

Solubility of *trans*-Co₂(CO)₆ [3,5-bis(CF₃)C₆H₃P(*i*-C₃H₇)₂] in dense carbon dioxide

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Abstract

The title compound (**1**) was prepared by the reaction of 3,5-bis(CF₃)C₆H₃P(*i*-C₃H₇)₂ (**L1**) and Co₂(CO)₈. Its solubility in supercritical carbon dioxide was measured at varying temperatures and pressures using a modified analytical extraction device. Solubility data were determined in the temperature and pressure ranges between 40 and 70 °C and between 100 and 300 bar, respectively. The solubility of **1** is lower compared to (*p*-CF₃C₆H₄)₃P, but much higher than for transition metal complexes bearing phosphines without fluorinated substituents.

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1. Introduction

Multi-phase catalysis allows for process steps, by means of which the reacting components can be separated easily for further purification or reprocessing of the catalyst and/or the unreacted substrate. Apart from aqueous systems and those based on ionic liquids, potential use of supercritical fluids (SCFs) was particularly investigated in the past decade [1–3]. SCFs in general allow to combine a homogeneous reaction in a single phase with a subsequent separation step, e.g., in a two-phase system only by adjusting temperature and/or pressure. Additionally, the high compressibilities of supercritical fluids in particular near their critical points are supposed to result in pressure effects on reactions in SCFs far more than at low pressures [4]. Investigations on variations of selectivities or rates of reactions in

SCFs as functions of pressure have been reported by several research groups and reviewed over the past years [5,6].

Our interest is directed towards the fundamental understanding of which parameters adequately characterize and influence a homogeneously catalyzed process using supercritical carbon dioxide (*sc* CO₂) as the reaction medium (*T*_c = 31.05 °C, *p*_c = 73.9 bar). When studying an integrated reaction-separation approach, knowledge of the phase behaviour of the participating components and especially of the catalyst solubilities, on basis of which a later process development can take place, obviously is indispensable. This knowledge allows for the setting of optimum reaction conditions [7] as well as for the design of an efficient post-reaction separation [8,9].

Recently, we studied the solubility of different substituted phosphines in *sc* CO₂, which may also act as co-ligands in homogeneously catalyzed reactions [10,11]. It was found that fluorination generally allows for higher solubilities compared to the unmodified derivatives.

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Based on empirical findings, similar fluorination concepts have been also transferred to homogeneous transition metal catalysis [12].

The present paper describes solubility measurements of an organometallic transition metal pre-catalyst $trans\text{-Co}_2(\text{CO})_6$ [3,5-bis(CF₃)C₆H₃P(*i*-C₃H₇)₂] (**1**), referring to a process variant, in which product separation is to be achieved by a thermally or pressure-controlled phase separation, while the catalyst remains dissolved in *sc* CO₂. Complexes like **1** with electron-rich substituents at the phosphorous are promising candidates for certain transition metal-catalyzed reactions, e.g., hydroformylation of long-chained olefins. Most recently, ethylene and propylene hydroformylation in *sc* CO₂ utilizing Co₂(CO)₆[P(4-CF₃C₆H₄)₃]₂ was demonstrated by Rathke and co-workers [13].

2. Results and discussion

2.1. Synthesis of 3,5-bis(CF₃)C₆H₃P(*i*-C₃H₇)₂ (**L1**) and $trans\text{-Co}_2(\text{CO})_6$ [3,5-bis(CF₃)C₆H₃P(*i*-C₃H₇)₂] (**1**)

1 is synthesized by the Grignard reaction of 3,5-(CF₃)₂C₆H₃MgBr with (*i*-C₃H₇)₂PCL in Et₂O with a yield of 90%. In analogy with the syntheses of other

derivatives of the $trans\text{-Co}_2(\text{CO})_6(\text{phosphine})_2$ type, further reaction of **L1** in a benzene solution with Co₂(CO)₈ yields **1** [14]. The spectroscopic data of **L1** and **1** listed in Section 4.1 are as expected for these types of compounds.

2.2. Molecular structure of the cobalt complex **1**

Red crystals of **1** were obtained from a mixture of dichloromethane and pentane. The molecular structure of **1** is shown in Fig. 1.

1 crystallizes in the monoclinic space group $P2(1)/c$ with a centre of inversion between the cobalt atoms. One of the CF₃ groups is rotatory disordered (F31, F32, F33). The phosphine ligands are orientated *trans* to each other, which is the usual conformation of complexes of the type Co₂(CO)₆(phosphine)₂. All bond lengths and angles are in the range of those of similar derivatives with the cobalt centres being trigonal-bipyramidally coordinated and carbonyl ligands showing a nearly perfect linearity [15–17].

2.3. Solubility measurements

To determine solubilities of solids in *sc* CO₂, an extraction apparatus was constructed as shown in

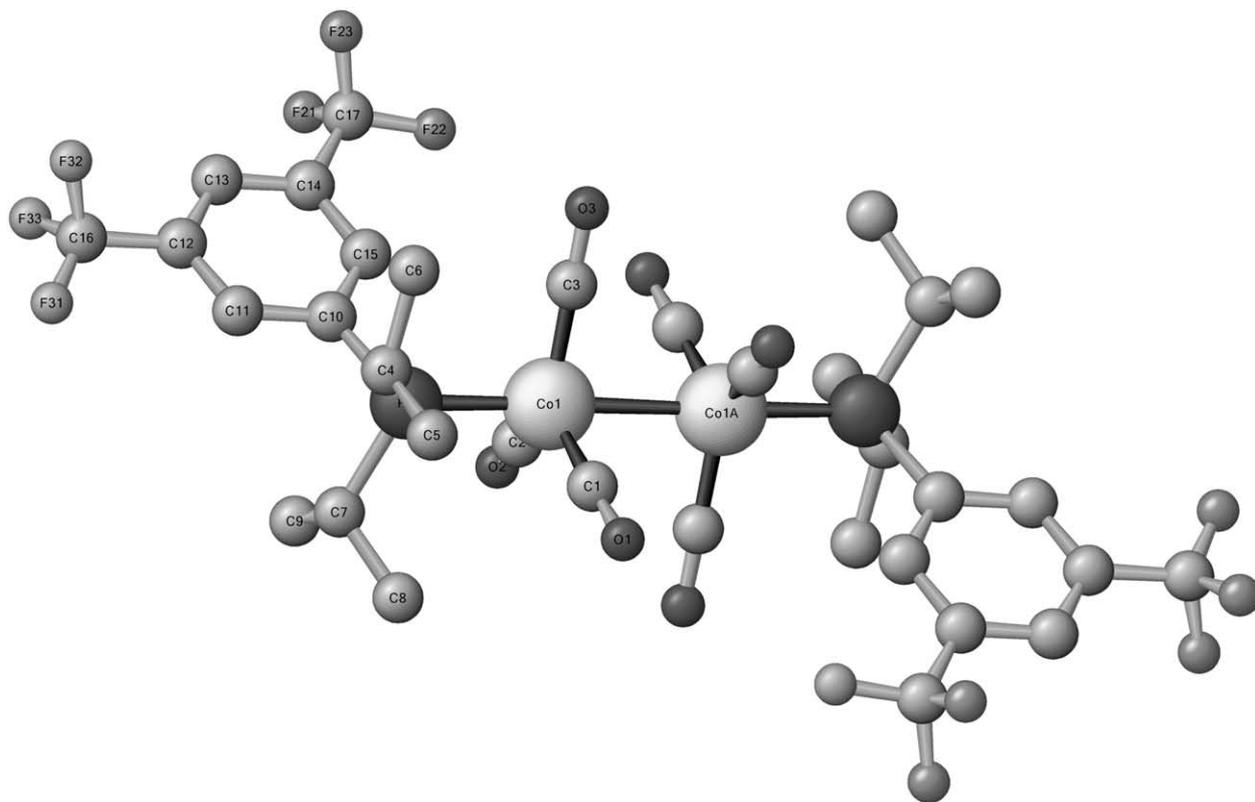


Fig. 1. Molecular structure of **1** in the crystal. Selected bond distances (pm) and angles (°): Co(1)–C(1) 177.9(2), Co(1)–P(1) 219.63(6), Co(1)–Co(1A) 267.08(6), P(1)–C(10) 184.1(2), F(21)–C(17) 132.2(3), O(1)–C(1) 115.2(3) and C(1)–Co(1)–C(2) 118.87(11), C(1)–Co(1)–P(1) 96.77(7), C(1)–Co(1)–Co(1A) 83.36(7), P(1)–Co(1)–Co(1A) 178.07(2), C(4)–P(1)–Co(1) 117.84(7), C(10)–P(1)–Co(1) 114.58(7).

Fig. 2 [18–21]. The apparatus mainly consists of a syringe pump (A) (ISCO model 100DX), a supercritical fluid extraction oven (C) containing an extraction vessel of 10 ml volume (D) (ISCO SXX2-10), and a back-pressure regulator (F) (JASCO model 880-81).

To allow for solubility measurements by a dynamic procedure, equilibrium conditions have to be established in the extraction cell. If a sufficiently low flow rate is adjusted, the CO₂ passing the extraction cell is loaded with an equilibrium substance amount in the steady state. To determine the optimum flow rate of *sc* CO₂, the solid is extracted at different flow rates. Starting from a somewhat high value, decrease of the flow rate results in an increased concentration of the solid in the fluid. At a certain flow rate, the solution is saturated. Further reduction of the flow rate does not cause any further changes of the concentration.

2.4. Solubility of **1**

The determination of solubilities of organometallic complexes, as described here with **1** being used as an example, yields reproducible data without any decomposition of the complex being observed. It is applicable to solids of low volatility and needs gram quantities of the sample. The extraction method applied here (for experimental details see Section 4.3) allows for the measurement of solubilities of **1** up to concentrations of approx. 20 g l⁻¹. At higher concentrations, the expansion tube of the back pressure regulator control may be plugged by complex particles occur. Hence it was possible to measure the solubility of **1** within the technically relevant range of 100 up to 237 bar, but not above 237 bar.

The appropriate flow rate for the extraction method was established in experiments at 200 bar and 50 °C using CO₂ flow rates of 1.25, 0.625, 0.313, and 0.125 ml min⁻¹. During the extraction, no decomposition of the complex was observed, as confirmed by IR

and ³¹P NMR spectroscopy. According to Fig. 3, the same complex quantities were extracted at flow rates of 0.313 and 0.125 ml min⁻¹ within the experimental error of ±5%. For this reason, a flow rate of 0.313 ml min⁻¹ was used for all further experiments.

The effect of temperature on the solubility of **1** was investigated in a series of experiments at the same CO₂ density (0.75 g cm⁻³). The results are shown in Table 1. The temperature which generally may affect the solubility of volatile compounds in compressed fluids has a minor impact only on the solubility of **1** in the investigated range. At temperatures of 50, 60, and 70 °C, approximately the same quantities of **1** are extracted. The lower solubility at 40 °C is believed to be mainly caused by the temperature influence on the solubility kinetics.

The solubilities of **1** at 50 °C and various pressures are compiled in Table 2. It is well known that one important factor influencing the solubility in *sc* CO₂ is the density of the fluid [3]. Compared to the solubility of different substituted phosphines in *sc* CO₂ published earlier, lower solubilities of the corresponding complexes may be expected in general [22]. The findings reported here confirm that **1**, a metal complex coordinated by fluorinated phosphine ligands, is less soluble than non-coordinating fluorinated phosphines (Fig. 4, ♦ and ●). Interestingly, the dependence of solubility on the CO₂ density shows a similar characteristic curve that exhibits a sharp bend for both complex **1** and the related ligand P(4-CF₃C₆H₄)₃ [10]. The significance and the cause of this bend are not yet clear and still under investigation. Compared to **1**, the non-fluorinated complex (Ph₃P)₂NiCl₂ (▲) [22] is poorly soluble in CO₂ even at a somewhat higher temperature, whereas the curve is similar shaped to that of Ph₃P (■) which again is much better soluble in *sc* CO₂ according to the other findings. Note that with the method reported here solubility measurement of the liquid phosphine **L1** is experimentally not feasible (see also Section 2.3).

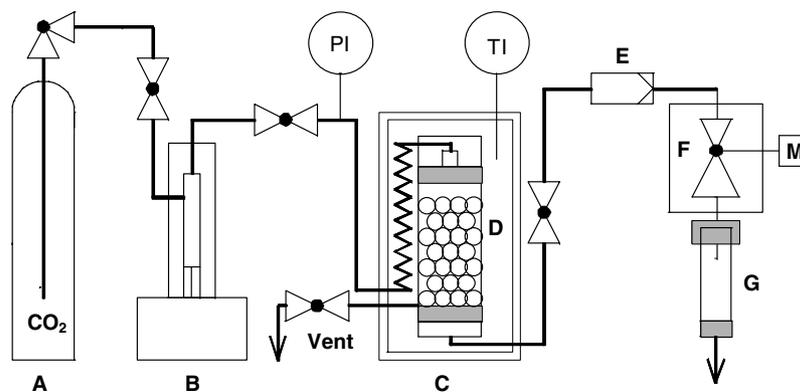
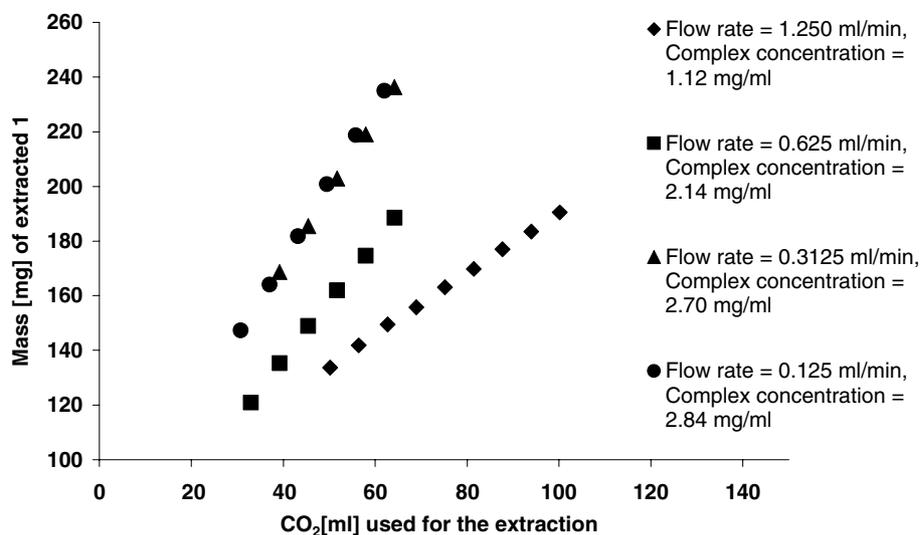


Fig. 2. Extraction device consisting of: A, CO₂ cylinder; B, syringe pump; C, oven; D, extraction cell; E, particle filter; F, back-pressure regulator; G, sampling tube.

Fig. 3. Extraction of **1** at 50 °C in CO₂ at varying flow rates.Table 1
Solubility of **1** in CO₂ (density 0.75 g cm⁻³) at various temperatures

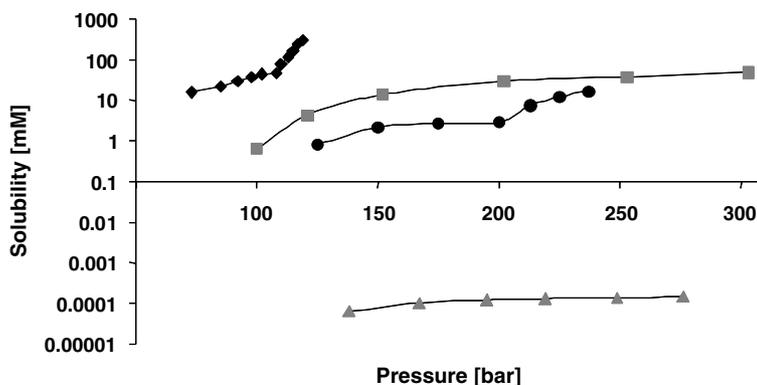
<i>T</i> (°C)	<i>p</i> (bar)	Solubility (g l ⁻¹)	Solubility (mM)
40	133.5	1.1 ± 0.1	1.1 ± 0.1
50	175.0	2.5 ± 0.3	2.7 ± 0.3
60	217.5	2.6 ± 0.3	2.7 ± 0.3
70	260.0	2.5 ± 0.5	2.6 ± 0.5

Table 2
Solubility of **1** at 50 °C in CO₂ at various pressures

<i>p</i> (bar)	CO ₂ density (g cm ⁻³)	Solubility (g l ⁻¹)	Solubility (mM)
100	0.38	<0.1	<0.1
125	0.61	0.7 ± 0.1	0.8 ± 0.1
150	0.70	2.1 ± 0.2	2.2 ± 0.2
175	0.75	2.5 ± 0.3	2.7 ± 0.3
200	0.78	2.7 ± 0.1	2.8 ± 0.1
213	0.80	7.0 ± 0.7	7.4 ± 0.7
225	0.81	11.4 ± 0.4	12.1 ± 0.4
237	0.82	15.8 ± 0.8	16.7 ± 0.9

3. Conclusion

The synthesis of [*trans*-Co₂(CO)₆{3,5-bis(CF₃)-C₆H₃P(*i*-C₃H₇)₂}] (**1**) and its solubility in supercritical carbon dioxide is reported. The solubility of **1** is low compared to non-coordinated phosphines, but high compared to transition metal complexes bearing phosphines without fluorinated substituents. Solubility data were determined between 40 and 70 °C, but at higher temperatures, however, a change of crystal structure or melting of the compound could result in a remarkable solubility change. Therefore, future measurements on liquids, for example, on non-coordinated phosphines or on solids which undergo melting induced by CO₂ pressure, would require an alternative approach for the determination of solubility data. The development of an appropriate experimental setup for such measurements is currently under way and will be reported in the near future.

Fig. 4. Solubility of (*p*-CF₃C₆H₄)₃P – ◆ (47 °C) [10], Ph₃P – ■ (47 °C) [10], **1** – ● (50 °C), and (Ph₃P)₂NiCl₂ – ▲ (55 °C) [22] in CO₂ at varying pressures.

4. Experimental

4.1. Syntheses

All manipulations were performed under an atmosphere of dry argon using standard Schlenk techniques. 3,5-Bis(CF₃)C₆H₄-Br (ABCR) and (*i*-C₃H₇)₂PCl (Aldrich) were distilled prior to use. Co₂(CO)₈ (Aldrich) was crystallized from pentane.

The NMR spectra of **1** and **L1** were recorded on a Bruker Avance 250 spectrometer at 298 K. ¹H NMR: 250.13 MHz, internal reference CDCl₃ (δ = 7.27) relative to SiMe₄ (δ = 0 ppm); ¹³C{¹H} NMR: 62.90 MHz, internal reference CDCl₃ (δ = 77.00) relative to SiMe₄ (δ = 0 ppm); ³¹P{¹H} NMR: 101.25 MHz, internal reference 85% H₃PO₄ (δ = 0 ppm). Chemical shifts and coupling constants are given in ppm and Hz, respectively. IR spectra were recorded on a Perkin–Elmer, System 2000 FT-IR, as a Nujol film (Nujol dried using K/Na alloy) or in KBr (KBr dried at 500 °C for several hours). EI-MS (70 eV) spectra were obtained from GC–MS analysis with a Hewlett–Packard 5890/5922 instrument. ESI-MS spectra were measured on a Hewlett–Packard Series 1100 MSD and HR-EI-MS on Micromass GCT.

Melting or decomposition points were determined on a Büchi B545 melting-point apparatus. Elemental analyses were performed by Hermann Kolbe Micro Analytical Laboratory (Mülheim/Ruhr, Germany).

4.1.1. (*i*-C₃H₇)₂PCoH₃(CF₃)₂, **L1**

A round flask with stirrer was charged with magnesium turnings (0.912 g, 37.50 mM) in 5 ml Et₂O with two drops of 1,2-dibromoethane. A solution of 3,5-(CF₃)₂C₆H₃ Br (10.00 g, 34.13 mM) in 50 ml Et₂O was added drop wise to the magnesium turnings under stirring. The reaction mixture was refluxed for two hours and then filtrated. Within 1 h at 0 °C, the Grignard solution was added to the solution of (*i*-C₃H₇)₂PCl (5.21 g, 34.13 mM) in 40 ml diethyl ether. After warming up, the mixture was hydrolyzed with degassed water. The organic layer was separated, dried over MgSO₄, and the solvent was removed in vacuum. The dark brownish liquid was distilled under vacuum at 60 °C, yielding 10.0 g (90%) of **L1** as a colorless liquid.

¹H NMR: 0.78 (dd, ³J_{H,H} = 6.87, ³J_{H,P} = 11.3 Hz, 6H, CH₃), 0.96 (dd, ³J_{H,H} = 7.12, ³J_{H,P} = 15.25, 6H, CH₃), 2.04 (m, 2H, CH–P), 7.74 (s, 1H, Ph), 7.80 (d, ³J_{C,P} = 5.5, 2H, Ph). ³¹P NMR: 13.38 (s). ¹³C NMR: 18.30 (d, ²J_{C,P} = 8.05, CH₃), 19.26 (d, ²J_{C,P} = 18.39, CH₃), 22.86 (d, ¹J_{C,P} = 13.79, CH–P), 122.57 (m, CH, Ph), 123.67 (q, ¹J_{C,F} = 273.5, CF₃), 131.40 (dq, ²J_{C,F} = 36.6, ³J_{C,P} = 6.90, C, Ph), 134.16 (d, ²J_{C,P} = 19.54, CH, Ph) 139.59 (d, ¹J_{C,P} = 27.58, C–P, Ph). IR (Nujol): 1355 s, 1279 s, 1183 s, b, 1140 vb, vs, 1098 w, 1023 vw, 899 w, 843 vw, 708 w, 682 w.

EI-MS, *m/z* (relative abundance): 330 (M⁺), 311 (C₁₄H₁₇F₅P⁺), 288 (C₁₁H₁₁F₆P⁺), 246 (C₈H₅F₆P⁺), 227 (C₈H₅F₅P⁺), 195 (C₈H₄F₅⁺) 43 (C₃H₇⁺). HR-EI-MS (C₁₄H₁₇F₆P), *m/z* (calculated): 330.0983 (330.0972).

4.1.2. Co₂(CO)₆ [3,5-bis(CF₃)C₆H₃P(*i*-C₃H₇)₂] (**1**)

The solution of **L1** (10.000 g, 30.27 mM) in 40 ml benzene was added drop wise to the solution of Co₂(CO)₈ (5.1778 g, 15.14 mM) in 60 ml benzene. The reaction mixture was refluxed and stirred for 6 hours. After cooling to 25 °C, the solvent was removed in vacuum and the red coloured crude product (yield >95%) was washed twice with *n*-pentane. **1** (MP 144 °C, decomposition) was crystallized from a 1:5 mixture of CH₂Cl₂/pentane (68% yield).

³¹P NMR: 90.3 (s). IR: 1973 w, 1952, vs, vb 1357 s, 1279 s, 1185 s, 1138 vs 1124 sh, 1096 w, 1052 vw, 905 w, 843 vw, 705 w, 683 w, 650 s, 536 w, 511 w, 500 w, 466 vw. ESI-MS, *m/z* (relative abundance): 984 (C₃₄F₁₂H₃₅NaNO₆P₂Co₂⁺; M⁺ + Na + NH₃ – 2H), 968 (C₃₄H₃₃F₁₂P₂O₆Co₂Na⁺, M⁺ + Na – H), 903 (C₃₁H₂₆F₁₂P₂O₆Co₂⁺, M⁺ – C₃H₇), 369 (C₁₄H₁₉F₆PNNa⁺; **L1** + Na – H + NH₃), 347 (C₁₄H₂₀F₆PN⁺; **L1** + NH₃).

Table 3
Crystallographic data of **1** (standard deviations in parentheses)

Empirical formula	C ₃₄ H ₃₄ Co ₂ F ₁₂ O ₆ P ₂
Formula weight	946.41
Crystal size (mm ³)	0.45 × 0.45 × 0.1
Crystal system	Monoclinic
Space group	<i>P</i> 2 ₁ / <i>c</i> (no. 14)
Unit cell dimensions	
<i>a</i>	1019.79(17)
<i>b</i>	1293.2(2)
<i>c</i>	1610.1(3)
<i>β</i>	108.034(3)
Volume (pm ³)	2019.1(6) × 10 ⁶
<i>Z</i>	2
<i>D</i> _{calc} (g cm ^{−3})	1.557
Diffractometer	Siemens SMART 1000 CCD diffractometer
Wavelength	Mo Kα, graphite monochromator
Temperature (K)	200
<i>θ</i> Range (°)	2.06 ≤ <i>θ</i> ≤ 28.31
Scan	<i>ω</i> scan, Δ <i>ω</i> = 0.3°
Index ranges	−13 ≤ <i>h</i> ≤ 13, −16 ≤ <i>k</i> ≤ 17, −21 ≤ <i>l</i> ≤ 21
Number of reflections measured	20697
Independent reflections	4844
Reflections observed	3974 (<i>I</i> > 2σ)
Number of parameters refined	290
Residual electron density (e pm ^{−3})	0.56 × 10 ^{−6}
Corrections	Lorentz and polarization, exp. absorption correction
Structure solution	Direct methods
Structure refinement	Full-matrix least-square on <i>F</i> ₂
Programs and weightings used	SHELX-97 [23], XPRMA, ZORTEP [24]
<i>R</i> indices	<i>R</i> ₁ = 0.0363 (<i>I</i> > 2σ) <i>R</i> _w = 0.0992 (all data against <i>F</i> ²)

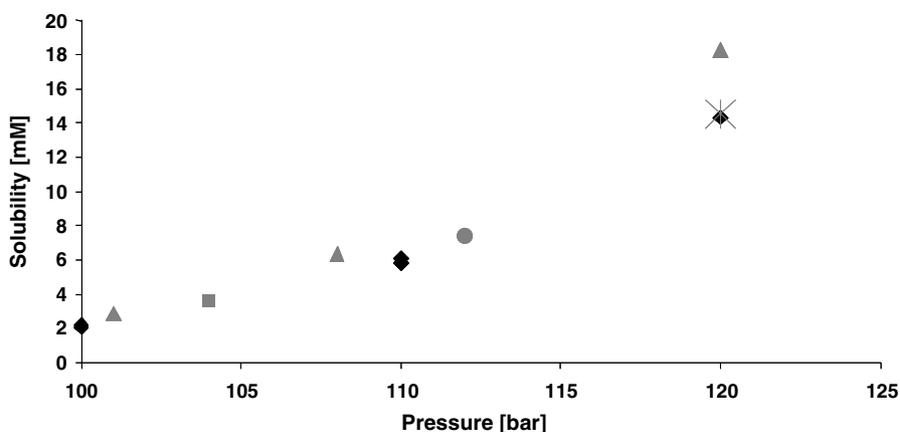


Fig. 5. Solubility of naphthalene in CO₂ at 55 °C. Own measurement – ◆, Chang/Morell [18] ●, McHugh/Paulaitis – ▲, [25], Mitra et al. – ■ [19], Lamb et al. – ☆ [26].

Elemental analysis: % found (calculated) C, 42.90 (43.13), H, 3.64 (3.62).

4.2. X-ray crystallography

Red crystals of **1** suitable for X-ray analysis were grown from CH₂Cl₂/pentane solution. All diffraction data were collected on a Siemens SMART 1000 CCD diffractometer (Table 3). Crystallographic data of the structure have been stored at the Cambridge Crystallographic Database Centre, Supplementary publication Nos. CCDC 242209 (**1**). Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or www: <http://www.ccdc.cam.ac.uk>).

4.3. Solubility measurements

The solid (up to 2 g) is placed on a bed of glass beads (0.25–0.5 mm) inside the extraction cell **D** (see Fig. 1, Section 2.1) having a volume of 10 ml which is heated to the temperature of measurement. To avoid the removal of solid particles from the cell, a metal frit with 2 μm porosity was used.

The *sc* CO₂ is compressed to the desired pressure using the syringe pump **B**. The experiment proceeds by pumping the *sc* CO₂ at a constant flow rate through the extraction cell. In the extraction cell, the solid dissolves in *sc* CO₂ and is thereby removed from the cell. The pressure is released through a back-pressure regulator (F). As separator, specially designed glass tubes (G) with frits were used. The separator is used to vent off the gas and collect the particles precipitating while the *sc* CO₂ solution expands. Knowing the flow rate, the time of sampling the solid, and the mass of the precipitated solid, its solubility is calculated.

The separation method was examined in an experiment with a known amount of triphenylphosphine being completely extracted from the cell and collected in the glass tube with an accuracy of 99.9%.

In order to validate the method with regard to accuracy and reproducibility, solubility of naphthalene was determined. As shown in Fig. 5, the values for the solubility of naphthalene are in good agreement with other published data [18,19,25,26].

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