Mechanochemical synthesis of aromatic sulfonamides.

Satenik Mkrtchyan,^a Viktor O. Iaroshenko.^{a,b,c*}

^aLaboratory of Homogeneous Catalysis and Molecular Design at the Centre of Molecular and Macromolecular Studies, Polish Academy of Sciences,

Sienkiewicza 112, PL-90-363 Łodź, Poland.

^bDepartment of Chemistry, University of Helsinki, A.I. Virtasen aukio 1, 00014 Helsinki, Finland.

https://researchportal.helsinki.fi/en/persons/viktor-iaroshenko

E-mail: iva108@gmail.com

^cDepartment of Chemistry, Faculty of Natural Sciences, Matej Bel University, Tajovkého 40, 97401 Banska Bystrica, Slovakia.

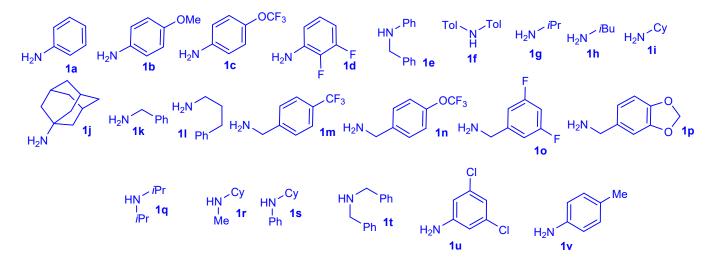
Table of Contents

(A) Experimental Section	53
(A-1) Scope of reagents used	<u>5</u> 3
(A-2) Reaction condition screening	<u>§</u> 5
(B) Characterization of products.	<u>5</u> 17
(C) Copies ¹ H and ¹³ C NMR spectra	<u>5</u> 44

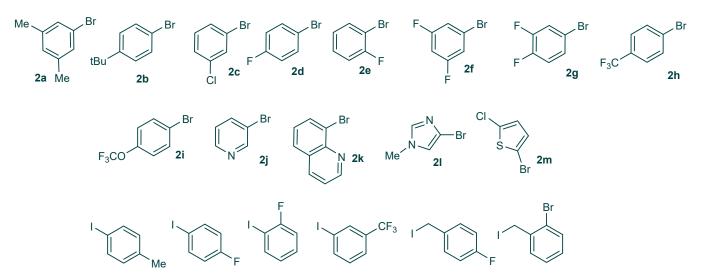
(A) Experimental Section.

Commercially available starting materials, reagents, catalysts, anhydrous and degassed solvents were used without further purification. Flash column chromatography was performed with Merck Silica gel 60 (230-400 mesh). The solvents for column chromatography were distilled before the use. Thin layer chromatography was carried out using Merck TLC Silica gel 60 F₂₅₄ and visualized by short-wavelength ultraviolet light or by treatment with potassium permanganate (KMnO₄) stain. ¹H, ¹³C and ¹⁹F NMR spectra were recorded on a Bruker 250, 400 and 500 MHz at 20°C. All ¹H NMR spectra are reported in parts per million (ppm) downfield of TMS and were measured relative to the signals for CHCl₃ (7.26 ppm) and DMSO (2.50 ppm). All ¹³C{¹H} NMR spectra were reported in perter to residual CHCl₃ (77.00 ppm) or DMSO (39.70 ppm) and were obtained with ¹H decoupling. Coupling constants, *J*, are reported in Hertz (Hz). Gas chromatographic analyses was performed on Gas Chromatograph Mass Spectrometer GCMS-QP2010 Ultra instrument. Mechanochemical synthesis was performed using the Retsch MM400 mill using the standard kit. Liquid chemicals were dosed using gas tight micro syringes. Isolation of obtained compounds was achieved by column chromatography on Silica gel. All commercially available compounds were purchased from appropriate vendors.

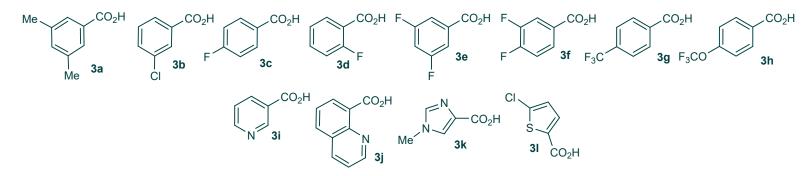
A-1. Scope of the reagents used.



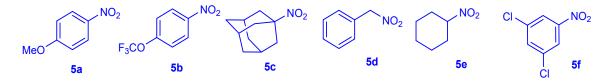
Scheme S1. List of amines.



Scheme S2. List of aryl bromides and aryl iodides.



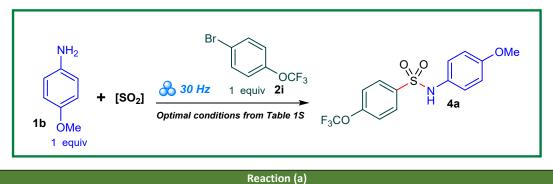
Scheme S3. List of aromatic carboxylic acids.



Scheme S4. List of nitro compounds.

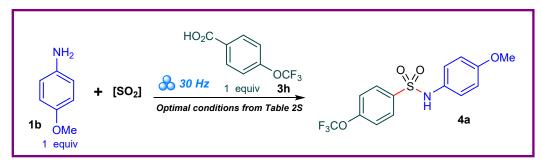
A-2. Reaction condition screening for arylation of *ortho*-hydroxyarylenaminones.

Table S1. Optimization of the reaction conditions.



entry	reaction components	frequency/time	yield (%) 4a ª
1	K ₂ S ₂ O ₅ (1.2 equiv.), cucurbit[7]uril (0.05 equiv.), CuBr ₂ (0.1 equiv.), r.t.	30Hz/60min	0
2	$K_2S_2O_5$ (1.2 equiv.), cucurbit[7]uril (0.05 equiv.), CuI (0.1 equiv.), r.t.	30Hz/60min	0
3	$K_2S_2O_5$ (1.2 equiv.), cucurbit[7]uril (0.05 equiv.), [RuCl ₂ (p-cymene)] ₂ (0.1 equiv.), r.t.	30Hz/60min	20
4	$K_2S_2O_5$ (1.2 equiv.), cucurbit[7]uril (0.05 equiv.), RuCl ₂ (PPh ₃) ₄ (0.1 equiv.), r.t.	30Hz/60min	35
5	K ₂ S ₂ O ₅ (1.2 equiv.), cucurbit[7]uril (0.05 equiv.), [Ir(1,5-cod)Cl] ₂ (0.03 equiv.), r.t., under Ar.	30Hz/60min	0
6	$K_2S_2O_5$ (1.2 equiv.), RhCl(PPh ₃) ₃ (0.05 equiv.), r.t., under Ar.	30Hz/60min	34
7	$K_2S_2O_5$ (1.2 equiv.), Pd(OAc) ₂ (0.05 equiv.), P(^t Bu) ₃ (0.1 equiv.), r.t., under Ar.	30Hz/60min	31
8	$K_2S_2O_5$ (1.2 equiv.), Pd(OAc) ₂ (0.05 equiv.), S-Phos (0.1 equiv.), r.t., under Ar.	30Hz/60min	37
9	$K_2S_2O_5$ (1.2 equiv.), $Pd_2(dba)_3$ (0.05 equiv.), Xantphos (0.05 equiv.), r.t., under Ar.	30Hz/60min	44
10	$K_2S_2O_5$ (1.2 equiv.), PdBr ₂ (0.05 equiv.), Xantphos (0.05 equiv.), r.t., under Ar.	30Hz/60min	37
11	K ₂ S ₂ O ₅ (1.2 equiv.), (PPh ₃) ₂ PdCl ₂ (0.03 equiv.), r.t.	30Hz/60min	43
12	$K_2S_2O_5$ (1.2 equiv.), cucurbit[7]uril (0.05 equiv.), (PPh ₃) ₂ PdCl ₂ (0.03 equiv.), r.t.	30Hz/60min	81
13	$K_2S_2O_5$ (1.2 equiv.), cucurbit[6]uril (0.05 equiv.), (PPh ₃) ₂ PdCl ₂ (0.03 equiv.), r.t.	30Hz/60min	80
14	DABSO (1.1 equiv.), cucurbit[6]uril (0.05 equiv.), (PPh ₃) ₂ PdCl ₂ (0.03 equiv.), r.t.	30Hz/60min	82
15	K ₂ S ₂ O ₅ (1.2 equiv.), cucurbit[6]uril (0.05 equiv.), RuCl ₂ (PPh ₃) ₂ (0.03 equiv.), r.t.	30Hz/60min	53
16	$K_2S_2O_5$ (1.2 equiv.), cucurbit[6]uril (0.05 equiv.), (PPh ₃) ₂ NiBr ₂ (0.05 equiv.), r.t.	30Hz/60min	0
17	$K_2S_2O_5$ (1.2 equiv.), cucurbit[6]uril (0.05 equiv.), (PPh ₃) ₂ Nil ₂ (0.05 equiv.), r.t.	30Hz/60min	0
18	K ₂ S ₂ O ₅ (1.2 equiv.), cucurbit[6]uril (0.05 equiv.), (PPh ₃) ₂ CoCl ₂ (0.05 equiv.), r.t.	30Hz/60min	0
	Reactions in solution		
19	$K_2S_2O_5$ (1.2 equiv.), cucurbit[6]uril (0.05 equiv.), (PPh ₃) ₂ PdCl ₂ (0.03 equiv.), r.t., in 1,4-dioxane under Ar.	/10h	0
20	$K_2S_2O_5$ (1.2 equiv.), cucurbit[6]uril (0.05 equiv.), (PPh ₃) ₂ PdCl ₂ (0.03 equiv.), reflux, in 1,4-dioxane under Ar.	/10h	17
21	$K_2S_2O_5$ (1.2 equiv.), cucurbit[6]uril (0.05 equiv.), (PPh ₃) ₂ PdCl ₂ (0.03 equiv.), r.t., in DMF under Ar.	/10h	0
22	$K_2S_2O_5$ (1.2 equiv.), cucurbit[6]uril (0.05 equiv.), (PPh ₃) ₂ PdCl ₂ (0.03 equiv.), 100 °C, in DMF under Ar.	/10h	13
23	$K_2S_2O_5$ (1.2 equiv.), cucurbit[6]uril (0.05 equiv.), (PPh ₃) ₂ PdCl ₂ (0.03 equiv.), r.t., in EtOH under Ar.	/10h	0
24	K ₂ S ₂ O ₅ (1.2 equiv.), cucurbit[6]uril (0.05 equiv.), (PPh ₃) ₂ PdCl ₂ (0.03 equiv.), reflux, in EtOH under Ar.	/10h	31
25	$K_2S_2O_5$ (1.2 equiv.), cucurbit[6]uril (0.05 equiv.), (PPh ₃) ₂ PdCl ₂ (0.03 equiv.), r.t., in CH ₂ Cl ₂ under Ar.	/10h	0
26	$K_2S_2O_5$ (1.2 equiv.), cucurbit[6]uril (0.05 equiv.), (PPh ₃) ₂ PdCl ₂ (0.03 equiv.), reflux, in CH ₂ Cl ₂ under Ar.	/10h	0
27	$K_2S_2O_5$ (1.2 equiv.), cucurbit[6]uril (0.05 equiv.), (PPh ₃) ₂ PdCl ₂ (0.03 equiv.), r.t., in NEt ₃ under Ar.	/10h	0
28	K ₂ S ₂ O ₅ (1.2 equiv.), cucurbit[6]uril (0.05 equiv.), (PPh ₃) ₂ PdCl ₂ (0.03 equiv.), reflux, in NEt ₃ under Ar.	/10h	0

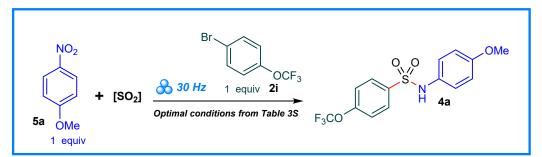
Table S2. Optimization of the reaction conditions.



	Reaction (b)		
entry	reaction components	frequency/time	yield (%) 4a ª
1	K ₂ S ₂ O ₅ (1.2 equiv.), cucurbit[6]uril (0.05 equiv.), (PPh ₃) ₂ PdCl ₂ (0.03 equiv.), r.t.	30Hz/60min	17
2	K ₂ S ₂ O ₅ (1.2 equiv.), cucurbit[6]uril (0.05 equiv.), (PPh ₃) ₂ PdCl ₂ (0.03 equiv.), Cul (1.4 equiv.), r.t.	30Hz/60min	31
3	K ₂ S ₂ O ₅ (1.2 equiv.), cucurbit[6]uril (0.05 equiv.), (PPh ₃) ₂ PdCl ₂ (0.03 equiv.), CuCl ₂ (1.4 equiv.), r.t.	30Hz/60min	37
4	K ₂ S ₂ O ₅ (1.2 equiv.), cucurbit[6]uril (0.05 equiv.), (PPh ₃) ₂ PdCl ₂ (0.03 equiv.), CuO (1.4 equiv.), r.t.	30Hz/60min	60
5	K ₂ S ₂ O ₅ (1.2 equiv.), cucurbit[6]uril (0.05 equiv.), (PPh ₃) ₂ PdCl ₂ (0.03 equiv.), CuBr ₂ (1.4 equiv.), r.t.	30Hz/60min	79
6	DABSO (1.1 equiv.), cucurbit[6]uril (0.05 equiv.), (PPh ₃) ₂ PdCl ₂ (0.03 equiv.), CuBr ₂ (1.4 equiv.), r.t.	30Hz/60min	83
	Reactions in solution	I	
7	$K_2S_2O_5$ (1.2 equiv.), cucurbit[6]uril (0.05 equiv.), (PPh ₃) ₂ PdCl ₂ (0.03 equiv.), CuBr ₂ (1.4 equiv.), reflux, in 1,4-dioxane under Ar.	/10h	0
8	K ₂ S ₂ O ₅ (1.2 equiv.), cucurbit[6]uril (0.05 equiv.), (PPh ₃) ₂ PdCl ₂ (0.03 equiv.), CuBr ₂ (1.4 equiv.), 130 °C, in DMF under Ar.	/10h	19
9	K ₂ S ₂ O ₅ (1.2 equiv.), cucurbit[6]uril (0.05 equiv.), (PPh ₃) ₂ PdCl ₂ (0.03 equiv.), CuBr ₂ (1.4 equiv.), reflux, in EtOH under Ar.	/10h	0
10	$K_2S_2O_5$ (1.2 equiv.), cucurbit[6]uril (0.05 equiv.), (PPh ₃) ₂ PdCl ₂ (0.03 equiv.), CuBr ₂ (1.4 equiv.), reflux, in CH ₂ Cl ₂ under Ar.	/10h	0
11	$K_2S_2O_5$ (1.2 equiv.), cucurbit[6]uril (0.05 equiv.), (PPh ₃) ₂ PdCl ₂ (0.03 equiv.), CuBr ₂ (1.4 equiv.), reflux, in NEt ₃ under Ar.	/10h	0

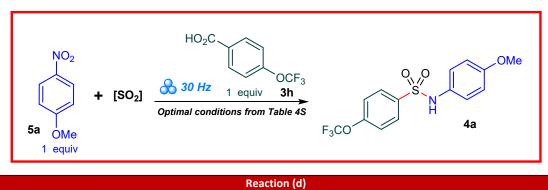
^a Isolated yield.

 Table S3. Optimization of the reaction conditions.



entry	reaction components	frequency/time	yield (%) 4 a
1	$K_2S_2O_5$ (1.2 equiv.), cucurbit[6]uril (0.05 equiv.), (PPh ₃) ₂ PdCl ₂ (0.03 equiv.), r.t.	30Hz/60min	21
2	$K_2S_2O_5$ (4.8 equiv.), cucurbit[6]uril (0.05 equiv.), (PPh ₃) ₂ PdCl ₂ (0.03 equiv.), r.t.	30Hz/60min	68
	Reactions in solution		
3	$K_2S_2O_5$ (4.8 equiv.), cucurbit[6]uril (0.05 equiv.), (PPh ₃) ₂ PdCl ₂ (0.03 equiv.), reflux, in 1,4-dioxane under Ar.	/10h	0
4	K ₂ S ₂ O ₅ (4.8 equiv.), cucurbit[6]uril (0.05 equiv.), (PPh ₃) ₂ PdCl ₂ (0.03 equiv.), 130 °C, in DMF under Ar.	/10h	17
5	$K_2S_2O_5$ (4.8 equiv.), cucurbit[6]uril (0.05 equiv.), (PPh ₃) ₂ PdCl ₂ (0.03 equiv.), reflux, in EtOH under Ar.	/10h	33
6	$K_2S_2O_5$ (4.8 equiv.), cucurbit[6]uril (0.05 equiv.), (PPh ₃) ₂ PdCl ₂ (0.03 equiv.), reflux, in CH ₂ Cl ₂ under Ar.	/10h	0
7	K ₂ S ₂ O ₅ (4.8 equiv.), cucurbit[6]uril (0.05 equiv.), (PPh ₃) ₂ PdCl ₂ (0.03 equiv.), reflux, in NEt ₃ under Ar.	/10h	0

Table S4. Optimization of the reaction conditions.



entry	reaction components	frequency/time	yield (%) 4a ^a
1	K ₂ S ₂ O ₅ (1.2 equiv.), cucurbit[6]uril (0.05 equiv.), (PPh ₃) ₂ PdCl ₂ (0.03 equiv.), CuBr ₂ (1.4 equiv.), r.t.	30Hz/60min	17
2	K ₂ S ₂ O ₅ (4.8 equiv.), cucurbit[6]uril (0.05 equiv.), (PPh ₃) ₂ PdCl ₂ (0.03 equiv.), CuBr ₂ (1.4 equiv.), r.t.	30Hz/60min	61
	Reactions in solution		
3	K ₂ S ₂ O ₅ (4.8 equiv.), cucurbit[6]uril (0.05 equiv.), (PPh ₃) ₂ PdCl ₂ (0.03 equiv.), CuBr ₂ (1.4 equiv.), reflux, in 1,4-dioxane under Ar.	/10h	0
4	K ₂ S ₂ O ₅ (4.8 equiv.), cucurbit[6]uril (0.05 equiv.), (PPh ₃) ₂ PdCl ₂ (0.03 equiv.), CuBr ₂ (1.4 equiv.), 130 °C, in DMF under Ar.	/10h	21
5	K ₂ S ₂ O ₅ (4.8 equiv.), cucurbit[6]uril (0.05 equiv.), (PPh ₃) ₂ PdCl ₂ (0.03 equiv.), CuBr ₂ (1.4 equiv.), reflux, in EtOH under Ar.	/10h	0
6	K ₂ S ₂ O ₅ (4.8 equiv.), cucurbit[6]uril (0.05 equiv.), (PPh ₃) ₂ PdCl ₂ (0.03 equiv.), CuBr ₂ (1.4 equiv.), reflux, in NEt ₃ under Ar.	/10h	0

^a Isolated yield.

Reaction procedures with optimised reaction conditions.

General procedure for the synthesis of sulfonamides 4 by the reaction between amines 1, aryl bromides 2 and $K_2S_2O_5$.

In air, to 5 mL grinding vessel (made of stainless) equipped with three balls (made of stainless, diameter: 5 mm) was placed consequently an appropriate aryl bromide (1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (267 mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50 mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21 mg, 0.03 mmol, 0.03 equiv.); then an appropriate amine (1.0 mmol, 1.0 equiv.) was added and the reaction vessel was properly capped. Finally, the reaction vessel was installed on the mill and subjected to milling at 30 Hz for 60 minutes. After completion of the reaction, the content of the vessel was generously treated with distilled water, filtered and washed two times with distilled water and finally properly dried in vacuum. The resulted crude was directly subjected to gradient flash chromatography on silica gel to isolate the desired compound. The gram scale synthesis was performed on 10 mmol of the starting amine in 25 mL grinding vessel using three 10 mm balls.

General procedure for the synthesis of sulfonamides 4 by the reaction between amines 1, aromatic carboxylic acids 3 and $K_2S_2O_5$.

In air, to 5 mL grinding vessel (made of stainless) equipped with three balls (made of stainless, diameter: 5 mm) was placed consequently an appropriate aromatic carboxylic acid (1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (267[°]mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50[°]mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°]mg, 0.03 mmol, 0.03 equiv.); CuBr₂ (313 mg, 1.4 mmol, 1.4 equiv.); then an appropriate amine (1.0 mmol, 1.0 equiv.) was added

and the reaction vessel was properly capped. Finally, the reaction vessel was installed on the mill and subjected to milling at 30 Hz for 60 minutes. After completion of the reaction, the content of the vessel was generously treated with distilled water, filtered and washed two times with distilled water and finally properly dried in vacuum. The resulted crude was directly subjected to gradient flash chromatography on silica gel to isolate the desired compound.

The gram scale synthesis was performed on 10 mmol of the starting amine in 25 mL grinding vessel using three 10 mm balls.

General procedure for the synthesis of sulfonamides 4 by the reaction between amines 1, aryl bromides 2 and DABSO.

In air, to 5 mL grinding vessel (made of stainless) equipped with three balls (made of stainless, diameter: 5 mm) was placed consequently an appropriate aryl bromide (1.0 mmol, 1.0 equiv.), DABSO (264 mg, 1.1 mmol, 1.1 equiv.), cucurbit[6]uril (50 mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21 mg, 0.03 mmol, 0.03 equiv.); then an appropriate amine (1.0 mmol, 1.0 equiv.) was added and the reaction vessel was properly capped. Finally, the reaction vessel was installed on the mill and subjected to milling at 30 Hz for 60 minutes. After completion of the reaction, the content of the vessel was generously treated with distilled water, filtered and washed two times with distilled water and finally properly dried in vacuum. The resulted crude was directly subjected to gradient flash chromatography on silica gel to isolate the desired compound. The gram scale synthesis was performed on 10 mmol of the starting amine in 25 mL grinding vessel using three 10 mm balls.

General procedure for the synthesis of sulfonamides 4 by the reaction between amines 1, aromatic carboxylic acids 3 and DABSO.

In air, to 5 mL grinding vessel (made of stainless) equipped with three balls (made of stainless, diameter: 5 mm) was placed consequently an appropriate aromatic carboxylic acid (1.0 mmol, 1.0 equiv.), DABSO (264° mg, 1.1 mmol, 1.1 equiv.), cucurbit[6]uril (50° mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21° mg, 0.03 mmol, 0.03 equiv.); CuBr₂ (313 mg, 1.4 mmol, 1.4 equiv.); then an appropriate amine (1.0 mmol, 1.0 equiv.) was added and the reaction vessel was properly capped. Finally, the reaction vessel was installed on the mill and subjected to milling at 30 Hz for 60 minutes. After completion of the reaction, the content of the vessel was generously treated with distilled water, filtered and washed two times with distilled water and finally properly dried in vacuum. The resulted crude was directly subjected to gradient flash chromatography on silica to isolate the desired compound.

The gram scale synthesis was performed on 10 mmol of the starting amine in 25 mL grinding vessel using three 10 mm balls.

General procedure for the synthesis of sulfonamides 4 by the reaction between nitro compounds 5, aryl bromides 2 and $K_2S_2O_5$.

In air, to 10 mL grinding vessel (made of stainless) equipped with three balls (made of stainless, diameter: 5 mm) was placed consequently an appropriate aryl bromide (1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (1.068 g, 4.8 mmol, 4.8 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.); then an appropriate nitro compound (1.0 mmol, 1.0 equiv.) was added and the reaction vessel was properly capped. Finally, the reaction vessel was installed on the mill and subjected to milling at 30 Hz for 60 minutes. After completion of the reaction, the content of the vessel was generously treated with distilled water, filtered and washed two times with distilled water and finally properly dried in vacuum. The resulted crude was directly subjected to gradient flash chromatography on silica to isolate the desired compound. The gram scale synthesis was performed on 10 mmol of the starting amine in 25 mL grinding vessel using three 10 mm balls.

General procedure for the synthesis of sulfonamides 4 by the reaction between nitro compounds 5, aromatic carboxylic acids 3 and $K_2S_2O_5$.

In air, to 10 mL grinding vessel (made of stainless) equipped with three balls (made of stainless, diameter: 5 mm) was placed consequently an appropriate aromatic carboxylic acid (1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (1.068 g, 4.8 mmol, 4.8 equiv.), cucurbit[6]uril (50 mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21 mg, 0.03 mmol, 0.03 equiv.); CuBr₂ (313 mg, 1.4 mmol, 1.4 equiv.); then an appropriate nitro compound (1.0 mmol, 1.0 equiv.) was added and the reaction vessel was properly capped. Finally, the reaction vessel was installed on the mill and subjected to milling at 30 Hz for 60 minutes. After completion of the reaction, the content of the vessel was generously treated with distilled water, filtered and washed two times with distilled water and finally properly dried in vacuum. The resulted crude was directly subjected to gradient flash chromatography on silica gel to isolate the desired compound.

The gram scale synthesis was performed on 10 mmol of the starting amine in 25 mL grinding vessel using three 10 mm balls.

General procedure for the synthesis of sulfonamides 4 by the reaction between amines 1, aryl iodides and $K_2S_2O_5$.

In air, to 5 mL grinding vessel (made of stainless) equipped with three balls (made of stainless, diameter: 5 mm) was placed consequently an appropriate aryl iodide (1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (267 mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.); then an appropriate amine (1.0 mmol, 1.0 equiv.) was added and the reaction vessel was properly capped. Finally, the reaction vessel was installed on the mill and subjected to milling at 30 Hz for 60 minutes. After completion of the reaction, the content of the vessel was generously treated with distilled water, filtered and washed two times with distilled water and finally properly dried in vacuum. The resulted crude was directly subjected to gradient flash chromatography on silica gel to isolate the desired compound.

Procedure for the synthesis of sulfonamide 4aa by the reaction between amine 1t, corresponding aryl palladium bromide intermediate 9 and $K_2S_2O_5$ (Scheme S6a).

In air, to 5 mL grinding vessel (made of stainless) equipped with three balls (made of stainless, diameter: 5 mm) was placed consequently the freshly prepared $CF_3Ph-Pd(PPh_3)_2$ -Br **9** (428 mg, 0.5 mmol, 1.0 equiv.), $K_2S_2O_5$ (134 mg, 0.6 mmol, 1.2 equiv.), cucurbit[6]uril (25° mg, 0.025 mmol, 0.05 equiv.); then an appropriate amine (99 mg, 0.5 mmol, 1.0 equiv.) was added and the reaction vessel was properly capped. Finally, the reaction vessel was installed on the mill and subjected to milling at 30 Hz for 60 minutes. After completion of the reaction, the content of the vessel was generously treated with distilled water, subsequently extracted two times by dichloromethane. The organic layers were combined washed with water and brine and then dried over MgSO₄. The dichloromethane solution was concentrated in vacuum and the resulted crude was directly subjected to gradient flash chromatography on silica to isolate the desired compound.

Procedure for the arylation of p-tolylamine by 1-bromo-4-methylbenzene (Scheme S6b).

In dry box, to 5 mL grinding vessel (made of stainless) equipped with three balls (made of stainless, diameter: 5 mm) was placed consequently 1bromo-4-methylbenzene (171 mg, 1.0 mmol, 1.0 equiv.), K_2CO_3 (166 mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50 mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21 mg, 0.03 mmol, 0.03 equiv.); then p-tolylamine (107 mg, 1.0 mmol, 1.0 equiv.) was added and the reaction vessel was properly capped. Finally, the reaction vessel was installed on the mill and subjected to milling at 30 Hz for 60 minutes. After completion of the reaction, the content of the vessel was generously treated with distilled water, subsequently extracted two times by dichloromethane. The organic layers were combined washed with water and brine and then dried over MgSO₄. The dichloromethane solution was concentrated in vacuum and the resulted crude was directly subjected to gradient flash chromatography on silica to isolate the desired compound.

Procedure for the arylation of p-tolylamine by 4-methylbenzoic acid (Scheme S6c).

In dry box, to 5 mL grinding vessel (made of stainless) equipped with three balls (made of stainless, diameter: 5 mm) was placed consequently an 4-methylbenzoic acid (136 mg, 1.0 mmol, 1.0 equiv.), K₂CO₃ (166 mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.); CuBr₂ (313 mg, 1.4 mmol, 1.4 equiv.); then p-tolylamine (107 mg, 1.0 mmol, 1.0 equiv.) was added and the reaction vessel was properly capped. Finally, the reaction vessel was installed on the mill and subjected to milling at 30 Hz for 60 minutes. After completion of the reaction, the content of the vessel was generously treated with distilled water, filtered and washed two times with distilled water and finally properly dried in vacuum. The resulted crude was directly subjected to gradient flash chromatography on silica gel to isolate the desired compound.

Procedure for the preparation of 1-(methylsulfonyl)-4-(trifluoromethoxy)benzene 11 by a reaction between 1-bromo-4-(trifluoromethoxy)benzene 10, MeI and K₂S₂O₅ (Scheme S6d).

In dry box, to 5 mL grinding vessel (made of stainless) equipped with three balls (made of stainless, diameter: 5 mm) was placed consequently 1bromo-4-(trifluoromethoxy)benzene **10** (241 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (267 mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.); then MeI (426 mg, 3.0 mmol, 3.0 equiv.) was added and the reaction vessel was properly capped. Finally, the reaction vessel was installed on the mill and subjected to milling at 30 Hz for 60 minutes. After completion of the reaction, the content of the vessel was generously treated with distilled water, subsequently extracted two times by dichloromethane. The organic layers were combined washed with water and brine and then dried over MgSO₄. The dichloromethane solution was concentrated in vacuum and the resulted crude was directly subjected to gradient flash chromatography on silica to isolate the desired compound.

Procedure for the preparation of sulfonyldibenzene 13 by a reaction between bromobenzene 12, phenyl boronic acid and K₂S₂O₅ (Scheme S6e).

In dry box, to 5 mL grinding vessel (made of stainless) equipped with three balls (made of stainless, diameter: 5 mm) was placed consequently bromobenzene **12** (157 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (267 mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.); then phenyl boronic acid (122 mg, 1.0 mmol, 1.0 equiv.) was added and the reaction vessel was properly capped. Finally, the reaction vessel was installed on the mill and subjected to milling at 30 Hz for 60 minutes. After completion of the reaction, the content of the vessel was generously treated with distilled water, subsequently extracted two times by dichloromethane. The organic layers were combined washed with water and brine and then dried over MgSO₄. The dichloromethane solution was concentrated in vacuum and the resulted crude was directly subjected to gradient flash chromatography on silica to isolate the desired compound.

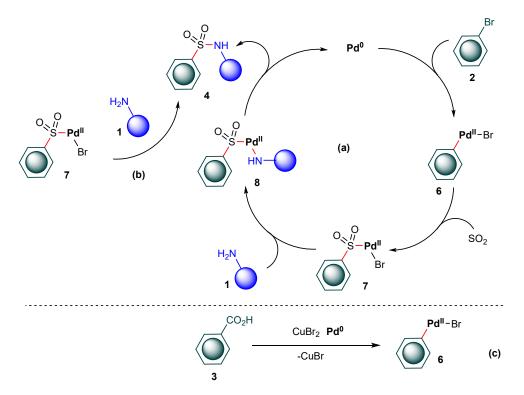
Procedure for the synthesis of 3,5-dichloroaniline 1u from nitro compound 5f (Scheme S6f).

In air, to 10 mL grinding vessel (made of stainless) equipped with three balls (made of stainless, diameter: 5 mm) was placed consequently nitro compound **5f** (192 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (1.068 g, 4.8 mmol, 4.8 equiv.), (PPh₃)₂PdCl₂ (21 mg, 0.03 mmol, 0.03 equiv.); then distilled water (0.036 mL, 2.0 mmol, 2.0 equiv.) was added and the reaction vessel was properly capped. Finally, the reaction vessel was installed on the mill and subjected to milling at 30 Hz for 35 minutes. After completion of the reaction, the content of the vessel was generously treated with distilled water and small portion of dichloromethane, subsequently extracted two times by dichloromethane. The organic layers were combined washed with water and brine and then dried over MgSO₄. The dichloromethane solution was concentrated in vacuum and the resulted crude was directly subjected to gradient flash chromatography on silica to isolate the desired compound.

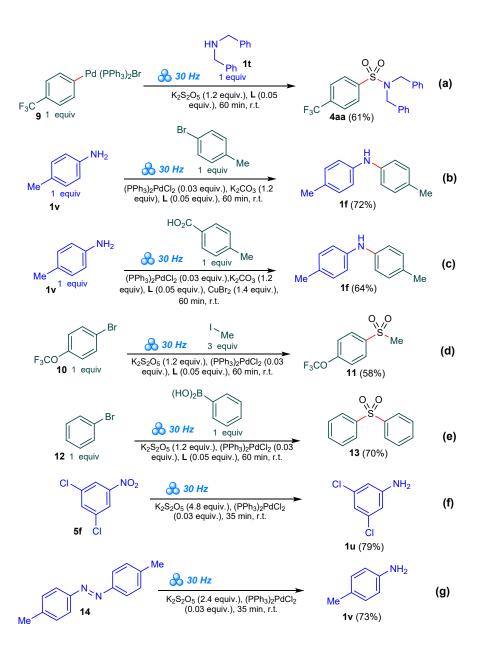
Procedure for the synthesis of p-toluidine 1v from 1,2-di-p-tolyldiazene 14 (Scheme S6g).

In air, to 10 mL grinding vessel (made of stainless) equipped with three balls (made of stainless, diameter: 5 mm) was placed consequently 1,2-dip-tolyldiazene **14** (210 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (0.53 g, 2.4 mmol, 2.4 equiv.), (PPh₃)₂PdCl₂ (21[°]₁mg, 0.03 mmol, 0.03 equiv.); then distilled water (0.036 mL, 2.0 mmol, 2.0 equiv.) was added and the reaction vessel was properly capped. Finally, the reaction vessel was installed on the mill and subjected to milling at 30 Hz for 35 minutes. After completion of the reaction, the content of the vessel was generously treated with distilled water, subsequently extracted two times by dichloromethane. The organic layers were combined washed with water and brine and then dried over MgSO₄. The dichloromethane solution was concentrated in vacuum and the resulted crude was directly subjected to gradient flash chromatography on silica to isolate the desired compound.

The reaction mechanisms for the developed synthetic protocols.



Scheme S5. Putative reaction mechanisms for the developed synthetic protocols.



Scheme S6. Control experiments.

(B) Characterization of products.

di-p-tolylamine 1f.

Me

This compound was prepared starting from 1-bromo-4-methylbenzene (171 mg, 1.0 mmol, 1.0 equiv.), K_2CO_3 (166 mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50 mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21 mg, 0.03 mmol, 0.03 equiv.) and p-tolylamine **1v** (107 mg, 1.0 mmol, 1.0 equiv.) The purification was performed by column chromatography on silica gel to provide the desired compound **1f** (142 mg, 0.72 mmol, 72%).

Alternatively, the title compound was prepared starting from 4-methylbenzoic acid (136 mg, 1.0 mmol, 1.0 equiv.), K_2CO_3 (166 mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50 mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21 mg, 0.03 mmol, 0.03 equiv.); CuBr₂ (313 mg, 1.4 mmol, 1.4 equiv.) and p-tolylamine **1v** (107 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **1f** (126 mg, 0.64 mmol, 64%).

Colorless solid, mp 79-80 °C. ¹H NMR (500 MHz, CDCl₃): δ 2.44 (s, 6H, 2xMe), 4.46 (br. s, H, NH), 7.08 (d, 4H, ³*J* = 8.4 Hz, CH_{Ar}), 7.21 (d, 4H, ³*J* = 8.4 Hz, CH_{Ar}).

¹³C{¹H} NMR (126 MHz, CDCl₃): δ 20.6, 117.8, 129.7, 129.9, 141.0. MS (GC, 70eV): m/z (%) = 197 (M⁺, 100), 180 (16), 91 (18).

Anal. calcd. for C₁₄H₁₅N: C, 85.24; H, 7.66; N, 7.10. Found: C, 85.19; H, 7.48, N, 7.33.

3,5-dichloroaniline 1u.

 NH_2

This compound was prepared starting from nitro compound **5f** (192 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (1.068 g, 4.8 mmol, 4.8 equiv.), (PPh₃)₂PdCl₂ (21^î mg, 0.03 mmol, 0.03 equiv.) and distilled water (0.036 mL, 2.0 mmol, 2.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **1u** (128 mg, 0.79 mmol, 79%). Colorless solid, mp 49-50 °C. ¹H NMR (500 MHz, DMSO-*d*₆): δ 3.76 (s, 2H, NH₂), 6.52 (d, 2H, ⁴J = 1.7 Hz, CH_{Ar}), 6.72 (t, 1H, ⁴J = 1.7 Hz, CH_{Ar}).

¹³C{¹H} NMR (126 MHz, CDCl₃): δ 113.1, 118.2, 135.3, 148.2.

MS (GC, 70eV): m/z (%) = 161 (M⁺, 100), 126 (18), 90 (22).

p-toluidine 1v.

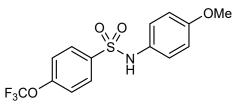
NH₂

This compound was prepared starting from 1,2-di-p-tolyldiazene **14** (210 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (0.53 g, 2.4 mmol, 2.4 equiv.), (PPh₃)₂PdCl₂ (21^î mg, 0.03 mmol, 0.03 equiv.) and distilled water (0.036 mL, 2.0 mmol, 2.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **1v** (78 mg, 0.73 mmol, 73%).

Colorless solid, mp 41-42 °C. ¹**H NMR** (500 MHz, DMSO-*d*₆): δ 2.13 (s, 3H, Me), 4.77 (s, 2H, NH₂), 6.47 (d, 2H, ³*J* = 7.7 Hz, CH_{Ar}), 6.81 (d, 2H, ³*J* = 8.4 Hz, CH_{Ar}).

¹³C{¹H} NMR (126 MHz, CDCl₃): δ 20.1, 114.1, 124.0, 129.3, 146.1.

N-(4-methoxyphenyl)-4-(trifluoromethoxy)benzenesulfonamide 4a.



This compound was prepared starting from aryl bromide **2i** (240 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (267 mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.) and amine **1b** (123 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4a** (278 mg, 0.80 mmol, 80%). The gram scale synthesis was performed on 10 mmol of the starting amine in 25 mL grinding vessel using three 10 mm balls and desired **4a** was prepared in 77% (2.67 g, 7.7 mmol) yield.

Alternatively, the title compound was prepared starting from aromatic carboxylic acid **3h** (206 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (267 mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50 mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21 mg, 0.03 mmol, 0.03 equiv.); CuBr₂ (313 mg, 1.4 mmol, 1.4 equiv.) and amine **1b** (123 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4a** (274 mg, 0.79 mmol, 79%). The gram scale synthesis was performed on 10 mmol of the starting amine in 25 mL grinding vessel using three 10 mm balls and desired **4a** was prepared in 72% (2.50 g, 7.2 mmol) yield.

Alternatively, the title compound was prepared starting from aryl bromide **2i** (240 mg, 1.0 mmol, 1.0 equiv.), DABSO (264 mg, 1.1 mmol, 1.1 equiv.), cucurbit[6]uril (50 mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21 mg, 0.03 mmol, 0.03 equiv.) and amine **1b** (123 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4a** (284 mg, 0.82 mmol, 82%). The gram scale synthesis was performed on 10 mmol of the starting amine in 25 mL grinding vessel using three 10 mm balls and desired **4a** was prepared in 74% (2.57 g, 7.4 mmol) yield.

Alternatively, the title compound was prepared starting from aromatic carboxylic acid **3h** (206 mg, 1.0 mmol, 1.0 equiv.), DABSO (264 mg, 1.1 mmol, 1.1 equiv.), cucurbit[6]uril (50 mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21 mg, 0.03 mmol, 0.03 equiv.); CuBr₂ (313 mg, 1.4 mmol, 1.4 equiv.) and amine **1b** (123 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide

the desired compound **4a** (288 mg, 0.83 mmol, 83%). The gram scale synthesis was performed on 10 mmol of the starting amine in 25 mL grinding vessel using three 10 mm balls and desired **4a** was prepared in 69% (2.39 g, 6.9 mmol) yield.

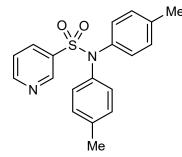
Alternatively, the title compound was prepared starting from aryl bromide **2i** (240 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (1.068 g, 4.8 mmol, 1.2 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.) and nitro compound **5a** (153 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4a** (236 mg, 0.68 mmol, 68%).

Alternatively, the title compound was prepared starting from aromatic carboxylic acid **3h** (206 mg, 1.0 equiv.), $K_2S_2O_5$ (1.068 g, 4.8 mmol, 1.2 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.); CuBr₂ (313 mg, 1.4 mmol, 1.4 equiv.) and nitro compound **5a** (153 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4a** (212 mg, 0.61mmol, 61%).

Yellow solid, mp 99 - 100 °C. ¹**H NMR** (500 MHz, CDCl₃): δ 3.84 (s, 3H, OMe), 6.88 (d, 2H, ³*J* = 8.8 Hz, CH_{Ar}), 6.92 (d, 2H, ³*J* = 8.8 Hz, CH_{Ar}), 7.54 (d, 2H, ³*J* = 7.5 Hz, CH_{Ar}), 7.64 (t, 2H, ³*J* = 7.9 Hz, CH_{Ar}), 7.76 (s, 2H, CH_{Ar}), 7.94 (d, 2H, ³*J* = 7.5 Hz, CH_{Ar}).

¹³C{¹H} NMR (126 MHz, CDCl₃): δ 55.5, 114.8, 120.3 (q, ¹*J*_{CF} = 256.2 Hz), 121.3, 125.7, 1266, 126.9, 130.8, 132.4, 141.0, 149.0, 161.3. HRMS (TOF MS ES+) m/z: [M - H]⁺: Calcd for C₁₄H₁₁NO₄SF₃ 346.0363. Found 346.0361.

N,N-di-p-tolylpyridine-3-sulfonamide 4b.



This compound was prepared starting from aryl bromide **2j** (157 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (267 mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.) and amine **1f** (197 mg, 1.0 mmol, 1.0 equiv.). The purification was

performed by column chromatography on silica gel to provide the desired compound **4b** (274 mg, 0.81 mmol, 81%). The gram scale synthesis was performed on 10 mmol of the starting amine in 25 mL grinding vessel using three 10 mm balls and desired **4b** was prepared in 80% (3.10 g, 8.0 mmol) yield.

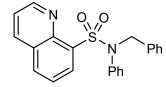
Alternatively, the title compound was prepared starting from aromatic carboxylic acid **3i** (123 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (267^î mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50^î mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21^î mg, 0.03 mmol, 0.03 equiv.); CuBr₂ (313 mg, 1.4 mmol, 1.4 equiv.) and amine **1f** (197 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4b** (294 mg, 0.87 mmol, 87%). The gram scale synthesis was performed on 10 mmol of the starting amine in 25 mL grinding vessel using three 10 mm balls and desired **4b** was prepared in 80% (3.10 g, 8.0 mmol) yield.

Yellowish solid, mp 118 - 120 °C. ¹H NMR (500 MHz, DMSO- d_6): δ 2.32 (s, 6H, 2xMe), 7.11 – 7.16 (m, 8H, CH_{Ar}), 7.41 – 7.44 (m, 1H, CH_{Ar}), 7.95 (dt, 1H, ³J = 8.0 Hz, ⁴J = 1.8 Hz, CH_{Ar}), 8.80 (d, 1H, ³J = 4.6 Hz, CH_{Ar}), 8.91 (dd, 1H, ⁴J = 2.0 Hz, CH_{Ar}).

¹³C{¹H} NMR (126 MHz, DMSO-*d*₆): δ 21.0, 123.5, 128.1, 130.1, 135.3, 137.0, 138.0, 138.3, 148.5, 153.1.

HRMS (TOF MS ES+) m/z: $[M + H]^+$: Calcd for C₁₉H₁₉N₂O₂S 339.1175. Found 339.1167.

N-benzyl-N-phenylquinoline-8-sulfonamide 4c.



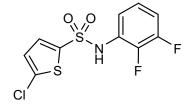
This compound was prepared starting from aryl bromide **2k** (207 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (267 mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.) and amine **1e** (183 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4c** (337 mg, 0.90 mmol, 90%). Alternatively, the title compound was prepared starting from aromatic carboxylic acid **3j** (173 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (267[°] mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.); CuBr₂ (313 mg, 1.4 mmol, 1.4 equiv.) and amine **1e** (183 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4c** (310 mg, 0.83 mmol, 83%).

Yellowish solid, mp 167 - 170 °C. ¹H NMR (500 MHz, DMSO- d_6): δ 5.40 (s, 2H, CH₂), 6.94 (d, 2H, ³*J* = 7.4 Hz, CH_{Ar}), 7.05 - 7.10 (m, 3H, CH_{Ar}), 7.22 - 7.24 (m, 1H, CH_{Ar}), 7.30 (t, 2H, ³*J* = 7.9 Hz, CH_{Ar}), 7.36 (d, 2H, ³*J* = 7.5 Hz, CH_{Ar}), 7.63 (t, 1H, ³*J* = 7.6 Hz, CH_{Ar}), 7.77 - 7.79 (m, 1H, CH_{Ar}), 8.19 (dd, 1H, ³*J* = 7.4 Hz, ⁴*J* = 1.2 Hz, CH_{Ar}), 8.28 (d, 1H, ³*J* = 7.8 Hz, CH_{Ar}), 8.58 (dd, 1H, ³*J* = 8.4 Hz, ⁴*J* = 1.7 Hz, CH_{Ar}), 9.24 - 9.25 (m, 1H, CH_{Ar}).

¹³C{¹H} NMR (126 MHz, DMSO-*d₆*): δ 56.4, 122.6, 125.6, 127.1, 127.2, 127.8, 127.9, 128.3, 128.6, 128.8, 133.0, 134.3, 136.2, 137.1, 138.1, 139.0, 143.2, 151.7.

HRMS (TOF MS ES+) m/z: [M + H]⁺: Calcd for C₂₂H₁₉N₂O₂S 375.1178. Found 375.1167.

5-chloro-N-(2,3-difluorophenyl)thiophene-2-sulfonamide 4d.



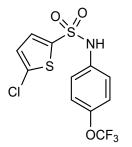
This compound was prepared starting from aryl bromide **2m** (196 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (267 mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.) and amine **1d** (129 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4d** (222 mg, 0.72 mmol, 72%). Alternatively, the title compound was prepared starting from aromatic carboxylic acid **3l** (162 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (267[°] mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.); CuBr₂ (313 mg, 1.4 mmol, 1.4 equiv.) and amine **1d** (129 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica to provide the desired compound **4d** (229 mg, 0.74 mmol, 1.0 equiv.).

Light brown solid, mp 129- 130 °C. ¹**H NMR** (500 MHz, CDCl₃): δ 6.97 – 7.00 (m, 2H, NH, CH_{Ar}), 7.15 (q, 1H, ³*J* = 5.4 Hz, CH_{Ar}), 7.33 (q, 1H, ³*J* = 8.1 Hz, CH_{Ar}), 7.59 (q, 2H, ⁴*J* = 3.7 Hz, CH_{Ar}).

¹³C{¹H} NMR (126 MHz, CDCl₃): δ 120.3 (d, J_{CF} = 16.8 Hz), 123.1 (d, J_{CF} = 9.9 Hz), 124.0 (m), 127.0, 127.8, 135.6, 135.9, 141.1, 148.8 (dd, ¹ J_{CF} = 260.7 Hz, J_{CF} = 12.8 Hz), 151.0 (dd, ¹ J_{CF} = 254.3 Hz, J_{CF} = 11.4 Hz).

HRMS (TOF MS ES+) m/z: [M - H]⁺: Calcd for C₁₀H₅NO₂S₂F₂Cl 307.9426. Found 307.9418.

5-chloro-N-(4-(trifluoromethoxy)phenyl)thiophene-2-sulfonamide 4e.



This compound was prepared starting from aryl bromide **2m** (196 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (267 mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.) and amine **1c** (177mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4e** (303 mg, 0.85 mmol, 85%).

Alternatively, the title compound was prepared starting from aromatic carboxylic acid **3I** (162 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (267[°] mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.); CuBr₂ (313 mg, 1.4 mmol, 1.4 equiv.) and amine **1c** (177 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4e** (289 mg, 0.81 mmol, 81%).

Alternatively, the title compound was prepared starting from aryl bromide **2m** (196 mg, 1.0 mmol, 1.0 equiv.), DABSO (264 mg, 1.1 mmol, 1.1 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.) and amine **1c** (177 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4e** (260 mg, 0.73 mmol, 73%).

Alternatively, the title compound was prepared starting from aromatic carboxylic acid **3I** (162 mg, 1.0 mmol, 1.0 equiv.), DABSO (264 mg, 1.1 mmol, 1.1 equiv.), cucurbit[6]uril (50 mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21 mg, 0.03 mmol, 0.03 equiv.); CuBr₂ (313 mg, 1.4 mmol, 1.4

equiv.) and amine **1c** (177 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4e** (281 mg, 0.79 mmol, 79%).

Alternatively, the title compound was prepared starting from aryl bromide **2m** (196 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (1.068 g, 4.8 mmol, 4.8 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.) and nitro compound **5b** (207 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4e** (207 mg, 0.58 mmol, 58%).

Alternatively, the title compound was prepared starting from aromatic carboxylic acid **3I** (162 mg, 1.0 equiv.), $K_2S_2O_5$ (1.068 g, 4.8 mmol, 4.8 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.); CuBr₂ (313 mg, 1.4 mmol, 1.4 equiv.) and nitro compound **5b** (207 mg, (1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4e** (225 mg, 0.63mmol, 63%).

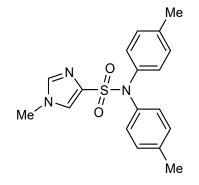
Yellow solid, mp 173 - 174 °C. ¹**H NMR** (500 MHz, CDCl₃): δ 7.00 (d, 2H, ⁴*J* = 3.2 Hz, CH_{Ar}), 7.18 (d, 2H, ³*J* = 8.3 Hz, CH_{Ar}), 7.25 (d, 2H, ³*J* = 8.3 Hz, CH_{Ar}), 7.56 (d, 2H, ⁴*J* = 3.2 Hz, CH_{Ar}).

¹³C{¹H} NMR (126 MHz, CDCl₃): δ 120.2 (q, ¹*J*_{CF} = 257.1 Hz), 127.0, 131.7, 132.7, 135.4, 136.1, 140.8, 150.7.

MS (GC, 70eV): m/z (%) = 357 (M⁺, 0.2), 298 (39), 292 (34), 257 (15), 183 (41), 181 (100), 133 (24), 105 (56).

Anal. calcd. for C₁₁H₇NO₃S₂F₃Cl: C, 36.93; H, 1.97; N, 3.92. Found: C, 36.99; H, 2.01, N, 3.92.

1-methyl-N,N-di-p-tolyl-1H-imidazole-4-sulfonamide 4f.



This compound was prepared starting from aryl bromide **2I** (160 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (267 mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.) and amine **1f** (197mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4f** (300 mg, 0.88 mmol, 88%).

Alternatively, the title compound was prepared starting from aromatic carboxylic acid **3k** (126 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (267 mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50 mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21 mg, 0.03 mmol, 0.03 equiv.); CuBr₂ (313 mg, 1.4 mmol, 1.4 equiv.) and amine **1f** (197 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4f** (280 mg, 0.82 mmol, 82%).

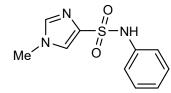
Yellowish solid, mp 120 - 121 °C. ¹H NMR (500 MHz, CDCl₃): δ 2.29 (s, 6H, 2xMe), 3.60 (s, 3H, Me), 7.09 (d, 4H, ³J = 7.2 Hz, CH_{Ar}), 7.24 (s, 1H, Imidazole), 7.36 (dd, 4H, ³J = 8.4 Hz, ⁴J = 1.8 Hz, CH_{Ar}), 7.50 (s, 1H, Imidazole).

¹³C{¹H} NMR (126 MHz, CDCl₃): δ 20.9, 33.8, 117.8, 125.4, 128.1, 129.6, 136.7, 138.9, 139.1, 139.7.

MS (GC, 70eV): m/z (%) = 341 (M⁺, 22), 197 (16), 196 (100), 181 (63).

Anal. calcd. for C₁₈H₁₉N₃O₂S: C, 63.32; H, 5.61; N, 12.31; Found: C, 63.18; H, 5.65; N, 12.37.

1-methyl-N-phenyl-1H-imidazole-4-sulfonamide 4g.

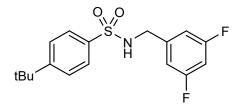


This compound was prepared starting from aryl bromide **2I** (160 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (267 mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.) and amine **1a** (93 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4g** (199 mg, 0.84 mmol, 84%). White solid, mp 203 - 204 °C. ¹**H NMR** (500 MHz, DMSO-*d*₆): δ 3.67 (s, 3H, Me), 7.00 (t, 1H, ³*J* = 7.5 Hz, CH_{Ar}), 7.19 – 7.20 (m, 2H, CH_{Ar}), 7.23 – 7.26 (m, 2H, CH_{Ar}), 7.76 (d, 1H, ⁴*J* = 0.8 Hz, Imidazole), 7.85 (d, 1H, ⁴*J* = 1.0 Hz, Imidazole), 10.27 (s, 1H, NH).

¹³C{¹H} NMR (126 MHz, DMSO-*d*₆): δ 33.5, 119.1, 123.2, 125.4, 128.9, 138.2, 138.5, 139.8.

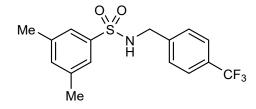
MS (GC, 70eV): m/z (%) = 237 (M⁺, 96), 173 (39), 145 (72), 132 (69), 92 (52), 65 (70), 42 (100). Anal. calcd. for $C_{10}H_{11}N_3O_2S$: C, 50.62; H, 4.67; N, 17.71; Found: C, 50.69; H, 4.73; N, 17.63.

4-(tert-butyl)-N-(3,5-difluorobenzyl)benzenesulfonamide 4h.



This compound was prepared starting from aryl bromide **2b** (212 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (267 mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.) and amine **1o** (143 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4h** (305 mg, 0.90 mmol, 90%). White solid, mp 99- 100 °C. ¹**H NMR** (500 MHz, CDCl₃): δ 1.31 (s, 9H, *t*Bu), 4.17 (d, 2H, ³*J* = 4.5 Hz, CH₂), 5.43 (s, 1H, NH), 6.59 – 6.64 (m, 1H, CH_{Ar}), 6.67 – 6.72 (m, 1H, CH_{Ar}), 7.17 – 7.22 (m, 1H, CH_{Ar}), 7.41 (d, 2H, ³*J* = 8.5 Hz, CH_{Ar}), 7.69 (d, 2H, ³*J* = 8.5 Hz, CH_{Ar}). ¹³C{¹H} NMR (126 MHz, CDCl₃): δ 31.0, 35.0, 40.7, 103.6 (t, *J*_{CF} = 26.7 Hz), 111.1 (dd, *J*_{CF} = 21.3 Hz, *J*_{CF} = 3.8 Hz), 119.6 (m), 125.9, 126.8, 131.0 (m), 136.8, 156.4, 160.5 (dd, ¹*J*_{CF} = 235.6 Hz, *J*_{CF} = 12.2 Hz), 162.5 (dd, ¹*J*_{CF} = 236.5 Hz, *J*_{CF} = 12.2 Hz). HRMS (TOF MS ES-) m/z: [M - H]⁺: Calcd for C₁₇H₁₈NO₂SF₂ (M-H) 338.1026. Found 338.1026.

3,5-dimethyl-N-(4-(trifluoromethyl)benzyl)benzenesulfonamide 4i.



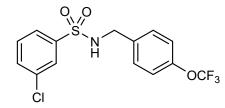
This compound was prepared starting from aryl bromide **2a** (184 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (267 mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.) and amine **1m** (191 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4i** (312 mg, 0.91 mmol, 91%).

Alternatively, the title compound was prepared starting from aromatic carboxylic acid **3a** (150 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (267[°] mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.); CuBr₂ (313 mg, 1.4 mmol, 1.4 equiv.) and amine **1m** (191 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4i** (322 mg, 0.94 mmol, 94%).

White solid, mp 129- 130 °C. ¹H NMR (500 MHz, CDCl₃): δ 2.31 (s, 6H, 2xMe), 4.17 (d, 2H, ³*J* = 6.0 Hz, CH₂), 5.49 (s, 1H, NH), 7.16 (s, 1H, CH_{Ar}), 7.31 (d, 2H, ³*J* = 7.9 Hz, CH_{Ar}), 7.42 (s, 2H, CH_{Ar}), 7.48 (d, 2H, ³*J* = 7.7 Hz, CH_{Ar}).

¹³C{¹H} NMR (126 MHz, CDCl₃): δ 21.1, 46.6, 123.9 (q, ¹J_{CF} = 272.0 Hz), 124.5, 125.3 (m), 128.0, 129.8 (q, ²J_{CF} = 32.2 Hz), 134.4, 139.2, 139.3, 140.6. HRMS (TOF MS ES+) m/z: [M + H]⁺: Calcd for C₁₆H₁₇NO₂SF₃ (M+H) 344.0939. Found 344.0932.

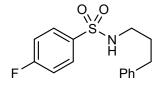
3-chloro-N-(4-(trifluoromethoxy)benzyl)benzenesulfonamide 4j.



This compound was prepared starting from aryl bromide **2c** (190 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (267 mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.) and amine **1n** (191 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4j** (312 mg, 0.95 mmol, 95%). Alternatively, the title compound was prepared starting from aromatic carboxylic acid **3b** (157 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (267[°] mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.); CuBr₂ (313 mg, 1.4 mmol, 1.4 equiv.) and amine **1n** (191 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound was prepared to provide the desired to provide the desired compound was prepared to provide the desired compound **4j** (322 mg, 0.91 mmol, 91%). Yellowish solid, mp 85 - 86 °C. ¹**H NMR** (500 MHz, CDCl₃): δ 4.16 (d, 2H, ³*J* = 6.3 Hz, NCH₂), 5.34 (br. s, 1H, NH), 7.09 (d, 2H, ³*J* = 8.1 Hz, CH_{Ar}), 7.21 (d, 2H, ³*J* = 8.6 Hz, CH_{Ar}), 7.40 (t, 1H, ³*J* = 8.1 Hz, CH_{Ar}), 7.52 (dd, 1H, ³*J* = 8.1 Hz, ⁴*J* = 0.9 Hz, CH_{Ar}), 7.68 (t, 1H, ³*J* = 7.9 Hz, CH_{Ar}), 7.79 (t, 1H, ⁴*J* = 1.8 Hz, CH_{Ar}).

¹³C{¹H} NMR (126 MHz, CDCl₃): δ 46.5, 120.3 (q, ¹*J*_{CF} = 257.3 Hz), 121.1, 125.0, 127.2, 129.3, 130.4, 132.9, 134.6, 135.3, 141.6, 148.8. MS (GC, 70eV): m/z (%) = 365 (M⁺, 1), 190 (100), 175 (13), 111 (17), 77 (17). Anal. calcd. for C₁₄H₁₁NO₃SClF₃: C, 45.97; H, 3.03; N, 3.83. Found: C, 46.09; H, 3.14; N, 3.95.

4-fluoro-N-(3-phenylpropyl)benzenesulfonamide 4k.

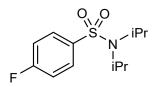


This compound was prepared starting from aryl bromide **2d** (174 mg, 1.0 mmol, 1.0 equiv.), K₂S₂O₅ (267 mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50[°]µmg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°]µmg, 0.03 mmol, 0.03 equiv.) and amine **1l** (135 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4k** (266 mg, 0.91 mmol, 91%). White solid, mp 62 - 63 °C. ¹**H NMR** (500 MHz, CDCl₃): δ 1.86 (quint, 2H, ³*J* = 7.3 Hz, CH₂), 2.67 (t, 2H, ³*J* = 7.4 Hz, CH₂), 3.02 (q, 2H, ³*J* = 6.6 Hz, CH₂), 5.14 (s, 1H, NH), 7.14 – 7.15 (m, 2H, CH_{Ar}), 7.21 – 7.26 (m, 3H, CH_{Ar}), 7.30 – 7.33 (m, 2H, CH_{Ar}), 7.93 – 7.96 (m, 2H, CH_{Ar}). ¹³C{¹H} NMR (126 MHz, CDCl₃): δ 31.0, 32.6, 42.5, 120.3 (d, *J*_{CF} = 22.3 Hz), 126.1, 128.3 (d, *J*_{CF} = 19.0 Hz), 129.7 (d, *J*_{CF} = 9.4 Hz), 135.8, 140.7, 165.0

 $(d, {}^{1}J_{CF} = 254.4 \text{ Hz}).$

HRMS (TOF MS ES+) m/z: [M + H]⁺: Calcd for C₁₅H₁₇NO₂SF 294.0968. Found 294.0964.

4-fluoro-N,N-diisopropylbenzenesulfonamide 41.

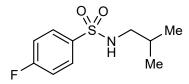


This compound was prepared starting from aryl bromide **2d** (174 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (267 mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.) and amine **1q** (101 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4l** (210 mg, 0.81 mmol, 81%). Alternatively, the title compound was prepared starting from aromatic carboxylic acid **3c** (140 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (267[°] mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.); CuBr₂ (313 mg, 1.4 mmol, 1.4 equiv.) and amine **1q** (101 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound sequiv.) and amine **1q** (101 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound of the desired compound **4l** (189 mg, 0.73 mmol, 73%).

White solid, mp 68 - 69 °C. ¹H NMR (500 MHz, CDCl₃): δ 1.23 (d, 12H, ³*J* = 7.1 Hz, 4xMe), 3.64 – 3.70 (m, 2H, 2xCH), 7.11 – 7.14 (m, 2H, CH_{Ar}), 7.84 – 7.86 (m, 2H, CH_{Ar}).

¹³C{¹H} NMR (126 MHz, CDCl₃): δ 21.8, 48.6, 115.8 (d, J_{CF} = 22.3 Hz), 129.6 (d, J_{CF} = 9.0 Hz), 137.2, 138.6, 164.6 (d, $^{1}J_{CF}$ = 257.2 Hz). HRMS (TOF MS ES+Na) m/z: [M + H]⁺: Calcd for C₁₂H₁₈NO₂SFNa 282.0945. Found 282.0940.

4-fluoro-N-isobutylbenzenesulfonamide 4m.



This compound was prepared starting from aryl bromide **2d** (174 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (267 mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.) and amine **1h** (73 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4m** (169 mg, 0.73 mmol, 73%).

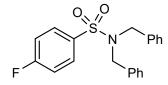
Alternatively, the title compound was prepared starting from aromatic carboxylic acid **3c** (140 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (267 mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50 mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21 mg, 0.03 mmol, 0.03 equiv.); CuBr₂ (313 mg, 1.4 mmol, 1.4 equiv.) and amine **1h** (73 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4m** (162 mg, 0.70 mmol, 70%).

White solid, mp 85 - 86 °C. ¹H NMR (500 MHz, CDCl₃): δ 0.83 (d, 6H, ³*J* = 6.7 Hz, 2xMe), 1.67 – 1.70 (m, 1H, 2xCH), 2.72 (t, 2H, CH₂), 5.18 (s, 1H, NH), 7.17 (t, 2H, ³*J* = 8.4 Hz, CH_{Ar}), 7.87 – 7.90 (m, 2H, CH_{Ar}).

¹³C{¹H} NMR (126 MHz, CDCl₃): 19.8, 28.3, 50.5, 116.2 (d, J_{CF} = 22.4 Hz), 129.7 (d, J_{CF} = 9.5 Hz), 136.0, 164.9 (d, ¹ J_{CF} = 256.0 Hz).

HRMS (TOF MS ES+) m/z: [M + H]⁺: Calcd for C₁₀H₁₅NO₂SF 232.0809. Found 232.0808.

N,N-dibenzyl-4-fluorobenzenesulfonamide 4n.



This compound was prepared starting from aryl bromide **2d** (174 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (267 mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.) and amine **1t** (197 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4n** (284 mg, 0.80 mmol, 80%).

Alternatively, the title compound was prepared starting from aromatic carboxylic acid **3c** (140 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (267[°] mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.); CuBr₂ (313 mg, 1.4 mmol, 1.4 equiv.) and amine **1t** (197 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4n** (294 mg, 0.83 mmol, 83%).

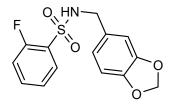
White solid, mp 89 - 90 °C. ¹H NMR (500 MHz, CDCl₃): δ 4.36 (s, 4H, 2xNCH₂), 7.08 – 7.10 (m, 4H, CH_{Ar}), 7.17 (t, 2H, ³J = 8.5 Hz, CH_{Ar}), 7.25 – 7.26 (m, 6H, CH_{Ar}), 7.83 – 7.86 (m, 2H, CH_{Ar}).

¹³C{¹H} NMR (126 MHz, CDCl₃): δ 50.4, 116.1 (d, J_{CF} = 21.1 Hz), 127.7, 128.4 (m), 129.7 (d, J_{CF} = 9.0 Hz), 135.3, 136.8 (m), 164.8 (d, ¹ J_{CF} = 254.4 Hz).

MS (GC, 70eV): m/z (%) = 355 (M⁺, 1), 264 (20), 196 (34), 91 (100).

Anal. calcd. for C₂₀H₁₈NO₂FS: C, 67.59; H, 5.10; N, 3.94. Found: C, 67.63; H, 5.22; N, 4.03.

N-(benzo[d][1,3]dioxol-5-ylmethyl)-2-fluorobenzenesulfonamide 40.



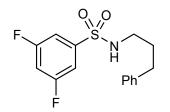
This compound was prepared starting from aryl bromide **2e** (174 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (267 mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.) and amine **1p** (151 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4o** (284 mg, 0.84 mmol, 84%).

Alternatively, the title compound was prepared starting from aromatic carboxylic acid **3d** (140 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (267[°] mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.); CuBr₂ (313 mg, 1.4 mmol, 1.4 equiv.) and amine **1p** (151 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4o** (294 mg, 0.82 mmol, 82%).

White solid, mp 108 - 109 °C. ¹H NMR (500 MHz, CDCl₃): δ 4.07 (d, 2H, ³*J* = 6.3 Hz, NCH₂), 5.17 (br. s, 1H, NH), 5.89 (s, 1H, OCH₂O), 6.21 – 6.66 (m, 3H, CH_{Ar}), 7.13 – 7.19 (m, 1H, CH_{Ar}), 7.24 (dt, 1H, ³*J* = 7.7 Hz, ⁴*J* = 0.8 Hz, CH_{Ar}), 7.52 – 7.56 (m, 1H, CH_{Ar}), 7.84 (dt, 1H, ³*J* = 7.5 Hz, ⁴*J* = 1.7 Hz, CH_{Ar}). ¹³C{¹H} NMR (126 MHz, CDCl₃): δ 47.2, 101.1, 108.2 (d, *J*_{CF} = 33.7 Hz), 116.7 (d, *J*_{CF} = 19.6 Hz,), 121.4, 121.4 (d, *J*_{CF} = 3.7 Hz,), 128.0 (d, *J*_{CF} = 13.5 Hz), 129.6, 134.8 (d, *J*_{CF} = 8.6 Hz), 147.5 (d, *J*_{CF} = 69.5 Hz), 158.6 (d, ¹*J*_{CF} = 253.5 Hz).

HRMS (TOF MS ES-) m/z: [M - H]⁺: Calcd for C₁₄H₁₁NO₄SF 308.0399. Found 308.0393.

3,5-difluoro-N-(3-phenylpropyl)benzenesulfonamide 4p.



This compound was prepared starting from aryl bromide **2f** (192 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (267 mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.) and amine **1l** (135 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4p** (230 mg, 0.74 mmol, 74%).

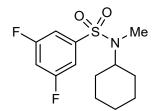
Alternatively, the title compound was prepared starting from aromatic carboxylic acid **3e** (158 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (267 mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50 mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21 mg, 0.03 mmol, 0.03 equiv.); CuBr₂ (313 mg, 1.4 mmol, 1.4 equiv.) and amine **1l** (135 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4p** (289 mg, 0.93 mmol, 93%).

White solid, mp 108 - 109 °C. ¹H NMR (500 MHz, CDCl₃): δ 1.91 (quint, 2H, ³*J* = 7.4 Hz, CH₂), 2.71 (t, 2H, ³*J* = 6.7 Hz, CH₂), 3.08 (q, 2H, ³*J* = 7.4 Hz, CH₂), 5.24 (br. s, 1H, NH), 7.08 – 7.12 (m, 1H, CH_{Ar}), 7.18 (d, 2H, ³*J* = 6.9 Hz, CH_{Ar}), 7.26 – 7.29 (m, 1H, CH_{Ar}), 7.33 – 7.36 (m, 2H, CH_{Ar}), 7.47 – 7.49 (m, 2H, CH_{Ar}).

¹³C{¹H} NMR (126 MHz, CDCl₃): δ 31.0, 32.5, 42.7, 108.2 (d, J_{CF} = 26.2 Hz), 110.4 (dd, J_{CF} = 21.3 Hz, J_{CF} = 7.1 Hz), 126.1, 128.3, 128.5, 140.5, 143.2 (d, J_{CF} = 8.1 Hz), 162.8 (dd, ¹ J_{CF} = 255.7 Hz, J_{CF} = 11.9 Hz).

HRMS (TOF MS ES+) m/z: $[M + H]^+$: Calcd for C₁₅H₁₆NO₂SF₂ 312.0868. Found 312.0870.

N-cyclohexyl-3,5-difluoro-N-methylbenzenesulfonamide 4q.



This compound was prepared starting from aryl bromide **2f** (192 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (267 mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.) and amine **1r** (113 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4q** (234 mg, 0.81 mmol, 81%).

Alternatively, the title compound was prepared starting from aromatic carboxylic acid **3e** (158 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (267 mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50 mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21 mg, 0.03 mmol, 0.03 equiv.); CuBr₂ (313 mg, 1.4 mmol, 1.4 equiv.) and amine **1r** (113 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4q** (257 mg, 0.89 mmol, 89%).

White solid, mp 77 - 78 °C. ¹H NMR (500 MHz, CDCl₃): δ 0.96 – 1.00 (m, 1H, Cycl.), 1.25 – 1.35 (m, 4H, Cycl.), 1.46 – 1.48 (m, 2H, Cycl.), 1.56 – 1.59 (m, 1H, Cycl.), 1.71 – 1.73 (m, 2H, Cycl.), 2.75 (s, 3H, Me), 3.70 – 3.73 (m, 1H, Cycl.), 6.98 (tt, 1H, ³*J* = 8.3 Hz, ⁴*J* = 2.3 Hz, CH_{Ar}), 7.30 – 7.31 (m, 2H, CH_{Ar}).

¹³C{¹H} NMR (126 MHz, CDCl₃): δ 25.1, 25.6, 28.6, 30.2, 57.1, 107.7 (t, J_{CF} = 25.1 Hz), 110.1 (dd, J_{CF} = 20.7 Hz, J_{CF} = 7.6 Hz), 143.7 (m), 162.7 (dd, ${}^{1}J_{CF}$ = 254.7 Hz, J_{CF} = 12.0 Hz).

MS (GC, 70eV): m/z (%) = 289 (M⁺, 22), 246 (100), 177 (77), 113 (30).

Anal. calcd. for C₁₃H₁₇NO₂F₂S: C, 53.97; H, 5.92; N, 4.84. Found: C, 54.08; H, 5.76; N, 4.96.

N-(adamantan-1-yl)-3,4-difluorobenzenesulfonamide 4r.

N

This compound was prepared starting from aryl bromide **2f** (192 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (267 mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.) and amine **1j** (151 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4r** (235 mg, 0.72 mmol, 72%).

Alternatively, the title compound was prepared starting from aromatic carboxylic acid **3e** (158 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (267 mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50 mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21 mg, 0.03 mmol, 0.03 equiv.); CuBr₂ (313 mg, 1.4 mmol, 1.4 equiv.) and amine **1j** (151 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4r** (229 mg, 0.70 mmol, 70%).

Alternatively, the title compound was prepared starting from aryl bromide **2f** (192 mg, 1.0 mmol, 1.0 equiv.), DABSO (264 mg, 1.1 mmol, 1.1 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.) and amine **1j** (151 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4r** (229 mg, 0.70 mmol, 70%).

Alternatively, the title compound was prepared starting from aromatic carboxylic acid **3e** (158 mg, 1.0 mmol, 1.0 equiv.), DABSO (264° mg, 1.1 mmol, 1.1 equiv.), cucurbit[6]uril (50° mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21° mg, 0.03 mmol, 0.03 equiv.); CuBr₂ (313 mg, 1.4 mmol, 1.4 equiv.) and amine **1j** (151 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4r** (219 mg, 0.67 mmol, 67%).

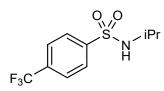
Alternatively, the title compound was prepared starting from aryl bromide **2f** (192 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (1.068 g, 4.8 mmol, 4.8 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.) and nitro compound **5c** (181 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4r** (199 mg, 0.61 mmol, 61%).

Alternatively, the title compound was prepared starting from aromatic carboxylic acid **3e** (158 mg, 1.0 equiv.), $K_2S_2O_5$ (1.068 g, 4.8 mmol, 4.8 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.); CuBr₂ (313 mg, 1.4 mmol, 1.4 equiv.) and nitro compound **5b** (207 mg, (1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4r** (190 mg, 0.58 mmol, 58%).

White solid, mp 144 - 145 °C. ¹**H NMR** (500 MHz, CDCl₃): δ 1.57 (q, 6H, ³*J* = 13.6 Hz, Adamantyl), 1.78 (s, 6H, Adamantyl), 2.01 (s, 3H, Adamantyl), 5.16 (s, 1H, NH), 7.28 (q, 1H, ³*J* = 8.4 Hz, CH_{Ar}), 7.70 – 7.77 (m, 2H, CH_{Ar}).

¹³C{¹H} NMR (126 MHz, CDCl₃): δ 29.4, 35.7, 42.9, 55.5, 116.8 (d, J_{CF} = 18.6 Hz), 118.0 (d, J_{CF} = 17.9 Hz), 123.8 (m), 140.9 (m), 149.8 (dd, $^{1}J_{CF}$ = 254.4 Hz, J_{CF} = 14.0 Hz), 152.6 (dd, $^{1}J_{CF}$ = 255.8 Hz, J_{CF} = 12.6 Hz). MS (GC, 70eV): m/z (%) = 327 (M⁺, 37), 270 (45), 135 (19), 113 (43), 93 (100). Anal. calcd. for C₁₆H₁₉NO₂F₂S: C, 58.70; H, 5.85; N, 4.28. Found: C, 58.72; H, 5.93; N, 4.11.

N-isopropyl-4-(trifluoromethyl)benzenesulfonamide 4s.



This compound was prepared starting from aryl bromide **2h** (224 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (267 mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.) and amine **1g** (59 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4s** (238 mg, 0.89 mmol, 89%).

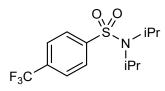
Alternatively, the title compound was prepared starting from aromatic carboxylic acid **3g** (190 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (267 mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50 mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21 mg, 0.03 mmol, 0.03 equiv.); CuBr₂ (313 mg, 1.4 mmol, 1.4 equiv.) and amine **1g** (59 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4s** (214 mg, 0.80 mmol, 80%).

White solid, mp 77 - 78 °C. ¹H NMR (500 MHz, CDCl₃): δ 1.05 (d, 6H, ³*J* = 6.8 Hz, 2xMe), 3.45 – 3.47 (m, 1H, CH.), 5.03 (s, 1H, NH), 7.73 (d, 2H, ³*J* = 8.2 Hz, CH_{Ar}), 8.0 (d, 2H, ³*J* = 8.2 Hz, CH_{Ar}).

¹³C{¹H} NMR (126 MHz, CDCl₃): δ 23.6, 46.4, 123.3 (q, ¹*J*_{*CF*} = 278.8 Hz), 126.2, 127.4, 134.1 (q, ²*J*_{*CF*} = 33.1 Hz), 144.8.

HRMS (TOF MS ES+) m/z: $[M + H]^+$: Calcd for $C_{10}H_{13}NO_2SF_3$ 268.0612. Found 268.0619.

N,N-diisopropyl-4-(trifluoromethyl)benzenesulfonamide 4t.



This compound was prepared starting from aryl bromide **2h** (224 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (267 mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.) and amine **1q** (101 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4t** (222 mg, 0.72 mmol, 72%).

Alternatively, the title compound was prepared starting from aromatic carboxylic acid **3g** (190 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (267[°] mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.); CuBr₂ (313 mg, 1.4 mmol, 1.4 equiv.) and amine **1q** (101 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4t** (185 mg, 0.60 mmol, 60%).

White solid, mp 100 - 101 °C. ¹H NMR (500 MHz, CDCl₃): δ 1.25 (d, 12H, ³*J* = 6.9 Hz, 4xMe), 3.72 – 3.75 (m, 2H, 2xCH.), 7.73 (d, 2H, ³*J* = 8.3 Hz, CH_{Ar}), 7.98 (d, 2H, ³*J* = 8.0 Hz, CH_{Ar}).

¹³C{¹H} NMR (126 MHz, CDCl₃): δ 21.9, 49.0, 123.3 (q, ¹*J*_{CF} = 274.2 Hz), 126.0 (m), 127.6, 133.5 (q, ²*J*_{CF} = 32.7 Hz), 146.2.

HRMS (TOF MS ES+Na) m/z: [M + Na]⁺: Calcd for C₁₃H₁₈NO₂SNaF₃ 332.0930. Found 332.0908.

N-benzyl-4-(trifluoromethyl)benzenesulfonamide 4u.

O,↓ S,N H

This compound was prepared starting from aryl bromide **2h** (224 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (267 mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.) and amine **1k** (107 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4u** (249 mg, 0.79 mmol, 79%).

Alternatively, the title compound was prepared starting from aromatic carboxylic acid **3g** (190 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (267[°] mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.); CuBr₂ (313 mg, 1.4 mmol, 1.4 equiv.) and amine **1k** (107 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4u** (277 mg, 0.88 mmol, 88%).

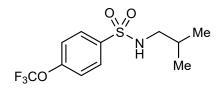
Alternatively, the title compound was prepared starting from aryl bromide **2h** (224 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (1.068 g, 4.8 mmol, 4.8 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.) and nitro compound **5d** (137 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4u** (202 mg, 0.64 mmol, 64%).

Alternatively, the title compound was prepared starting from aromatic carboxylic acid **3g** (190 mg, 1.0 equiv.), $K_2S_2O_5$ (1.068 g, 4.8 mmol, 4.8 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.); CuBr₂ (313 mg, 1.4 mmol, 1.4 equiv.) and nitro compound **5d** (137 mg, (1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4u** (208 mg, 0.66 mmol, 66%).

White solid, mp 121 - 122 °C. ¹H NMR (500 MHz, CDCl₃): δ 4.16 (d, 2H, ³*J* = 6.2 Hz, CH₂), 5.35 (s, 1H, NH), 7.14 – 7.16 (m, 2H, CH_{Ar}), 7.22 – 7.26 (m, 3H, CH_{Ar}), 7.70 (d, 2H, ³*J* = 8.2 Hz, CH_{Ar}), 7.92 (d, 2H, ³*J* = 8.2 Hz, CH_{Ar}).

¹³C{¹H} NMR (126 MHz, CDCl₃): δ 47.2, 123.1 (q, ¹*J*_{CF} = 268.2 Hz), 126.1 (m), 127.5, 127.8, 128.0, 128.7, 134.2 (q, ²*J*_{CF} = 35.0 Hz), 135.7. HRMS (TOF MS ES-) m/z: [M - H]⁺: Calcd for C₁₄H₁₁NO₂SF₃ 314.0467. Found 314.0463.

N-isobutyl-4-(trifluoromethoxy)benzenesulfonamide 4v.



This compound was prepared starting from aryl bromide **2i** (240 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (267 mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.) and amine **1h** (73 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4v** (211 mg, 0.71 mmol, 71%).

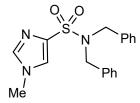
Alternatively, the title compound was prepared starting from aromatic carboxylic acid **3h** (206 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (267 mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50 mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21 mg, 0.03 mmol, 0.03 equiv.); CuBr₂ (313 mg, 1.4 mmol, 1.4 equiv.) and amine **1h** (73 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4v** (175 mg, 0.59 mmol, 59%).

White solid, mp 120 - 122 °C. ¹**H NMR** (500 MHz, CDCl₃): δ 0.86 (d, 6H, ³*J* = 6.8 Hz, 2xMe), 1.69 – 1.74 (m, 1H, CH), 2.76 (t, 2H, ³*J* = 6.4 Hz, CH₂), 5.12 (s, 1H, NH), 7.32 (d, 1H, ³*J* = 8.8 Hz, CH_{Ar}), 7.92 (d, 1H, ³*J* = 8.8 Hz, CH_{Ar}).

¹³C{¹H} NMR (126 MHz, CDCl₃): δ 19.8, 28.4, 50.5, 120.2 (q, ¹*J*_{CF} = 259.2 Hz), 121.0, 129.1, 138.4, 152.0.

HRMS (TOF MS ES+) m/z: [M + H]⁺: Calcd for C₁₁H₁₅NO₃SF₃ 298.0725. Found 298.0725.

N,*N*-dibenzyl-1-methyl-1H-imidazole-4-sulfonamide 4w.

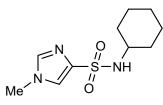


This compound was prepared starting from aryl bromide **2l** (160 mg, 1.0 mmol, 1.0 equiv.), K₂S₂O₅ (267 mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50[°]µmg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°]µmg, 0.03 mmol, 0.03 equiv.) and amine **1t** (197 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4w** (252 mg, 0.74 mmol, 74%). Alternatively, the title compound was prepared starting from aromatic carboxylic acid **3k** (126 mg, 1.0 mmol, 1.0 equiv.), K₂S₂O₅ (267[°]µmg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50[°]µmg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°]µmg, 0.03 mmol, 0.03 equiv.); CuBr₂ (313 mg, 1.4 mmol, 1.4 equiv.) and amine **1t** (197 mg, 1.0 mmol, 1.0 equiv.). The purification was prepared by column chromatography on silica gel to provide the provide the desired compound **3k** (126 mg, 1.0 mmol, 1.0 equiv.), K₂S₂O₅ (267[°]µmg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50[°]µmg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°]µmg, 0.03 mmol, 0.03 equiv.); CuBr₂ (313 mg, 1.4 mmol, 1.4 equiv.) and amine **1t** (197 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide

the desired compound 4w (280 mg, 0.82 mmol, 82%).

White solid, mp 120 - 122 °C. ¹H NMR (500 MHz, CDCl₃): δ 3.71 (s, 3H, Me), 4.30 (s, 4H, 2xCH₂), 7.13 – 7.15 (m, 4H, CH_{Ar}), 7.19 – 7.23 (m, 6H, CH_{Ar}), 7.86 (s, 1H, Imidazole), 7.86 (s, 1H, Imidazole). ¹³C{¹H} NMR (126 MHz, CDCl₃): δ 33.5, 51.4, 125.2, 127.2, 128.1, 128.2, 136.5, 138.5, 139.9. MS (GC, 70eV): m/z (%) = 341 (M⁺, 9), 145 (33), 95 (100). Anal. calcd. for C₁₈H₁₉N₃O₂S: C, 63.32; H, 5.61; N, 12.31. Found: C, 63.33; H, 5.693; N, 12.21.

N-cyclohexyl-1-methyl-1H-imidazole-4-sulfonamide 4x.



This compound was prepared starting from aryl bromide **2I** (160 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (267 mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.) and amine **1i** (99 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4x** (170 mg, 0.70mmol, 70%).

Alternatively, the title compound was prepared starting from aromatic carboxylic acid **3k** (126 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (267 mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50 mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21 mg, 0.03 mmol, 0.03 equiv.); CuBr₂ (313 mg, 1.4 mmol, 1.4 equiv.) and amine **1i** (99 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4x** (209 mg, 0.86 mmol, 86%).

Alternatively, the title compound was prepared starting from aryl bromide **2l** (160 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (1.068 g, 4.8 mmol, 4.8 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.) and nitro compound **5e** (99 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4x** (148 mg, 0.55 mmol, 55%).

Alternatively, the title compound was prepared starting from aromatic carboxylic acid **3g** (190 mg, 1.0 equiv.), $K_2S_2O_5$ (1.068 g, 4.8 mmol, 4.8 equiv.), cucurbit[6]uril (50 mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21 mg, 0.03 mmol, 0.03 equiv.); CuBr₂ (313 mg, 1.4 mmol, 1.4 equiv.) and

nitro compound **5e** (99 mg, (1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4x** (139 mg, 0.57 mmol, 57%).

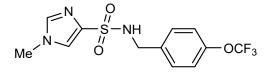
White solid, mp 177-178 °C. ¹H NMR (500 MHz, DMSO-*d*₆): δ 1.00 – 1.17 (m, 5H, Cycl.), 1.43 – 1.45 (m, 1H, Cycl.), 1.58 – 1.66 (m, 4H, Cycl.), 2.96 – 2.97 (m, 1H, Cycl.), 3.68 (s, 3H, Me), 7.36 (d, 1H, ³*J* = 7.3 Hz, NH), 7.65 (d, 1H, ⁴*J* = 1.1 Hz, Imidazole), 7.73 (d, 1H, ⁴*J* = 0.7 Hz, Imidazole).

¹³C{¹H} NMR (126 MHz, DMSO-*d*₆): δ 24.6, 25.0, 33.40, 33.44, 52.1, 123.6, 139.4, 141.0.

MS (GC, 70eV): m/z (%) = 243(M⁺, 1), 200 (31), 145 (65), 136 (21), 123 (21), 98 (100), 82 (74).

Anal. calcd. for C₁₀H₁₇N₃O₂S: C, 49.36; H, 7.04; N, 17.27. Found: C, 49.45; H, 7.11; N, 17.21.

1-methyl-N-(4-(trifluoromethoxy)benzyl)-1H-imidazole-4-sulfonamide 4y.



This compound was prepared starting from aryl bromide **2I** (160 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (267 mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.) and amine **1n** (191 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4y** (228 mg, 0.68 mmol, 68%). Alternatively, the title compound was prepared starting from aromatic carboxylic acid **3k** (126 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (267[°] mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.); CuBr₂ (313 mg, 1.4 mmol, 1.4 mmol, 1.4 mmol, 1.2 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.); CuBr₂ (313 mg, 1.4 mmol, 1.4 mmol, 1.4 mmol, 1.2 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.); CuBr₂ (313 mg, 1.4 mmol, 1.4 mmol, 1.4 mmol, 1.2 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.); CuBr₂ (313 mg, 1.4 mmol, 1.4 mmol,

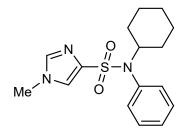
equiv.) and amine **1n** (191 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4y** (261 mg, 0.78 mmol, 78%).

White solid, mp 170 - 171 °C. ¹H NMR (500 MHz, DMSO-*d*₆): δ 3.60 (s, 3H, Me), 4.07 (s, 2H, CH₂), 7.27 (d, 2H, ³*J* = 8.0 Hz, CH_{Ar}), 7.39 (d, 2H, ³*J* = 8.7 Hz, CH_{Ar}), 7.71 (d, 1H, ⁴*J* = 1.1 Hz, Imidazole), 7.62 (d, 1H, ⁴*J* = 1.0 Hz, Imidazole), 8.00 (br. s, 1H, NH).

¹³C{¹H} NMR (126 MHz, DMSO- d_6): δ 33.4, 45.3, 120.1 (q, ¹ J_{CF} = 259.9 Hz), 120.8, 124.2, 129.4, 137.9, 139.6, 139.7, 147.3.

HRMS (TOF MS ES+) m/z: [M + H]⁺: Calcd for C₁₂H₁₃N₃O₃SF₃ 336..0631. Found 336.0630.

N-cyclohexyl-1-methyl-N-phenyl-1H-imidazole-4-sulfonamide 4z.



This compound was prepared starting from aryl bromide **2I** (160 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (267 mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.) and amine **1s** (175 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4z** (230 mg, 0.72 mmol, 72%).

Alternatively, the title compound was prepared starting from aromatic carboxylic acid **3k** (126 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (267[°] mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.); CuBr₂ (313 mg, 1.4 mmol, 1.4 equiv.) and amine **1s** (175 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4z** (191 mg, 0.60 mmol, 60%).

White solid, mp 194 - 195 °C. ¹H NMR (500 MHz, CDCl₃): δ 0.78 – 0.83 (m, 1H, Cy), 1.00 – 1.04 (m, 2H, Cy), 1.29 – 1.31 (m, 2H, Cy), 1.48 (br. s, 1H, Cy), 1.67 (br. s, 2H, Cy), 1.98 (br. s, 2H, Cy), 3.61 (s, 3H, Me), 4.08 – 4.13 (m, 1H, Cy), 7.13 – 7.28 (m, 6H, CH_{Ar}), 7.44 – 7.45 (m, 1H, CH_{Ar}). ¹³C{¹H} NMR (126 MHz, CDCl₃): δ 25.0, 25.8, 32.7, 33.8, 59.1, 124.3, 128.2, 128.5, 132.3, 135.8, 138.8, 141.0.

HRMS (TOF MS ES+) m/z: $[M + H]^+$: Calcd for C₁₅H₂₂N₅OS 320.1576. Found 320.1545.

N,N-dibenzyl-4-(trifluoromethyl)benzenesulfonamide 4aa.

This compound was prepared starting from $CF_3Ph-Pd(PPh_3)_2$ -Br (428 mg, 0.5 mmol, 1.0 equiv.), $K_2S_2O_5$ (134 mg, 0.6 mmol, 1.2 equiv.), cucurbit[6]uril (25 mg, 0.025 mmol, 0.05 equiv.) and amine **1t** (99 mg, 0.5 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **4aa** (122 mg, 0.31 mmol, 61%).

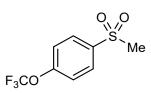
White solid, mp 91 - 92 °C. ¹H NMR (500 MHz, CDCl₃): δ 4.40 (s, 4H, 2xCH₂), 7.07 – 7.09 (m, 4H, CH_{Ar}), 7.25 – 7.26 (m, 4H, CH_{Ar}), 7.74 (d, 2H, ³*J* = 7.3 Hz, CH_{Ar}), 7.92 (d, 2H, ³*J* = 7.3 Hz, CH_{Ar}).

¹³C{¹H} NMR (126 MHz, CDCl₃): δ 50.6, 123.2 (q, ¹*J*_{CF} = 274.9 Hz), 126.2 (m), 127.5, 127.8, 128.4, 128.5, 134.0 (q, ²*J*_{CF} = 32.9 Hz), 135.0.

MS (GC, 70eV): m/z (%) = 405 (M⁺, 2), 314 (12), 195 (19), 91 (100).

Anal. calcd. for C₂₁H₁₈F₃NO₂S: C, 62.21; H, 4.48; N, 3.45. Found: C, 62.18; H, 4.53; N, 3.52.

1-(methylsulfonyl)-4-(trifluoromethoxy)benzene 11.



This compound was prepared starting from 1-bromo-4-(trifluoromethoxy)benzene **10** (241 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (267 mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.) and MeI (426 mg, 3.0 mmol, 3.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **11** (139 mg, 0.58 mmol, 58%).

White solid, mp 67-69 °C. ¹H NMR (500 MHz, CDCl₃): δ 3.06 (s, 3H, Me), 7.37(d, 2H, ³J = 8.7 Hz, CH_{Ar}), 7.98 (dt, 2H, ³J = 8.9 Hz, ⁴J = 2.1 Hz, CH_{Ar}). ¹³C{¹H} NMR (126 MHz, CDCl₃): δ 44.4, 120.1 (q, ¹J_{CF} = 257.9 Hz), 121.1, 129.6 (m), 129.7, 138.7, 152.9. Anal. calcd. for C₈H₇F₃O₃S: C, 40.00; H, 2.94. Found: C, 40.12; H, 3.06.

sulfonyldibenzene 13.

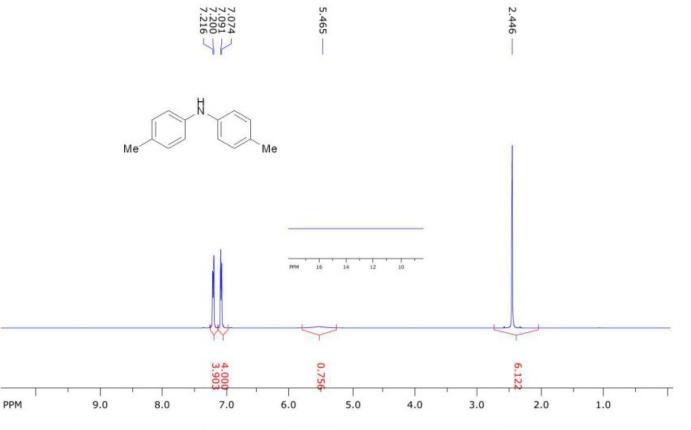
This compound was prepared starting from bromobenzene **12** (157 mg, 1.0 mmol, 1.0 equiv.), $K_2S_2O_5$ (267 mg, 1.2 mmol, 1.2 equiv.), cucurbit[6]uril (50[°] mg, 0.05 mmol, 0.05 equiv.), (PPh₃)₂PdCl₂ (21[°] mg, 0.03 mmol, 0.03 equiv.) and boronic acid (122 mg, 1.0 mmol, 1.0 equiv.). The purification was performed by column chromatography on silica gel to provide the desired compound **13** (153 mg, 0.70 mmol, 70%).

White solid, mp 125-126 °C. ¹H NMR (500 MHz, CDCl₃): δ 7.45 (t, 4H, ³*J* = 8.4 Hz, CH_{Ar}), 7.50 – 7.52 (m, 2H, CH_{Ar}), 7.92 (d, 4H, ³*J* = 7.6 Hz, CH_{Ar}). ¹³C{¹H} NMR (126 MHz, CDCl₃): δ 124.4, 129.1, 133.1, 141.3.

Anal. calcd. for C₁₂H₁₀O₂S: C, 66.03; H, 4.62. Found: C, 66.13; H, 4.69.

(C) Copies ¹H and ¹³C NMR spectra.

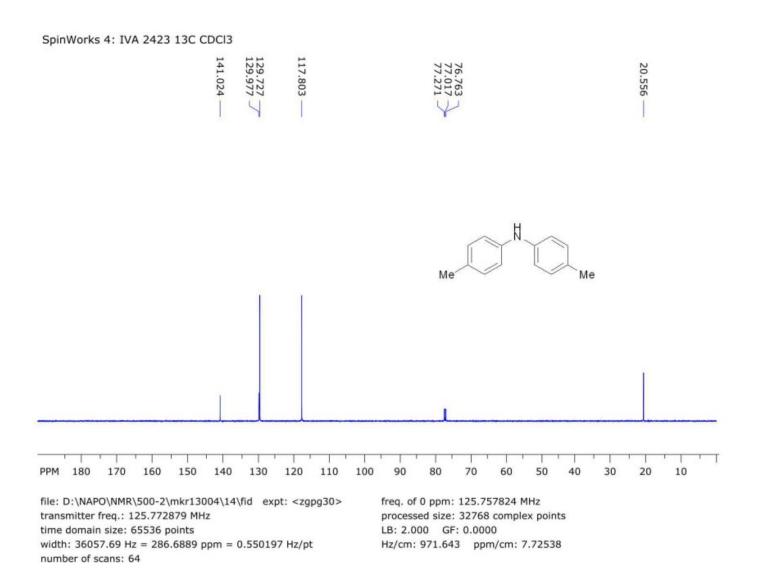
Compound 1f



file: D:\NAPO\NMR\500-2\mkr13004\13\fid expt: <zg30> transmitter freq.: 500.133001 MHz time domain size: 65536 points width: 12335.53 Hz = 24.6645 ppm = 0.188225 Hz/pt number of scans: 24

freq. of 0 ppm: 500.130022 MHz processed size: 65536 complex points LB: 0.300 GF: 0.0000 Hz/cm: 213.416 ppm/cm: 0.42672

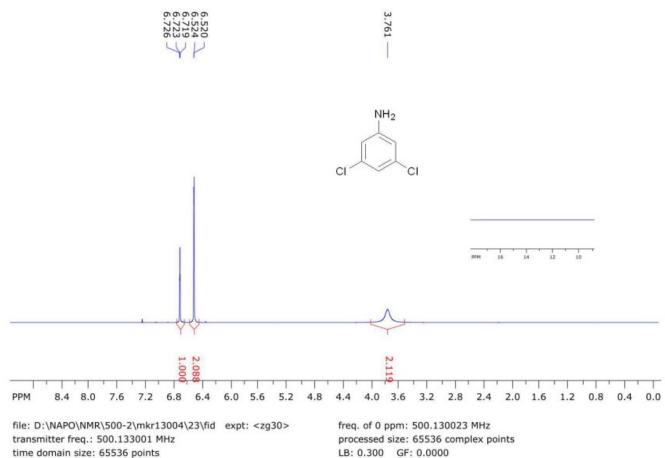
Compound 1f



Compound 1u

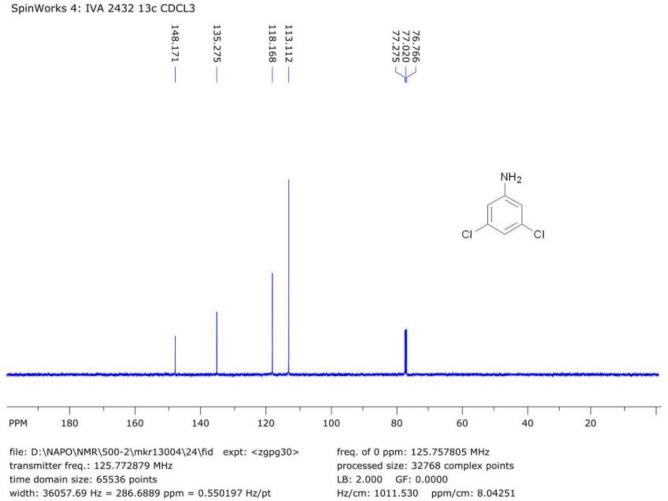
width: 12335.53 Hz = 24.6645 ppm = 0.188225 Hz/pt

number of scans: 24



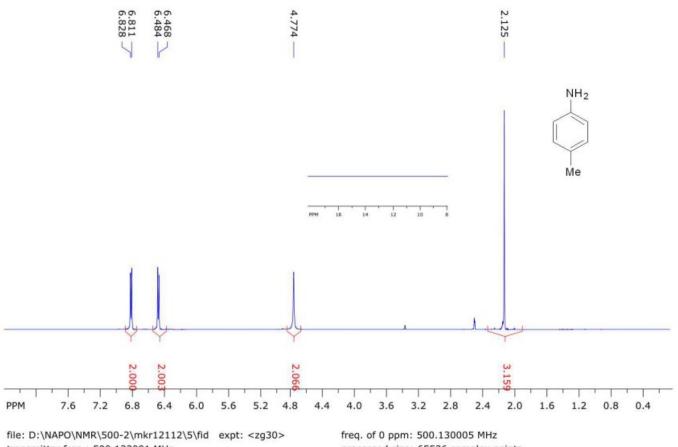
Hz/cm: 185.033 ppm/cm: 0.36997

Compound 1u



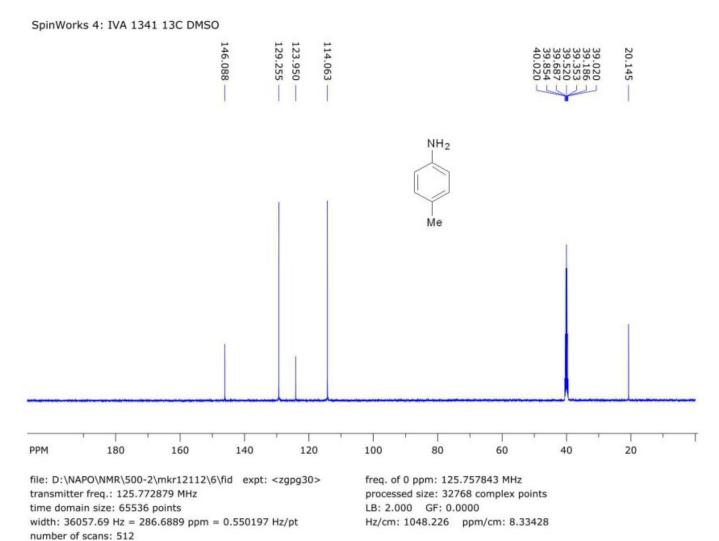
number of scans: 64

Compound 1v



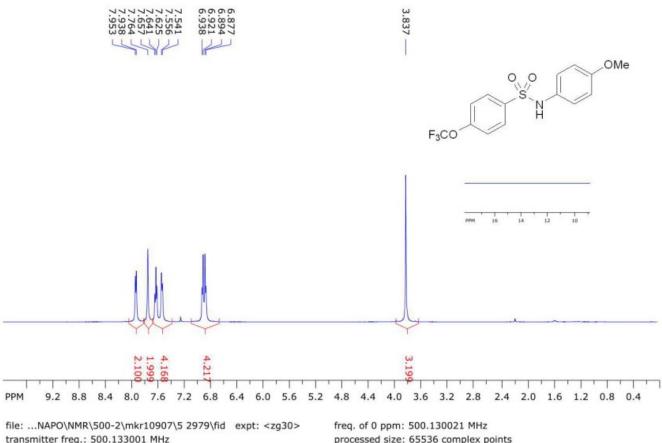
transmitter freq.: 500.133001 MHz time domain size: 65536 points width: 12335.53 Hz = 24.6645 ppm = 0.188225 Hz/pt number of scans: 24 freq. of 0 ppm: 500.130005 MHz processed size: 65536 complex points LB: 0.300 GF: 0.0000 Hz/cm: 167.567 ppm/cm: 0.33504

Compound 1v



Compound 4a

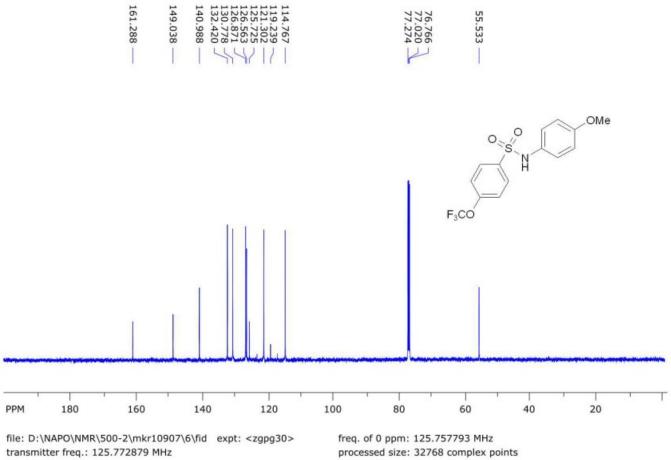
SpinWorks 4: IVA 2979 1H CDCl3



file: ...NAPO\NMR\500-2\mkr10907\5 2979\fid expt: <zg3 transmitter freq.: 500.133001 MHz time domain size: 65536 points width: 12335.53 Hz = 24.6645 ppm = 0.188225 Hz/pt number of scans: 24 freq. of 0 ppm: 500.130021 MHz processed size: 65536 complex points LB: 0.300 GF: 0.0000 Hz/cm: 199.224 ppm/cm: 0.39834

Compound 4a

SpinWorks 4: IVA 2979 13C CDCl3



time domain size: 65536 points width: 36057.69 Hz = 286.6889 ppm = 0.550197 Hz/pt number of scans: 512

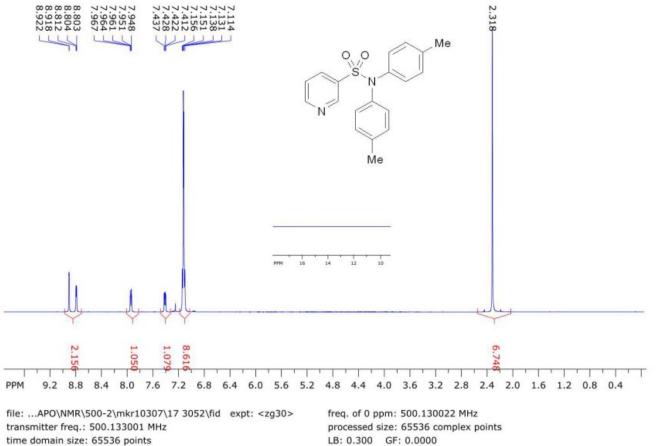
LB: 2.000 GF: 0.0000 Hz/cm: 1016.316 ppm/cm: 8.08057

Compound 4b

SpinWorks 4: IVA 3052 1H CDCl3

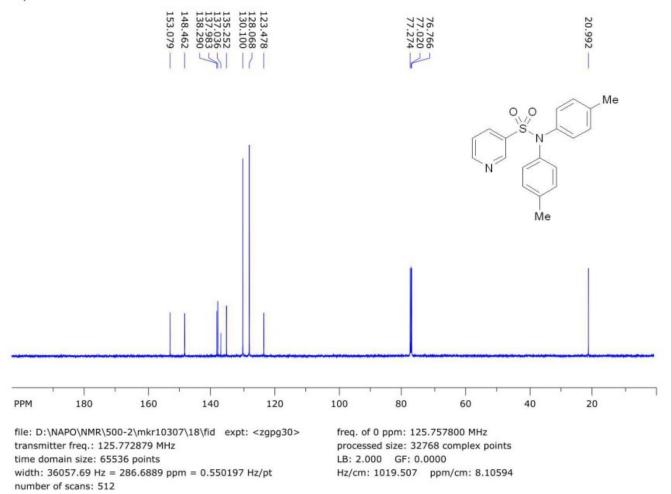
width: 12335.53 Hz = 24.6645 ppm = 0.188225 Hz/pt

number of scans: 24



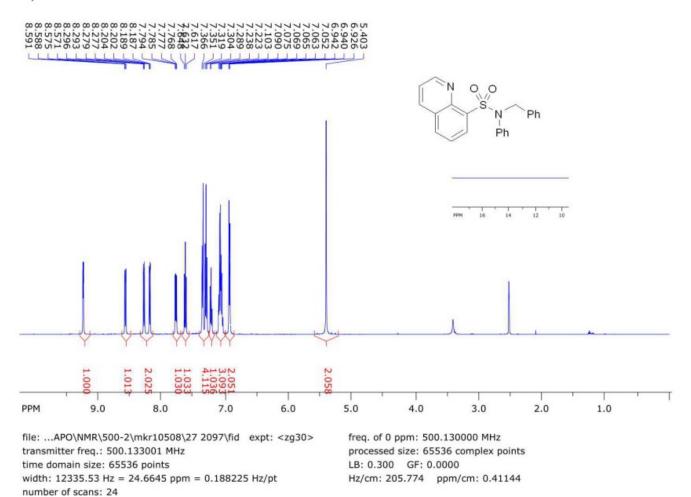
Hz/cm: 199.770 ppm/cm: 0.39943

Compound 4b



Compound 4c

SpinWorks 4: IVA 2097 1H DMSO

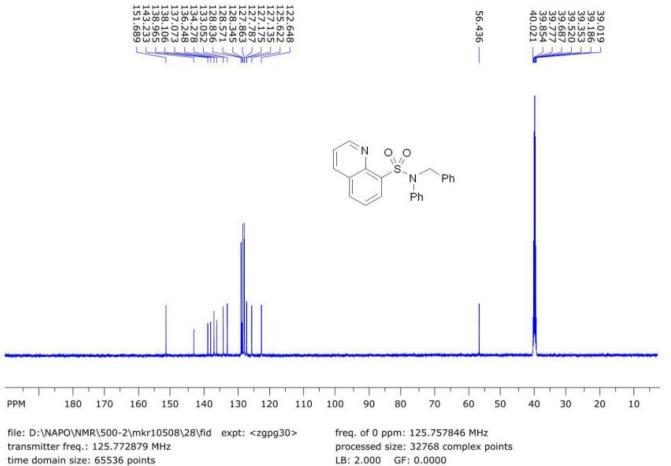


Compound 4c

SpinWorks 4: IVA 2097 13C DMSO

width: 36057.69 Hz = 286.6889 ppm = 0.550197 Hz/pt

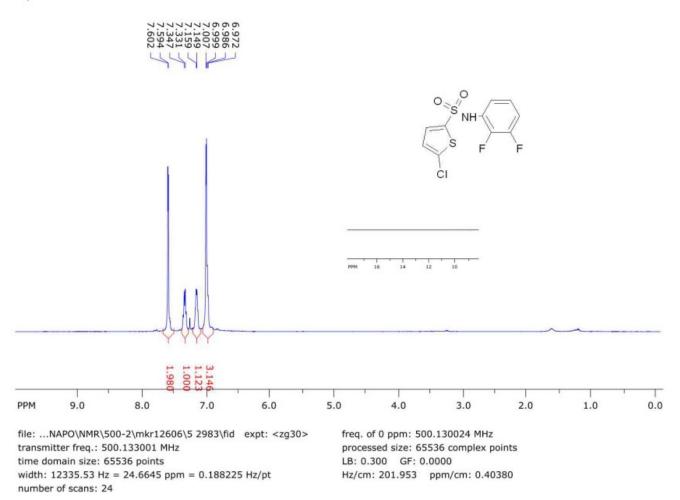
number of scans: 512



Hz/cm: 998.766 ppm/cm: 7.94103

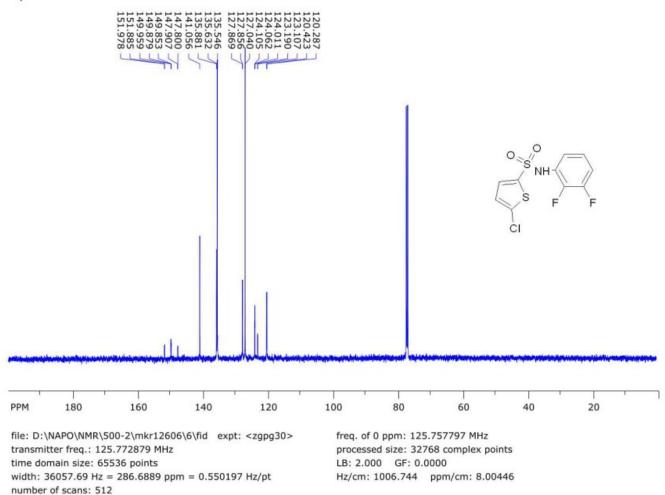
Compound 4d

SpinWorks 4: IVA 2983 1H CDCl3



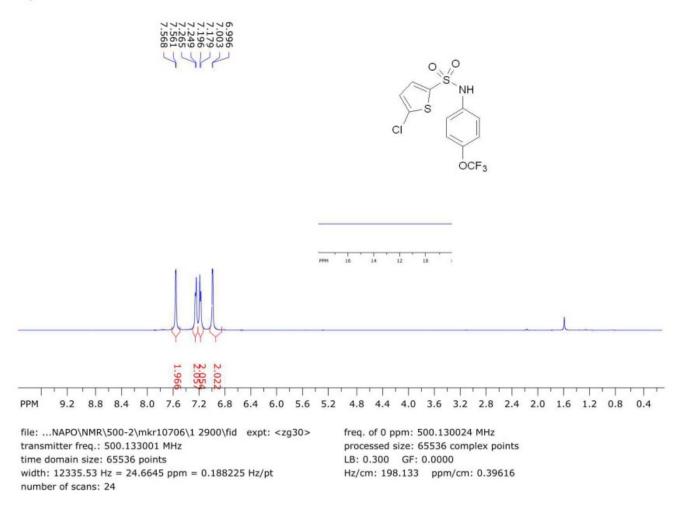
Compound 4d

SpinWorks 4: IVA 2983 13C CDCL3



Compound 4e

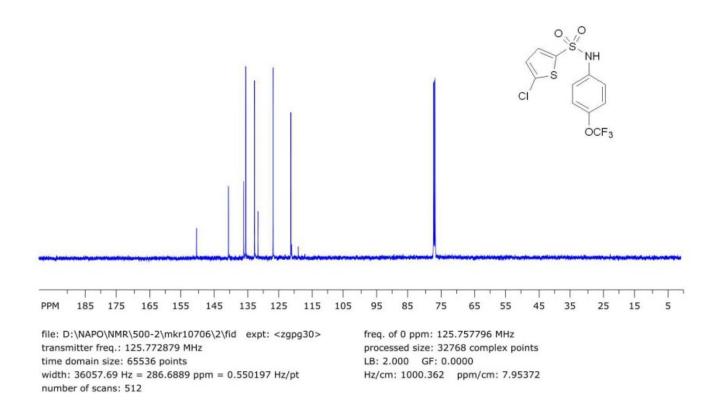
SpinWorks 4: IVA 2900 1H



Compound 4e

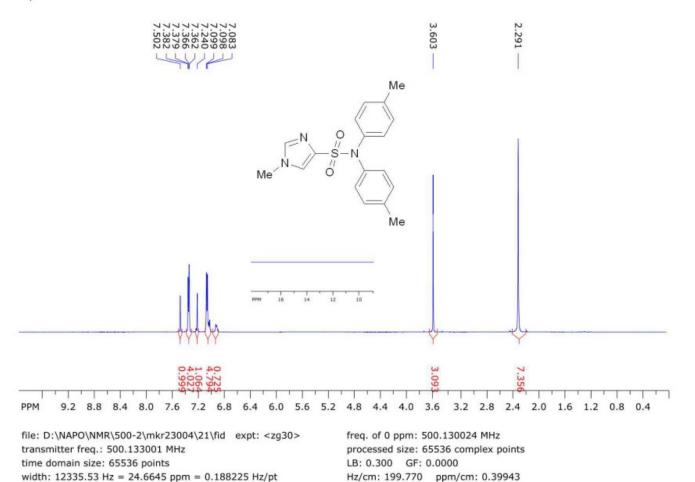
SpinWorks 4: IVA 2900 13C

150.689	127.01 131.67 132.73 135.44 136.07 140.81	119.18 121.24 121.49
	1441	66



Compound 4f

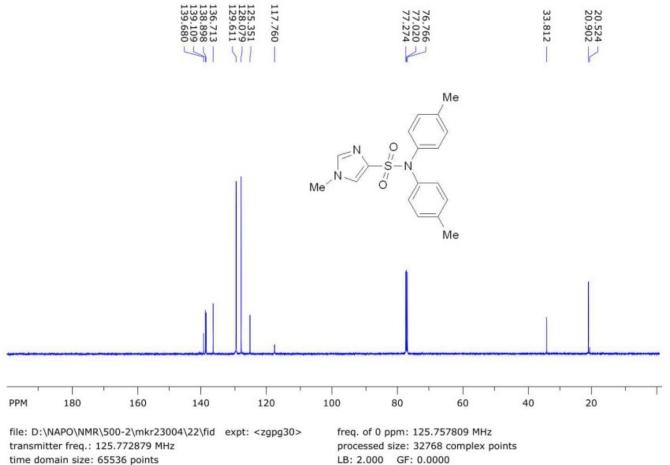
number of scans: 24



Compound 4f

width: 36057.69 Hz = 286.6889 ppm = 0.550197 Hz/pt

number of scans: 512

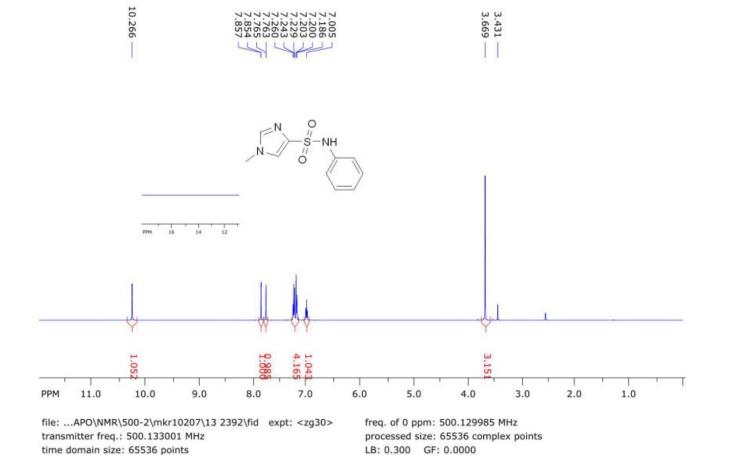


Hz/cm: 1013.125 ppm/cm: 8.05520

Compound 4g

width: 12335.53 Hz = 24.6645 ppm = 0.188225 Hz/pt

number of scans: 24



Hz/cm: 240.707 ppm/cm: 0.48128

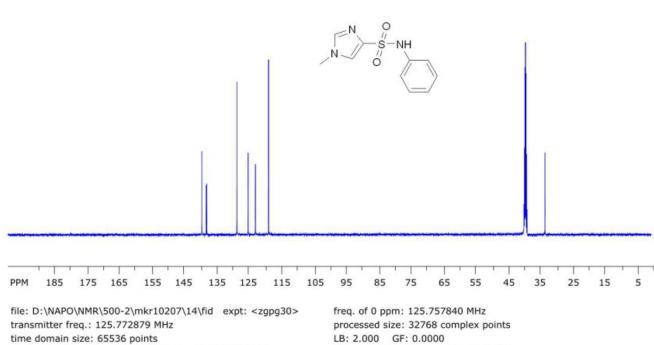
Compound 4g

SpinWorks 4: IVA 2392 13C

width: 36057.69 Hz = 286.6889 ppm = 0.550197 Hz/pt

number of scans: 512

138.236 138.478 139.790	119.107 123.180 125.440 128.928	33,495 39,183 39,520 39,520 39,520 39,520 39,687 39,854 40,021
4		

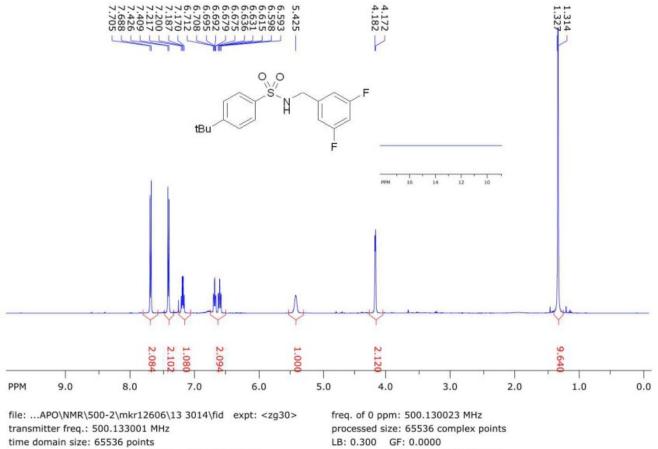


Hz/cm: 1001.957 ppm/cm: 7.96640

Compound 4h

width: 12335.53 Hz = 24.6645 ppm = 0.188225 Hz/pt

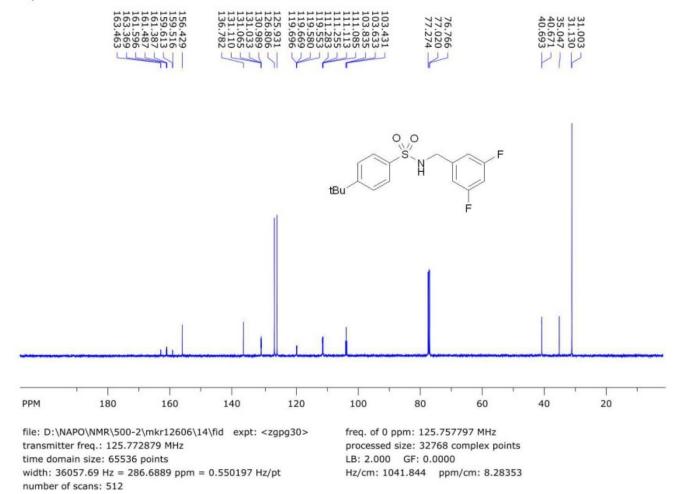
number of scans: 24



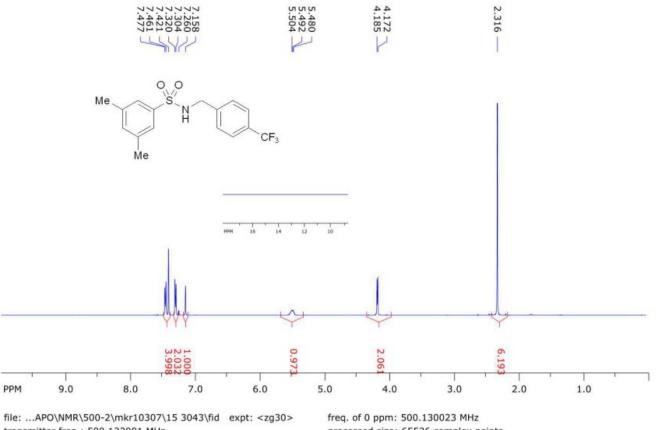
Hz/cm: 201.512 ppm/cm: 0.40292

Compound 4h

SpinWorks 4: IVA 3014 13C CDCl3



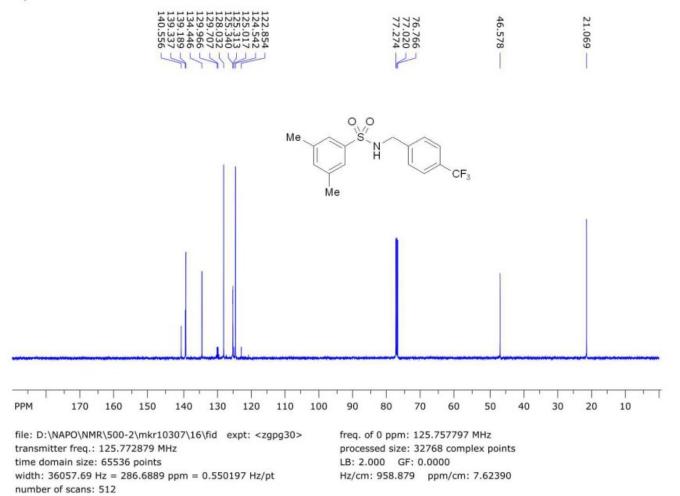
Compound 4i



transmitter freq.: 500.133001 MHz time domain size: 65536 points width: 12335.53 Hz = 24.6645 ppm = 0.188225 Hz/pt number of scans: 24

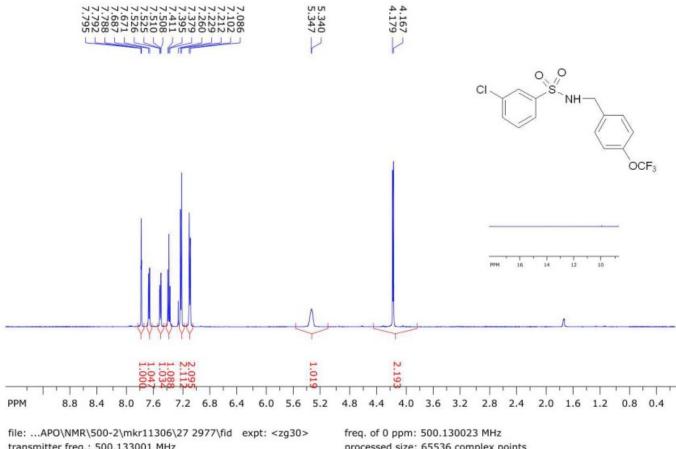
freq. of 0 ppm: 500.130023 MHz processed size: 65536 complex points LB: 0.300 GF: 0.0000 Hz/cm: 200.316 ppm/cm: 0.40053

Compound 4i



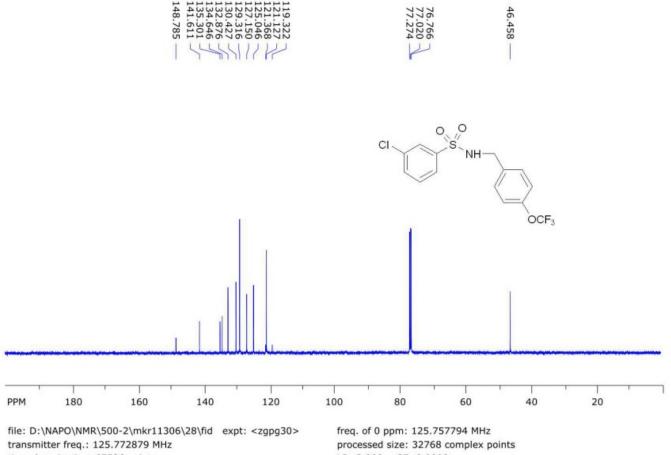
Compound 4j

SpinWorks 4: IVA 2977 1H CDCl3



transmitter freq.: 500-2(mkr11306/2/297/(ld expt: <2g30 transmitter freq.: 500.133001 MHz time domain size: 65536 points width: 12335.53 Hz = 24.6645 ppm = 0.188225 Hz/pt number of scans: 24 freq. of 0 ppm: 500.130023 MHz processed size: 65536 complex points LB: 0.300 GF: 0.0000 Hz/cm: 192.674 ppm/cm: 0.38525

Compound 4j

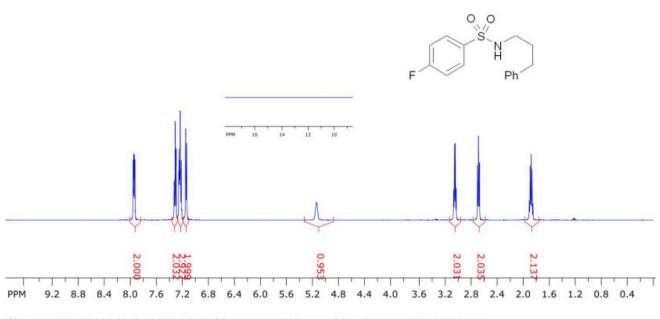


time domain size: 65536 points width: 36057.69 Hz = 286.6889 ppm = 0.550197 Hz/pt number of scans: 512

LB: 2.000 GF: 0.0000 Hz/cm: 1009.934 ppm/cm: 8.02983

Compound 4k



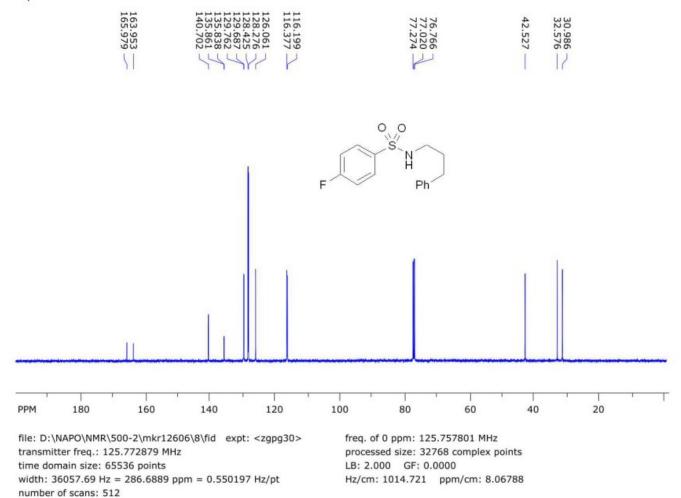


file: ...NAPO\NMR\500-2\mkr12606\7 2993\fid expt: <zg30> transmitter freq.: 500.133001 MHz time domain size: 65536 points width: 12335.53 Hz = 24.6645 ppm = 0.188225 Hz/pt number of scans: 24

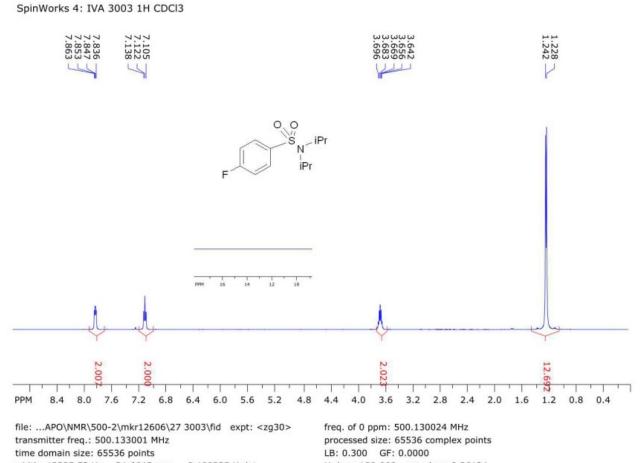
freq. of 0 ppm: 500.129992 MHz processed size: 65536 complex points LB: 0.300 GF: 0.0000 Hz/cm: 198.678 ppm/cm: 0.39725

Compound 4k

SpinWorks 4: IVA 2993 13C CDCl3



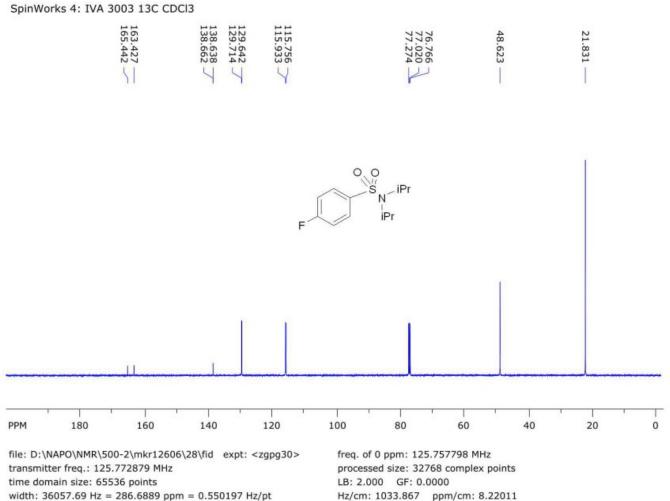
Compound 4I



width: 12335.53 Hz = 24.6645 ppm = 0.188225 Hz/pt number of scans: 24

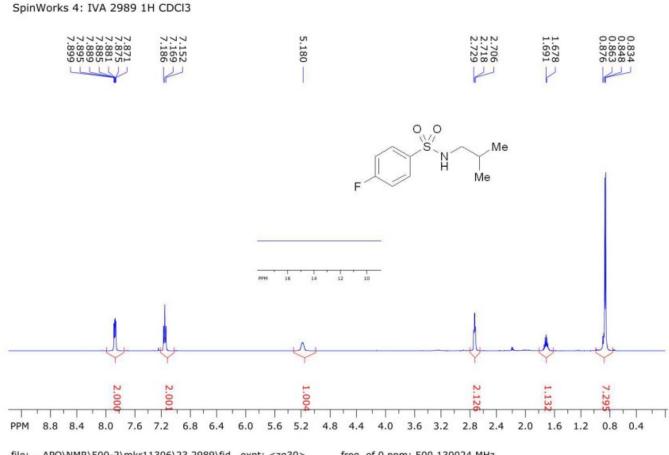
Hz/cm: 180.666 ppm/cm: 0.36124

Compound 4I



number of scans: 512

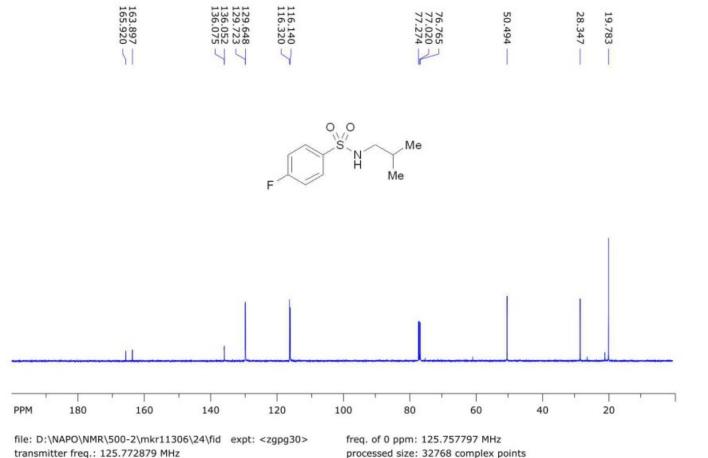
Compound 4m



file: ...APO\NMR\500-2\mkr11306\23 2989\fid expt: <zg30> transmitter freq.: 500.133001 MHz time domain size: 65536 points width: 12335.53 Hz = 24.6645 ppm = 0.188225 Hz/pt number of scans: 24 freq. of 0 ppm: 500.130024 MHz processed size: 65536 complex points LB: 0.300 GF: 0.0000 Hz/cm: 188.854 ppm/cm: 0.37761

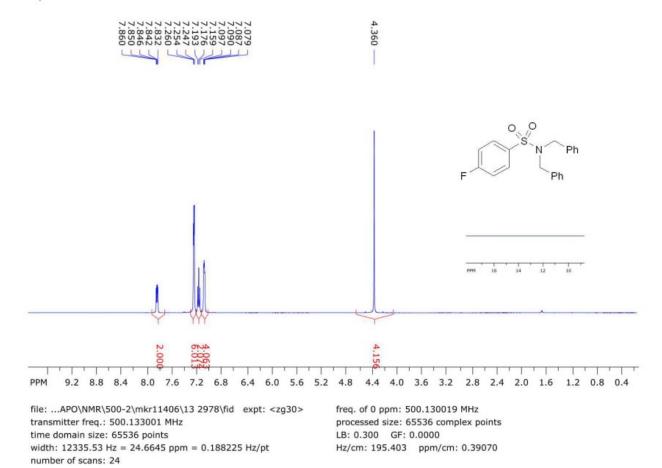
Compound 4m





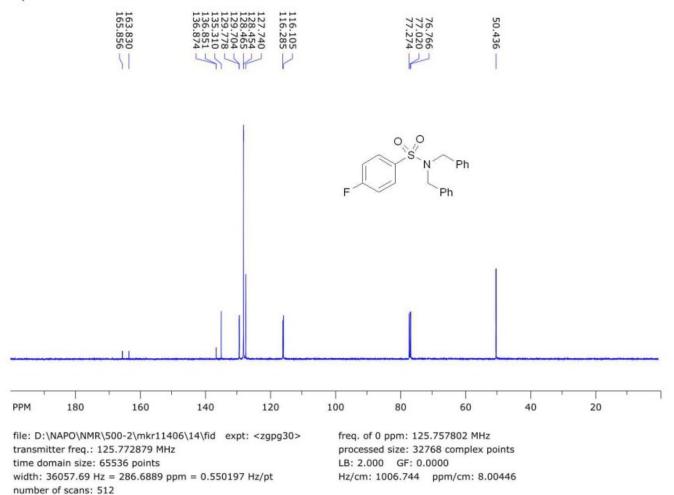
transmitter freq.: 125.772879 MHz time domain size: 65536 points width: 36057.69 Hz = 286.6889 ppm = 0.550197 Hz/pt number of scans: 512 rreq. of 0 ppm: 125.757797 MHz processed size: 32768 complex points LB: 2.000 GF: 0.0000 Hz/cm: 1006.744 ppm/cm: 8.00446

Compound 4n



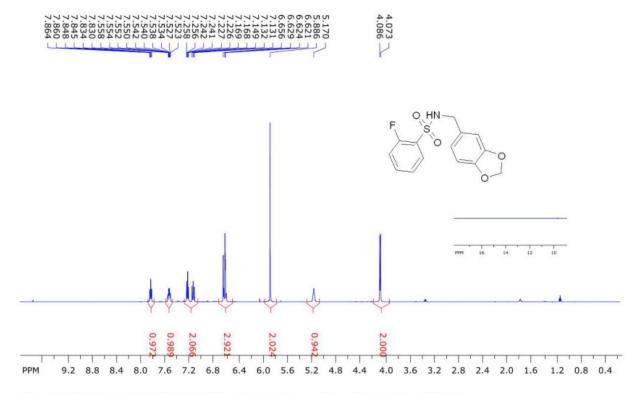
Compound 4n

SpinWorks 4: IVA 2978 13C CDCl3

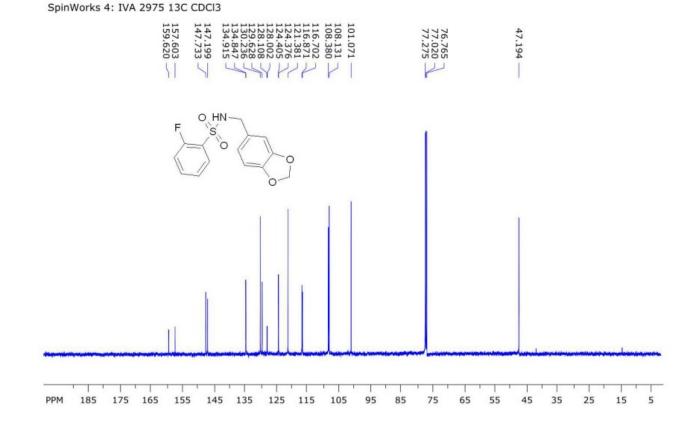


Compound 4o

SpinWorks 4: IVA 2975 1H CDL3

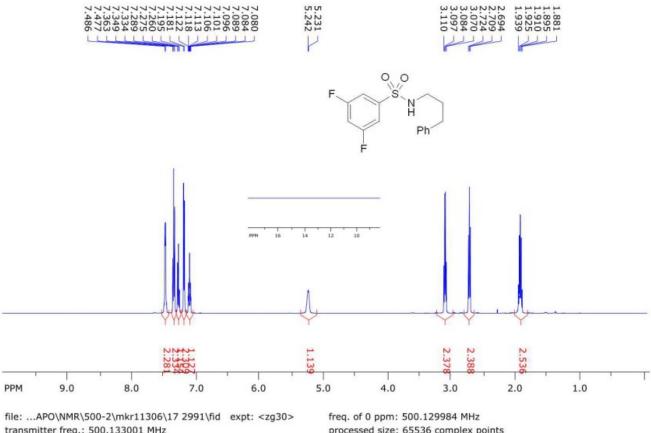


file: ...NAPO\NMR\500-2\mkr12606\9 2975\fid expt: <zg30> transmitter freq.: 500.133001 MHz time domain size: 65536 points width: 12335.53 Hz = 24.6645 ppm = 0.188225 Hz/pt number of scans: 24 freq. of 0 ppm: 500.130024 MHz processed size: 65536 complex points LB: 0.300 GF: 0.0000 Hz/cm: 197.041 ppm/cm: 0.39398 **Compound 4o**



file: D:\NAPO\NMR\500-2\mkr12606\10\fid expt: <zgpg30> transmitter freq.: 125.772879 MHz time domain size: 65536 points width: 36057.69 Hz = 286.6889 ppm = 0.550197 Hz/pt number of scans: 512 freq. of 0 ppm: 125.757800 MHz processed size: 32768 complex points LB: 2.000 GF: 0.0000 Hz/cm: 997.171 ppm/cm: 7.92834

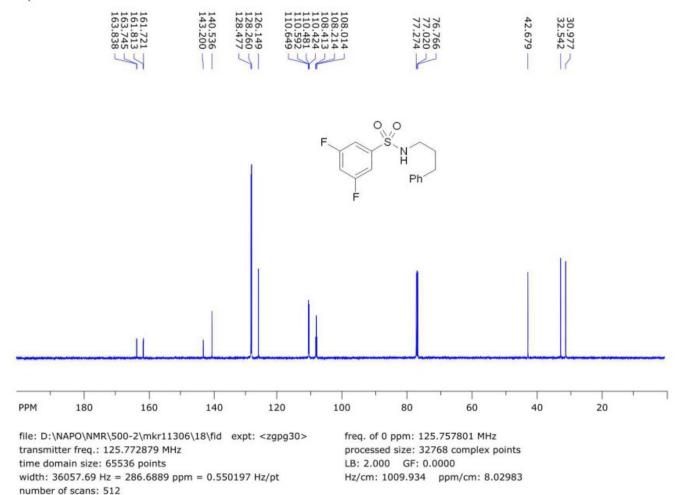
Compound 4p



transmitter freq.: 500.133001 MHz time domain size: 65536 points width: 12335.53 Hz = 24.6645 ppm = 0.188225 Hz/pt number of scans: 24 freq. of 0 ppm: 500.129984 MHz processed size: 65536 complex points LB: 0.300 GF: 0.0000 Hz/cm: 202.499 ppm/cm: 0.40489

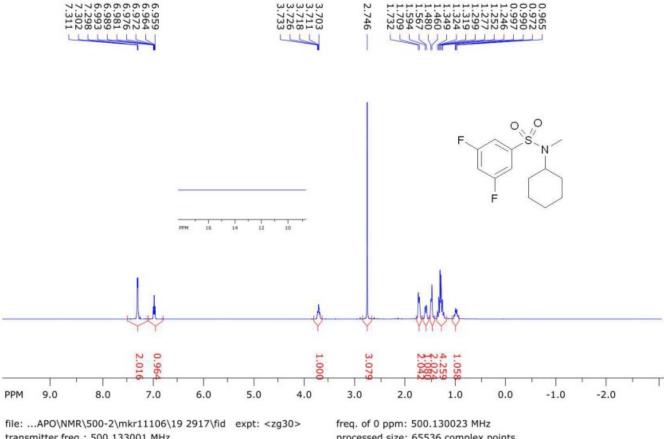
Compound 4p

SpinWorks 4: IVA 2087 13C CDCl3



Compound 4q

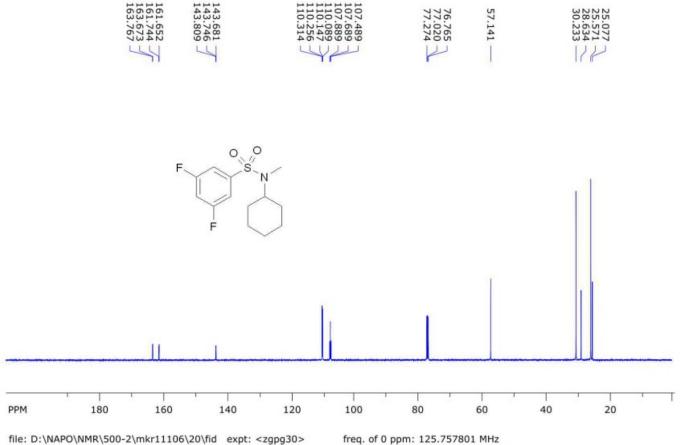
SpinWorks 4: IVA 2917 1H



transmitter freq.: 500-133001 MHz time domain size: 65536 points width: 12335.53 Hz = 24.6645 ppm = 0.188225 Hz/pt number of scans: 24 freq. of 0 ppm: 500.130023 MHz processed size: 65536 complex points LB: 0.300 GF: 0.0000 Hz/cm: 261.993 ppm/cm: 0.52385

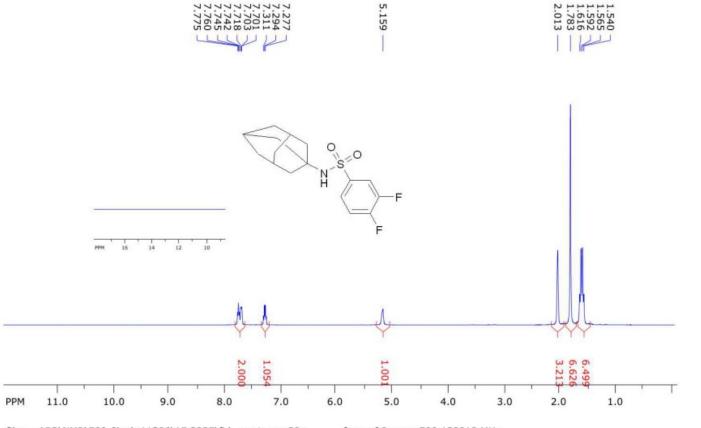
Compound 4q

SpinWorks 4: IVA 2917 13C



file: D:\NAPO\NMR\500-2\mkr11106\20\fid expt: <zgpg305 transmitter freq.: 125.772879 MHz time domain size: 65536 points width: 36057.69 Hz = 286.6889 ppm = 0.550197 Hz/pt number of scans: 512 freq. of 0 ppm: 125.757801 MHz processed size: 32768 complex points LB: 2.000 GF: 0.0000 Hz/cm: 1056.203 ppm/cm: 8.39770

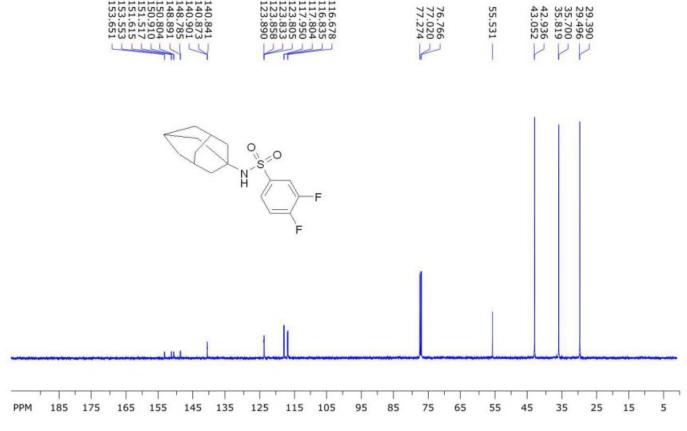
Compound 4r



file: ...APO\NMR\500-2\mkr11306\15 2985\fid expt: <zg30> transmitter freq.: 500.133001 MHz time domain size: 65536 points width: 12335.53 Hz = 24.6645 ppm = 0.188225 Hz/pt number of scans: 24 freq. of 0 ppm: 500.130018 MHz processed size: 65536 complex points LB: 0.300 GF: 0.0000 Hz/cm: 241.798 ppm/cm: 0.48347

Compound 4r

SpinWorks 4: IVA 2985 13C CDCl3

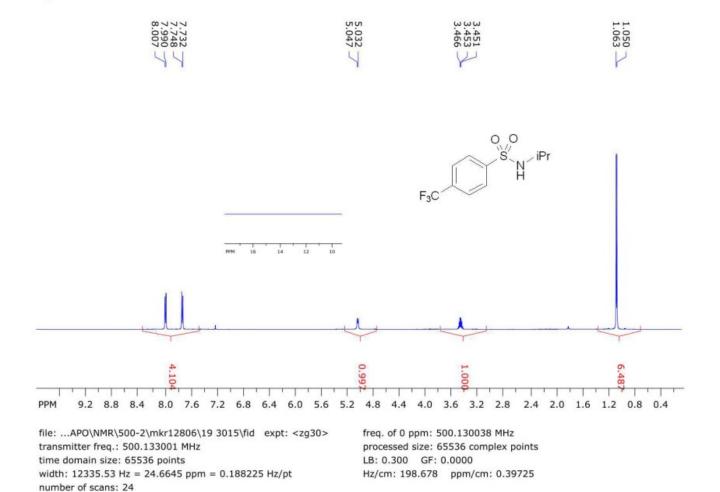


file: D:\NAPO\NMR\500-2\mkr11306\16\fid expt: <zgpg30> transmitter freq.: 125.772879 MHz time domain size: 65536 points width: 36057.69 Hz = 286.6889 ppm = 0.550197 Hz/pt number of scans: 512

freq. of 0 ppm: 125.757796 MHz processed size: 32768 complex points LB: 2.000 GF: 0.0000 Hz/cm: 1001.957 ppm/cm: 7.96640

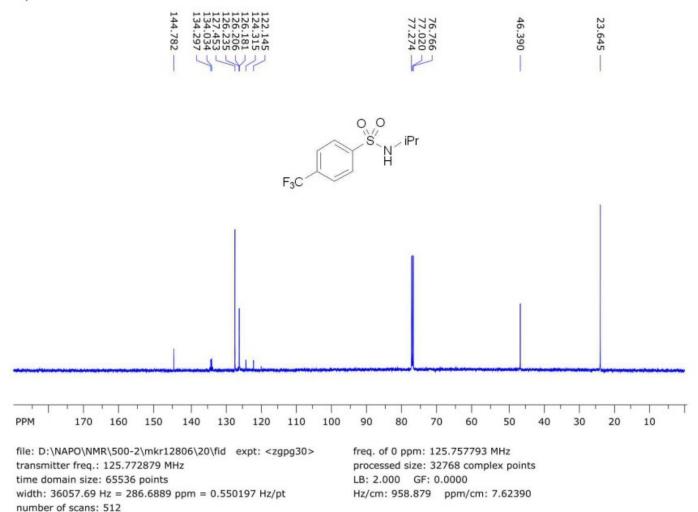
Compound 4s



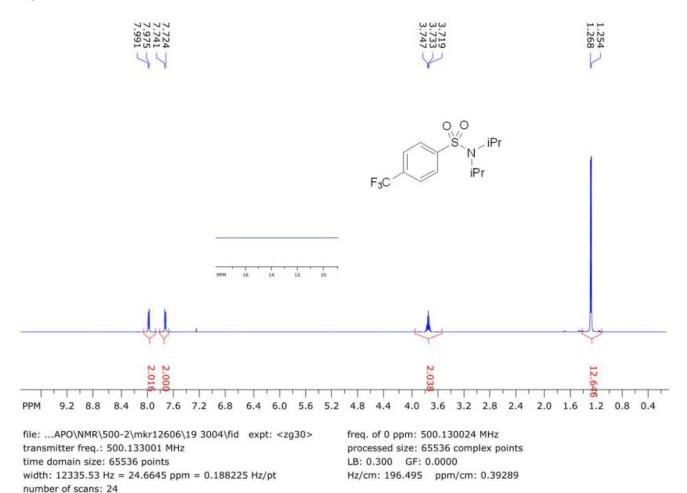


Compound 4s

SpinWorks 4: IVA 3015 13C CDCl3

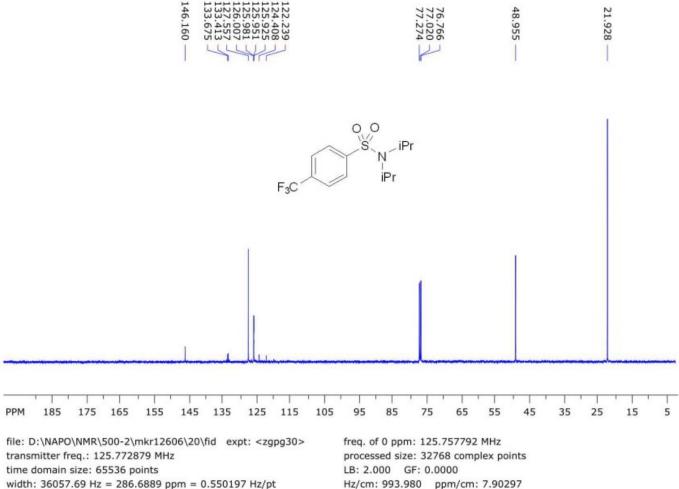


Compound 4t



Compound 4t

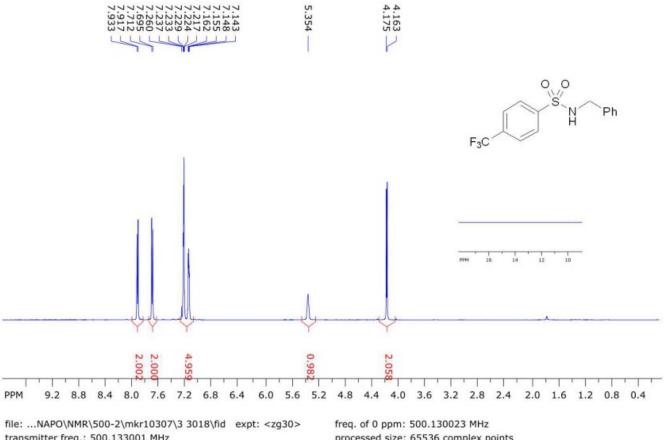
SpinWorks 4: IVA 3004 13C CDCl3



number of scans: 512

Hz/cm: 993.980 ppm/cm: 7.90297

Compound 4u

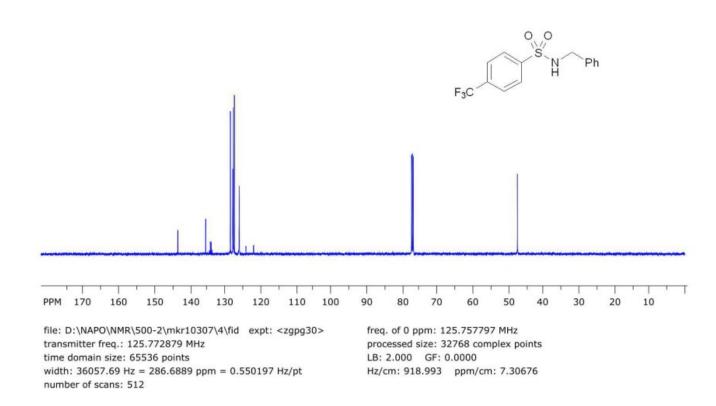


transmitter freq.: 500.133001 MHztime domain size: 65536 pointswidth: 12335.53 Hz = 24.6645 ppm = 0.188225 Hz/ptnumber of scans: 24 freq. of 0 ppm: 500.130023 MHz processed size: 65536 complex points LB: 0.300 GF: 0.0000 Hz/cm: 198.133 ppm/cm: 0.39616

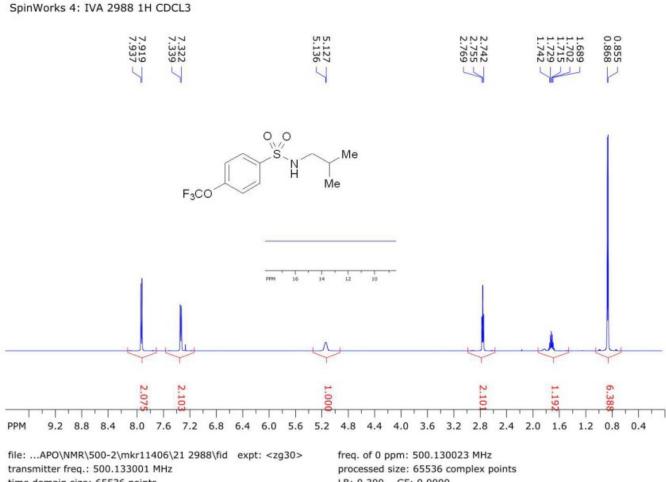
Compound 4u

SpinWorks 4: IVA 3018 13C CDCl3





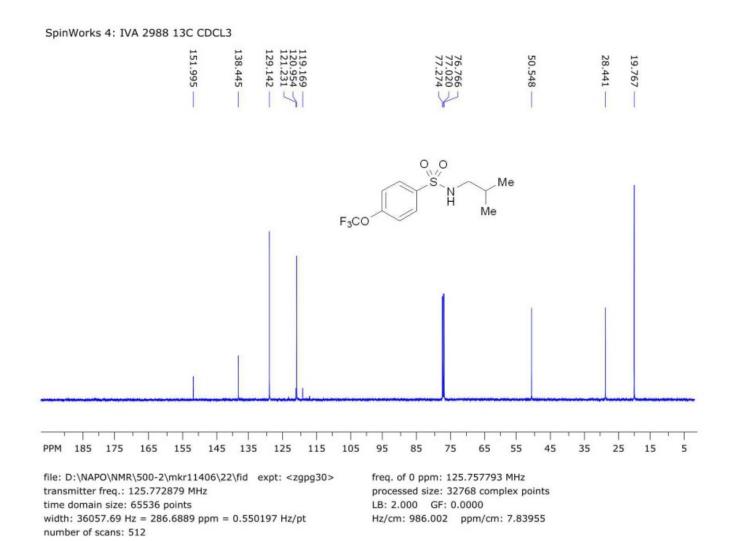
Compound 4v



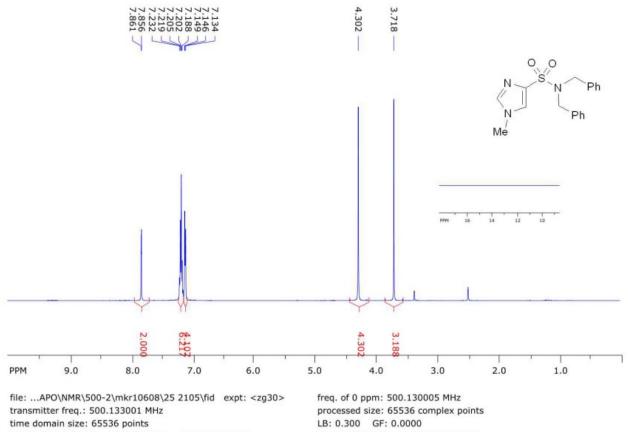
time domain size: 65536 points width: 12335.53 Hz = 24.6645 ppm = 0.188225 Hz/pt number of scans: 24

LB: 0.300 GF: 0.0000 Hz/cm: 199.224 ppm/cm: 0.39834

Compound 4v



Compound 4w

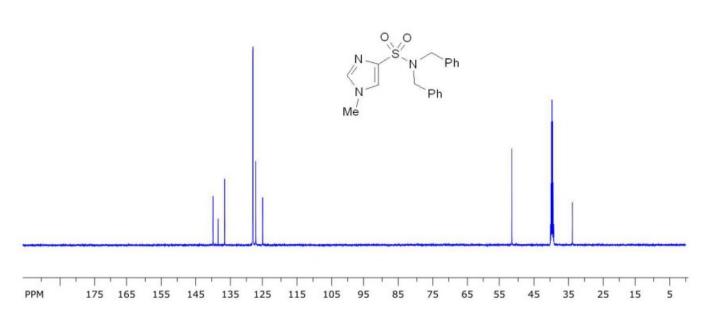


width: 12335.53 Hz = 24.6645 ppm = 0.188225 Hz/pt number of scans: 24

Hz/cm: 200.862 ppm/cm: 0.40162

Compound 4w

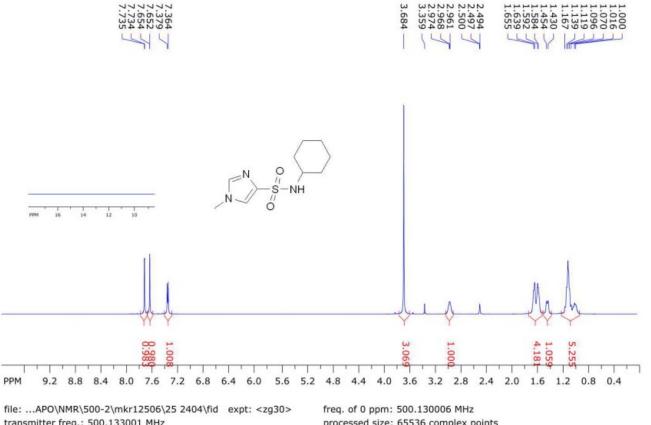
125.211 127.243 127.243 127.243 128.101 128.101 138.490 138.491 139.936	33.512 39.1820 39.520 39.520 39.687 39.687 51.437 51.437
41-41	



file: D:\NAPO\NMR\500-2\mkr10608\26\fid expt: <zgpg30> transmitter freq.: 125.772879 MHz time domain size: 65536 points width: 36057.69 Hz = 286.6889 ppm = 0.550197 Hz/pt number of scans: 512 freq. of 0 ppm: 125.757846 MHz processed size: 32768 complex points LB: 2.000 GF: 0.0000 Hz/cm: 987.598 ppm/cm: 7.85223

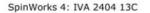
Compound 4x

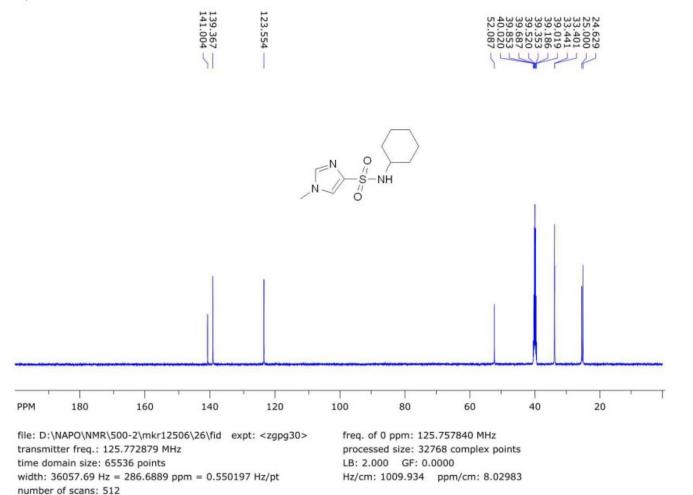
SpinWorks 4: IVA 2404 1H



transmitter freq.: 500.133001 MHztime domain size: 65536 points width: 12335.53 Hz = 24.6645 ppm = 0.188225 Hz/ptnumber of scans: 24 freq. of 0 ppm: 500.130006 MHz processed size: 65536 complex points LB: 0.300 GF: 0.0000 Hz/cm: 198.678 ppm/cm: 0.39725

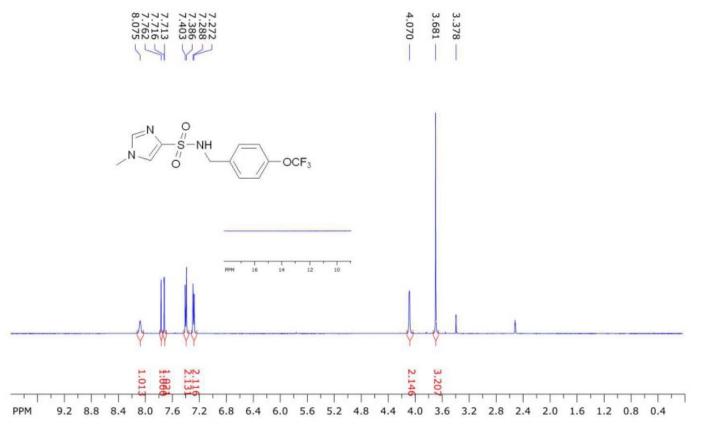
Compound 4x





Compound 4y

SpinWorks 4: IVA 2399 1H



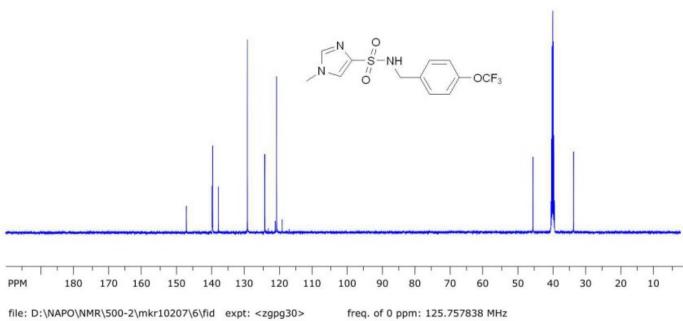
file: ...NAPO\NMR\500-2\mkr10207\5 2399\fid expt: <zg30> transmitter freq.: 500.133001 MHz time domain size: 65536 points width: 12335.53 Hz = 24.6645 ppm = 0.188225 Hz/pt number of scans: 24

freq. of 0 ppm: 500.130005 MHz processed size: 65536 complex points LB: 0.300 GF: 0.0000 Hz/cm: 199.770 ppm/cm: 0.39943

Compound 4y

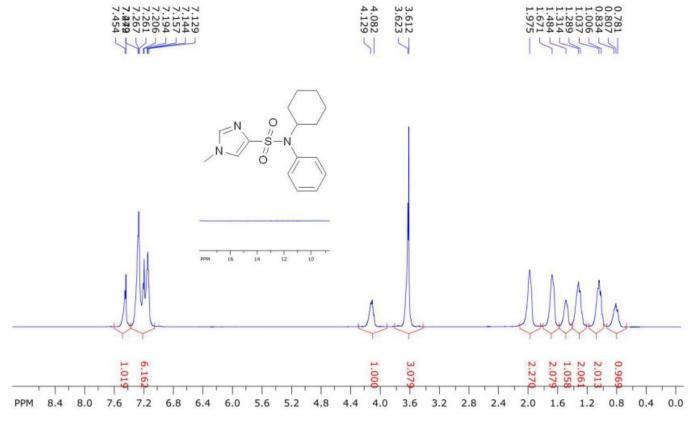
SpinWorks 4: IVA 2399 13C

137.916 139.592 139.704 147.330	119.125 120.764 121.161 124.242 129.372	33.408 39.1819 39.35339.3533 39.3533 39.3533 39.854 45.346 87 45.467 87 45.346 87 45.3467 87 45.3467 87 45.3467 87 45.3467 87 45.3467 87 45.3467 87 45.3467 87 45.3467 87 45.3467 87 45.3467 87 45.3467 87 45.3467 87 45.3467 47 45.3467 47 45.3467 47 45.3467 47 45.3467 47 47 47 47 47 47 47 47 47 47 47 47 47
4	1441	



transmitter freq.: 125.772879 MHz time domain size: 65536 points width: 36057.69 Hz = 286.6889 ppm = 0.550197 Hz/pt number of scans: 512 freq. of 0 ppm: 125.757838 MHz processed size: 32768 complex points LB: 2.000 GF: 0.0000 Hz/cm: 998.766 ppm/cm: 7.94103

Compound 4z



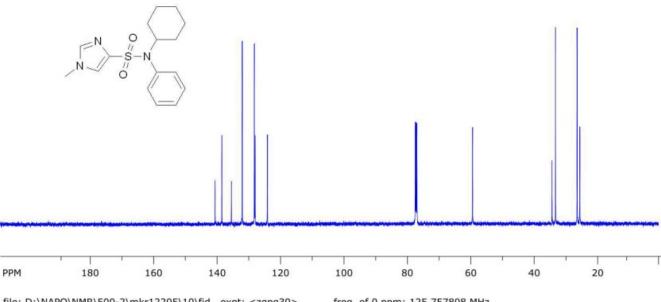
file: ...\NMR\500-2\mkr12205\9 iva 2391\fid expt: <zg30> transmitter freq.: 500.133001 MHz time domain size: 65536 points width: 12335.53 Hz = 24.6645 ppm = 0.188225 Hz/pt number of scans: 24

freq. of 0 ppm: 500.130024 MHz processed size: 65536 complex points LB: 0.300 GF: 0.0000 Hz/cm: 181.212 ppm/cm: 0.36233

Compound 4z

SpinWorks 4: IVA 2391 13C



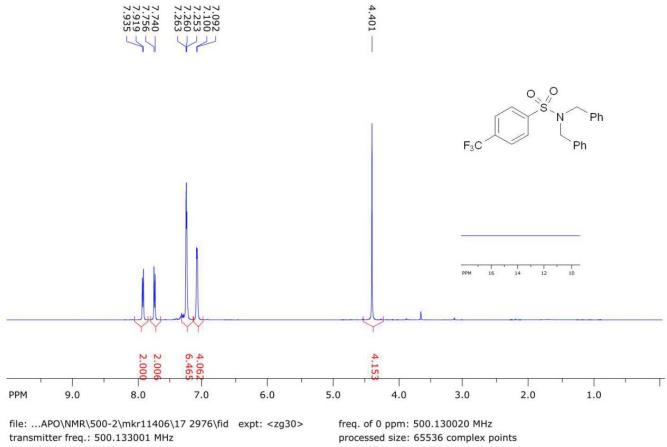


file: D:\NAPO\NMR\500-2\mkr12205\10\fid expt: <zgpg30> transmitter freq.: 125.772879 MHz time domain size: 65536 points width: 36057.69 Hz = 286.6889 ppm = 0.550197 Hz/pt number of scans: 512

freq. of 0 ppm: 125.757808 MHz processed size: 32768 complex points LB: 2.000 GF: 0.0000 Hz/cm: 1053.012 ppm/cm: 8.37233

Compound 4aa

SpinWorks 4: IVA 2976 1H CDCl3

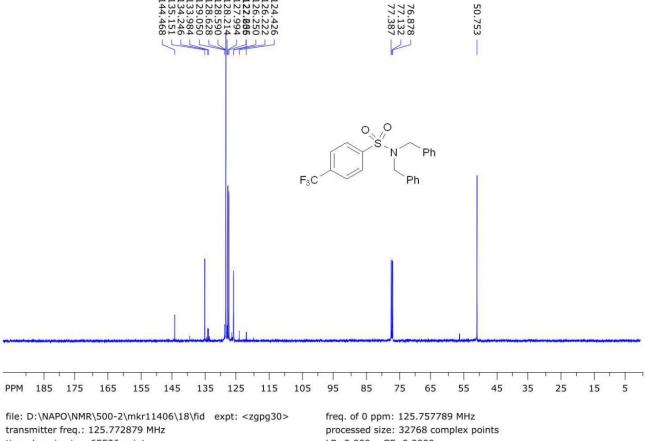


time domain size: 65536 points width: 12335.53 Hz = 24.6645 ppm = 0.188225 Hz/pt number of scans: 24

LB: 0.300 GF: 0.0000 Hz/cm: 201.953 ppm/cm: 0.40380

Compound 4aa

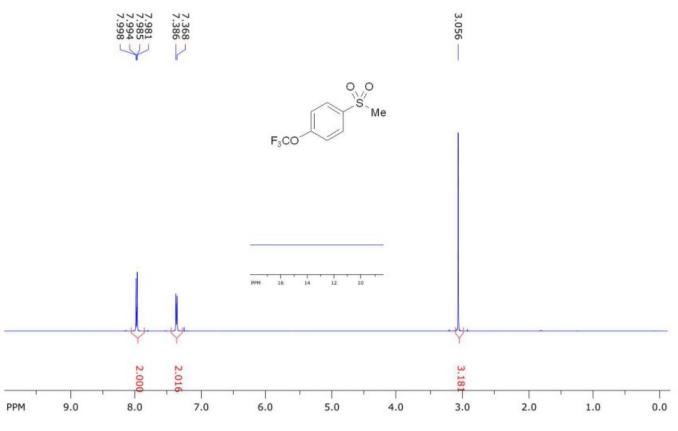
SpinWorks 4: IVA 2976 13C CDCl3



time domain size: 65536 points width: 36057.69 Hz = 286.6889 ppm = 0.550197 Hz/pt number of scans: 512

LB: 2.000 GF: 0.0000 Hz/cm: 993.980 ppm/cm: 7.90297

SpinWorks 4: IVA 2489 1H CDCl3

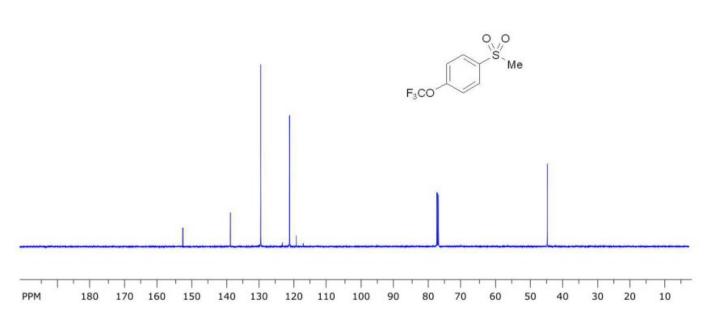


file: ...APO\NMR\500-2\mkr11906\13 2489\fid expt: <zg30> transmitter freq.: 500.133001 MHz time domain size: 65536 points width: 12335.53 Hz = 24.6645 ppm = 0.188225 Hz/pt number of scans: 24

freq. of 0 ppm: 500.130023 MHz processed size: 65536 complex points LB: 0.300 GF: 0.0000 Hz/cm: 203.591 ppm/cm: 0.40707

SpinWorks 4: IVA 2489 13C CDCl3

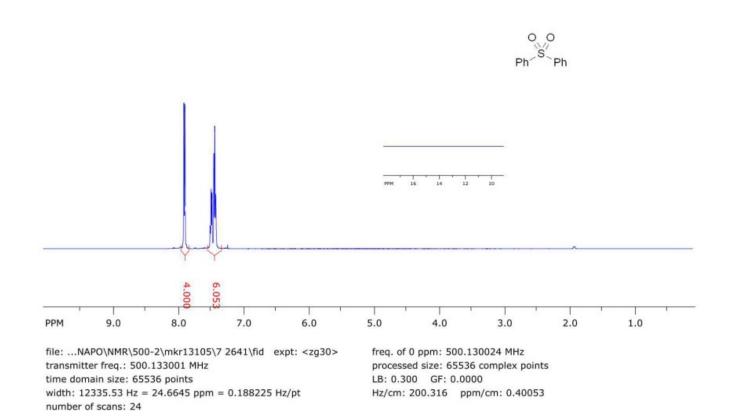




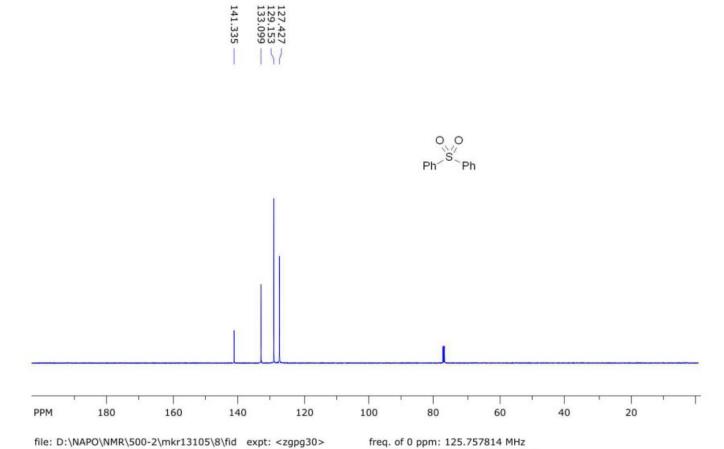
file: D:\NAPO\NMR\500-2\mkr11906\14\fid expt: <zgpg30> transmitter freq.: 125.772879 MHz time domain size: 65536 points width: 36057.69 Hz = 286.6889 ppm = 0.550197 Hz/pt number of scans: 512 freq. of 0 ppm: 125.757798 MHz processed size: 32768 complex points LB: 2.000 GF: 0.0000 Hz/cm: 1001.957 ppm/cm: 7.96640

SpinWorks 4: IVA 2641 1H CDCl3

7.437 7.454 7.468 7.497 7.510 7.914 7.930







TIIE: D:\NAPO\NMK\500-2\mkr13105\8\fid expt: <zgpg30> transmitter freq.: 125.772879 MHz time domain size: 65536 points width: 36057.69 Hz = 286.6889 ppm = 0.550197 Hz/pt number of scans: 512 freq. of 0 ppm: 125.757814 MHz processed size: 32768 complex points LB: 2.000 GF: 0.0000 Hz/cm: 1029.080 ppm/cm: 8.18205