstituted anilines will be found also with other anilines, *i.e.* it is unlikely to be a general result.

The ΔH^0 changes in all the systems will arise mainly as a result of changes in electronic substituent effects and in steric repulsions; the variations in the overall level of ΔS^0 perhaps reflect the effects of adduct formation on the motions of the base molecule. These suggestions could account for the rather random, although roughly parallel, variations found in the different series.

The results for the 2- or 6-substituted anilines, for which steric repulsions are likely to be of particular importance in the metal halide adducts, show that both ΔH^0 and ΔS^0 can be affected adversely.

(2) For *a given base with a series of acids* $-\Delta H^0$ decreases in the sequence $PhSnCl_3 > SnCl_4 > GaCl_3 \geqslant ZnCl_2$, whereas the corresponding K values follow the sequence $SnCl_4 > GaCl_3 > PhSnCl_3 > ZnCl_2$.

Further it is clear that, for a given base, ΔS^0 depends markedly on the acid. Therefore the assumption that entropy factors can be ignored in comparisons of Lewis acidity [18] is unjustified. Values of ΔS^0 decrease significantly along the sequence $PhSnCl_3 > SnCl_4 > ZnCl_2 > GaCl_3$ and to some extent changes in ΔH^0 and ΔS^0 actually oppose one another. In all we conclude therefore that values of ΔH^0 are an entirely unreliable guide to Lewis acid strength as measured by ΔG^0.

For a given base the absolute values of ΔH^0 and ΔS^0 for the different acids are clearly determined by a variety of factors including gross differences in adduct bond strength, in co-ordination geometry, and in adduct polarity leading to differences of solvation. However, while granting that important differences must exist for different acids engaging in formally the same type of equilibrium process [*e.g.* equation (1)], nevertheless the ΔH^0 and ΔS^0 values for $PhSnCl_3$ do seem exceptionally large by comparison with those for the other acids. Thus for $GaCl_3$, $ZnCl_2$, and $SnCl_4$ ΔS^0 covers the approximate range -30 to -70 J mol^{-1} K^{-1}, whereas for $PhSnCl_3$ ΔS^0 *ca.* -135 J mol^{-1} K^{-1}. This suggests to us that a different chemical process may be occurring for $PhSnCl_3$. Normally in these acid–base equilibria it would be expected that the incoming aniline displaces a co-ordinated solvent molecule from the central metal atom [equation (1)]. If the rather weak and bulky

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* $MeSnCl_3$, Ph_2SnCl_2, and $HgCl_2$ were too weak to study quantitatively.

[14] D. P. N. Satchell and R. S. Satchell, *Quart. Rev.*, 1971, **25**, 171.

[15] P. D. Bolton and F. M. Hall, *Austral. J. Chem.*, 1967, **20**, 1797.

[16] P. D. Bolton and F. M. Hall, *Austral. J. Chem.*, 1968, **21**, 939.

[17] C. L. Liotta, E. M. Perdue, and H. P. Hopkins, *J. Amer. Chem. Soc.*, 1973, **95**, 2439.

[18] A. Mohammad and D. P. N. Satchell, *J. Chem. Soc. (B)*, 1966, 527.

$PhSnCl_3$ were only to form a 1 acid–1 dioxan adduct in solution. instead of the 1 acid–2 dioxan adduct expected, for example with $SnCl_4$, then the incoming aniline molecule could simply add to the tin atom without displacing solvent [reaction (2)]. This could account for the anomalously negative values of ΔS^0 and ΔH^0 found with $PhSnCl_3$.

[5/1875 *Received, 29th September*, 1975]