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A Study on the Formation of Molecular Complex between Benzophenone and Indole

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〈Abstract〉

Solid-liquid phase equilibria were used to determine the presence of a molecular complex between Benzophenone and Indole system. The phase diagram of the system yield a pattern indicating the formation of an unstable complex. The infrared spectra of the 1 : 1 mixture of Benzophenone and Indole also indicated that hydrogen bonding is the reason for the complex formation.

Benzophenone과 Indole 사이의 착물 형성에 관한 연구

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〈요 약〉

고체-액체 상평형 그림으로 Benzophenone과 Indole 사이의 착물 형성 여부를 확인하였다. 이들 두 화합물 사이에는 불안정한 1 : 1 착물이 형성됨을 알았다. 또한 두 화합물의 1 : 1 혼합물의 적외선 흡수 스펙트럼은 착물 형성이 수소결합에 의한 것임을 나타내었다.

I. Introduction

It is difficult to define the term molecular complex because its usage has been so varied. It is described as the product of a weak reversible interaction between two or more components in general.

In this paper the term will be taken to mean the formation of a substance by the interaction of two components, which may have independant crystal structures. Then, such a molecular complex will reversely dissociate into its components on dissolution. The existence of these complexes is readily determined by using solid-liquid phase equilibria.

Landauer and McConnell[1] used ultraviolet spectroscopy to study the complex formation and attributed the complex to a donor-acceptor phenomenon but have ignored the distinct possibility of hydrogen bonding. Andrews and keefer[2] have studied that complex formation can be detected by infrared spectra. The bond length of complexes increases which occur are generally accompanied by a decrease in vibrational frequencies of the individual components. The presence of these phenomena was taken as evidence for the existence of a complex.

Therefore the purpose of this investigation was to inspect the phase diagram and infrared absorption spectra of the Benzophenone—Indole system in order to determine the presence or absence of complexes which form are due to a charge transfer process or hydrogen bonding.

Ⅱ. Experimental

All of the compounds used in this study were commercially available, TCI analytical grade Benzophenone (Diphenyl ketone) and Baker analyzed reagent grade Indole (Benzopyrrole) were used after recrystallization from ethanol and dryied in a vacuum oven. Melting points and infrared spectra were used to check the purity of materials. Reagents were considered acceptable for use when melting points agree to within 1.0°C of the literature values[3] and the i.r. spectra showed no interference peaks. Infrared spectra were recorded on the Shimadzu IR-430 Infrared Spectrophotometer.

Experimental method used to construct the phase equilibria diagram is transition point method which is same as the one described by Thorne et al.[4] It consisted of using the inner tube assembly from the Beckman apparatus and heating the contents untill all solid had melted. The transition point is taken as the temperature at which the last amount of solid had disappeared. In all instances, temperature measurements should be accurate to with in 0.5°C.

The phase diagram are constructed by plotting the mole fraction of Benzophenone vs. the transition point temperatures for the mixture under study. With the construction of the solid-liquid equilibria phase diagram the presence of a complex is verified by the presence of a peritectic point.

Ⅲ. Results and Discussion

The system of this investigation were studied over a wide range of mole fractions. A solid-liquid phase equilibria diagram obtained for the Benzophenone and Indole system is presented on Fig. 1, respectively. The phase diagram for the Benzophenone—Indole system indicates that a peritectic point is observed. This showed that

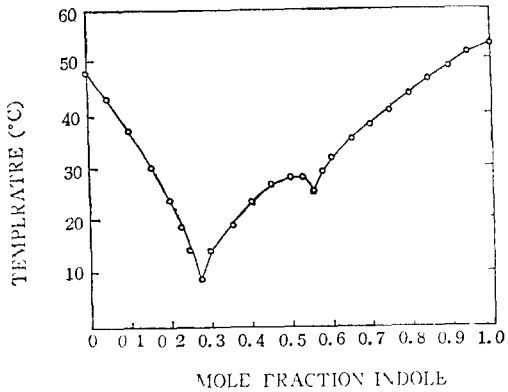

Fig. 1. Phase Diagram for the Benzophenone-Indole System.

a weak bonded unstable complex is formed. The peritectic point occurs at 50 mole% Benzophenone indicating the presence of 1 : 1 complex. The fact showing weak compound formation leads one to believe that hydrogen bonding is a very real possibility.

The eutectic compositions and eutectic temperature obtained in this study are 27mole% 8°C and 55mole% 28°C for the Benzophemone-Indole system. The infrared absorption spectra of the complex in liquid thin film indicate that complex formation does take place[5] (Fig. 2). Examination of the spectra of pure Benzophenone and Indole shows a large number of absorption bands over the range of the infrared spectrum. The majority of these bands are in the finger print region of the spectrum and this makes conclusion about the spectral shift somewhat difficult. However, the N-H stretch region of the Indole does provide a proper point of reference. There are two reasons for using this band as the reference point. First, the absorption is a well-defined singlet peak with strong intensity in an area where there are no interferences from the Benzophenones spectra. Secondly, this is the actual site of the hydrogen bonding with in the complex and it would seem logical that the absorption shift and intensity reduction can be pronounced at this absorption frequency.

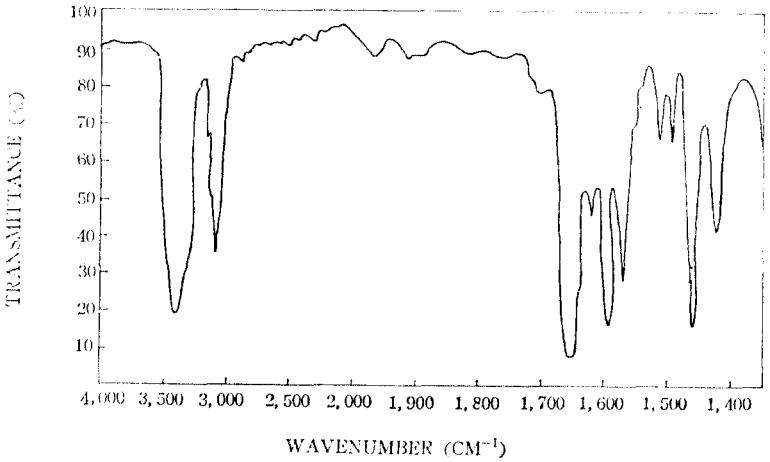

Fig. 2. IR Spectra of the 1 : 1 Mixture of Benzophenone and Indole.

It was observed that the N-H stretching absorption of Indole shifted to a lower wave number (from 3420 to 3340) and a decrease occured in intensity. This indicates that there is complex formation and such complexing may be due to hydrogen bonding.

Ⅳ. Conclusions

The phase diagram of the system involving Benzophenone and Indole showed a pattern indicating the formation of an unstable complex of some type. The infrared spectra of the 1 : 1 mixture of Benzophenone with Indole indicated that hydrogen bonding is the reason for the complex formation, particulary because of the absorption shift of the infrared band of the Indole N-H bond to lower wave number.

The results of this study indicate that while complex formation take places beween Benzophenone and Indole and the complex formed is due to hydrogen bonding.

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