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Synthesis and characterization of new pentafluorophenylbismuth(V) derivatives [1]

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Abstract

The reactions of tris(pentafluorophenyl)bismuth difluoride with chlorotrimethylsilane, trimethylsilicontrifluoroacetate and trimethylsilicontrifluoromethanesulphonate gives tris(pentafluorophenyl)bismuth dichloride, bis(trifluoroacetate) and bis(trifluoromethanesulphonate) in nearly quantitative yields. The products were identified by ^{19}F and $^{13}C\{^{19}F\}$ NMR spectroscopy. The products of the thermal decomposition are described. © 1997 Elsevier Science S.A.

Keywords: Decomposition; Ligand exchange reactions; NMR spectra; Pentafluorophenylbismuth(V) compounds

1. Introduction

Non-fluorinated arylbismuth(V) compounds have been known for more than 100 years since Michaelis and coworkers prepared triphenylbismuth dihalides by oxidizing $Bi(C_6H_5)_3$ with elemental chlorine and bromine [1,2]. The preparations, properties and applications of a large number of these compounds have been summarized in several reviews [3,4]. Since 1985, Barton et al. [5–7] and Dodonov et al. [8–10] have described many applications of non-fluorinated arylbismuthanes in organic synthesis, especially in ligand exchange reactions and O- and C-phenylation. The importance of arylbismuth(III) and arylbismuth(V) compounds as strong arylating reagents is still increasing and has been extended to $Bi(C_6F_5)_3$ as a versatile C_6F_5 group transfer reagent in metathesis reactions with fluorine compounds [11,12].

Hitherto tris(pentafluorophenyl)bismuth(V) difluoride has been described [13] as the only member of the class of $Bi(C_6F_5)_3X_2$ compounds. It is unstable in solution and as a solid. In 1989, we obtained $Bi(C_6F_5)_3F_2$ from the reaction of $Bi(C_6F_5)_3$ with XeF_2 or fluoroxenon metallates. Similar results have been obtained by Schmuck and Seppelt [14] who also described a synthetic route to penta-arylbismuth compounds with two different aryl groups, e.g. $Bi(C_6H_5)_3(C_6F_5)_2$ [14,15].

The direct synthesis of pentafluorophenylbismuth(V) derivatives starting with $Bi(C_6F_5)_3$ and a xenon-oxo compound gave only spectroscopic evidence for $Bi(C_6F_5)_3(OTeF_5)_2$ [16]; reactions with xenonperfluorocarboxylates have not given identifiable products to date [17].

In 1971, de Ketelaere et al. [18] reported the synthesis of partly fluorinated tris(fluorophenyl)bismuthanes, such as $Bi(3\text{-}FC_6H_4)_3$ and $Bi(4\text{-}FC_6H_4)_3$. Tris(fluorophenyl)-bismuth(III) derivatives with the 2,6-difluorophenyl and 2,4,6-trifluorophenyl ligands have been prepared by our group [19]. Tris(2,6-difluorophenyl)bismuth(V) dihalides were prepared by the oxidation of $Bi(2,6\text{-}F_2C_6H_3)_3$ with XeF_2 or elemental Cl_2 [20]. $Bi(2,6\text{-}F_2C_6H_3)_3(OCOCF_3)_2$ and $Bi(2,6\text{-}F_2C_6H_3)_3(OSO_2CF_3)_2$ can be synthesized via ligand exchange reaction of the difluoride with the corresponding trimethylsilyl derivatives or reaction of the dichlorides with the appropriate silver salts. The successful use of trimethylsilicon derivatives has already been demonstrated in exchange reactions in iodine and xenon chemistry [21].

In this paper, we report the preparation of new pentafluorophenylbismuth(V) derivatives obtained from the metathesis reactions of $Bi(C_6F_5)_3F_2$ with $(CH_3)_3SiCl$, $(CH_3)_3SiOCOCF_3$ and $(CH_3)_3SiOSO_2CF_3$.

2. Results and discussion

Continuing our work on the ligand exchange reactions of trimethylsilicon derivatives with fluorophenylbismuth com-

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[1] Dedicated to Professor Alois Haas on the occasion of his 65th birthday.

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pounds [20], we reacted tris(pentafluorophenyl)bismuth difluoride with chlorotrimethylsilane and trimethylsilylesters.

The reactions proceeded in dichloromethane solution and were monitored by ^{19}F NMR spectroscopy. The formation of fluorotrimethylsilane was an indicator for the course of the reaction. Exchanges were complete within 5 days at -40 °C ($(CH_3)_3SiOSO_2CF_3$ and $(CH_3)_3SiOCOCF_3$) and within 2 days at -20 °C ($(CH_3)_3SiCl$)

$$Bi(C_6F_5)_3F_2 + 2(CH_3)_3SiOR \xrightarrow[-40\ ^\circ C]{CH_2Cl_2} Bi(C_6F_5)_3(OR)_2 + 2(CH_3)_3SiF \quad (1)$$

$$Bi(C_6F_5)_3F_2 + 2(CH_3)_3SiCl \xrightarrow[-20\ ^\circ C]{CH_2Cl_2} Bi(C_6F_5)_3Cl_2 + 2(CH_3)_3SiF \quad (2)$$

where $R \equiv SO_2CF_3$ or $COCF_3$.

All volatile compounds were distilled off in vacuo; the new compounds were obtained as analytically pure yellow solids in nearly quantitative yields. They were stored in Schlenk tubes in a dry nitrogen atmosphere for several weeks at -30 °C without noticeable decomposition. Fluorophenylbismuth(V) derivatives appear to be insensitive towards daylight.

Table 1
Decomposition points (°C) of selected triarylbismuth(V) compounds

	$Ar \equiv 2,6-F_2C_6H_3$ [a]	$Ar \equiv C_6F_5$	$Ar \equiv C_6H_5$
$BiAr_3F_2$	207	155–162[b]	>190[c,f]
$BiAr_3Cl_2$	131	42	159–160[c]
$BiAr_3(OSO_2CF_3)_2$	119	-15[g]	155[d]
$BiAr_3(OCOCF_3)_2$	151	83	133[e]

[a] Ref. [20].
[b] Ref. [14].
[c] Ref. [1].
[d] Ref. [22].
[e] Ref. [23].
[f] Melting point, 127 °C.
[g] In CH_2Cl_2.

$Bi(C_6F_5)_3Cl_2$ and $Bi(C_6F_5)_3(OCOCF_3)_2$ are stable solids at room temperature. As a solid, the dichloride decomposes at 42 °C and the bis(trifluoroacetate) at 83 °C. In CH_2Cl_2 solution, both compounds are stable up to the boiling point of CH_2Cl_2 (40 °C). $Bi(C_6F_5)_3(OSO_2CF_3)_2$ decomposes in dichloromethane solution below -15 °C (in acetonitrile solution between 0 and $+10$ °C) to give $Bi(C_6F_5)_3$, $C_6F_5OSO_2CF_3$, $Bi(C_6F_5)_2OSO_2CF_3$ and several unidentified fluorine-containing compounds. All perfluorophenylbismuth(V) derivatives decompose according to

$$Bi(C_6F_5)_3X_2 \xrightarrow{\Delta} Bi(C_6F_5)_3 + C_6F_5X + Bi(C_6F_5)_2X + \ldots$$

where $X \equiv Cl$, $OCOCF_3$ or OSO_2CF_3.

In comparison with the corresponding tris(2,6-difluorophenyl)bismuth(V) compounds, the new pentafluorophenyl derivatives exhibit a significant decrease in thermal stability (see Table 1), which may be caused by the general electron-withdrawing influences of each of the five ligands.

The new tris(pentafluorophenyl)bismuth(V) derivatives were characterized by ^{19}F and $^{13}C\{^{19}F\}$ NMR spectroscopy. The ^{19}F NMR data of the new compounds and of some 2,6-difluorophenylbismuth derivatives are summarized in Table 2.

In general, the oxidation of $Bi(C_6F_5)_3$ to $Bi(C_6F_5)_3X_2$ produces a significant high-field shift of the F2,6 atoms of approximately 9 ppm. However, the resonances of the F4 atom and the F3,5 atoms are deshielded by approximately 10 ppm and 5 ppm respectively.

We have already mentioned this effect [13,20], which appears to be general for fluoroaryl derivatives of all main group V–VII elements [21,25,26]. These results demonstrate that ^{19}F NMR data are a very helpful tool for the rapid identification of fluorine-containing arylelement derivatives in different oxidation states.

Table 2
^{19}F NMR data of tris(fluorophenyl)bismuth compounds (ppm)

Compound	δ(F2,6)	δ(F4)	δ(F3,5)	$\delta(R_f)$	Solvent
$Bi(2,6-F_2C_6H_3)_3$ [a]	-89.9				CD_3CN
$Bi(2,6-F_2C_6H_3)_3Cl_2$ [b]	-98.7				CD_3CN
$Bi(2,6\text{-}F_2C_6H_3)_3F_2$ [b]	-98.0			-75.5	CD_3CN
$Bi(2,6\text{-}F_2C_6H_3)_3(OCOCF_3)_2$ [b]	-97.6			-74.5	$(C_2H_5)_2O$
$Bi(2,6\text{-}F_2C_6H_3)_3(OSO_2CF_3)_2$ [b]	-96.8			-78.7	CH_2Cl_2
$Bi(C_6F_5)_3$ [c]	-117.5	-152.4	-160.4		CH_3CN
$Bi(C_6F_5)_3Cl_2$	-127.0	-142.3	-154.3		$CDCl_3$
$Bi(C_6F_5)_3F_2$ [d]	-126.9	-143.3	-155.2	-59.0	CH_3CN
$Bi(C_6F_5)_3(OCOCF_3)_2$	-125.1	-140.6	-154.3	-74.9	$CDCl_3$
$Bi(C_6F_5)_3(OSO_2CF_3)_2$	-123.9	-135.2	-152.0	-77.7	$CDCl_3$

[a] Ref. [19].
[b] Ref. [20].
[c] Ref. [24].
[d] Ref. [14].

Table 3
^{19}F NMR data of $Bi(C_6F_5)_3(OCOCF_3)_2$ in different solvents (ppm)[a]

Solvent	DN[b]	δ(F2,6)	δ(F4)	δ(F3,5)	$\delta(CF_3)$
$CDCl_3$	0.1	− 125.1	− 140.6	− 154.3	− 74.9
CH_2Cl_2	–	− 125.5	− 141.7	− 155.3	− 75.5
$C_6H_5NO_2$	4.4	− 125.7	− 142.6	− 155.7	− 75.6
CH_3CN	14.1	− 125.9	− 142.7	− 156.3	− 75.7
$(C_2H_5)_2O$	19.2	− 126.4	− 142.1	− 156.2	− 76.4

[a] In THF (DN 20.0), DMF (DN 24.0) and DMSO (DN 29.8), only resonances of decomposition products are detected.
[b] Donor number, taken from Ref. [27].

The fluorophenylbismuth compounds $Bi(Ar_f)_3X_2$ ($Ar_f \equiv C_6F_5$, 2,6-$F_2C_6H_3$) must be considered to be stronger Lewis acids than the non-fluorinated analogues. One argument for this consideration is the strong dependence of the chemical shift of the BiF_2 resonance of $Bi(C_6F_5)_3F_2$ and $Bi(2,6\text{-}F_2C_6H_3)_3F_2$ on the donor properties of the solvent [13,20]. The ^{19}F NMR spectra of $Bi(C_6F_5)_3(OCOCF_3)_2$, recorded in different solvents, show that the fluorine chemical shift is dependent on the donor properties of the solvent. The resonances of the F2,6, F4 and F3,5 atoms show a characteristic shift to higher field with increasing donor number (Table 3). Even the chemical shift of the CF_3CO_2 resonance appears to be dependent on the donor number of the solvent. Compared with the data measured for the difluoride [13], these changes in chemical shift are unambiguously indicative of a Lewis acid. Therefore a mainly covalent character for the bismuth–trifluoroacetate bond is assumed, although similar changes in chemical shifts are also observed in the ^{19}F NMR spectra of, for exemple, more ionic bis(pentafluorophenyl)iodine salts [25].

In solvents with stronger donor properties than $(C_2H_5)_2O$, i.e. tetrahydrofuran (THF), dimethylsulphoxide (DMSO) and *N*,*N*-dimethylformamide (DMF), decomposition into $Bi(C_6F_5)_3$ and further unidentified fluorine-containing products is observed immediately after adding the solvent to the solid.

In Fig. 1, a typical $^{13}C\{^{19}F\}$ NMR spectrum of a pentafluorophenylbismuth compound is given. Several low intensity resonances covering the region from 137.0 to 148.0 ppm are assigned to decomposition products in solution (e.g. $Bi(C_6F_5)_3$, $Bi(C_6F_5)_2X$ and C_6F_5X).

A comparison of the $^1J(^{19}F-^{13}C)$ and $^2J(^{19}F-^{13}C)$ couplings of covalent trifluoroacetates (e.g. $(CH_3)_3SiOCOCF_3$) and ionic trifluoroacetates ($AgOCOCF_3$) exhibits an increase in the $^1J(^{19}F-^{13}C)$ value (285 Hz vs. 293 Hz) accompanied by a decrease in the $^2J(^{19}F-^{13}C)$ value (42 Hz vs. 35 Hz) (Table 4). The absolute values measured for $Bi(C_6F_5)_3(OCOCF_3)_2$ (283 Hz, 36 Hz) indicate that the character of the Bi–O bonds is neither purely ionic nor purely covalent.

However, the results of the molecular weight determinations show that $Bi(C_6F_5)_3X_2$ ($X \equiv Cl$, $OCOCF_3$) dissolve in acetonitrile as molecular species. The values determined agree with the calculated value, within the accuracy of the measurement, indicating an undissociated species with presumably covalent character of the Bi–Cl and Bi–O bonds.

3. Experimental details

^{19}F NMR spectra were recorded on a Bruker FT-NMR AC 200 spectrometer operating at 188.3 MHz. ^{13}C and $^{13}C\{^{19}F\}$ NMR spectra were obtained on a Bruker FT-NMR AMX 300 spectrometer at 75.5 MHz. $(CH_3)_4Si$ (^{13}C) and

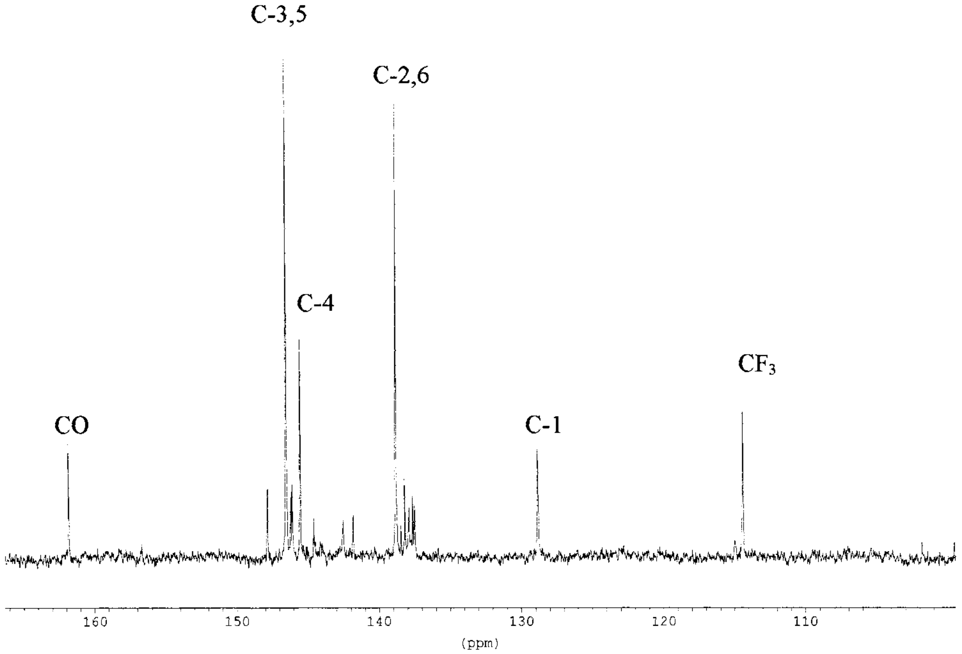

Fig. 1. $^{13}C\{^{19}F\}$ NMR spectrum of $Bi(C_6F_5)_3(OCOCF_3)_2$ ($CDCl_3$, 21 °C).

Table 4
^{13}C NMR data of selected compounds with a CF_3COO group (δ in ppm; coupling constants in Hz)

Compound	$\delta(CF_3)$	$^1J(^{19}F-^{13}C)$	$\delta(CO_2)$	$^2J(^{19}F-^{13}C)$	Solvent
$(CH_3)_3SiOCOCF_3$	116.0	285	157.8	42	CD_3CN
CF_3COOH	115.9	285	158.4	41	CD_3CN
$Bi(C_6F_5)_3(OCOCF_3)_2$	114.5	283	161.8	36	CD_3CN
$Bi(2,6\text{-}F_2C_6H_3)_3(OCOCF_3)_2$ [a]	114.8	290	160.2	39	$CDCl_3$
$KOCOCF_3$	117.5	293	161.2	37	CD_3CN
$Bi(C_6H_5)_4OCOCF_3$ [b]	116.5	–[c]	159.6	–[c]	$CDCl_3$
$AgOCOCF_3$	118.7	293	161.6	35	CD_3CN

[a] Ref. [20].
[b] Ref. [7].
[c] Couplings are not published in Ref. [7].

CCl_3F (^{19}F) were used as external standards. A negative chemical shift denotes a resonance occurring to low frequency (high field) of the reference substance.

Electron impact (EI) mass spectra were run on a modified Varian MAT CH5 spectrometer. No ion current was observed at a sample temperature below the decomposition points of $Bi(C_6F_5)_3Cl_2$ and $Bi(C_6F_5)_3(OCOCF_3)_2$. Therefore the mass spectrometric data must be regarded as the spectra of the decomposition products. Differential thermal analysis (DTA) measurements were recorded with a Mettler TA1 thermoanalyser.

Fluorine was analysed according to Ref. [28], chlorine according to Ref. [29] and bismuth according to Ref. [30]. The molecular weight was determined using a Knauer vapour pressure osmometer.

All reactions were carried out in a dry nitrogen atmosphere using Schlenk techniques. Solvents were purified by standard methods [31]. $Bi(C_6F_5)_3$ was prepared according to Ref. [24], $Bi(C_6F_5)_3F_2$ according to Ref. [13] and XeF_2 according to Ref. [32]. $AgOCOCF_3$ and $KOCOCF_3$ were prepared by reacting the corresponding carbonates with CF_3COOH and drying the resulting solids in vacuo. $(CH_3)_3SiCl$ (Merck-Schuchardt), $(CH_3)_3SiOSO_2CF_3$ and $(CH_3)_3SiOCOCF_3$ (Aldrich) were used as received and CF_3COOH (Solvay Fluor und Derivate GmbH) after distilling in vacuo.

3.1. General synthetic procedure

The trimethylsilyl derivative (2.4 mmol) was added dropwise to a solution of $Bi(C_6F_5)_3F_2$ (0.75 g, 1.0 mmol) dissolved in 15 ml of carefully dried CH_2Cl_2 at -40 °C (-20 °C). The mixture was stirred for 5 days (2 days) at this temperature and then allowed to warm to room temperature in the case of $Bi(C_6F_5)_3Cl_2$ and $Bi(C_6F_5)_3(OCOCF_3)_2$. The solvent and other volatile compounds were distilled off in vacuo. The new compounds were isolated as yellow solids in nearly quantitative yields and recrystallized from $CHCl_3$.

3.2. General procedure of the thermal decomposition reaction

The tris(pentafluorophenyl)bismuth(V) derivative (1.0 mmol) was heated in an evacuated Schlenk tube above the corresponding decomposition point for 1 h. The tube was cooled to -196 °C; 5 ml of $CDCl_3$ was added and the mixture was allowed to warm to room temperature. The solutions of the decomposition products were studied by ^{19}F NMR spectroscopy. $Bi(C_6F_5)_3$ was identified by adding authentic material to the solution.

3.3. Spectroscopic and analytical data of $Bi(C_6F_5)_3Cl_2$

Elemental analysis (%) for $C_{18}Cl_2F_{15}Bi$ (values in parentheses are calculated): Bi, 23.9 (26.8); F, 34.9 (36.5); Cl, 8.7 (9.1). Molecular weight determination (CH_2Cl_2): 792.3 g mol^{-1} (781.1 g mol^{-1}). ^{19}F NMR ($CDCl_3$, 21 °C) δ: -127.0 (s, $\Delta_{1/2}=41$ Hz, 6F, F2,6); -142.3 (m, $^3J(^{19}F-^{19}F)=20$ Hz, 3F, F4); -154.3 (m, $^3J(^{19}F-^{19}F)=20$ Hz, 6F, F3,5) ppm. $^{13}C\{^{19}F\}$ NMR ($CDCl_3$, 21 °C) δ: 145.6 (s, C3,5); 145.1 (s, C4); 138.2 (s, C2,6); 132.2 (s, $\Delta_{1/2}=37$ Hz, C1) ppm. MS (EI, 17 eV, 100 °C, m/e): 710 (5.1%) $[Bi(C_6F_5)_3]^+$; 543 (20.9%) $[Bi(C_6F_5)_2]^+$; 395 (6.2%) $[Bi(C_6F_5)F]^+$; 376 (100.0%) $[Bi(C_6F_5)]^+$; 368 (5.2%) $[BiC_4F_4Cl]^+$; 295 (6.9%) $[BiC_4F_2]^+$; 280 (5.1%) $[BiCl_2]^+$; 244 (8.4%) $[BiCl]^+$; 209 (40.2%) $[Bi]^+$; 202 (50.8%) $[C_6F_5Cl]^+$; 168 (37.9%) $[C_6F_5H]^+$; 167 (11.8%) $[C_6F_5]^+$; 117 (6.2%) $[C_5F_3]^+$; 99 (7.1%) $[C_5F_2H]^+$. Fragments with intensities of less than 5% are omitted. Decomposition point, 42 °C.

3.4. Spectroscopic and analytical data of $Bi(C_6F_5)_3(OCOCF_3)_2$

Elemental analysis (%) of $C_{22}F_{21}O_4Bi$ (values in parentheses are calculated): Bi, 23.3 (22.3); F, 39.6 (42.6). Molecular weight determination (CH_3CN): 889.0 g mol^{-1} (936.2 g mol^{-1}). ^{19}F NMR ($CDCl_3$, 21 °C) δ: -74.9 (s, 6F, $OCOCF_3$); -125.1 (m, $^3J(^{19}F-^{19}F)=21$ Hz, 6F, F2,6); -140.6 (t, $^3J(^{19}F-^{19}F)=21$ Hz, 3F, F4); -154.3 (m, $^3J(^{19}F-^{19}F)=21$ Hz, 6F, F3,5) ppm. $^{13}C\{^{19}F\}$ NMR ($CDCl_3$, 21 °C) δ: 161.8 (s, CO_2); 146.5 (s, C3,5); 145.5 (s, C4); 138.8 (s, C2,6); 128.8 (s, C1); 114.4 (s, CF_3) ppm. MS (EI, 20 eV, 100 °C, m/e): 823 (35.7%) $[Bi(C_6F_5)_3OCOCF_3]^+$; 543 (19.4%) $[Bi(C_6F_5)_2]^+$; 489 (8.9%) $[Bi(C_6F_5)OCOCF_3]^+$; 395 (22.4%)

$[Bi(C_6F_5)F]^+$; 376 (100.0%) $[Bi(C_6F_5)]^+$; 322 (5.1%) $[BiOCOCF_3]^+$; 280 (5.3%) $[C_6F_5OCOCF_3]^+$; 228 (5.1%) $[BiF]^+$; 209 (35.2%) $[Bi]^+$; 168 (18.4%) $[C_6F_5H]^+$; 155 (5.1%) $[C_5F_5]^+$; 99 (5.9%) $[C_5F_2H]^+$. Fragments with intensities of less than 5% are omitted. DTA measurement: exothermic decomposition at 83 °C.

3.5. Spectroscopic data of $Bi(C_6F_5)_3(OSO_2CF_3)_2$

^{19}F NMR ($CDCl_3$, −20 °C) δ: −77.7 (s, 6F, OSO_2CF_3); −123.9 (m, 6F, F2,6); −135.2 (m, 3F, F4); −152.0 (m, 6F, F3,5) ppm. $^{13}C\{^{19}F\}$ NMR ($CDCl_3$, −20 °C) δ: 146.7 (s, C3,5); 146.1 (s, C4); 139.9 (s, C2,6); 126.5 (s, C1); 118.5 (s, CF_3) ppm. Decomposition point: −15 °C (in dichloromethane solution).

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