0.45 (50:50 EtOAc–cyclohexane); mp 153 °C (EtOAc–petroleum ether); IR ($CHCl_3$ solution) 3438, 3318, 1658, 1630 $cm^{-1}$; $^1H$ NMR ($CDCl_3$) δ 0.95 (m, 4 H), 3.60 (s, 2 H), 6.83 (br s, 1 H), 7.09 (m, 2 H), 7.30–7.52 (m, 9 H), 7.62 (dd, 2 H), 7.78 (dd, 2 H); $^{13}C$ NMR ($CDCl_3$) δ 12.3 (t), 34.9 (s), 58.6 (t), 127.0, 127.7, 128.2, 128.6, 130.1, 131.5 (each d), 135.1, 136.8, 139.6, 167.9, 169.4 (each s); MS (CI) *m/e* 355 ($MH^+$). Anal. Calcd for $C_{24}H_{22}N_2O$: C, 81.33; H, 6.26; N, 7.90. Found: C, 80.85; H, 6.21; N, 8.09.

**1-Amino-1-(benzamidomethyl)cyclopropane (10).** A solution of **9** (300 mg, 0.84 mmol) in MeOH (20 mL) was treated with $Pd(OH)_2$–C catalyst (100 mg) and the mixture subjected to Parr hydrogenation at 25 psi of overpressure for 24 h. The catalyst was removed by filtration through Celite, and the Celite was washed several times with MeOH. The filtrate was concentrated, and 1 M HCl (15 mL) was added. The resulting solution was washed with EtOAc (4 × 10 mL) and then lyophilized to leave the hydrochloride hemihydrate salt as a hygroscopic white solid (190 mg, 96%): mp 215 °C (MeOH–$Et_2O$); IR (mull) 3260, 1647 $cm^{-1}$; $^1H$ NMR ($D_2O$) δ 1.04 (m, 4 H), 3.64 (s, 2 H), 7.51 (t, 2 H), 7.59 (t, 1 H), 7.77 (d, 2 H); $^{13}C$ NMR ($D_2O$) δ 9.9 (t), 36.0 (s), 44.9 (t), 128.3, 129.9, 133.5 (each d), 134.0 (s), 172.8 (s); MS (CI) *m/e* 191 ($MH^+$). Anal. Calcd for $C_{11}H_{14}N_2O{\cdot}HCl{\cdot}0.5H_2O$: C, 56.05; H, 6.84; N, 11.88. Found: C, 56.37; H, 6.41; N, 11.34.

**1-(Aminomethyl)-1-benzamidocyclopropane (12). Method A.** To a solution of NaOH (80 mg, 2.00 mmol) in absolute EtOH (17 mL) was added **11** (187 mg, 1.00 mmol) and then Raney nickel (100 mg). The mixture was subjected to Parr hydrogenation at 50 psi of overpressure for 6 h. The catalyst was removed by careful vacuum filtration through Celite, and the Celite was washed with MeOH (4 × 10 mL). Concentrated HCl (3.2 mL) was added, the solvents were evaporated, and the residue was dried in vacuo over $P_2O_5$. The hydrated hydrochloride salt was crystallized from a $CHCl_3$ solution containing a minimum of MeOH by slow addition of $Et_2O$, giving clear plates (186 mg, 76%): mp 136–138 °C; IR (KBr) 3315, 1645 $cm^{-1}$; $^1H$ NMR ($D_2O$) δ 1.08 (s, 4 H), 3.23 (s, 2 H), 7.49 (t, 2 H), 7.60 (t, 1 H), 7.74 (d, 2 H); $^{13}C$ NMR ($D_2O$) δ 13.7 (t), 31.8 (s), 47.3 (t), 128.2, 129.8, 133.5 (each d), 134.0 (s), 173.5 (s); MS (CI) *m/e* 191 ($MH^+$). Anal. Calcd for $C_{11}H_{14}N_2O{\cdot}HCl{\cdot}H_2O$: C, 53.99; H, 7.00; N, 11.45. Found: C, 54.11; H, 6.63; N, 11.35.

**Method B.** A mixture of unpurified **13** (1.20 g, 3.39 mmol), $Et_2O$ (15 mL), and 1 M HCl (25 mL) was stirred at rt for 18 h. The aqueous phase was collected, washed with $Et_2O$ (3 × 10 mL), and then lyophilized to give a white solid (580 mg). The hydrated hydrochloride salt (428 mg, 52%) was obtained by crystallization as above.

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# Synthesis of Bicyclo[2.2.1]heptane-2,3,5,6-tetracarboxylic 2,3:5,6-Dianhydrides

Masao Yamada, Masatoshi Kusama, Toshihiko Matsumoto, and Toshikazu Kurosaki*

*Department of Industrial Chemistry, Faculty of Engineering, Tokyo Institute of Polytechnics, 1583 Iiyama, Atsugi, Kanagawa 243-02, Japan*

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Aromatic polyimides have been recently noted as high-performance polymers.[1] However, since their utilization has been limited by poor solubility in organic solvents,[2] we have studied the synthesis of aliphatic tetracarboxylic anhydrides with a bicyclic structure in order to enhance the solubility of the polyimides without degrading their thermal stability. Although Florey et al.[3] have reported the synthesis of tetramethyl bicyclo[2.2.1]heptane-2-*exo*,3-*endo*,5-*exo*,6-*endo*-tetracarboxylate, the synthetic route involved more than five steps. Moreover, it would be difficult to convert the tetramethyl ester into the desired dianhydride because of the trans configuration of the vicinal methoxycarbonyl groups. Palladium(0) and palladium(II) chloride are know to catalyze the bismethoxycarbonylation of alkenes and alkynes with carbon monoxide in the presence of CuCl or $CuCl_2$.[4-12] The present work relates the facile synthesis of two novel compounds, bicyclo[2.2.1]heptane-2-*endo*,3-*endo*,5-*exo*,6-*exo*-tetracarboxylic 2,3:5,6-dianhydride (**4**) and its *all-exo* isomer (**8**) from 2-*endo*,3-*endo*- and 2-*exo*,3-*exo*-anhydrides (**1** and **5**), respectively, by bismethoxycarbonylation of the double bond.

## Results and Discussion

The synthetic route to the compounds is illustrated in Scheme I. Bismethoxycarbonylation of **1** with carbon monoxide in methanol afforded 2-*endo*,3-*endo*,5-*exo*,6-*exo*-tetracarboxylate **2** in the presence of catalytic amounts of 5% Pd/C and stoichiometric amounts of $CuCl_2$. James and Stille[10] reported that in the palladium(II)-catalyzed bismethoxycarbonylation of norbornene the two methoxycarbonyl groups were introduced with exo configuration to give the 2-*exo*,3-*exo*-dicarboxylate. The stereochemistry of the methoxycarbonyl groups introduced into **2** was shown to be C-5(*exo*) and C-6(*exo*) by their $^{13}C$ NMR carbon signals at 172.09 (*exo*-CO) and 173.43 (*endo*-CO). The tetracarboxylic acid **3** was prepared by the acid-catalyzed hydrolysis of **2**. Alternatively, base-catalyzed hydrolysis afforded a mixture of the stereoisomers, which was confirmed easily by $^{13}C$ NMR. The *exo,endo*-dianhydride **4** was synthesized by dehydration reaction of **3** with thionyl chloride. It was also prepared from **2** by a one-pot method without isolating **3**.

In a manner similar to that mentioned above, **8** was prepared by employing **5** as a starting material. The *all-exo* ester **6** was synthesized directly by the tetramethoxycarbonylation of norbornadiene (**9**) in the presence of 8 molar equiv of $CuCl_2$, although the yield was somewhat low (30%). Interestingly, thermal dehydration of **3**, at 200 °C for 2 h with removal of water under vacuum, afforded the *all-exo* dianhydride **8**. Isomerization of the two carboxyl groups at the C-2 and C-3 positions was considered to occur by heating, which was confirmed by the $^{13}C$ NMR spectrum in which only one signal (171.4 ppm) was observed in the carbonyl region. Studies on the preparation of polyimides from these dianhydrides and diamines will be reported elsewhere in the near future.

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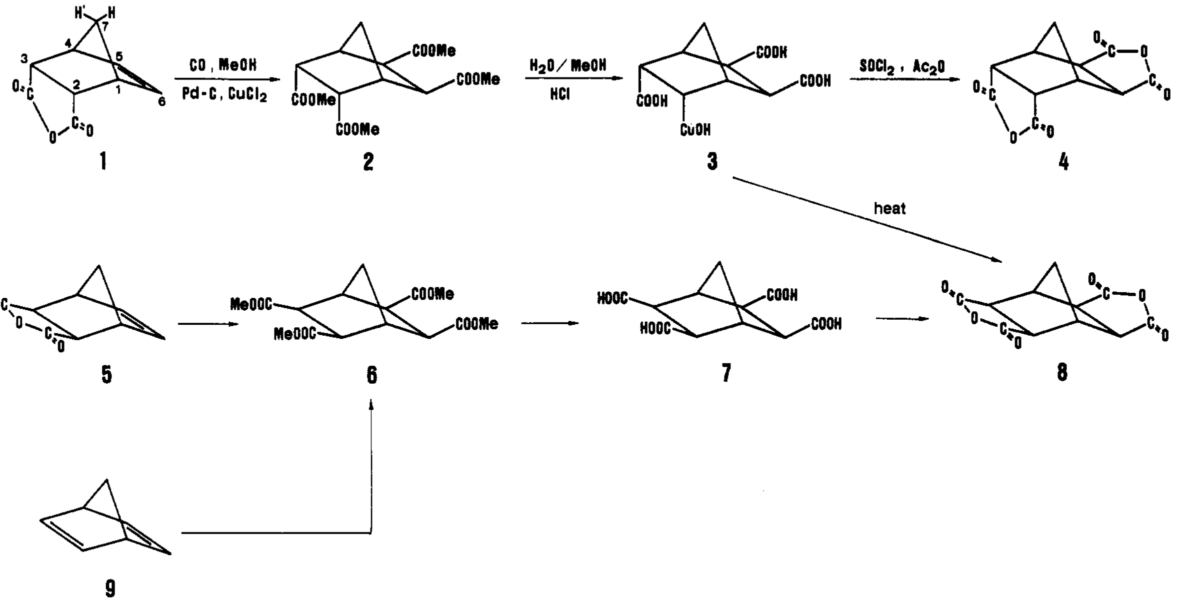
**Scheme I**



## Experimental Section

$^1$H and $^{13}$C NMR spectra were recorded at 400 and 100 MHz, respectively. Proton and carbon signals in $^1$H, $^{13}$C NMR spectra were assigned in H,H- and H,C-COSY and NOESY (with eliminating *J* correlation) spectra. Signals are singlets where no multiplicity is shown.

**Tetramethyl Bicyclo[2.2.1]heptane-2-*endo*,3-*endo*,5-*exo*,6-*exo*-tetracarboxylate (2).** In a 2-L three-necked flask were placed $CuCl_2$ (268.9 g, 2.00 mol), bicyclo[2.2.1]hept-7-ene-2-*endo*,3-*endo*-dicarboxylic anhydride (1) (82.10 g, 0.50 mol), and absolute methanol (760 mL). After the mixture was cooled overnight in a refrigerator, 5% Pd/C (7.58 g, 3.56 mmol as Pd) was added to the suspension. The air in the flask was replaced with CO, and then it was fitted with a 2-L rubber balloon. The reaction was allowed to proceed with magnetic stirring at rt, CO being sometimes introduced, until no more CO consumption was observed (~6 h). The solvent was removed under reduced pressure, the residue was stirred with a mixture of water (700 mL) and chloroform (700 mL), and the resulting suspension was filtered through Celite. The organic layer was separated and washed throughout with an aqueous saturated $NaHCO_3$ solution and water and then dried over $MgSO_4$. The solution was concentrated under reduced pressure to give an oily substance, which was solidified by adding hexane. The resulting solid was recrystallized from water, and 2 was obtained as a white crystalline solid (131 g; 80%): mp 82.5–83.5 °C; $^1$H NMR ($CDCl_3$) 1.46 (1 H, d, $J_{7',7}$ = 10.7, H'-7), 2.29 (1 H, d, $J_{7,7'}$ = 10.7, H-7), 2.84 (2 H, H-1,4), 3.08 (2 H, H-2,3), 3.42 (2 H, H-5,6), 3.63 (6 H, *exo*-$CH_3$), 3.66 (6 H, *endo*-$CH_3$); $^{13}$C NMR (100 MHz, $CDCl_3$) 37.25 (C-7), 43.27 (C-1,4), 44.75 (C-5,6), 46.09 (C-2,3), 51.77 ($CH_3$), 172.09 (*exo*-CO), 173.43 (*endo*-CO); IR (KBr) 1750, 1725, 1440, 1375, 1350, 1305, 1270, 1245, 1205, 1185, 1150, 1140, 1105, 1040, 925, 780 cm$^{-1}$. Anal. Calcd for $C_{15}H_{20}O_8$: C, 54.87; H, 6.14. Found: C, 54.68; H, 6.40.

**Bicyclo[2.2.1]heptane-2-*endo*,3-*endo*,5-*exo*,6-*exo*-tetracarboxylic Acid (3).** A mixture of concd HCl (40 mL) and water (30 mL) was added to a suspension of 2 (10 g, 0.03 mol) in methanol (30 mL) and then stirred at rt for 2 days. The reaction mixture was concentrated under reduced pressure to give a white solid. Recrystallization of the solid gave a white crystalline product (6.2 g, 75%): mp 161.5–162.0 °C; $^1$H NMR (400 MHz, DMSO-$d_6$) 1.37 (1 H, d, $J_{7',7}$ = 9.8, H'-7), 2.07 (1 H, d, $J_{7,7'}$ = 9.8, H-7), 2.58 (2 H, H-1,4), 2.98 (2 H, H-2,3), 3.16 (2 H, H-5,6), 12.15 (4 H, COOH); $^{13}$C NMR (100 MHz, DMSO-$d_6$), 35.90 (C-7), 42.63 (C-1,4), 44.40 (C-5,6), 45.56 (C-2,3), 173.11 (CO), 174.01 (CO); IR (KBr) 3000 (br), 1700, 1405, 1320, 1300, 1270, 1235, 900 cm$^{-1}$. Anal. Calcd for $C_{11}H_{12}O_8$: C, 48.53; H, 4.44. Found: C, 48.63; H, 4.69.

**Bicyclo[2.2.1]heptane-2-*endo*,3-*endo*,5-*exo*,6-*exo*-tetracarboxylic 2,3:5,6-Dianhydride (4).** To a solution of 3 (10 g, 0.037 mol) in acetic anhydride (100 mL) was added $SOCl_2$ (18.7 mL, 0.096 mol), and the mixture was heated at 100 °C until evolution of HCl ceased (~3 h). The reaction mixture was poured into ether (about 500 mL), and the precipitate was removed by filtration. Recrystallization of the solid from acetic anhydride gave 4 as a powdery product (5.7 g, 65%): mp 248–249 °C; $^1$H NMR (400 MHz, DMSO-$d_6$) 1.52 (1 H, d, $J_{7',7}$ = 12.0, H'-7), 1.81 (1 H, d, $J_{7,7'}$ = 12.0, H-7), 2.97 (2 H, d, $J_{5,4}$ = $J_{6,1}$ = 2.0, H-5,6), 3.09 (2 H, dd, $J_{1,2}$ = $J_{4,3}$ = 3.2, $J_{1,6}$ = $J_{4,5}$ = 2.0, H-1,4), 3.73 (2 H, d, $J_{2,1}$ = $J_{3,4}$ = 3.2, H-2,3); $^{13}$C NMR (100 MHz, DMSO-$d_6$) 37.45 (C-7), 42.36 (C-1,4), 46.65 (C-5,6), 48.01 (C-2,3), 172.72 (CO), 174.18 (CO); IR (KBr) 1850, 1775, 1720, 1440, 1350, 1220, 1090, 925, 910, 740 cm$^{-1}$. Anal. Calcd for $C_{11}H_8O_6$: C, 55.94; H, 3.41. Found: C, 55.89; H, 3.73. One-pot method: A mixture of of 2 (10 g, 0.03 mol), catalytic amounts of *p*-toluenesulfonic acid, and formic acid (150 mL) was heated at 90 °C for 8 h and then concentrated under reduced pressure. The residue was dissolved in formic acid (100 mL), to the solution was added acetic anhydride (150 mL) dropwise at 90 °C, and then the mixture was allowed to react for 5 h. The reaction mixture was evaporated to dryness under reduced pressure. The resulting solid was washed with ether and recrystallized from acetic anhydride to give 4 (4.7 g, 65% based on 2).

**Tetramethyl Bicyclo[2.2.1]heptane-2-*exo*,3-*exo*,5-*exo*,6-*exo*-tetracarboxylate (6).** In a manner similar to that mentioned above, 6 was prepared by the palladium(0)-catalyzed methoxycarbonylation of 5 (67% from 5): mp 141.5–142.5 °C; $^1$H NMR (400 MHz, $CDCl_3$) 2.10 (2 H, H-7), 2.76 (4 H, H-2,3,5,6), 2.90 (2 H, H-1,4), 3.64 (12 H, $CH_3$); $^{13}$C NMR (100 MHz, $CDCl_3$) 33.74 (C-7), 43.23 (C-1,4), 50.11 (C-2,3,5,6), 51.92 ($CH_3$), 172.44 (CO); IR (KBr) 1740, 1430, 1340, 1260, 1195, 1175, 1145, 1040, 930 cm$^{-1}$. Anal. Calcd for $C_{15}H_{20}O_8$: C, 54.87; H, 6.14. Found: C, 54.85; H, 6.33.

**Bicyclo[2.2.1]heptane-2-*exo*,3-*exo*,5-*exo*,6-*exo*-tetracarboxylic Acid (7).** As described above for compound 3, 7 was synthesized from 6 by the acid-catalyzed hydrolysis (70%): mp 161–162 °C; $^1$H NMR (400 MHz, DMSO-$d_6$) 1.89 (2 H, H-7), 2.55 (4 H, H-2,3,5,6), 2.72 (2 H, H-1,4), 12.13 (4 H, COOH); $^{13}$C NMR (100 MHz, DMSO-$d_6$) 32.90 (C-7), 42.78 (C-1,4), 49.07 (C-2,3,5,6), 173.63 (COOH); IR (KBr) 3000, 1705, 1415, 1275, 910 cm$^{-1}$. Anal. Calcd for $C_{11}H_{12}O_8$: C, 48.53; H, 4.44. Found: C, 48.71; H, 4.66.

**Bicyclo[2.2.1]heptane-2-*exo*,3-exo,5-*exo*,6-*exo*-tetracarboxylic 2:3,5:6-Dianhydride (8).** The *all-exo* dianhydride 8 was prepared by the chemical dehydration reaction of 7 with $SOCl_2$ in acetic anhydride as described above for compound 4, 60%: mp 254–256 °C; $^1$H NMR (400 MHz, DMSO-$d_6$) 1.30 (2 H, H-7), 2.94 (2 H, H-1,4), 3.41 (4 H, H-2,3,5,6); $^{13}$C NMR (100 MHz, DMSO-$d_6$) 30.91 (C-7), 43.29 (C-1,4), 47.18 (C-2,3,5,6), 172.27 (CO); IR (KBr) 1850, 1775, 1490, 1350, 1320, 1235, 1158, 1100, 940, 905, 890, 810, 740 cm$^{-1}$. Anal. Calcd for $C_{11}H_8O_6$: C, 55.94;

H, 3.41. Found: C, 56.04; H, 3.55.

**Supplementary Material Available:** $^1H$ and/or $^{13}C$ NMR spectra of 2–4 and 6–8 (10 pages). This material is contained in many libraries on microfiche, immediately follows this article in the microfilm version of the journal, and can be ordered from the ACS; see any current masthead page for ordering information.

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# Solubility of $C_{60}$ in Organic Solvents

N. Sivaraman, R. Dhamodaran, I. Kaliappan,
T. G. Srinivasan, P. R. Vasudeva Rao, and C. K. Mathews*

*Radiochemistry Programme, IGCAR, Kalpakkam 603 102, India*

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A large number of reports have appeared in the literature in the last couple of years on the preparation, properties, and reactions of $C_{60}$ (Buckminsterfullerene).[1–7] The solubility of $C_{60}$ in organic solvents plays a crucial role in its extraction, chromatographic separation, and reactions in solvent media. In fact, the poor solubility of $C_{60}$ in most organic solvents has been one of the main impediments to studying the properties of $C_{60}$. However, there has been no systematic investigation of the solubility of $C_{60}$ in different solvents, though qualitative observations have been made on the solubility behavior. Ajie et al.[8] reported that the solubility of a mixture of $C_{60}$ and $C_{70}$ in benzene was "about 5 mg/mL" at 25 °C. They also mentioned that the compound dissolved with difficulty in chloroform, dichloromethane, tetrachloromethane, diethyl ether, etc. In a recent report on the preparative separation of $C_{60}$ and $C_{70}$, Meier and Selegue[9] employed toluene solutions saturated with fullerenes, and they reported a concentration of ca. 6–8 mg/mL. In this paper, we report the solubility of pure $C_{60}$ in various organic solvents and note that the solubility shows a correlation with the solubility parameter of the solvent.

The solubility of $C_{60}$ in organic solvents at 303 K as measured in our work is given in Table I. The values given are averages of duplicate measurements. The solubility is rather low; in fact, even in aromatic solvents the solubility is comparable to that of inert gases. For example, the solubility of $C_{60}$ in benzene is 1.44 mg/mL, which corresponds to a mole fraction of $1.78 \times 10^{-4}$. The solubility of $C_{60}$ in benzene may be compared to the reported solubility of neon in benzene (mole fraction $1.07 \times 10^{-4}$ at 298 K for a pressure of 1 atm).[10] $C_{60}$ has been described as a van der Waals solid, similar to the solid form of inert gases, with little interaction between the molecules. The solubility of $C_{60}$ can thus be expected to be generally low because the energy required for the breaking of a large number of solvent bonds during the dissolution is not adequately compensated for by the interaction between the solute and the solvent. The higher solubility of $C_{60}$ in aromatic solvents as compared to aliphatic solvents can be explained on the basis of a better interaction between the solute and the solvent molecules in the former case.

**Table I. Solubility of $C_{60}$ in Organic Solvents (at 303 K)**

| solvent | solubility parameter[a] $(J/cm^3)^{1/2}$ | solubility ($\mu g/mL$) |
|---|---|---|
| isooctane | 14.17 | 26 |
| pentane | 14.52 | 4 |
| hexane | 14.85 | 40 |
| octane | 15.45 | 25 |
| decane | 15.81 | 70 |
| dodecane | 16.07 | 91 |
| tetradecane | 16.24 | 126 |
| cyclohexane | 16.77 | 51 |
| carbon tetrachloride | 17.59 | 447 |
| mesitylene | 18.04 | 997 |
| toluene | 18.20 | 2150 |
| benzene | 18.82 | 1440 |
| methylene chloride | 20.04 | 254 |
| dioxane | 20.50 | 41 |
| carbon disulfide | 20.50 | 5160 |

[a] Refers to the value at 25 °C.

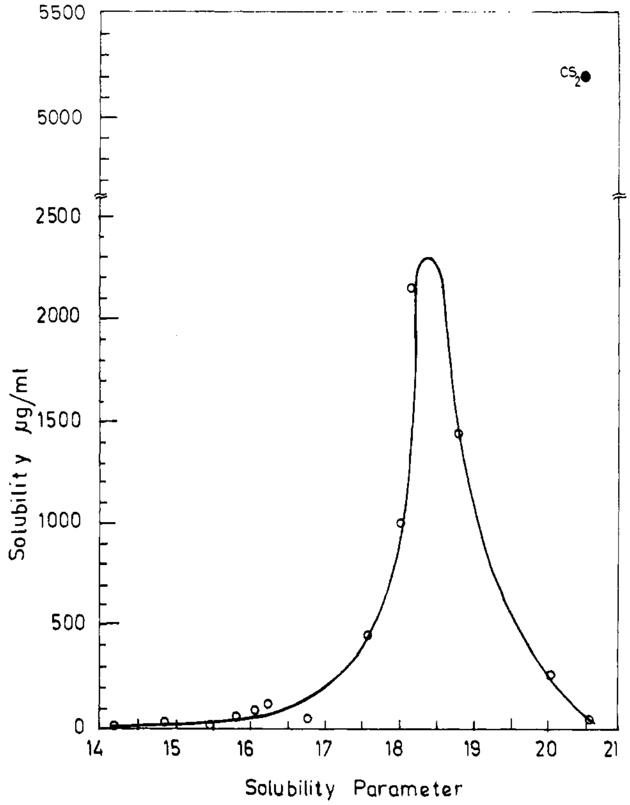

**Figure 1.** Variation of the solubility of $C_{60}$ in different solvents as a function of the solvent solubility parameter ($\delta$).

It is interesting to note that the solubility of $C_{60}$ in aliphatic hydrocarbons increases with the carbon number of the solvent. This can be explained in terms of Hildebrand's theory of regular solutions,[10] in which the solubility

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