Supporting Information

Polyfluoroalkylated Antipyrines In Pd-Catalyzed Transformations

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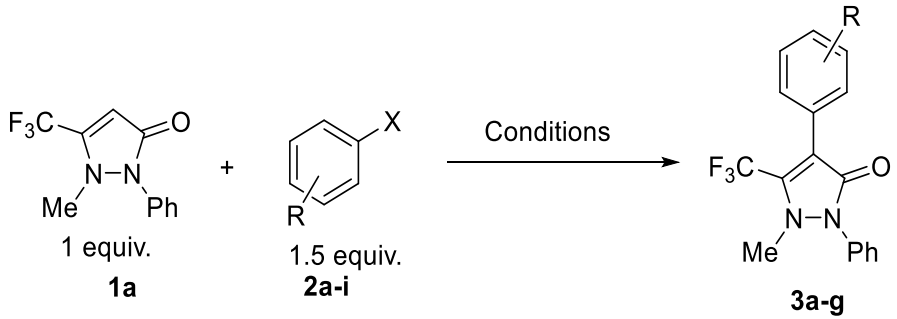
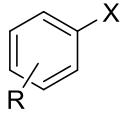
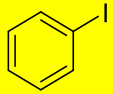
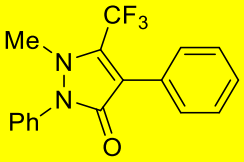
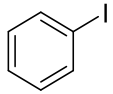
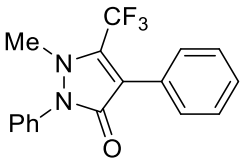
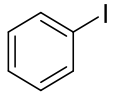
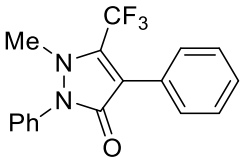
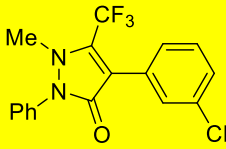
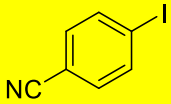
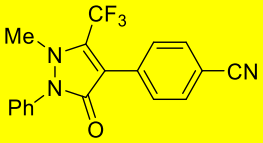
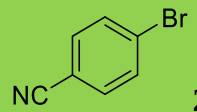
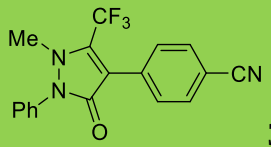
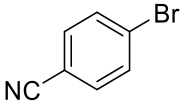
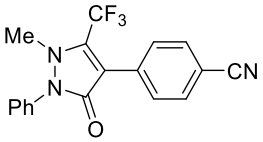
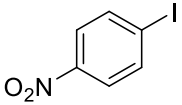
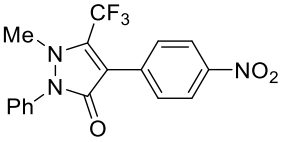
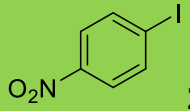
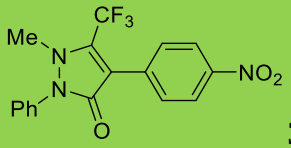
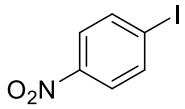
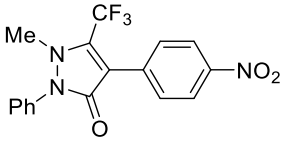
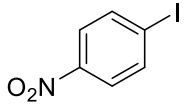
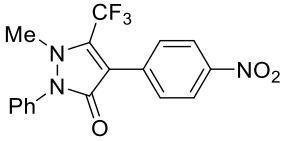
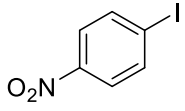
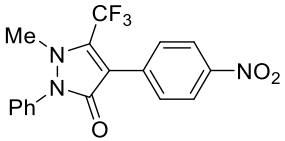
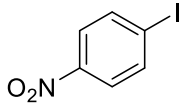
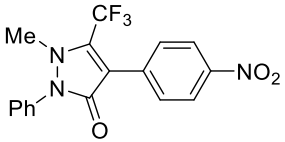
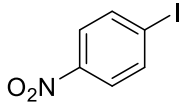
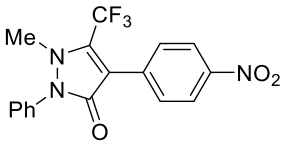
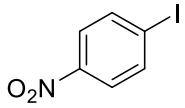
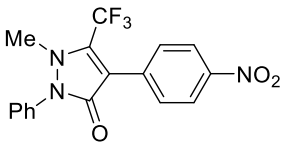
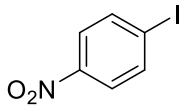
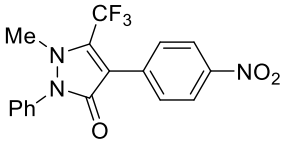
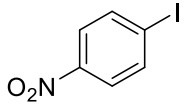
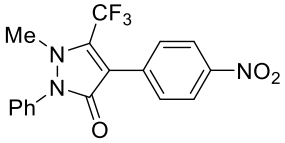
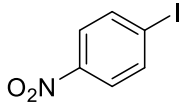
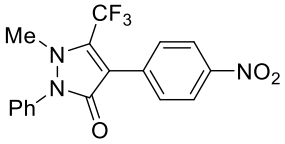
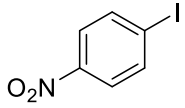
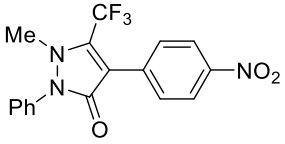
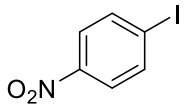
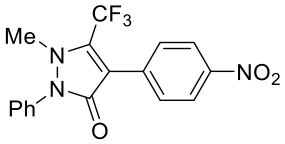
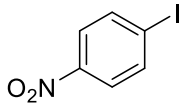
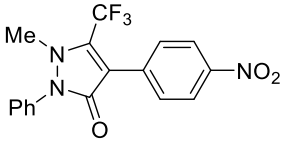
Table S1. Direct C—H arylation of CF_3-antipyrine **1a** (the best conditions for each type of aryl halogenide **2a-i** are highlighted in color: yellow – compound is not isolated; green – compound is yielded in the pure form).



Entry	Conditions		Products **3a-g**	Ratio of components (based on GLC mass spectrometry)			Preparative yield, %
	 **2a-g**	Catalyst, base, solvent, temperature, time		Compound **3**	The initial antipyrine **1a**	By-products	
1.	 **2a**	0.005 equiv. $Pd(OAc)_2$, 2 equiv. AcOK, $CO(OEt)_2$ 150 °C, 24h	 **3a**	5	60	35	0
2.	 **2a**	0.02 equiv. $Pd_2(dba)_3$, 2 equiv. AcOK, $CO(OEt)_2$ 150-160 °C, 24h	 **3a**	2	60	38	-
3.	 **2a**	0.02 equiv. $Pd_2(dba)_3$, 0.04 equiv. XPhos, 2.5 equiv. K_2CO_3, EtOH-H_2O	 **3a**	0	89	11	-

		100 °C, 24h					
4.	 **2b**	0.02 equiv. $Pd(OAc)_2$, 1.3 equiv. K_3PO_4, $CO(OEt)_2$ 150-160 °C, 24h	 **3b**	12	54	34	0
5.	 **2c**	0.02 equiv. $Pd(OAc)_2$, 1.3 equiv. K_3PO_4, $CO(OEt)_2$ 150-160 °C, 24h	 **3c**	13	56	31	0
6.	 **2d**	0.02 equiv. $Pd(OAc)_2$, 1.3 equiv. K_3PO_4, $CO(OEt)_2$ 150-160 °C, 24h	 **3c**	21	54	25	10
7.	 **2d**	0.02 equiv. $Pd(OAc)_2$, 1.3 equiv. AcOK, $CO(OEt)_2$ 150-160 °C, 24h	 **3c**	10	55	35	-
8.	 **2e**	0.02 equiv. $Pd(OAc)_2$, 1 equiv. AcOK, $CO(OEt)_2$ 150-160 °C, 24h	 **3d**	16	74	9	-
9.	 **2e**	0.02 equiv. $Pd(OAc)_2$, 1.3 equiv. AcOK, $CO(OEt)_2$ 150-160 °C, 24h	 **3d**	20	56	23	12

10.	 **2e**	0.02 equiv. $Pd(OAc)_2$, 1.5 equiv. AcOK, $CO(OEt)_2$ 150-160 °C, 24h	 **3d**	6	82	12	-
11.	 **2e**	0.02 equiv. $Pd(OAc)_2$, 2 equiv. AcOK, $CO(OEt)_2$ 150-160 °C, 24h	 **3d**	9	47	43	-
12.	 **2e**	0.02 equiv. $Pd(OAc)_2$, 1.3 equiv. AcOK, DMA 150-160 °C, 24h	 **3d**	resinification			-
13.	 **2e**	0.02 equiv. $Pd(OAc)_2$, 1.3 equiv. AcOK, toluene 150-160 °C, 24h	 **3d**	1	79	20	-
14.	 **2e**	0.02equiv. $Pd(OAc)_2$, 1.3 equiv. AcOK, 1,4-dioxane 150-160 °C, 24h	 **3d**	5	73	22	-
15.	 **2e**	0.02 equiv. $Pd(OAc)_2$, 1.3 equiv. K_2CO_3, $CO(OEt)_2$ 150-160 °C, 24h	 **3d**	4	80	15	-

16.	 **2e**	0.02 equiv. $Pd(OAc)_2$, 1.3 equiv. AcOK, toluene 150-160 °C, 24h	 **3d**	1	79	20	-
17.	 **2e**	0.02 equiv. $Pd(OAc)_2$, 1.3 equiv. AcOK, 1,4-dioxane 150-160 °C, 24h	 **3d**	5	73	22	-
18.	 **2e**	0.02 equiv. $Pd(OAc)_2$, 1.3 equiv. K_2CO_3, $CO(OEt)_2$ 150-160 °C, 24h	 **3d**	4	80	15	-
19.	 **2e**	0.02 equiv. $Pd(OAc)_2$, 1.3 equiv. Cs_2CO_3, $CO(OEt)_2$ 150-160 °C, 24h	 **3d**	0	81	15	-
20.	 **2e**	0.02 equiv. $Pd(OAc)_2$, 1.3 equiv. K_3PO_4, $CO(OEt)_2$ 150-160 °C, 24h	 **3d**	6	76	18	-
21.	 **2e**	0.02 equiv. $Pd_2(dba)_3$, 0.04 equiv. XPhos, 2.5 equiv. K_2CO_3, EtOH-H_2O 100 °C, 8h	 **3d**	0	53	27	-

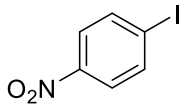
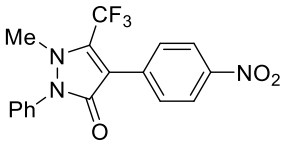
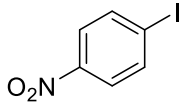
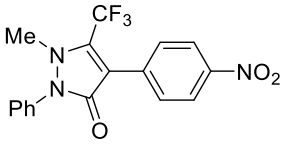
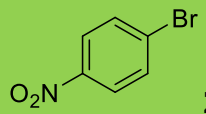
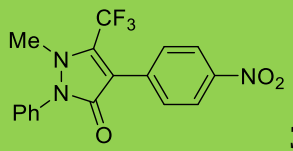
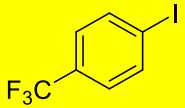
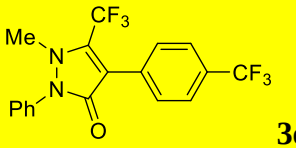
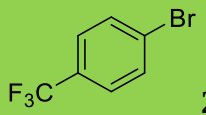
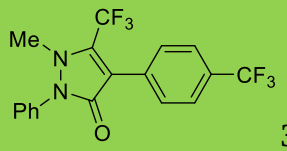
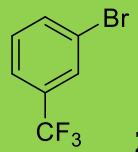
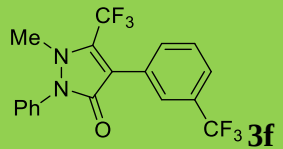
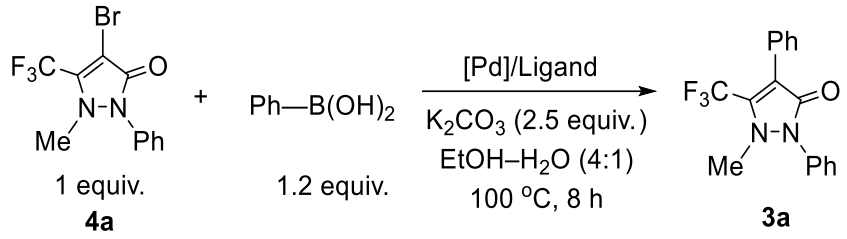
22.	 **2e**	0.02 equiv. $Pd_2(dba)_3$, 0.04 equiv. XPhos, 2 equiv. AcOK, $CO(OEt)_2$ 150-160 °C, 24h	 **3d**	5	45	50	-
23.	 **2e**	0.02 equiv. $Pd_2(dba)_3$, 1.3 equiv. K_3PO_4, $CO(OEt)_2$ 150-160 °C, 24h	 **3d**	8	78	12	-
24.	 **2f**	0.02 equiv. $Pd(OAc)_2$, 1.3 equiv. K_3PO_4, $CO(OEt)_2$ 150-160 °C, 24h	 **3d**	42	38	20	27
25.	 **2g**	0.02 equiv. $Pd(OAc)_2$, 1.3 equiv. K_3PO_4, $CO(OEt)_2$ 150-160 °C, 24h	 **3e**	7	54	39	0
26.	 **2h**	0.02 equiv. $Pd(OAc)_2$, 1.3 equiv. K_3PO_4, $CO(OEt)_2$ 150-160 °C, 24h	 **3e**	40	47	13	25
27.	 **2i**	0.02 equiv. $Pd(OAc)_2$, 1.3 equiv. K_3PO_4, $CO(OEt)_2$ 150-160 °C, 24h	 **3f**	32	52	16	23

Table S2. Optimization of the conditions for synthesis of 4-phenyl-5-trifluoromethyl-antipyrine **3a** from bromo-antipyrine **4a**

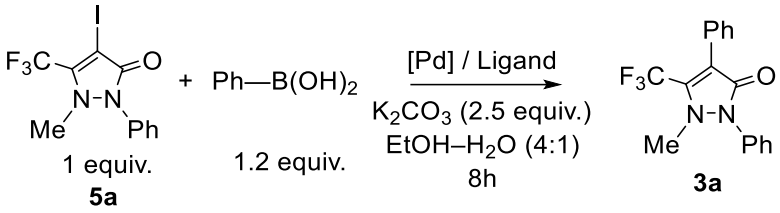


Entry	Conditions		Ratio of components (based on GLC mass spectrometry)			
	[Pd]	Ligand	Product **3a**	Initial Br-antipyrine **4a**	Debromination product – antipyrine **1a**	Impurities
1[a]	0.05 equiv. $Pd(PPh_3)_4$		2	85	3	10
2	0.05 equiv. $Pd(PPh_3)_4$		72	-	10	18
3	0.02 equiv. $Pd_2(dba)_3$	0.04 equiv. P(*o*-Tol)$_3$	76	2	**11**	**11**
4	0.02 equiv. $Pd_2(dba)_3$	0.04 equiv. P(*p*-Tol)$_3$	62	3	14	21
5	**0.02 equiv. $Pd_2(dba)_3$**	**0.04 equiv. XPhos**	**86**	-	**6**	**8**
6	0.02 equiv. $Pd_2(dba)_3$	0.04 equiv. Xantphos	81	-	4	15
7	0.02 equiv. $Pd_2(dba)_3$	0.04 equiv. BINAP	76	-	8	16
8[b]	0.02 equiv. $Pd_2(dba)_3$	0.04 equiv. XPhos	62	-	5	33

[a] Solvent - THF-H_2O (3:4)

[b] Reaction was performed in a closed vial in microwave synthesizer "CEM Discover", MW 100 Wt, 100°C, 8 h

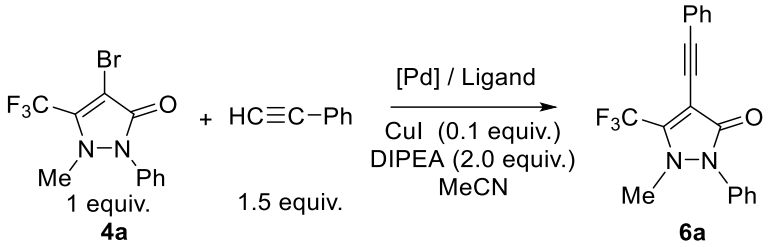
Table S3. Optimization of the conditions for synthesis of 4-phenyl-5-trifluoromethyl-antipyrine **3a** from iodo-antipyrine **5a**



Entry	Conditions			Ratio of components (based on GLC mass spectrometry)			
	[Pd]	Ligand	T, °C	Product **3a**	Initial I-antipyrine **5a**	Deiodation product – antipyrine **1a**	Impurities
1	0.05 equiv. $Pd(PPh_3)_4$		100	25	-	47	28
2	0.02 equiv. $Pd_2(dba)_3$	0.04 equiv. Xantphos	100	33	-	59	8
3	0.02 equiv. $Pd_2(dba)_3$	0.04 equiv. BINAP	100	27	-	57	16
4	0.02 equiv. $Pd_2(dba)_3$	0.04 equiv. XPhos	100	32	-	55	13
5[a]	0.02 equiv. $Pd_2(dba)_3$	0.04 equiv. XPhos	100	32	-	60	8
6[b]	**0.02 equiv. $Pd_2(dba)_3$**	**0.04 equiv. XPhos**	**rt**	**100**	-	-	-
7[b]	0.05 equiv. $Pd(PPh_3)_4$		rt	31	-	42	27

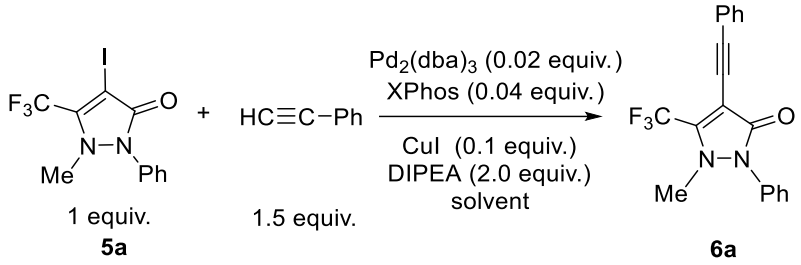
[a] Base Cs_2CO_3 (2.5 equiv.)
[b] Time of reaction is 72 h

Table S4. Optimization of the conditions for synthesis of 4-(phenylethynyl)-5-(trifluoromethyl)-antipyrine **6a** from bromo-antipyrine **4a**



Entry	Conditions				Ratio of components (based on GLC mass spectrometry)			
	[Pd]	Ligand	T, °C	Time, h	Product **6a**	Initial Br-antipyrine **4a**	Debromination product **1a**	1,4-diphenyl-1,3-butadiyne + impurities
1	0.05 equiv. $Pd(PPh_3)_4$		80	12	29	36	-	35
2	0.05 equiv. $Pd(PPh_3)_4$		100	6	35	12	-	53
3	0.02 equiv. $Pd_2(dba)_3$	0.04 equiv. BINAP	80	12	8	49	-	43
4	0.02 equiv. $Pd_2(dba)_3$	0.04 equiv. Xantphos	80	12	30	34	-	36
5	0.02 equiv. $Pd_2(dba)_3$	0.04 equiv. XPhos	80	12	18	37	1	44
6	0.02 equiv. $Pd_2(dba)_3$	0.04 equiv. XPhos	100	6	36	17	1	46

Table S5. Optimization of the conditions for synthesis of 4-(phenylethynyl)-5-(trifluoromethyl)-antipyrine **6a** from iodo-antipyrine **5a**



Entry	Conditions			Ratio of components (based on GLC mass spectrometry)			
	Solvent	T, °C	Time, h	Product **6a**	Initial I-antipyrine **5a**	Deiodation product **1a**	1,4-diphenyl-1,3-butadiyne + impurities
1	Toluene	100	14	2	-	8	90
2	1,4-Dioxane	100	14	13	-	48	39
3	Acetonitrile	100	14	25	-	45	30
4	Acetonitrile	50	4	35	-	43	23
5	Acetonitrile	rt	4	25	70	-	5
6	**Acetonitrile**	**rt**	**72**	**75**	**-**	**-**	**25**

Experimental

All solvents, chemicals, and reagents were obtained commercially and used without purification. Melting points were measured in open capillaries on a Stuart SMP30 melting point apparatus and were uncorrected. The IR spectra were recorded on Perkin Elmer «Spectrum Two» using frustrated total internal reflection accessory with diamond crystal at ν 4000–400 cm^{-1}. The ^{1}H and ^{19}F NMR spectra were registered on a Bruker DRX-400 (400 or 376 MHz, respectively) or a Bruker Avance^{III} 500 (500 or 470 MHz, respectively). The ^{13}C NMR spectra were recorded on a Bruker Avance^{III} 500 (125 MHz). The internal standard is $SiMe_4$ (for ^{1}H and ^{13}C NMR spectra) and C_6F_6 (for ^{19}F NMR spectra, δ –162.9 ppm). The microanalyses (C, H, N) were carried out on a PerkinElmer PE 2400 series II elemental analyzer. The column chromatography was performed on Silica gel 60 (0.062-0.2 mm).

Crystallographic data

The X-ray studies were performed on "Xcalibur 3 CCD" diffractometer with graphite monochromator, *ω* scanning with 1° step, *λ*(Mo*K*α) 0.71073 Å radiation, *T* 295(2) K. Empirical absorption correction was applied. Using Olex2 [1], the structure was solved with the ShelXS structure solution program using Direct Methods and refined with the ShelXL [2] refinement package using Least Squares minimization. All non-hydrogen atoms were refined in the anisotropic approximation; H-atoms of the OH-groups were localized from the electron density peaks and refined independently; H-atoms at the C-H bonds were refined in the "rider" model with dependent displacement parameters. Empirical absorption correction was carried out through spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm by a program "CrysAlisPro 1.171.39.38a" (Rigaku Oxford Diffraction, 2017).

Synthesis of 5-(polyfluoroalkyl)-antipyrines 1a-d

The initial antipyrines **1a-d** were synthesized by the known method [3].

Synthesis of 4-halogen-5-polyfluoroalkyl-antipyrines 4a-d, 5a,b.

A mixture of antipyrine **1a-d** (1 mmol) and N-halogensuccinimide (1.2 mmol) in $CHCl_3$ (20 ml) was stirring at the room temperature for 3-4 h. Then, it was added water (20 ml), the organic lay was separated, dried over Na_2SO_4 and distilled off on a rotary evaporator. The residue was re-crystallized from *n*-hexane.

4-Bromo-1-methyl-2-phenyl-5-(trifluoromethyl)-1,2-dihydro-3*H*-pyrazol-3-one (4a). Yield 85%, light-yellow powder, mp 79-80 °C. ^{1}H NMR (DMSO-d_6, 400 MHz): δ 3.22 (s, 3H, Me); 7.46-7.49, 7.46-7.61 (all m, 5H, Ph). ^{13}C NMR (DMSO-d_6, 125 MHz): δ 37.74 (Me); 92.80 (q, *J* 2.8 Hz, C—Br); 118.96 (q, *J* 272.8 Hz, CF_3); 125.76, 128.66, 129.49, 132.39 (Ph); 140.23 (q, *J* 37.0 Hz, C—CF_3); 159.11 (C═O). ^{19}F NMR (DMSO-d_6, 376 MHz): δ 102.85 (s, CF_3). IR: ν

1690 (C═O); 1489, 1457, 1400 (C═C); 1061-1172 (CF), 704-759 (CBr) cm^{-1}. Anal. calcd for $C_{11}H_8BrF_3N_2O$: C, 41.15; H, 2.51; N, 8.72. Found: C, 41.23; H, 2.67; N, 8.79.

4-Bromo-1-methyl-5-(pentafluoroethyl)-2-phenyl-1,2-dihydro-3*H*-pyrazol-3-one (4b). Yield 83%, white powder, mp 95 °C. ^1H NMR ($CDCl_3$, 400 MHz): δ 3.20 (s, 3H, Me); 7.41-7.43, 7.51-7.53 (both m, 5H, Ph). ^{13}C NMR ($CDCl_3$, 125 MHz): δ 38.60-38.66 (m, Me); 98.74 (t, *J* 1.9 Hz, C—Br); 109.98 (tq, *J* 258.5, 41.3 Hz, CF_2); 118.37 (qt, *J* 287.5, 37.0 Hz, CF_3); 125.11, 128.59, 129.64, 132.84 (Ph); 140.67 (t, *J* 27.1 Hz, C—CF_2); 160.09 (C═O). ^{19}F NMR ($CDCl_3$, 376 MHz): δ 49.22-49.23 (m, 2F, CF_2), 78.60 (t, *J* 3.3 Hz, 3F, CF_3). IR: ν 1687 (C═O); 1487, 1458, 1330 (C═C); 1113-1210 (CF) cm^{-1}. Anal. calcd for $C_{12}H_8BrF_5N_2O$: C, 38.84; H, 2.17; N, 7.55. Found: C, 38.96; H, 2.17; N, 7.71.

4-Bromo-5-(heptafluoropropyl)-1-methyl-2-phenyl-1,2-dihydro-3*H*-pyrazol-3-one (4c). Yield 79%, white powder, mp 97-98 °C. ^1H NMR ($CDCl_3$, 400 MHz): δ 3.20 (s, 3H, Me); 7.41-7.42, 7.51-7.53 (both m, 5H, Ph). ^{13}C NMR ($CDCl_3$, 125 MHz): δ 38.64-38.71 (m, Me); 99.03 (unsolv. t, C—Br); 108.76 (tq, *J* 266.8, 33.6 Hz, β-CF_2); 112.07 (tt, *J* 259.7, 38.5 Hz, α-CF_2); 117.49 (qt, *J* 288.0, 33.6 Hz, CF_3); 125.19, 128.65, 129.62, 132.85 (Ph); 140.65 (t, *J* 27.5 Hz, C—CF_2); δ 160.13 (C═O). ^{19}F NMR ($CDCl_3$, 376 MHz): δ 36.34-36.40 (m, 2F, CF_2); 52.15-52.21 (m, 2F, CF_2); 81.68 (t, *J* 9.7 Hz, 3F, CF_3). IR: ν 1695 (C═O), 1497, 1459, 1388, 1337 (C═C), 1236-1109 (CF) cm^{-1}. Anal. calcd for $C_{13}H_8BrF_7N_2O$: C, 37.08; H, 1.19; N, 6.75. Found: C, 37.09; H, 2.14; N, 6.75.

4-Bromo-1-methyl-5-(nonafluorobuthyl)-2-phenyl-1,2-dihydro-3*H*-pyrazol-3-one (4d). Yield 75%, white powder, mp 99-100 °C. ^1H NMR ($CDCl_3$, 400 MHz): δ 3.20 (s, 3H, Me); 7.40-7.44, 7.51-7.55 (both m, 5H, Ph). ^{13}C NMR ($CDCl_3$, 125 MHz): δ 38.67-38.68 (m, Me); 99.14 (unsolv. t, C—Br); 108.50-114.76 (m, 3CF_2); 117.25 (qt, *J* 288.6, 33.0 Hz, CF_3); 125.19, 128.65, 129.66, 132.85 (Ph); 140.66 (t, *J* 27.6 Hz, C—CF_2); δ 160.13 (C═O). ^{19}F NMR ($CDCl_3$, 376 MHz): δ 35.83-35.92, 39.97-40.00, 52.87-52.94 (all m, 6F, 3CF_2); 80.94-81.00 (m, 3F, CF_3). IR: ν 1677 (C═O), 1497, 1462, 1396 (C═C), 1114-1217 (CF) cm^{-1}. Anal. calcd for $C_{14}H_8BrF_9N_2O$: C, 35.69; H, 1.71; N, 3.40. Found: C, 35.78; H, 1.91; N, 6.09.

4-Iodo-1-methyl-2-phenyl-5-(trifluoromethyl)-1,2-dihydro-3*H*-pyrazol-3-one (5a). Yield 80%, yellow powder, mp 85°C. ^1H NMR ($CDCl_3$, 400 MHz): δ 3.24 (s, 3H, Me); 7.40-7.43, 7.49-7.53 (both м, 5H, Ph). ^{13}C NMR ($CDCl_3$, 125 MHz): δ 38.26 (q, *J* 2.0 Hz, Me); 64.90 (q, *J* 2.1 Hz, C—I); 118.86 (q, *J* 273.3 Hz, CF_3); 124.78, 128.31, 129.56, 132.99 (Ph); 145.97 (q, *J* 37.1 Hz, C—CF_3); 162.12 (C═O). ^{19}F NMR ($CDCl_3$, 376 MHz): δ 101.31 (s, CF_3). IR: ν 1661 (C═O), 1594, 1552, 1496 (C═C), 1024-1245 (CF) cm^{-1}. Anal. calcd for $C_{11}H_8F_3IN_2O$: C, 35.89; H, 2.19; N, 7.61. Found: C, 36.10; H, 2.23; N, 7.69.

4-Iodo-1-methyl-5-(pentafluoroethyl)-2-phenyl-1,2-dihydro-3*H*-pyrazol-3-one (5b). Yiled 75 %, yellow powder, mp 80 °C. ^{1}H NMR ($CDCl_3$, 400 MHz): δ 3.23 (s, 3H, Me); 7.40-7.42, 7.50-7.54 (both m, 5H, Ph). ^{13}C NMR ($CDCl_3$, 125 MHz): δ 38.72 (br.s, Me); 68.08 (unsolv. t, C–I); 109.75 (tq, *J* 258.7, 41.1 Hz, CF_2); 118.31 (qt, *J* 287.7, 37.0 Hz, CF_2); 125.17, 128.54, 129.60 (Ph); 143.92 (t, *J* 27.0 Hz, C—CF_2); 162.20 (C═O). ^{19}F NMR ($CDCl_3$, 376 MHz): δ 50.33 (q, *J* 3.5 Hz, CF_2), 78.96 (t, *J* 3.5 Hz, CF_3). IR: ν 1667 (C═O), 1496, 1485, 1457 (C═C), 1019-1213 (CF) cm^{-1}. Anal. calcd for $C_{12}H_8F_5IN_2O$: C, 34.73; H, 1.93; N, 6.70. Found: C, 34.69; H, 1.91; N, 6.86.

Synthesis of 4-aryl-5-(polyfluoroalkyl)-antipyrines 3a,c-l

Method A. A screw-neck vial was flushed with argon and charged with 4-bromo-5-(polyfluoroalkyl)-antipyrine **4a-d** (1 mmol), arylboronic acid (1.2 mmol) (phenylboronic acid - 122 mg, (4-(methylthio)phenyl)boronic acid – 168 mg), XPhos (0.04 mmol, 19 mg), $Pd_2(dba)_3$ (0.02 mmol, 18 mg), K_2CO_3 (2.5 mmol, 345 mg), EtOH (4 ml), and water (1 ml). The vial was repeatedly flushed with argon, closed tightly and heated at 100 °C for 8 h. After cooling, another portion of ethanol (20 ml) was added and a mixture was filtered through a small quantity of silica gel. The solvent was evaporated in vacuo, and the residue was purified by column chromatography on silica gel using a mixture of $CHCl_3$ and EtOAc (4:1).

Method B. Similarly to method A from 1 mmol of 4-iodo-5-(polyfluoroalkyl)-antipyrine **5a,b** at the room temperature for 72 h.

Method C. A screw-neck vial was flushed with argon and charged with 5-(trifluoromethyl)-antipyrine **1a** (1 mmol, 242 mg), arylhalogenide (1.5 mmol), $Pd(OAc)_2$ (0.02 mmol, 4.5 mg), K_3PO_4 (1.3 mmol, 275 mg), and diethyl carbonate (4 ml). The vial was repeatedly flushed with argon, closed tightly and heated at 150-160 °C for 20 h. After cooling, the solvent was evaporated in vacuo, and the residue was purified by column chromatography on silica gel using a mixture of *n*-hexane and EtOAc (4:1).

2,4-Diphenyl-1-methyl-5-(trifluoromethyl)-1,2-dihydro-3*H*-pyrazol-3-one (3a). Yield 69% (*method A*), 89% (*method B*), white powder, mp 94-95 °C. ^{1}H NMR ($CDCl_3$, 500 MHz): δ 3.25 (s, 3H, Me); 7.34-7.43, 7.47-7.49, 7.50-7.52 (all m, 10H, 2 Ph). ^{13}C NMR ($CDCl_3$, 125 MHz): δ 38.13 (Me); 117.93 (═C—Ph); 119.91 (q, *J* 272.9 Hz, CF_3); 124.46, 127.67, 128.09, 128.15, 128.59, 129.42, 129.77, 133.40 (2 Ph); 140.86 (q, *J* 36.8 Hz, C—CF_3); 162.44 (C═O). ^{19}F NMR ($CDCl_3$, 470 MHz): δ 102.96 (s, CF_3). IR: 1678 (C═O), 1413, 1450, 1495, 1596 (C═C), 1076-1153 (CF) cm^{-1}. Anal. calcd for $C_{17}H_{13}F_3N_2O$: C, 64.15; H, 4.12; N, 8.80. Found: C, 63.67; H, 4.00; N, 8.56.

1-Methyl-2-phenyl-5-(trifluoromethyl)-4-[4-(cyano)phenyl]-1,2-dihydro-3*H*-pyrazol-3-one (3c):antipyrine (1a) – 83:17. Yield 10% (*method C*). ^{1}H NMR ($CDCl_3$, 500 MHz): δ 3.32 (s, 3H, Me); 7.40-7.56 (m, 5H, Ph); 7.61, 7.70 (both d, *J* 8.3 Hz, 4H, C_6H_4). ^{19}F NMR ($CDCl_3$, 470 MHz): δ 103.05 (s, CF_3).

1-Methyl-2-phenyl-5-(trifluoromethyl)-4-[4-(nitro)phenyl]-1,2-dihydro-3H-pyrazol-3-one (3d). Yield 27% (*method C*), off-white powder, mp 103-104 °C. ^{1}H NMR ($CDCl_3$, 500 MHz): δ 3.35 (s, 3H, Me); 7.42-7.49, 7.54-7.57 (both m, 5H, Ph); 7.68, 8.28 (both d, *J* 8.6 Hz, 4H, C_6H_4). ^{13}C NMR ($CDCl_3$, 125 MHz): δ 37.67 (q, *J* 2.0 Hz, Me); 114.22 (═C—Ar); 119.62 (q, *J* 273.0 Hz, CF_3); 123.36, 125.14, 128.48, 129.67, 130.83, 132.81, 135.14, 147.65 (C_6H_4 and Ph); 140.46 (q, *J* 37.2 Hz, C^4—CF_3); 161.58 (C═O). ^{19}F NMR ($CDCl_3$, 470 MHz): δ 103.07 (s, CF_3). IR: 1660 (C═O), 1627, 1595, 1493, 1455, 1441, 1420 (C═C), 1155-1095 (CF) cm^{-1}. Anal. calcd for $C_{17}H_{12}F_3N_3O_3$: C, 56.20; H, 3.33; N, 11.57. Found: C, 56.32; H, 3.27; N, 11.39.

1-Methyl-2-phenyl-5-(trifluoromethyl)-4-[4-(trifluoromethyl)phenyl]-1,2-dihydro-3H-pyrazol-3-one (3e). Yield 25% (*method C*), off-white powder, mp 99-100 °C. ^{1}H NMR ($CDCl_3$, 500 MHz): δ 3.30 (s, 3H, Me); 7.39-7.42, 7.48-7.50, 7.52-7.55 (all m, 5H, Ph); 7.61, 7.68 (both d, *J* 8.1 Hz, 4H, C_6H_4). ^{13}C NMR ($CDCl_3$, 125 MHz): δ 37.81 (q, *J* 1.9 Hz, Me); 115.62 (═C—Ar); 119.70 (q, *J* 273.0 Hz, CF_3); 121.86 (q, *J* 270.0 Hz, C_6H_4–CF_3); 124.85, 128.15, 129.56, 133.01 (Ph); 125.13 (q, *J* 3.7 Hz, C^o Ar^F); 130.22 (q, *J* 1.2 Hz, C^m Ar^F); 130.77 (q, *J* 32.6 Hz, C^i Ar^F); 131.98 (q, *J* 1.1 Hz, C^p Ar^F); 140.72 (q, *J* 37.2 Hz, C^4—CF_3); 161.95 (C═O). ^{19}F NMR ($CDCl_3$, 470 MHz): δ 98.99, 103.01 (both s, 6F, $2CF_3$). IR: 1666 (C═O), 1620, 1593, 1496, 1457, 1442, 1421 (C═C), 1165-1068 (CF) cm^{-1}. Anal. calcd for $C_{18}H_{12}F_6N_2O$: C, 55.97; H, 3.13; N, 7.25. Found: C, 55.69; H, 3.05; N, 7.36.

Colorless single crystals of compound **3e** were obtained by crystallization from a mixture of $CHCl_3$:*n*-hexane:EtOAc – 4:4:1. Main crystallographic data for **3e**: $C_{18}H_{12}F_6N_2O$, *M* 386.30, space group $P\bar{1}$, triclinic, *a* 8.6416(7), *b* 11.0188(9), *c* 18.0722(14) Å, *α* 93.734(6), *β* 90.935(6), *γ* 95.221(6)°, *V* 1709.6(2) Å^3, *Z* 4, D_{calc} 1.501 g·cm^{-3}, μ 0.139 mm^{-1}, 584 refinement parameters, 8397 reflections measured, 3326 unique reflections which were used in all calculations. The final R is 0.057. CCDC 2110385 contains the supplementary crystallographic data for this compound.

1-Methyl-2-phenyl-5-(trifluoromethyl)-4-[3-(trifluoromethyl)phenyl]-1,2-dihydro-3H-pyrazol-3-one (3f). Yield 23% (*method C*), white powder, mp 105-106 °C. ^{1}H NMR ($CDCl_3$, 500 MHz): δ 3.30 (s, 3H, Me); 7.39-7.42, 7.49-7.54, 7.63-7.68, 7.77 (all m, 9H, C_6H_4 and Ph). ^{13}C NMR ($CDCl_3$, 125 MHz): δ 37.90 (q, *J* 1.9 Hz, Me); 115.76 (═C—Ar); 119.74 (q, *J* 273.0 Hz, CF_3); 121.84 (q, *J* 255.8 Hz, C_6H_4–CF_3); 124.80, 128.12, 129.57, 133.11 (Ph); 125.33 (q, *J* 3.7 Hz, C Ar^F); 130.75 (q, *J* 32.6 Hz, $C^{3'}$ Ar^F); 126.81, 128.69, 129.07, 133.17 (4C Ar^F); 140.85 (q, *J* 37.1Hz, C^4—CF_3); 162.05 (C═O). ^{19}F NMR ($CDCl_3$, 470 MHz): δ 99.00, 102.91 (both s, 6F,

2CF_3). IR: 1675 (C═O), 1624, 1596, 1496, 1459, 1411 (C═C), 1154-1074 (CF) cm^{-1}. Anal. calcd for $C_{18}H_{12}F_6N_2O$: C, 55.97; H, 3.13; N, 7.25. Found: C, 55.72; H, 3.15; N, 7.29.

1-Methyl-4-(4-(methylthio)phenyl)-5-(trifluoromethyl)-2-phenyl-1,2-dihydro-3*H*-pyrazol-3-one (3g). Yield 59% (*method A*), 79% (*method B*), beige powder, mp 113-115 °C. ^{1}H NMR (DMSO-d_6, 500 MHz): δ 2.51 (s, 3H, SMe); 3.23 (s, 3H, Me); 7.34-7.35, 7.50-7.51, 7.57-7.59 (all m, 10H, 2 Ph). ^{13}C NMR (DMSO-d_6, 125 MHz): δ 14.31 (SMe); 39.16 (Me); 115.28 (unsolv. q, ═C—Ar); 119.76 (q, *J* 272.7 Hz, CF_3); 124.57, 125.17, 125.21, 127.97, 129.41, 130.06, 132.95, 138.91 (2 Ph); 138.92 (q, 36.2 Hz, C—CF_3); 161.29 (C═O). ^{19}F NMR (DMSO-d_6, 470 MHz): δ 104.68 (s, CF_3). IR: 1680 (C═O), 1412, 1429, 1456, 1494 (C═C), 1094-1157 (CF) cm^{-1}. Anal. calcd for $C_{18}H_{15}F_3N_2OS$. C, 59.33; H, 4.15; N, 7.69. Found: C, 56.47; H, 3.66; N, 6.27.

2,4-Diphenyl-1-methyl-5-(pentafluoroethyl)-1,2-dihydro-3*H*-pyrazol-3-one (3h). Yield 68% (*method A*), 86% (*method B*), beige powder, mp 155 °C. ^{1}H NMR ($CDCl_3$, 500 MHz): δ 3.23 (s, 3H, Me); 7.31-7.45, 7.48-7.57 (all m, 10H, 2 Ph). ^{13}C NMR ($CDCl_3$, 125 MHz): δ 38.70 (Me); 110.60 (tq, *J* 257.4, 40.7 Hz, CF_2); 118.45 (qt, *J* 287.3, 37.0 Hz, CF_2); 121.49 (═C—Ph); 124.66, 127.77, 127.97, 128.33, 128.45, 129.42, 129.65, 130.07 (2 Ph); 133.50 (t, *J* 27.4 Hz, C—CF_2); 162.63 (C═O). ^{19}F NMR ($CDCl_3$, 470 MHz): δ 52.38 (br.s, 2F, CF_2), 78.37 (t, *J* 3.2 Hz, 3F, CF_3). IR: 1670 (C═O), 1122, 1139, 1184, 1216, 1223 (C═C), 1018-1079 (CF) cm^{-1}. Anal. calcd for $C_{18}H_{13}F_5N_2O$: C, 58.70; H, 3.56; N, 7.61. Found: C, 57.88; H, 3.34; N, 7.44.

1-Methyl-4-(4-(methylthio)phenyl)-5-(pentafluoroethyl)-2-phenyl-1,2-dihydro-3*H*-pyrazol-3-one (3i). Yield 54% (*method A*), 73% (*method B*), beige powder, mp 99-100 °C. ^{1}H NMR ($CDCl_3$, 500 MHz): δ 2.50 (s, 3H, SMe); 3.22 (s, 3H, Me); 7.26-7.31, 7.38-7.40, 7.48-7.54 (all m, 10H, 2 Ph). ^{13}C NMR ($CDCl_3$, 125 MHz): δ 15.47 (SMe); 38.69 (Me); 110.60 (tq, *J* 257.3, 40.6 Hz, CF_2); 118.45 (qt, *J* 287.3, 37.1 Hz, CF_2); 120.85 (═C—Ar); 124.71, 124.83, 125.82, 127.85, 129.44, 130.42, 133.41, 139.24 (2 Ph); 138.97 (t, *J* 27.5 Hz, C—CF_2); 162.59 (C═O). ^{19}F NMR ($CDCl_3$, 470 MHz): δ 52.42 (br.s, 2F, CF_2), 78.40 (t, *J* 3.3 Hz, 3F, CF_3). IR: 1671 (C═O), 1460, 1459, 1587 (C═C), 1147-1219 (CF) cm^{-1}. Anal. calcd for $C_{19}H_{15}F_5N_2OS$: C, 55.07; H, 3.65; N, 6.76. Found: C, 55.27; H, 3.60; N, 6.47.

2,4-Diphenyl-5-(heprtafluoropropyl)-1-methyl-1,2-dihydro-3*H*-pyrazol-3-one (3j). Yield 65% (*method A*), beige powder, mp 160 °C. ^{1}H NMR ($CDCl_3$, 500 MHz): δ 3.23 (s, 3H, Me); 7.37-7.38, 7.48-7.54 (all m, 10H, 2 Ph). ^{13}C NMR ($CDCl_3$, 125 MHz): δ 38.84 (Me); 108.68 (tq, *J* 266.7, 38.0 Hz, β-CF_2); 112.62 (tt, *J* 258.0, 32.9 Hz, α-CF_2); 117.56 (qt, *J* 288.0, 33.7 Hz, CF_3); 121.84 (═C—Ph); 124.68, 127.81, 127.96, 128.34, 128.47, 129.45, 130.09 (2 Ph); 139.39 (t, *J* 27.7 Hz, C—CF_2); 162.67 (C═O). ^{19}F NMR ($CDCl_3$, 470 MHz): δ 36.34-36.41 (m, 2F, CF_2); 55.19 (q, *J* 9.7 Hz, 2F, CF_2); 81.66 (t, *J* 9.7 Hz, 3F, CF_3). IR: 1671 (C═O), 1338, 1498, 1593

(C═C), 1116-1235 (CF) cm^{-1}. Anal. calcd for $C_{19}H_{13}F_7N_2O$: C, 54.55; H, 3.13; N, 6.70. Found: C, 52.76; H, 3.16; N, 6.48.

5-(Heptafluoropropyl)-1-methyl-4-(4-(methylthio)phenyl)-2-phenyl-1,2-dihydro-3*H*-pyrazol-3-one (3k). Yield 52% (*method A*), beige powder, mp 155-156 °C. ^{1}H NMR ($CDCl_3$, 500 MHz): δ 2.50 (s, 3H, SMe); 3.22 (s, 3H, Me); 7.26-7.28, 7.36-7.40, 7.47-7.52 (all m, 10H, 2 Ph). ^{13}C NMR ($CDCl_3$, 125 MHz): δ 15.49 (SMe); 38.86-38.88 (m, Me); 107.73 (tq, *J* 266.8, 37.9 Hz, β-CF_2); 112.66 (tt, *J* 258.0, 32.9 Hz, α-CF_2); 117.59 (qt, *J* 288.2, 33.8 Hz, CF_3); 121.25 (═C—Ph); 124.77, 124.89, 125.82, 127.91, 129.50, 130.48, 133.48, 139.29 (2 Ph); 139.01 (t, *J* 28.0 Hz, C—CF_2); 162.67 (C═O). ^{19}F NMR ($CDCl_3$, 470 MHz): δ 36.36-36.46 (m, 2F, CF_2), 55.21-55.23 (m, 2F, CF_2), 81.70 (t, *J* 9.8 Hz, 3F, CF_3). IR: 1674 (C═O), 1405, 1459, 1587 (C═C), 1094-1240 (CF) cm^{-1}. Anal. calcd for $C_{20}H_{15}F_7N_2OS$: C, 51.73; H, 3.26; N, 6.03. Found: C, 51.92; H, 3.33; N, 6.01.

2,4-Diphenyl-1-methyl-5-(nonafluorobutyl)-1,2-dihydro-3*H*-pyrazol-3-one (3l). Yield 67% (*method A*), beige powder, mp 97-98 °C. ^{1}H NMR ($CDCl_3$, 500 MHz): δ 3.23 (s, 3H, Me); 7.36-7.42, 7.48-7.54 (all m, 10H, 2 Ph). ^{3}C NMR ($CDCl_3$, 125 MHz): δ 38.84-38.88 (m, Me); 106.39-115.55 (m, 3CF_2); 117.25 (qt, *J* 288.3, 33.2 Hz, CF_3); 121.92 (═C—Ph); 124.69, 127.81, 127.96, 128.34, 128.36, 129.45, 130.09 (2 Ph); 139.25 (t, *J* 27.8 Hz, C—CF_2); 162.67 (C═O). ^{19}F NMR ($CDCl_3$, 470 MHz): δ 35.84-35.93, 39.90-39.97, 55.86-55.93 (all m, 3CF_2), 80.92 (t, *J* 9.8 Hz, 3F, CF_3). IR: 1669 (C═O), 1458, 1498, 1593, 1626 (C═C), 1130-1239 (CF) cm^{-1}. Anal. calcd for $C_{20}H_{13}F_9N_2O$: C, 51.29; H, 2.80; N, 5.98.Found: C, 51.29; H, 2.88; N, 5.91.

Synthesis of 1-methyl-2-phenyl-4-(phenylethynyl)-5-(polyfluoroalkyl)-1,2-dihydro-3*H*-pyrazolon-3-ones 6a,b.

A screw-neck vial was flushed with argon and charged with 4-iodo-5-(polyfluoroalkyl)-antipyrine **5a,b** (1 mmol), phenylacetylene (1.5 mmol, 153 mg), XPhos (0.04 mmol, 19 mg), $Pd_2(dba)_3$ (0.02 mmol, 18 mg), CuI (0.1 mmol, 19 mg), DIPEA (2 mmol, 258 mg), and MeCN (4 ml). The vial was repeatedly flushed with argon, closed tightly and a mixture was stirring at the room temperature for 72 h. After cooling, the solvent was evaporated in vacuo and the crosslinking product - 1,4-diphenyl-1,3-butadiyne was removed by flash-chromatography on silica gel (eluent – $CHCl_3$). Then, the product **6a** was purified by column chromatography on silica gel using a mixture of *n*-hexane and EtOAc (4:1) or the compound **6b** was re-crystallized from a mixture of *n*-hexane and EtOAc (4:1).

1-Methyl-2-phenyl-4-(phenylethynyl)-5-(trilfuoromethyl)-1,2-dihydro-3*H*-pyrazolon-3-one (6a). Yield 60 %, brown crystals, mp 124-125 °C. ^{1}H NMR ($CDCl_3$, 500 MHz): δ 3.27 (s, 3H, Me); 7.33-7.34, 7.41-7.43, 7.51-7.55 (all m, 10H, 2 Ph). ^{13}C NMR ($CDCl_3$, 125

MHz): δ 37.27-37.28 (m, Me); 76.45, 97.75 (–C≡C–); 99.95 (unsolv. q, C^4); 119.22 (q, *J* 272.9 Hz, CF_3); 122.59, 125.40, 128.25, 128.54, 128.73, 129.63, 131.78, 131.74 (2 Ph); 142.83 (q, *J* 37.2 Hz, C—CF_3); 161.85 (C═O). ^{19}F NMR ($CDCl_3$, 470 MHz): δ 100.83 (s, CF_3). IR: 1678 (C═O); 1416, 1457, 1585 (C═C); 1118-1174 (CF) cm^{-1}. Anal. calcd for $C_{19}H_{13}F_3N_2O$: C, 66.67; H, 3.83; N, 6.76. Found: C, 66.66; H, 3.86; N, 7.91.

1-Methyl-5-(pentafluoroethyl)-2-phenyl-4-(phenylethynyl)-1,2-dihydro-3*H*-pyrazolon-3-one (6b). Yield 65 %, brown crystals, mp 129-130 °C. ^{1}H NMR ($CDCl_3$, 500 MHz): δ 3.26 (s, 3H, Me); 7.33, 7.40-7.44, 7.53 (all m, 10H, 2 Ph). ^{13}C NMR ($CDCl_3$, 125 MHz): δ 37.85-37.93 (m, Me); 76.62, 97.92 (–C≡C–); 102.72 (unsolv. t, C^4); 110.15 (tq, *J* 258.2, 41.2 Hz, CF_2); 118.16 (qt, *J* 287.5, 37.1 Hz, CF_3); 122.66, 125.65, 128.24, 128.67, 128.73, 129.64, 131.76, 132.77 (2 Ph); 141.45 (t, *J* 27.0 Hz, C—CF_2); 161.75 (C═O). ^{19}F NMR ($CDCl_3$, 470 MHz): δ 49.24 (br.s, 2F, CF_2); 100.83 (br.s, 3F, CF_3). IR: 1677 (C═O); 1574, 1589, 1488 (C═C); 1207-1157 (CF) cm^{-1}. Anal. calcd for $C_{20}H_{13}F_5N_2O$. C, 61.23; H, 3.34; N, 7.14. Найдено (%): C, 61.35; H, 3.23; N, 7.10.

Biological testing

Viruses and cells. We used the transplantable Vero cell cultures (green monkey kidney cells) obtained from the collection of cell cultures of the Vector Institute (Koltsovo, Novosibirsk region) and MDCK (ATCC CCL-34) from the collection of cell lines of the Saint Petersburg Pasteur Institute of Epidemiology and Microbiology. Vero cells were cultured in 96-well culture plates in DMEM medium with additive of 2% fetal bovine serum («HyClone», USA), 40 U/ml of gentamicin sulfate, and 2.5 U/ml of amphotericin B. MDCK cells were cultured in MEM medium with additive of 10% fetal bovine serum («HyClone», USA), 40 U/ml gentamicin sulfate and 2.5 U/ml amphotericin B. Cell suspension with a concentration of 105 cells/ml was placed in the wells of the plates in volume of 100 μl and cultured until a complete monolayer formation for 24 h at 37 °C in the presence of 5% CO_2.

The same medium without serum was used as a support medium for culturing cells with viruses.

We used influenza virus A/Puerto Rico/8/34 (H1N1) from the collection of the Saint Petersburg Pasteur Institute of Epidemiology and Microbiology, and vaccine virus (VV, strain Copenhagen), herpes simplex virus type 1 (HSV-1, strain VR-3) and bovine diarrhea virus (BDV) (strain VC-1) obtained from the collection of the Vector Institute (Koltsovo, Novosibirsk region). The infectious titers of viruses were determined by titration in 96-well plates with monolayers of the corresponding line cells [4]. The results were evaluated visually according to the presence of the virus cytopathic action, the virus titer was calculated by the Spearman-Kerber method and represented in decimal logarithms of 50% tissue cytopathic doses in ml (lg TCD_{50}/ml) [5].

Evaluation of cytotoxic properties of compounds

The investigation of toxicity of compounds was carried out based on evaluation of the cells viability using the reduction reaction of the tetrazolium dye MTT (3-(4,5-dimethyl-2-thiazolyl)-2,5-diphenyl-2*H*-tetrazolium bromide) by cells in culture. Its intensity shows the degree of cell viability as a result of dye reduction by mitochondrial and partially cytoplasmic dehydrogenases.

The test compounds in the concentration range of 4-1000 μg/ml dissolved in the medium for cell cultivation were added to the plate wells in a volume of 200 μl and incubated for 72 h at 36 °C in an atmosphere of 5% CO_2. At the end of the incubation period, the cells were washed with MEM medium, and 100 μl of a solution (0.5 mg/ml) of MTT in the cell medium was added to the plate wells. The cells were incubated at 36 °C in an atmosphere of 5% CO_2 for 2 h and washed for 5 min with saline. The precipitate was dissolved in 100 μl of DMSO per well, and the optical density was measured using a Multiscan FC plate analyzer (Thermo Scientific) at a wavelength of 540 nm. Based on the obtained data, the 50% cytotoxic concentration (CC_{50}) was calculated, i.e. the concentration of the compound, which reduces the optical density in the wells by half compared to control cells without drugs.

Evaluation of antiviral activity of compounds

Determination of activity in relation to influenza virus A/Puerto Rico/8/34 (H1N1)

The compounds in appropriate concentrations were added to MDCK cells (0.1 ml per well). After 1 h of incubation, cells were infected with influenza virus A/Puerto Rico/8/34 (H1N1) (moi 0.01) and incubated for 72 h at 36 °C and 5% CO_2. After that, cell viability was assessed by MTT test as described in [6]. The cytoprotective activity of compounds was considered as their ability to increase the values of OD comparing to control wells (with virus only, no drugs). Based on the results obtained, the values of IC_{50}, i.e. concentration of compounds that result in 50% cells protection were calculated using GraphPad Prism 6.01 software. Values of IC_{50} obtained in μg/ml were then calculated into micromoles (μM). For each compound the value of selectivity index (SI) was calculated as ratio of CC_{50} to IC_{50}.

Determination of activity in relation to SVV, HSV-1, and VDC

The studied compounds were preliminarily dissolved in DMSO at a concentration of 20 mg/ml and stored in a freezer at –70 °C.

To test the antiviral activity of the compounds against VV, HSV-1, and BDV, a series of 5-fold dilutions of the samples were prepared (starting with a concentration of 400 μg/ml).

VV, HSV-1, and BDV were added to the plate wells at doses corresponding to a multiplicity of infection (moi) of 0.01. Viruses were cultured in cells in the presence of test compounds for 3 days. After this period, the cells viability in culture was assessed as described above. Based on the obtained data, the value of 50% inhibitory concentration (IC_{50}) was calculated,

i.e., the concentration of the compound leading to a 50% decrease in the cytodestructive effect of the virus.

Based on the CC_{50} and IC_{50} values, the selectivity index (SI) or the therapeutic index was calculated showing how many times the cytotoxic concentration is greater than the virus inhibiting concentration of the drug: $SI = CC_{50}/IC_{50}$.

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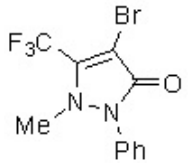
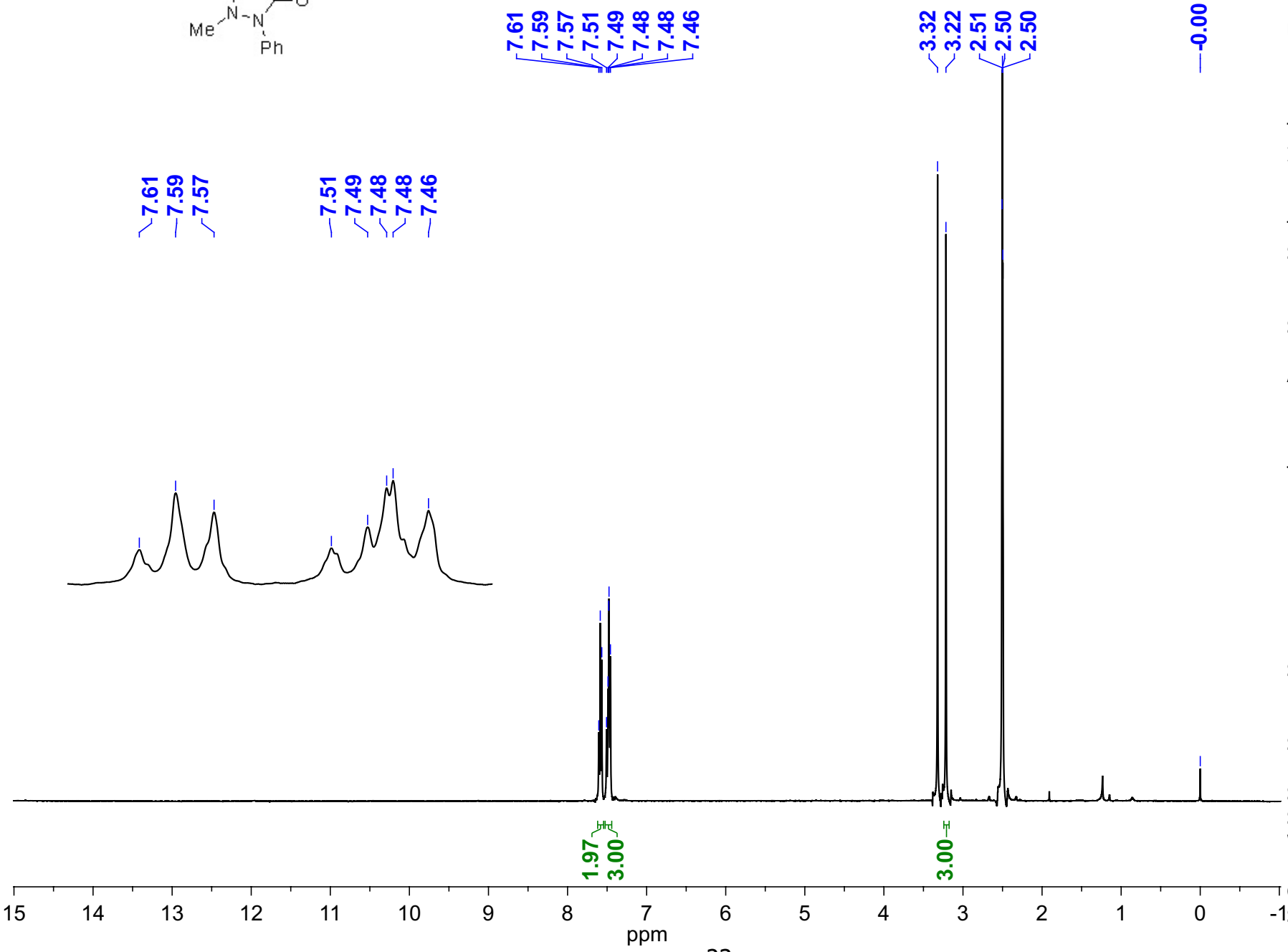
Copies of ^{1}H, ^{13}C, and ^{19}F NMR spectra of compounds

^{1}H NMR spectrum of compound **4a** in DMSO-d_6





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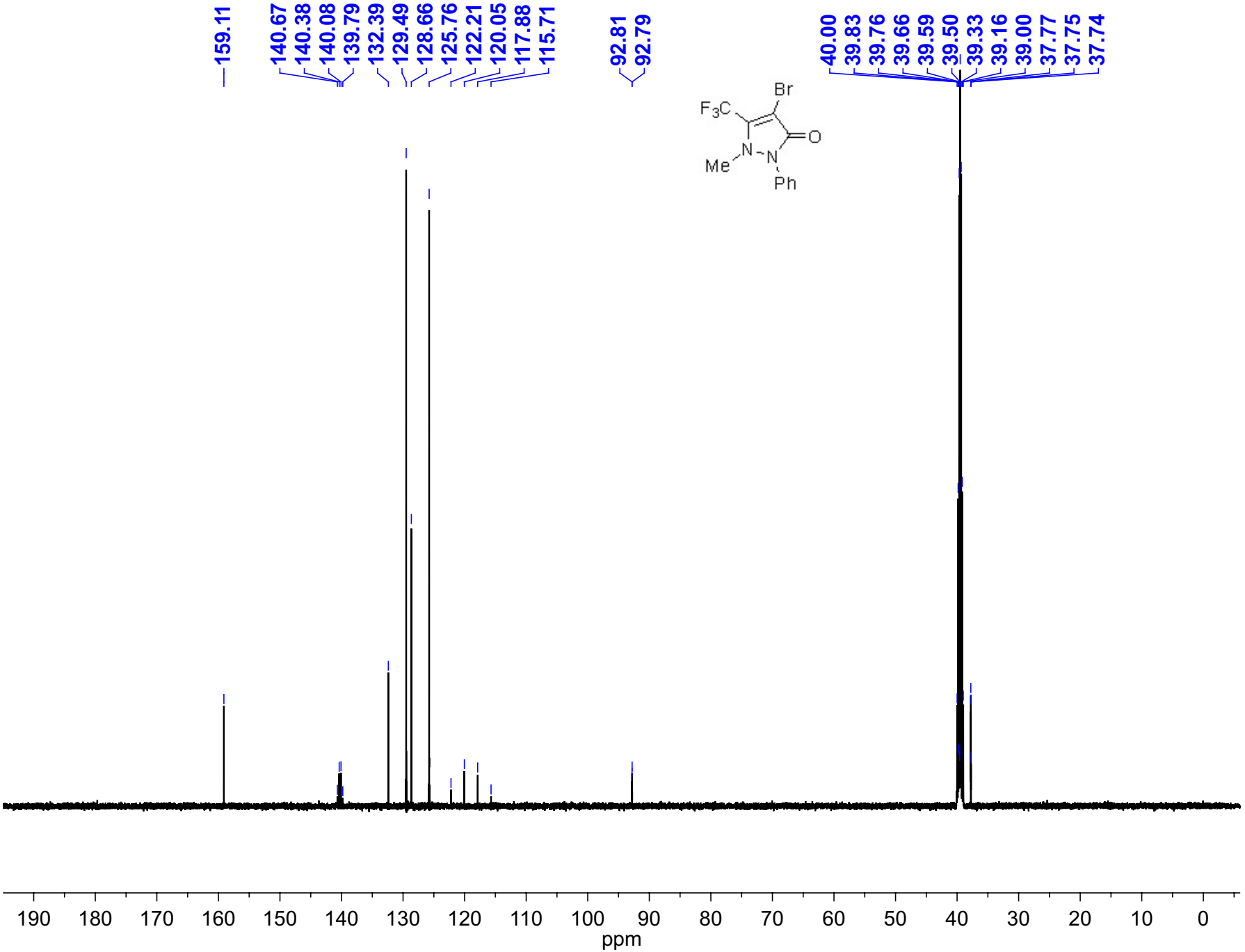
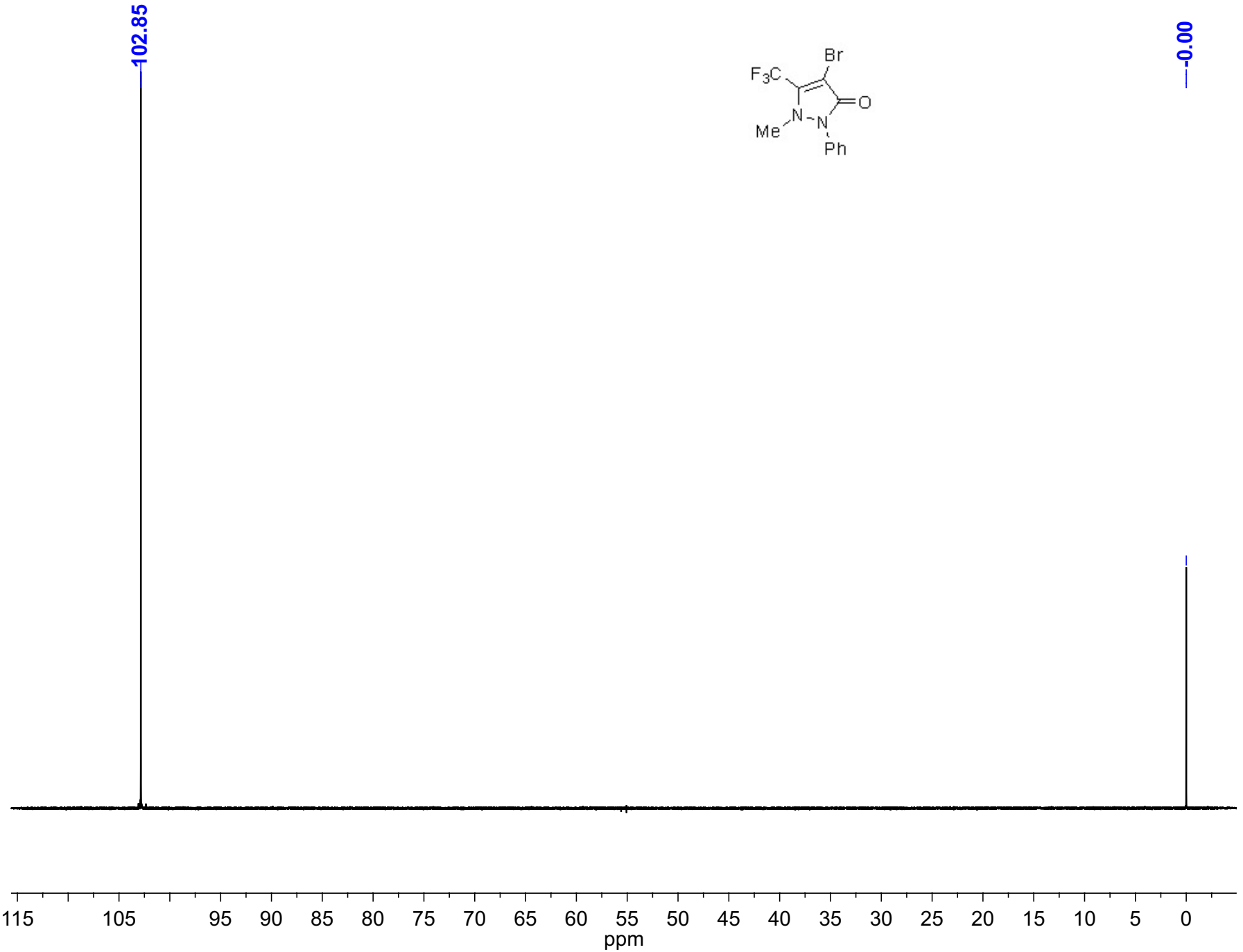
WDW EM
LB 0.00 Hz
GB 0
SSB 0
PC 4.00

^{13}C NMR spectrum of compound **4a** in DMSO-d_6



NAME	NNA304
EXPNO	13
PROCNO	1
USER	uralnmr
Date_	20190123
Time	13.27
INSTRUM	AV500
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
SOLVENT	DMSO
TD	32768
SW	200.7838 ppm
O1P	95.000 ppm
FIDRES	0.770646 Hz
NS	1024
DS	8
AQ	0.6488564 sec
RG	203
TE	296.9 K
DE	6.50 usec
D1	0.80000001 sec
D11	0.03000000 sec
TD0	1
==== CHANNEL f1 =====	
NUC1	13C
P1	10.00 usec
PL1	0.00 dB
PL1W	115.29558563 W
SFO1	125.7697360 MHz
==== CHANNEL f2 ======	
CPDPRG2	waltz16
NUC2	1H
PCPD2	75.00 usec
PL2	120.00 dB
PL12	16.30 dB
PL13	19.30 dB
PL2W	0.00000000 W
PL12W	0.47519693 W
PL13W	0.23816262 W
SFO2	500.1320005 MHz
SI	32768
HZpPT	0.770646 Hz
SR	63.68 Hz
WDW	EM
LB	0.00 Hz
GB	0
SSB	0

^{19}F NMR spectrum of compound **4a** in DMSO-d_6

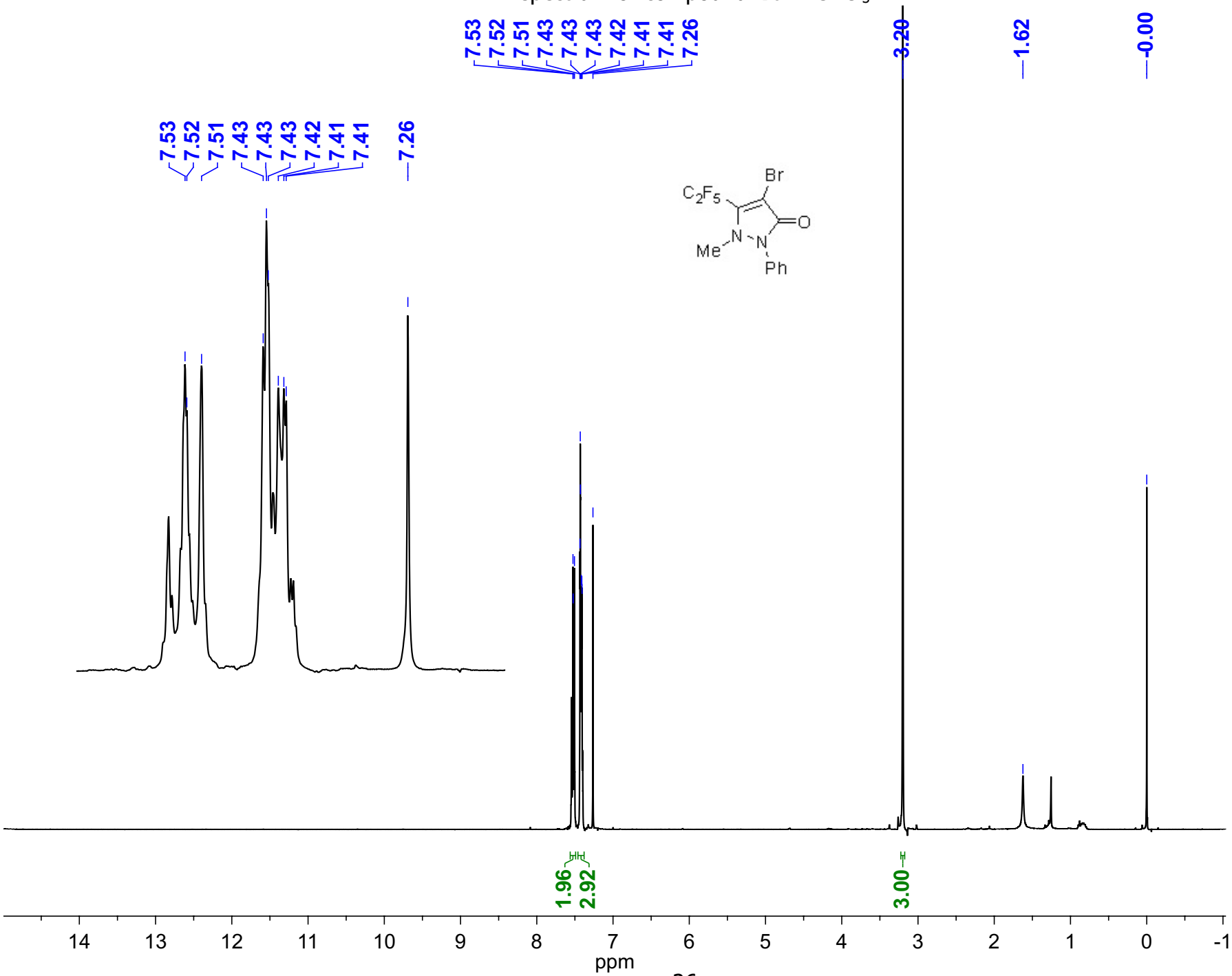


Current Data Parameters
NAME NNA304
EXPNO 19
PROCNO 1
USER uralnmr

F2 - Acquisition Parameters
Date_ 20181212
Time 13.15
INSTRUM DRX400
PROBHD 5 mm SEF 19F-1
PULPROG zg30
TD 131072
SOLVENT DMSO
NS 8
DS 2
SWH 45351.473 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 4597.6
DW 11.025 usec
DE 6.50 usec
TE 297.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
==== CHANNEL f1 =====
NUC1 19F
P1 10.10 usec
PL1 0.00 dB
SFO1 376.4579891 MHz

F2 - Processing parameters
SI 131072
HZpPT 0.346004 Hz
SF 376.4371603 MHz
SR -124.73 Hz
WDW EM
LB 0.00 Hz
GB 0
SSB 0
PC 4.00

^{1}H NMR spectrum of compound **4b** in $CDCl_3$



Current Data Parameters

NAME	ESh705a
EXPNO	1
PROCNO	1
USER	uralnmr

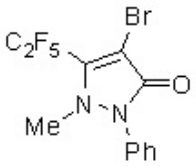
F2 - Acquisition Parameters

Date_	20191219
Time	13.31
INSTRUM	DRX400
PROBHD	5 mm SEF 19F-1
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	6410.256 Hz
FIDRES	0.195625 Hz
AQ	2.5559540 sec
RG	512
DW	78.000 usec
DE	6.50 usec
TE	298.2 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec
===== CHANNEL f1 ======	
NUC1	1H
P1	20.00 usec
PL1	0.00 dB
SFO1	400.1328009 MHz

F2 - Processing parameters

SI	32768
HZpPT	0.195625 Hz
SF	400.1300087 MHz
SR	8.72 Hz
WDW	EM
LB	0.00 Hz
GB	0
SSB	0
PC	4.00

^{13}C NMR spectrum of compound **4b** in $CDCl_3$



160.09 140.89 140.67 140.46 132.84 129.64 128.59 125.11 119.81 119.51 119.22 117.52 117.23 110.14 109.81 98.74 77.25 77.00 76.75 38.63 38.62 -0.06

140.89 140.67 140.46

122.09 121.80 121.50 119.81 119.51 119.22 117.52 117.23 116.93 114.94 112.20 111.87 110.47 110.14 109.81 109.49 108.09 107.76

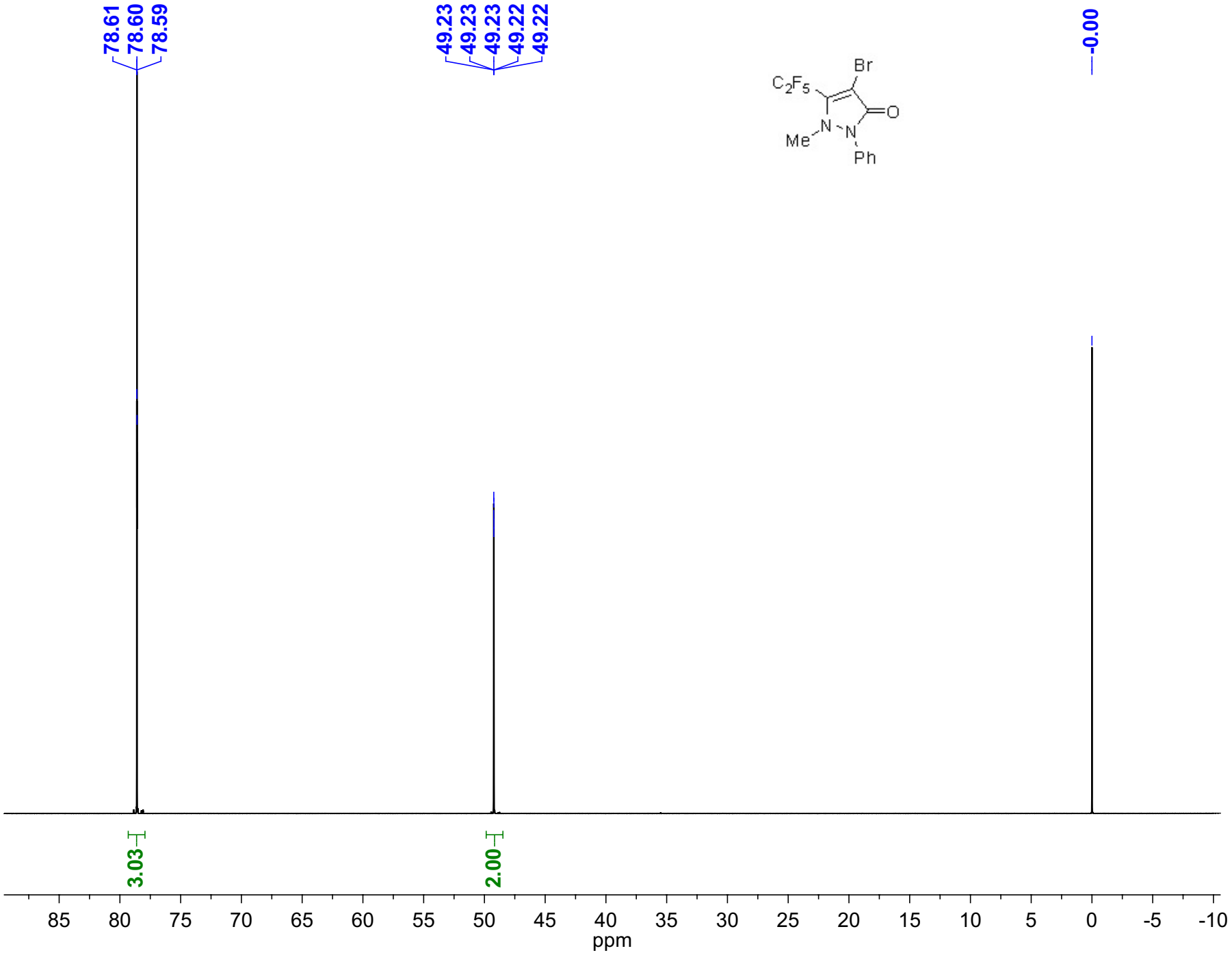
B (qt)
118.37
J(36.95, 287.49)

A (tq)
109.98
J(41.28, 258.47)

190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
ppm

NAME	ESh705a
EXPNO	13
PROCNO	1
USER	uralnmr
Date_	20191225
Time	19.36
INSTRUM	AV500
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
SOLVENT	CDCl3
TD	65536
SW	200.7838 ppm
O1P	95.000 ppm
FIDRES	0.385323 Hz
NS	16384
DS	8
AQ	1.2976629 sec
RG	203
TE	295.8 K
DE	6.50 usec
D1	1.00000000 sec
D11	0.03000000 sec
TD0	16
==== CHANNEL f1 ======	
NUC1	13C
P1	10.00 usec
PL1	0.00 dB
PL1W	115.29558563 W
SFO1	125.7697360 MHz
===== CHANNEL f2 =====	
CPDPRG2	waltz16
NUC2	1H
PCPD2	75.00 usec
PL2	120.00 dB
PL12	16.30 dB
PL13	19.30 dB
PL2W	0.00000000 W
PL12W	0.47519693 W
PL13W	0.23816262 W
SFO2	500.1320005 MHz
SI	65536
HZpPT	0.385323 Hz
SR	2.23 Hz
WDW	EM
LB	1.00 Hz
GB	0
SSB	0

^{19}F NMR spectrum of compound **4b** in $CDCl_3$



Current Data Parameters

NAME	ESh705a
EXPNO	19
PROCNO	1
USER	uralnmr

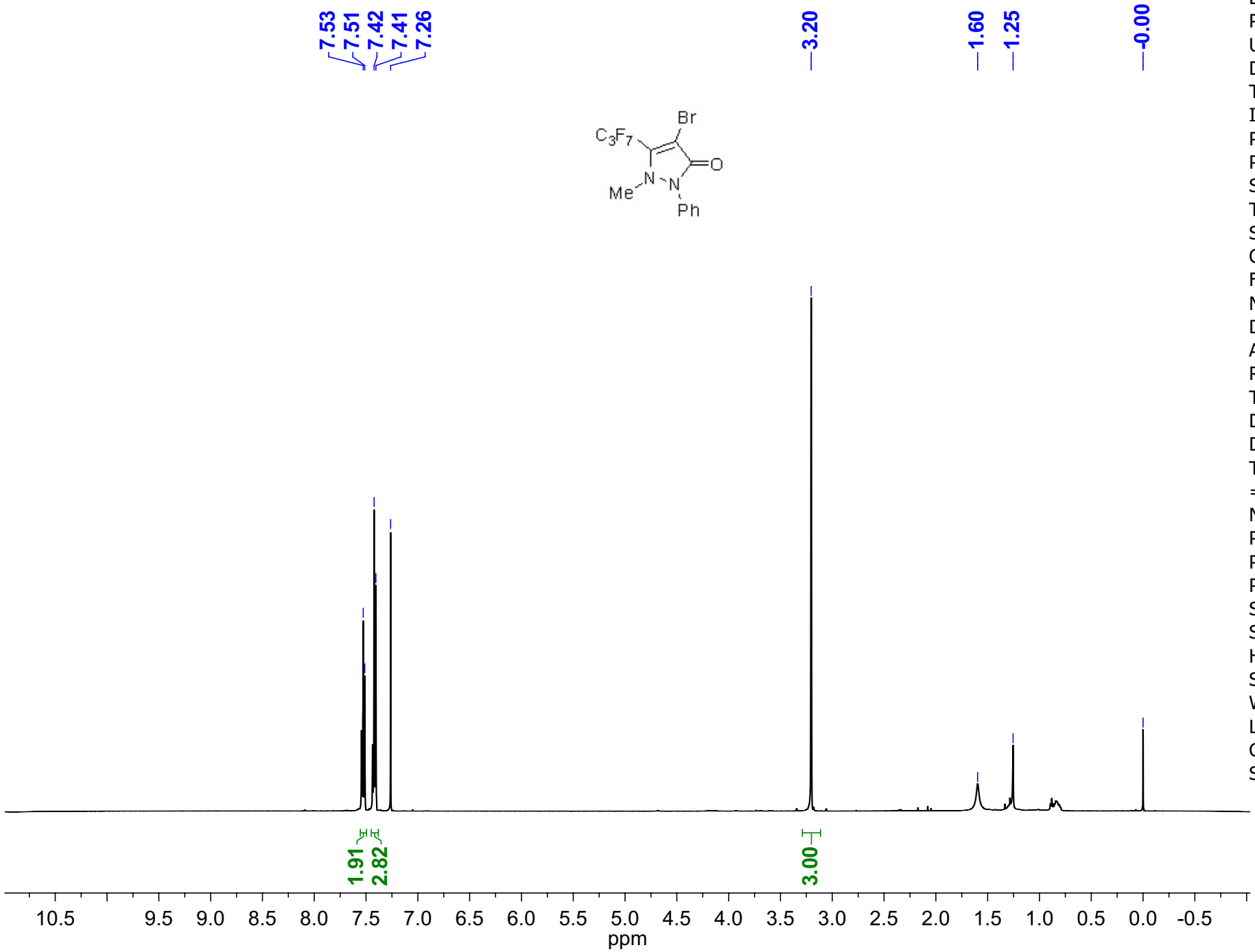
F2 - Acquisition Parameters

Date_	20191219
Time	13.40
INSTRUM	DRX400
PROBHD	5 mm SEF 19F-1
PULPROG	zg30
TD	262144
SOLVENT	CDCl3
NS	16
DS	2
SWH	37664.785 Hz
FIDRES	0.143680 Hz
AQ	3.4800117 sec
RG	912.3
DW	13.275 usec
DE	6.50 usec
TE	298.2 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec
===== CHANNEL f1 =====	
NUC1	19F
P1	10.10 usec
PL1	0.00 dB
SFO1	376.4523425 MHz

F2 - Processing parameters

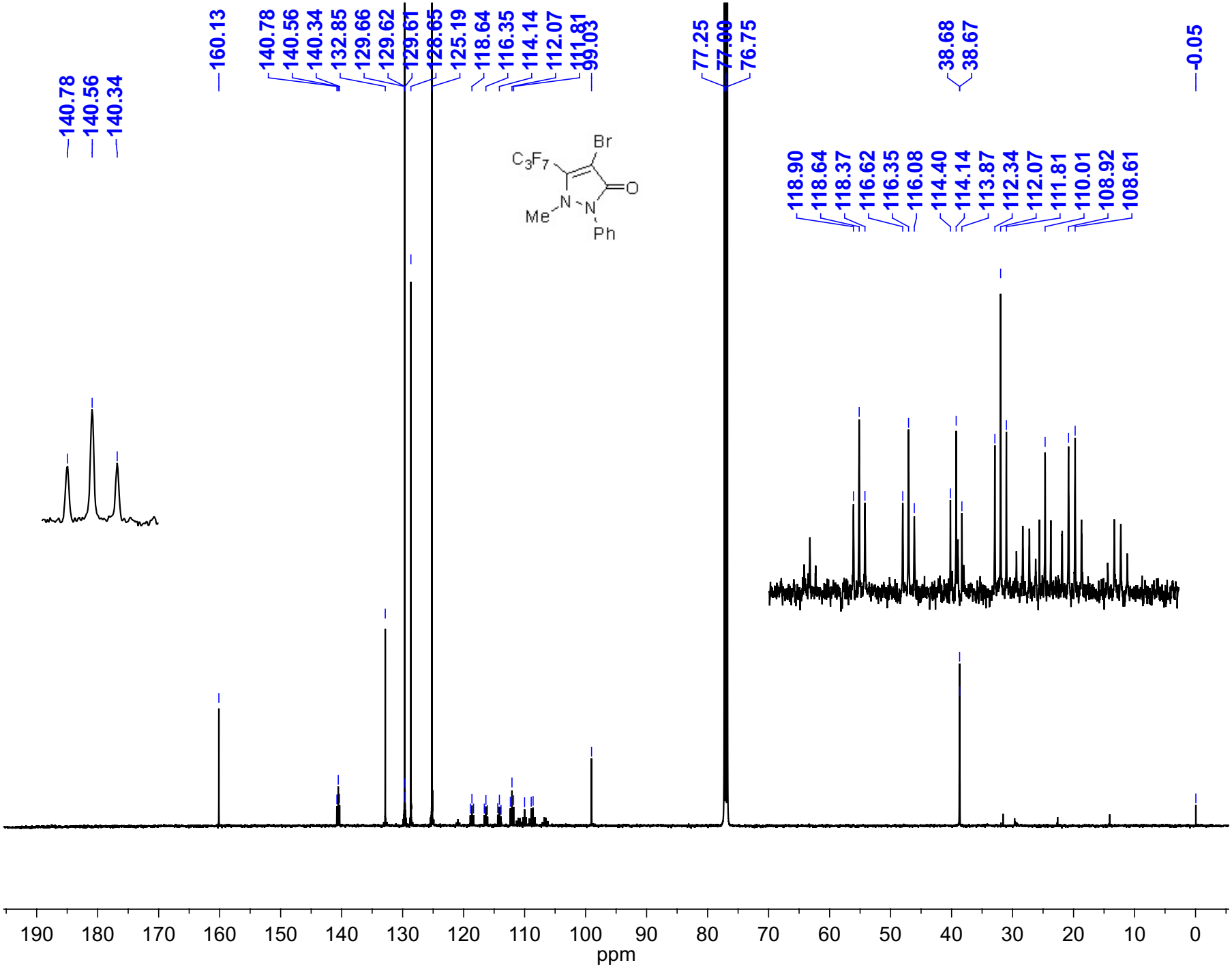
SI	131072
HZpPT	0.287360 Hz
SF	376.4374801 MHz
SR	195.11 Hz
WDW	EM
LB	0.20 Hz
GB	0
SSB	0
PC	3.00

^{1}H NMR spectrum of compound **4c** in $CDCl_3$



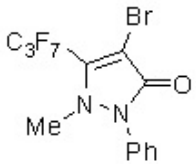
NAME	ESh706
EXPNO	1
PROCNO	1
USER	uralnmr
Date_	20200110
Time	14.21
INSTRUM	AV500
PROBHD	5 mm PABBO BB-
PULPROG	zg30
SOLVENT	CDCl3
TD	32768
SW	12.0160 ppm
O1P	5.000 ppm
FIDRES	0.183399 Hz
NS	16
DS	2
AQ	2.7263477 sec
RG	203
TE	295.6 K
DE	6.50 usec
D1	1.00000000 sec
TD0	1
==== CHANNEL f1 =====	
NUC1	1H
P1	12.00 usec
PL1	0.30 dB
PL1W	18.91792679 W
SFO1	500.1325007 MHz
SI	32768
HZpPT	0.183399 Hz
SR	11.57 Hz
WDW	EM
LB	0.00 Hz
GB	0
SSB	0

^{13}C NMR spectrum of compound **4c** in $CDCl_3$

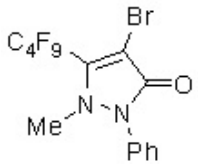


NAME	ESh706
EXPNO	13
PROCNO	1
USER	uralnmr
Date_	20200205
Time	18.28
INSTRUM	AV500
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
SOLVENT	CDCl3
TD	32768
SW	200.7838 ppm
O1P	95.000 ppm
FIDRES	0.770646 Hz
NS	28672
DS	8
AQ	0.6488564 sec
RG	203
TE	296.5 K
DE	6.50 usec
D1	1.00000000 sec
D11	0.03000000 sec
TD0	28
===== CHANNEL f1 =====	
NUC1	13C
P1	9.00 usec
PL1	0.00 dB
PL1W	115.29558563 W
SFO1	125.7697360 MHz
==== CHANNEL f2 =====	
CPDPRG2	waltz16
NUC2	1H
PCPD2	75.00 usec
PL2	120.00 dB
PL12	17.00 dB
PL13	20.00 dB
PL2W	0.00000000 W
PL12W	0.40445811 W
PL13W	0.20270923 W
SFO2	500.1320005 MHz
SI	32768
HZpPT	0.770646 Hz
SR	1.55 Hz
WDW	EM
LB	1.00 Hz
GB	0
SSB	0

^{19}F NMR spectrum of compound **4c** in $CDCl_3$



NAME	ESh706
EXPNO	19
PROCNO	1
USER	uralnmr
Date_	20200110
Time	14.26
INSTRUM	AV500
PROBHD	5 mm PABBO BB-
PULPROG	zg30
SOLVENT	CDCl3
TD	131072
SW	99.6202 ppm
O1P	45.000 ppm
FIDRES	0.357628 Hz
NS	16
DS	2
AQ	1.3981513 sec
RG	203
TE	295.6 K
DE	6.50 usec
D1	1.00000000 sec
TD0	1
===== CHANNEL f1 ====	
NUC1	19F
P1	15.50 usec
PL1	-5.00 dB
PL1W	46.07103729 W
SFO1	470.5370532 MHz
SI	131072
HZpPT	0.357628 Hz
SR	400.40 Hz
WDW	EM
LB	0.00 Hz
GB	0
SSB	0



^{1}H NMR spectrum of compound **4d** in $CDCl_3$

Current Data Parameters

NAME	ESh717
EXPNO	1
PROCNO	1
USER	uralnmr

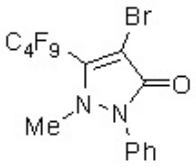
F2 - Acquisition Parameters

Date_	20200206
Time	12.38
INSTRUM	DRX400
PROBHD	5 mm SEF 19F-1
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	5592.841 Hz
FIDRES	0.170680 Hz
AQ	2.9295092 sec
RG	1149.4
DW	89.400 usec
DE	6.00 usec
TE	297.2 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec
==== CHANNEL f1 =====	
NUC1	1H
P1	20.00 usec
PL1	0.00 dB
SFO1	400.1324008 MHz

F2 - Processing parameters

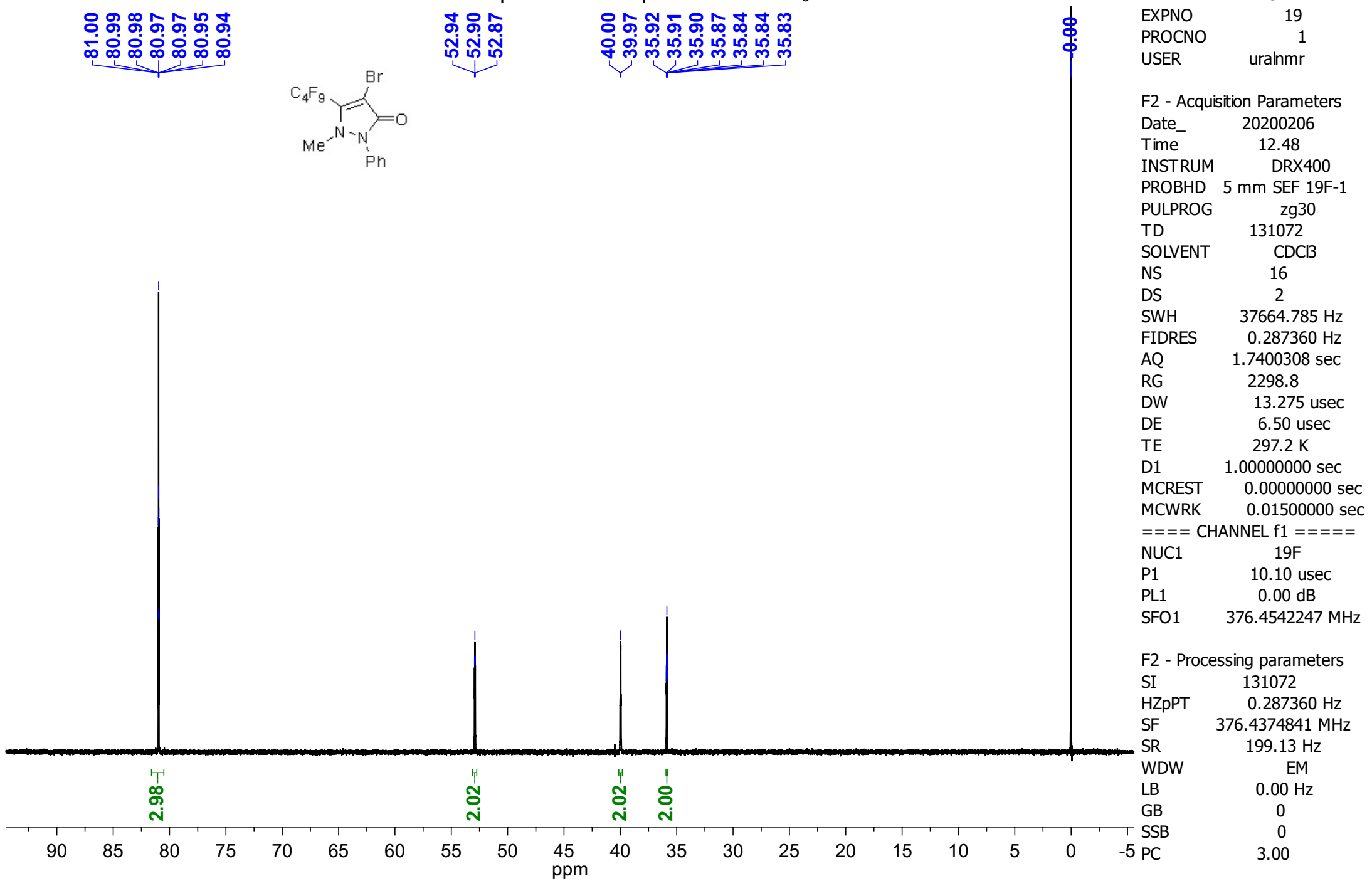
SI	32768
HZpPT	0.170680 Hz
SF	400.1300091 MHz
SR	9.05 Hz
WDW	EM
LB	0.00 Hz
GB	0
SSB	0
PC	4.00

^{13}C NMR spectrum of compound **4d** in $CDCl_3$



NAME	ESh717
EXPNO	13
PROCNO	1
USER	uralnmr
Date_	20200213
Time	17.13
INSTRUM	AV500
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
SOLVENT	CDCl3
TD	32768
SW	200.7838 ppm
O1P	95.000 ppm
FIDRES	0.770646 Hz
NS	34816
DS	8
AQ	0.6488564 sec
RG	203
TE	295.7 K
DE	6.50 usec
D1	1.00000000 sec
D11	0.03000000 sec
TD0	34
==== CHANNEL f1 =====	
NUC1	13C
P1	9.00 usec
PL1	0.00 dB
PL1W	115.29558563 W
SFO1	125.7697360 MHz
==== CHANNEL f2 ======	
CPDPRG2	waltz16
NUC2	1H
PCPD2	75.00 usec
PL2	120.00 dB
PL12	17.00 dB
PL13	20.00 dB
PL2W	0.00000000 W
PL12W	0.40445811 W
PL13W	0.20270923 W
SFO2	500.1320005 MHz
SI	32768
HZpPT	0.770646 Hz
SR	1.43 Hz
WDW	EM
LB	1.50 Hz
GB	0
SSB	0

^{19}F NMR spectrum of compound **4d** in $CDCl_3$

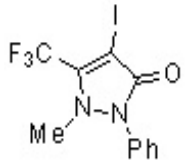


Current Data Parameters
NAME ESh717
EXPNO 19
PROCNO 1
USER uralnmr

F2 - Acquisition Parameters
Date_ 20200206
Time 12.48
INSTRUM DRX400
PROBHD 5 mm SEF 19F-1
PULPROG zg30
TD 131072
SOLVENT CDCl3
NS 16
DS 2
SWH 37664.785 Hz
FIDRES 0.287360 Hz
AQ 1.7400308 sec
RG 2298.8
DW 13.275 usec
DE 6.50 usec
TE 297.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
==== CHANNEL f1 =====
NUC1 19F
P1 10.10 usec
PL1 0.00 dB
SFO1 376.4542247 MHz

F2 - Processing parameters
SI 131072
HZpPT 0.287360 Hz
SF 376.4374841 MHz
SR 199.13 Hz
WDW EM
LB 0.00 Hz
GB 0
SSB 0
PC 3.00

^{1}H NMR spectrum of compound **5a** in $CDCl_3$



Current Data Parameters

NAME	ESh811b
EXPNO	1
PROCNO	1
USER	uralnmr

F2 - Acquisition Parameters

Date_	20210416
Time	13.41
INSTRUM	DRX400
PROBHD	5 mm SEF 19F-1
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	6410.256 Hz
FIDRES	0.195625 Hz
AQ	2.5559540 sec
RG	1625.5
DW	78.000 usec
DE	16.00 usec
TE	297.2 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

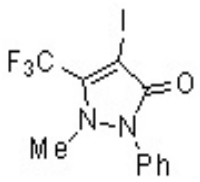
==== CHANNEL f1 ====

NUC1	1H
P1	32.50 usec
PL1	-4.00 dB
SFO1	400.1328009 MHz

F2 - Processing parameters

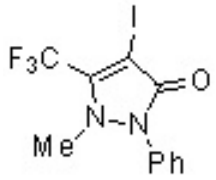
SI	32768
HZpPT	0.195625 Hz
SF	400.1300092 MHz
SR	9.16 Hz
WDW	EM
LB	0.00 Hz
GB	0
SSB	0
PC	4.00

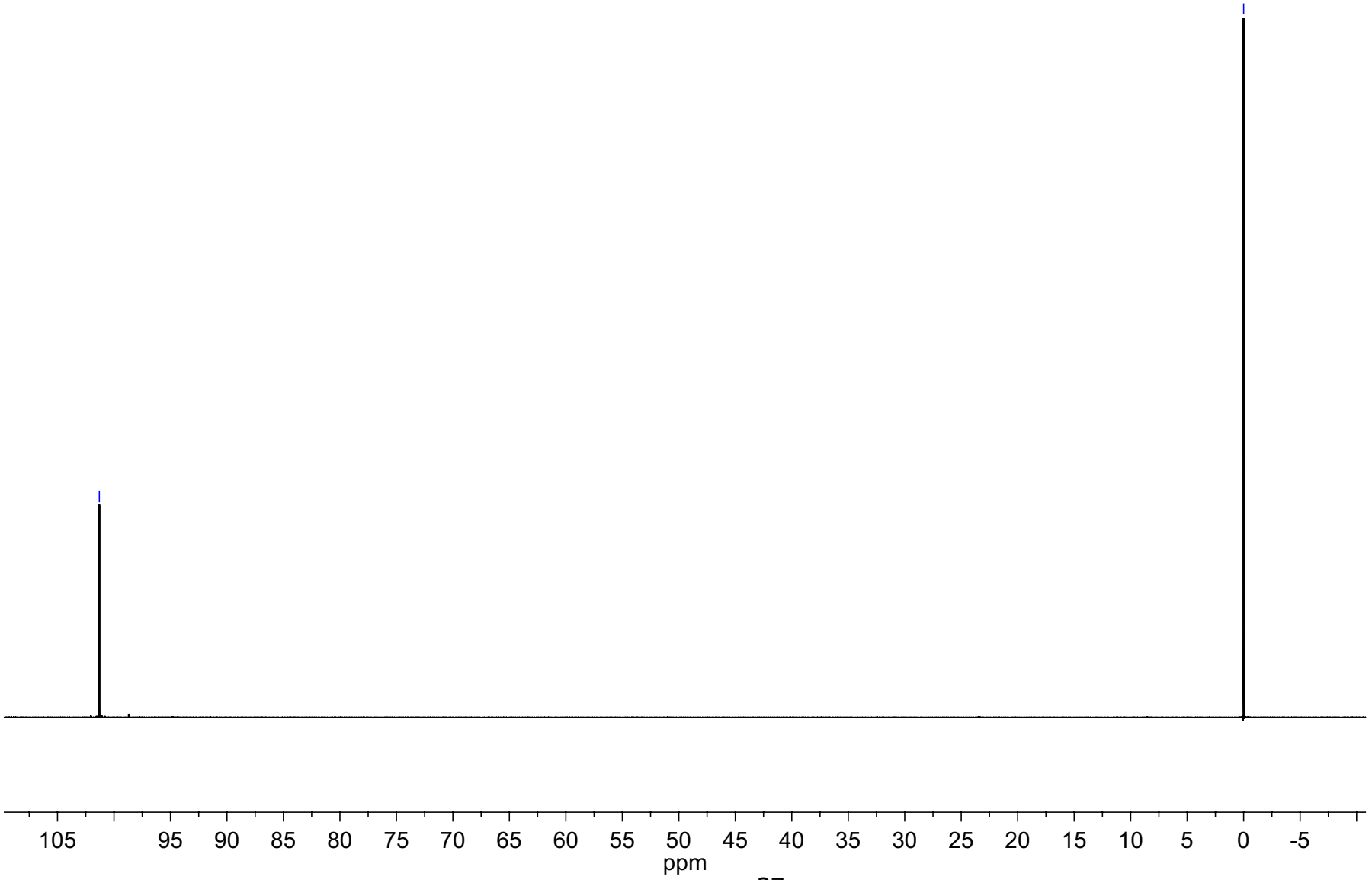
^{13}C NMR spectrum of compound **5a** in $CDCl_3$



NAME	ESh811b
EXPNO	13
PROCNO	1
USER	uralnmr
Date_	20210611
Time	14.23
INSTRUM	AV500
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
SOLVENT	CDCl3
TD	32768
SW	200.7838 ppm
O1P	95.000 ppm
FIDRES	0.770646 Hz
NS	1024
DS	8
AQ	0.6488564 sec
RG	203
TE	296.1 K
DE	6.50 usec
D1	1.00000000 sec
D11	0.03000000 sec
TD0	1
=== CHANNEL f1 =====	
NUC1	13C
P1	10.00 usec
PL1	0.00 dB
PL1W	115.29558563 W
SFO1	125.7697360 MHz
====CHANNEL f2 =====	
CPDPRG2	waltz16
NUC2	1H
PCPD2	75.00 usec
PL2	120.00 dB
PL12	16.30 dB
PL13	19.30 dB
PL2W	0.00000000 W
PL12W	0.47519693 W
PL13W	0.23816262 W
SFO2	500.1320005 MHz
SI	32768
HZpPT	0.770646 Hz
SR	5.28 Hz
WDW	EM
LB	1.00 Hz
GB	0
SSB	0

^{19}F NMR spectrum of compound **5a** in $CDCl_3$





Current Data Parameters

NAME	ESh811b
EXPNO	19
PROCNO	1
USER	uralnmr

F2 - Acquisition Parameters

Date_	20210416
Time	13.47
INSTRUM	DRX400
PROBHD	5 mm SEF 19F-1
PULPROG	zg30
TD	131072
SOLVENT	CDCl3
NS	16
DS	2
SWH	45351.473 Hz
FIDRES	0.346004 Hz
AQ	1.4451188 sec
RG	2048
DW	11.025 usec
DE	6.50 usec
TE	297.2 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

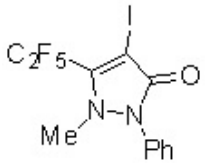
=== CHANNEL f1 =====

NUC1	19F
P1	20.25 usec
PL1	0.00 dB
SFO1	376.4561069 MHz

F2 - Processing parameters

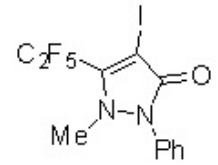
SI	131072
HZpPT	0.346004 Hz
SF	376.4374880 MHz
SR	202.99 Hz
WDW	EM
LB	0.01 Hz
GB	0
SSB	0
PC	4.00

^{1}H NMR spectrum of compound **5b** in $CDCl_3$



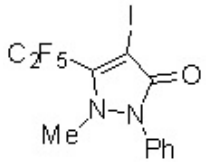
NAME	ESh752
EXPNO	1
PROCNO	1
USER	uralnmr
Date_	20210408
Time	15.42
INSTRUM	AV500
PROBHD	5 mm PABBO BB-
PULPROG	zg30
SOLVENT	CDCl3
TD	32768
SW	14.0019 ppm
O1P	6.000 ppm
FIDRES	0.213709 Hz
NS	16
DS	2
AQ	2.3396852 sec
RG	203
TE	296.4 K
DE	6.50 usec
D1	1.00000000 sec
TD0	1
==== CHANNEL f1 =====	
NUC1	1H
P1	12.00 usec
PL1	0.30 dB
PL1W	18.91792679 W
SFO1	500.1330008 MHz
SI	32768
HZpPT	0.213709 Hz
SR	12.73 Hz
WDW	EM
LB	0.00 Hz
GB	0
SSB	0

^{13}C NMR spectrum of compound **5b** in $CDCl_3$



NAME	ESh752
EXPNO	13
PROCNO	1
USER	uralnmr
Date_	20201105
Time	18.12
INSTRUM	AV500
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
SOLVENT	CDCl3
TD	32768
SW	200.7838 ppm
O1P	95.000 ppm
FIDRES	0.770646 Hz
NS	32768
DS	8
AQ	0.6488564 sec
RG	203
TE	296.9 K
DE	6.50 usec
D1	1.00000000 sec
D11	0.03000000 sec
TD0	32
===== CHANNEL f1 ====	
NUC1	13C
P1	9.00 usec
PL1	0.00 dB
PL1W	115.29558563 W
SFO1	125.7697360 MHz
=== CHANNEL f2 =====	
CPDPRG2	waltz16
NUC2	1H
PCPD2	75.00 usec
PL2	120.00 dB
PL12	17.00 dB
PL13	20.00 dB
PL2W	0.00000000 W
PL12W	0.40445811 W
PL13W	0.20270923 W
SFO2	500.1320005 MHz
SI	32768
HZpPT	0.770646 Hz
SR	1.28 Hz
WDW	EM
LB	1.00 Hz
GB	0
SSB	0

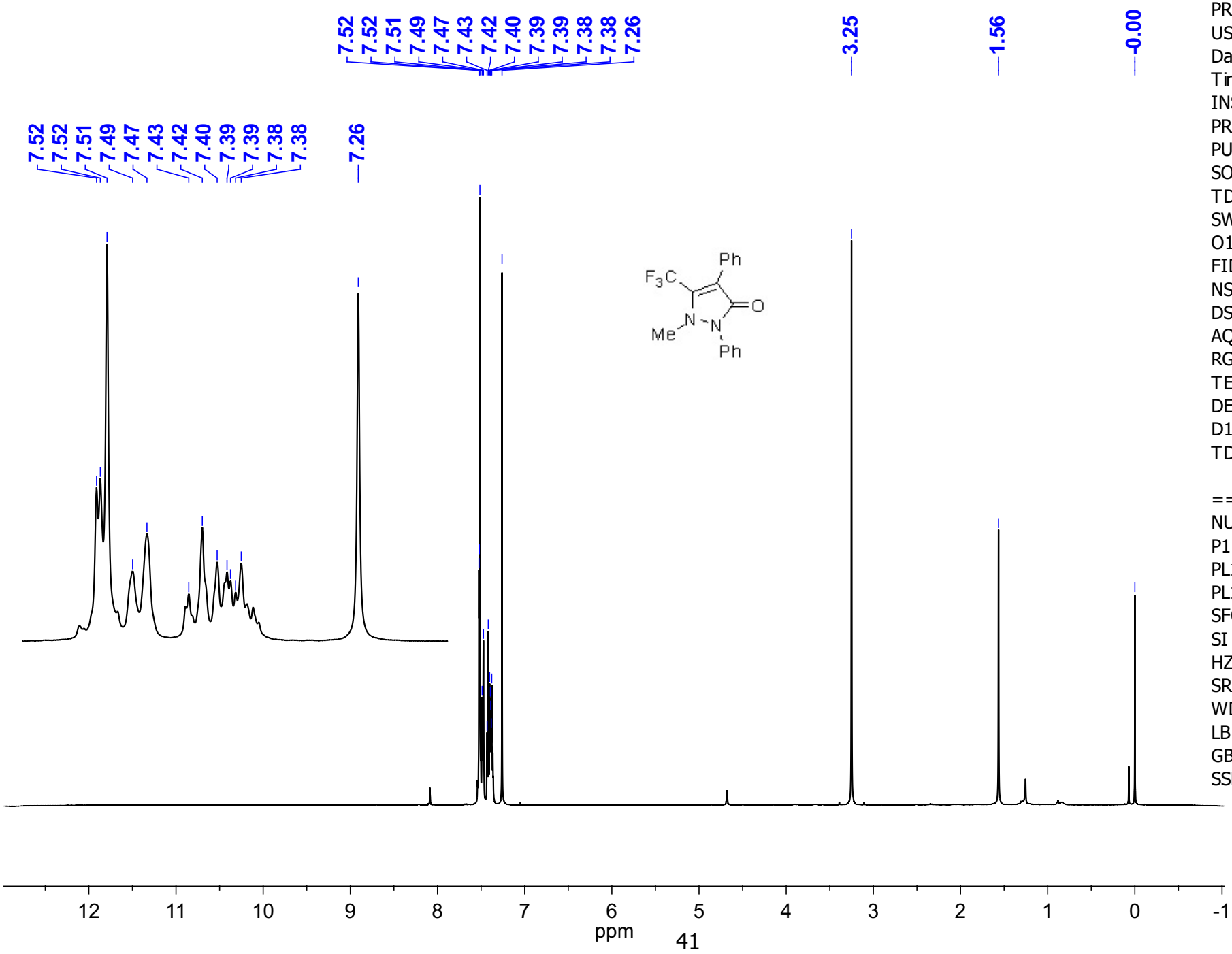
^{19}F NMR spectrum of compound **5b** in $CDCl_3$



78.96
78.95
78.95
50.34
50.33
0.00
3.20
2.00
90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 -5
ppm

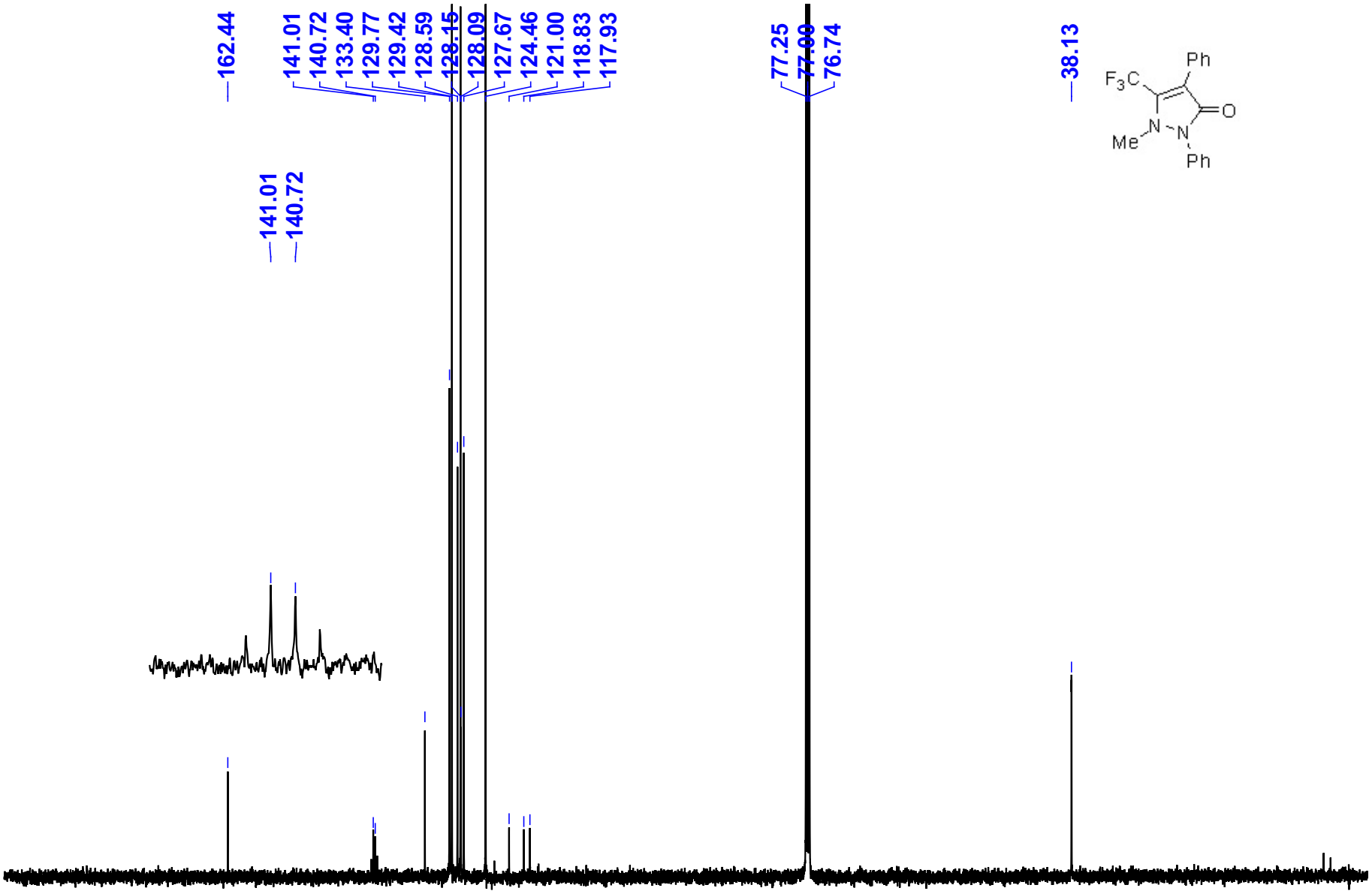
NAME	ESh752
EXPNO	19
PROCNO	1
USER	uralnmr
Date_	20210408
Time	15.46
INSTRUM	AV500
PROBHD	5 mm PABBO BB-
PULPROG	zg30
SOLVENT	CDCl3
TD	131072
SW	99.6202 ppm
O1P	45.000 ppm
FIDRES	0.357628 Hz
NS	16
DS	2
AQ	1.3981513 sec
RG	203
TE	296.4 K
DE	6.50 usec
D1	1.00000000 sec
TD0	1
=== CHANNEL f1 ====	
NUC1	19F
P1	15.50 usec
PL1	-5.00 dB
PL1W	46.07103729 W
SFO1	470.5370532 MHz
SI	131072
HZpPT	0.357628 Hz
SR	403.23 Hz
WDW	EM
LB	0.00 Hz
GB	0
SSB	0

^{1}H NMR spectrum of compound **3a** in $CDCl_3$



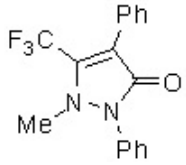
NAME	ESh695
EXPNO	1
PROCNO	1
USER	uralnmr
Date_	20190725
Time	13.25
INSTRUM	AV500
PROBHD	5 mm PABBO BB-
PULPROG	zg30
SOLVENT	CDCl3
TD	32768
SW	14.0019 ppm
O1P	6.000 ppm
FIDRES	0.213709 Hz
NS	16
DS	2
AQ	2.3396852 sec
RG	181
TE	295.8 K
DE	6.50 usec
D1	1.00000000 sec
TD0	1
====== CHANNEL f1 ======	
NUC1	1H
P1	12.00 usec
PL1	0.30 dB
PL1W	18.91792679 W
SFO1	500.1330008 MHz
SI	32768
HZpPT	0.213709 Hz
SR	13.62 Hz
WDW	EM
LB	0.00 Hz
GB	0
SSB	0

^{13}C NMR spectrum of compound **3a** in $CDCl_3$

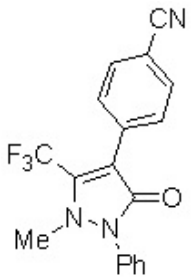


NAME	ESh695
EXPNO	13
PROCNO	1
USER	uralnmr
Date_	20190731
Time	10.39
INSTRUM	AV500
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
SOLVENT	CDCl3
TD	32768
SW	200.7838 ppm
O1P	95.000 ppm
FIDRES	0.770646 Hz
NS	1024
DS	8
AQ	0.6488564 sec
RG	203
TE	297.3 K
DE	6.50 usec
D1	1.00000000 sec
D11	0.03000000 sec
TD0	1
===== CHANNEL f1 ====	
NUC1	13C
P1	10.00 usec
PL1	0.00 dB
PL1W	115.29558563 W
SFO1	125.7697360 MHz
==== CHANNEL f2 ======	
CPDPRG2	waltz16
NUC2	1H
PCPD2	75.00 usec
PL2	0.30 dB
PL12	16.30 dB
PL13	19.30 dB
PL2W	18.91792679 W
PL12W	0.47519693 W
PL13W	0.23816262 W
SFO2	500.1320005 MHz
SI	65536
HZpPT	0.385323 Hz
SR	5.26 Hz
WDW	EM
LB	1.00 Hz
GB	0
SSB	0

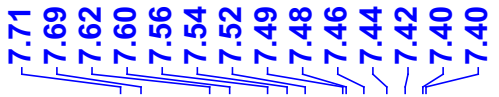
^{19}F NMR spectrum of compound **3a** in $CDCl_3$



NAME	ESh695
EXPNO	19
PROCNO	1
USER	uralnmr
Date_	20190725
Time	13.32
INSTRUM	AV500
PROBHD	5 mm PABBO BB-
PULPROG	zg30
SOLVENT	CDCl3
TD	131072
SW	120.7506 ppm
O1P	55.000 ppm
FIDRES	0.433488 Hz
NS	16
DS	2
AQ	1.1534836 sec
RG	203
TE	295.8 K
DE	6.50 usec
D1	1.00000000 sec
TD0	1
===== CHANNEL f1 =====	
NUC1	19F
P1	15.50 usec
PL1	-5.00 dB
PL1W	46.07103729 W
SFO1	470.5417584 MHz
SI	131072
HZpPT	0.433488 Hz
SR	405.65 Hz
WDW	EM
LB	0.00 Hz
GB	0
SSB	0



^{1}H NMR spectrum of compound **3c** in $CDCl_3$



Current Data Parameters

NAME	ESh788c
EXPNO	1
PROCNO	1
USER	uralnmr

F2 - Acquisition Parameters

Date_	20210421
Time	14.57
INSTRUM	DRX400
PROBHD	5 mm SEF 19F-1
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	6410.256 Hz
FIDRES	0.195625 Hz
AQ	2.5559540 sec
RG	228.1
DW	78.000 usec
DE	16.00 usec
TE	297.2 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

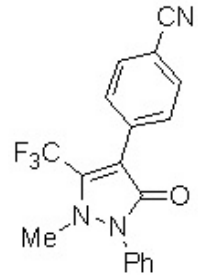
=== CHANNEL f1 ====

NUC1	1H
P1	32.50 usec
PL1	-4.00 dB
SFO1	400.1328009 MHz

F2 - Processing parameters

SI	32768
HZpPT	0.195625 Hz
SF	400.1300086 MHz
SR	8.58 Hz
WDW	EM
LB	0.00 Hz
GB	0
SSB	0
PC	3.00

^{19}F NMR spectrum of compound **3c** in $CDCl_3$



Current Data Parameters

NAME	ESh788c
EXPNO	19
PROCNO	1
USER	uralnmr

F2 - Acquisition Parameters

Date_	20210421
Time	15.04
INSTRUM	DRX400
PROBHD	5 mm SEF 19F-1
PULPROG	zg30
TD	262144
SOLVENT	CDCl3
NS	16
DS	2
SWH	49019.609 Hz
FIDRES	0.186995 Hz
AQ	2.6739187 sec
RG	724.1
DW	10.200 usec
DE	6.50 usec
TE	297.2 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

==== CHANNEL f1 =====

NUC1	19F
P1	20.25 usec
PL1	0.00 dB
SFO1	376.4579891 MHz

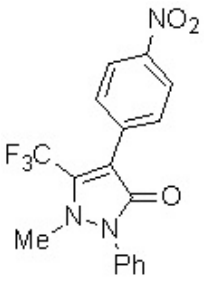
F2 - Processing parameters

SI	262144
HZpPT	0.186995 Hz
SF	376.4374830 MHz
SR	198.00 Hz
WDW	EM
LB	0.00 Hz
GB	0
SSB	0
PC	4.00

^{1}H NMR spectrum of compound **3d** in $CDCl_3$

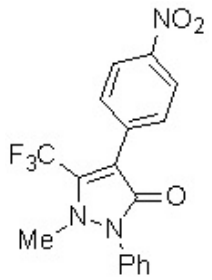
NAME	ESh828
EXPNO	1
PROCNO	1
USER	uralnmr
Date_	20210903
Time	14.04
INSTRUM	AV500
PROBHD	5 mm PABBO BB-
PULPROG	zg30
SOLVENT	CDCl3
TD	32768
SW	16.0214 ppm
O1P	7.000 ppm
FIDRES	0.244532 Hz
NS	16
DS	2
AQ	2.0447731 sec
RG	181
TE	296.0 K
DE	6.50 usec
D1	1.00000000 sec
TD0	1
===== CHANNEL f1 =====	
NUC1	1H
P1	12.00 usec
PL1	0.30 dB
PL1W	18.91792679 W
SFO1	500.1335009 MHz
SI	32768
HZpPT	0.244532 Hz
SR	11.22 Hz
WDW	EM
LB	0.00 Hz
GB	0
SSB	0

^{13}C NMR spectrum of compound **3d** in $CDCl_3$



NAME	ESh828
EXPNO	13
PROCNO	1
USER	uralnmr
Date_	20210903
Time	14.12
INSTRUM	AV500
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
SOLVENT	CDCl3
TD	32768
SW	200.7838 ppm
O1P	95.000 ppm
FIDRES	0.770646 Hz
NS	3072
DS	8
AQ	0.6488564 sec
RG	203
TE	296.0 K
DE	6.50 usec
D1	0.85000002 sec
D11	0.03000000 sec
TD0	1
==== CHANNEL f1 ====	
NUC1	13C
P1	10.00 usec
PL1	0.00 dB
PL1W	115.29558563 W
SFO1	125.7697360 MHz
==== CHANNEL f2 =====	
CPDPRG2	waltz16
NUC2	1H
PCPD2	75.00 usec
PL2	120.00 dB
PL12	16.30 dB
PL13	19.30 dB
PL2W	0.00000000 W
PL12W	0.47519693 W
PL13W	0.23816262 W
SFO2	500.1320005 MHz
SI	65536
HZpPT	0.385323 Hz
SR	1.37 Hz
WDW	EM
LB	1.00 Hz
GB	0
SSB	0

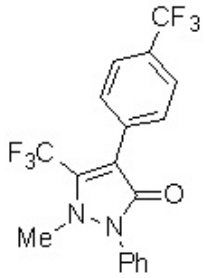
^{19}F NMR spectrum of compound **3d** in $CDCl_3$



NAME	ESh828
EXPNO	19
PROCNO	1
USER	uralnmr
Date_	20210903
Time	14.09
INSTRUM	AV500
PROBHD	5 mm PABBO BB-
PULPROG	zg30
SOLVENT	CDCl3
TD	131072
SW	120.7512 ppm
O1P	50.000 ppm
FIDRES	0.433488 Hz
NS	16
DS	2
AQ	1.1534836 sec
RG	203
TE	296.0 K
DE	6.50 usec
D1	1.00000000 sec
TD0	1

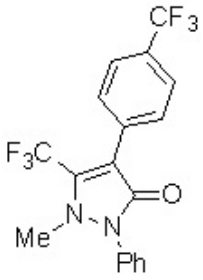
=== CHANNEL f1 ====	
NUC1	19F
P1	15.50 usec
PL1	-5.00 dB
PL1W	46.07103729 W
SFO1	470.5394058 MHz
SI	131072
HZpPT	0.433488 Hz
SR	400.51 Hz
WDW	EM
LB	0.00 Hz
GB	0
SSB	0

^{1}H NMR spectrum of compound **3e** in $CDCl_3$



NAME	ESh825
EXPNO	1
PROCNO	1
USER	uralnmr
Date_	20210816
Time	13.39
INSTRUM	AV500
PROBHD	5 mm PABBO BB-
PULPROG	zg30
SOLVENT	CDCl3
TD	32768
SW	16.0214 ppm
O1P	7.000 ppm
FIDRES	0.244532 Hz
NS	16
DS	2
AQ	2.0447731 sec
RG	101
TE	295.3 K
DE	6.50 usec
D1	1.00000000 sec
TD0	1
=== CHANNEL f1 =====	
NUC1	1H
P1	12.00 usec
PL1	0.30 dB
PL1W	18.91792679 W
SFO1	500.1335009 MHz
SI	32768
HZpPT	0.244532 Hz
SR	13.20 Hz
WDW	EM
LB	0.00 Hz
GB	0
SSB	0

^{13}C NMR spectrum of compound **3e** in $CDCl_3$



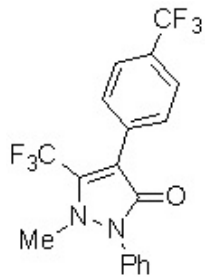
161.95 140.87 140.57 133.01 131.97 130.60 130.34 130.23 130.22 129.56 128.15 125.17 125.14 125.11 125.08 124.85 122.93 120.78 118.61 115.62 77.25 77.00 76.75 37.82 37.81

131.98 131.97 130.60 130.34 130.23 130.22 130.08 125.14 125.11 125.08 37.82 37.81 140.87 140.57

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
ppm

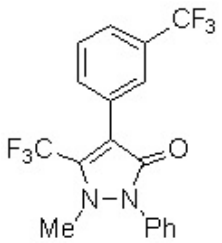
NAME	ESh825
EXPNO	13
PROCNO	1
USER	uralnmr
Date_	20210816
Time	13.53
INSTRUM	AV500
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
SOLVENT	CDCl3
TD	65536
SW	209.2368 ppm
O1P	100.000 ppm
FIDRES	0.401547 Hz
NS	2048
DS	8
AQ	1.2452340 sec
RG	203
TE	296.3 K
DE	6.50 usec
D1	1.00000000 sec
D11	0.03000000 sec
TD0	1
==== CHANNEL f1 ======	
NUC1	13C
P1	10.00 usec
PL1	0.00 dB
PL1W	115.29558563 W
SFO1	125.7703648 MHz
===== CHANNEL f2 =====	
CPDPRG2	waltz16
NUC2	1H
PCPD2	75.00 usec
PL2	120.00 dB
PL12	16.30 dB
PL13	19.30 dB
PL2W	0.00000000 W
PL12W	0.47519693 W
PL13W	0.23816262 W
SFO2	500.1320005 MHz
SI	65536
HZpPT	0.401547 Hz
SR	4.27 Hz
WDW	EM
LB	1.00 Hz
GB	0
SSB	0

^{19}F NMR spectrum of compound **3e** in $CDCl_3$



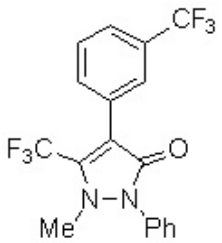
NAME	ESh825
EXPNO	19
PROCNO	1
USER	uralnmr
Date_	20210816
Time	13.43
INSTRUM	AV500
PROBHD	5 mm PABBO BB-
PULPROG	zg30
SOLVENT	CDCl3
TD	131072
SW	120.7512 ppm
O1P	50.000 ppm
FIDRES	0.433488 Hz
NS	16
DS	2
AQ	1.1534836 sec
RG	203
TE	295.3 K
DE	6.50 usec
D1	1.00000000 sec
TD0	1
==== CHANNEL f1 ======	
NUC1	19F
P1	15.50 usec
PL1	-5.00 dB
PL1W	46.07103729 W
SFO1	470.5394058 MHz
SI	131072
HZpPT	0.433488 Hz
SR	390.01 Hz
WDW	EM
LB	0.00 Hz
GB	0
SSB	0

^{1}H NMR spectrum of compound **3f** in $CDCl_3$



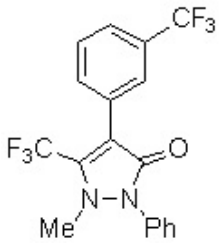
NAME	ESh826
EXPNO	1
PROCNO	1
USER	uralnmr
Date_	20210820
Time	16.45
INSTRUM	AV500
PROBHD	5 mm PABBO BB-
PULPROG	zg30
SOLVENT	CDCl3
TD	32768
SW	16.0214 ppm
O1P	7.000 ppm
FIDRES	0.244532 Hz
NS	32
DS	2
AQ	2.0447731 sec
RG	203
TE	296.4 K
DE	6.50 usec
D1	1.00000000 sec
TD0	1
==== CHANNEL f1 =====	
NUC1	1H
P1	12.00 usec
PL1	0.30 dB
PL1W	18.91792679 W
SFO1	500.1335009 MHz
SI	32768
HZpPT	0.244532 Hz
SR	13.45 Hz
WDW	EM
LB	0.00 Hz
GB	0
SSB	0

^{13}C NMR spectrum of compound **3f** in $CDCl_3$



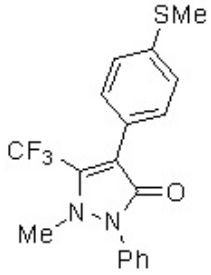
NAME	ESh826
EXPNO	13
PROCNO	1
USER	uralnmr
Date_	20210820
Time	16.57
INSTRUM	AV500
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
SOLVENT	CDCl3
TD	32768
SW	200.7838 ppm
O1P	95.000 ppm
FIDRES	0.770646 Hz
NS	49152
DS	8
AQ	0.6488564 sec
RG	203
TE	297.1 K
DE	6.50 usec
D1	1.00000000 sec
D11	0.03000000 sec
TD0	48
==== CHANNEL f1 =====	
NUC1	13C
P1	10.00 usec
PL1	0.00 dB
PL1W	115.29558563 W
SFO1	125.7697360 MHz
=== CHANNEL f2 =====	
CPDPRG2	waltz16
NUC2	1H
PCPD2	75.00 usec
PL2	120.00 dB
PL12	16.30 dB
PL13	19.30 dB
PL2W	0.00000000 W
PL12W	0.47519693 W
PL13W	0.23816262 W
SFO2	500.1320005 MHz
SI	65536
HZpPT	0.385323 Hz
SR	0.05 Hz
WDW	EM
LB	1.00 Hz
GB	0
SSB	0

^{19}F NMR spectrum of compound **3f** in $CDCl_3$



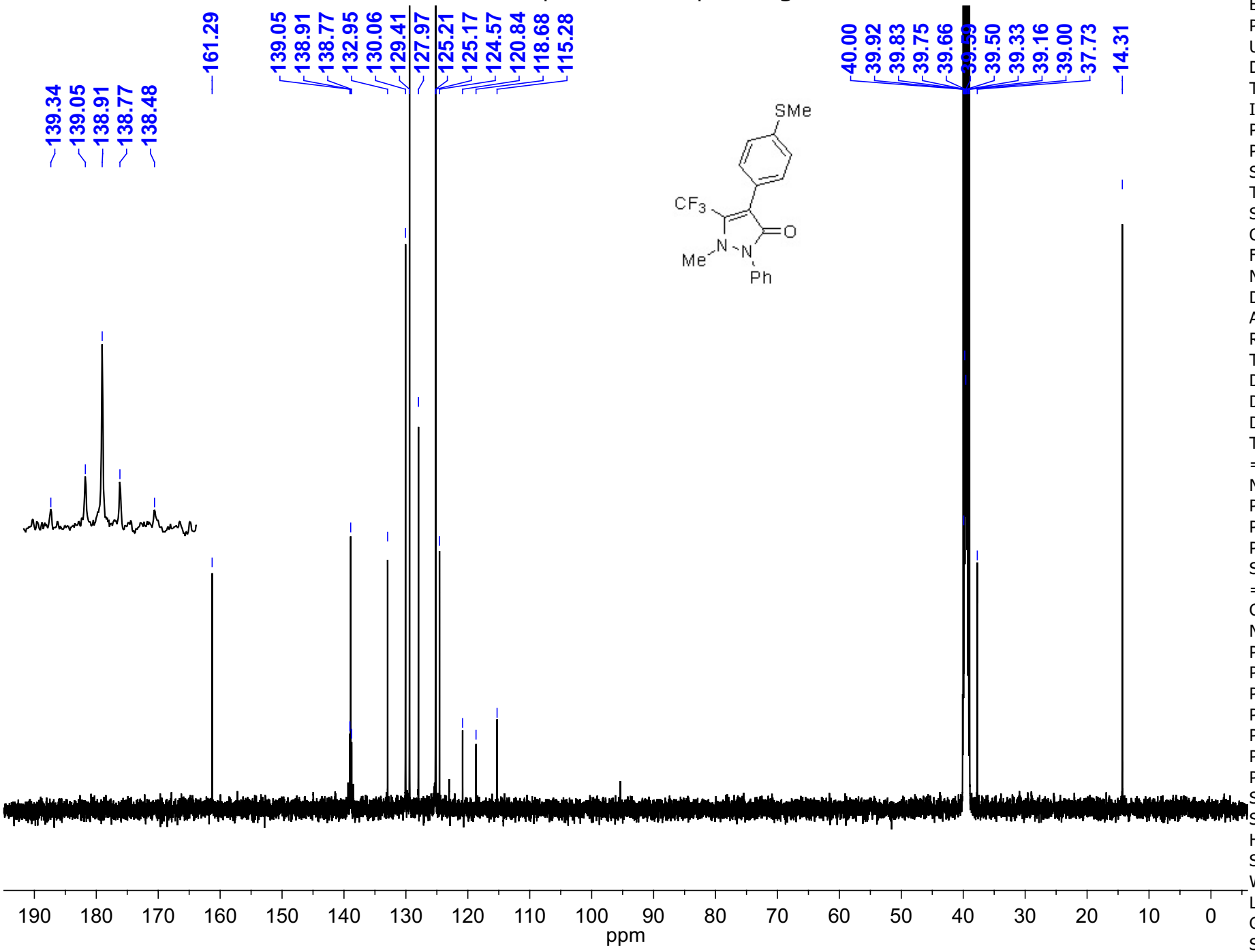
NAME	ESh826
EXPNO	19
PROCNO	1
USER	uralnmr
Date_	20210820
Time	16.49
INSTRUM	AV500
PROBHD	5 mm PABBO BB-
PULPROG	zg30
SOLVENT	CDCl3
TD	131072
SW	120.7506 ppm
O1P	55.000 ppm
FIDRES	0.433488 Hz
NS	16
DS	2
AQ	1.1534836 sec
RG	203
TE	296.4 K
DE	6.50 usec
D1	1.00000000 sec
TD0	1
====CHANNEL f1 =====	
NUC1	19F
P1	15.50 usec
PL1	-5.00 dB
PL1W	46.07103729 W
SFO1	470.5417584 MHz
SI	131072
HZpPT	0.433488 Hz
SR	398.60 Hz
WDW	EM
LB	0.00 Hz
GB	0
SSB	0

^{1}H NMR spectrum of compound **3g** in DMSO-d_6



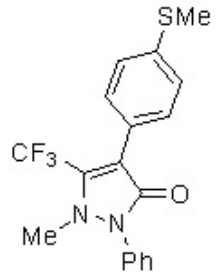
NAME	ESh698
EXPNO	1
PROCNO	1
USER	uralnmr
Date_	20191001
Time	11.08
INSTRUM	AV500
PROBHD	5 mm PABBO BB-
PULPROG	zg30
SOLVENT	DMSO
TD	32768
SW	12.0160 ppm
O1P	5.000 ppm
FIDRES	0.183399 Hz
NS	16
DS	2
AQ	2.7263477 sec
RG	203
TE	296.6 K
DE	16.00 usec
D1	1.00000000 sec
TD0	1
===== CHANNEL f1 =====	
NUC1	1H
P1	12.00 usec
PL1	0.30 dB
PL1W	18.91792679 W
SFO1	500.1325007 MHz
SI	32768
HZpPT	0.183399 Hz
SR	5.33 Hz
WDW	EM
LB	0.00 Hz
GB	0
SSB	0

^{13}C NMR spectrum of compound **3g** in DMSO-d_6



NAME	ESh698
EXPNO	13
PROCNO	1
USER	uralnmr
Date_	20191002
Time	10.57
INSTRUM	AV500
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
SOLVENT	DMSO
TD	32768
SW	200.7838 ppm
O1P	95.000 ppm
FIDRES	0.770646 Hz
NS	2048
DS	8
AQ	0.6488564 sec
RG	203
TE	297.1 K
DE	6.50 usec
D1	1.00000000 sec
D11	0.03000000 sec
TD0	2
==== CHANNEL f1 =====	
NUC1	13C
P1	10.00 usec
PL1	0.00 dB
PL1W	115.29558563 W
SFO1	125.7697360 MHz
==== CHANNEL f2 =====	
CPDPRG2	waltz16
NUC2	1H
PCPD2	75.00 usec
PL2	120.00 dB
PL12	16.30 dB
PL13	19.30 dB
PL2W	0.00000000 W
PL12W	0.47519693 W
PL13W	0.23816262 W
SFO2	500.1320005 MHz
SI	65536
HZpPT	0.385323 Hz
SR	62.03 Hz
WDW	EM
LB	1.00 Hz
GB	0
SSB	0

^{19}F NMR spectrum of compound **3g** in DMSO-d_6



104.68

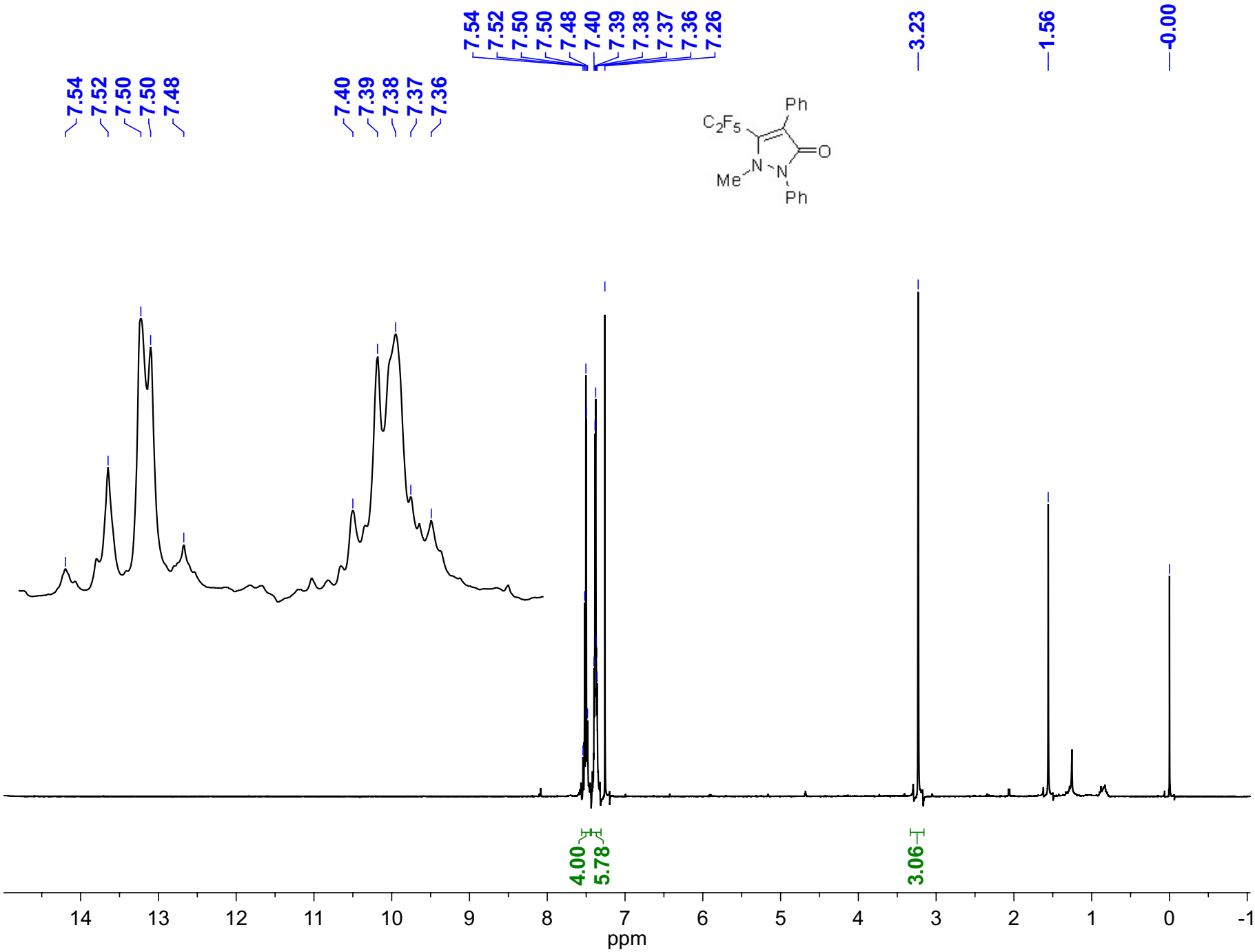
0.00

NAME	ESh698
EXPNO	19
PROCNO	1
USER	uralnmr
Date_	20191001
Time	11.14
INSTRUM	AV500
PROBHD	5 mm PABBO BB-
PULPROG	zg30
SOLVENT	DMSO
TD	131072
SW	120.7506 ppm
O1P	55.000 ppm
FIDRES	0.433488 Hz
NS	16
DS	2
AQ	1.1534836 sec
RG	203
TE	296.6 K
DE	6.50 usec
D1	1.00000000 sec
TD0	1
=== CHANNEL f1 =====	
NUC1	19F
P1	15.50 usec
PL1	-5.00 dB
PL1W	46.07103729 W
SFO1	470.5417584 MHz
SI	131072
HZpPT	0.433488 Hz
SR	-3.69 Hz
WDW	EM
LB	0.00 Hz
GB	0
SSB	0

115 105 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 -5

ppm

^{1}H NMR spectrum of compound **3h** in $CDCl_3$



Current Data Parameters

NAME	ESh707
EXPNO	1
PROCNO	1
USER	uralnmr

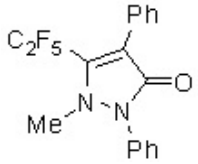
F2 - Acquisition Parameters

Date_	20200131
Time	12.50
INSTRUM	DRX400
PROBHD	5 mm SEF 19F-1
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	6410.256 Hz
FIDRES	0.195625 Hz
AQ	2.5559540 sec
RG	574.7
DW	78.000 usec
DE	6.00 usec
TE	297.2 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec
==== CHANNEL f1 =====	
NUC1	1H
P1	20.00 usec
PL1	0.00 dB
SFO1	400.1328009 MHz

F2 - Processing parameters

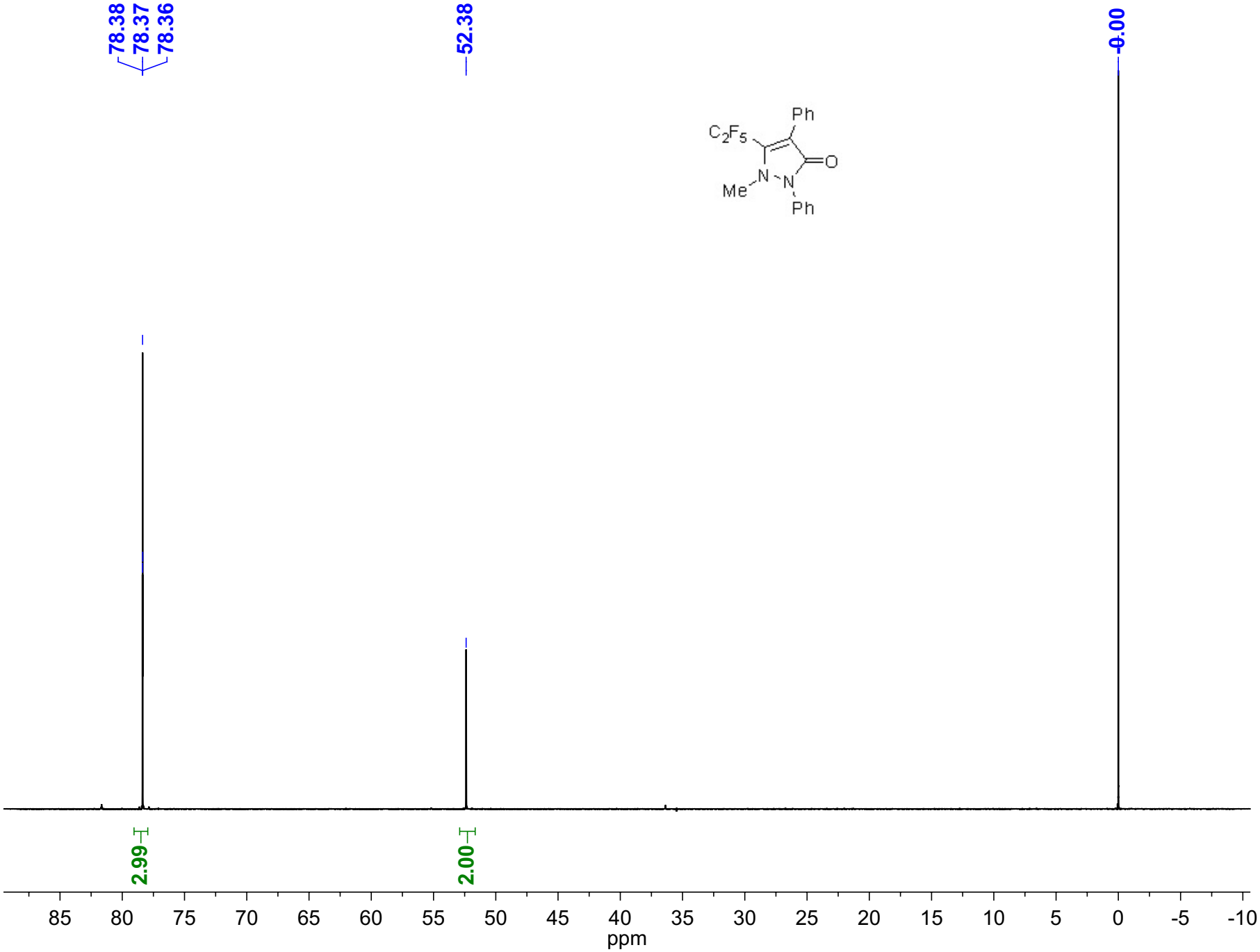
SI	32768
HZpPT	0.195625 Hz
SF	400.1300099 MHz
SR	9.93 Hz
WDW	EM
LB	0.00 Hz
GB	0
SSB	0
PC	4.00

^{13}C NMR spectrum of compound **3h** in $CDCl_3$



NAME	ESh707
EXPNO	13
PROCNO	1
USER	uralnmr
Date_	20200211
Time	19.46
INSTRUM	AV500
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
SOLVENT	CDCl3
TD	65536
SW	209.2368 ppm
O1P	100.000 ppm
FIDRES	0.401547 Hz
NS	20480
DS	8
AQ	1.2452340 sec
RG	203
TE	296.6 K
DE	6.50 usec
D1	1.00000000 sec
D11	0.03000000 sec
TD0	20
=== CHANNEL f1 ====	
NUC1	13C
P1	9.00 usec
PL1	0.00 dB
PL1W	115.29558563 W
SFO1	125.7703648 MHz
=== CHANNEL f2 ====	
CPDPRG2	waltz16
NUC2	1H
PCPD2	75.00 usec
PL2	120.00 dB
PL12	17.00 dB
PL13	20.00 dB
PL2W	0.00000000 W
PL12W	0.40445811 W
PL13W	0.20270923 W
SFO2	500.1320005 MHz
SI	32768
HZpPT	0.803094 Hz
SR	3.36 Hz
WDW	EM
LB	1.50 Hz
GB	0
SSB	0

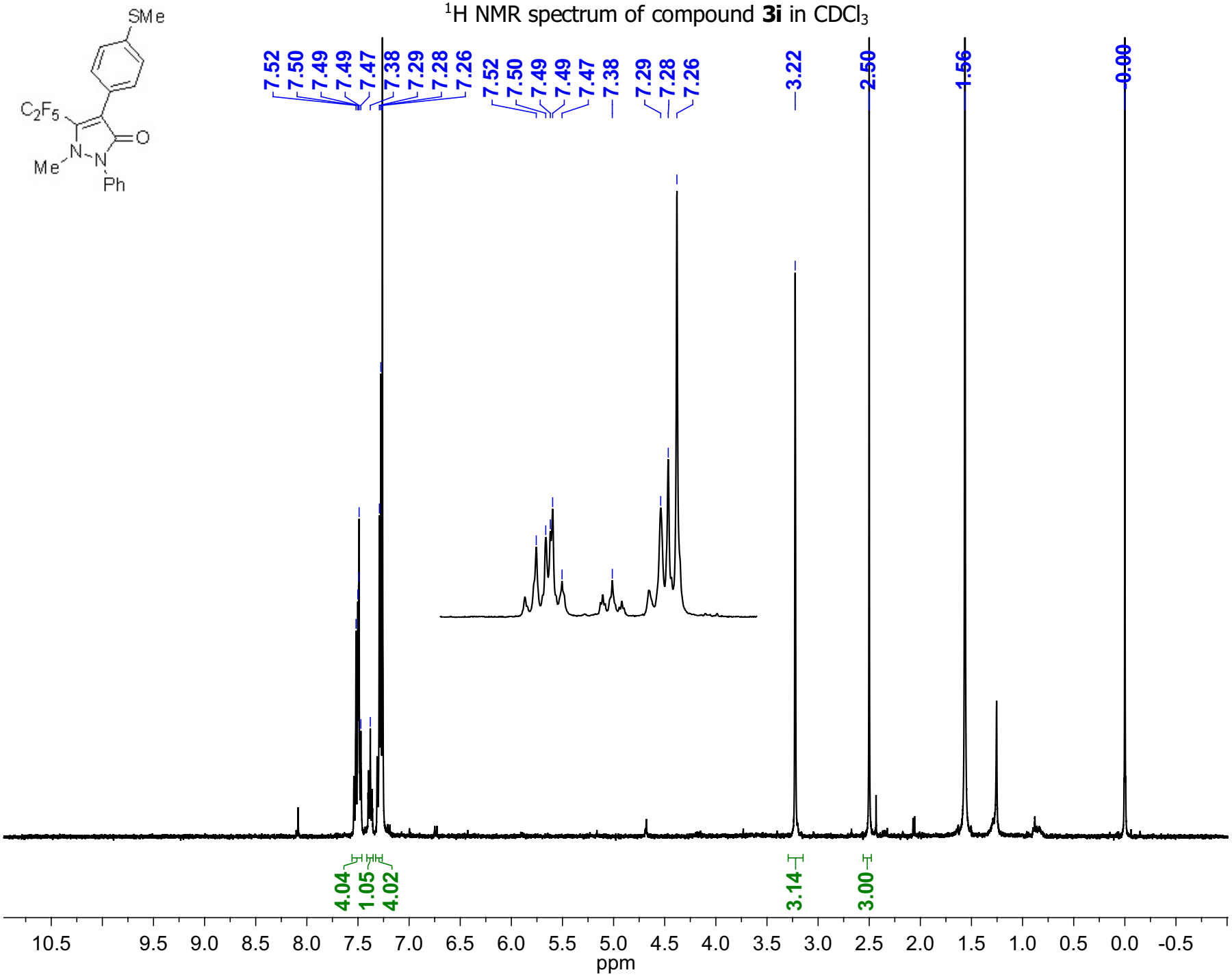
^{19}C\F NMR spectrum of compound **3h** in $CDCl_3$



Current Data Parameters
NAME ESh707
EXPNO 19
PROCNO 1
USER uralnmr

F2 - Acquisition Parameters
Date_ 20200131
Time 12.54
INSTRUM DRX400
PROBHD 5 mm SEF 19F-1
PULPROG zg30
TD 131072
SOLVENT CDCl3
NS 16
DS 2
SWH 37664.785 Hz
FIDRES 0.287360 Hz
AQ 1.7400308 sec
RG 2298.8
DW 13.275 usec
DE 6.50 usec
TE 297.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
===== CHANNEL f1 =====
NUC1 19F
P1 10.10 usec
PL1 0.00 dB
SFO1 376.4523425 MHz

F2 - Processing parameters
SI 131072
HZpPT 0.287360 Hz
SF 376.4374855 MHz
SR 200.54 Hz
WDW EM
LB 0.00 Hz
GB 0
SSB 0
PC 3.00



Current Data Parameters

NAME	ESh714
EXPNO	1
PROCNO	1
USER	uralnmr

F2 - Acquisition Parameters

Date_	20200312
Time	14.27
INSTRUM	DRX400
PROBHD	5 mm SEF 19F-1
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	4789.272 Hz
FIDRES	0.146157 Hz
AQ	3.4210291 sec
RG	812.7
DW	104.400 usec
DE	6.00 usec
TE	298.2 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

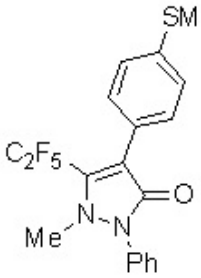
==== CHANNEL f1 =====

NUC1	1H
P1	20.00 usec
PL1	0.00 dB
SFO1	400.1320007 MHz

F2 - Processing parameters

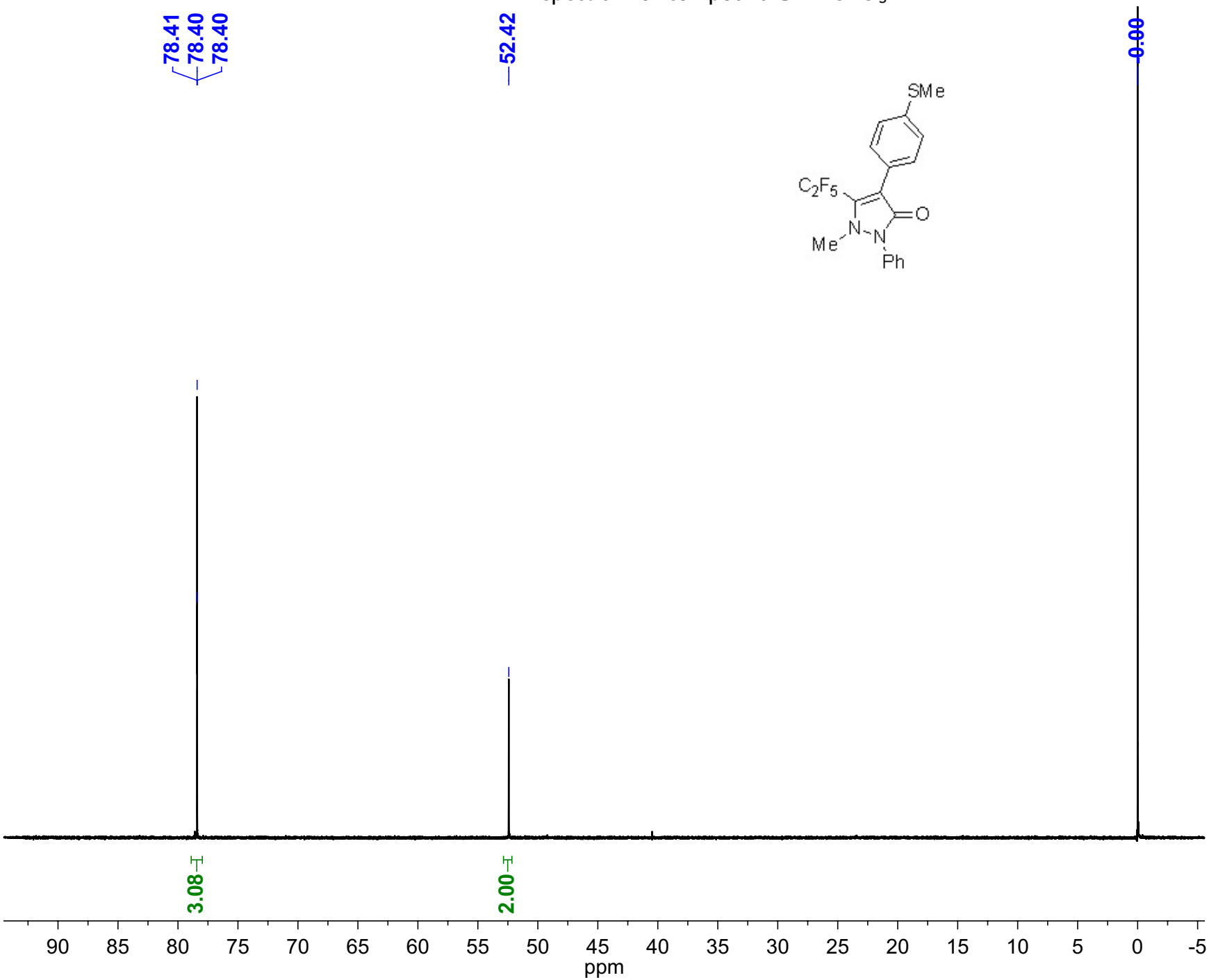
SI	32768
HZpPT	0.146157 Hz
SF	400.1300090 MHz
SR	8.97 Hz
WDW	EM
LB	0.00 Hz
GB	0
SSB	0
PC	4.00

^{13}C NMR spectrum of compound **3i** in $CDCl_3$



NAME	ESh714
EXPNO	13
PROCNO	1
USER	uralnmr
Date_	20200330
Time	19.09
INSTRUM	AV500
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
SOLVENT	CDCl3
TD	65536
SW	200.7838 ppm
O1P	95.000 ppm
FIDRES	0.385323 Hz
NS	32768
DS	8
AQ	1.2976629 sec
RG	203
TE	297.2 K
DE	6.50 usec
D1	1.20000005 sec
D11	0.03000000 sec
TD0	32
==== CHANNEL f1 =====	
NUC1	13C
P1	9.00 usec
PL1	0.00 dB
PL1W	115.29558563 W
SFO1	125.7697360 MHz
==== CHANNEL f2 =====	
CPDPRG2	waltz16
NUC2	1H
PCPD2	75.00 usec
PL2	120.00 dB
PL12	17.00 dB
PL13	20.00 dB
PL2W	0.00000000 W
PL12W	0.40445811 W
PL13W	0.20270923 W
SFO2	500.1320005 MHz
SI	65536
HZpPT	0.385323 Hz
SR	3.54 Hz
WDW	EM
LB	1.00 Hz
GB	0
SSB	0

^{19}F NMR spectrum of compound **3i** in $CDCl_3$



Current Data Parameters

NAME	ESh714
EXPNO	19
PROCNO	1
USER	uralnmr

F2 - Acquisition Parameters

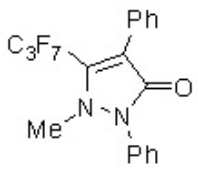
Date_	20200312
Time	14.32
INSTRUM	DRX400
PROBHD	5 mm SEF 19F-1
PULPROG	zg30
TD	131072
SOLVENT	CDCl3
NS	16
DS	2
SWH	37664.785 Hz
FIDRES	0.287360 Hz
AQ	1.7400308 sec
RG	2580.3
DW	13.275 usec
DE	6.50 usec
TE	298.2 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

==== CHANNEL f1 ====

NUC1	19F
P1	10.10 usec
PL1	0.00 dB
SFO1	376.4542247 MHz

F2 - Processing parameters

SI	131072
HZpPT	0.287360 Hz
SF	376.4374841 MHz
SR	199.13 Hz
WDW	EM
LB	0.00 Hz
GB	0
SSB	0
PC	3.00



^{1}H NMR spectrum of compound **3j** in $CDCl_3$

Current Data Parameters

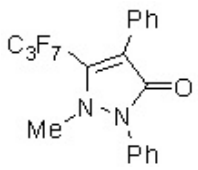
NAME	ESh708
EXPNO	1
PROCNO	1
USER	uralnmr

F2 - Acquisition Parameters

Date_	20200131
Time	13.13
INSTRUM	DRX400
PROBHD	5 mm SEF 19F-1
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	6410.256 Hz
FIDRES	0.195625 Hz
AQ	2.5559540 sec
RG	574.7
DW	78.000 usec
DE	6.00 usec
TE	297.2 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec
===CHANNEL f1 ======	
NUC1	1H
P1	20.00 usec
PL1	0.00 dB
SFO1	400.1328009 MHz

F2 - Processing parameters

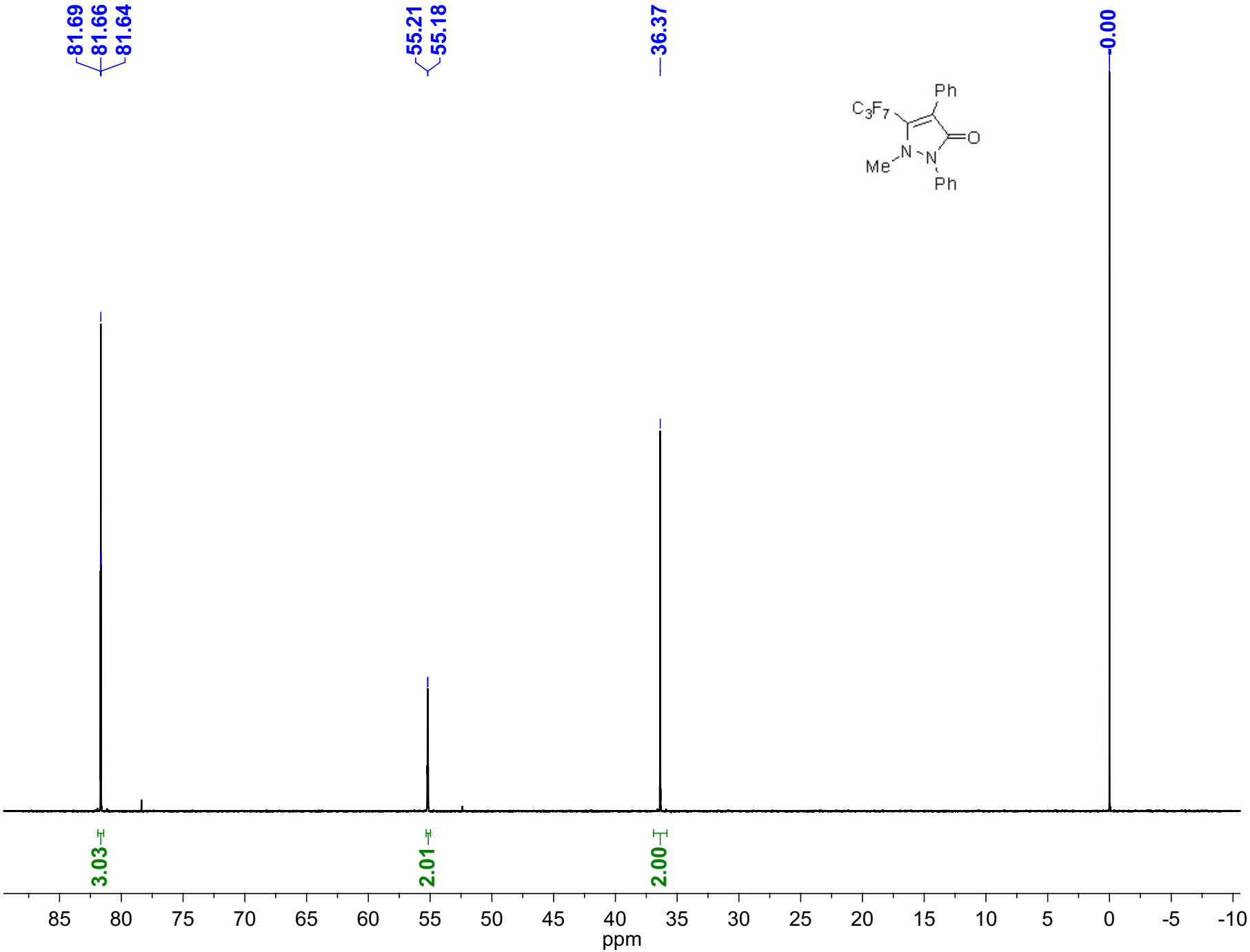
SI	32768
HZpPT	0.195625 Hz
SF	400.1300102 MHz
SR	10.25 Hz
WDW	EM
LB	0.00 Hz
GB	0
SSB	0
PC	4.00



^{13}C NMR spectrum of compound **3j** in $CDCl_3$

NAME	ESh708
EXPNO	13
PROCNO	1
USER	uralnmr
Date_	20200210
Time	17.43
INSTRUM	AV500
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
SOLVENT	CDCl3
TD	32768
SW	200.7838 ppm
O1P	95.000 ppm
FIDRES	0.770646 Hz
NS	32768
DS	8
AQ	0.6488564 sec
RG	203
TE	295.9 K
DE	6.50 usec
D1	1.00000000 sec
D11	0.03000000 sec
TD0	32
==== CHANNEL f1 =====	
NUC1	13C
P1	9.00 usec
PL1	0.00 dB
PL1W	115.29558563 W
SFO1	125.7697360 MHz
==== CHANNEL f2 =====	
CPDPRG2	waltz16
NUC2	1H
PCPD2	75.00 usec
PL2	120.00 dB
PL12	17.00 dB
PL13	20.00 dB
PL2W	0.00000000 W
PL12W	0.40445811 W
PL13W	0.20270923 W
SFO2	500.1320005 MHz
SI	32768
HZpPT	0.770646 Hz
SR	2.16 Hz
WDW	EM
LB	1.50 Hz
GB	0
SSB	0

^{19}F NMR spectrum of compound **3j** in $CDCl_3$



Current Data Parameters

NAME	ESh708
EXPNO	19
PROCNO	1
USER	uralnmr

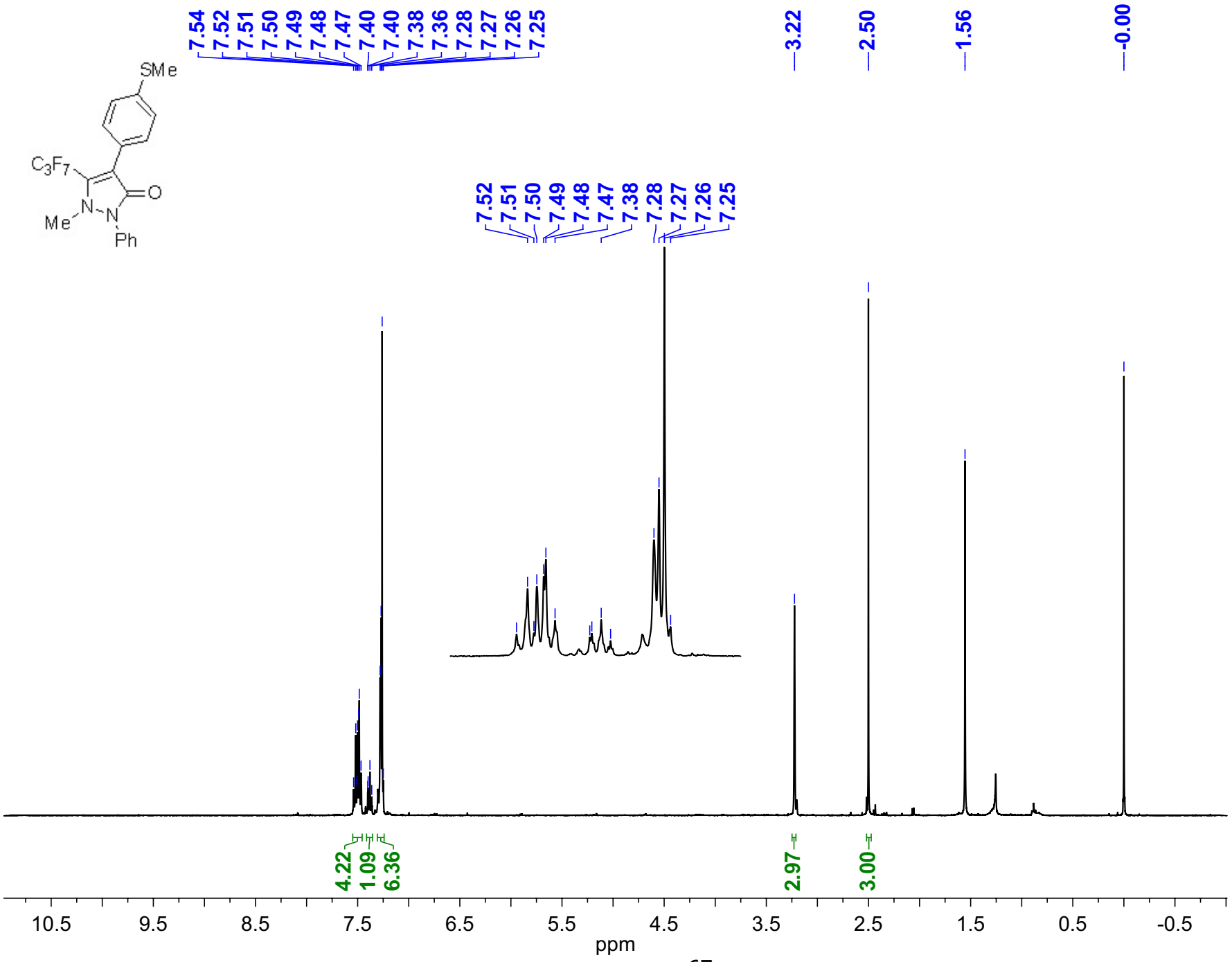
F2 - Acquisition Parameters

Date_	20200131
Time	13.17
INSTRUM	DRX400
PROBHD	5 mm SEF 19F-1
PULPROG	zg30
TD	131072
SOLVENT	CDCl3
NS	16
DS	2
SWH	37664.785 Hz
FIDRES	0.287360 Hz
AQ	1.7400308 sec
RG	2298.8
DW	13.275 usec
DE	6.50 usec
TE	297.2 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec
==== CHANNEL f1 =====	
NUC1	19F
P1	10.10 usec
PL1	0.00 dB
SFO1	376.4523425 MHz

F2 - Processing parameters

SI	131072
HZpPT	0.287360 Hz
SF	376.4374848 MHz
SR	199.78 Hz
WDW	EM
LB	0.00 Hz
GB	0
SSB	0
PC	3.00

^{1}H NMR spectrum of compound **3k** in $CDCl_3$



Current Data Parameters

NAME	ESh715
EXPNO	1
PROCNO	1
USER	uralnmr

F2 - Acquisition Parameters

Date_	20200312
Time	14.39
INSTRUM	DRX400
PROBHD	5 mm SEF 19F-1
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	4789.272 Hz
FIDRES	0.146157 Hz
AQ	3.4210291 sec
RG	812.7
DW	104.400 usec
DE	6.00 usec
TE	298.2 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

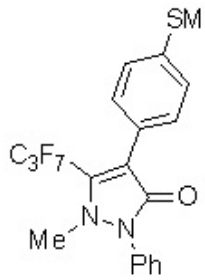
===== CHANNEL f1 ====

NUC1	1H
P1	20.00 usec
PL1	0.00 dB
SFO1	400.1320007 MHz

F2 - Processing parameters

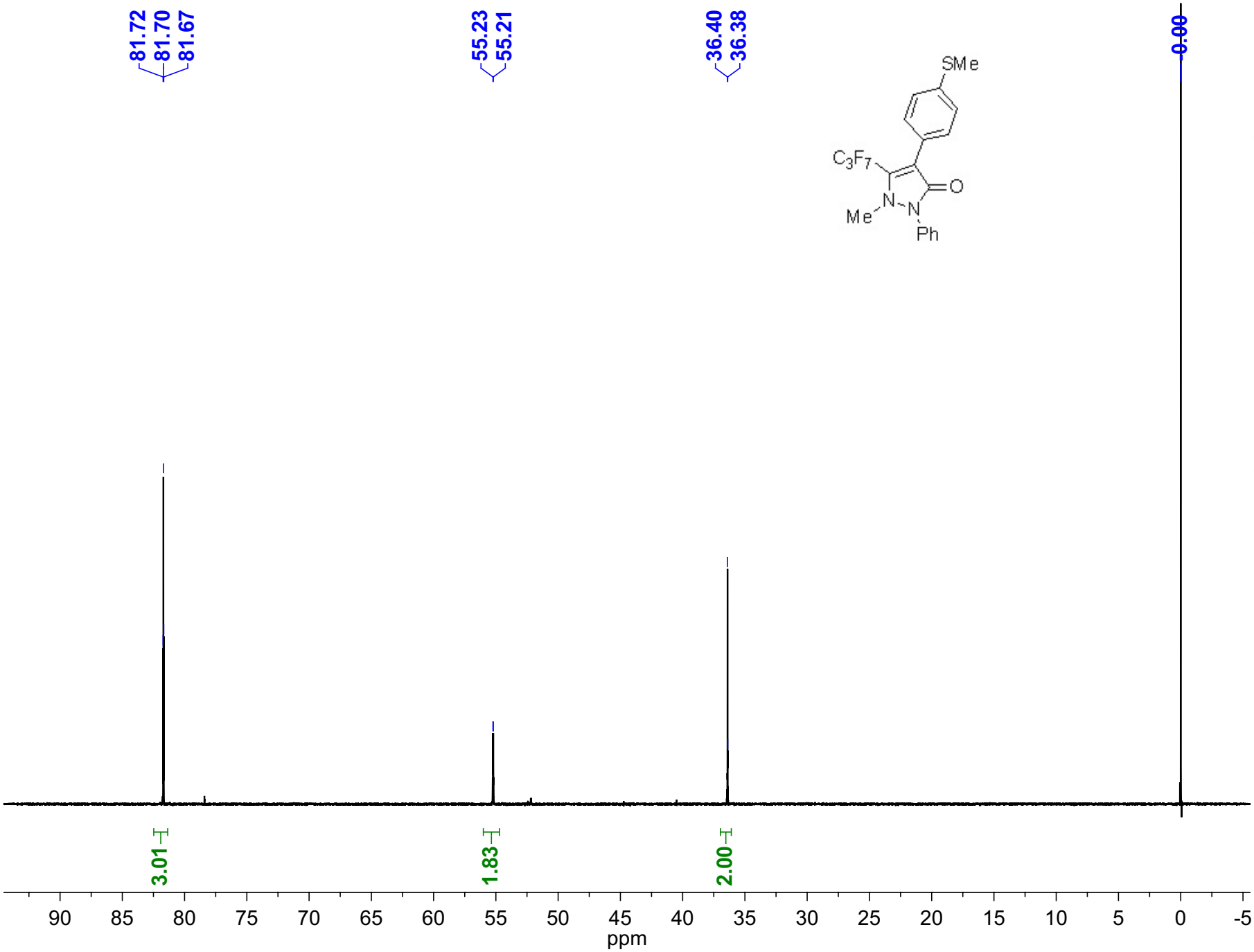
SI	32768
HZpPT	0.146157 Hz
SF	400.1300092 MHz
SR	9.23 Hz
WDW	EM
LB	0.00 Hz
GB	0
SSB	0
PC	4.00

^{13}C NMR spectrum of compound **3k** in $CDCl_3$



NAME	ESh715
EXPNO	13
PROCNO	1
USER	uralnmr
Date_	20200325
Time	19.03
INSTRUM	AV500
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
SOLVENT	CDCl3
TD	32768
SW	200.7838 ppm
O1P	95.000 ppm
FIDRES	0.770646 Hz
NS	30720
DS	8
AQ	0.6488564 sec
RG	203
TE	296.6 K
DE	6.50 usec
D1	1.00000000 sec
D11	0.03000000 sec
TD0	30
==== CHANNEL f1 =====	
NUC1	13C
P1	9.00 usec
PL1	0.00 dB
PL1W	115.29558563 W
SFO1	125.7697360 MHz
==== CHANNEL f2 ====	
CPDPRG2	waltz16
NUC2	1H
PCPD2	75.00 usec
PL2	120.00 dB
PL12	17.00 dB
PL13	20.00 dB
PL2W	0.00000000 W
PL12W	0.40445811 W
PL13W	0.20270923 W
SFO2	500.1320005 MHz
SI	65536
HZpPT	0.385323 Hz
SR	-2.56 Hz
WDW	EM
LB	1.00 Hz
GB	0
SSB	0

^{13}C NMR spectrum of compound **3k** in $CDCl_3$



Current Data Parameters

NAME	ESh715
EXPNO	19
PROCNO	1
USER	uralnmr

F2 - Acquisition Parameters

Date_	20200312
Time	14.42
INSTRUM	DRX400
PROBHD	5 mm SEF 19F-1
PULPROG	zg30
TD	131072
SOLVENT	CDCl3
NS	16
DS	2
SWH	37664.785 Hz
FIDRES	0.287360 Hz
AQ	1.7400308 sec
RG	2580.3
DW	13.275 usec
DE	6.50 usec
TE	298.2 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

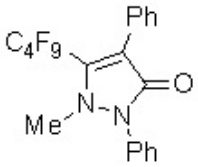
==== CHANNEL f1 =====

NUC1	19F
P1	10.10 usec
PL1	0.00 dB
SFO1	376.4542247 MHz

F2 - Processing parameters

SI	131072
HZpPT	0.287360 Hz
SF	376.4374837 MHz
SR	198.74 Hz
WDW	EM
LB	0.00 Hz
GB	0
SSB	0
PC	3.00

^{1}H NMR spectrum of compound **3l** in $CDCl_3$

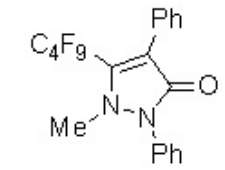


Current Data Parameters
NAME ESh722
EXPNO 1
PROCNO 1
USER uralnmr

F2 - Acquisition Parameters
Date_ 20200312
Time 14.49
INSTRUM DRX400
PROBHD 5 mm SEF 19F-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 812.7
DW 104.400 usec
DE 6.00 usec
TE 298.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 20.00 usec
PL1 0.00 dB
SFO1 400.1320007 MHz

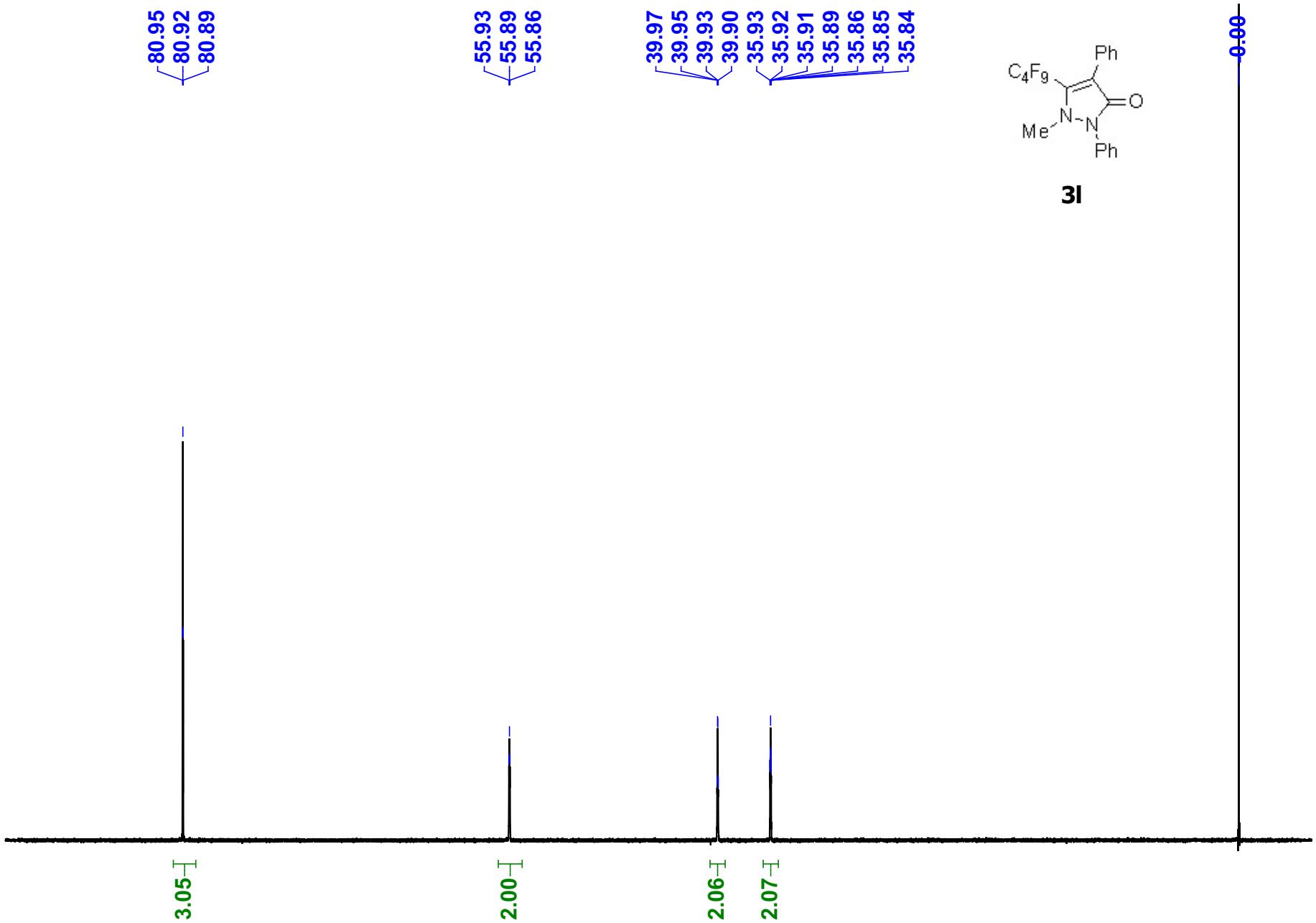
F2 - Processing parameters
SI 32768
HZpPT 0.146157 Hz
SF 400.1300093 MHz
SR 9.28 Hz
WDW EM
LB 0.00 Hz
GB 0
SSB 0
PC 4.00



^{13}C NMR spectrum of compound **3l** in $CDCl_3$

NAME	ESh722
EXPNO	13
PROCNO	1
USER	uralnmr
Date_	20200320
Time	18.26
INSTRUM	AV500
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
SOLVENT	CDCl3
TD	65536
SW	200.7838 ppm
O1P	95.000 ppm
FIDRES	0.385323 Hz
NS	32768
DS	8
AQ	1.2976629 sec
RG	203
TE	296.6 K
DE	6.50 usec
D1	1.00000000 sec
D11	0.03000000 sec
TD0	32
===== CHANNEL f1 =====	
NUC1	13C
P1	9.00 usec
PL1	0.00 dB
PL1W	115.29558563 W
SFO1	125.7697360 MHz
===== CHANNEL f2 =====	
CPDPRG2	waltz16
NUC2	1H
PCPD2	75.00 usec
PL2	120.00 dB
PL12	17.00 dB
PL13	20.00 dB
PL2W	0.00000000 W
PL12W	0.40445811 W
PL13W	0.20270923 W
SFO2	500.1320005 MHz
SI	131072
HZpPT	0.192661 Hz
SR	1.58 Hz
WDW	EM
LB	1.00 Hz
GB	0
SSB	0

^{19}F NMR spectrum of compound **3l** in $CDCl_3$



3l

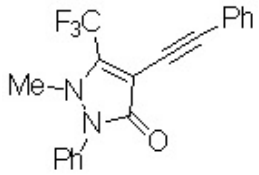
Current Data Parameters
NAME ESh722
EXPNO 19
PROCNO 1
USER uralnmr

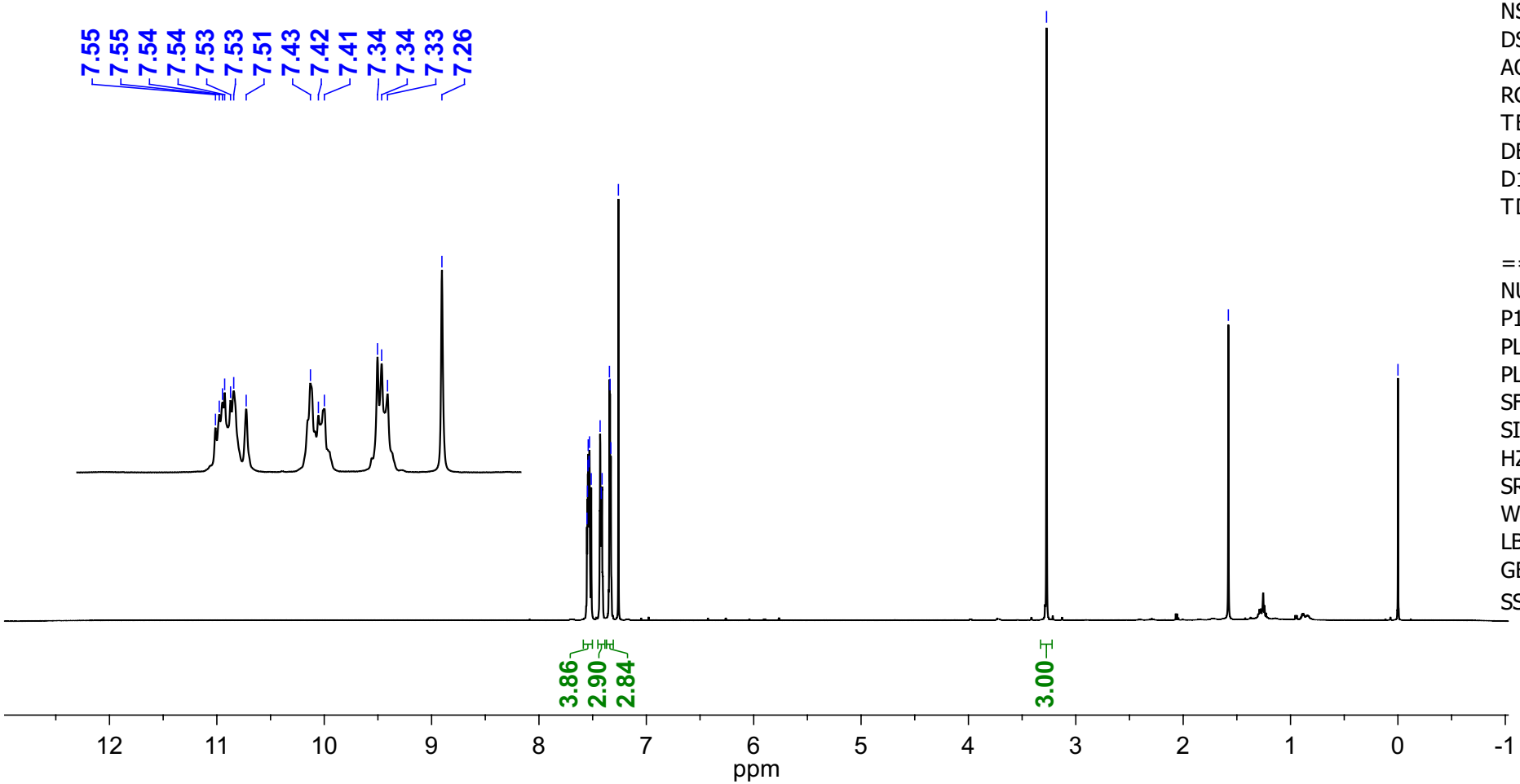
F2 - Acquisition Parameters
Date_ 20200312
Time 14.52
INSTRUM DRX400
PROBHD 5 mm SEF 19F-1
PULPROG zg30
TD 131072
SOLVENT CDCl3
NS 16
DS 2
SWH 37664.785 Hz
FIDRES 0.287360 Hz
AQ 1.7400308 sec
RG 2580.3
DW 13.275 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

=== CHANNEL f1 =====
NUC1 19F
P1 10.10 usec
PL1 0.00 dB
SFO1 376.4542247 MHz

F2 - Processing parameters
SI 131072
HZpPT 0.287360 Hz
SF 376.4374823 MHz
SR 197.29 Hz
WDW EM
LB 0.00 Hz
GB 0
SSB 0
PC 3.00

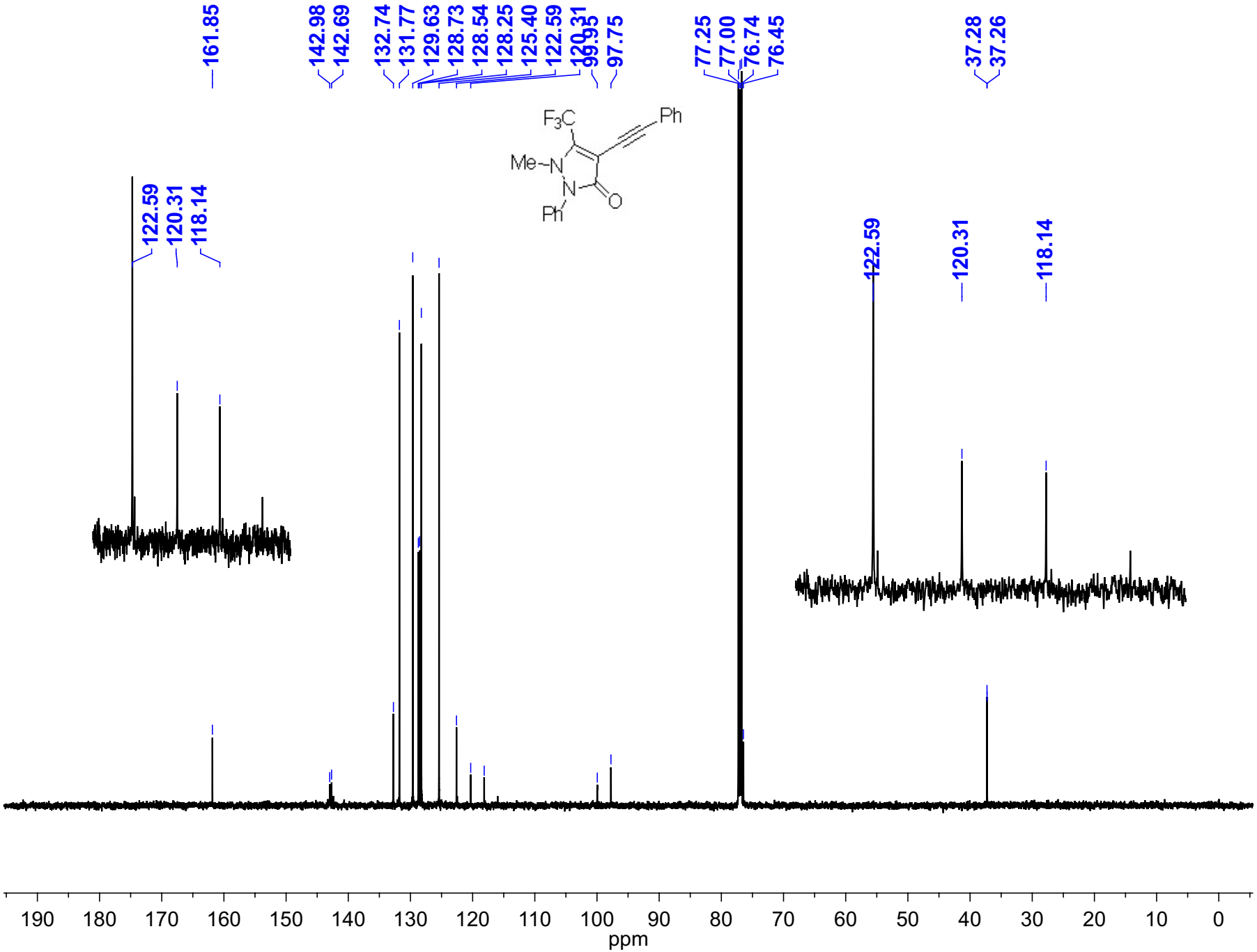
^{1}H NMR spectrum of compound **6a** in $CDCl_3$





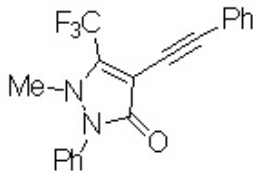
NAME	ESh782
EXPNO	1
PROCNO	1
USER	uralnmr
Date_	20210322
Time	14.35
INSTRUM	AV500
PROBHD	5 mm PABBO BB-
PULPROG	zg30
SOLVENT	CDCl3
TD	32768
SW	14.0019 ppm
O1P	6.000 ppm
FIDRES	0.213709 Hz
NS	16
DS	0
AQ	2.3396852 sec
RG	203
TE	296.3 K
DE	6.50 usec
D1	1.00000000 sec
TD0	1
==== CHANNEL f1 ====	
NUC1	1H
P1	12.00 usec
PL1	0.30 dB
PL1W	18.91792679 W
SFO1	500.1330008 MHz
SI	32768
HZpPT	0.213709 Hz
SR	12.68 Hz
WDW	EM
LB	0.00 Hz
GB	0
SSB	0

^{13}C NMR spectrum of compound **6a** in $CDCl_3$



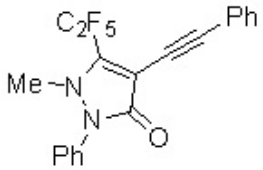
NAME	ESH782
EXPNO	13
PROCNO	1
USER	uralnmr
Date_	20210405
Time	10.56
INSTRUM	AV500
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
SOLVENT	CDCl3
TD	32768
SW	200.7838 ppm
O1P	95.000 ppm
FIDRES	0.770646 Hz
NS	1024
DS	8
AQ	0.6488564 sec
RG	203
TE	297.0 K
DE	6.50 usec
D1	0.85000002 sec
D11	0.03000000 sec
TD0	1
==== CHANNEL f1 =====	
NUC1	13C
P1	10.00 usec
PL1	0.00 dB
PL1W	115.29558563 W
SFO1	125.7697360 MHz
===== CHANNEL f2 =====	
CPDPRG2	waltz16
NUC2	1H
PCPD2	75.00 usec
PL2	120.00 dB
PL12	16.30 dB
PL13	19.30 dB
PL2W	0.00000000 W
PL12W	0.47519693 W
PL13W	0.23816262 W
SFO2	500.1320005 MHz
SI	65536
HZpPT	0.385323 Hz
SR	5.13 Hz
WDW	EM
LB	1.00 Hz
GB	0
SSB	0

^{19}H NMR spectrum of compound **6a** in $CDCl_3$



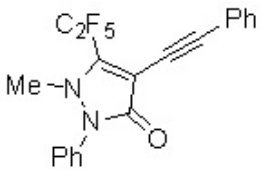
NAME	ESh782
EXPNO	19
PROCNO	1
USER	uralnmr
Date_	20210322
Time	14.37
INSTRUM	AV500
PROBHD	5 mm PABBO BB-
PULPROG	zg30
SOLVENT	CDCl3
TD	131072
SW	120.7506 ppm
O1P	55.000 ppm
FIDRES	0.433488 Hz
NS	8
DS	2
AQ	1.1534836 sec
RG	203
TE	296.3 K
DE	6.50 usec
D1	1.00000000 sec
TD0	1
==== CHANNEL f1 =====	
NUC1	19F
P1	15.50 usec
PL1	-5.00 dB
PL1W	46.07103729 W
SFO1	470.5417584 MHz
SI	131072
HZpPT	0.433488 Hz
SR	402.41 Hz
WDW	EM
LB	0.00 Hz
GB	0
SSB	0

^{1}H NMR spectrum of compound **6b** in $CDCl_3$



NAME	ESh824
EXPNO	1
PROCNO	1
USER	uralnmr
Date_	20210809
Time	17.38
INSTRUM	AV500
PROBHD	5 mm PABBO BB-
PULPROG	zg30
SOLVENT	CDCl3
TD	32768
SW	14.0019 ppm
O1P	6.000 ppm
FIDRES	0.213709 Hz
NS	16
DS	2
AQ	2.3396852 sec
RG	101
TE	295.1 K
DE	6.50 usec
D1	1.50000000 sec
TD0	1
==== CHANNEL f1 =====	
NUC1	1H
P1	12.00 usec
PL1	0.30 dB
PL1W	18.91792679 W
SFO1	500.1330008 MHz
SI	32768
HZpPT	0.213709 Hz
SR	12.89 Hz
WDW	EM
LB	0.00 Hz
GB	0
SSB	0

^{13}C NMR spectrum of compound **6b** in $CDCl_3$

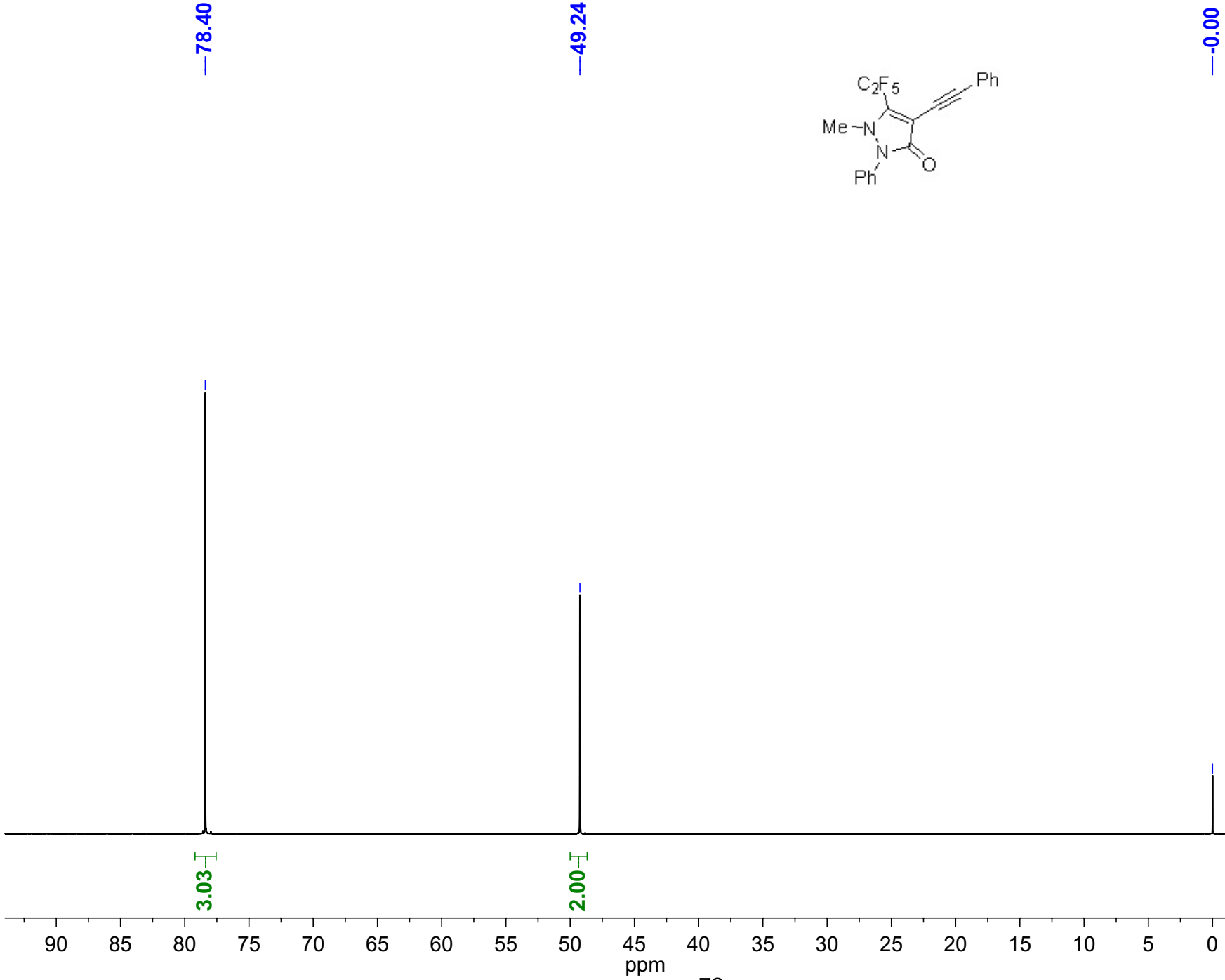


161.75
141.66
141.45
141.23
132.77
131.76
129.64
128.73
128.67
128.24
125.62
122.66
119.76
117.47
102.72
97.92
77.26
77.00
76.75
76.62
37.90
122.66
120.05
119.76
119.46
117.77
117.47
112.36
112.03
110.31
109.98
109.65
108.26
107.93

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
ppm

NAME	ESh824
EXPNO	13
PROCNO	1
USER	uralnmr
Date_	20210809
Time	18.01
INSTRUM	AV500
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
SOLVENT	CDCl3
TD	65536
SW	209.2368 ppm
O1P	100.000 ppm
FIDRES	0.401547 Hz
NS	24576
DS	8
AQ	1.2452340 sec
RG	203
TE	296.3 K
DE	6.50 usec
D1	1.00000000 sec
D11	0.03000000 sec
TD0	24
==== CHANNEL f1 ======	
NUC1	13C
P1	10.00 usec
PL1	0.00 dB
PL1W	115.29558563 W
SFO1	125.7703648 MHz
==== CHANNEL f2 ======	
CPDPRG2	waltz16
NUC2	1H
PCPD2	75.00 usec
PL2	120.00 dB
PL12	16.30 dB
PL13	19.30 dB
PL2W	0.00000000 W
PL12W	0.47519693 W
PL13W	0.23816262 W
SFO2	500.1320005 MHz
SI	32768
HZpPT	0.803094 Hz
SR	3.29 Hz
WDW	EM
LB	1.00 Hz
GB	0
SSB	0

^{19}F NMR spectrum of compound **6b** in $CDCl_3$



NAME	ESh824
EXPNO	19
PROCNO	1
USER	uralnmr
Date_	20210809
Time	17.44
INSTRUM	AV500
PROBHD	5 mm PABBO BB-
PULPROG	zg30
SOLVENT	CDCl3
TD	131072
SW	99.6202 ppm
O1P	45.000 ppm
FIDRES	0.357628 Hz
NS	16
DS	2
AQ	1.3981513 sec
RG	203
TE	295.1 K
DE	6.50 usec
D1	1.00000000 sec
TD0	1
==== CHANNEL f1 =====	
NUC1	19F
P1	15.50 usec
PL1	-5.00 dB
PL1W	46.07103729 W
SFO1	470.5370532 MHz
SI	131072
HZpPT	0.357628 Hz
SR	395.82 Hz
WDW	EM
LB	0.00 Hz
GB	0
SSB	0