

**THERMODYNAMIC CHARACTERISTICS OF THE DISSOLUTION
OF FULLERENE C₆₀ IN BENZENE, TOLUENE, *o*-XYLENE,
o-DICHLOROBENZENE, AND CARBON DISULFIDE
AT DIFFERENT TEMPERATURES**

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Abstract

The enthalpy of the fullerene C₆₀ dissolution in carbon disulfide CS₂ at 298.15 K was measured in a calorimeter with an isothermal shell. The value of the standard enthalpy of the fullerene C₆₀ dissolution in this substance was found. Standard thermodynamic functions of fullerene C₆₀ dissolution in benzene, chlorobenzene, bromobenzene, carbon disulfide, toluene, *o*-xylene, and *o*-dichlorobenzene at 298.15 K were determined based on the results of thermometric measurements and theoretical data. The standard thermodynamic functions have also been determined for toluene, *o*-xylene, and *o*-dichlorobenzene at temperatures of 288.15 and 308.15 K. The possible causes of the abnormal temperature dependence of the fullerene C₆₀ solubility in some solvents, as well as sign changes in their dissolution enthalpies, in the temperature range of 288.15–308.15 K have been discussed

Keywords

Enthalpy, dissolution Gibbs energy and enthalpy, crystallo-solvates, clusters, anomalous temperature dependence of solubility

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Introduction. C_{*n*} fullerenes are a group of allotropic modifications of carbon. In dependence on the number of atoms *n* in the molecule, they are conditionally subdivided into the lowest (*n* = 20–70) and the highest (*n* > 70) ones. In both, the carbon atoms are connected in such a way that the crystalline lattices resemble a football shape.

The fullerenes C₆₀ and C₇₀ are of practical interest, as the most common and most studied. They are characterized by high sorption capacity and chemical

stability, nonlinear optical properties, photoconductivity, low surface energy, good mechanical strength and elasticity, etc.

One of the most interesting features of the behavior of fullerene C_{60} is its unusual temperature dependence of solubility in some organic solvents. It was first discovered and described in Ref. [1], where the results of C_{60} solubility in n-hexane, toluene, and carbon disulfide in the temperature range of 200–400 K are given. The mentioned peculiarity lies in the fact that the solubility passes through the maximum observed at about 300 K. Dependencies of this kind were found for C_{60} in *o*-xylene [2], *o*-dichlorobenzene, 1,3-diphenylacetone, tetraline [3], benzene, and n-hexane [1]. In order to analyze the causes of this phenomenon, the measurements of fullerene C_{60} enthalpy were carried out in benzene, chlorobenzene, bromobenzene, toluene, *o*-xylene, *o*-dichlorobenzene at 298.15 K [4]. For the last three solvents, the dissolution enthalpy was also measured at temperatures of 288.15 and 308.15 K [5].

The aim of the present work is to present the results of the measurement of the dissolution enthalpy of fullerene C_{60} at 298.15 K carbon disulfide CS_2 . The carbon disulfide molecule, like the benzene molecule, has a dipole moment equal to zero, i.e., it is non-polar. Accumulated thermochemical material together with the data on the solubility of fullerene C_{60} in the studied solvents allows one to analyze the thermodynamic characteristics of dissolution, including their relationship to the polarity of the solvents.

Experimental. The same sample of fullerene as in Ref. [5] was used for the thermochemical measurements. The organic solvent used for its dissolution — carbon disulfide — was of “chemically pure” grade and was not additionally purified. Calorimetric measurements were performed using a device with an isothermal shell, the main characteristics of which are given in Ref. [5]. Thermal value of the calorimeter filled with 220.0 g of carbon disulfide was determined via electric method in a series of six experiments: it was equal to 1.278 ± 0.007 J/ Ω . All other results of the fullerene C_{60} dissolution enthalpy measurements in carbon disulfide CS_2 at 298.15 K are presented in Table 1.

Results and discussion. According to the data presented in Table 1, the concentration dependence of the enthalpy of fullerene C_{60} dissolution in carbon disulfide is insignificant or practically absent. This aspect was taken into account when the standard enthalpy of the fullerene C_{60} dissolution in carbon disulfide CS_2 was found; the value of this characteristic was equal to -18.90 ± 0.10 kJ/mol.

In addition to the unusual temperature dependence of fullerene C_{60} dissolution in some organic solvents, the change in the sign of the dissolution

enthalpy of fullerene C₆₀ in toluene, *o*-xylene, and *o*-dichlorobenzene, established in Ref. [5], attracts attention. Moreover, at temperatures of 288.15 and 298.15 K, corresponding to the region of increase in solubility with increasing temperature, an exothermic dissolution is observed, and endothermic dissolution is found at 308.15 K. The Gibbs — Helmholtz equation linking the solubility with the dissolution enthalpy requires opposite signs of the dissolution enthalpy. This clearly indicates that the temperature dependence of solubility is not only due to the dissolution process, but also due to another process (or processes). It should be noted that the negative value of entropy of fullerene C₆₀ dissolution in all investigated solvents [5, 6] decreases with temperature increase.

Table 1

The results of measurement of the enthalpy of fullerene C₆₀ dissolution in carbon disulfide CS₂ at 298.1 K

| Initial temperature of the calorimetric experiment (minus 10 000 Ω) t_0 , Ω | Heat transfer correction δ , Ω | Corrected temperature change ΔR_c , Ω | Fullerene mass m_w , mg | Amount of heat released upon dissolution Q , J | Dissolution enthalpy ΔH_d , kJ/mol |
|--|---------------------------------------|---|---------------------------|--|--|
| $\Delta H_{d, av} = -19.03$ kJ/mol; $\sigma = 0.005$ kJ/mol; $\sigma \times t_{0.05} = 0.010$ kJ/mol; $C_m = 0.00012$ mol/kg | | | | | |
| 24.362 | -0.065 | 0.312 | 15.15 | 0.3984 | -18.95 |
| 21.925 | -0.150 | 0.382 | 18.35 | 0.4889 | -19.20 |
| 20.183 | -0.140 | 0.434 | 21.00 | 0.5548 | -19.04 |
| 31.927 | 0.046 | 0.430 | 20.90 | 0.5493 | -18.94 |
| 19.768 | -0.205 | 0.404 | 19.55 | 0.5165 | -19.00 |
| $\Delta H_{d, av} = -19.12$ kJ/mol; $\sigma = 0.003$ kJ/mol; $\sigma \times t_{0.05} = 0.008$ kJ/mol; $C_m = 0.00022$ mol/kg | | | | | |
| 31.212 | 0.186 | 0.773 | 37.20 | 0.9885 | -19.15 |
| 29.927 | 0.154 | 0.691 | 33.45 | 0.8833 | -19.03 |
| 28.753 | 0.137 | 0.664 | 31.90 | 0.8426 | -19.17 |
| 25.909 | 0.178 | 0.678 | 32.75 | 0.8662 | -19.06 |
| 23.572 | 0.264 | 0.620 | 29.75 | 0.7926 | -19.20 |
| $\Delta H_{d, av} = -19.15$ kJ/mol; $\sigma = 0.003$ kJ/mol; $\sigma \times t_{0.05} = 0.010$ kJ/mol; $C_m = 0.00027$ mol/kg | | | | | |
| 31.751 | 0.177 | 0.857 | 41.05 | 1.095 | -19.22 |
| 35.217 | 0.122 | 0.845 | 40.80 | 1.010 | -19.17 |
| 30.783 | 0.076 | 0.917 | 44.15 | 1.172 | -19.06 |
| 35.932 | 0.081 | 0.984 | 47.25 | 1.257 | -19.20 |
| $\Delta H_{d, av}$ is the average dissolution enthalpy value; σ is the standard deviation of the average result; $t_{0.05}$ is the Student's t test; C_m is the solution molar concentration. | | | | | |

According to the data presented in Ref. [1], the reason of nonmonotonic temperature dependence of solubility of fullerenes is the orientation phase transition, well known for crystalline fullerene C_{60} [7]. It consists in changing of the type of crystalline lattice from simple cubic to face-centred cubic one at a temperature of about 260 K. The authors [1] considered the argument in favor of this to be the fact that the solubility maxima for the three solvents they studied (n-hexane, toluene, and carbon disulfide) are close to this temperature, and the observed minor differences in the values were explained by the experimental errors. In addition, they assumed that the surrounding of solid fullerene by a solvent, called solvation, could alter the characteristics of the phase transition.

The observed discrepancies in the solubility maxima positions (273–303 K) can hardly be explained by the experimental errors. Moreover, the enthalpy of the orientation phase transition amounts to about 7 kJ/mol; its low value does not explain the sharp change in the form of the solubility temperature dependence.

The model, according to which nonmonotone temperature dependence of solubility is associated with the possibility of formation and existence of clusters-aggregates (*conglomerates*) containing several fullerene molecules at once in solutions was proposed in Refs. [8, 9]. Such complex cluster formations should significantly affect the properties of fullerene solutions. The phenomenon of aggregation of molecules of the latter in the solution changes their thermodynamic characteristics. It leads to shift of phase equilibrium, and thus to change of solubility. The observed decrease in solubility of fullerenes with increasing temperature is treated as a consequence of the thermal decomposition of clusters, leading to an increase in the energy of interaction of fullerene molecules with solvent molecules and, consequently, to a shift in phase equilibrium in the direction of the solid phase. The ideology of cluster nature of the fullerene solubility gives the principal possibility of quantitative description of nonmonotone solubility temperature dependence. Decrease of the solubility with increasing temperature is observed in the range of temperature values, where existence of clusters consisting of a large number of fullerene molecules in the solution is thermodynamically beneficial. The observed decrease in solubility with increasing temperature is connected with thermal destruction of the largest clusters. At low temperature values ($T < 260$ K), to which the increasing section of the solubility temperature curve corresponds, the role of clusters is insignificant. This is due to a different structure of fullerenes in the crystalline phase, characterized by a higher value of interaction energy of their molecules with the solvent.

The authors of [10] measured the solubility of fullerenes C₆₀ and C₇₀ in tetraline, 1,3-diphenylacetone, and 1,2-dichlorobenzene. For both fullerenes in all three solvents, the maximum is noted on the temperature curve. It has been assumed that this phenomenon can be caused by both the formation of C₆₀ or C₇₀ fullerene aggregates and changes in the solid phase structure. The authors of [10] hypothesized that at temperature values lower than the maximum solubility temperature, the dissolution process is typical; dissolution occurs either from individual crystalline or solvated crystalline matter into a solution, in which C₆₀ (or C₇₀) fullerene molecules are associated. At temperatures above the maximum solubility temperature, dissolution occurs from a strongly solvated and disordered solid solution to a solution, in which the fullerene molecules are also associated. This principally explains the change in the enthalpy sign of the fullerenes dissolution. However, the authors of the above-mentioned work unreasonably believed that dissolution at temperatures below the maximum solubility temperature is an endothermic process, and is exothermic at the temperatures above the maximum solubility temperature. The results of this work do not confirm this.

When explaining the nonmonotonic dependence of the solubility of fullerenes, various authors rely either on the phenomenon of association of fullerene molecules in solutions [8, 11, 12] or on the formation of solvated crystals [10, 13]. The decomposition temperature of fullerene C₆₀ crystallosolvate with 1,2-dichlorobenzene is close to the maximum solubility temperature established by the authors of [10].

Another hypothesis explaining the observed course of the temperature dependence was made in Refs. [13–15], and later it received a good experimental confirmation [16, 17]. According to this hypothesis, at different temperature values, there are different crystalline phases in equilibrium with saturated fullerene solution: crystallosolvate at lower temperature values, individual unsolvated fullerene C₆₀ or crystallosolvate of other compositions at higher values. Decomposition (incongruent melting) of the solvate with the formation of individual fullerene and saturated solution of fullerene C₆₀ occurs at the maximum solubility temperature. Such a feature is a fairly common case for binary aqueous-salt systems, such as Na₂SO₄–H₂O, CuSO₄–H₂O, and others [17, 18]. According to the stated approach, the maximum temperature on the solubility curve is associated with the formation of crystalline solvate (I), melting incongruently with the release of another solvate (II), and formation of a saturated solution of fullerene C₆₀ in this solvent. Of course, the incongruent melting point must coincide with the maximum solubility temperature. At

temperature values below the incongruent melting point, the saturated solution is in equilibrium with the solvent (I). At the temperature values above the incongruent melting point, the saturated solution is in equilibrium with solvent (II), which, in most cases, is crystalline fullerene C_{60} .

According to the notions given in Refs. [13, 15], upon formation of crystallosolvate C_{60} -solvent S: $C_{60} \times nS$, the ascending (low-temperature) branch of the solubility curve is described by the equation

$$\frac{d \ln X_{C_{60} \times nS}}{dT} = \frac{\Delta_{sol} H^0 - \Delta_r H}{RT^2}, \quad (1)$$

where $X_{C_{60} \times nS}$ is the molar fraction of the crystallosolvate; $\Delta_{sol} H^0$ is the standard enthalpy of fullerene dissolution in the solvent; $\Delta_r H$ is the enthalpy of the $C_{60} \times nS$ crystallosolvate formation process.

After the solubility extremum, the dissolution of unsolvated fullerene should be described by a different equation:

$$\frac{d \ln X_{C_{60}}}{dT} = \frac{\Delta_{sol} H^0}{RT^2}. \quad (2)$$

In accordance with (1) and (2), the extreme course of the temperature dependence of solubility should be determined by a certain ratio between $\Delta_{sol} H^0$ and $\Delta_r H$. It is clear that in order to realize the solubility maximum $\Delta_{sol} H^0$, the enthalpy should be negative, and the enthalpy of crystallosolvate formation $\Delta_r H$, being also negative, should exceed the absolute value of fullerene dissolution enthalpy. Equations (1) and (2) are strict thermodynamic ratios; they do not contain any assumptions on the structure of the saturated solution. Similarly, it is possible to describe the situation where the crystallosolvate is a phase of variable composition, i.e., a solid solution. In this case, the solubility maximum can be observed at the peritectic point [16].

The adequacy of the model presented is confirmed on the fullerene C_{60} -solvent systems, for which the data on solubility at different temperature values are obtained [13, 14, 16, 19, 20]. The nonmonotonic temperature dependence, i.e., the presence of the solubility maximum, is due to the values of fullerene dissolution enthalpy and thermal stability of the formed crystalline solvents. The question arises: how well do the data on dissolution enthalpies correspond to the thermodynamic model described? Let us start with the fact that the information on the dissolution enthalpies of fullerenes, and particularly fullerene C_{60} , in individual solvents is very limited [13, 21–26] and refers to the temperature of 298.15 K.

Experimental determination of the dissolution enthalpy of fullerenes is characterized by some difficulties. Firstly, their very low solubility requires measurement of minuscule amounts of heat when determining dissolution enthalpies. In the present work, 0.5–1.0 Joules of heat were measured for determination of the dissolution enthalpy of fullerene C₆₀ in carbon disulfide CS₂. Not all results given in the above mentioned works meet the requirements of reliability and accuracy. Secondly, apart from low solubility, fullerenes are slow soluble substances, in most cases. This leads to long (time-wise) thermochemical experiments, and thus — especially in the case of calorimeters with isothermal shell — to increase of the error of the obtained values and reduction of their reliability. Thirdly, the propensity of fullerene molecules to form crystallosolvates with solvent molecules contributes to certain errors in the dissolution enthalpy values. The formation of these crystallosolvates may lead to the undervaluation of the dissolution enthalpy values at a given temperature. Probably, all these reasons are responsible for the observed differences in the values of the obtained values of fullerene solubility values. For example, in the case of benzene, the divergence reaches 20 times [16, 28], in the case of carbon disulfide — 15 times [13, 21, 27].

As described above, the most popular model of solvation processes that occur upon the dissolution of fullerenes, fullerene C₆₀ in particular, assumes its exothermic dissolution and does not imply a change in the sign of dissolution enthalpy in the temperature range of 285–310 K. Positive values of the enthalpy of dissolution were obtained for three solvents at a temperature of 308.15 K [5]. In other words, the signs of the dissolution enthalpies are opposite to those given by the Gibbs — Helmholtz equation for the temperature dependence of solubility with a maximum. This indicates the formation of crystallosolvates, thus changing the traditional dissolution thermodynamics.

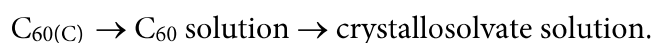
Due to the low solubility, the fullerene in calorimetric experiments falls to the bottom of the vessel after breaking the ampoule, where the formation of crystallosolvates occurs, which then dissolve. The enthalpy of the first process is negative and larger than the positive enthalpy of the second process in absolute value.

At a higher temperature value (308.15 K), only weak crystallosolvates may form, whose formation enthalpy is less than their dissolution enthalpy.

The Hesse law allows for the presentation of a transition



as




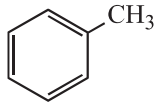
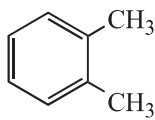
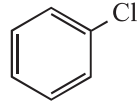
Here, one can verify if this model is correct. For this purpose, it is necessary to measure the dissolution enthalpy of fullerene C_{60} at higher temperature values when the formation of crystalloolvates is unlikely. Then it is possible to measure the actual dissolution enthalpy of fullerene C_{60} .

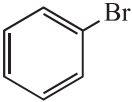
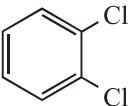
As for the mismatch between the signs of the values of enthalpy of the fullerene C_{60} dissolution and the Gibbs — Helmholtz solubility equation with the maximum temperature, it is true for some crystalloolvate(s). The thermodynamic calculations with the measured dissolution enthalpies are not quite correct, since these very dissolution enthalpies are the sum of at least two values.

The values of the measured standard enthalpy of the dissolution of fullerene C_{60} in seven solvents at 298.15 K, standard enthalpies of fullerene C_{60} transfer into the solvent from benzene, and the values of dipole moments of solvent molecules, taken from [17], are given in Table 2.

Table 2

Correlations between the organic solvent, standard enthalpy of fullerene $C_{60(C)}$ dissolution in it, and standard enthalpy of $C_{60(C)}$ transfer from benzene into it

| Organic solvent | | | Standard enthalpy of, kJ/mol | |
|---------------------------------|---|---------------------------------|--------------------------------------|--|
| Name and gross formula | Structural formula | Molecule dipole moment μ, D | C_{60} dissolution, ΔH_d^0 | C_{60} transfer from benzene into other solvent $\Delta H_t^0 = \Delta H_d^0 - \Delta H_b^0$ |
| Benzene C_6H_6 |  | 0.00 | -6.80 ± 0.15 | — |
| Toluene C_7H_8 |  | 0.37 | -8.65 ± 0.10 | -1.85 ± 0.19 |
| <i>o</i> -Xylene C_8H_{10} |  | 0.62 | -10.00 ± 0.10 | -3.20 ± 0.19 |
| Chlorobenzene C_6H_5Cl |  | 1.69 | -12.50 ± 0.40 | -5.70 ± 0.40 |

| Organic solvent | | | Standard enthalpy of, kJ/mol | |
|--|---|---------------------------------|---|---|
| Name and gross formula | Structural formula | Molecule dipole moment μ, D | C ₆₀ dissolution, ΔH_d^0 | C ₆₀ transfer from benzene into other solvent $\Delta H_t^0 = \Delta H_d^0 - \Delta H_b^0$ |
| Bromobenzene C ₆ H ₅ Br |  | 1.77 | -13.50 ± 0.40 | -6.70 ± 0.40 |
| <i>o</i> -Dichlorobenzene C ₆ H ₄ Cl ₂ |  | 2.16 | -12.90 ± 0.12 | -6.10 ± 0.19 |
| Carbon disulfide CS ₂ | S = C = S | 0.00 | -18.90 ± 0.10 | -12.10 ± 0.20 |

According to the presented data, there is a clear correlation between dissolution enthalpy, transfer enthalpy, and dipole moments of solvent molecules for benzene derivatives: the values of the first two characteristics increase in absolute value with increasing dipole moment of the solvent molecules, i.e., with increasing induction interaction.

The dissolution of fullerene C₆₀ in non-polar benzene and carbon disulfide is related to the ability of fullerenes to form charge transfer complexes with intramolecular donors [28, 29]. This ability is due to the fact that fullerenes have an electron affinity energy of about 7 eV, which is several times higher than the electron affinity of organic molecules intramolecular acceptors.

Conclusion. In addition to the unusual temperature dependence of the solubility of fullerene C₆₀ in organic solvents (maximum at about 300 K), the listed systems, at least with toluene, *o*-xylene, *o*-dichlorobenzene, are characterized by a change in the sign of the dissolution enthalpy in the temperature range of 295–300 K. Possible models of interactions in these systems are discussed to explain the observed phenomena. There is a clear correlation between the dissolution enthalpy of C_{60(C)} and the dipole moments of the solvent molecules — benzene and its derivatives. The dissolution of C_{60(C)} in non-polar solvents such as benzene and carbon disulfide is caused by the ability of fullerenes to form charge transfer complexes with intramolecular donors.

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