



Electric and bond moments of some carbon, silicon and titanium organic compounds
by Walter Katz

A THESIS Submitted to the Graduate Faculty in partial fulfillment of the requirements for the degree
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Abstract:

A further study of the dielectric and chemical properties of titanium-tetraethylate has been made. This compound forms aggregates in solution up to approximately a trimer, a type of association which is apparently not based upon hydrogen bonding but rather upon the coordination valence of titanium. X-ray diffractions of titanium-tetraethylate crystals could be used to determine whether this is actually true. The dipole moments of $\text{Ti}(\text{OC}_2\text{H}_5)_4$, $\text{Ti}(\text{OC}_3\text{H}_7)_4$, $\text{Ti}(\text{OC}_4\text{H}_9)_4$, $\text{TiCl}(\text{OC}_2\text{H}_5)_3$, $\text{SiCl}(\text{OC}_2\text{H}_5)_3$ and $\text{HC}(\text{OC}_2\text{H}_5)_3$ were determined in hexane solution at 25°C and found to be 1.58, 1.32, 1.04, 2.50, 1.75 and 0.7 Debye units respectively. The method used for the determination of the electric moments consisted of measuring the dielectric constants and specific volumes of dilute solutions of the compounds in hexane and extrapolating the resulting straight lines to infinite dilution. The slopes and intercepts were then used to calculate the molar polarisation from an equation derived by Halverstadt and Kumler (8). The electronic and atomic polarisations were obtained either from refractive index and density measurements of the pure compound or by measuring the refractive indices of dilute solutions of the compound in hexane solution and using the slope and intercept of the plot of the square of the refractive index against mole fraction in the Halverstadt and Kumler equation. The molar polarisations of titanium tetraethylate at low temperatures were corrected and the polarization plotted against $1/T$.

ELECTRIC AND BOND MOMENTS OF SOME CARBON,
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A THESIS

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I. ABSTRACT

A further study of the dielectric and chemical properties of titanium-tetraethylate has been made. This compound forms aggregates in solution up to approximately a trimer, a type of association which is apparently not based upon hydrogen bonding but rather upon the coordination valence of titanium. X-ray diffractions of titanium-tetraethylate crystals could be used to determine whether this is actually true. The dipole moments of $Ti(OC_2H_5)_4$, $Ti(OC_3H_7)_4$, $Ti(OC_4H_9)_4$, $TiCl(OC_2H_5)_3$, $SiCl(OC_2H_5)_3$ and $Hf(OC_2H_5)_4$ were determined in hexane solution at 25°C and found to be 1.58, 1.32, 1.08, 2.50, 1.75 and 0.7 Debye units respectively. The method used for the determination of the electric moments consisted of measuring the dielectric constants and specific volumes of dilute solutions of the compounds in hexane and extrapolating the resulting straight lines to infinite dilution. The slopes and intercepts were then used to calculate the molar polarization from an equation derived by Halverstadt and Kusler (8). The electronic and atomic polarizations were obtained either from refractive index and density measurements of the pure compound or by measuring the refractive indices of dilute solutions of the compound in hexane solution and using the slope and intercept of the plot of the square of the refractive index against mole fraction in the Halverstadt and Kusler equation. The molar polarizations of titanium tetraethylate at low temperatures were measured and the polarization plotted against $1/T$.

II. INTRODUCTION

The inorganic titanates have been known and used as dielectrics for many years. The organic derivatives of $TiCl_4$ have been prepared (1), but practically no work has been done to determine physical properties or structures. Since the inorganic titanates have very unusual dielectric properties it is reasonable to inquire into the properties of organic derivatives of $TiCl_4$. Furthermore, determination of structures and physical properties of these compounds has so far been neglected. Thus it is the purpose of this work to study their dielectric and other properties.

Crowe (4) determined the dipole moments of titaniumtetraethylate, monochlorotriethoxytitanium and trichloromonophenoxytitanium, using benzene as a solvent. Theoretical calculation indicates the possibility of some solvent effect. The author has redetermined the dipole moments of titaniumtetraethylate and monochlorotriethoxytitanium in hexane to study the effect of using a different solvent. Crowe also noticed that titaniumtetraethylate associates in benzene solution; thus molecular weights have been determined to consider the extent of the association.

Although the work is primarily concerned with titanium compounds, the dipole moments of the two similar compounds of carbon and silicon were measured to establish some correlation between dipole moment and molecular structure among analogous organic compounds of carbon, silicon and titanium. The moments of ethylorthoformate and monochlorotriethoxysilicon have not been measured before, although some dielectric studies have been made on certain esters of carbon (6) and silicon (23) tetrachlorides.

III. LITERATURE STUDY

A great many publications have appeared in the last twenty years in the field of dielectric constants, dipole moments and related phenomena. One cannot adequately cover in a literature study all the articles. Excellent reviews are found in Debye's "Polar Molecules" (5) and Smyth's "Dielectric Constant and Molecular Structure" (22). Onsager (16) has modified the Debye theory of dielectrics by deriving an alternative expression for the internal field induced by a dipole placed in an electric field. Onsager considers a spherical, polar liquid molecule as the dipole. The environment of the dipole is treated as being a homogeneous continuum. This is, of course, a simplification of the actual situation. Kirkwood (14) has pointed out that fluctuations in the induced moment of a molecule lead to correction terms in the Lorentz equation (5), which is used for the derivation of the Debye equation.

An extensive search through the literature revealed that very little is known concerning the dielectric properties of titanium organic compounds, particularly the esters of $TiCl_4$. Bischoff and Adkins (2) and Jennings, Wardlaw and Way (13) have prepared various esters of $TiCl_4$ and chloro derivatives of the esters. The physical properties of these titanium organic compounds were not studied, however. Schumann (21) reacted $TiCl_4$ with phenol and obtained $Ti(O_6C_5H_5)_4 \cdot HCl$ but no extensive study of the physical properties of the compound was made. Hauser and Levite (11) prepared compounds from the reaction of $TiCl_4$ with aminophenols and Rosenheim and Sorge (20) analyzed the product from the reaction of

TiCl_4 with oxy-carboxylic acid titanates and polyphenolic titanates. These authors also failed to report on the physical properties of the compounds prepared.

The theoretical calculations of molecular and bond moments have been considered by many investigators for simple and complex molecules. The simplest and most common method is vector addition of the group or bond moments constituting the molecule. The water molecule, for example, has a large permanent dipole moment and, therefore, it cannot be a symmetrical linear molecule. From an examination of the spectrum of water vapor (9) the angle between the O-H linkages has been found to be 106° and, therefore, knowledge of the total dipole moment enables one to calculate, with a fair degree of accuracy, the O-H bond moment. For diatomic molecules, such as HCl, the observed dipole moment is identical with the H-Cl bond moment. The problem, in the latter case, however, is complicated by the deformation of the electron system of the halogen atom by the hydrogen atom. Thus the dipole value for HCl of 6.1 Debye units calculated from infrared measurements of the distance between the electrical centers differs markedly from the observed value of 1.03 Debye units.

The theoretical calculation of dipole moments is further complicated by the restricted rotation of polar groups. The ethylene dichloride molecule (22) illustrates the effect of restricted rotation, the restriction being due to the mutual interaction of the two CH_2Cl groups rotating about the C-C axis. Using the C-Cl valency angle as 70° and the C-Cl bond moment as 1.5, leads to a dipole moment of 2.1 as a limit. Observed values in various solvents, in different states and at different temperatures show

none higher than 1.8 D. Meyer (7) has made a further study of the effect of restricted rotation on the dipole moment by taking into consideration the mutual potential energy of the two rotating dipoles. He concludes that if this energy is appreciably less than the energy of thermal agitation, kT , then rotation can occur freely. However, the mutual potential energy of the two rotating dipoles in ethylene dichloride is about two to three times kT and, therefore, rotation will be hindered. Hydroquinone diethyl ether (9) shows a mutual potential energy of rotating groups very much smaller than kT , since the $O-C_2H_5$ groups are separated by at least 6×10^{-18} cm and the group moments are only 1.1×10^{-18} . Free rotation is possible and the resultant moment, 1.73 compares favorably with the calculated value of 1.46.

Eyring, (8) assuming free rotation, has developed an expression for the resultant moment of complex rotating molecules by means of matrix algebra. His formula is actually an extension of the law of cosines which is employed in the vector addition of moments in simple molecules. The Eyring formula states in words, "For free rotation about connecting lines the mean square sum of n vectors is equal to the sum of the squares of the lengths of the separate vectors plus twice all the possible products of the lengths of the two vectors multiplied by the cosine of the angle, θ_{jk} , made by the directed lines connecting the pair". The mathematical expression is as follows:

$$C_m^2 = \sum C_n^2 + 2 \sum C_j C_k \cos \theta_{jk}$$

This equation offers a means of calculating the theoretical dipole moments

of numerous compounds if one has a knowledge of the individual bond moments. Pauling (17) has shown that a relation exists between the electronegativity scale, based upon single bond energies, and the bond moments. According to this, the difference in electronegativity should equal the bond moment in Debye units. The bond moment for As-H, obtained in this manner, is identical with the observed bond moment. Likewise the values for P-Cl, As-F, Sb-I, H-Cl and others, agree very closely with the actual values. On the other hand, while C-O is 0.7, the bond moment from electronegativity is 1.0. C-Cl yields a bond moment of only 0.5 when computed according to the Pauling table when it is actually 1.5. Numerous metal-halogen moments exhibit the same marked difference, including that of Ti-Cl. Ti-O appears in the same category as C-O; a corrected value for this bond moment was used for the theoretical calculation of the Ti-Cl bond moment.

Hucken and Meyer (7) have analyzed the dipole constituents of a molecule and have taken into consideration the directions of operation of the constituents. Vectorial addition of the various linkages gave the standard bond moments for C-C, H-C, C-O, C-Cl, (the more positive element being placed first) which are used in dipole calculations. No attempt was made on their part to consider the probable altering effects on the bond moments by the environment in which the bond finds itself in the molecule. Such an inductive effect is evidenced in the substitution of a methyl radical for a hydrogen atom in the following compounds: CHCl_3 , CH_2Cl_2 , CH_3Cl . The resulting compounds, CH_3Cl_3 , CH_3CHCl_2 and $\text{CH}_3\text{CH}_2\text{Cl}$ show variation of dipole moment of 0.8, 0.5 and 0.2, from the values of CHCl_3 .

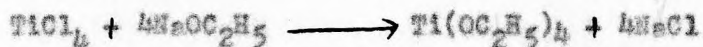
CH_2Cl_2 and CH_3Cl , which one would not predict on the basis of the substitution of a hydrogen atom by a methyl radical.

An attempt to use the same moment for a particular bond in different compounds by calculating quantitatively the charges induced by the other bonds in the molecule was made by Kemick (19). This author applies the generally accepted English electronic theory to the calculation of dipole moments and thereby obtains a more nearly valid result for theoretically calculated electric moments. So far, this procedure has not been used for many compounds but it has possibilities.

In general we can say that bond moments are at best only approximations of the actual physical situation; the computations which appear here are justified under that restriction.

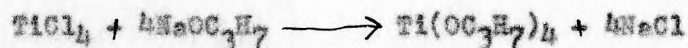
IV. PREPARATION OF COMPOUNDS USED

1. Titaniumtetraethylate, $Ti(OC_2H_5)_4$: This compound was prepared according to the method of Bischoff and Adkins (2) by the reaction:



The details of the preparation are described by Crowe (4). Upon standing titaniumtetraethylate changes to a pale yellow liquid which soon deepens in color eventually becoming an orange, red or brown liquid. Purification was accomplished by vacuum distillation, a little activated charcoal being added to remove the color. This procedure was found to be effective in obtaining a colorless, pure compound. An analysis for titanium by means of hydrolysis to titanium dioxide and ignition gave a titanium content of 21.10, 21.06 and 21.00%. The theoretical value is 21.05%.

2. Titaniumtetrapropylate, $Ti(OC_3H_7)_4$: This compound was prepared in a manner analogous to the method employed in the preparation of titaniumtetraethylate. A colorless, viscous liquid was obtained which did not hydrolyze as rapidly in air as did the titaniumtetraethylate; after a few weeks the compound turned a pale yellow color but did not become deeper in color. It is apparently a much more stable compound. The reaction is as follows:

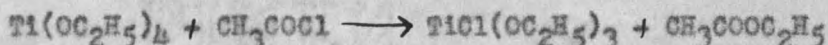


An analysis for the titanium content gave values of 16.85 and 16.53%. The theoretical value is 16.84.

3. Titaniumtetrabutylate, $Ti(OC_4H_9)_4$: It was possible to prepare this compound in a manner analogous to that used in preparing titanium-

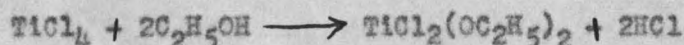
tetraethylate; in this case butyl alcohol was used. Found Ti: 14.06, 13.96%.
Theoretical Ti content: 14.08%.

4. Monochlorotriethoxytitanium, $TiCl(OC_2H_5)_3$: This compound was prepared according to the method of Jennings, Wardlaw and Way (13) by the reaction:



It maintained a pale yellow color even after repeated distillation and was kept in a sealed flask to prevent hydrolysis. A titanium analysis gave a titanium percentage of 21.77. The theoretical value is 21.97%. Cl found: 16.37, theoretical 16.22%.

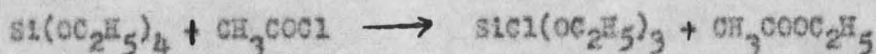
5. Dichlorodiethoxytitanium, $TiCl_2(OC_2H_5)_2$: 100 grams of $TiCl_4$ and 100 grams of absolute ethyl alcohol were refluxed according to the reaction (2):



At the end of twenty-four hours a white viscous liquid was obtained which was distilled under reduced pressure. A white sublimate distilled over and began to settle in the side arm of the distilling flask. The residue then gave off HCl, apparently decomposing in the process and no further distillate was obtained. An analysis of the yellowish-white liquid, which soon set to a solid, gave a titanium composition of 20.39%, whereas 22.91 is required. Apparently a very low pressure in the distillation process is necessary for purification. The electric moment of this material was not determined since it was considered impure.

6. Monochlorotriethoxysilicon, $SiCl(OC_2H_5)_3$: 30 cc. of acetyl

chloride was added dropwise to 110 cc of ethyl orthosilicate over a period of two hours, according to the method of Post and Horton (15).



The mixture was distilled and at 157°C at atmospheric pressure a colorless liquid came over and the temperature remained virtually constant during the distillation process. Monochlorotriethoxysilicon distills at 157-8°C at atmospheric pressure. The yield was very good; the compound has a pungent odor similar to ethyl orthosilicate and is soluble in hexane. The refractive index is 1.3857; the density 0.9271 at 25°C. The literature gives 1.0483 as the density of monochlorotriethoxysilicon at 0°C. No refractive index constant was found in available literature. Therefore the purity of this compound has not been established.

2. Ethyl Orthoformate, $\text{HC}(\text{OC}_2\text{H}_5)_3$: This compound was obtained from Eastman Kodak Company and employed without further purification. The refractive index of the compound was found to be 1.3923. The literature gives 1.3922 as the refractive index of ethyl orthoformate.

V. ELECTRIC MOMENT DETERMINATIONS

1. Method Used for Determination and Calculation of Electric Moment:

The apparatus used for the electric moment determinations was the same as previously described (4). The cell constant was calculated using distilled benzene, the dielectric constant of which was taken as 2.273 at 25°. Anhydrous benzene was prepared from Baker C. P. Thiophene-free benzene by drying over sodium for several hours and distilling directly from the flask, keeping only the middle third of the distillate. The anhydrous benzene was used almost immediately after preparation. Petroleum hexane was used as the solvent for the electric moment determinations and dried in the same manner. From a comparison of the cell capacitance with air as the dielectric and then with benzene, the cell constant could be calculated by the formula

$$C = \frac{C_x - C_a}{\epsilon_x + \epsilon_a}$$

The cell constant was checked using carbon tetrachloride, the dielectric constant of which is given as 2.2273 at 25°C in the Critical Tables. The results are shown in Tables I and II.

Table I. Calculation of Cell Constant

Trials No.	C _x	C _a	C	Ave.
1	70.16	34.75	27.82	
2	70.00	34.37	27.69	27.75

Table II. Determination of Dielectric Constant of CCl_4

Trial No.	C_x	C_a	x	x
1	68.84	34.83	27.75	2.2256
2	68.84	34.75	27.75	2.2281
3	68.74	34.72	27.75	2.2260

The total molar polarization was determined from dielectric constant and density measurements on dilute solutions of the compounds in hexane at 25°C. The dielectric constant and specific volume were found to be a linear function of the mole fraction of solute; thus the slopes and intercepts of the straight lines were obtained by using the method of least squares applied to the experimental points. These values were inserted into the equation of Halverstadt and Kuwler (10) which gives the total molar polarization of the compound:

$$P = \frac{\epsilon_1 - 1}{\epsilon_1 + 2} (M_2 v_1 + M_1 B) + \frac{3AV_1 M_1}{(\epsilon_1 + 2)^2}$$

in which ϵ_1 and v_1 are the intercepts of the dielectric constant and specific volume straight lines, respectively, and A and B are the respective slopes. M_1 is the molecular weight of the solvent and M_2 is the molecular weight of the solute. The identical procedure was used on all the compounds measured.

In order to calculate the electric moment it is necessary to calculate the electronic and atomic polarizations and subtract their sum from the total polarization to obtain P_m . Thus the dipole moment can be calculated from the Debye equation in the form: $\mu = 0.0127\sqrt{(P - P_e - P_a)}$ where μ is the dipole moment in Debye units, P is the total polarization and $P_e + P_a$ is

the sum of the electronic and atomic polarizations. The electronic polarization was measured in two different ways; (1) the density and refractive index of the pure compound were measured using an Oswald type pycnometer and a dipping refractometer; the values obtained were used for the calculation of the electronic polarization from the following formula of Lorentz (5):

$$P_e = \frac{n^2 - 1}{n^2 + 2} \cdot \frac{M}{d}$$

in which n is the refractive index, M the molecular weight, and d the density of the pure compound. The atomic polarization was assumed to be 5% of the electronic polarization and then added to the latter. (2) Several dilute solutions of the compound in hexane were made up and the refractive indices taken. A plot of the square of the refractive index against mole fraction of solute was a straight line. The values for the slope and intercept and this straight line were used in place of the ones for dielectric constant and inserted into the Halverstadt and Kuwler formula. The constants v_1 and B previously determined for the compounds were used again for this calculation. P_e for titaniumtetraethylate, monochlorotriethoxysilicon and ethyl orthoformate were determined by method (1). The electronic polarizations of titaniumtetrapropylate, titaniumtetrabutylate and monochlorotriethoxytitanium were determined by method (2).

The following tables and graphs show the experimental results used to calculate the values given in Table 17.

2. Titaniumtetraethylate. $Ti(OC_2H_5)_4$:

Table 3 (See Fig. 1)

Dielectric Constants of Solutions of $Ti(OC_2H_5)_4$ in Hexane

Solution No.	Mole Fraction	Dielectric Constant
1	0.004265	1.928
2	0.005091	1.929
3	0.008050	1.941
4	0.010560	1.946

Table 4 (See Fig. 2)

Specific Volumes of Solutions of $Ti(OC_2H_5)_4$ in Hexane

Solution No.	Mole Fraction	Dielectric Constant
1	0.004769	1.4579
2	0.007158	1.4492
3	0.016280	1.4360
4	0.007518	1.4491
5	0.002709	1.4575
6	0.017508	1.4334

2. Titaniumtetrpropylate, $Ti(OC_3H_7)_4$:

Table 5 (See Fig. 3)

Dielectric Constants of Solutions of $Ti(OC_3H_7)_4$ in Hexane

Solution No.	Mole Fraction	Dielectric Constant
1	0.006640	1.943
2	0.01050	1.952
3	0.01250	1.957
4	0.00564	1.940
5	0.01020	1.954
6	0.00251	1.933

Table 6 (See Fig. 4)

Specific Volumes of Solutions of $Ti(OC_3H_7)_4$ in Hexane

Solution No.	Mole Fraction	Specific Volume
1	0.002621	1.4618
2	0.007565	1.4537
3	0.01480	1.4371
4	0.009114	1.4502

Table 7 (See Fig. 5)

Refractive Indices of Solutions of $Ti(OC_3H_7)_4$ in Hexane

Solution No.	Mole Fraction	Refractive Index	Square of R.I.
1	0.003730	1.38517	1.92036
2	0.004652	1.38599	1.92066
3	0.01504	1.38954	1.93082
4	0.01439	1.38874	1.92860

3. Titaniumtetrabutylate, $Ti(OC_4H_9)_4$:

Table 8 (See Fig. 6)

Dielectric Constants of Solutions of $Ti(OC_4H_9)_4$ in Hexane

Solution No.	Mole Fraction	Dielectric Constant
1	0.004923	1.937
2	0.002479	1.929
3	0.002285	1.928
4	0.0003458	1.924

Table 9 (See Fig. 7)

Specific Volumes of Solutions of $Ti(OC_4H_9)_4$ in Hexane

Solution No.	Mole Fraction	Specific Volume
1	0.004923	1.4490
2	0.002489	1.4556
3	0.002289	1.4557
4	0.001497	1.4578

Table 10 (See Fig. 8)

Refractive Indices of Solutions of $Ti(OC_4H_9)_4$ in Hexane

Solution No.	Mole Fraction	Refractive Index	Square of R. I.
1	0.002360	1.38090	1.90688
2	0.005314	1.38195	1.90840
3	0.007660	1.38271	1.91189
4	0.009174	1.38309	1.91294

4. Monochlorotriethoxytitanium, $TiCl(O_2C_2H_5)_3$:

Table 11 (See Fig. 9)

Dielectric Constants of Solutions of $TiCl(O_2C_2H_5)_3$ in Hexane

Solution No.	Mole Fraction	Dielectric Constant
1	0.00781	1.978
2	0.00285	1.947
3	0.00373	1.952
4	0.00904	1.982
5	0.00820	1.979
6	0.00806	1.982
7	0.00623	1.964
8	0.01310	2.001
9	0.00646	1.964
10	0.00565	1.968

Table 12 (See Fig. 10)

Specific Volumes of Solutions of $TiCl(O_2C_2H_5)_3$ in Hexane

Solution No.	Mole Fraction	Specific Volume
1	0.00781	1.4486
2	0.00285	1.4589
3	0.00565	1.4543
4	0.01210	1.4400

5. Monochlorotriethoxysilicon, $\text{SiCl}(\text{OC}_2\text{H}_5)_3$:

Table 13 (See Fig. 11)

Dielectric Constants of Solutions of $\text{SiCl}(\text{OC}_2\text{H}_5)_3$ in Hexane

Solution No.	Mole Fraction	Dielectric Constant
1	0.009580	1.945
2	0.086774	2.104
3	0.062035	2.052
4	0.026490	1.977
5	0.058030	2.041

Table 14 (See Fig. 12)

Specific Volumes of Solutions of $\text{SiCl}(\text{OC}_2\text{H}_5)_3$ in Hexane

Solution No.	Mole Fraction	Specific Volume
1	0.009580	1.4546
2	0.030321	1.4382
3	0.058030	1.4193
4	0.086774	1.3950

6. Ethyl orthoformate, $\text{HC}(\text{OC}_2\text{H}_5)_3$:

Table 15 (See Fig. 13)

Dielectric Constants of Solutions of $\text{HC}(\text{OC}_2\text{H}_5)_3$ in Hexane

Solution No.	Mole Fraction	Dielectric Constant
1	0.18676	1.971
2	0.17689	1.969
3	0.20385	1.974

4	0.07068	1.941
5	0.33067	2.007

Table 16 (See Fig. 14)

Specific Volumes of Solutions of $\text{HC}(\text{OC}_2\text{H}_5)_3$ in Hexane

Solution No.	Mole Fraction	Specific Volume
1	0.18676	1.4523
2	0.17689	1.4535
3	0.20385	1.4485
4	0.07068	1.4623
5	0.04149	1.4649
6	0.35692	1.4420
7	0.42759	1.4383

Table 17

Summary of Electric Moment Determinations

Compound	A	ϵ_1	B	V_1	P	P_e	μ
$\text{Ti}(\text{OC}_2\text{H}_5)_4$	3.0684	1.9146	-1.6992	1.4612	119.3	64.4	1.58
$\text{Ti}(\text{OC}_3\text{H}_7)_4$	2.4882	1.9266	-2.0371	1.4681	118.4	82.3	1.32
$\text{Ti}(\text{OC}_4\text{H}_9)_4$	2.6444	1.9227	-2.5789	1.4617	129.7	102.0	1.04
$\text{TiCl}(\text{OC}_2\text{H}_5)_3$	7.1230	1.9210	-2.0815	1.4652	185.1	54.9	2.51
$\text{SiCl}(\text{OC}_2\text{H}_5)_3$	2.5957	1.9237	-0.7629	1.4620	116.7	50.3	1.75
$\text{HC}(\text{OC}_2\text{H}_5)_3$	0.02515	1.9237	-0.06378	1.4655	51.74	39.4	0.7

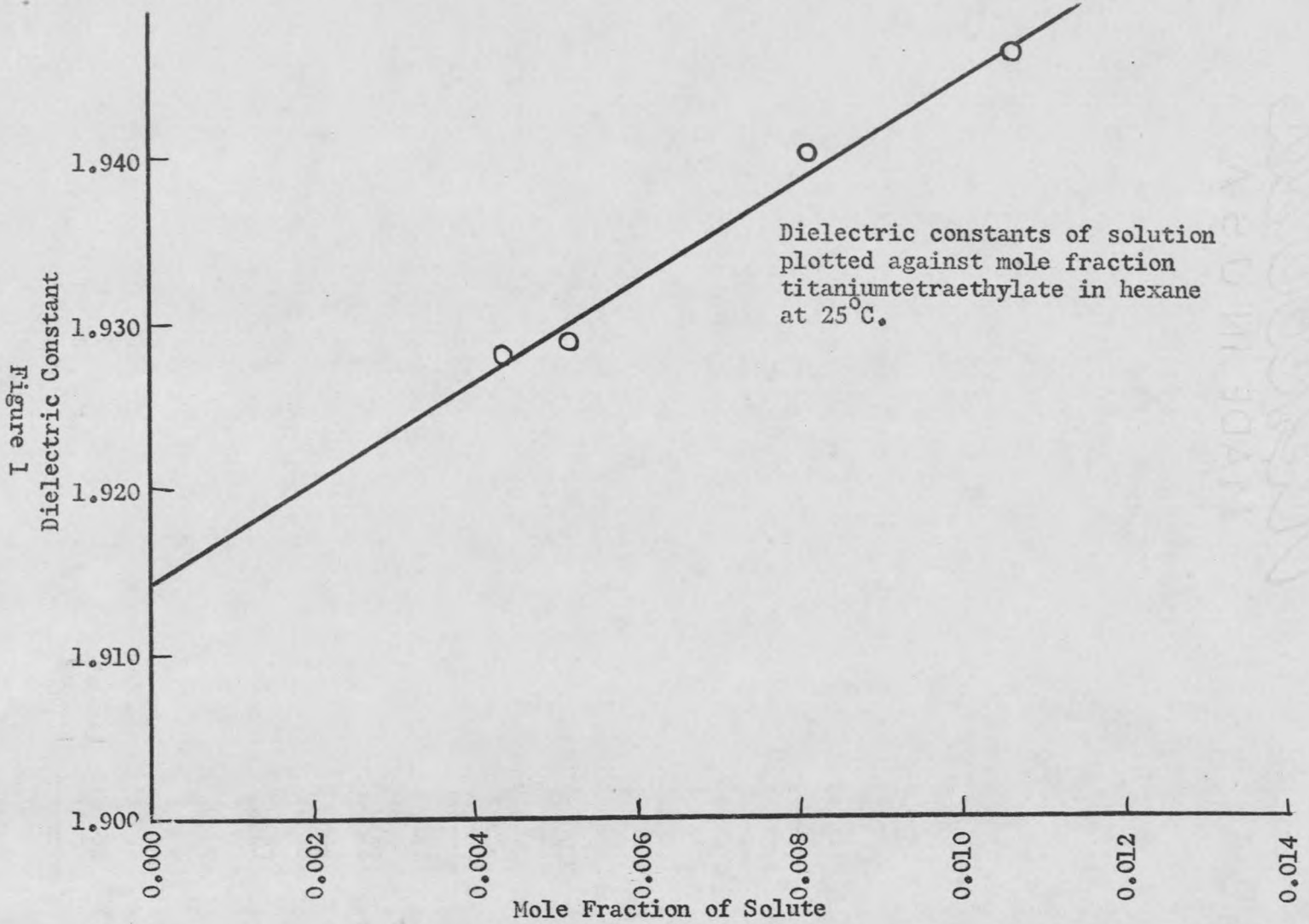
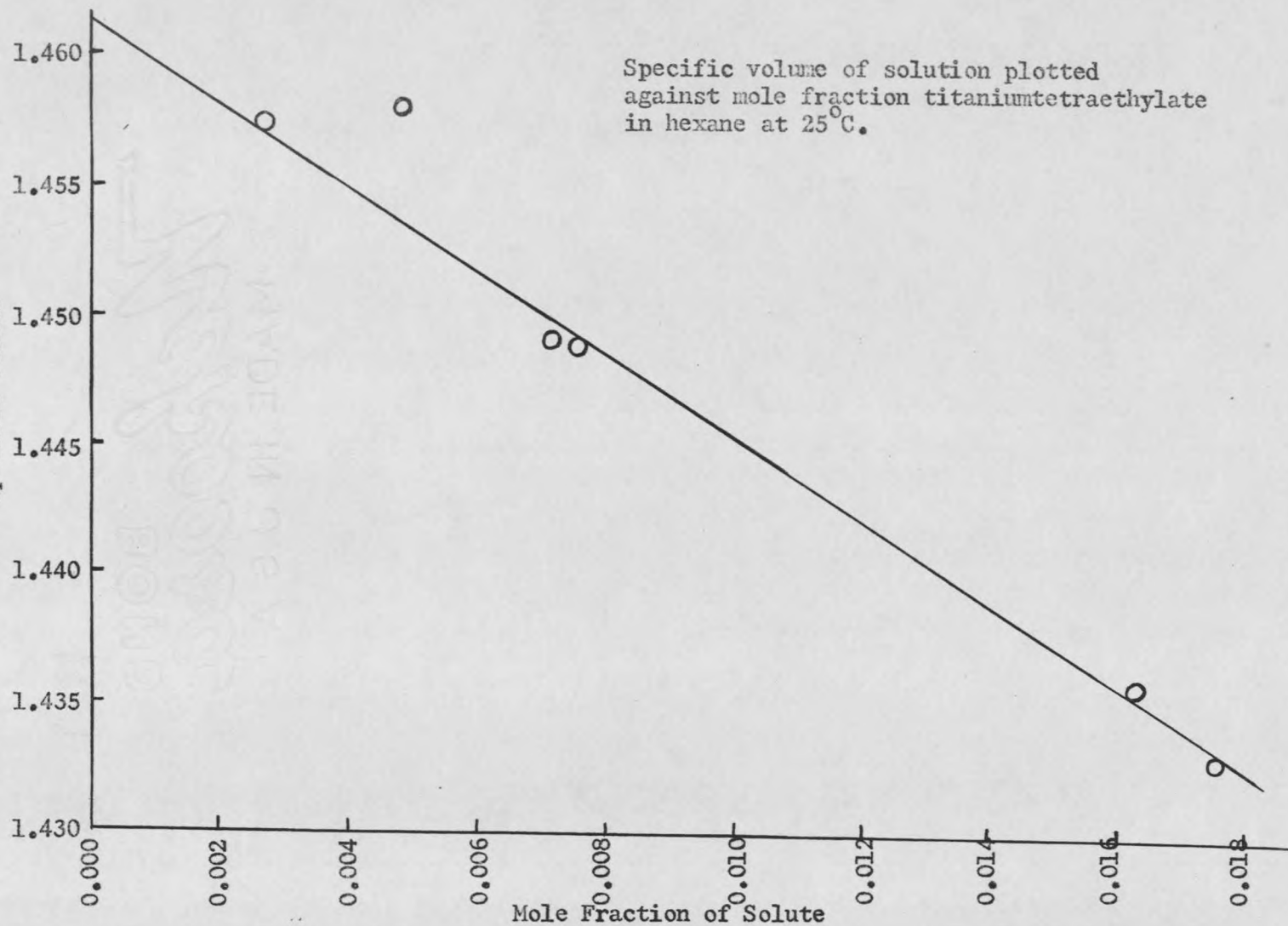
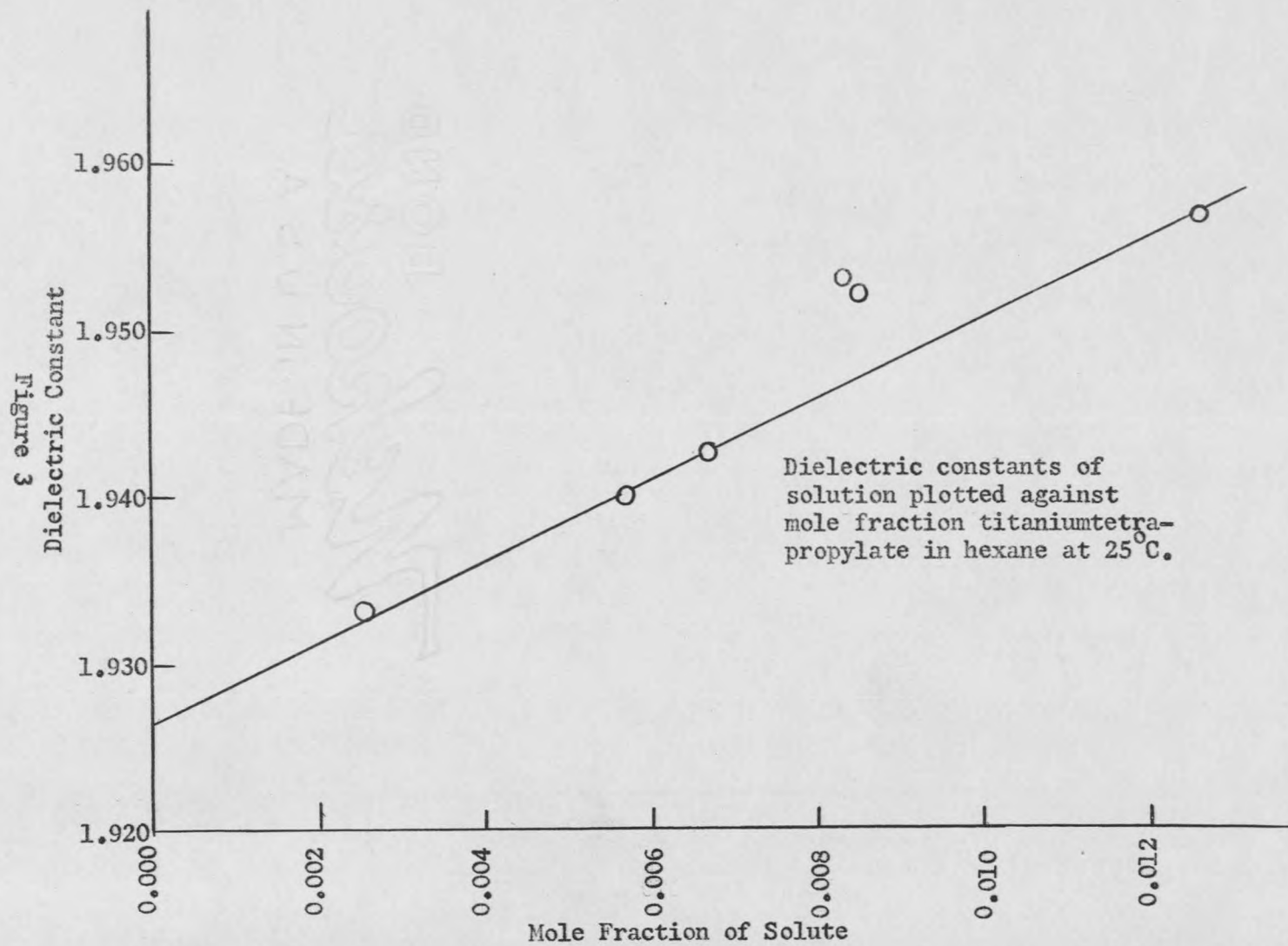


Figure 2
Specific Volume





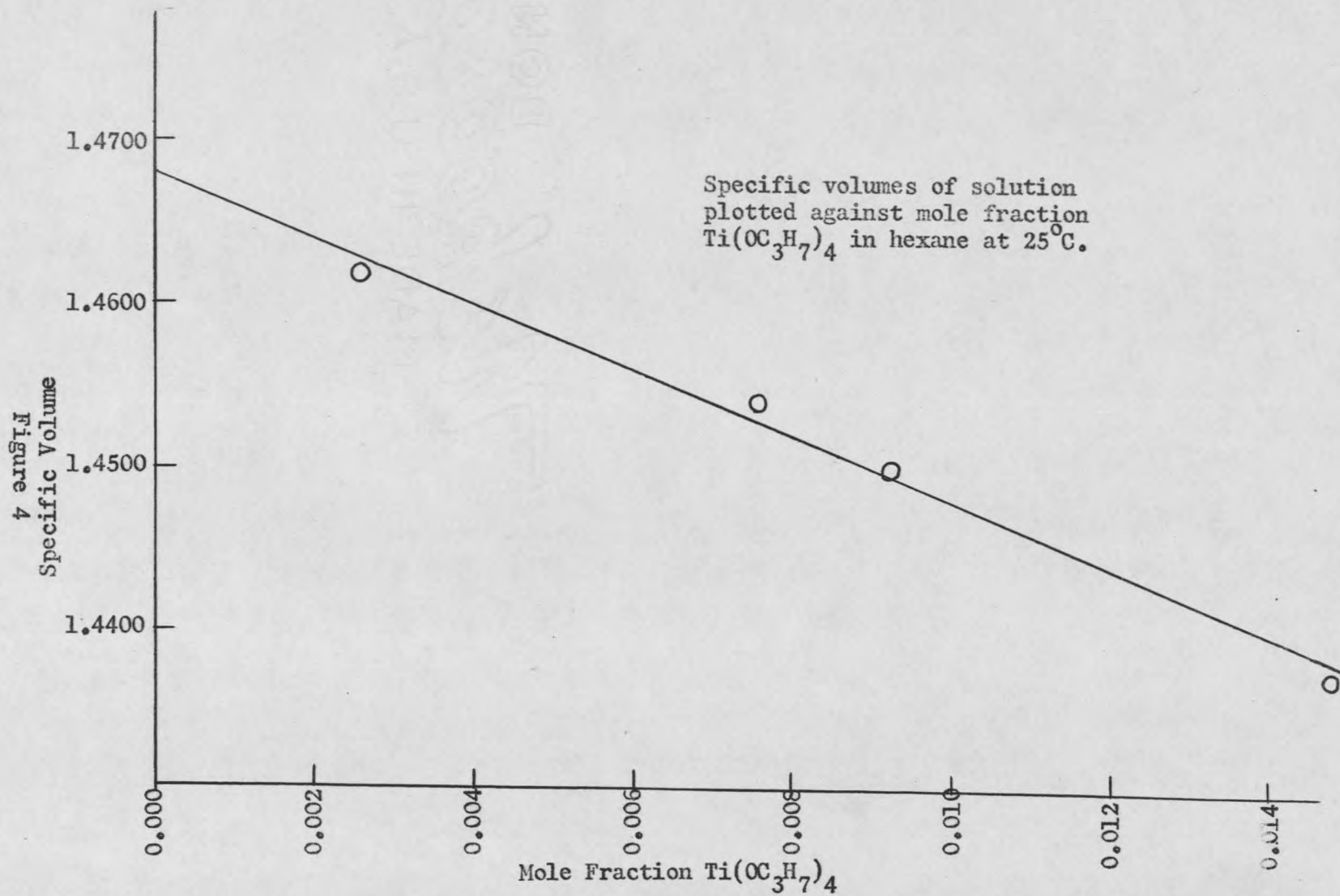


Figure 5

