Synthesis and X-Ray Single Crystal Structure of Dialkyl 2-[1-(2,2-Dimethylpropionyl)-3,3-dimethyl-2-oxobutyl]-3-(triphenylphosphoranylidene)succinates

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A one-pot synthesis of sterically congested phosphorus ylides in fairly high yields by the reaction of 2,2,6,6-tetramethyl-3,5-heptanedione, dialkyl acetylenedicarboxylates and triphenylphosphine is reported. The structures of these compounds were confirmed by IR, ^{1}H, ^{31}P and ^{13}C NMR spectroscopy, and X-ray single crystal structure determination. The NMR spectra (CDCl$_3$ as solvent) indicated that the compounds contained two rotamers for each ylide.

Key words: 2,2,6,6-Tetramethyl-3,5-heptanedione, Phosphorus Ylide, Acetylenic Ester, Vinyltriphenylphosphonium Salts

Introduction

Phosphorus ylides are a class of special type of zwitterions, which bear strongly nucleophilic electron rich carbanions. The electron distribution around the P$^+$–C$^-$ bond and its consequent chemical implications had been probed and assessed through theoretical, spectroscopic and crystallographic investigations [1]. They are excellent ligands and excel in their ligating functions the unstabilized ylides because of their ambidentate and chemically differentiating character. Proton affinity of these ylides can be used as a molecular guide to assess their utility as synthetic reagents and their function as ligands in coordination and organometallic chemistry [2]. The nucleophilicity at the ylidic carbon is a factor of essential mechanistic importance in the use of these ylides as Wittig reagents. Phosphorus ylides are important reagents in synthetic organic chemistry, especially in the synthesis of naturally occurring products, compounds with biological and pharmacological activity [3]. These ylides are usually prepared by treatment of a phosphonium salt with a base, and phosphonium salts are usually obtained from a phosphane and an alkyl halide. Phosphonium salts are also prepared by Michael addition of phosphorus nucleophiles to activated olefins and in other ways [4]. The phosphonium salts are most often converted to the ylide by treatment with a strong base, though weaker bases can be used if the salt is acidic enough. In recent years, we have established a one-pot method for the synthesis of stabilized ylides [5 – 8]. In this paper, we wish to describe the preparation of sterically congested phosphorus ylides from the CH-acid 2,2,6,6-tetramethyl-3,5-heptanedione in fairly high yields.

Results and Discussion

The ylide **5** may result from initial addition of triphenylphosphane (**1**) to the acetylenic ester **2** and concomitant protonation of the 1 : 1 adduct, followed by attack of the CH-acid anion on the vinyltriphenylphosphonium cation to form the stabilized phosphorane **5** (Scheme 1). The NMR spectra indicated that solutions of compound **5** (CDCl$_3$ as solvent) contain two rotamers (**5E** and **5Z**). The relative percentages of rotamers in CDCl$_3$ for each ylide **5** were determined from the ^{1}H NMR spectra. The structures **5a, b** were deduced from their ^{1}H, ^{13}C and ^{31}P NMR spectra.

Description of the crystal structure of **5a**

The crystal of **5a** is built up from ylide molecules (Fig. 1) and disordered solvent molecules of different type (see Experimental Section). In the investigated crystal, the chiral atom C(22) (Fig. 1, Table 1) had the *S* configuration.
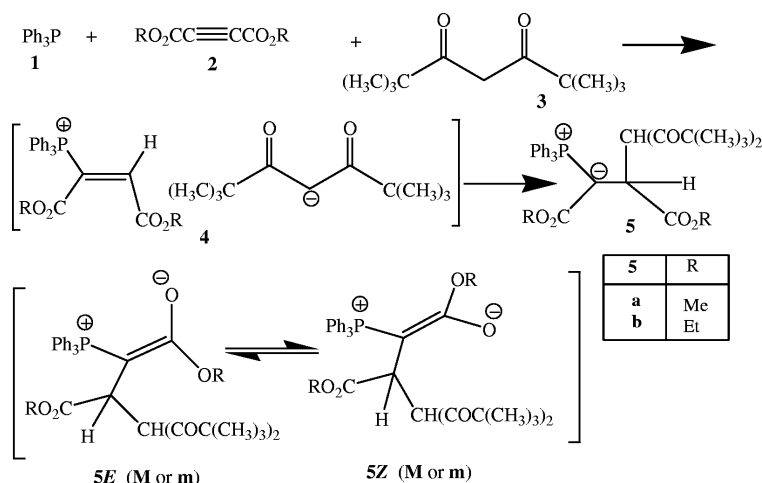
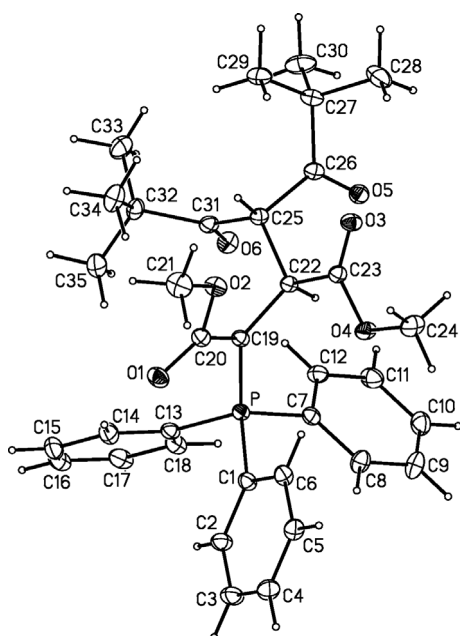Scheme 1. **M** = major rotamer and **m** = minor rotamer.

Fig. 1. The molecular structure of the ylide **5a** showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

The molecular geometry observed in **5a** corresponds well with the knowledge on stabilized phosphonium ylides [1, 9]. The values of P–C(19) (1.731(2) Å), C(19)–C(20) (1.421(2) Å) and C(20)=O(1) (1.241(2) Å) bond lengths are in agreement with the previously observed ones and are consistent with electronic delocalization involving the P–C–(C=O) system. That leads to a nearly planar, trigonal environment of the ylidic atom C(19) (it is

close to sp^2 hybridization), which is reflected in the distance of the P atom from the C(19)/C(20)/C(22) plane (0.36 Å) and in the sum of the angles about C(19) close to 360° (358.5(2)° with the mean value of 120°). Such small degree of the pyramidalization of the ylidic C atom in the stabilized ylide is accompanied by other structural features; namely, shortening of the C_{ylide}–C_α [C(19)–C(20)] distance and the lengthening of the C=O [C(20)=O(1)] distance (see Table 2 and compare with the unaffected C(22)–C(23) distance and the whole (C(23)=O(3))–O4–C24 ester group).

The partial double bond character of C(19)–C(20) results in some increase of the rotational barrier around it. Consequently, the two rotamers (**5E** and **5Z** in Scheme 1, with different O(1) to P orientation) may be expected and were shown indeed by NMR to exist in equilibrium in CDCl$_3$ solution. However, the X-ray data demonstrated that, in the solid state, this orientation may be opposite even in compounds of highly related structures [9, 10]. The ylide molecules in **5a** and in the crystal of analogous compound described recently (ethyl ester of a phenyl derivative [9]) adopt different orientation of the carbonyl O(1) atom with respect to the P atom. In **5a**, the O(1) is in *syn*, and in the related compound – in *anti* orientation. This is reflected in the values of the torsion angle P–C(19)–C(20)–O(1), which is −16.0(2)° in **5a** and 165.0(2)° in related compounds [9, 10]. However, all that shows that the ester groups are slightly twisted out of the plane of the ylidic atom C(19), reducing delocalization to some extent. It is to note here, that a search of the Cambridge Structural Database [10] revealed about 60 hits of stabilized ylides of related structures, with

Table 1. Crystal data and structure refinement details for **5a**.

Crystal data	
Empirical formula	$C_{35} H_{41} O_6 P$
Formula weight (g mol^{-1})	588.65
Crystal system, space group	orthorhombic, $P2_12_12_1$
Unit cell dimensions [Å]	$a = 10.401(2)$
	$b = 14.433(3)$
	$c = 27.400(4)$
V [Å^3]	4113.2(13)
Z	4
D_{calc} [g cm^{-3}]	0.951
μ [mm^{-1}]	0.863
$F(000)$	1256
Crystal size [mm]	$0.40 \times 0.25 \times 0.25$
Crystal color and form	Colorless needle
Data collection	
Diffractometer	Xcalibur PX
Monochromator	Graphite
Radiation type, wavelength, λ [Å]	Cu-K_α, 1.5418
T [K]	100(2)
θ Range [°]	4.45 – 76.12
h, k, l Ranges	$-12 \leq h \leq 12$,
	$-11 \leq k \leq 18$,
	$-32 \leq l \leq 29$
Absorption correction	analytical
T_{min}/T_{max}	0.747/0.869
Measured reflections	36083
Independent reflections	8035 [$R_{int} = 0.0327$]
Observed refl. ($I > 2\sigma(I)$)	7582
Completeness to $\theta = 70.00°$	0.997
Refinement	
Refinement on	F^2
Data/restraints/parameters	8035/0/389
$R(F_o^2 > 2\sigma(F_o^2))$	$R1 = 0.0366$, $wR2 = 0.1020$
R (all data)	$R1 = 0.0379$, $wR2 = 0.1031$
GooF = S	1.097
Weighting parameter a/b	0.0792/0.0
$\Delta\rho_{max}/\Delta\rho_{min}$ [e Å^{-3}]	0.33/-0.28
Flack parameter	0.045(14)

$R1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$; $wR2 = \sqrt{\Sigma[w(F_o^2 - F_c^2)^2]/\Sigma[w(F_o^2)^2]}$. Weighting scheme: $w = 1/[\sigma^2(F_o^2) + (aP)^2 + bP]$ where $P = (F_o^2 + 2F_c^2)/3$.

Table 2. Selected interatomic distances [Å], valence angles [°] and torsion angles [°] in **5a**.

P-C(1)	1.813(2)	O(4)-C(23)	1.345(2)
P-C(7)	1.806(2)	O(4)-C(24)	1.442(2)
P-C(13)	1.821(2)	O(5)-C(26)	1.221(2)
P-C(19)	1.731(2)	O(6)-C(31)	1.215(2)
O(1)-C(20)	1.241(2)	C(19)-C(20)	1.421(2)
O(2)-C(20)	1.369(2)	C(19)-C(22)	1.519(2)
O(2)-C(21)	1.433(2)	C(22)-C(23)	1.527(2)
O(3)-C(23)	1.210(2)		
C(1)-P-C(7)	105.96(7)	C(20)-O(2)-C(21)	115.26(13)
C(1)-P-C(19)	112.76(7)	C(23)-O(4)-C(24)	115.06(12)
C(1)-P-C(13)	104.10(7)	C(20)-C(19)-P	114.60(11)
C(7)-P-C(13)	106.29(7)	C(22)-C(19)-P	123.41(11)
C(7)-P-C(19)	109.60(7)	C(20)-C(19)-C(22)	120.52(13)
C(13)-P-C(19)	117.34(7)		
P-C(19)-C(20)-O(1)	−16.0(2)	C(7)-P-C(19)-C(22)	5.6(2)
C(7)-P-C(19)-C(20)	−160.7(2)	C(1)-P-C(19)-C(22)	123.3(2)
C(1)-P-C(19)-C(20)	−42.9(2)	C(13)-P-C(19)-C(22)	−115.7(2)
C(13)-P-C(19)-C(20)	78.1(2)		

most of them existing in *anti* rather than *syn* coplanar conformation. Most of them also showed the ester group slightly twisted.

Both ester moieties in **5a**, (C(20)=O(1))–O(2)–C(21) and (C(23)=O(3))–O(4)–C(24) are almost planar with the methyl C(21) and C(24) atoms being nearly coplanar with the rest. The distances of atoms C(21) or C(24) from the planes C(20)/O(1)/O(2) and C(23)/O(3)/O(4) are about 0.03 and 0.04 Å, respectively. This is also reflected in the torsion angle C(21)–O(2)–C(20)–O(1) and C(24)–O(4)–C(23)–O(3) of 1.5(2) and 1.9(2)°, respectively.

The mutual orientation around the ylidic atoms P and C(19) atoms in **5a** may be described as *eclipsed* or *parallel* [1], which means that one of the C(19) substituents (atom C(22)) is in *synperiplanar* conformation with respect to one of the P-bonded phenyl substituents (atom C(7)); the torsion angle C(7)–P–C(19)–C(22) is 5.6(2)°. On the other hand, the analysis of the ylide P–C distances and the C–P–C(19) angle reveals that P–C(13) (1.821(2) Å) and C(13)–P–C(19) (117.34(7)°) are larger than the other values, which could make the C(13) phenyl group the so-called *unique* substituent, which, in combination with the torsion angle C(13)–P–C(19)–C(20) (78.1(2)°) could imply the so-called *planar* or *perpendicular* [1] conformation around the P–C(19) bond. Nevertheless, with the additional lack of the characteristic bend of the ylide substituents at the ylidic carbon C(19) [C(20), C(22)] toward the C(13) phenyl group, the *eclipsed* conformation gives a better description of the conformation around the P–C(19) bond.

The geometry at the phosphorus atom reveals a deformed tetrahedral shape with the largest deformations observed for the bond distance P–C(19) and the bond angle C(13)–P–C(19) (Table 2), which is a common feature of compounds with related structure.

The molecular structure of **5a** is stabilized by intra- and intermolecular C–H···O interactions (Fig. 2), the geometry of which is given in Table 3. Additionally, the *syn* orientation of O(1) atom to P atom favors short intramolecular P···O contacts with a P···O(1) distance of 2.961(2) Å. Every phenyl ring is involved in one intramolecular C–H···O interaction and additional intermolecular contacts of the same type. The adjacent molecules are joined to each other to form two-
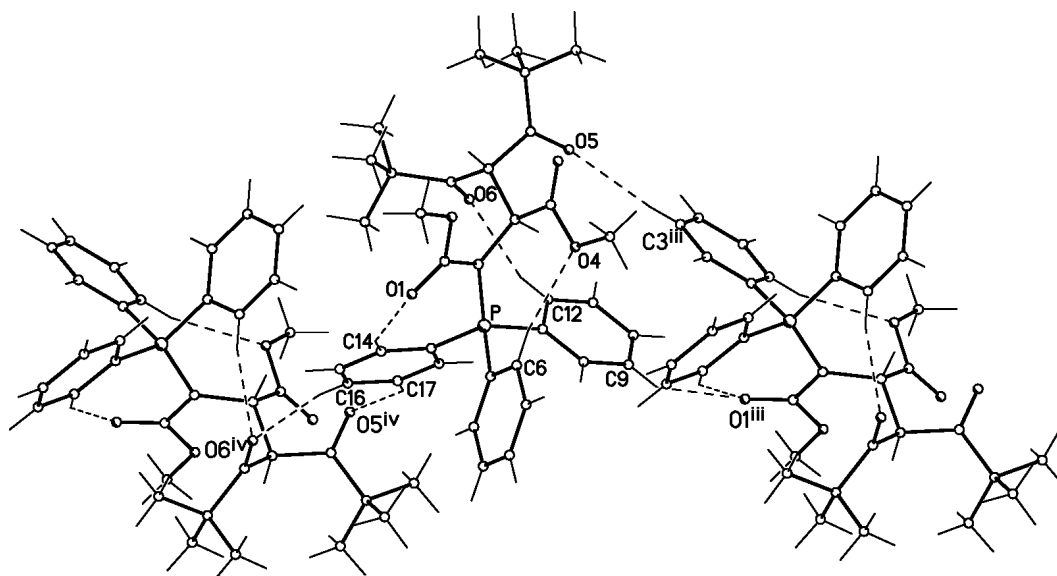
Fig. 2. The arrangement of the ylide molecules in the crystal **5a**. Dashed lines show intra- and intermolecular C–H⋯O close contacts. Symmetry codes are given in Table 3.

Table 3. Geometry of proposed C–H⋯O close contacts for **5a** (Å, °).

D-H⋯A	D-H (Å)	H⋯A (Å)	D⋯A (Å)	D-H⋯A (°)
C(3)-H(3)⋯O(5)^i	0.95	2.65	3.475(2)	146
C(5)-H(5)⋯O(6)^{ii}	0.95	2.54	3.477(2)	170
C(6)-H(6)⋯O(4)	0.95	2.55	3.418(2)	153
C(9)-H(9)⋯O(1)^{iii}	0.95	2.30	3.145(2)	147
C(12)-H(12)⋯O(6)	0.95	2.40	3.279(2)	153
C(14)-H(14)⋯O(1)	0.95	2.35	3.161(2)	143
C(16)-H(16)⋯O(6)^{iv}	0.95	2.46	3.395(2)	170
C(17)-H(17)⋯O(5)^{iv}	0.95	2.50	3.272(2)	138
C(29)-H(29C)⋯O(3)^v	0.98	2.64	3.581(2)	160
C(34)-H(34A)⋯O(2)	0.98	2.64	3.474(2)	143

Symmetry codes: ^{i} $-x, y + 1/2, -z + 1/2$; ^{ii} $x - 1, y, z$; ^{iii} $-x, y - 1/2, -z + 1/2$; ^{iv} $-x + 1, y + 1/2, -z + 1/2$; ^{v} $x + 1/2, -y + 1/2, -z + 1$.

dimensional, folded molecular layers parallel to the (001) plane. The channels formed between the layers are occupied by highly disordered solvent molecules (see Experimental Section).

Concluding remarks

In conclusion, we have developed a convenient, one-pot method for preparing stabilized ylides (**5a – b**) utilising *in situ* generation of the phosphonium salts. Other aspects of this process are under investigation. The X-ray structure of **5a** revealed a number of common features of stabilized ylides: deformed tetrahedral P centre, near planar ylide carbon atom, short $C_{ylide}–C_{\alpha}$ and long C=O distance, and coplanar $P–C_{ylide}–C_{\alpha}–O_{carbonyl}$ conformation, which all indicate the effective electronic delocalization.

Experimental Section

Melting points were measured on an Electrothermal 9100 apparatus and are uncorrected. Elemental analyses were performed using a Heraeus CHN-O-Rapid analyzer. IR spectra were recorded on a Shimadzu IR-460 spectrometer. ^{1}H, ^{13}C and ^{31}P NMR spectra were measured with a BRUKER DRX-500 AVANCE spectrometer at 500, 125 and 202.44 MHz, respectively. **5a** was analyzed with CD (spectropolarimeter Jasco J-600) and GC/MS (EI) (Hewlett Packard gas chromatograph HP 5890II with mass detector HP 5971A).

*Preparation of 2-[1-(2,2-dimethylpropionyl)-3,3-dimethyl-2-oxobutyl]-3-(triphenylphosphoranylidene)succinic acid dimethyl ester (*5a*)*

General procedure

To a magnetically stirred solution of triphenylphosphane (0.066 g, 0.25 mmol) and 2,2,6,6-tetramethyl-3,5-heptanedione (0.05 ml, 0.25 mmol) in dichloromethane (5 ml) was added dropwise a mixture of dimethyl acetylenedicarboxylate (0.03 ml, 0.25 mmol) in dichloromethane (2 ml) at -10 °C over 15 min. The mixture was allowed to warm up to r. t. The mixture was cloudy white, and it was filtered, the filtrate being evaporated to dryness as a white solid. Then colorless crystals were obtained from

dichloromethane/petroleum ether (20 – 25 °C). Yield: 0.14 g (95 %); m. p. 187.0 – 188.7 °C. – IR (KBr): $\nu = 1726, 1626, 1441$ cm^{-1}. – ^{1}H NMR (CDCl$_3$, major rotamer (M) 50.4 % and minor rotamer (m) 49.6 %): $\delta = 1.06, 1.14, 1.22$ and 1.24 (4s, 18 H, CH$_3$), 3.13, 3.53 and 3.59 (3s, 6H, OCH$_3$), 3.74 – 3.80 (m, 1 H, CHCO$_2$Me), 5.90 (1H, d, $^3J_{HH} = 10.36$ Hz) and 6.13 (1H, d, $^3J_{HH} = 9.85$ Hz, CH(COtBu)$_2$); 7.47 – 7.68 (3m, 15H, H$_{arom}$). – ^{13}C NMR (CDCl$_3$): $\delta = 27.41, 27.50, 27.97$ and 28.12 (4 C(CH$_3$)$_3$); 40.73 (d, $^1J_{PC} = 121.63$ Hz) and 42.02 (d, $^1J_{PC} = 128.58$ Hz) (2 P=C); 45.09, 45.28, 45.77 and 45.94 (4C(CH$_3$)$_3$); 49.41 (d, $^2J_{PC} = 13.04$ Hz) and 49.90 (d, $^2J_{PC} = 13.10$ Hz) (2P=C-C); 48.70, 49.90, 51.29 and 51.37 (4 OCH$_3$); 55.94 (d, $^3J_{PC} = 2.77$ Hz) and 56.69 (d, $^3J_{PC} = 5.98$ Hz) (2 CH(COtBu)$_2$); 128.26 – 135.42 (fairly complex, C$_{arom}$); 169.55 (d, $^2J_{PC} = 14.21$ Hz), 171.10 (d, $^2J_{PC} = 19.75$ Hz), 175.21 (d, $^3J_{PC} = 3.20$ Hz) and 175.47 (d, $^3J_{PC} = 4.02$ Hz) (4 CO of esters); 210.00, 210.05, 210.15 and 210.54 (4 CO of ketones). – ^{31}P NMR (CDCl$_3$): $\delta = 23.80, 24.51$. – C$_{35}$H$_{41}$O$_6$P (588.26): calcd. C 71.41, H 7.02; found C 69.73, H 7.98.

Selected data for 2-[1-(2,2-dimethylpropionyl)-3,3-dimethyl-2-oxobutyl]-3-(triphenylphosphoranylidene)succinic acid diethyl ester **5b**

Colorless crystals. – Yield: 0.14 g (91 %); m. p. 169.0 – 171.0 °C. – IR (KBr): $\nu = 1726, 1626, 1487, 1441$ cm^{-1}. – ^{1}H NMR (CDCl3, major rotamer (M) 57.4 % and minor rotamer (m) 42.6 %): $\delta = 1.05, 1.14, 1.23$ and 1.24 (4s, 18 H, CH$_3$); 0.54, 1.12, 1.17 and 1.27 (4t, 6H, $^3J_{HH} = 7.1$ Hz, OCH$_2$C**H**$_3$); 3.53 – 3.60 (m, 1 H, C**H**CO$_2$Et); 3.70 – 3.86, 3.92 – 4.04 and 4.08 – 4.11 (3m, 4 H, OCH$_2$); 5.91 (d, 1 H, $^3J_{HH} = 10.4$ Hz) and 6.13 (d, 1 H, $^3J_{HH} = 10.1$ Hz) (C**H**(COtBu)$_2$); 7.45 – 7.71 (3m, 15 H, H$_{arom}$). – ^{13}C NMR (CDCl$_3$): $\delta = 14.17, 14.19, 14.22$ and 15.01 (4 OCH$_2$**C**H$_3$); 27.38, 27.44, 28.02 and 28.20 (4 C(CH$_3$)$_3$); 40.26 (d, $^1J_{PC} = 121.31$ Hz) and 41.86 (d, $^1J_{PC} = 129.13$ Hz) (2 P=C); 45.04, 45.30, 45.81 and 46.00 (4 **C**(CH$_3$)$_3$); 50.08 (d, $^2J_{PC} = 13.05$ Hz) and 50.59 (d, $^2J_{PC} = 13.14$ Hz) (2P=C-**C**); 57.37, 58.16, 60.49 and 60.52 (4 O**C**H$_2$CH$_3$); 56.02 (d, $^3J_{PC} = 5.66$ Hz) and 56.66 (d, $^3J_{PC} = 5.03$ Hz) (2 **C**H(COtBu)$_2$); 128.16 – 134.32 (fairly complex, C$_{arom}$); 169.17 (d, $^2J_{PC} = 14.46$ Hz), 170.86 (d, $^2J_{PC} = 19.75$ Hz), 174.76 (d, $^3J_{PC} = 3.65$ Hz) and 175.09 (d, $^3J_{PC} = 4.02$ Hz) (4 CO of esters); 210.09, 210.15, 210.20 and 210.74 (4 CO of ketones). – ^{31}P NMR (CDCl$_3$): $\delta = 23.58, 24.30$. – C$_{37}$H$_{45}$O$_6$P (616.30): calcd. C 72.06, H 7.35; found C 69.73, H 7.98.

Crystal structure determination of **5a**

The crystallographic measurement was performed on a Xcalibur PX automated four-circle diffractometer with the graphite-monochromatized Cu-K$_\alpha$ radiation. The data for the crystal were collected at 100(2) K using the Oxford Cryosystems cooler. A summary of the conditions for the data collection and the structure refinement parameters are given in Table 1. The data were corrected for Lorentz and polarization effects. Analytical absorption correction was applied. Data collection, cell refinement, and data reduction and analysis were carried out with the Xcalibur PX software (Oxford Diffraction Poland): CrysAlis CCD and CrysAlis RED, respectively [11]. The structure was solved by direct methods using the SHELXS-97 program [12] and refined by a full-matrix least-squares technique using SHELXL-97 [13] with anisotropic thermal parameters for non-H atoms of the **5a** molecule. All H atoms were placed in calculated positions and were treated as riding atoms, with C–H distances of 0.95 – 1.00 Å, and with U_{iso}(H) values of 1.5 U_{eq}(C) for methyl or 1.2 U_{eq}(C) for aromatic and CH groups. All figures were made using XP program [14]. The absolute configuration was determined by the anomalous dispersion effect [15]. The extinction was also refined with the final extinction coefficient of 0.0034(3).

After the diffraction experiment, the crystal was dissolved in dichloromethane. The optical rotation was not possible to be measured, due to low concentration of the solution (2.17 · 10^{-4} M). Nevertheless, the CD spectrum could be recorded.

In the crystal of **5a**, channels along the *a* axis are present, in which the molecules of the crystallization solvent are located. Since the compound has been recrystallized from dichloromethane/petroleum ether, the voids in the crystals are filled up with the molecules of different type, and of different occupation factors. The GC/MS analysis of the crystal revealed presence of at least five different compounds in the crystal of **5a**, with the most intense chromatographic peak ascribed to *n*-hexane.

To eliminate the possibility that the disorder in the solvent region was due to the immediate chilling of the crystal (by direct putting it in the gas stream of 100 K), the next diffraction experiment was performed (the results of which are presented here), in which the crystal was put in the gas stream of r. t. and then the temperature was slowly decreased (with the rate of 120 K/h). However, this procedure did not prevent the disorder in the crystal. The molecules of the solvent used in the synthesis and crystallization were highly disordered and could not be recognized in the disordered electron density. Therefore, the contribution of the solvent to the diffraction pattern was incorporated in the model using the SQUEEZE option in PLATON [16]. Disordered solvent is present in two channels per unit cell, each located on a 2_1 axis. A total of 89 electrons were found in every channel of about 614 Å^3 each [located at $(x, 1/4, 0)$ and $(x, 3/4, 1/2)$].

Supplementary material

Crystallographic data for the structure have been deposited with the Cambridge Crystallographic Data Centre,

CCDC-606706 and 606707 (with or without SQUEEZE procedure). Copies of the data can be obtained free of charge on application to The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: int.code+(1223)336 – 033; e-mail for inquiry: fileserv@ccdc.cam.ac.uk).

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