



A study of the reaction between titanium tetrachloride and ethylene glycol : the dielectric constant of tetraethoxytitanium at radio frequencies and low temperatures, the specific conductivity of monochlorotriethoxytitanium

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A THESIS Submitted to the Graduate Faculty in partial fulfillment of the requirements for the degree of Master of Science in Chemistry

Montana State University

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Abstract:

Part I A slightly yellow, viscous, polymer-like compound was prepared by reacting ethylene glycol with titanium tetrachloride. A method of purification by precipitation of the compound from a solution in ethyl alcohol with dry diethyl ether was tried.

An analysis of the product of the reaction indicates the presence of 21.98 percent titanium, 16.53 percent chlorine, 5.34 percent hydrogen, 26.92 percent carbon and 29.23 percent oxygen. The empirical formulae calculated from these data are $TiClH_{12}C_5O_4$ based on one Ti atom, or $C_6H_{15}O_5$ based on 6 carbon atoms.

The molecular weight determined by the freezing point method is 30. This value is too low to be correct.

The conductivity of a solution of the product of the reaction between ethylene glycol and titanium tetrachloride was measured. A concentration of four grams of solute in 1000 grams of ethylene glycol exhibited a specific conductivity of 10.9×10^{-5} reciprocal ohms. The specific conductivity was found to decrease linearly with increasing dilution. The values obtained for the solution of the compound were compared with values for a solution of HCl and a solution of $TiCl(OC_2H_5)_3$ in glycol and found to be more like $TiCl(OC_2H_5)_3$.

This compound is decomposed rapidly by heat and slowly with time, with a subsequent loss of chlorine. A second compound which is similar in behavior was made by reacting monochlorotriethoxytitanium with ethylene glycol. Its properties were not tested since it was also decomposed by heat and with time.

The apparatus used for analysis is described and an attempt made to explain the experimental results.

Part II.

Tetraethoxytitanium has been prepared and purified. The behavior of the dielectric constant of the pure liquid was studied at frequencies ranging from 30 to 100 megacycles, and at temperatures from $-54^\circ C$ to $25^\circ C$.

The dielectric constant did not decrease with increasing frequency within the range of measurements, and, therefore does not exhibit anomalous dispersion at those temperatures and frequencies.

Monochlorotriethoxytitanium was prepared and an attempt made to measure the dielectric constant at 1000 cycles and $25^\circ C$. It was found, however, that monochlorotriethoxytitanium conducts electricity and the dielectric constant was not measurable with the equipment available.

The specific conductivity of pure monochlorotriethoxytlenium was measured at 25°C and found to be 1.34×10^{-6} mhos.

A discussion of the difficulties involved in the use of a Q meter is presented.

The apparatus used for measurements is described.

Part I. A STUDY OF THE REACTION BETWEEN TITANIUM TETRACHLORIDE AND
ETHYLENE GLYCOL

Part II. THE DIELECTRIC CONSTANT OF TETRAETHOXYTITANIUM AT RADIO FREQUENCIES
AND LOW TEMPERATURES. THE SPECIFIC CONDUCTIVITY OF MONOCHLOROTRI-
ETHOXYTITANIUM

By

WILLIAM HODGSON

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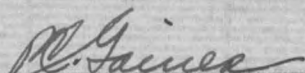
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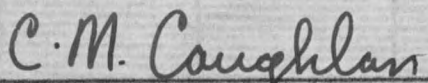
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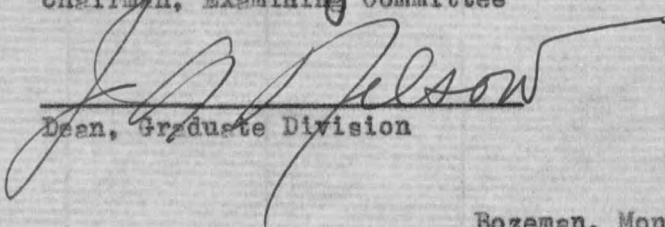
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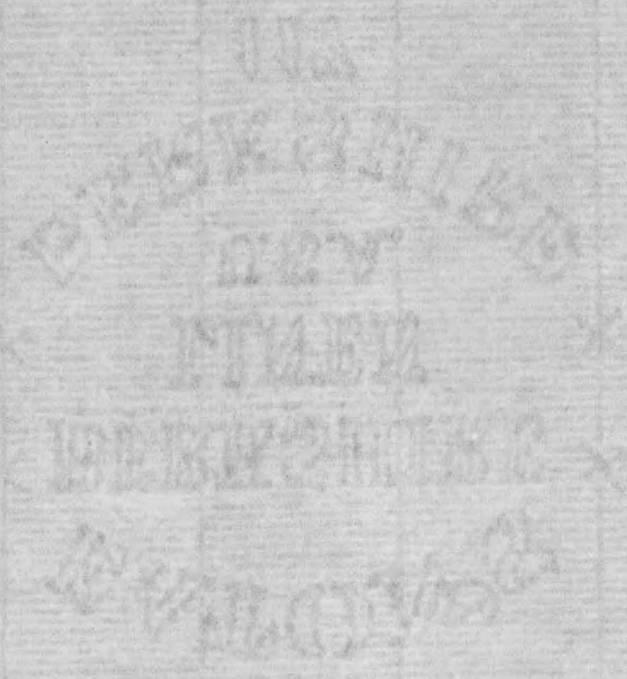
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Part I.

I. ABSTRACT

A slightly yellow, viscous, polymer-like compound was prepared by reacting ethylene glycol with titanium tetrachloride. A method of purification by precipitation of the compound from a solution in ethyl alcohol with dry diethyl ether was tried.

An analysis of the product of the reaction indicates the presence of 21.98 percent titanium, 16.53 percent chlorine, 5.34 percent hydrogen, 26.92 percent carbon and 29.23 percent oxygen. The empirical formulae calculated from these data are $TiClH_{12}C_5O_4$ based on one Ti atom, or $C_6H_{15}O_5TiCl$ based on 6 carbon atoms.

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