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Regioselective Synthesis of Tetraalkynylarenes by Consecutive Dual Sonogashira Coupling Reactions of the Bis(triflate) of 4,5-Diiodobenzene-1,2-diol

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Abstract

The regioselective synthesis of nonsymmetric tetraalkynylarenes has been readily achieved through consecutive sets of Sonogashira cross-coupling reactions of the bis(triflate) derivative of 4,5-diiodobenzene-1,2-diol. The initial coupling reactions proceeded with nearly complete selectivity for the reaction at the Ar–I linkages. Subsequent coupling reactions at the Ar–OTf linkages were efficiently conducted. The tetraalkynylarene products are of interest as components of organic molecular materials.

Keywords: Cross-coupling, Regioselectivity, Palladium, Alkynes

Introduction

The palladium-catalyzed cross-coupling reactions of aryl halides or triflates with terminal alkynes is a popular method for the synthesis of arylalkynes.¹ One class of reaction products, tetraalkynylarenes, is of particular interest in the field of organic materials.² Tetraalkynylbenzenes containing four identical alkyne side-chains can be prepared from nonselective coupling reactions of tetrahaloarenes.³ However, regio-defined synthesis of unsymmetrical tetraalkynylarenes also requires control of the sequence of cross-coupling steps. A reported solution to this challenge exploits the greater reactivity of aryl iodides relative to aryl bromides.^{1,2} However, in some cases, the sequential coupling reactions of iodo/bromoarenes have resulted in low yields of tetraalkynylarenes, presumably reflecting the lower reactivity and cross-coupling efficiency of aryl bromides and/or the corresponding requirement for more forcing reaction conditions.⁴ Indeed, in the course of investigations requiring the preparation of unsymmetrical tetraalkynes, we found that attempted two-fold Sonogashira cross-coupling of model 1,2-dibromoarenes failed to give bis(alkyne) products. As a result, we became interested in the possibility of performing consecutive dual Sonogashira coupling reactions with an arene bearing both *ortho*-diiodo and *ortho*-bis(triflate) units.

The higher reactivity of aryl iodides relative to aryl triflates as substrates for C–C cross-coupling reactions is well established,⁵ and we were encouraged by a few reports describing the successful use of arene-1,2-diyl bis(triflates) as substrates for Sonogashira coupling reactions.⁶ Herein, we report the synthesis of the bis(triflate) ester of 4,5-diiodobenzene-1,2-diol and the application of this previously unreported electrophile in the regiocontrolled synthesis of tetraalkynylarenes (Scheme 1).

Scheme 1. Synthetic approach to tetraalkynylarenes.

Results and Discussion

Diiodo bis(triflate) **1** was readily prepared in three steps from 1,2-dimethoxybenzene as shown in Scheme 2. Electrophilic iodination of 1,2-dimethoxybenzene proceeded in 86% yield.⁷ Bis(demethylation), followed by disulfonylation of the resulting catechol proceeded quantitatively to furnish **1**. We used this procedure to prepare up to 20 g of **1**, which is stable in the dark at room temperature for months.

Scheme 2. Synthesis of **1**.

The selectivity of Sonogashira coupling reactions of **1** was investigated by using the benzyl ether of 4-pentynol as a model alkyne (Table 1). [PdCl₂(PPh₃)₂] proved to be an efficient catalyst at a loading of 6 mol-% (Entry 1). Good yields

Table 1. Optimization of the alkyne coupling reaction with **1**.^[a]

Entry	Pd cat. (amount [mol-%])	Alkyne equiv.	Conv. [%]	Yield ^[b] [%]
1	[PdCl ₂ (PPh ₃) ₂] (6)	2.3	>99	84
2	[PdCl ₂ (PPh ₃) ₂] (3)	2.3	68	16
3	[PdCl ₂ (PPh ₃) ₂] (1)	2.3	67	2
4 ^[c]	[PdCl ₂ (PPh ₃) ₂] (3)	2.3	>99	70
5 ^[d]	[PdCl ₂ (PPh ₃) ₂] (3)	2.3	>99	nd
6	[PdCl ₂ (PPh ₃) ₂] (6)	2.1	>95	69
7	[PdCl ₂ (PPh ₃) ₂] (6)	2.5	>99	81
8	[Pd(PPh ₃) ₄] (6)	2.3	52	trace
9	[PdCl ₂ (dppf)] (6)	2.3	47	trace

[a] Reagents: **1** (0.4 mmol), CuI/Pd (2:1); nd = not determined.

[b] Isolated yield. [c] Time = 10 h. [d] T = 60 °C.

of the 1,2-bis(alkynyl) product could be obtained at lower catalyst loadings but at the cost of extended reaction times (Entry 4). Complete conversion of the starting material at lower catalyst loadings could be achieved by performing the reaction at higher temperatures (Entry 5), but these conditions resulted in the formation of copious amounts of by-products that were difficult to separate from the desired diyne. Alternative sources of Pd^{II} or Pd⁰ provided lower conversions and only trace amounts of the product (Entries 8 and 9). A 2:1 ratio of Cu^I/Pd^{II} proved optimal for the transformation. Complete selectivity for the coupling of the aryl iodides was found when 2.3 equiv. of alkyne were used; no appreciable amounts of products resulting from coupling to the triflates were detected. However, performing the reaction in the presence of a large excess (5 equiv.) of alkyne did lead to the formation of significant amounts of inseparable byproducts, which appeared to include the tri- and tetraalkynylbenzene (not shown).

The selectivity of the reaction is significant. One might envisage that the introduction of an alkyne in the first Sonagashira coupling might direct the second coupling away from the *ortho* iodide and towards one of the electron-deficient, but sterically unencumbered triflate units; electron-poor aryl halides are typically favored as electrophiles in alkyne cross-coupling reactions.⁸ However, the selective Sonogashira coupling of aryl iodides in the presence of aryl triflates has previously been demonstrated.⁵ Interestingly, the reaction of **1** with only 1.1 equiv. of (4-methoxyphenyl) ethyne under typical reaction conditions generated the diyne and monoynone in a 1.3:1 ratio in a combined yield of 81% (not shown). The modest preference for dialkynylation could reflect a directing effect of the *ortho*-alkyne⁹ or, alternatively, a more facile insertion into the hindered aryl iodide due to the diffusion-controlled proximity of the Pd catalyst to the electrophile.¹⁰

With the optimized reaction conditions in hand, we investigated the scope of the selective cross-coupling reaction of **1** with a variety of alkynes (Table 2). Although most reactions were complete in 3 h, some alkynes required

Table 2. Substrate scope for the selective coupling of **1**.^[a]

Entry	R	t [h]	Product, yield ^[b] [%]
1	(CH ₂) ₃ OBn	3	2a , 84
2	(CH ₂) ₃ OBz	5	2b , 71
3	(CH ₂) ₃ CN	3	2c , 78
4	(CH ₂) ₄ OTBS	3	2d , 94
5	(CH ₂) ₄ Cl	3	2e , 76
6	TIPS	3	2f , 46
7	4-MeOC ₆ H ₄	3	2g , 78
8	2-Py	3	2h , 69
9	2-Py	3	2h , 84 ^[c]
10	(CH ₂) ₂ OTHP	3	2i , 85
11	C(CH ₃) ₂ OH	3	2j , 72
12	CH(OEt) ₂	3	2k , 69
13	CH ₂ OBz	3	2l , 46
14	CH ₂ OBn	5	2m , 43

[a] Reagents: **1** (0.4 mmol), THF (1 mL). [b] Isolated yield.

[c] On a 3.2 mmol scale.

longer times for the complete consumption of the starting material (Entries 2 and 14). The reactions proceeded smoothly with alkynes functionalized with esters (Entry 2), nitriles (Entry 3), silyl ethers (Entry 4), alkyl chlorides (Entry 5), THP ethers (Entry 10), and an acetal (Entry 12). Aromatic alkynes also proved to be excellent coupling partners (Entries 7–9). Coupling reactions with nonpolar alkynes (e.g., heptyne) appeared to proceed to completion (TLC); however, because of the difficulty in completely separating the products from recovered alkyne and nonpolar by-products by silica chromatography, these examples are not reported. Cross-coupling with a tertiary propargyl alcohol proceeded in good yield (Entry 11), whereas reactions with unhindered propargyl ethers (Entries 13 and 14) provided lower yields of the bis(alkynes). Given the frequent application of propargyl alcohols and ethers as nucleophiles in Sonagashira coupling reactions,¹ we are uncertain as to why their use has limitations in this setting.

We next investigated the conditions for displacing both triflates in a second set of Sonogashira coupling reactions (Table 3). By using conditions related to those reported by Powell and Rychnovsky,^{6a} diyne **2a** was coupled with the TBS ether of 5-hexynol to yield tetrayne **3a** in 72% yield (Entry 1). Further modification of the reported conditions did not lead to any notable improvement (data not shown). The scope of the second set of coupling reactions was then investigated (Entries 2–9).

Finally, we compared our new approach directly against an existing methodology for the synthesis of **3i**, a target previously prepared in four steps and 11% yield from 1,2-dibromo-4,5-diiodobenzene (Scheme 3).^{2a} By using our method, **3i** could be prepared from diiododifluoride **1** in two steps and 61% yield (or five steps and 53% overall yield from 1,2-dimethoxybenzene).

Table 3. Synthesis of tetraalkynylarenes by dual coupling of dialkynylarenediyl bis(triflates).^[a]

Entry	R	R'	Product, yield ^[b] [%]
1	(CH ₂) ₃ OBn	(CH ₂) ₄ OTBS	3a , 72
2	(CH ₂) ₃ OBn	(CH ₂) ₃ OBz	3b , 64
3	(CH ₂) ₃ OBn	TMS	3c , 64
4	(CH ₂) ₃ OBn	Ph	3d , 83
5	(CH ₂) ₃ OBn	4-MeOC ₆ H ₄	3e , 79
6	(CH ₂) ₃ OBn	4-FC ₆ H ₄	3f , 74
7 ^[c]	(CH ₂) ₄ OTBS	(CH ₂) ₃ OBz	3g , 68
8	4-MeOC ₆ H ₄	TMS	3h , 57
9	2-Py	4-(Bu ₂ N)C ₆ H ₄	3i , 73

[a] Reagents and conditions: on a 0.25 mmol scale, DMF/TEA (5:1) (1.5 mL), sealed vial. [b] Isolated yield. [c] KI (3 equiv.) used instead of TBAI.

Scheme 3. Comparison of the protocol developed here with an existing methodology.

Conclusions

The selective Sonogashira cross-coupling reaction of 1,2-diiodobenzene-4,5-diyl bis(triflate) proceeds with almost complete selectivity displacing both iodine atoms to furnish a 4,5-bis(alkynyl)benzene-1,2-diyl bis(triflate), which can be subjected to a second set of alkyne coupling reactions to efficiently give nonsymmetric 1,2,4,5-tetraalkynylarenes. Structurally related 1,2-diyl bis(triflates) have been shown to undergo dual Suzuki coupling reactions,^{6b} which suggests our results could potentially be extended to other transition-metal-catalyzed cross-coupling reactions.

Experimental Section

General: All the reactions were carried out in flame-dried glassware under dry nitrogen with magnetic stirring. Solvents were used as purchased with the exception of THF and CH₂Cl₂, which were distilled from Na/Ph₂CO and CaH₂, respectively. TLC was performed on 0.25 mm hard-layer silica G plates; developed plates were visualized with a UV lamp and/or by staining: vanillin (general stain, after charring), 1% aq. KMnO₄ (for

unsaturated compounds), I₂, or phosphomolybdic acid (general stain, after charring). NMR spectra were recorded in CDCl₃ (by using residual CHCl₃: δ = 7.286 ppm) at 300/400/500/600 MHz (¹H) or 75/100/125/150 MHz (¹³C), as indicated. ¹H NMR chemical shifts are reported as δ in ppm as follows: chemical shift [multiplicity, coupling constant(s) in Hz, integration]. ¹³C NMR chemical shifts are reported as δ in ppm. IR spectra were recorded as neat ATR films with selected absorbances reported in wavenumbers (cm⁻¹).

1,2-Diiodo-4,5-dimethoxybenzene: This compound was prepared according to the procedure of Lacour et al.⁷ H₂IO₆ (0.41 equiv., 25.6 mmol, 5.84 g) and MeOH (36 mL) were added to a flame-dried 100 mL round-bottomed flask equipped with a short air condenser. The mixture was stirred at room temp., and then I₂ (0.8 equiv., 50.2 mmol, 12.76 g) was added. The reaction mixture was stirred vigorously for 10 min, after which 1,2-dimethoxybenzene (1 equiv., 63 mmol, 8.7 g, 8.0 mL) was added in one portion through a syringe. The reaction mixture was heated at 70 °C in an oil bath for 5 h, which resulted in the formation of a white solid that made stirring difficult; however, the reaction proceeded even without efficient stirring. The hot solution was poured into dilute aqueous Na₂S₂O₅ (ca. 100 mL), and the mixture was cooled to room temp. The solid collected by filtration through a glass frit was washed quickly with two 30 mL portions of cold MeOH and dried in vacuo to afford 1,2-diiodo-4,5-dimethoxybenzene (21.07 g, 54 mmol, 86%) as a white solid that was deemed pure by NMR spectroscopy and used without further purification. *R*_f = 0.49 (20% ethyl acetate/hexane). M.p. 134.5–136.0 °C (ref.⁷ 134 °C). ¹H NMR (600 MHz): δ = 7.25 (s, 2 H), 3.85 (s, 6 H) ppm. ¹³C NMR (150 MHz): δ = 149.6, 121.7, 96.1, 56.2 ppm.

1,2-Dihydroxy-4,5-diiodobenzene: A flame-dried 250 mL round-bottomed flask was charged with 1,2-diiodo-4,5-dimethoxybenzene (1 equiv., 10 mmol, 3.90 g) and then evacuated/back-filled with nitrogen (3×) before addition of CH₂Cl₂ (70 mL). The solution was cooled to 0 °C, and BBr₃ (2.5 equiv., 25 mmol, 25 mL of a 1.0 M solution in CH₂Cl₂) was added through a syringe pump over 20 min. The reaction mixture was stirred at 0 °C for 4 h and then quenched with H₂O (50 mL). The separated aqueous layer was extracted with Et₂O (2×75 mL). The combined organic layers were dried with MgSO₄, filtered through a pad of silica, and concentrated in vacuo to afford 1,2-dihydroxy-4,5-diiodobenzene (3.61 g, 9.99 mmol, quant.) as an off-white solid that was deemed pure by NMR spectroscopy and used without further purification. *R*_f = 0.50 (50% ethyl acetate/hexane). M.p. 116.0–116.5 °C. ¹H NMR (400 MHz, [D₆]acetone): δ = 8.48 (br. s, 2 H), 7.38 (s, 2 H) ppm. ¹³C NMR (150 MHz, [D₆]acetone): δ = 146.5, 125.6, 93.7 ppm.

4,5-Diiodo-1,2-phenylene Bis(trifluoromethanesulfonate) (1): 1,2-Dihydroxy-4,5-diiodobenzene (1 equiv., 7.85 mmol, 2.84 g), CH₂Cl₂ (55 mL), and pyridine (5 equiv., 39 mmol, 3.10 g, 3.16 mL) were added to a flame-dried 100 mL round-bottomed flask. The solution was cooled to 0 °C, and Tf₂O (2.2 equiv., 17.3 mmol, 4.88 g, 2.91 mL) was added dropwise through a syringe over 10 min. The reaction mixture was stirred for 6 h, while warming to ambient temperature, then cooled to 0 °C, and quenched with H₂O (30 mL). The separated aqueous layer was extracted with CH₂Cl₂ (2×30 mL). The combined organic layers were dried with MgSO₄ and filtered through a tall pad of silica. The pad was washed carefully with CH₂Cl₂ to avoid the elution of impurities, and the filtrate was concentrated in vacuo to afford **1** (4.90 g, 7.82 mmol, quant.) as an off-white solid that was deemed pure by NMR spectroscopy and used without further purification. (Note: For reactions in which small amounts of impurities were observed after filtration, the product could be obtained in

pure form by column chromatography utilizing 10% ethyl acetate/hexane as the mobile phase.) $R_f = 0.60$ (10% ethyl acetate/hexane). M.p. 46.5–47.7 °C. ^1H NMR (400 MHz): $\delta = 7.91$ (s, 2 H) ppm. ^{13}C NMR (100 MHz): $\delta = 139.6, 133.4, 118.5$ (q, $J_{\text{CF}} = 321.0$ Hz), 108.0 ppm. FTIR: $\bar{\nu} = 1429, 1335, 1215, 1125, 1105, 868, 788, 745, 689\text{ cm}^{-1}$. HRMS (ESI): calcd. for $\text{C}_8\text{H}_2\text{F}_6\text{I}_2\text{NaO}_6\text{S}_2$ [$\text{M} + \text{Na}$] $^+$ 648.7184; found 648.7164.

General Procedure A. Sonogashira Coupling of 1: A flame-dried 8 mL vial fitted with a screw-top septum cap was charged with $[\text{Pd}(\text{PPh}_3)_2\text{Cl}_2]$ (0.06 equiv., 0.024 mmol, 16.8 mg), CuI (0.12 equiv., 0.048 mmol, 9.2 mg), and **1** (1 equiv., 0.40 mmol, 250 mg) and then evacuated/back-filled with nitrogen (3 $\times$). A solution of the alkyne (2.3 equiv., 0.92 mmol) in THF (1 mL) was then added, followed by Et_3N (3 equiv., 1.2 mmol, 0.17 mL). The mixture was stirred at room temp. for the required time (3–5 h) until the reaction was complete (TLC) and then filtered through a plug of silica, which was washed with Et_2O . The combined eluents were concentrated in vacuo and purified by flash chromatography (ethyl acetate/hexane) to afford the diyne.

4,5-Bis(5-benzyloxy-pent-1-ynyl)-1,2-phenylene Bis(trifluoromethanesulfonate) (2a): Yield: 241 mg, 0.336 mmol, 84%. Mobile phase: step gradient hexane to 10% ethyl acetate/hexane. $R_f = 0.27$ (10% ethyl acetate/hexane). ^1H NMR (600 MHz): $\delta = 7.42$ (s, 2 H), 7.27–7.39 (10 H), 4.56 (s, 4 H), 3.65 (t, $J = 6.0$ Hz, 4 H), 2.62 (t, $J = 7.1$ Hz, 4 H), 1.92–1.98 (m, 4 H) ppm. ^{13}C NMR (150 MHz): $\delta = 138.8, 138.3, 128.4, 128.2, 127.59, 127.56, 126.3, 118.5$ (q, $J_{\text{CF}} = 321.1$ Hz), 98.1, 77.4, 73.0, 68.5, 28.6, 16.5 ppm. FTIR: $\bar{\nu} = 2859, 2230, 1489, 1433, 1210, 1178, 1135, 1080, 732\text{ cm}^{-1}$. HRMS (ESI): calcd. for $\text{C}_{32}\text{H}_{28}\text{F}_6\text{O}_8\text{S}_2\text{Na}$ [$\text{M} + \text{Na}$] $^+$ 741.1027; found 741.1039.

5,5'-[4,5-Bis(trifluoromethylsulfonyloxy)-1,2-phenylene]dipent-4-ynyl Dibenzoate (2b): Yield: 211 mg, 0.284 mmol, 71%. Mobile phase: step gradient hexane to 12% ethyl acetate/hexane. $R_f = 0.20$ (10% ethyl acetate/hexane). ^1H NMR (400 MHz): $\delta = 8.01$ –8.11 (4 H), 7.53–7.60 (2 H), 7.39–7.48 (6 H), 4.51 (t, $J = 6.3$ Hz, 4 H), 2.71 (t, $J = 7.0$ Hz, 4 H), 2.12 (quint, $J = 6.5$ Hz, 4 H) ppm. ^{13}C NMR (150 MHz): $\delta = 166.5, 138.9, 133.0, 130.1, 129.6, 128.4, 128.0, 126.5, 118.5$ (q, $J_{\text{CF}} = 321.4$ Hz), 97.2, 77.8, 63.5, 27.6, 16.7 ppm. FTIR: $\bar{\nu} = 2210, 1716, 1489, 1433, 1271, 1246, 1209, 1177, 1134, 1085, 1070, 1027, 863, 808, 708\text{ cm}^{-1}$. HRMS (ESI): calcd. for $\text{C}_{32}\text{H}_{24}\text{F}_6\text{O}_{10}\text{S}_2\text{Na}$ [$\text{M} + \text{Na}$] $^+$ 769.0613; found 769.0609.

4,5-Bis(5-cyanopent-1-ynyl)-1,2-phenylene Bis(trifluoromethanesulfonate) (2c): Yield: 173 mg, 0.312 mmol, 78%. M.p. 56.8–58.0 °C. Mobile phase: step gradient 25–35% ethyl acetate/hexane. $R_f = 0.35$ (40% ethyl acetate/hexane). ^1H NMR (600 MHz): $\delta = 7.49$ (s, 2 H), 2.73 (t, $J = 6.7$ Hz, 4 H), 2.62 (t, $J = 6.7$ Hz, 4 H), 2.03 (quint, $J = 6.7$ Hz, 4 H) ppm. ^{13}C NMR (150 MHz): $\delta = 139.2, 127.5, 126.7, 121.7, 118.5$ (q, $J_{\text{CF}} = 321.0$ Hz), 95.4, 78.7, 24.2, 18.6, 16.2 ppm. FTIR: $\bar{\nu} = 2231, 1490, 1431, 1401, 1246, 1223, 1212, 1131, 1084, 907, 858, 799, 749\text{ cm}^{-1}$. HRMS (ESI): calcd. for $\text{C}_{20}\text{H}_{14}\text{F}_6\text{N}_2\text{O}_6\text{S}_2\text{Na}$ [$\text{M} + \text{Na}$] $^+$ 579.0095; found 579.0092.

4,5-Bis[6-(tert-butylidimethylsilyloxy)hex-1-ynyl]-1,2-phenylene Bis(trifluoromethanesulfonate) (2d): Yield: 298 mg, 0.376 mmol, 94%. Mobile phase: step gradient hexane to 2% ethyl acetate/hexane. $R_f = 0.27$ (2.5% ethyl acetate/hexane). ^1H NMR (400 MHz): $\delta = 7.44$ (s, 2 H), 3.66–3.72 (4 H), 2.49–2.61 (4 H), 1.66–1.77 (8 H), 0.92 (s, 18 H), 0.08 (s, 12 H) ppm. ^{13}C NMR (150 MHz): $\delta = 138.8, 128.3, 126.3, 118.5$ (q, $J_{\text{CF}} = 320.9$ Hz), 98.6, 77.3, 62.5, 31.9, 25.9, 24.9, 19.4, 18.3, –5.3 ppm. FTIR: $\bar{\nu} = 2945, 2930, 2858, 2230, 1489, 1436, 11247, 1211, 1179, 1137, 1087, 834, 807, 774\text{ cm}^{-1}$. HRMS (ESI): calcd. for $\text{C}_{32}\text{H}_{48}\text{F}_6\text{O}_8\text{S}_2\text{Na}$ [$\text{M} + \text{Na}$] $^+$ 817.2131; found 817.2120.

4,5-Bis(6-chlorohex-1-ynyl)-1,2-phenylene Bis(trifluoromethanesulfonate) (2e): Yield: 185 mg, 0.304 mmol, 76%. Mobile phase: 4% ethyl acetate/hexane, $R_f = 0.27$ (5% ethyl acetate/hexane). ^1H NMR (600 MHz): $\delta = 7.46$ (s, 2 H), 3.63 (t, $J = 6.6$ Hz, 4 H), 2.57 (t, $J = 7.0$ Hz, 4 H), 1.97–2.04 (4 H), 1.80–1.86 (4 H) ppm. ^{13}C NMR (150 MHz): $\delta = 138.9, 128.0, 126.5, 118.5$ (q, $J_{\text{CF}} = 320.7$ Hz), 97.7, 77.7, 44.4, 31.6, 25.6, 19.0 ppm. FTIR: $\bar{\nu} = 2946, 2230, 1489, 1432, 1302, 1246, 1209, 1179, 1085, 862, 802, 597\text{ cm}^{-1}$. HRMS (ESI): calcd. for $\text{C}_{20}\text{H}_{18}\text{F}_6\text{O}_8\text{S}_2\text{Cl}_2\text{Na}$ [$\text{M} + \text{Na}$] $^+$ 624.9724; found 624.9716.

4,5-Bis[2-(triisopropylsilyl)ethynyl]-1,2-phenylene Bis(trifluoromethanesulfonate) (2f): Yield: 136 mg, 0.184 mmol, 46%. M.p. 42.3–43.5 °C. Mobile phase: 100% hexane. $R_f = 0.43$ (100% hexane). ^1H NMR (600 MHz): $\delta = 7.52$ (s, 2 H), 1.14–1.21 (42 H) ppm. ^{13}C NMR (150 MHz): $\delta = 139.3, 128.0, 127.3, 118.5$ (q, $J_{\text{CF}} = 320.9$ Hz), 102.1, 100.7, 18.7, 11.2 ppm. FTIR: $\bar{\nu} = 2944, 2867, 2363, 1485, 1436, 1385, 1211, 1167, 1135, 881, 834, 574\text{ cm}^{-1}$. HRMS (EI): calcd. for $\text{C}_{30}\text{H}_{44}\text{F}_6\text{O}_6\text{S}_2\text{Si}_2\text{Na}$ [M] $^+$ 734.2022; found 734.2020.

4,5-Bis[2-(4-methoxyphenyl)ethynyl]-1,2-phenylene Bis(trifluoromethanesulfonate) (2g): Yield: 197 mg, 0.312 mmol, 78%. M.p. 113.4–114.3 °C. Mobile phase: step gradient hexane to 7% ethyl acetate/hexane. $R_f = 0.30$ (10% ethyl acetate/hexane). ^1H NMR (400 MHz): $\delta = 7.59$ (s, 2 H), 7.51–7.57 (4 H), 6.90–6.97 (4 H), 3.88 (s, 6 H) ppm. ^{13}C NMR (150 MHz): $\delta = 160.6, 139.0, 133.5, 127.8, 125.9, 118.5$ (q, $J_{\text{CF}} = 321.0$ Hz), 114.3, 114.0, 97.7, 84.6, 55.4 ppm. FTIR: $\bar{\nu} = 2205, 1605, 1513, 1485, 1435, 1414, 1290, 1248, 1213, 1130, 1027, 896, 828\text{ cm}^{-1}$. HRMS (ESI): calcd. for $\text{C}_{26}\text{H}_{16}\text{F}_6\text{O}_8\text{S}_2\text{Na}$ [$\text{M} + \text{Na}$] $^+$ 657.0088; found 657.0083.

4,5-Bis[2-(pyridin-2-yl)ethynyl]-1,2-phenylene Bis(trifluoromethanesulfonate) (2h): Yield: 158 mg, 0.276 mmol, 69%. M.p. 119.1–120.2 °C. Mobile phase: 40% ethyl acetate/hexane. $R_f = 0.30$ (40% ethyl acetate/hexane). ^1H NMR (400 MHz): $\delta = 8.65$ –8.77 (2 H), 7.67–7.83 (6 H), 7.31–7.40 (m, 2 H) ppm. ^{13}C NMR (150 MHz): $\delta = 150.4, 142.2, 140.0, 136.3, 128.1, 127.3, 126.8, 123.8, 118.5$ (q, $J_{\text{CF}} = 320.8$ Hz), 96.4, 84.2 ppm. FTIR: $\bar{\nu} = 1581, 1561, 1493, 1429, 1208, 1131, 1054, 1040, 904, 885, 833, 782, 732\text{ cm}^{-1}$. HRMS (ESI): calcd. for $\text{C}_{22}\text{H}_{10}\text{F}_6\text{N}_2\text{O}_6\text{S}_2\text{Na}$ [$\text{M} + \text{Na}$] $^+$ 598.9782; found 598.9783.

4,5-Bis[4-(tetrahydro-2H-pyran-2-yloxy)but-1-ynyl]-1,2-phenylene Bis(trifluoromethanesulfonate) (2i): Yield: 230 mg, 0.340 mmol, 85%. Mobile phase: step gradient 10–20% ethyl acetate/hexane. $R_f = 0.22$ (15% ethyl acetate/hexane). ^1H NMR (600 MHz): $\delta = 7.45$ (s, 2 H), 4.69–4.73 (2 H), 3.88–3.98 (4 H), 3.69 (dt, $J = 9.7, 7.0$ Hz, 2 H), 3.52–3.58 (2 H), 2.81 (t, $J = 7.0$ Hz, 4 H), 1.81–1.91 (2 H), 1.71–1.79 (2 H), 1.51–1.69 (8 H) ppm. ^{13}C NMR (150 MHz): $\delta = 138.9, 128.0, 126.4, 118.5$ (q, $J_{\text{CF}} = 320.4$ Hz), 98.9, 95.6, 77.8, 65.3, 62.2, 30.5, 25.4, 21.2, 19.4 ppm. FTIR: $\bar{\nu} = 2944, 2873, 2234, 1489, 1434, 1247, 1210, 1180, 1133, 1082, 976, 892, 859, 804, 733\text{ cm}^{-1}$. HRMS (ESI): calcd. for $\text{C}_{22}\text{H}_{28}\text{F}_6\text{O}_{10}\text{S}_2\text{Na}$ [$\text{M} + \text{Na}$] $^+$ 701.0926; found 701.0925.

4,5-Bis(3-hydroxy-3-methylbut-1-ynyl)-1,2-phenylene Bis(trifluoromethanesulfonate) (2j): Yield: 155 mg, 0.288 mmol, 72%. Mobile phase: step gradient 30–40% ethyl acetate/hexane. $R_f = 0.22$ (30% ethyl acetate/hexane). ^1H NMR (600 MHz): $\delta = 7.48$ (s, 2 H), 3.65 (br. s, 2 H), 1.66 (s, 12 H) ppm. ^{13}C NMR (150 MHz): $\delta = 139.3, 127.5, 125.8, 118.5$ (q, $J_{\text{CF}} = 321.0$ Hz), 102.2, 78.2, 65.6, 31.1 ppm. FTIR: $\bar{\nu} = 3371, 2985, 1486, 1431, 1210, 1134, 1062, 952, 866, 826, 799, 734\text{ cm}^{-1}$. HRMS (ESI): calcd. for $\text{C}_{18}\text{H}_{16}\text{F}_6\text{O}_8\text{S}_2\text{Na}$ [$\text{M} + \text{Na}$] $^+$ 561.0088; found 561.0094.

4,5-Bis(3,3-diethoxyprop-1-ynyl)-1,2-phenylene Bis(trifluoromethanesulfonate) (2k): Yield: 172 mg, 0.276 mmol, 69%. Mobile phase: step gradient hexane to 5% ethyl ac-

etate/hexane. $R_f = 0.22$ (5% ethyl acetate/hexane). ^1H NMR (600 MHz): $\delta = 7.58$ (s, 2 H), 5.52 (s, 2 H), 3.79–3.9 (4 H), 3.64–3.74 (4 H), 1.29 (t, $J = 7.2$ Hz, 12 H) ppm. ^{13}C NMR (150 MHz): $\delta = 139.8, 127.0, 126.7, 118.5$ (q, $J_{\text{CF}} = 320.9$ Hz), 92.51, 91.5, 80.3, 61.3, 15.1 ppm. FTIR: $\nu = 3675, 2979, 2901, 1490, 1435, 1248, 1211, 1178, 1135, 1076, 1050, 868, 814$ cm^{-1} . HRMS (ESI): calcd. for $\text{C}_{22}\text{H}_{24}\text{F}_6\text{O}_{10}\text{S}_2\text{Na}$ $[\text{M} + \text{Na}]^+$ 649.0613; found 649.0610.

3,3'-(4,5-Bis(trifluoromethylsulfonyloxy)-1,2-phenylene]diprop-2-ynyl Dibenzoate (2l): Yield: 127 mg, 0.184 mmol, 46%. M.p. 78.4–79.5 °C. Mobile phase: step gradient hexane to 8% ethylacetate/hexane. $R_f = 0.35$ (10% ethyl acetate/hexane). ^1H NMR (400 MHz): $\delta = 8.04$ –8.17 (4 H), 7.54–7.64 (4 H), 7.42–7.53 (4 H), 5.18 (s, 4 H) ppm. ^{13}C NMR (150 MHz): $\delta = 165.7, 139.8, 133.5, 129.8, 129.3, 128.5, 127.1, 126.8, 118.5$ (q, $J_{\text{CF}} = 320.8$ Hz), 91.6, 81.7, 52.7 ppm. FTIR: $\nu = 1724, 1490, 1434, 1265, 1246, 1210, 1177, 1134, 1089, 1069, 869, 803, 709$ cm^{-1} . HRMS (ESI): calcd. for $\text{C}_{28}\text{H}_{16}\text{F}_6\text{O}_{10}\text{S}_2\text{Na}$ $[\text{M} + \text{Na}]^+$ 712.9987; found 712.9988.

4,5-Bis(3-benzyloxyprop-1-ynyl)-1,2-phenylene Bis(trifluoromethanesulfonate) (2m): Yield: 114 mg, 0.172 mmol, 43%. Mobile phase: step gradient hexane to 10% ethyl acetate/hexane. $R_f = 0.27$ (10% ethyl acetate/hexane). ^1H NMR (600 MHz): $\delta = 7.55$ (s, 2 H), 7.30–7.43 (10 H), 4.67 (s, 4 H), 4.43 (s, 4 H) ppm. ^{13}C NMR (150 MHz): $\delta = 139.6, 137.1, 128.5, 128.11, 128.07, 127.2, 126.8, 118.5$ (q, $J_{\text{CF}} = 320.4$ Hz), 93.5, 82.2, 72.0, 57.6 ppm. FTIR: $\nu = 3676, 2988, 2901, 1489, 1433, 1247, 1209, 1178, 1133, 1072, 864, 807, 737$ cm^{-1} . HRMS (ESI): calcd. for $\text{C}_{28}\text{H}_{20}\text{F}_6\text{O}_8\text{S}_2\text{Na}$ $[\text{M} + \text{Na}]^+$ 685.0402; found 685.0397.

General Procedure B. Sonogashira Coupling of Bis(triflates): A flame-dried 8 mL vial fitted with a septum-containing screw-top cap was charged with $[\text{Pd}(\text{PPh}_3)_2\text{Cl}_2]$ (0.06 equiv., 0.015 mmol, 10.6 mg), CuI (0.3 equiv., 0.075 mmol, 14.3 mg), and Bu_4NI (3 equiv., 0.75 mmol, 277 mg). The vessel was evacuated/back-filled with nitrogen (3 $\times$), after which was added a solution of the bis(triflate) (1 equiv., 0.25 mmol) in DMF/TEA (5:1, 1 mL). The mixture was stirred at room temp. for 5 min, whereupon a solution of alkyne (4 equiv., 1 mmol) in DMF/TEA (5:1, 0.5 mL) was added. The capped vial was placed in an oil bath (70 °C) for 5.5 h and then cooled to room temp. The reaction mixture was filtered through a plug of silica, which was washed with Et_2O . The combined eluents were concentrated in vacuo and purified by flash chromatography.

1,2-Bis(5-benzyloxyprop-1-ynyl)-4,5-bis(6-tert-butyl dimethylsilyloxyhex-1-ynyl)benzene (3a): Yield: 151 mg, 0.18 mmol, 72%. Mobile phase: 6% ethyl acetate/hexane. $R_f = 0.54$ (10% ethyl acetate/hexane). ^1H NMR (600 MHz): $\delta = 7.32$ –7.40 (10 H), 7.27–7.31 (2 H), 4.55 (s, 4 H), 3.69 (t, $J = 5.9$ Hz, 4 H), 3.65 (t, $J = 6.2$ Hz, 4 H), 2.59 (t, $J = 7.0$ Hz, 4 H), 2.51 (t, $J = 6.6$ Hz, 4 H), 1.93 (quint, $J = 6.7$ Hz, 4 H), 1.67–1.78 (8 H), 0.93 (s, 18 H), 0.09 (s, 12 H) ppm. ^{13}C NMR (150 MHz): $\delta = 138.5, 135.2, 128.4, 127.60, 127.56, 125.2, 125.0, 95.4, 94.7, 79.2, 79.1, 73.0, 68.8, 62.7, 32.0, 28.9, 26.0, 25.1, 19.5, 18.4, 16.6, -5.3$ ppm. FTIR: $\nu = 2928, 2855, 2209, 1487, 1253, 1102, 900, 834, 774, 733, 696$ cm^{-1} . HRMS (ESI): calcd. for $\text{C}_{54}\text{H}_{74}\text{O}_4\text{Si}_2\text{Na}$ $[\text{M} + \text{Na}]^+$ 865.5023; found 865.5006.

5,5'-(4,5-Bis(5-benzyloxyprop-1-ynyl)-1,2-phenylene]dipent-4-ynyl Dibenzoate (3b): Yield: 127 mg, 0.16 mmol, 64%. Mobile phase: 15% ethyl acetate/hexane. $R_f = 0.39$ (20% ethyl acetate/hexane). ^1H NMR (600 MHz): $\delta = 8.04$ –8.10 (4 H), 7.54–7.60 (2 H), 7.42–7.47 (4 H), 7.39–7.41 (2 H), 7.33–7.38 (8 H), 7.27–7.31 (2 H), 4.55 (s, 4 H), 4.53 (t, $J = 6.3$ Hz, 4 H), 3.66 (t, $J = 6.2$ Hz, 4 H), 2.70 (t, $J = 7.0$ Hz, 4 H), 2.60 (t, $J = 7.0$ Hz, 4 H), 2.11 (quint, $J = 6.6$ Hz, 4 H), 1.94 (quint, $J = 6.6$ Hz, 4 H) ppm. ^{13}C NMR (150 MHz): $\delta = 166.5, 138.5, 135.3, 133.0, 130.2, 129.6, 128.38, 128.36, 127.60, 127.56, 125.3, 124.9, 95.0, 93.9, 79.6, 79.1, 73.0, 68.7, 63.7, 28.9, 28.0, 16.7, 16.6$ ppm. FTIR: $\nu = 3675, 2988, 2972, 2901, 2229,$

1716, 1451, 1394, 1269, 1107, 1068, 1027, 900 cm^{-1} . HRMS (ESI): calcd. for $\text{C}_{54}\text{H}_{50}\text{O}_6\text{Na}$ $[\text{M} + \text{Na}]^+$ 817.3505; found 817.3503.

1,2-Bis(5-benzyloxyprop-1-ynyl)-4,5-bis(2-trimethylsilylethynyl)benzene (3c): Yield: 98 mg, 0.16 mmol, 64%. Mobile phase: 6% ethyl acetate/hexane. $R_f = 0.41$ (10% ethyl acetate/hexane). ^1H NMR (600 MHz): $\delta = 7.48$ (s, 2 H), 7.28–7.41 (10 H), 4.56 (s, 4 H), 3.66 (t, $J = 6.1$ Hz, 4 H), 2.61 (t, $J = 7.0$ Hz, 4 H), 1.94 (quint, $J = 6.4$ Hz, 4 H), 0.32 (s, 18 H) ppm. ^{13}C NMR (150 MHz): $\delta = 138.5, 135.6, 128.4, 127.62, 127.61, 126.0, 124.5, 102.4, 100.0, 95.6, 79.0, 73.0, 68.7, 28.9, 16.6, 0.01$ ppm. FTIR: $\nu = 2956, 2856, 2226, 2155, 1481, 1454, 1249, 1103, 1077, 1028, 839, 758, 731, 696, 635$ cm^{-1} . HRMS (ESI): calcd. for $\text{C}_{40}\text{H}_{46}\text{O}_2\text{Si}_2\text{Na}$ $[\text{M} + \text{Na}]^+$ 637.2934; found 637.2922.

1,2-Bis(5-benzyloxyprop-1-ynyl)-4,5-bis(2-phenylethynyl)benzene (3d): Yield: 130 mg, 0.208 mmol, 83%. Mobile phase: step gradient 5–10% ethyl acetate/hexane. $R_f = 0.32$ (10% ethyl acetate/hexane). ^1H NMR (600 MHz): $\delta = 7.57$ –7.63 (6 H), 7.34–7.42 (14 H), 7.29–7.34 (2 H), 4.58 (s, 4 H), 3.69 (t, $J = 6.1$ Hz, 4 H), 2.64 (t, $J = 7.0$ Hz, 4 H), 1.97 (quint, $J = 6.6$ Hz, 4 H) ppm. ^{13}C NMR (150 MHz): $\delta = 138.5, 135.1, 131.7, 128.7, 128.5, 128.4, 127.63, 127.61, 125.9, 124.6, 123.1, 95.6, 94.9, 87.6, 79.2, 73.0, 68.7, 28.9, 16.6$ ppm. FTIR: $\nu = 2856, 2224, 1597, 1497, 1364, 1102, 1076, 1027, 899, 754, 734, 688$ cm^{-1} . HRMS (ESI): calcd. for $\text{C}_{46}\text{H}_{38}\text{O}_6\text{Na}$ $[\text{M} + \text{Na}]^+$ 645.2770; found 645.2759.

1,2-Bis(5-benzyloxyprop-1-ynyl)-4,5-bis[2-(4-methoxyphenyl)ethynyl]benzene (3e): Yield: 135 mg, 0.198 mmol, 79%. Mobile phase: 15% ethyl acetate/hexane. $R_f = 0.37$ (20% ethyl acetate/hexane). ^1H NMR (600 MHz): $\delta = 7.49$ –7.56 (6 H), 7.34–7.42 (8 H), 7.28–7.38 (2 H), 6.88–6.93 (4 H), 4.56 (s, 4 H), 3.86 (s, 6 H), 3.68 (t, $J = 6.1$ Hz, 4 H), 2.62 (t, $J = 7.0$ Hz, 4 H), 1.95 (quint, $J = 6.6$ Hz, 4 H) ppm. ^{13}C NMR (150 MHz): $\delta = 159.2, 138.5, 134.8, 133.2, 128.4, 127.62, 127.59, 125.5, 124.7, 115.3, 114.1, 95.2, 94.9, 86.6, 79.3, 73, 68.7, 55.3, 28.9, 16.6$ ppm. FTIR: $\nu = 2932, 2857, 2209, 1604, 1568, 1511, 1289, 1246, 1172, 1141, 1104, 1027, 899, 829, 734, 696$ cm^{-1} . HRMS (ESI): calcd. for $\text{C}_{48}\text{H}_{42}\text{O}_4\text{Na}$ $[\text{M} + \text{Na}]^+$ 705.2981; found 705.2974.

1,2-Bis(5-benzyloxyprop-1-ynyl)-4,5-bis[2-(4-fluorophenyl)ethynyl]benzene (3f): Yield: 122 mg, 0.185 mmol, 74%. Mobile phase: 8% ethyl acetate/hexane. $R_f = 0.36$ (10% ethyl acetate/hexane). ^1H NMR (600 MHz): $\delta = 7.52$ –7.58 (6 H), 7.34–7.42 (8 H), 7.28–7.34 (2 H), 7.05–7.11 (4 H), 4.57 (s, 4 H), 3.68 (t, $J = 6.1$ Hz, 4 H), 2.63 (t, $J = 7.0$ Hz, 4 H), 1.96 (quint, $J = 6.6$ Hz, 4 H) ppm. ^{13}C NMR (150 MHz): $\delta = 162.8$ (d, $J_{\text{CF}} = 250.5$ Hz), 138.5, 135.0, 133.5 (d, $J_{\text{CF}} = 8.6$ Hz), 128.4, 127.61, 127.60, 126.0, 124.3, 119.2 (d, $J_{\text{CF}} = 4.4$ Hz), 115.8 (d, $J_{\text{CE}} = 22.5$ Hz), 95.7, 93.7, 87.3, 79.1, 73.0, 68.7, 28.9, 16.6 ppm. FTIR: $\nu = 2926, 2856, 2225, 1600, 1567, 1507, 1227, 1155, 1102, 899, 833, 792, 734, 696$ cm^{-1} . HRMS (ESI): calcd. for $\text{C}_{46}\text{H}_{36}\text{F}_2\text{O}_2\text{Na}$ $[\text{M} + \text{Na}]^+$ 681.2581; found 681.2567.

5,5'-(4,5-Bis[6-(tert-butyl dimethylsilyloxy)hex-1-ynyl]-1,2-phenylene]dipent-4-ynyl Dibenzoate (3g): Yield: 149 mg, 0.170 mmol, 68%. Mobile phase: 6% ethyl acetate/hexane. $R_f = 0.36$ (10% ethyl acetate/hexane). ^1H NMR (600 MHz): $\delta = 8.03$ –8.08 (4 H), 7.34–7.42 (8 H), 7.52–7.59 (2 H), 7.38–7.47 (6 H), 4.50 (t, $J = 6.3$ Hz, 4 H), 3.68 (t, $J = 6.0$ Hz, 4 H), 2.67 (t, $J = 6.8$ Hz, 4 H), 2.50 (t, $J = 6.5$ Hz, 4 H), 2.09 (quint, $J = 6.5$ Hz, 4 H), 1.65–1.77 (8 H), 0.92 (s, 18 H), 0.07 (s, 12 H) ppm. ^{13}C NMR (150 MHz): $\delta = 166.5, 135.2, 132.9, 130.2, 129.6, 128.3, 125.4, 124.8, 95.6, 93.8, 79.6, 79.0, 63.7, 62.6, 31.9, 28.0, 26.0, 25.1, 19.5, 18.3, 16.6, -5.3$ ppm. FTIR: $\nu = 2951, 2928, 2856, 2231, 1719, 1270, 1107, 1070, 834, 709$ cm^{-1} . HRMS (ESI): calcd. for $\text{C}_{54}\text{H}_{70}\text{O}_6\text{Si}_2\text{Na}$ $[\text{M} + \text{Na}]^+$ 893.4609; found 893.4631.

1,2-Bis[2-(4-methoxyphenyl)ethynyl]-4,5-bis(2-trimethylsilylethynyl)benzene (3h): Yield: 76 mg, 0.143 mmol, 57%. Mo-

bile phase: 4% ethyl acetate/hexane. R_f = 0.38 (10% ethyl acetate/hexane). ^1H NMR (600 MHz): δ = 7.66 (s, 2 H), 7.47–7.55 (4 H), 6.86–6.93 (4 H), 3.85 (s, 6 H), 0.31 (s, 18 H) ppm. ^{13}C NMR (150 MHz): δ = 160.0, 135.2, 133.2, 125.6, 124.8, 115.1, 114.1, 102.4, 100.4, 95.7, 86.4, 55.3, 0.0 ppm. FTIR: ν = 2959, 2213, 2156, 1605, 1513, 1290, 1246, 1167, 1031, 828 cm^{-1} . HRMS (EI): calcd. for $\text{C}_{34}\text{H}_{34}\text{O}_2\text{Si}_2$ [M] $^+$ 530.2097; found 530.2093.

1,2-Bis[2-[4-(dibutylamino)phenyl]ethynyl]-4,5-bis[2-(2-pyridyl)ethynyl]benzene (3i): Yield: 134 mg, 0.183 mmol, 73%. Mobile phase: 35% ethyl acetate/hexane. R_f = 0.35 (40% ethyl acetate/hexane). ^1H NMR (600 MHz): δ = 8.64–8.70 (2 H), 7.78 (s, 2 H), 7.73 (d, J = 7.6 Hz, 2 H), 7.67 (td, J = 7.8, 1.9 Hz, 2 H), 7.45 (d, J = 8.7 Hz, 2 H), 7.23–7.28 (2 H), 6.61 (d, J = 8.7 Hz, 4 H), 3.32 (t, J = 7.9 Hz, 8 H), 1.61 (quint, J = 7.8 Hz, 8 H), 1.39 (sext, J = 7.6 Hz, 8 H), 0.99 (t, J = 7.3 Hz, 12 H) ppm. ^{13}C NMR (150 MHz): δ = 150.1, 148.3, 143.5, 136.1, 134.8, 133.3, 127.8, 126.7, 123.4, 122.9, 111.2, 108.5, 98.0, 93.9, 87.4, 86.0, 50.7, 29.4, 20.3, 14.0 ppm.

Supporting Information

^1H and ^{13}C NMR spectra for all compounds are presented following the References.

Acknowledgments — We are grateful for financial support from the National Science Foundation (NSF) (EPS 10040 94). Preliminary experiments were conducted by Mitch Trauernicht.

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SUPPORTING INFORMATION

DOI: 10.1002/ejoc.201200079

Title: Regioselective Synthesis of Tetraalkynylarenes by Consecutive Dual Sonogashira Coupling Reactions of the Bis(triflate) of 4,5-Diodobenzene-1,2-diol

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¹H and ¹³C Spectra:

1,2-diiodo-4,5-dimethoxybenzene

1,2-dihydroxy-4,5-dimethoxybenzene

4,5-Diiodo-1,2-phenylene bistrifluoromethanesulfonate (**1**)

4,5-Bis(5-benzyloxypent-1-yn-1-yl)-1,2-phenylene bistriflate (**2a**)

4,5-Bis(trifluoromethylsulfonyloxy)-1,2-phenylene-bis(pent-4-yne-1-ol-4-yl, benzoate ester) (**2b**)

4,5-Bis(hexanenitrile-6-yl)-1,2-phenylene bistriflate (**2c**)

4,5-Bis(6-(tert-butyldimethylsilyloxy)hex-1-yn-1-yl)-1,2-phenylene bistriflate (**2d**)
(¹H, ¹³C)

4,5-Bis(6-chlorohex-1-yn-1-yl)-1,2-phenylene bistriflate (**2e**)

4,5-Bis(2-triisopropylsilylethynyl)-1,2-phenylene bistriflate (**2f**)

4,5-Bis(2-(4-methoxyphenyl)ethynyl)-1,2-phenylene bistriflate (**2g**)

4,5-Bis(2-(pyridin-2-yl)ethynyl)-1,2-phenylene bistriflate (**2h**)

4,5-Bis(4-((tetrahydro-2H-pyran-2-yl)oxy)but-1-yn-1-yl)-1,2-phenylene bistriflate (**2i**)

4,5-Bis(2-methyl-3-butyn-1-ol-4-yl)-1,2-phenylene bistriflate (**2j**)

4,5-Bis(3,3-diethoxyprop-1-yn-1-yl)-1,2-phenylene bistriflate (**2k**)

(4,5-Bis(trifluoromethylsulfonyloxy)-1,2-phenylene)bis(prop-2-yne-1-ol-3-yl, benzoate ester) (**2l**)

4,5-Bis(3-benzyloxypent-1-yn-1-yl)-1,2-phenylene bistriflate (**2m**)

4,5-Bis(5-benzyloxypent-1-yn-1-yl)-1,2-phenylene bis
(6-tert-butyldimethylsilyloxyhex-1-yn-1-yl) (**3a**)

4,5-Bis(5-benzyloxypent-1-yn-1-yl)-1,2-phenylene-bis(pent-4-yne-1-ol-5-yl, benzoate ester) (**3b**)

4,5-Bis(5-benzyloxypent-1-yn-1-yl)-1,2-phenylene bis(2-trimethylsilylethyn-1-yl) (**3c**)

4,5-Bis(5-benzyloxypent-1-yn-1-yl)-1,2-phenylene bis(2-phenylethyn-1-yl) (**3d**)

4,5-Bis(5-benzyloxypent-1-yn-1-yl)-1,2-phenylene) bis(2-(4-methoxyphenyl) ethyn-1-yl) (**3e**)

4,4'-((4,5-Bis(5-benzyloxypent-1-yn-1-yl)-1,2-phenylene)bis-2-(4-fluorophenyl)ethyn-1-yl) (**3f**)

(4,5-Bis(6-tert-butyldimethylsilyloxyhex-1-yn-1-yl)-1,2-phenylene) bis(pent-4-yne-1-ol-5-yl, benzoate ester) (**3g**)

4,5-Bis(2-(4-methoxyphenyl)ethyn-1-yl)-1,2-phenylene) bis(2-trimethylsilylethynyl-1-yl) (**3h**)

4,5-Bis(2-(4-pyridyl)ethynyl)-1,2-phenylene)bis-2-(4-(*N,N'*-dibutylanilino)ethyn-1-yl) (**3i**)

NAME TFish0773_product_022411
EXPNO 1
PROCNO 1
Date_ 20110224
Time 14.02
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 12376.237 Hz
FIDRES 0.188846 Hz
AQ 2.6477449 sec
RG 181
DW 40.400 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.50 usec
PL1 0.20 dB
PL1W 35.98249435 W
SFO1 600.1827064 MHz
SI 32768
SF 600.1789953 MHz
WDW EK
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

```

NAME      TFish0773_product_022411
EXPNO     2
PROCNO    1
Date_     20110224
Time      14.06
INSTRUM   spect
PROBHD    5 mm BBO BB-1H
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         129
DS         4
SWH        35971.223 Hz
FIDRES     0.548877 Hz
AQ         0.9110143 sec
RG         9195.2
DW         13.900 usec
DE         6.50 usec
TE         298.1 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

```

```

===== CHANNEL f1 =====
NUC1       13C
P1         9.70 usec
PL1        1.00 dB
PL1W       59.85786819 W
SFO1       150.9302211 MHz

```

```

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2        1H
PCPD2      60.00 usec
PL2         0.20 dB
PL12       12.54 dB
PL13       12.54 dB
PL2W       35.98249435 W
PL12W      2.09938097 W
PL13W      2.09938097 W
SFO2       600.1814007 MHz
SI         32768
SF         150.9151300 MHz
WDW         EM
SSB         0
LB         1.00 Hz
GB          0
PC         1.40

```

```

NAME      TFish0774_product_022411
EXPNO     1
PROCNO    1
Date_     20110224
Time      23.24
INSTRUM   spect
PROBHD    5 mm QNP 1H/13
PULPROG   zg30
TD         65536
SOLVENT   Acetone
NS         16
DS         2
SWH        8278.146 Hz
FIDRES     0.126314 Hz
AQ         3.9584243 sec
RG         574.7
DW         60.400 usec
DE         6.50 usec
TE         298.0 K
D1         1.00000000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1      1H
P1         12.00 usec
PL1        -2.30 dB
SFO1      400.1324710 MHz
SI         65536
SF         400.1300000 MHz
WDW        EM
SSB         0
LB         0.30 Hz
GB          0
PC          1.00
  
```

```

NAME      TFish0774_2_product_030511
EXPNO     2
PROCNO    1
Date_     20110305
Time      20.41
INSTRUM   spect
PROBHD    5 mm BBO BB-1H
PULPROG   zgpg30
TD         65536
SOLVENT   Acetone
NS         152
DS         4
SWH        35971.223 Hz
FIDRES     0.548877 Hz
AQ         0.9110143 sec
RG         7298.2
DW         13.900 usec
DE         6.50 usec
TE         298.2 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

```

```

===== CHANNEL f1 =====
NUC1       13C
P1         9.70 usec
PL1        1.00 dB
PL1W       59.85786819 W
SFO1       150.9302211 MHz

```

```

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2        1H
PCPD2      60.00 usec
PL2         0.20 dB
PL12       12.54 dB
PL13       12.54 dB
PL2W       35.98249435 W
PL12W      2.09938097 W
PL13W      2.09938097 W
SFO2       600.1814007 MHz
SI         32768
SF         150.9151300 MHz
WDW         EM
SSB         0
LB          1.00 Hz
GB          0
PC          1.40

```

NAME TFish0776_crude_022511
EXPNO 1
PROCNO 1
Date_ 20110225
Time 20.48
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 456.1
DW 60.400 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 12.00 usec
PL1 -2.30 dB
SFO1 400.1324710 MHz
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

```

NAME      TFish0776_crude_022511
EXPNO     2
PROCNO    1
Date_     20110225
Time      20.52
INSTRUM   spect
PROBHD    5 mm QNP 1H/13
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         661
DS         4
SWH        23980.814 Hz
FIDRES     0.365918 Hz
AQ         1.3664756 sec
RG         4597.6
DW         20.850 usec
DE         6.50 usec
TE         298.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1       13C
P1         10.00 usec
PL1        0.50 dB
SFO1       100.6228298 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2        1H
PCPD2       70.00 usec
PL2         -3.35 dB
PL12        13.34 dB
PL13        13.34 dB
SFO2        400.1316005 MHz
SI          32768
SF          100.6127690 MHz
WDW         EM
SSB         0
LB          1.00 Hz
GB          0
PC          1.40
  
```

NAME TFish0786_product_030511
 EXPNO 3
 PROCNO 1
 Date_ 20110306
 Time 14.14
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 12376.237 Hz
 FIDRES 0.188846 Hz
 AQ 2.6477449 sec
 RG 71.8
 DW 40.400 usec
 DE 6.50 usec
 TE 298.0 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 14.50 usec
 PL1 0.20 dB
 PL1W 35.98249435 W
 SF01 600.1827064 MHz
 SI 32768
 SF 600.1789953 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

```

NAME      TFish0786_product_030511
EXPNO     2
PROCNO    1
Date_     20110306
Time      14.21
INSTRUM   spect
PROBHD    5 mm BBO BB-1H
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         701
DS         4
SWH        35971.223 Hz
FIDRES     0.548877 Hz
AQ         0.9110143 sec
RG         11585.2
DW         13.900 usec
DE         6.50 usec
TE         298.1 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1       13C
P1         9.70 usec
PL1        1.00 dB
PL1W       59.85786819 W
SFO1       150.9302211 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
PCPD2      60.00 usec
PL2        0.20 dB
PL12       12.54 dB
PL13       12.54 dB
PL2W       35.98249435 W
PL12W      2.09938097 W
PL13W      2.09938097 W
SFO2       600.1814007 MHz
SI         32768
SF         150.9151355 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
  
```

NAME TFish0882_5_product_101211
 EXPNO 5
 PROCNO 1
 Date_ 20111013
 Time 18.29
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 362
 DW 60.400 usec
 DE 6.50 usec
 TE 298.0 K
 D1 1.00000000 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 10.00 usec
 PL1 -4.00 dB
 SFO1 400.1324710 MHz
 SI 65536
 SF 400.1299990 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

```

NAME      IFish0882_5_product_101211
EXPNO     2
PROCNO    1
Date_     20111023
Time      12.40
INSTRUM   spect
PROBHD    5 mm BBO BB-1H
PULPROG   zgpg30
TO        65536
SOLVENT   CDCl3
NS         1024
DS         4
SWH        35971.223 Hz
FIDRES     0.548877 Hz
AQ         0.9110143 sec
RG         5160.6
DW         13.900 usec
DE         6.50 usec
TE         298.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1      13C
P1         10.00 usec
PL1        -4.30 dB
PL1W       202.82490540 W
SFO1       150.9302211 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2       1H
PCPD2      80.00 usec
PL2         0.00 dB
PL12       16.66 dB
PL13       16.66 dB
PL1W       37.67829514 W
PL12W      0.81300133 W
PL13W      0.81300133 W
SFO2       600.1814007 MHz
SI         32768
SF         150.9151300 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
  
```

NAME TFish0881_4_product_101411
 EXPNO 1
 PROCNO 1
 Date_ 20111014
 Time 14.47
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 161.3
 DW 60.400 usec
 DE 6.50 usec
 TE 298.0 K
 D1 1.00000000 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 10.00 usec
 PL1 -4.00 dB
 SFO1 400.1324710 MHz
 SI 65536
 SF 400.1299990 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

```

NAME      TFish0883_4_product_102311
EXPNO     2
PROCNO    1
Date_     20111023
Time      13.46
INSTRUM   spect
PROBHD    5 mm BBO BB-1H
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         1024
DS         4
SWH        35971.223 Hz
FIDRES     0.348877 Hz
AQ         0.9110143 sec
RG         18390.4
DW         13.900 usec
DE         6.50 usec
TE         298.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1       13C
P1         10.00 usec
PL1        -4.30 dB
PL1W       202.82490540 W
SFO1       150.9302211 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2        1H
PCPD2       80.00 usec
PL2         0.00 dB
PL12        16.66 dB
PL13        16.66 dB
PL2W        37.67829514 W
PL12W       0.81300133 W
PL13W       0.81300133 W
SFO2        600.1814007 MHz
SI          32768
SF          150.9151300 MHz
WDW         EM
SSB         0
LB          1.00 Hz
GB          0
PC          1.40
  
```

```

NAME      TFish0880_2_product_100511
EXPNO     2
PROCNO    1
Date_     20111005
Time      13.22
INSTRUM   spect
PROBHD    5 mm BBO BB-1H
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         1024
DS         4
SWH        35971.223 Hz
FIDRES     0.548877 Hz
AQ          0.9110143 sec
RG          11585.2
DW          13.900 usec
DE          6.50 usec
TE          298.0 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0        1

```

```

===== CHANNEL f1 =====
NUC1       13C
P1          10.00 usec
PL1         -4.30 dB
PL1W       202.82490540 W
SF01       150.9302211 MHz

```

```

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2        1H
PCPD2       80.00 usec
PL2         0.00 dB
PL12        16.66 dB
PL13        16.66 dB
PL2W        37.67829514 W
PL12W       0.81300133 W
PL13W       0.81300133 W
SF02       600.1814007 MHz
SI          32768
SF          150.9151300 MHz
WDW         EM
SSB         0
LB          1.00 Hz
GB          0
PC          1.40

```

NAME TFish0892_2_product_110911
 EXPNO 3
 PROCNO 1
 Date_ 20111227
 Time 16.54
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 12376.237 Hz
 FIDRES 0.188846 Hz
 AQ 2.6477449 sec
 RG 114
 DW 40.400 usec
 DE 6.50 usec
 TE 298.0 K
 D1 1.00000000 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 11.25 usec
 PL1 -2.30 dB
 PL1W 63.98692703 W
 SFO1 600.1827064 MHz
 SI 65536
 SF 600.1789965 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

```

NAME      TFish0892_2_product_110911
EXPNO     4
PROCNO    1
Date_     20111227
Time      17.01
INSTRUM   spect
PROBHD    5 mm BBO BB-1H
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         1024
DS         4
SWH        35971.223 Hz
FIDRES     0.548877 Hz
AQ         0.9110143 sec
RG         2580.3
DW         13.900 usec
DE         6.50 usec
TE         298.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1      13C
P1        10.00 usec
PL1       -5.00 dB
PL1W      238.29847717 W
SFO1      150.9302211 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2       1H
PCPD2     80.00 usec
PL2        -2.30 dB
PL12      14.74 dB
PL13      14.75 dB
PL2W      63.98692703 W
PL12W     1.26500225 W
PL13W     1.26209271 W
SFO2      600.1814007 MHz
SI         32768
SF        150.9151300 MHz
WDW        EM
SSB         0
LB          1.00 Hz
GB          0
PC          1.40
  
```

NAME TFish0892_3_product_110911
 EXPNO 1
 PROCNO 1
 Date_ 20111109
 Time 17.17
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zg30
 TD 65536
 SOLVENT CDCl₃
 NS 16
 DS 2
 SWH 12376.237 Hz
 FIDRES 0.188846 Hz
 AQ 2.6477449 sec
 RG 64
 DW 40.400 usec
 DE 6.50 usec
 TE 298.0 K
 D1 1.00000000 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 11.25 usec
 PL1 -2.30 dB
 PL1W 63.98692703 W
 SFO1 600.1827064 MHz
 SI 32768
 SF 600.1789965 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

NAME TFish0892_3_product_110911
EXPNO 2
PROCNO 1
Date_ 20111109
Time 17.20
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 35971.223 Hz
FIDRES 0.548877 Hz
AQ 0.9110143 sec
RG 3649.1
DW 13.900 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 10.00 usec
PL1 -5.00 dB
PL1W 238.29847717 W
SF01 150.9302211 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.30 dB
PL12 14.74 dB
PL13 14.75 dB
PL2W 63.98692703 W
PL12W 1.26500225 W
PL13W 1.26209271 W
SF02 600.1814007 MHz
SI 32768
SF 150.9151300 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

```

NAME      TFish0882_1_product_101111
EXPNO     1
PROCNO    1
Date_     20111011
Time      18.10
INSTRUM   spect
PROBHD    5 mm QNP 1H/13
PULPROG   zg30
TD        65536
SOLVENT   CDCl3
NS         16
DS         2
SWH        8278.146 Hz
FIDRES     0.126314 Hz
AQ         3.9584243 sec
RG         512
DW         60.400 usec
DE         6.50 usec
TE         298.0 K
D1         1.00000000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1      1H
P1        10.00 usec
PL1       -4.00 dB
SFO1      400.1324710 MHz
SI        65536
SF        400.1299990 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
  
```

```

NAME      TFish0882_1_product_101111
EXPNO     2
PROCNO    1
Date_     20111012
Time      18.11
INSTRUM   spect
PROBHD    5 mm BBO BB-1H
PULPROG   zgpg30
TD        65536
SOLVENT   CDCl3
NS         1024
DS         4
SWH        35971.223 Hz
FIDRES     0.548877 Hz
AQ         0.9110143 sec
RG         9195.2
DW         13.900 usec
DE         6.50 usec
TE         298.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1       13C
P1         10.00 usec
PL1        -4.30 dB
PL1W       202.82490540 W
SFO1       150.9302211 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2        1H
PCPD2       80.00 usec
PL2         0.00 dB
PL12        16.66 dB
PL13        16.66 dB
PL2W        37.67829514 W
PL12W       0.81300133 W
PL13W       0.81300133 W
SFO2        600.1814007 MHz
SI          32768
SF          150.9151300 MHz
WDW         EM
SSB         0
LB          1.00 Hz
GB          0
PC          1.40
  
```

```

NAME      TFish0897_2_product_120711
EXPNO     1
PROCNO    1
Date_     20111207
Time      10.20
INSTRUM   spect
PROBHD    5 mm QNP 1H/13
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         16
DS         2
SWH        8278.146 Hz
FIDRES     0.126314 Hz
AQ         3.9584243 sec
RG         362
DW         60.400 usec
DE         6.50 usec
TE         298.0 K
D1         1.00000000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1       1H
P1         10.00 usec
PL1        -4.00 dB
SFO1       400.1324710 MHz
SI         65536
SF         400.1299991 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
  
```

```

NAME      TFish0881_3_product_100611
EXPNO     2
PROCNO    1
Date_     20111006
Time      11.09
INSTRUM   spect
PROBHD    5 mm BBO BB-1H
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         1024
DS         4
SWH        35971.223 Hz
FIDRES     0.548877 Hz
AQ         0.9110143 sec
RG         3251
DW         13.900 usec
DE         6.50 usec
TE         298.1 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1      13C
P1         10.00 usec
PL1        -4.30 dB
PL1W       202.82490540 W
SFO1       150.9302211 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2       1H
PCPD2      80.00 usec
PL2         0.00 dB
PL12       16.66 dB
PL13       16.66 dB
PL2W       37.67829514 W
PL12W      0.81300133 W
PL13W      0.81300133 W
SFO2       600.1814007 MHz
SI         32768
SF         150.9151300 MHz
WDW        EM
SSB         0
LB          1.00 Hz
GB          0
PC          1.40
  
```

NAME TFish0881_3_product_100611
 EXPNO 3
 PROCNO 1
 Date_ 20111227
 Time 17.51
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 12376.237 Hz
 FIDRES 0.188846 Hz
 AQ 2.6477449 sec
 RG 64
 DW 40.400 usec
 DE 6.50 usec
 TE 298.0 K
 D1 1.0000000 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 11.25 usec
 PL1 -2.30 dB
 PL1W 63.98692703 W
 SFO1 600.1827064 MHz
 SI 32768
 SF 600.1789961 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

```

NAME      TFish0881_3_product_100611
EXPNO     2
PROCNO    1
Date_     20111006
Time      11.09
INSTRUM   spect
PROBHD    5 mm BBO BB-1H
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         1024
DS         4
SWH        35971.223 Hz
FIDRES     0.548877 Hz
AQ         0.9110143 sec
RG         3251
DW         13.900 usec
DE         6.50 usec
TE         298.1 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1      13C
P1         10.00 usec
PL1        -4.30 dB
PL1W       202.82490540 W
SFO1       150.9302211 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2       1H
PCPD2      80.00 usec
PL2         0.00 dB
PL12       16.66 dB
PL13       16.66 dB
PL2W       37.67829514 W
PL12W      0.81300133 W
PL13W      0.81300133 W
SFO2       600.1814007 MHz
SI         32768
SF         150.9151300 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
  
```

```

NAME      TFish0879_3_product_093011
EXPNO     1
PROCNO    1
Date_     20110930
Time      16.47
INSTRUM   spect
PROBHD    5 mm BBO BB-1H
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         16
DS         2
SWH        12376.237 Hz
FIDRES     0.188846 Hz
AQ         2.6477449 sec
RG         181
DW         40.400 usec
DE         6.50 usec
TE         298.0 K
D1         1.00000000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1      1H
P1         11.75 usec
PL1        0.00 dB
PL1W       37.67829514 W
SFO1       600.1827064 MHz
SI         32768
SF         600.1789965 MHz
WDW        EM
SSB         0
LB         0.30 Hz
GB         0
PC         1.00
  
```

```

NAME      TFish0879_3_product_093011
EXPNO     2
PROCNO    1
Date_     20110930
Time      16.30
INSTRUM   spect
PROBHD    5 mm BBO BB-1H
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         1024
DS         4
SWH        35971.223 Hz
FIDRES     0.548877 Hz
AQ         0.9110143 sec
RG         2896.3
DW         13.900 usec
DE         6.50 usec
TE         298.1 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

```

```

===== CHANNEL f1 =====
NUC1       13C
P1         10.00 usec
PL1        -4.30 dB
PL1W       202.82490540 W
SF01       150.9302211 MHz

```

```

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2        1H
PCPD2       80.00 usec
PL2         0.00 dB
PL12        16.66 dB
PL13        16.66 dB
PL2W        37.67829514 W
PL12W       0.81300133 W
PL13W       0.81300133 W
SF02       600.1814007 MHz
SI          32768
SF         150.9151300 MHz
WDW         EM
SSB         0
LB          1.00 Hz
GB          0
PC          1.40

```

```

NAME      TFish0881_5_product_100711
EXPNO     1
PROCNO    1
Date_     20111007
Time      10.45
INSTRUM   spect
PROBHD    5 mm BBO BB-1H
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         16
DS         2
SWH        12376.237 Hz
FIDRES     0.188846 Hz
AQ         2.6477449 sec
RG         128
DW         40.400 usec
DE         6.50 usec
TE         298.0 K
D1         1.00000000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1      1H
P1         11.75 usec
PL1        0.00 dB
PL1W       37.67829514 W
SFO1       600.1827064 MHz
SI         65536
SF         600.1789964 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
  
```

```

NAME      TFish0881_5_product_100711
EXPNO     2
PROCNO    1
Date_     20111007
Time      12.07
INSTRUM   spect
PROBHD    5 mm BBO BB-1H
PULPROG   zgpg30
TD        65536
SOLVENT   CDCl3
NS         1024
DS         4
SWH        35971.223 Hz
FIDRES     0.548877 Hz
AQ         0.9110143 sec
RG         4096
DW         13.900 usec
DE         6.50 usec
TE         298.1 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1       13C
P1         10.00 usec
PL1        -4.30 dB
PL1W       202.82490540 W
SFO1       150.9302211 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2        1H
PCPD2       80.00 usec
PL2         0.00 dB
PL12        16.66 dB
PL13        16.66 dB
PL2W        37.67829514 W
PL12W       0.81300133 W
PL13W       0.81300133 W
SFO2        600.1814007 MHz
SI          32768
SF          150.9151300 MHz
WDW         EM
SSB         0
LB          1.00 Hz
GB          0
PC          1.40
  
```

```

NAME      TFish0880_6_product_100711
EXPNO     3
PROCNO    1
Date_     20111227
Time      11.48
INSTRUM   spect
PROBHD    5 mm QNP 1H/13
PULPROG   zg30
TD        65536
SOLVENT   CDCl3
NS         16
DS         2
SWH        8278.146 Hz
FIDRES     0.126314 Hz
AQ         3.9584243 sec
RG         362
DW         60.400 usec
DE         6.50 usec
TE         298.0 K
D1         1.00000000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1      1H
P1        10.00 usec
PL1       -4.00 dB
SFO1      400.1324710 MHz
SI        32768
SF        400.1299991 MHz
WDW       EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
  
```

```

NAME      TFish0880_6_product_100711
EXPNO     2
PROCNO    1
Date_     20111007
Time      11.05
INSTRUM   spect
PROBHD    5 mm BBO BB-1H
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         1024
DS         4
SWH        35971.223 Hz
FIDRES     0.548877 Hz
AQ         0.9110143 sec
RG         16384
DW         13.900 usec
DE         6.50 usec
TE         298.1 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1       13C
P1         10.00 usec
PL1        -4.30 dB
PL1W       202.82490540 W
SF01       150.9302211 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2        1H
PCPD2       80.00 usec
PL2         0.00 dB
PL12        16.66 dB
PL13        16.66 dB
PL2W        37.67829514 W
PL12W       0.81300133 W
PL13W       0.81300133 W
SF02        600.1814007 MHz
SI          32768
SF          150.9151300 MHz
WDW         EM
SSB         0
LB          1.00 Hz
GB          0
PC          1.40
  
```

```

NAME      TFish0881_6_product_100711
EXPNO     1
PROCNO    1
Date_     20111007
Time      10.51
INSTRUM   spect
PROBHD    5 mm BBO BB-1H
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         16
DS         2
SWH        12376.237 Hz
FIDRES     0.188846 Hz
AQ         2.6477449 sec
RG         256
DW         40.400 usec
DE         6.50 usec
TE         298.0 K
D1         1.00000000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1      1H
P1         11.75 usec
PL1        0.00 dB
PL1W       37.67829514 W
SFO1      600.1827064 MHz
SI         32768
SF         600.1789964 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
  
```

```

NAME      TFish0881_6_product_100711
EXPNO     2
PROCNO    1
Date_     20111007
Time      13.31
INSTRUM   spect
PROBHD    5 mm BBO BB-1H
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         1024
DS         4
SWH        35971.223 Hz
FIDRES     0.548877 Hz
AQ         0.9110143 sec
RG         3251
DW         13.900 usec
DE         6.50 usec
TE         298.1 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1      13C
P1         10.00 usec
PL1        -4.30 dB
PL1W       202.82490540 W
SFO1      150.9302211 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2       1H
PCPD2      80.00 usec
PL2         0.00 dB
PL12       16.66 dB
PL13       16.66 dB
PL2W       37.67829514 W
PL12W      0.81300133 W
PL13W      0.81300133 W
SFO2      600.1814007 MHz
SI         32768
SF         150.9151300 MHz
WDW         EM
SSB         0
LB          1.00 Hz
GB          0
PC          1.40
  
```

```

NAME      TFish0909_1_product_122011
EXPNO     1
PROCNO    1
Date_     20111220
Time      10.20
INSTRUM   spect
PROBHD    5 mm BBO BB-1H
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         16
DS         2
SWH        12376.237 Hz
FIDRES     0.188846 Hz
AQ         2.6477449 sec
RG         35.9
DW         40.400 usec
DE         6.50 usec
TE         298.0 K
D1         1.0000000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1      1H
P1         11.25 usec
PL1        -2.30 dB
PL1W       63.98692703 W
SFO1       600.1827064 MHz
SI         32768
SF         600.1790000 MHz
WDW        EM
SSB         0
LB         0.30 Hz
GB         0
PC         1.00
  
```

```

NAME      TFish0909_1_product_122011
EXPNO     2
PROCNO    1
Date_     20111220
Time      11.55
INSTRUM   spect
PROBHD    5 mm BBO BB-1H
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         987
DS         4
SWH        45454.547 Hz
FIDRES     0.693381 Hz
AQ         0.7209570 sec
RG         2298.8
DW         11.000 usec
DE         6.50 usec
TE         298.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1       13C
P1         10.00 usec
PL1        -5.00 dB
PL1W       238.29847717 W
SFO1       150.9302211 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2        1H
PCPD2       80.00 usec
PL2         -2.30 dB
PL12        14.74 dB
PL13        14.75 dB
PL2W        63.98692703 W
PL12W       1.26500225 W
PL13W       1.26209271 W
SFO2        600.1814007 MHz
SI          32768
SF          150.9151300 MHz
WDW         EM
SSB         0
LB          1.00 Hz
GB          0
PC          1.40
  
```

NAME TFish0787_product_030611
 EXPNO 1
 PROCNO 1
 Date_ 20110307
 Time 8.04
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 12376.237 Hz
 FIDRES 0.188846 Hz
 AQ 2.6477449 sec
 RG 71.8
 DW 40.400 usec
 DE 6.50 usec
 TE 298.0 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 14.50 usec
 PL1 0.20 dB
 PL1W 35.98249435 W
 SF01 600.1827064 MHz
 SI 65536
 SF 600.1789954 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

```

NAME      TFish0787_product_030611
EXPNO     2
PROCNO    1
Date_     20110307
Time      8.13
INSTRUM   spect
PROBHD    5 mm BBO BB-1H
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         1024
DS         4
SWH        35971.223 Hz
FIDRES     0.548877 Hz
AQ         0.9110143 sec
RG         14596.5
DW         13.900 usec
DE         6.50 usec
TE         298.2 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1       13C
P1         9.70 usec
PL1        1.00 dB
PL1W       59.85786819 W
SFO1       150.9302211 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2        1H
PCPD2      60.00 usec
PL2         0.20 dB
PL12       12.54 dB
PL13       12.54 dB
PL2W       35.98249435 W
PL12W      2.09938097 W
PL13W      2.09938097 W
SFO2       600.1814007 MHz
SI         32768
SF         150.9151300 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
  
```

NAME TFish0885_product_120111
 EXPNO 1
 PROCNO 1
 Date_ 20111220
 Time 16.37
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 12376.237 Hz
 FIDRES 0.188846 Hz
 AQ 2.6477449 sec
 RG 32
 DW 40.400 usec
 DE 6.50 usec
 TE 298.0 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 11.25 usec
 PL1 -2.30 dB
 PL1W 63.98692703 W
 SF01 600.1827064 MHz
 SI 32768
 SF 600.1790000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

```

NAME      TFish0885_product_120111
EXPNO     2
PROCNO    1
Date_     20111220
Time      16.41
INSTRUM   spect
PROBHD    5 mm BBO BB-1H
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         1024
DS         4
SWH        35971.223 Hz
FIDRES     0.548877 Hz
AQ         0.9110143 sec
RG         32768
DW         13.900 usec
DE         6.50 usec
TE         298.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1       13C
P1         10.00 usec
PL1        -5.00 dB
PL1W       238.29847717 W
SFO1       150.9302211 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2        1H
PCPD2       80.00 usec
PL2         -2.30 dB
PL12        14.74 dB
PL13        14.75 dB
PL2W        63.98692703 W
PL12W       1.26500225 W
PL13W       1.26209271 W
SFO2        600.1814007 MHz
SI          32768
SF          150.9151300 MHz
WDW         EM
SSB         0
LB          1.00 Hz
GB          0
PC          1.40
  
```

NAME TPish0909_2_product_122011
 EXPNO 3
 PROCNO 1
 Date_ 20111220
 Time 12.45
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 12376.237 Hz
 FIDRES 0.188846 Hz
 AQ 2.6477449 sec
 RG 71.8
 DW 40.400 usec
 DE 6.50 usec
 TE 298.0 K
 D1 1.00000000 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 11.25 usec
 PL1 -2.30 dB
 PL1W 63.98692703 W
 SFO1 600.1827064 MHz
 SI 32768
 SF 600.1790000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

NAME TFish0909_2_product_122011
 EXPNO 2
 PROCNO 1
 Date_ 20111220
 Time 12.49
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 987
 DS 4
 SWH 35971.223 Hz
 FIDRES 0.548877 Hz
 AQ 0.9110143 sec
 RG 4096
 DW 13.900 usec
 DE 6.50 usec
 TE 298.0 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 10.00 usec
 PL1 -5.00 dB
 PL1W 238.29847717 W
 SF01 150.9302211 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -2.30 dB
 PL12 14.74 dB
 PL13 14.75 dB
 PL2W 63.98692703 W
 PL12W 1.26500225 W
 PL13W 1.26209271 W
 SF02 600.1814007 MHz
 SI 32768
 SF 150.9151300 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

```

NAME      TFish0909_3_product_122011
EXPNO     2
PROCNO    1
Date_     20111220
Time      13.44
INSTRUM   spect
PROBHD    5 mm BBO BB-1H
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         1024
DS         4
SWH        35971.223 Hz
FIDRES     0.548877 Hz
AQ         0.9110143 sec
RG         8192
DW         13.900 usec
DE         6.50 usec
TE         298.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

```

```

===== CHANNEL f1 =====
NUC1       13C
P1         10.00 usec
PL1        -5.00 dB
PL1W       238.29847717 W
SFO1       150.9302211 MHz

```

```

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2        1H
PCPD2       80.00 usec
PL2         -2.30 dB
PL12        14.74 dB
PL13        14.75 dB
PL2W        63.98692703 W
PL12W       1.26500225 W
PL13W       1.26209271 W
SFO2        600.1814007 MHz
SI          32768
SF          150.9151300 MHz
WDW         EM
SSB         0
LB          1.00 Hz
GB          0
PC          1.40

```

NAME TPish0909_4_product_122011
 EXPNO 3
 PROCNO 1
 Date_ 20111220
 Time 14.48
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 12376.237 Hz
 FIDRES 0.188846 Hz
 AQ 2.6477449 sec
 RG 71.8
 DW 40.400 usec
 DE 6.50 usec
 TE 298.0 K
 D1 1.00000000 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 11.25 usec
 PL1 -2.30 dB
 PL1W 63.98692703 W
 SFO1 600.1827064 MHz
 SI 32768
 SF 600.1790000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

```

NAME      TFish0909_4_product_122011
EXPNO     2
PROCNO    1
Date_     20111220
Time      14.52
INSTRUM   spect
PROBHD    5 mm BBO BB-1H
PULPROG   zgpg30
TD        65536
SOLVENT   CDCl3
NS         976
DS         4
SWH        35971.223 Hz
FIDRES     0.548877 Hz
AQ         0.9110143 sec
RG         5792.6
DW         13.900 usec
DE         6.50 usec
TE         298.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

```

```

===== CHANNEL f1 =====
NUC1      13C
P1        10.00 usec
PL1       -5.00 dB
PL1W      238.29847717 W
SF01      150.9302211 MHz

```

```

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2       1H
PCPD2     80.00 usec
PL2       -2.30 dB
PL12      14.74 dB
PL13      14.75 dB
PL2W      63.98692703 W
PL12W     1.26500225 W
PL13W     1.26209271 W
SF02      600.1814007 MHz
SI        32768
SF        150.9151300 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40

```

NAME TFish0904_4_product_122111
 EXPNO 3
 PROCNO 1
 Date_ 20111221
 Time 14.05
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 12376.237 Hz
 FIDRES 0.188846 Hz
 AQ 2.6477449 sec
 RG 45.3
 DW 40.400 usec
 DE 6.50 usec
 TE 298.0 K
 D1 1.00000000 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 11.25 usec
 PL1 -2.30 dB
 PL1W 63.98692703 W
 SFO1 600.1827064 MHz
 SI 32768
 SF 600.1790000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

NAME TFish0904_4_product_122111
 EXPNO 4
 PROCNO 1
 Date_ 20111221
 Time 14.12
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 226
 DS 4
 SWH 35971.223 Hz
 FIDRES 0.548877 Hz
 AQ 0.9110143 sec
 RG 6502
 DW 13.900 usec
 DE 6.50 usec
 TE 298.0 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 10.00 usec
 PL1 -5.00 dB
 PL1W 238.29847717 W
 SF01 150.9302211 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -2.30 dB
 PL12 14.74 dB
 PL13 14.75 dB
 PL2W 63.98692703 W
 PL12W 1.26500225 W
 PL13W 1.26209271 W
 SF02 600.1814007 MHz
 SI 32768
 SF 150.9151300 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

NAME TFish0898_product_120111
 EXPNO 3
 PROCNO 1
 Date_ 20111220
 Time 15.42
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 12376.237 Hz
 FIDRES 0.188846 Hz
 AQ 2.6477449 sec
 RG 71.8
 DW 40.400 usec
 DE 6.50 usec
 TE 298.0 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 11.25 usec
 PL1 -2.30 dB
 PL1W 63.98692703 W
 SF01 600.1827064 MHz
 SI 32768
 SF 600.1790000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

```

NAME      TFish0898_product_120111
EXPNO     2
PROCNO    1
Date_     20111220
Time      15.46
INSTRUM   spect
PROBHD    5 mm BBO BB-1H
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         1024
DS         4
SWH        35971.223 Hz
FIDRES     0.548877 Hz
AQ         0.9110143 sec
RG         3251
DW         13.900 usec
DE         6.50 usec
TE         298.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1       13C
P1         10.00 usec
PL1        -5.00 dB
PL1W       238.29847717 W
SFO1       150.9302211 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2        1H
PCPD2      80.00 usec
PL2         -2.30 dB
PL12        14.74 dB
PL13        14.75 dB
PL2W       63.98692703 W
PL12W      1.26500225 W
PL13W      1.26209271 W
SFO2       600.1814007 MHz
SI          32768
SF         150.9151300 MHz
WDW         EM
SSB         0
LB          1.00 Hz
GB          0
PC          1.40
  
```

NAME TFish0916_product_011012
 EXPNO 1
 PROCNO 1
 Date_ 20120110
 Time 13.34
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 12376.237 Hz
 FIDRES 0.188846 Hz
 AQ 2.6477449 sec
 RG 57
 DW 40.400 usec
 DE 6.50 usec
 TE 298.0 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 11.25 usec
 PL1 -2.30 dB
 PL1W 63.98692703 W
 SF01 600.1827064 MHz
 SI 32768
 SF 600.1789961 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

NAME	TFish0916_product_011012	
EXNO	1	
PROCNO	2	
Date	20120110	
Time	13.38	
INSTRUM	spect	
PROBHD	5 mm BBO	BB-1H
PULPROG	zgpg30	
TD	65536	
SOLVENT	CDCl3	
NS	300	
DS	4	
SWH	35971.223	Hz
FIDRES	0.548877	Hz
AQ	0.9110143	sec
RG	7298.2	
DW	13.900	usec
DE	6.50	usec
TE	298.0	K
D1	2.0000000	sec
D11	0.0300000	sec
TD0	1	

```
===== CHANNEL f1 =====
NUC1                13C
P1                  10.00 usec
PL1                 -5.00 dB
PL1W                238.29847717 W
SFO1                150.9302211 MHz
```

```

===== CHANNEL f2 =====
CPDRP2                waltz16
NUC2                   1H
PCPD2                 80.00  usec
PL2                   -2.30  dB
PL12                  14.74  dB
PL13                  14.75  dB
PL2W                  63.98692703  W
PL12W                 1.26500225  W
PL13W                 1.26209271  W
SF02                  600.1814007  MHz
SI                    32768
SF                    150.95151300  MHz
WDW                    EM
SSB                    0
LB                    1.00  Hz
GB                    0
PC                    1.40

```