(m, 15 H, $-O-CH_2-C\underline{H}_3$, 6 CH_2); 1.59 (m, 2 H, $C\underline{H}_2-CH_2-COO-$); 1.96 (s, 4 H, 2 $C\underline{H}_2-CH=$); 2.17 (t, 2 H, $CH_2-C\underline{H}_2-COO-$, J = 7.0 Hz); 4.02 (q, 2 H, $-O-C\underline{H}_2-CH_3$, J = 7.1 Hz); 5.30 (m, 2 H, CH); 5.27 (s, 2 H, *Z*-H—C=C—H); 5.31 (s, 2 H, *E*-H—C=C—H). IR, ν/cm^{-1}: 710 (*Z*-H—C=C—H); 950 (*E*-H—C=C—H).

(*Z,E*)-Tetradec-11-en-1-ol (5). $LiAlH_4$ (1.9 g, 0.05 mol) and anhydrous THF (25 mL) were placed in a flask with a stirrer, reflux condenser, and dropping funnel. Then a solution of ester **4** (4.2 g, 0.0165 mol) in THF (10 mL) was added dropwise with stirring to the suspension. The mixture was heated to 50 °C and stirred for 1 h. Then excess $LiAlH_4$ was decomposed with HCl (7 mL) in H_2O (14 mL). THF was removed *in vacuo*, and the residue was extracted with hexane. The extract was washed with water, and dried with $MgSO_4$. Alkenol **5** (3.3 g, 94 %) was obtained after removal of hexane.

(*Z,E*)-Tetradec-11-enyl acetate (6). A mixture of alcohol **5** (3.3 g, 0.0156 mol) and pyridine (2.3 g, 0.03 mol) in anhydrous hexane (7 mL) were placed in a flask with a stirrer, reflux condenser, thermometer, and dropping funnel. Then a solution of AcCl (2.1 g, 0.027 mol) in hexane (5 mL) was added at 12 °C with stirring so that the temperature did not exceed 20 °C. The mixture was stirred for 2 h, then the reaction mass was treated with water, and the product was extracted with hexane. The extract was washed (H_2O) and dried ($MgSO_4$). Compound **6** (3.9 g, 98 %) was obtained after the removal of hexane.

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The weak interaction between C_{60} and molecular iodine in solution

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Fullerene C_{60} forms a weak molecular complex with iodine. Its stability constant is much less (<0.1 L^{-1} mol^{-1}) than that determined in another study.

Key words: fullerene C_{60}, iodine, molecular complex, stability constant.

We found a few years ago that C_{60} forms a molecular complex with iodine in different organic solvents.[1] However, this complex is so unstable that the stability constant could not be determined. We estimated that the stability constant at room temperature is smaller than 0.1 L mol^{-1} and planned to make measurements at a much lower temperature, down to −50 °C, where a measurable stability constant could be expected.

It was most surprising to learn from the paper by Turanov and Kremenskaya[2] that they found the formation of a very stable complex of C_{60} with I_2 in the same organic solvents at 20 °C. They used three different methods: i) spectrophotometry, ii) effect of iodine on the solubility of C_{60}, and iii) effect of C_{60} on the extraction of iodine from a toluene solution by aqueous potassium iodine solution. The composition of the complex was determined spectrophotometrically using the Job method[3] and the ratio of C_{60} : I_2 was found to be strictly 1 : 3; no indication of the formation of other complexes was determined from the data obtained by the third method and was found to be as great as $10^{8.9}$ L^3 mol^{-3}.

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Because of the obvious discrepancy with our former experience, we carefully repeated the experiments of Turanov and Kremenskaya using measurements of all three types.

Experimental

C_{60} of 99.8 % purity (Merck) was used without further purification. All the other materials were of analytical grade quality. The spectrophotometric measurements were made with an HP 8452A instrument. In the spectrophotometric experiments on the assumed interaction, a tandem cuvette was used. The spectra were recorded before and after the mixing of the content of the two halves of the cell. The temperature in all experiments was 20±2 °C.

Results and Discussion

In agreement with our former experiments, no deviation from the additivity was observed in toluene solutions of C_{60} and I_2 in the visible spectrum. There is a small difference from the additivity in the UV-region, which can be observed in a dichloromethane solution. This deviation is proportional to the concentration of iodine, therefore no stability constant can be calculated from these data.

According to our experiments, iodine has no effect on the solubility of C_{60} in chloroform. (We would like to mention that we are currently making a detailed study of the solubility of C_{60} in different organic solvents.[4])

In the extraction of iodine from toluene solutions of C_{60} and iodine with aqueous solutions of potassium iodide, we found that absorbance of the aqueous solution was slightly but significantly higher when the toluene phase contains C_{60}. This is in contrast to the results of Turanov and Kremenskaya, who found much smaller absorbances in the aqueous phase. They regarded this finding as evidence that C_{60} forms a stable complex with I_2 and thereby retains iodine in the organic phase.

Based on our former and present experiments it is obvious that C_{60} does not form a stable complex with iodine in different organic solvents at room temperature. Considering the available data on the donor-acceptor complexes of different organic compounds with iodine,[5] the value of this constant is certainly less than 1, and is probably less than 0.1 L mol^{-1}. Considering the structure of C_{60}, obviously not only one complex, but a series of weak complexes can be formed. However, it is most likely that the ratios of the stability constants of these complexes correspond to the statistical ones. The findings of Turanov and Kremenskaya[2] are inconceivable for us.

The increase in the absorbance of the aqueous phase is probably due to the dissolution of a minute amount (less than 10^{-5} mol L^{-1}) of C_{60} in a colloid form or the formation of an unstable triiodide complex of C_{60}.

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