[Note]

Equilibrium Constants for the Dissociation of Thiourea Adducts of Dialkylnaphthalenes

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Various 2,6-dialkylnaphthalenes, 2,3-dimethylnaphthalene, and 2-methyl-7-*t*-butylnaphthalene adducts with thiourea were formed to determine the equilibrium constants at temperatures of 273.2 to 303.2 K. No adducts of thiourea with 2,6-dimethylnaphthalene, 2-methyl-6-ethylnaphthalene, and 2-methyl-6-isopropyl-naphthalene were detected at these temperatures. Equilibrium constants of dialkylnaphthalene–thiourea systems, except for the 2-methyl-7-*t*-butylnaphthalene–thiourea system, were greater than those of the hexadecane–urea system and less than those of isoparaffin– or naphthene–thiourea systems, indicating that stability of dialkylnaphthalene–thiourea adducts is relatively high, whereas *n*-paraffin–urea adducts are more stable than thiourea adducts with various guest molecules. The order of adduct formation was as follows: 2,6-di-*t*-butylnaphthalene > 2,3-dimethylnaphthalene > 2,6-diisopropylnaphthalene ~ 2,6-diethylnaphthalene > 2-methyl-6-*t*-butylnaphthalene > 2-methyl-7-*t*-butylnaphthalene. The molar ratios of thiourea to substrate were in the range of 5.0 to 6.7, depending on the size of the dialkylnaphthalenes or bulkiness of the alkyl groups.

The formation of the thiourea adducts was exothermic and were favored at lower temperatures. The heats of formation ($-\Delta H_G$) were between 28 and 37 kJ mol⁻¹, which are of the same order of magnitude as those of other thiourea adducts with branched paraffins or cycloparaffins. Deviations were somewhat large, although the values of ($-\Delta H_G$) were correlated with *m*, suggesting that the structure of thiourea adducts is more flexible than that of urea adducts.

1. Introduction

Thiourea forms channel adducts with highly branched paraffins, cycloparaffins and alkylaromatics^{1)–5)}. The alkylaromatic adduct crystals form only in the presence of suitable guest molecules such as 2,6-dialkylnaphthalenes (*i.e.*, 2,6-diisopropyl-, 2,6-di-*t*-butylnaphthalene), 2,3-dimethylnaphthalene and 2,3,6-trimethylnaphthalene³⁾. These results suggest that thiourea addition under suitable conditions may separate alkylnaphthalenes of specific structures from mixtures. Both 2,3- and 2,6-dialkylnaphthalenes are potential feed stocks for speciality high-performance polymers and liquid crystals. Application of thiourea adducts to the separation process requires fundamental information such as equilibrium constants and enthalpy changes for the adduct formation. The composition of the adducts (molar ratio of thiourea to substrate) is also important to design an efficient separation process. However, no systematic studies are available except those in our laboratory on the adduct formation of dialkylnaphthalenes with thiourea^{6)–11)}. Our previous work investigated the equilibrium constants and compositions of thiourea adducts with 2,6-diethyl-, and 2,6-

diisopropylnaphthalene⁷⁾.

In the present study, equilibrium constants for thiourea adducts of various dialkylnaphthalenes, *i.e.*, 2,6-di-*t*-butylnaphthalene, 2,3-dimethylnaphthalene, 2-methyl-6-, and 2-methyl-7-*t*-butylnaphthalenes were determined to elucidate adduct formation between dialkylnaphthalenes and thiourea. Heats of formation and compositions of the adducts were also measured.

2. Experimental

2.1. Substrates and Reagents

A mixture consisting of di-*t*-butylnaphthalene (97.0 wt%), 2-*t*-butylnaphthalene (2.0 wt%), and others (1.0 wt%) was obtained from the *t*-butylation products of naphthalene by a conventional distillation method. A mixture of 2-methyl-6-*t*-butylnaphthalene (50.9 wt%), 2-methyl-7-*t*-butylnaphthalene (49.1 wt%) was also synthesized by *t*-butylation of 2-methylnaphthalene. 2,6-Di-*t*-butylnaphthalene and 2-methyl-6-*t*-butylnaphthalene were isolated from these mixtures by repeated thiourea adduct formation. 2-Methyl-7-*t*-butylnaphthalene was crystallized at 15°C from a methanol solution of the methyl-*t*-butylnaphthalenes mixture. The purity of these isolated dialkylnaphthalene substrates were better than 99 wt% by GC analysis.

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Table 1 Equilibrium Constants for the Dissociation of Thiourea Adducts of Dialkyl-naphthalenes

Temperature [K]	2,6-DEN-TU ^{a),7)}	2,6-DIPN-TU ^{b),7)}	2,6-DtBN-TU ^{c)}	2M6tBN-TU ^{d)}	2M7tBN ^{e)}	2,3-DMN-TU ^{f)}
273.2	0.0156	0.0138	0.00428	0.0176	0.0415	0.0098
283.2	0.0219	0.0273	0.00617	—	—	0.0143
293.2	0.0335	0.0400	0.00813	0.0302	0.0523	0.0221
303.2	0.0518	0.0685	0.00428	—	—	0.0348

a) 2,6-diethylnaphthalene-thiourea adduct. b) 2,6-diisopropylnaphthalene-thiourea adduct. c) 2,6-di-*t*-butylnaphthalene-thiourea adduct. d) 2-methyl-6-*t*-butylnaphthalene-thiourea adduct. e) 2-methyl-7-*t*-butylnaphthalene-thiourea adduct. f) 2,3-dimethylnaphthalene-thiourea adduct.

2,3-Dimethylnaphthalene (98 wt%) extra pure grade reagent was obtained from Tokyo Chemical Industry Co. and used without further purification. Thiourea, methanol, heptane, and diethyl ether extra pure grade reagents (Nacalai Tesque, Inc.) were dried to remove trace amounts of water before use.

2. 2. Procedure and Analysis

The non-aqueous solution method^{7),12)} was adapted to measure the equilibrium constant for thiourea adduction. A mixture of the substrate (dialkyl-naphthalene, 0.1 g) and heptane solvent (0.9 g) was charged into a reaction vessel made of Pyrex glass (20 ml in volume) equipped with a sampling port. Excess thiourea (0.4–0.5 g) moisturized with methanol (100 μ l) was added to the vessel. The vessel was kept at the desired temperature between 273.2 and 303.2 K with periodic monitoring the substrate concentration. When the reaction had nearly reached equilibrium, thiourea and/or a small amount of methanol was additionally charged into the system to confirm the establishment of the equilibrium. The reaction was regarded to be in equilibrium when no change in substrate concentration was observed after this addition. Concentrations of the substrates were analyzed by gas chromatography using 3.2 mm (dia.) $\times$ 1.0 m column packed with OV-1.

The composition of the adduct, *m* (molar ratio of thiourea to the substrate), was determined by elemental analysis using a CHN coder (MT-3, Yanagimoto Inc.).

3. Results and Discussion

3. 1. Equilibrium Constant

The equilibrium constant, *K*, for the dissociation reaction of the thiourea adduct,

is given by Eq. (2)¹³⁾,

$$K = (a_{\text{DAN}})(a_{\text{TU}})^m / (a_{\text{ADD}}) \quad (2)$$

where *a*_{DAN}, *a*_{ADD}, and *a*_{TU} are the activities of the substrate in solution, the activity of the adduct, and the activity of thiourea, respectively. Assuming an ideal solution Eq. (2) can be simplified since thiourea and the adduct are in the solid state. Thus, *K* is expressed simply by the molar fraction of the substrate in the solution^{7),21)}

$$K = X_{\text{DAN}} \quad (3)$$

a) 2,6-diethylnaphthalene-thiourea adduct. b) 2,6-diisopropylnaphthalene-thiourea adduct. c) 2,6-di-*t*-butylnaphthalene-thiourea adduct. d) 2,3-dimethylnaphthalene-thiourea adduct. e) 2-methyl-6-*t*-butylnaphthalene-thiourea adduct. f) 2-methyl-7-*t*-butylnaphthalene-thiourea adduct. g) cyclohexane-thiourea adduct. h) hexadecane-urea adduct.

Fig. 1 Equilibrium Constants for Adduct Decomposition

The values of equilibrium constants for the dissociation of 2,6-di-*t*-butylnaphthalene, 2-methyl-6-*t*-butylnaphthalene, 2-methyl-7-*t*-butylnaphthalene, and 2,3-dimethylnaphthalene thiourea adducts are listed in Table 1. Figure 1 shows the equilibrium constants for the decomposition of the thiourea adducts as a function of reciprocal temperature. Literature values for 2,6-diethylnaphthalene, 2,6-diisopropylnaphthalene⁷⁾, and cyclohexane-thiourea adduct²⁾ are also plotted for comparison. Equilibrium constants for the hexadecane-urea system¹³⁾, which forms one of the most stable urea adducts, are also included. The values of the equilibrium constants found in this study, except for 2-methyl-7-*t*-butylnaphthalene-thiourea system, are lower than those of the cyclohexane-thiourea system extrapolated to the temperature range of the present study (0–30°C), and one order of magnitude greater than those of the hexadecane-urea system. The equilibrium constants obtained in the present study are also lower than those for branched paraffin-thiourea systems¹⁵⁾ as well

Table 2 Compositions of the Thiourea Adducts

	<i>m</i>	Ref.
2,6-DEN-TU ^{a)}	6.0	6)
2,6-DIPN-TU ^{b)}	6.0	6)
2,6-DtBN-TU ^{c)}	6.7	this work
2M6tBN-TU ^{d)}	6.7	this work
2,3-DMN-TU ^{e)}	5.0	this work

a) 2,6-diethylnaphthalene-thiourea adduct. b) 2,6-diisopropylnaphthalene-thiourea adduct. c) 2,6-di-*t*-butylnaphthalene-thiourea adduct. d) 2-methyl-6-*t*-butylnaphthalene-thiourea adduct. e) 2,3-dimethylnaphthalene-thiourea adduct.

m is molar ratio of thiourea to substrate.

as cycloparaffin-thiourea systems¹⁵⁾, indicating that the combination of dialkylnaphthalenes and thiourea is favorable for the formation of stable thiourea adducts.

Judging from the values of the equilibrium constants, the order of adduct forming potential is as follows: 2,6-Di-*t*-butylnaphthalene > 2,3-dimethylnaphthalene > 2,6-diisopropylnaphthalene ~ 2,6-diethylnaphthalene > 2-methyl-6-*t*-butylnaphthalene > 2-methyl-7-*t*-butylnaphthalene. It is interesting to note that the *t*-butyl group at the β -position of naphthalene seems to enhance the formation of the adduct. Our preliminary experiment shows that 2,6-dimethylnaphthalene, 2-methyl-6-ethylnaphthalene, and 2-methyl-6-isopropylnaphthalene do not form the thiourea adduct at the temperatures used in this study, whereas the equilibrium constants for the 2-methyl-6-*t*-butylnaphthalene system are similar to those for the 2,6-diethyl and 2,6-diisopropylnaphthalene systems.

3. 2. Compositions of Thiourea Adducts

Analysis of molar ratio of reagent (host) to substrate (guest) of the adducts, *m*, can be achieved by several methods^{1),6),13),14)}. The thiourea (or urea) content of the adducts can be obtained from elemental analysis. The amount of guest component may be established by the weight loss upon dissociation of the adduct as well as elemental analysis. Measurement of concentration changes in the reaction liquid during adduct formation is also applicable to certain systems. Considering the potential errors inherent in these procedures, the elemental analysis method was adopted in the present study. The values of the ratio, *m*, obtained from the contents of carbon, hydrogen, nitrogen, and sulfur (diff.) in the thiourea adducts are listed in Table 2.

The values of *m* are 5.0 to 6.7, depending on the size of guest molecules. The less compact dialkylnaphthalenes with bulky alkyl groups are associated with more thiourea in the adduct than are compact dialkylnaphthalenes. Interaction between the guest molecules with bulky alkyl groups and thiourea molecules is greater in comparison with compact guest molecules. The fit of the alkyl groups in the guest molecules with the channels of the adduct to make close contact with

a) 2,2-dimethylbutane-thiourea¹⁵⁾. b) 2,2,3-trimethylbutane-thiourea¹⁵⁾. c) 2,2,4-trimethylpentane-thiourea¹⁵⁾. d) cyclopentane-thiourea¹⁵⁾. e) cyclohexane-thiourea¹⁵⁾. f) isopropylcyclohexane-thiourea¹⁵⁾. g) 2,6-diethylnaphthalene-thiourea⁷⁾. h) 2,6-diisopropylnaphthalene-thiourea⁷⁾. i) 2,6-di-*t*-butylnaphthalene-thiourea. j) 2-methyl-6-*t*-butylnaphthalene-thiourea. k) 2,3-dimethylnaphthalene-thiourea. l) $-\Delta H_G$ for *n*-paraffin-urea¹⁵⁾.

Fig. 2 Relationship between Heat of Formation of Adduct and Composition Ratio *m*

the rugged surfaces of the thiourea channels may be important. The size or the degree of branching in the alkyl groups, especially the *t*-butyl group at the β -position of naphthalene, apparently has a significant effect on the stability of the thiourea adduct.

3. 3. Heats of Formation of Thiourea Adduct

The negative slope of the plots in Fig. 1 shows that the formation of thiourea adducts is exothermic and that lower temperatures are favorable to adduct formation. As shown in the Fig. 1, the log *K* values in this study change linearly with the reciprocal temperatures. The heats of formation, $-\Delta H_G$ (substrate basis), were calculated using the van't Hoff equation, and the results are plotted in Fig. 2 as a function of ratio *m* in 3. 2. Literature values^{7),15)} are included for comparison.

The $-\Delta H_G$ value is well correlated with *m* in the case of *n*-paraffin-urea adducts (the bold line in Fig. 2). Figure 2 shows that the values of $-\Delta H_G$ for dialkylnaphthalene-thiourea system fall in the same range obtained for isoparaffins, cycloparaffin-thiourea systems¹⁵⁾ and *n*-paraffin-urea systems¹⁵⁾, and that the heats of thiourea adduct formation are also correlated with *m* although the slope is a little different and the deviations are somewhat large (the dotted line for $-\Delta H_G = 5.67m - 3.45$, $r^2 = 0.634$ in Fig. 2 was obtained using the least squares method). These results suggest that the structure of thiourea adducts is more flexible than that of urea adducts. Various types of guest molecules can be incorporated in the adduct of

Table 3 Standard Free Energy Change and Standard Entropy Change for Adduct Formation

	$-\Delta G^0$ [kJ (mol of guest) ⁻¹]	$-\Delta S^0$ [JK ⁻¹ (mol of guest) ⁻¹]	S^0_{sol} [JK ⁻¹ (mol of guest) ⁻¹]	S^0_{add} [JK ⁻¹ (mol of guest) ⁻¹]
2,6-DEN-TU ^{a)}	-7.9 ⁷⁾	-67 ⁷⁾	550	483
2,6-DIPN-TU ^{b)}	-7.3 ⁷⁾	-99 ⁷⁾	540	441
2,6-DtBN-TU ^{c)}	-10.4	-61	697	636
2M6tBN-TU ^{d)}	-6.8	-91	575	484
2M7tBN-TU ^{e)}	-6.5	-3	578	575
2,3-DMN-TU ^{f)}	-8.8	-68	474	406
CH-TU ^{g)}	-6.9 ²⁾	-21 ²⁾	345	324
HD-U ^{h)}	-14.9 ¹³⁾	-225 ¹³⁾	862	637

a) 2,6-diethylnaphthalene-thiourea adduct. b) 2,6-diisopropylnaphthalene-thiourea adduct. c) 2,6-di-*t*-butylnaphthalene-thiourea adduct. d) 2-methyl-6-*t*-butylnaphthalene-thiourea adduct. e) 2-methyl-7-*t*-butylnaphthalene-thiourea adduct. f) 2,3-dimethylnaphthalene-thiourea adduct. g) cyclohexane-thiourea adduct. h) hexadecane-urea adduct.

thiourea. Changes in the structural features of the adducts, *e.g.*, the crystal system, cell parameters, and the shapes of the adduct channels depend on the guest molecules^{16)~19)}. Thus, the structural variety of thiourea adducts is associated with the low correlation between $-\Delta H_G$ and m . In contrast, the linear relationship between $-\Delta H_G$ and m in *n*-paraffin-urea adducts reflects the rigid structure of the urea adduct and the strictly limited shape and dimensions of the urea adduct channels^{20),21)}.

Standard free energy change ($-\Delta G^0$) and standard entropy change ($-\Delta S^0$) for adduct formation are listed in **Table 3**. The values of ($-\Delta S^0 = -(S^0_{\text{sol}} - S^0_{\text{add}})$) reflect the difference in the randomness of the guest molecules in the solution and in the channels of the adduct. The values of S^0_{sol} can be evaluated from the standard entropy of the guest molecules estimated by Benson's method²²⁾ and the entropy of mixing of the guest molecules with the solvents assuming an ideal solution. As S^0_{add} corresponds to randomness of the adduct, the regularity of the guest molecules in the adducts may be evaluated through S^0_{add} . The values of S^0_{add} for dialkylnaphthalene-thiourea system fall between those for the cyclohexane-thiourea system and hexadecane-urea system. This indicates that the regularity of the dialkylnaphthalene molecules in the channel is inferior to that of cyclohexane-thiourea system, assuming that the regularity of thiourea in the adducts does not differ significantly. The regularity of cyclohexane in the thiourea adduct was studied by the X-ray diffraction method. Both molecular structure and crystal structure of cyclohexane in the adduct were not refined, but those of thiourea in the adduct were determined, suggesting that molecules of cyclohexane were included as disordered structures in the channel of the thiourea adduct^{16),23)}. As mentioned in **3. 1.**, dialkylnaphthalenes of specific structures form stable adducts with thiourea. However, moderate values of $-\Delta S^0$ or S^0_{add} for thiourea-dialkylnaphthalenes suggest that the shape and/or size of the substrates may match modestly with the shape and/or size of the channels formed by

thiourea. Dialkylnaphthalenes are embedded in the distorted hexagonal channel of thiourea molecules⁶⁾. The naphthalene ring is like a plate, so some empty spaces can be expected between the naphthalene ring and the walls of the channels when such plate-like guest molecules are included. The void space in the channel of the thiourea adduct may be related to the randomness of dialkylnaphthalenes in the thiourea adducts.

4. Conclusion

The fundamental characteristics of the separation of 2,6- and 2,3-dialkylnaphthalenes by thiourea adduct formation were studied by the measurement of the equilibrium constants and heats of adduct formation as well as the compositions of the adducts. Equilibrium constants for the decomposition of the thiourea adduct of dialkylnaphthalenes are higher than those for the urea adduct of hexadecane and lower than those for thiourea adducts of isoparaffins and naphthenes. The order of adduct forming ability is as follows: 2,6-Di-*t*-butylnaphthalene > 2,3-dimethylnaphthalene > 2,6-diisopropylnaphthalene ~ 2,6-diethylnaphthalene > 2-methyl-6-*t*-butylnaphthalene > 2-methyl-7-*t*-butylnaphthalene. The heats of adduct formation are between 28 and 37 kJ (mol dialkylnaphthalene)⁻¹ and are higher than those of other thiourea adducts. Compositions of the adducts have a host : guest ratio of 5.0 to 6.7 according to the compactness of the dialkylnaphthalenes.

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要 旨

ジアルキルナフタレン類のチオ尿素付加物の解離平衡定数

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2,3-ジメチルナフタレン, 2-メチル-7-*t*-ブチルナフタレンおよび種々の2,6-ジアルキルナフタレンのチオ尿素付加反応を273.2~303.2 Kの温度範囲で行い, 平衡定数を測定した。2,6-ジメチルナフタレン, 2-メチル-6-エチルナフタレンおよび2-メチル-6-イソプロピルナフタレンはこの温度範囲においては付加物を形成しなかった。2-メチル-7-*t*-ブチルナフタレンを除き, 本研究で得られたジアルキルナフタレン類のチオ尿素付加物分解反応の平衡定数は, シクロヘキサン-チオ尿素付加物の値よりも小さく, ヘキサデカン-チオ尿素付加物の値より大きかった。このことは, ジアルキルナフタレン-チオ尿素付加物の安定性がチオ尿素付加物の中では相対的に高く, 代表的な*n*-パラフィン-チオ尿素付加物のそれより低いことを示している。付加物形成能の序列は2,6-ジ-*t*-ブチルナフタレン>2,3-ジメチルナ

フタレン>2,6-ジイソプロピルナフタレン~2,6-ジエチルナフタレン>2-メチル-6-*t*-ブチルナフタレン>2-メチル-7-*t*-ブチルナフタレンであった。また, 付加物組成*m*(付加物を構成するゲスト分子に対するチオ尿素分子の比)は5.0~6.7の範囲にあり, 包接される分子の大きさ, アルキル基のかさ高さに依存した。

チオ尿素付加物の形成は発熱反応であり, 低温ほど有利であった。ジアルキルナフタレン類のチオ尿素付加物の生成熱は28~37 kJ mol⁻¹であり, イソパラフィンあるいはシクロパラフィン類のチオ尿素付加物の値と同程度であった。生成熱は付加物組成*m*と弱い相関を示すことから, チオ尿素付加物の構造の多様性が示唆された。

Keywords

Thiourea adduct, Separation, Equilibrium constant, Dialkyl naphthalenes