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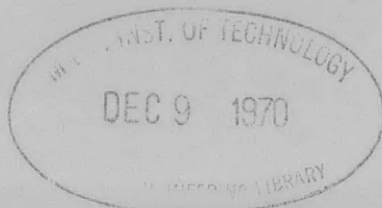
APPLICATION OF A THEORY OF BINARY SOLUTION SURFACE TENSION TO ADSORPTION PHENOMENA

By
Donald J. Cotton

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October 1969



Report 3065

Application of a Theory of Binary Solution Surface
Tension to Adsorption Phenomena

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ABSTRACT

A theory of surface tension of binary solutions is extended to apply to adsorption from solutions. Experimental work on the adsorption of several fatty acids from aqueous solutions by Nichrome V powder is presented and the theory is used in the analysis. The theory is also tested with literature data on adsorption from aqueous solutions of aliphatic and aromatic compounds by carbon blacks and graphites. A method for the determination of specific surface areas of adsorbents from solution is presented and applied.

FOREWORD

Adsorption of polar molecules from solution by solid surfaces is important to the Navy inasmuch as most naval machinery is exposed to liquid environments (water, lubricants) in which polar molecules either exist as contaminants or are intentionally incorporated as additives. Adsorption processes in such systems determine both the effectiveness of additives and the damage of contaminants. This is to present the results of research which has been conducted to increase knowledge of the effect of polar molecules at metal surfaces.

ADMINISTRATIVE INFORMATION

This research is a product of IR/IED Program, Sub-project Z-R011 01 01, Task 05600, Assignment 821-147.

This report was submitted, in part, as a paper, coauthored by Dr. J. L. Shereshfsky, Professor Emeritus, Howard University, Washington, D.C., to the Journal of Physical Chemistry.

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INTRODUCTION

In this report, a theory of binary solution surface tension¹ is extended to adsorption phenomena on solid adsorbents in contact with a binary solution. The adsorption process is regarded as an exchange between different types of interfaces accompanied by a decrease in interfacial free energy, and the solid-solution interface is assumed to be a region consisting of solid-solvent and of solid-solute interfaces in equilibrium with the solution.

THEORETICAL CONSIDERATIONS

When solute is adsorbed on the surface of the adsorbent, the change in interfacial free energy is given by

$$\Delta\sigma = \sigma_{13} - \sigma \quad \dots (1)$$

where σ_{13} is the tension of the solvent-solid interface, and σ is the tension of the solution-solid interface which is assumed to obey the law of additivity as given by

$$\sigma = \sigma_{13}x_{1s} + \sigma_{23}x_{2s} \quad \dots (2)$$

where σ_{23} is the tension of the solute-solid interface, and x_{1s} and x_{2s} are, respectively, the solvent and the solute mole fractions in the interfacial region. It is assumed that all solvent or solute molecules in the interfacial region within a hemisphere of attraction affect the tension of the interface and that the overall interfacial tension results from the interaction of the solid with all such molecules; consequently, σ_{13} and σ_{23} are not necessarily monolayer tensions of solvent or of solute molecules adsorbed on the solid.

The interfacial tension change due to adsorption is related to solution concentration by

$$\sigma_{13} - \sigma = \frac{\Delta\sigma_0 x_{2b} e^{\Delta F_s/RT}}{1 + x_{2b} \left(e^{\Delta F_s/RT} - 1 \right)} \quad \dots (3)$$

¹Shereshefsky, J. L., J. Colloid and Interface Sci., Vol. 24, 1967, p. 317

where

$$\Delta\sigma_0 = \sigma_{13} - \sigma_{23}, \quad \dots (4)$$

$$\Delta F_s = N\Delta\sigma_0 A_2/t, \quad \dots (5)$$

x_{2b} is the solute mole-fraction in the solution, N is Avogadro's number, A_2 is the solute area per molecule in a monolayer, and t is the number of molecular layers in the interface.

Equation (3) is identical in form with the equation describing the surface tension of a binary solution derived by Shereshefsky¹ and can be derived in an analogous manner. If the boundaries defining the interfacial region are chosen so that the Gibbs excess in the interface is limited to the solute, then

$$\Delta\sigma = RT \int_0^a \Gamma_2 d \ln(a_2) \quad \dots (6)$$

relates the decrease in interfacial tension to the surface excess Γ_2 of the solute and its activity in the solution a_2 . For dilute solutions, mole-fractions may be used instead of activities, and Γ_2 may be obtained from adsorption data by using Guggenheim and Adam's² equation.

The reciprocal form of Equation (3) is more tractable and when combined with Equation (6) becomes

$$\frac{x_{2b}}{\Delta\sigma} = \frac{1}{\Delta\sigma_0} e^{-\Delta F_s/RT} + \frac{x_{2b}}{\Delta\sigma_0} \left(1 - e^{-\Delta F_s/RT} \right), \quad \dots (7)$$

from which it follows that $x_{2b}/\Delta\sigma$ is a linear function of concentration, x_{2b} , and that the constants $\Delta\sigma_0$ and ΔF_s can be obtained from the slope, m , and intercept, b , with the following relationships:

$$\frac{m}{b} = e^{\Delta F_s/RT} - 1 \quad \dots (8)$$

and

$$m + b = \frac{1}{\Delta\sigma_0} \quad \dots (9)$$

²Guggenheim, E. A., and N. K. Adam, Proc. Roy. Soc. (London), Vol. A139, 1933, p. 218

When the specific surface area of the adsorbent under the conditions of the experiment, α , is unknown, Equation (7) can be expressed by

$$\frac{x_2 b}{\Sigma} = \frac{1}{\Sigma_0} e^{-\Delta F_s / RT} + \frac{x_2 b}{\Sigma_0} \left(1 - e^{-\Delta F_s / RT} \right) \quad \dots (10)$$

where $\Sigma = \alpha \Delta \sigma = RT \int_0^a \alpha \Gamma_2 d \ln(a_2)$, and $\Sigma_0 = \alpha \Delta \sigma_0$.

The parameters ΔF_s and Σ_0 are of special interest because they can be utilized, as will be shown later, for the determination of specific surface area.

In recent years, other equations derived from thermodynamic considerations and dealing with adsorption from solution have been reported in the literature.^{3,4,5,6} These equations, while similar in form to Equation (3), differ from it in several aspects: (1) The terms corresponding to ΔF_s have different meanings, and (2) These equations have no term equivalent to $\Delta \sigma_0$ or A_2 , the area of the adsorbed molecule, or t , the thickness of the interfacial region.

TESTS OF THE EQUATION

Studies of adsorption from aqueous solutions of butanoic, pentanoic, hexanoic, and heptanoic acid by Nichrome V powder were made, and data by Hansen and co-workers⁷ on adsorption from solutions of butyl alcohol, phenol, cyclohexanol, and amyl alcohol by several different carbons were used to test the equation.

ADSORPTION ONTO NICHROME V

The Nichrome V powder consisted of spherical particles, with known radii distribution, manufactured by the Linde Company. It contained less than 2% nonspherical particles with diameters ranging from 10 to 150 microns. The apparent specific area, determined by graphical integration of the particle size distribution curve supplied by the manufacturer, was 175 cm² per gram.*

³Shay, G., Acta Chim Acad. Sci. Hung., Vol. 10, 1956, p. 281

⁴Siskova, M., and E. Erdos, Coll. Czech. Chem. Comm., Vol. 25, 1960, pp. 1729, 3086

⁵Everett, D. H., Trans. Faraday Soc., Vol. 60, 1964, p. 1803

⁶Suri, S. K., and V. Ramakrishna, Journ. Phys. Chem., Vol. 72, 1968, p. 1555

⁷Hansen, R. S., Y. Fu. and F. E. Bartell, Journ. Phys. Chem., Vol. 53, 1949, p. 1141

*Abbreviations used in this text are from the GPO Style Manual, 1967, unless otherwise noted.

The surface of the powder, heated in a quartz tube, was reduced for 4 hours in a stream of hydrogen gas. Solutions were made by weight from Eastman Kodak chromophotographic grade chemicals and doubly demineralized water.

An approximately 20-gram sample of pretreated powder, weighed into a glass-stoppered bottle, was covered with 5 ml of solution. The sealed bottle and contents were vigorously shaken for several minutes and then were agitated overnight in a constant temperature bath at $25^{\circ} \pm 0.01^{\circ}$ C. The solution was then decanted into an interferometer cell, and the concentration was measured against pure water with a Rayleigh differential interferometer. Adsorption was calculated from the initial and final concentration, c_i and c_f , according to Equation (11).

$$\underline{a} = v \Delta c / m \underline{\alpha} \quad \dots (11)$$

where $\Delta c = c_i - c_f$, v is the volume of the solution, m is the weight of powder, and $\underline{\alpha}$ is the apparent specific surface area determined independently. Because of the high dilution of the solution, \underline{a} was considered equivalent to Gibbs Γ_2 . These surface excess values were fitted to a seventh order polynomial in terms of concentration as shown by Equation (12).

$$\Gamma_2 = \sum_{n=1}^N a_n c^n \quad \dots (12)$$

where a_n is a constant, c is the equilibrium concentration, and N is the number of data points. The polynomial was integrated as shown in Equation (13) to obtain interfacial tension changes, $\Delta \sigma'$.

$$\Delta \sigma' = RT \int_0^c \Gamma_2 d \ln c = RT \sum_{n=1}^N \frac{a_n}{n} c^n \quad \dots (13)$$

where the prime indicates that an apparent specific surface area determined under different experimental conditions was employed.

The applicability of Equation (7) to the data is shown in Figure 1, where $x_{2b} / \Delta \sigma'$ versus x_{2b} plots are given. The slopes and intercepts of the linear plots were used to calculate $\Delta \sigma'_0$, ΔF_s , and $A_2(\text{exp})$ per molecule, assuming $t = 1$. These values are given in Table 1.

Table 1
Surface Energy and Surface Area Constants of
Various Organic Acids Adsorbed onto Nichrome V

Acid	$\Delta \sigma'_0$ erg/cm ²	$\Delta F_s \times 10^{-9}$ erg/mole	$A_2(\text{exp.})$ \AA^2	$A_2(\text{vert.})^8$ \AA^2	α'
Butanoic	44.9	216.4	79.7	24.5	0.31
Pentanoic	45.1	174.9	64.3	23.6	0.37
Hexanoic	41.6	212.7	84.9	23.2	0.27
Heptanoic	40.6	273.9	112.6	23.0	0.20

ADSORPTION ONTO VARIOUS CARBONS

The data by Hansen and co-workers on the adsorption of butyl alcohol, phenol, cyclohexanol, and amyl alcohol from aqueous solution by various carbon powders includes a quantity ϕ which corresponds to $\Delta\sigma'$, the integral of Equation (6). The ratio of x_{2b}/ϕ was plotted against x_{2b} . A least square fit of each data set was made (Figures 2-5), and the constants $\Delta\sigma'_0$ and ΔF_s were calculated by employing Equations (8) and (9). Results are summarized in Table 2.

Table 2
Surface Energy and Surface Area Constants for Several Alcohols Adsorbed onto Four Carbons

Adsorbate	$\Delta\sigma'_0$ erg/cm ²	$\Delta F_s \times 10^{-9}$ erg/mole	A_2 (exp.) $\overset{\circ}{A}^2$	α'	t	t*
Sugar Charcoal	32.1	148.5	76.4	0.32	2	1
Carbon A	38.5	131.2	56.4	0.43	3	3
Carbon B	39.8	136.7	57.2	0.43	3	3
Channel Black	41.7	128.4	51.1	0.48	3	3
Phenol ($A_2 = 41.\overset{\circ}{A}^2$) ⁹						
Sugar Charcoal	35.4	159.4	74.8	0.56	2	1
Carbon A	63.9	149.8	38.9	1.10	4	3
Carbon B	48.7	171.9	58.7	0.72	3	3
Channel Black	48.3	152.9	52.6	0.80	3	3
Cyclohexanol ($A_2 = 39.\overset{\circ}{A}^2$) ⁹						
Sugar Charcoal	20.8	167.1	133.4	0.29	1	1
Carbon A	39.3	163.9	69.6	0.56	2	2
Carbon B	45.7	154.9	56.3	0.70	2	2
Channel Black	50.6	142.8	52.4	0.75	2	2
Amyl Alcohol ($A_2 = 24.5\overset{\circ}{A}^2$) ⁸						
Sugar Charcoal	29.0	159.9	91.6	0.27	2	1
Carbon A	37.0	158.0	66.8	0.36	3	3
Carbon B	50.6	164.7	54.1	0.45	3	3
Channel Black	42.6	155.0	60.4	0.41	3	3

The data confirm the derived equation for the several sets of measurements. The ΔF_s , defined as free energy change due to the displacement of an area of solvent-adsorbent interface equivalent to the area of the solute-adsorbent interface formed by a mole of solute, is approximately of the same magnitude for the different carbons. On the other hand, the molecular areas of the adsorbates on sugar charcoal are considerably higher than on the other adsorbents. The specific area of sugar charcoal is 790 cm² per gram while the other carbons are from 7 to 50 times less. In the solution phase, the specific area of this finely divided powder is probably considerably reduced by coalescence, as will be shown later.

DETERMINATION OF SPECIFIC SURFACE AREA

ΔF_s is independent of the specific surface area of the adsorbent. Consequently, by using an adsorbate of known molecular area and employing Equation (5), $\Delta\sigma_o$ can be evaluated, even when the specific area, α , of the adsorbent for the experimental condition is unknown. This combined with Σ_o , and $\Delta\sigma_o'$, obtained using an apparent specific area, permits the calculation of the specific surface area α or the specific surface coefficient α' as is seen from the following definition:

$$\Sigma_o = \alpha \Delta\sigma_o = \underline{\alpha} \Delta\sigma_o' \quad \dots (14)$$

and

$$\alpha' = \alpha / \underline{\alpha} = \Delta\sigma_o' / \Delta\sigma_o \quad \dots (15)$$

In the Nichrome V study, one of the adsorbates was butanoic acid. Ward⁸ reported the cross-sectional area of a butanol molecule as 24.5 sq. Å. In Table 1 the experimental area per molecule for butanoic acid is 79.7 sq. Å and $\Delta\sigma_o$ is 44.9 erg/cm². Inasmuch as the molecular area for butanoic acid is 24.5 sq. Å, the high value of approximately 80 sq. Å indicates that either the specific surface area of 175 cm² per gram assigned to the Nichrome V powder was erroneous or that only a fraction of the apparent area was effective as an adsorbent from solution.

If the molecular area of the acid on the Nichrome is 24.5 sq. Å, and $t = 1$, then

$\Delta\sigma_o = t \Delta F_s / A_2 = 146.6 \text{ erg/cm}^2$, and the specific surface coefficient, α' is 0.31 which means that the effective specific surface area of the Nichrome V is 54 cm² per gram.

The data on the carbon adsorption study, summarized in Table 2, demonstrate how Equation (1) can be utilized for specific surface area determination. Consider n-butanol, for the purpose of the analysis. If the molecular area of n-butanol is unknown, but is approximately the same on all the carbons, then

$$A_2 = (t_F \Delta F_s / \Delta\sigma_o') = (t_A \Delta F_s / \Delta\sigma_o') = (t_B \Delta F_s / \Delta\sigma_o') = (t_H \Delta F_s / \Delta\sigma_o') \quad \dots (16)$$

where F, A, B, H are the different carbons, as designated by Hansen and co-workers.⁷ If the ΔF_s 's and the $\Delta\sigma_o$'s in Table 2 are substituted in the above,

$$A_2 = 4.6t_F = 3.4t_A = 3.4t_B = 3.1t_H$$

From this it follows that $t_F = 2$, and $t_A = t_B = t_H = 3$. These values of t , with the exception of that for sugar charcoal (F), agree with the calculated number of monolayers found by the original investigators. In Table 2, t was calculated from Equation (16) and molecular areas by Ward⁸ and Smith;⁹ t^* was calculated by Hansen from the B. E. T. equations. Generally, agreement is good. Disagreement in the case of sugar charcoal can be explained by the change in the specific area this adsorbent undergoes in solution.

⁸Ward, A. F. H., Trans. Faraday Soc., Vol. 41, 1949, p. 399

⁹Smith, R. N., C. Pierce, and H. Cordes, Journ. Amer. Chem. Soc., Vol. 72, 1950, p. 5595

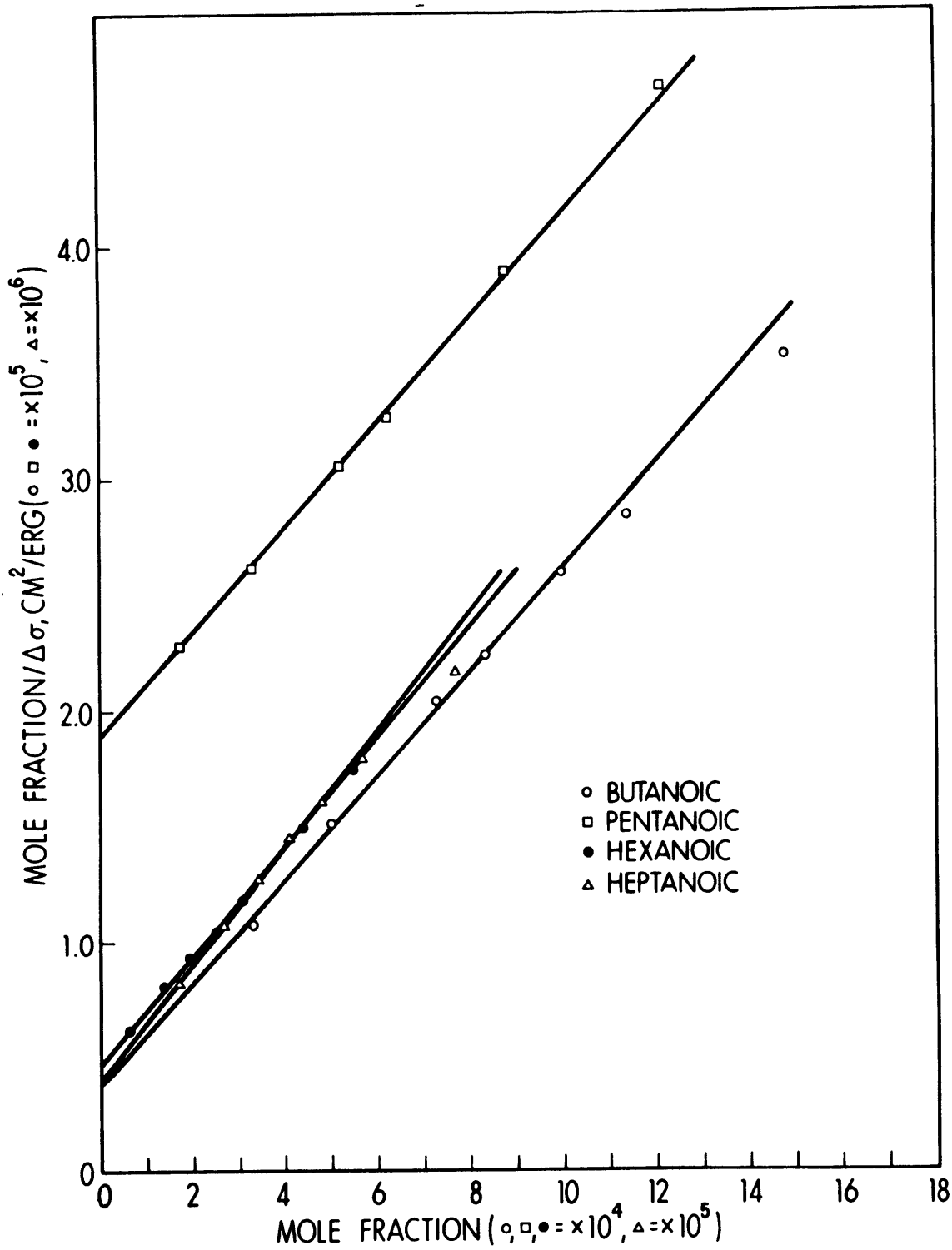


Figure 1
 Mole Fraction Divided by $\Delta\sigma$ Versus Mole Fraction for
 Aqueous Solutions of Butanoic, Pentanoic, Hexanoic, and
 Heptanoic Acid Against Nichrome V Power at 25° C

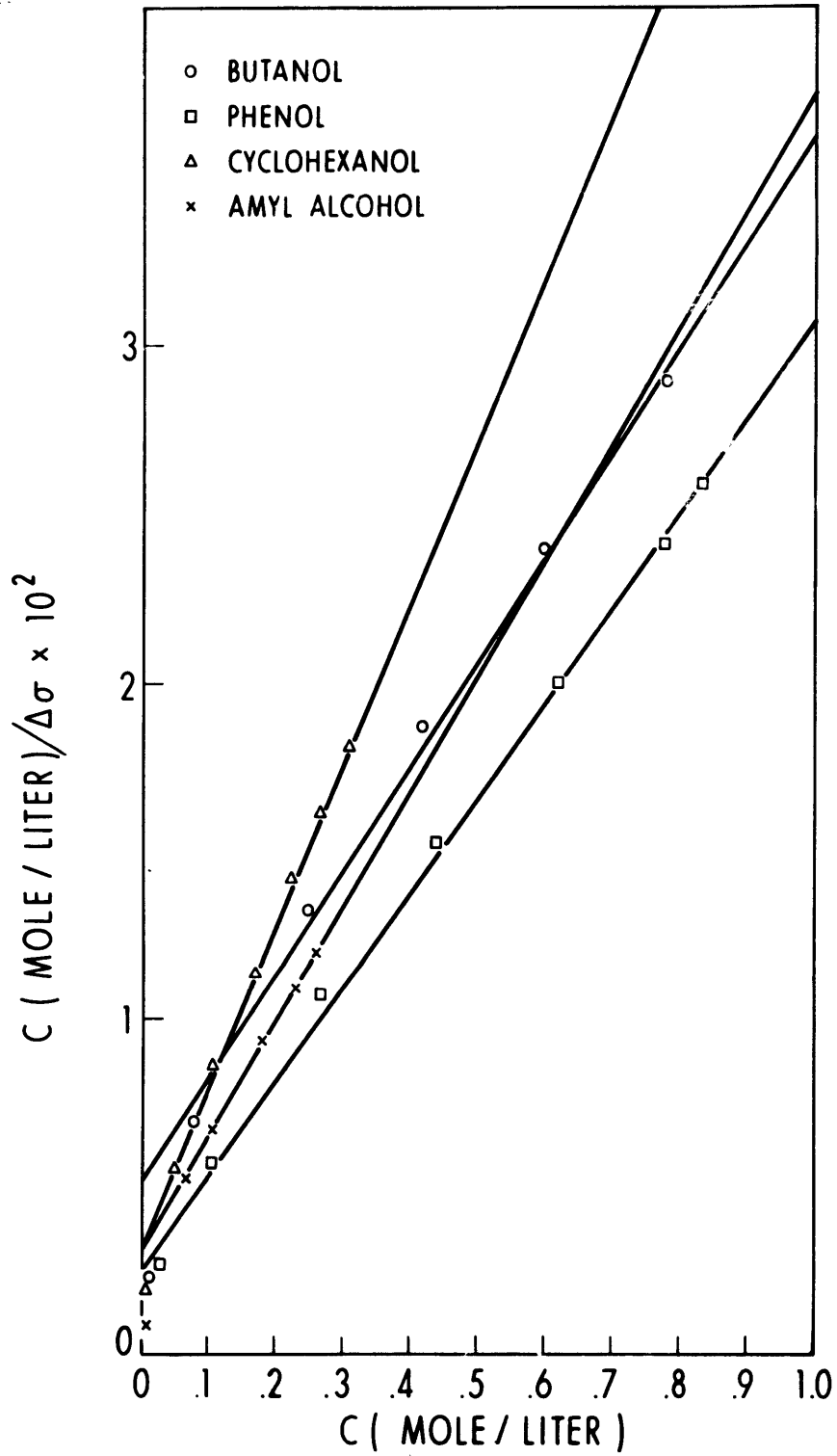


Figure 2
 Concentration Divided by $\Delta\sigma$ Versus
 Concentration for Aqueous Solutions of Various
 Alcohols Against Sugar Charcoal at 25° C

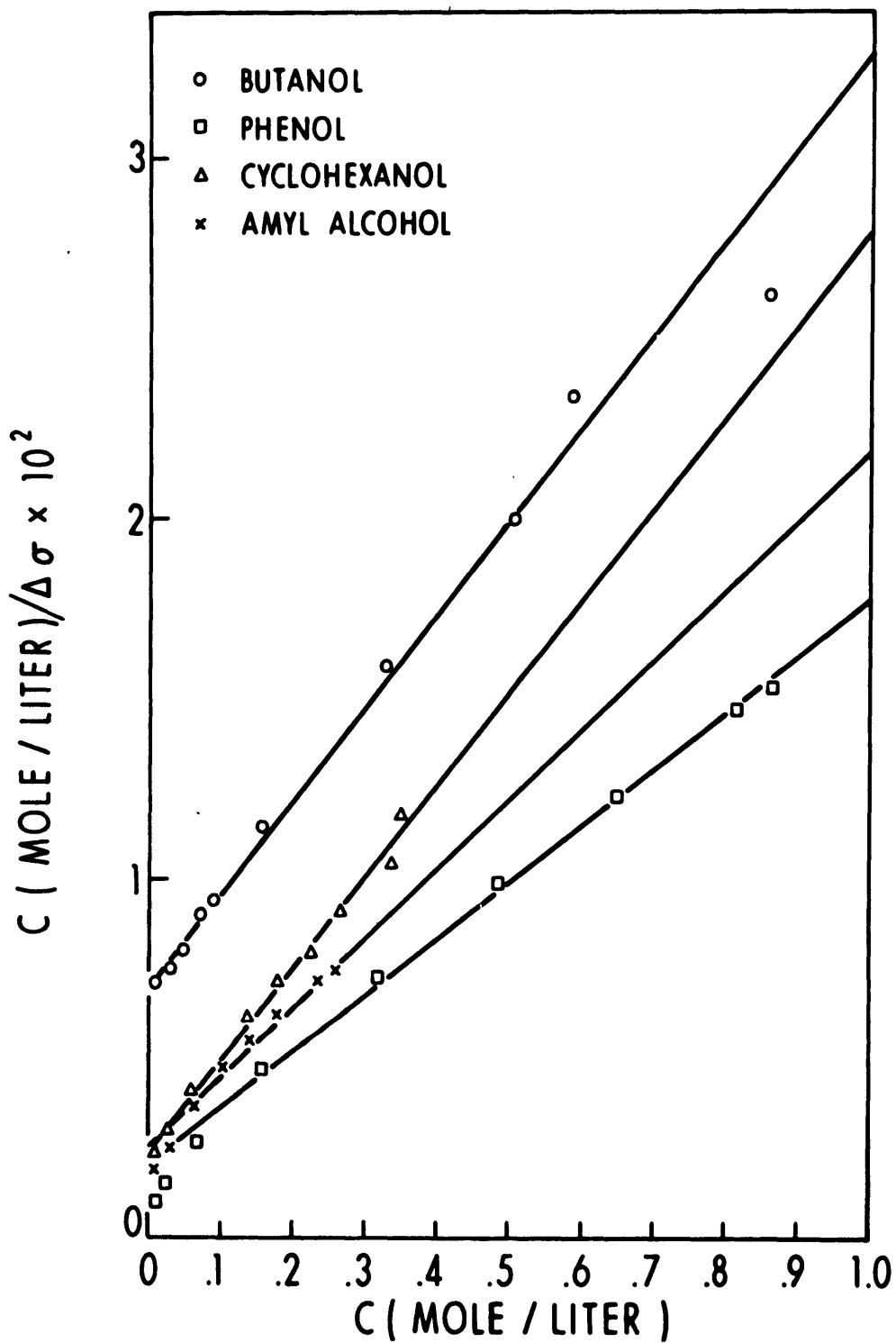


Figure 3
 Concentration Divided by $\Delta\sigma$ Versus
 Concentration for Aqueous Solutions of Various
 Alcohols Against Carbon A at 25° C

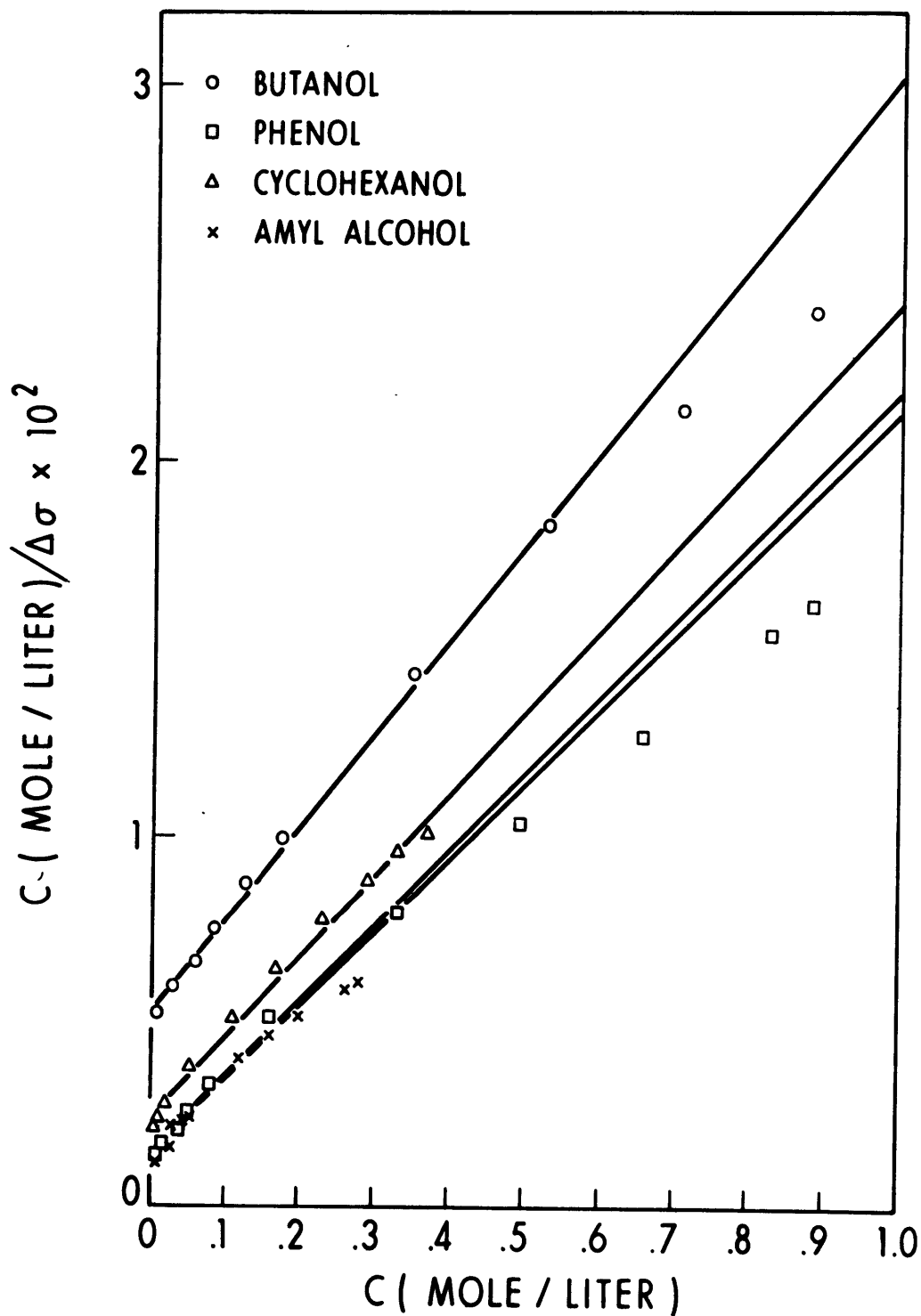


Figure 4
 Concentration Divided by $\Delta\sigma$ Versus
 Concentration for Aqueous Solutions of Various
 Alcohols Against Charcoal B at 25° C

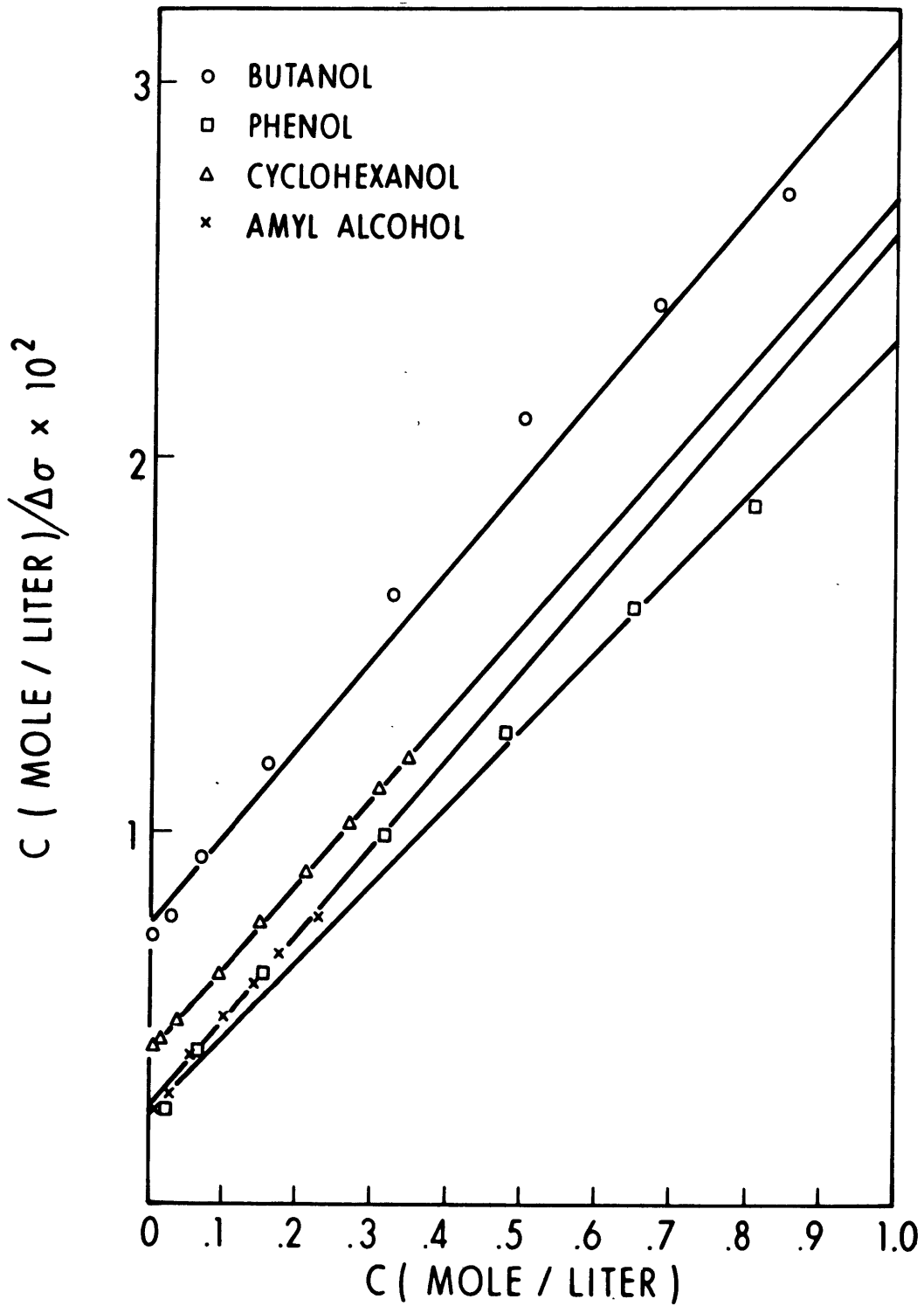


Figure 5
 Concentration Divided by $\Delta\sigma$ Versus
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<p>NAVSHIPRANDLAB Annapolis Report 3065 APPLICATION OF A THEORY OF BINARY SOLUTION SURFACE TENSION TO ADSORPTION PHENOMENA, by Donald J. Cotton. October 1969. 18 pp. Figs. UNCLASSIFIED</p> <p>A theory of surface tension of binary solutions is extended to apply to adsorption from solutions. Experimental work on the adsorption of several fatty acids from aqueous solutions by Nichrome V powder is presented and the theory is used in the analysis. The adsorption from aqueous solutions of aliphatic and aromatic compounds by carbon blacks and graphites. A method for the determination of specific surface areas of adsorbents from solution is presented and applied. (Task Area Z-R011 01 01, Task 05600)</p>	<p>1. Adsorption 2. Binary Solution 3. Surface Tension 4. Shereshefsky's Equation 5. Interfacial Tension I. Cotton, Donald J. II. Title... III. Report 3065</p> <p>UNCLASSIFIED</p>	<p>NAVSHIPRANDLAB Annapolis Report 3065 APPLICATION OF A THEORY OF BINARY SOLUTION SURFACE TENSION TO ADSORPTION PHENOMENA, by Donald J. Cotton. October 1969. 18 pp. Figs. UNCLASSIFIED</p> <p>A theory of surface tension of binary solutions is extended to apply to adsorption from solutions. Experimental work on the adsorption of several fatty acids from aqueous solutions by Nichrome V powder is presented and the theory is used in the analysis. The theory is also tested with literature data on adsorption from aqueous solutions of aliphatic and aromatic compounds by carbon blacks and graphites. A method for the determination of specific surface areas of adsorbents from solution is presented and applied. (Task Area Z-R011 01 01, Task 05600)</p>	<p>1. Adsorption 2. Binary Solution 3. Surface Tension 4. Shereshefsky's Equation 5. Interfacial Tension I. Cotton, Donald J. II. Title... III. Report 3065</p> <p>UNCLASSIFIED</p>
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APPLICATION OF A THEORY OF BINARY SOLUTION SURFACE TENSION TO ADSORPTION PHENOMENA, by Donald J. Cotton. October 1969. 18 pp. Figs. UNCLASSIFIED

A theory of surface tension of binary solutions is extended to apply to adsorption from solutions. Experimental work on the adsorption of several fatty acids from aqueous solutions by Nichrome V powder is presented and the theory is used in the analysis. The theory is also tested with literature data on adsorption from aqueous solutions of aliphatic and aromatic compounds by carbon blacks and graphites. A method for the determination of specific surface areas of adsorbents from solution is presented and applied. (Task Area Z-ROLL 01 01, Task 05600)

1. Adsorption
2. Binary Solution
3. Surface Tension
4. Sherashafsky's Equation
5. Interfacial Tension
- I. Cotton, Donald J.
- II. Title...
- III. Report 3065

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