Reaction pathways towards 1,1,2,2-tetra'butyldistannanes

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Abstract

1,2-Dichlorotetra'butyldistannane (**1**) is synthesized as a byproduct in the reaction of 'butylmagnesium chloride with tin tetrachloride or by the reaction of di'butyltindihydride with di'butyltin dichloride in the presence of amines. Reaction of **1** with lithium aluminumhydride yielded the dihydrido derivative **2**. The treatment of **2** with bromoform gave the dibromo distannane **3** nearly quantitatively. Reactions of **2** with one equivalent of LDA or KH yielded unsymmetrically substituted alkali metal distannanes of the type $M'Bu_2Sn–Sn(H)'Bu_2$ (**4**, M = Li; **5**, M = K). Reaction of either two equivalents of KH with **2**, or one equivalent of KH with **5** gave the dipotassium distannide **6**. The molecular structure of the dichloro derivative **1** was determined by X-ray diffraction. © 2002 Elsevier Science B.V. All rights reserved.

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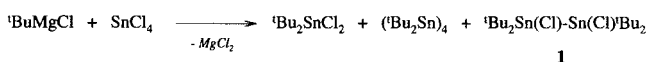
1. Introduction

A large number of hexaorgano substituted distannanes are known. These compounds are widely used in different types of organic and organometallic reactions [1]. However, only a limited number of distanna compounds of type $XR_2Sn–SnR_2X$ are described in the literature in which the substituent X is halogen, alkali metal or hydrogen. Many of these compounds are only known as minor byproducts from various reactions and only a small number could be isolated as pure substances (examples: $XSnR_2–Sn(X)R_2$ (X = Cl, Br, I) [2–18], $HSnR_2–Sn(H)R_2$ [19–21], and $MSnR_2–Sn(M)R_2$ (M = alkali metal) [22,23]). We describe here the synthesis and isolation of a number of symmetrically and unsymmetrically substituted 1,1,2,2-tetra'butyl distannanes.

2. Synthesis and discussions

A relatively convenient access towards the 1,2-dichlorotetra'butyldistannane (**1**) is its formation as a byproduct in the reaction of 'butylmagnesiumchloride with tin tetrachloride [24] (Scheme 1). The main product of this reaction is di'butyltin dichloride. However, **1** and also octa'butylcyclotetrastannane are formed as byproducts in yields of about 30 g related to 1 mol of the starting tin tetrachloride. Alternatively, compound **1** is obtained by reaction of di'butyltindihydride with di'butyltin dichloride in the presence of amines (Eq. (1)). A number of similar reactions have been reported in literature [1–3,15].

$$'Bu_2SnH_2 + 'Bu_2SnCl_2 \xrightarrow[-H_2]{+ amine} \underset{\mathbf{1}}{'Bu_2Sn(Cl)–Sn(Cl)'Bu_2} \tag{1}$$
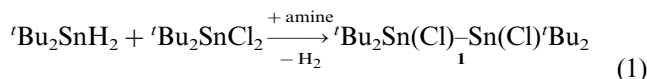
Treatment of **1** with lithium aluminumhydride yielded the dihydrido derivative **2** (Eq. (2)).

$$'BuMgCl + SnCl_4 \xrightarrow[-MgCl_2]{} 'Bu_2SnCl_2 + ('Bu_2Sn)_4 + \underset{\mathbf{1}}{'Bu_2Sn(Cl)\text{-}Sn(Cl)'Bu_2}$$

Scheme 1. Reaction of 'butylmagnesium chloride with tin tetrachloride.

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Scheme 2. Reaction of **2** with LDA or KH.

Fig. 1. Crystal structure of **1** showing 30% probability displacement ellipsoids and the atom numbering. Hydrogen atoms bonded to carbon have been removed for clarity.

Table 1
Crystallographic data for compound **1**

Empirical formula	$C_{16}H_{36}Cl_2Sn_2$
Formula weight	536.73
Crystal system	Monoclinic
Space group	$P2(1)/n$
Unit cell dimensions	
a (Å)	10.6984(6)
b (Å)	14.7300(8)
c (Å)	15.3657(9)
β (°)	106.9650(10)
V (Å^3)	2316.1(2)
Z	4
D_{calc} (g cm^{-3})	1.539
Absorption coefficient (mm^{-1})	2.380
Crystal size (mm)	$0.10 \times 0.10 \times 0.04$
$F(000)$	1064
Diffractometer	CCD area detector
Theta range for data collection (°)	1.96–28.28
Temperature (K)	86(2)
Wavelength (Å)	0.71073
Reflections collected	15 418
Independent reflections	5624 [$R_{int} = 0.0577$]
Completeness to theta = 28.28°	97.8%
Absorption correction	Semi-empirical from equivalents
Max/min transmission	0.9108/0.7967
R_1 ($I > 2\sigma(I)$)	0.0424
wR_2 (all data)	0.0867

$$^{t}Bu_2Sn(Cl)\text{–}Sn(Cl)^{t}Bu_2 \xrightarrow[-\text{LiCl/AlCl}_3]{+\text{LiAlH}_4} {}^{t}Bu_2Sn(H)\text{–}Sn(H)^{t}Bu_2$$

$$\underset{\mathbf{1}}{} \qquad \underset{\mathbf{2}}{}$$

(2)

Compound **2** was mentioned by Maire and Dufermont [21] in 1967 as one of the products of the thermal decomposition of di^{t}butyltindihydride (^{t}Bu$_2$SnH$_2$). However, as a synthetic target we were able to isolate it by a fractionated distillation with a yield of about 55% (Eq. (2)).

The treatment of **2** with bromoform gave the 1,2-dibromodistannane **3** as a colorless solid in almost quantitative yield (Eq. (3)). A reaction temperature of 0 °C or lower is strictly required to avoid Sn–Sn bond cleavage in this reaction. A similar conversion is possible by using chloroform instead of bromoform yielding the dichloro derivative **1**.

$$^{t}Bu_2Sn(H)\text{–}Sn(H)^{t}Bu_2 \xrightarrow[-2CH_2X_2]{+2CHX_3} {}^{t}Bu_2Sn(X)\text{–}Sn(X)^{t}Bu_2$$

$$\underset{\mathbf{2}}{} \qquad \underset{\mathbf{1},\ X=Cl}{}$$

(3)

Reactions of **2** with one equivalent of lithium diisopropylamide (hereafter referred to as LDA) or KH [25] in THF yielded the unsymmetrically substituted alkali metal distannanes of type M^{t}Bu$_2$Sn–Sn(H)^{t}Bu$_2$ (**4**, M = Li; **5**, M = K). Reaction of two equivalents of potassium hydride with the tetraorganodistannane derivative **2**, or one equivalent of KH with the potassium stannide **5** gave the dipotassium distannide **6** (Scheme 2). Similar reactions with an excess of LDA gave only the monometalated stannide **4**. There is no evidence in the resulting products for the existence of a dilithiated distannide. Although there is only little experimental evidence, we believe that the different reaction products obtained with either LDA or potassium hydride are the result of (i) the higher basicity of the potassium hydride, and (ii) the formation of hydrogen is shifting the reaction in favor of **6**.

The molecular structure of compound **1** as determined by single X-ray diffraction is shown in Fig. 1. Unit cell data, refinement details, and selected interatomic details are summarized in Tables 1 and 2. Both tin centers are four coordinate with two Sn–C, one Sn–Cl and one Sn–Sn contact. The Sn–Sn bond distance is 2.829(1) Å and therefore in a similar range as observed for ^{t}Bu$_3$Sn–Sn^{t}Bu$_3$ with 2.894 Å [4]. Sn–Cl distances range from 2.393(1) to 2.395(5) Å and the Sn–C contacts are observed between 2.182(5) and 2.196(5) Å. The tin atoms display approximate tetrahedral geometry with carbon–tin–carbon angles of 117.6(2) and 115.7(2)°, and chloro–tin–tin angles of 103.81(3) and 103.13(3)°. These slight distortions from ideal geometry are due to the different steric demands of the ligands. Compound **1** displays the expected staggered conformation.

Table 2
Selected bond lengths (Å) and bond angles (°) for compound **1**

Bond lengths			
Sn(1)–C(5)	2.187(4)	Sn(2)–C(9)	2.182(5)
Sn(1)–C(1)	2.196(5)	Sn(2)–C(13)	2.190(5)
Sn(1)–Cl(1)	2.395(1)	Sn(2)–Cl(2)	2.393(1)
Sn(1)–Sn(2)	2.8299(5)		
Bond angles			
C(5)–Sn(1)–C(1)	117.61(17)	C(9)–Sn(2)–C(13)	115.69(18)
C(5)–Sn(1)–Cl(1)	103.15(12)	C(9)–Sn(2)–Cl(2)	102.77(13)
C(1)–Sn(1)–Cl(1)	101.01(13)	C(13)–Sn(2)–Cl(2)	102.72(14)
C(5)–Sn(1)–Sn(2)	113.42(12)	C(9)–Sn(2)–Sn(1)	114.27(12)
C(1)–Sn(1)–Sn(2)	115.01(13)	C(13)–Sn(2)–Sn(1)	115.60(13)
Cl(1)–Sn(1)–Sn(2)	103.81(3)	Cl(2)–Sn(2)–Sn(1)	103.13(3)

Table 3 summarizes the ^{119}Sn-NMR data of compounds **1**–**6** in comparison with the previously described hexa^{t}butyldistannane [26]. The numerical values of the $^1J(^{119}$Sn–^{119}Sn) coupling constants are given without determination of the sign. In accordance with empirical predictions, the $^1J_{Sn-Sn}$ coupling constants depends mainly on the s-character at the tin atoms and inductive effects of the substituents [27]. Such a dependence is followed by the halogen-, hydrido-, ^{t}butyl- and dipotassio-substituted compounds **1**, **3**, **2**, **7** and **6** (decreasing the $^1J(^{119}$Sn–^{119}Sn) coupling constants, see Table 3). However, the magnitude of the unexpected large coupling constants for the monolithium and monopotassium substituted compounds **4**–**5** (^{t}Bu$_2$Sn-(M)–Sn(H)^{t}Bu$_2$) is still unexplained regardless of what the sign of the numerical value may be.

Reaction of the monometalated species **4** and **5** with equimolar amounts of DCl/D$_2$O yielded the monodeuterated ^{t}Bu$_2$Sn(D)–Sn(H)^{t}Bu$_2$ (95% in a mixture with **2**), while reaction of **6** with DCl/D$_2$O gives the 1,2-dideutero-1,1,2,2-tetra^{t}butyldistannane (^{t}Bu$_2$Sn(D)–Sn(D)^{t}Bu$_2$, 80% in a mixture with **2** and ^{t}Bu$_2$Sn(D)–Sn(H)^{t}Bu$_2$). These conversion rates were determined by ^{119}Sn-NMR spectroscopy.

Reactions of the alkali metal substituted compounds **4**–**6** with different difunctionalized organosilanes or organogermanes (R$_2$SiX$_2$, R$_2$GeX$_2$; X = Cl, OSO$_2$CF$_3$) are complex and will be discussed in a forthcoming publication.

3. Experimental

All reactions were carried out under an atmosphere of inert gas (N$_2$ or Ar) using Schlenk techniques. All solvents were dried by standard methods and freshly distilled prior to use. Lithiumdiisopropylamide (LDA) was freshly prepared according to published procedures [28]. All other chemicals used as starting materials were obtained commercially and used without further purification. NMR spectra were recorded on Bruker DRX 300 (^{119}Sn: 111.92 MHz) and DPX 400 (^{1}H: 400.15 MHz, ^{13}C: 100.63 MHz, ^{119}Sn: 149.21 MHz) spectrometers and referred against Me$_4$Si for ^{1}H, ^{13}C or Me$_4$Sn for ^{119}Sn. Mass spectra were obtained using a MAT 8200 mass spectrometer. Elemental analyses were performed on a LECO-CHNS-932 analyzer.

3.1. Synthesis of 1,2-dichlorotetra^{t}butyldistannane ^{t}Bu$_2$Sn(Cl)–Sn(Cl)^{t}Bu$_2$ (**1**) [24]

A solution of ^{t}butylchloride (231 g, 275 ml, 2.5 mol) and 500 ml of THF is placed in a dropping funnel and added dropwise to 61 g (2.5 mol) of magnesium and 300 ml of THF in a 2 l three-neck flask equipped with a mechanical stirrer and a reflux condenser. The reaction mixture was stirred at room temperature (r.t.) for another 10 h and then heated at reflux for another 4 h. The reaction mixture was separated from unreacted magnesium by decanting the liquids in a dropping funnel. A 4 l three necked flask with stopper, a mechanical stirrer and a reflux condenser with nitrogen inlet was charged with 260 g (1 mol) tin tetrachloride and

Table 3
^{119}Sn-NMR data of compounds of type ^{t}Bu$_2$Sn(X)–Sn(Y)^{t}Bu$_2$

Number	Substituents		^{119}Sn-NMR chemical shifts		$^1J_{Sn-Sn}$ coupling constants	
	X	Y	δ_{Sn-X} (ppm)	δ_{Sn-Y} (ppm)	^{119}Sn–^{117}Sn (Hz)	^{119}Sn–^{119}Sn (Hz)
1	Cl	Cl	112.9	–	1559	–
3	Br	Br	92.7	–	~1500	–
2	H	H	−83.7	–	1260	–
7 [26]	^{t}Bu	^{t}Bu	−3.4	–	<60	–
4	Li	H	107.8 [a]	−48.4 [b]	6610	6930
5	K	H	134.8 [c]	−43.2 [d]	7320	7665
6	K	K	16.8	–	45	–

[a] $^3J_{Sn-H}$: 28 Hz.
[b] $^1J_{Sn-H}$: 793 Hz; $^3J_{Sn-H}$: 45 Hz.
[c] $^3J_{Sn-H}$: 24 Hz.
[d] $^1J_{Sn-H}$: 760 Hz; $^3J_{Sn-H}$: 42 Hz.

500 ml THF, and the Grignard-solution was added dropwise. The reaction mixture was heated under reflux for 2 h. After being cooled to r.t. the solvent was removed in vacuo. The residue was placed in a Soxhlet apparatus and extracted with *n*-hexane for 72 h. The solvent was removed in vacuo (25 torr) and $^tBu_2SnCl_2$ is separated by sublimation at 80–100 °C/0.1 torr. Dissolving the residue in 200 ml of *n*-hexane. Filtration (G3) and subsequent removal of the solvent in vacuo gave 20 g of a colorless solid. Recrystallization from *n*-pentane or *n*-hexane yielded 12.5 g (5%) of **1** as a colorless solid. m.p.: 62 °C.

^{1}H-NMR (400.13 MHz, CDCl$_3$, ppm): 1.41 [s, 36H, $^3J(^1H-^{119/117}Sn) = 88/84$ Hz, $^4J(^1H-Sn) = 7$ Hz]; ^{13}C{^{1}H}-NMR (100.63 MHz, CDCl$_3$, ppm): 30.4 [Sn(C(CH$_3$)$_3$)$_2$, $^2J(^{13}C-Sn) =$ not observed], 35.1 [Sn(*C*(CH$_3$)$_3$)$_2$, $^1J(^{13}C-^{119/117}Sn) = 269/257$ Hz, $^2J(^{13}C-Sn) = 11$ Hz]; ^{119}Sn{^{1}H}-NMR (111.92 MHz, D$_2$O-cap./*n*-hexane, ppm): 112.9, $^1J(^{119}Sn-^{117}Sn) = 1559$ Hz; MS (EI) *m/z* (%): 536 (2) [M$^+$], 479 (4) [M$^+ -$ ^tBu], 423 (7) [M$^+ - 2 \times {}^tBu$], 309 (4) [M + $-4 \times {}^tBu$], 235 (21) [Sn$^tBu_2^+$], 57 (100) [$^tBu^+$]; EA calc. C$_{16}$H$_{36}$Cl$_2$Sn$_2$ (536.79): C, 35.80; H, 6.76. found: C, 35.3; H, 6.9%.

3.2. Synthesis of 1,1,2,2-tetra^{t}butyldistannane $^tBu_2Sn(H)$–$Sn(H)^tBu_2$ (2)

A solution of **1** (15 g, 28 mmol) in 250 ml of Et$_2$O was added dropwise to a cooled suspension (0 °C) of LiAlH$_4$ (1.1 g, 30 mmol) in 250 ml of Et$_2$O. The reaction mixture was stirred for 1 h at 0 °C and for 36 h at r.t. The excess LiAlH$_4$ was hydrolyzed with 100 ml of oxygen-free water at 0 °C. The solution was warmed up to r.t., the organic layer separated and treated with 100 ml of a saturated solution of sodium potassium tartrate. The organic layer was separated again and dried over calcium chloride. After filtration (G3) the solvent was removed in vacuo (30 torr). The resulting residue was purified by distillation (120 °C, $p = 10^{-3}$ torr) yielding 6.93 g (53%) **4** as a colorless oil.

^{1}H-NMR (400.13 MHz, C$_6$D$_6$, ppm): 1.37 [s, 36H, Sn(C(CH$_3$)$_3$)$_2$, $^3J(^1H-Sn) = 69$ Hz], 5.38 [s, 2H, Sn(C(CH$_3$)$_3$)$_2$*H*, $^1J(^1H-^{119/117}Sn) = 1260/1202$ Hz, $^2J(^1H-Sn) = 83$ Hz]; ^{13}C{^{1}H}-NMR (100.63 MHz, C$_6$D$_6$, ppm): 28.6 [Sn(*C*(CH$_3$)$_3$)$_2$, $^1J(^{13}C-^{119/117}Sn) = 314/300$ Hz, $^2J(^{13}C-Sn) = 30$ Hz], 33.6 [Sn(C(*C*H$_3$)$_3$)$_2$, $^2J(^{13}C-Sn) =$ not observed]; ^{119}Sn{^{1}H}-NMR (111.92 MHz, D$_2$O-cap./*n*-hexane, ppm): −83.7, $^1J(^{119}Sn-^{117}Sn) = 1260$ Hz; MS (EI) *m/z* (%): 468 (21) [M$^+$], 411 (36) [M$^+ - {}^tBu$], 355 (87) [M$^+ - 2 \times {}^tBu$], 297 (55) [M$^+ - 3 \times {}^tBu$], 241 (30) [M$^+ - 4 \times {}^tBu$], 177 (28) [SnH$^tBu^+$], 57 (100) [$^tBu^+$]; IR (Nujol) $v(Sn-H) = 1777$ cm^{-1}; EA calc. for C$_{16}$H$_{38}$Sn$_2$ (467.90): C, 41.07; H, 8.19. found: C, 41.2; H, 8.2%.

3.3. Synthesis of 1,2-dibromotetra^{t}butyldistannane $^tBu_2Sn(Br)$–$Sn(Br)^tBu_2$ (3)

3.85 mmol CHBr$_3$ or CHCl$_3$ dissolved in 10 ml of THF are dropwise added to a cooled solution (−20 °C) of 1.8 g (3.85 mmol) **1** ($^tBu_2SnH)_2$ in 20 ml of THF. The reaction mixture was stirred at −20°C for 4 h. The solution was slowly warmed up to r.t. and the solvent removed in vacuo. The resulting crude product was recrystallized from chloroform to yield 1.9 g (80%) of **3** or 1.45 (70%) of **2** as colorless solids. m.p.: 73–74 °C.

MS (EI) *m/z* (%): 625 (20) [M$^+$], 545 (10) [M$^+ -$ Br], 558 (75) [M$^+ - {}^tBu$], 511 (50) [M$^+ - 2 \times {}^tBu$], 397 (25) [M$^+ - 4 \times {}^tBu$], 80 (20) [Br$^+$], 57 (100) [$^tBu^+$]; EA calc. for C$_{16}$H$_{36}$Br$_2$Sn$_2$ (625.68): C, 30.71; H, 5.8. found: C, 32.2; H, 5.6%.

3.4. Potassium di^{t}butyl(di^{t}butylstannyl)stannide (4)

2.0 g (4.25 mmol) **1** ($^tBu_2SnH)_2$ are added dropwise to a suspension of 0.17 g (4.25 mmol) KH in 10 ml of THF at r.t. After the hydrogen development was complete, the reaction mixture was stirred for 2 h during which the color of the solution changed to yield a transparent orange liquid. The reaction mixture was examined by ^{119}Sn-NMR spectroscopy (conversion rate determined by ^{119}Sn-NMR spectroscopy: > 98%, see Table 3).

3.5. Lithium di^{t}butyl(di^{t}butylstannyl)stannide (5)

A solution of 2.14 mmol freshly prepared LDA was added dropwise to a cooled solution (−60 °C) of 1.0 g (2.14 mmol) **1** ($^tBu_2SnH)_2$ dissolved in 20 ml of THF. The reaction mixture was stirred for 15 min at −60°C. The solution was slowly warmed to r.t. and stirred for 2 h. The reaction mixture was examined by ^{119}Sn-NMR spectroscopy (conversion rate determined by ^{119}Sn-NMR spectroscopy: > 98%, see Table 3).

3.6. 1,2-Dipotassium tetra^{t}butyldistannide

1.0 g (2.14 mmol) **1** ($^tBu_2SnH)_2$ are added dropwise to a suspension of 0.4 g (10 mmol) KH in 15 ml of THF at r.t. The reaction mixture was stirred for 14 h during which the color of the solution changed from orange (after 2 h) to deep red (12 h). Subsequently, the mixture was filtered (G3) and 75% of the solvent was removed in vacuo. The residue was examined by ^{1}H-, ^{13}C-, and ^{119}Sn-NMR spectroscopy (yield determined by NMR spectroscopy: > 99%).

^{1}H-NMR (400.13 MHz, C$_6$D$_6$–THF (1:1 mixture), ppm): 1.7 ppm [Sn–*CMe*, $^3J(^{119}Sn-^1H)$: 29 Hz], 1.61 [THF, (O–CH$_2$–*CH*$_2$–)], 3.45 [THF, (O–*CH*$_2$–CH$_2$–)]. ^{13}C{^{1}H}-NMR (100.63 MHz, C$_6$D$_6$–THF (1:1 mixture), ppm) 24.3 ppm [4 C, Sn(*C*(CH$_3$)$_3$), $^1J(^{119}Sn-^{13}C$):

137 Hz], 36,1 [12 C, $Sn(C(CH_3)_3)$, $^2J(^{13}C–Sn) =$ not observed], 25.3 [THF, $(O–CH_2–CH_2–)$], 66.8 [THF, $(O–CH_2–CH_2–)$]. $^{119}Sn\{^1H\}$-NMR (111.92, D_2O-cap.– THF, ppm): see Table 3.

3.7. Reaction of **1** with two equivalents of LDA

A solution of 2.32 mmol freshly prepared LDA was added dropwise to a cooled solution (-60 °C) of 0.54 g (1.16 mmol) **1** ($^tBu_2SnH)_2$ dissolved in 10 ml of THF. The reaction mixture was stirred for 15 min at -60°C. The solution was slowly warmed to r.t. and stirred for 2 h. The reaction mixture was examined by ^{119}Sn-NMR spectroscopy (conversion rate determined by ^{119}Sn-NMR spectroscopy: $> 97\%$).

$^{119}Sn\{^1H\}$-NMR (111.92, D_2O-cap.–THF, ppm): $-$ 48,4 ppm [1 Sn, $^1J(^{119}Sn–^{117}Sn)$: 6618 Hz, $^1J(^{119}Sn–^{119}Sn)$: 6930 Hz, $^1J(^{119}Sn–^1H)$: 793 Hz], 107.8 ppm [1 Sn, $^1J(^{119}Sn–^{117}Sn)$: 6612 Hz, $^1J(^{119}Sn–^{119}Sn)$: 6930 Hz].

3.8. X-ray crystallographic studies

The crystal was mounted on the diffractometer as described previously [29]. Intensity data were collected at -182 °C with graphite monochromated Mo–K_α radiation ($\lambda = 0.71073$ Å), using a Siemens SMART system, complete with three-circle goniometer and CCD detector operating at -54 °C. Details regarding instrumentation and data treatment have been reported previously [30]. An absorption correction was applied utilizing the program SADABS [31]. The crystal structure was solved by Direct Methods, as included in the SHELXTL-Plus program package [32]. Hydrogen atoms were placed geometrically and refined using a riding model with U_{iso} constrained at 1.5 for methyl groups times U_{eq} of the carrier C atom. The structure was refined by full-matrix least-squares refinement on F^2 (SHELX-93) [33]. Scattering factors were those provided with the SHELX program system. All non-hydrogen atoms, with the exception of some disordered or restrained positions were refined anisotropically.

4. Supplementary material

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Centre, CCDC No. 169217. 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).

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