

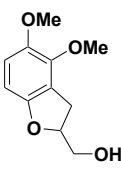
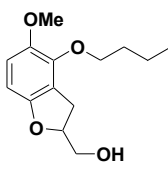
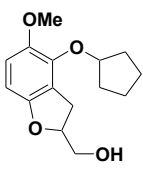
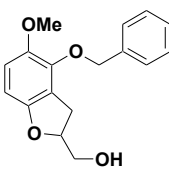
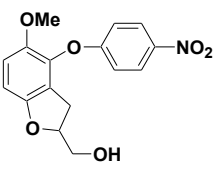
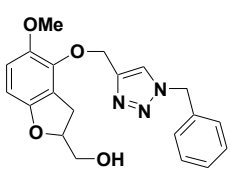
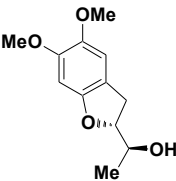
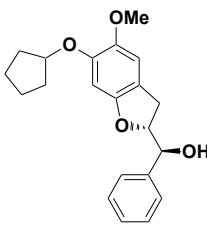
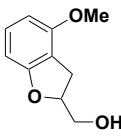
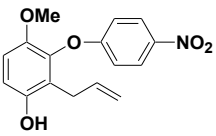
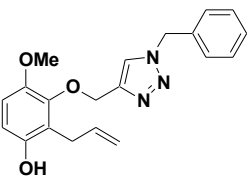
Supporting Information

Synthesis of 2-Hydroxymethyl-2,3-dihydrobenzofurans

Meng-Yang Chang,* Shin-Ying Lin and Chieh-Kai Chan

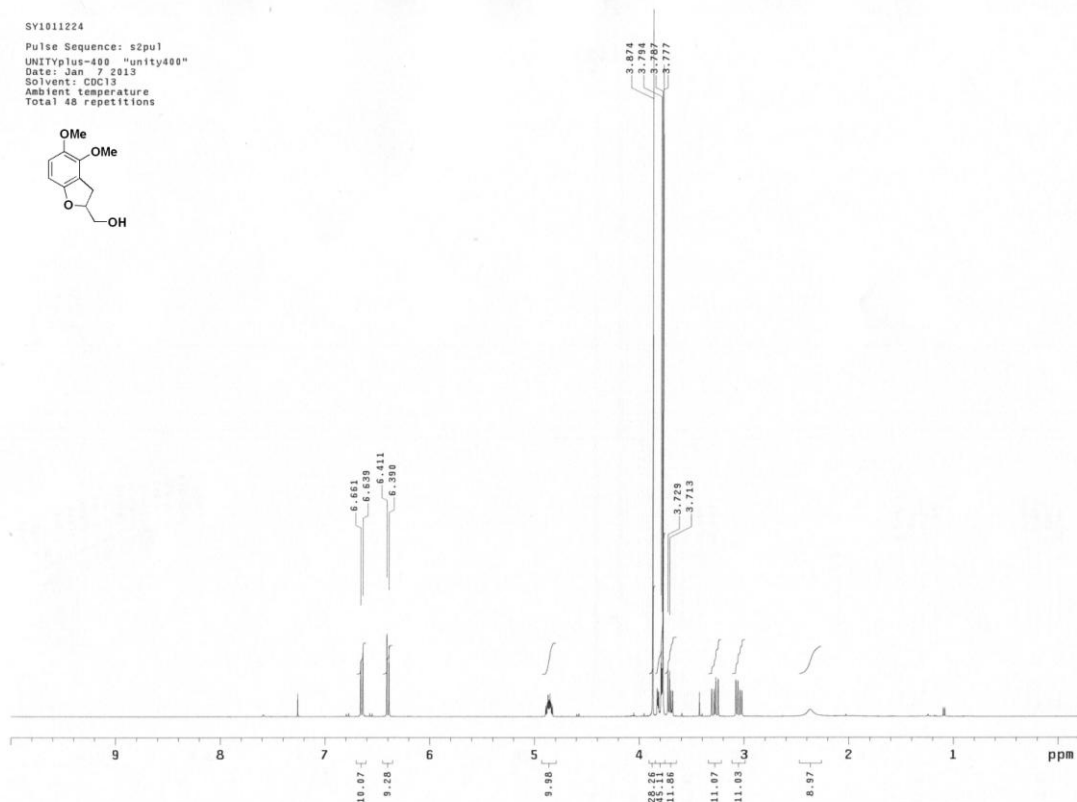
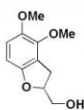
Department of Medicinal and Applied Chemistry, Kaohsiung Medical University, Kaohsiung 807, Taiwan

- (1) Additional scanned photocopies (pages S-2~S-12)
(2) X-ray crystal data of compound **2b** (pages S-13~S-20)

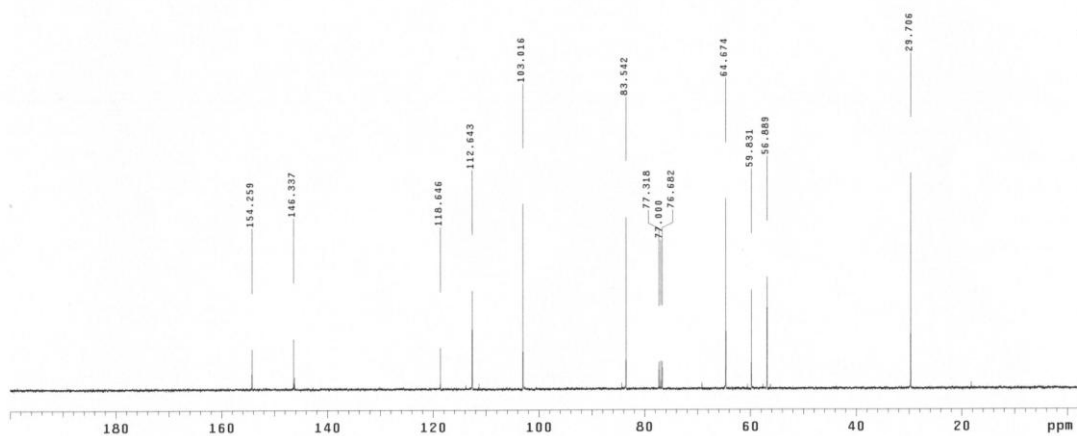
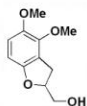
S-2 (1a) 	S-3 (1b) 	S-4 (1c) 	S-5 (1d) 	S-6 (1e) 
S-7 (1f) 	S-8 (1g) 	S-9 (1h) 	S-10 (1i) 	S-11 (2a) 
S-12 (2b) 				

Compound 1a

SV1011224
Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Jan 7 2013
Solvent: CDCl3
Ambient temperature
Total 48 repetitions

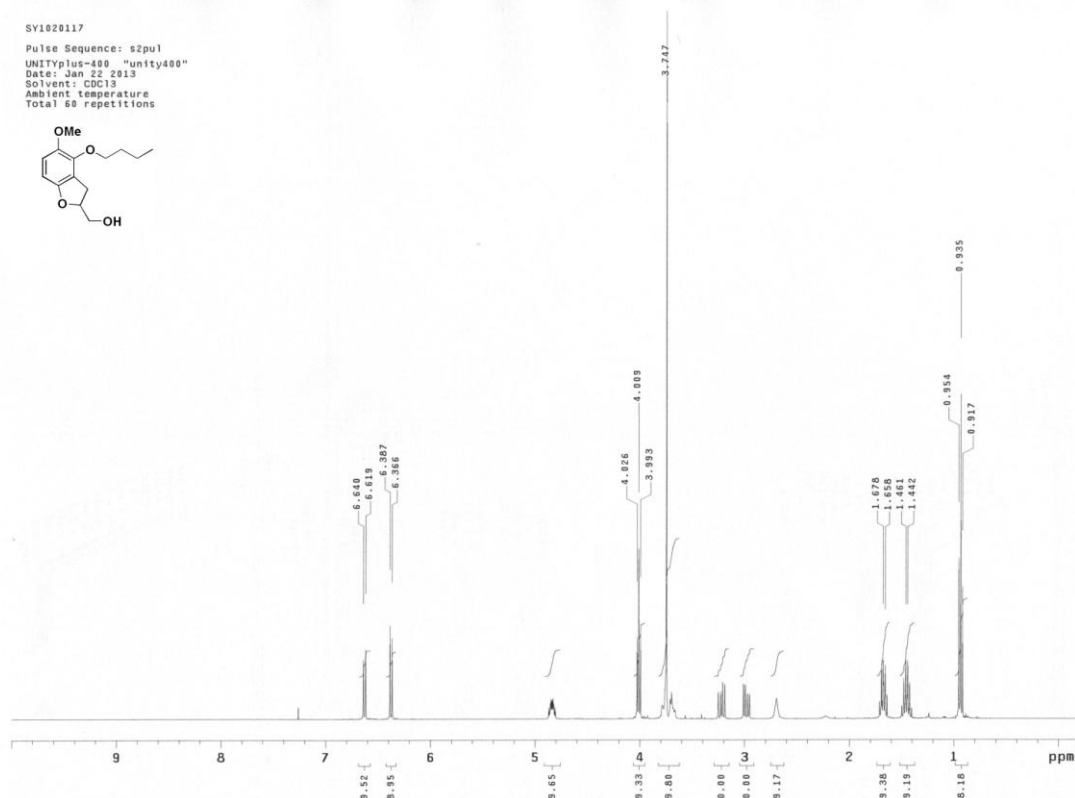
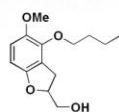


SV1011224
Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Jan 7 2013
Solvent: CDCl3
Ambient temperature
Total 752 repetitions

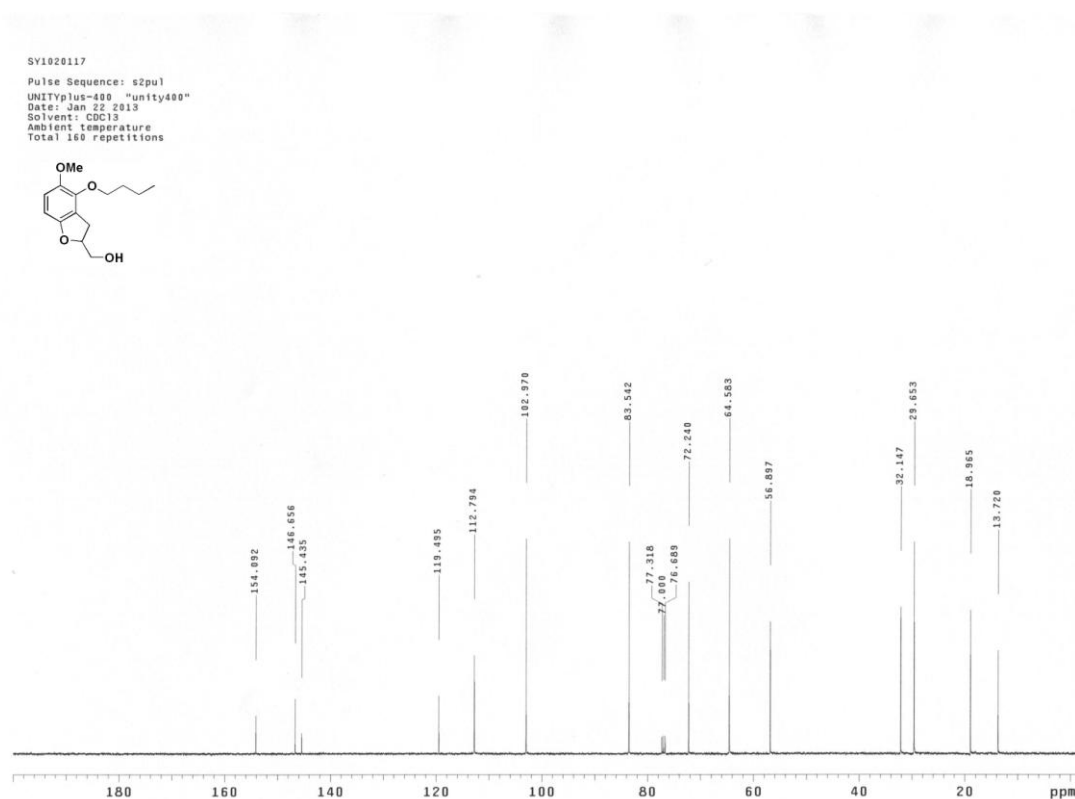
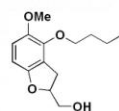


Compound 1b

SV1020117
Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Jan 22 2013
Solvent: CDCl3
Ambient temperature
Total 60 repetitions

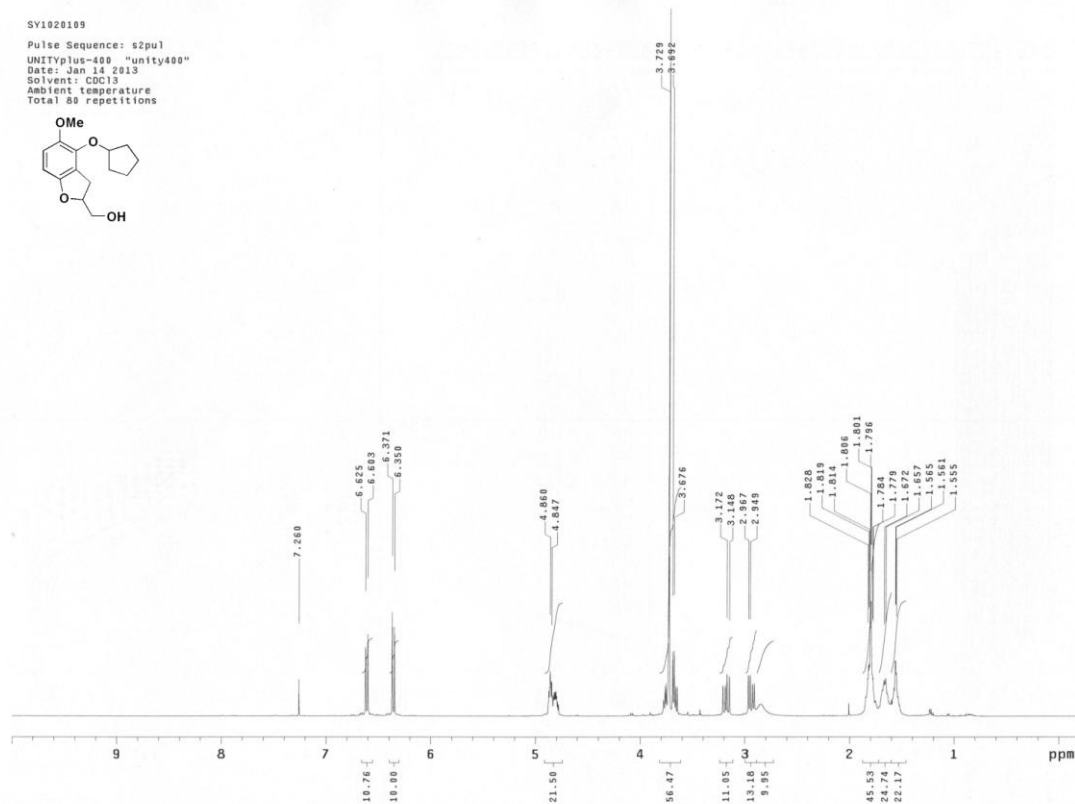
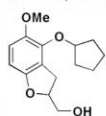


SV1020117
Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Jan 22 2013
Solvent: CDCl3
Ambient temperature
Total 160 repetitions

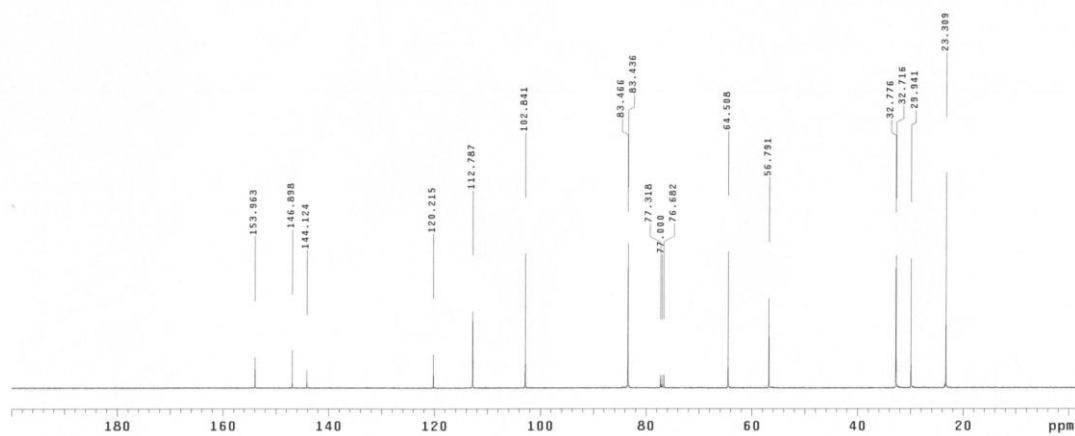
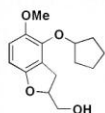


Compound 1c

SV1020109
Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Jan 14 2013
Solvent: CDCl3
Ambient temperature
Total 80 repetitions



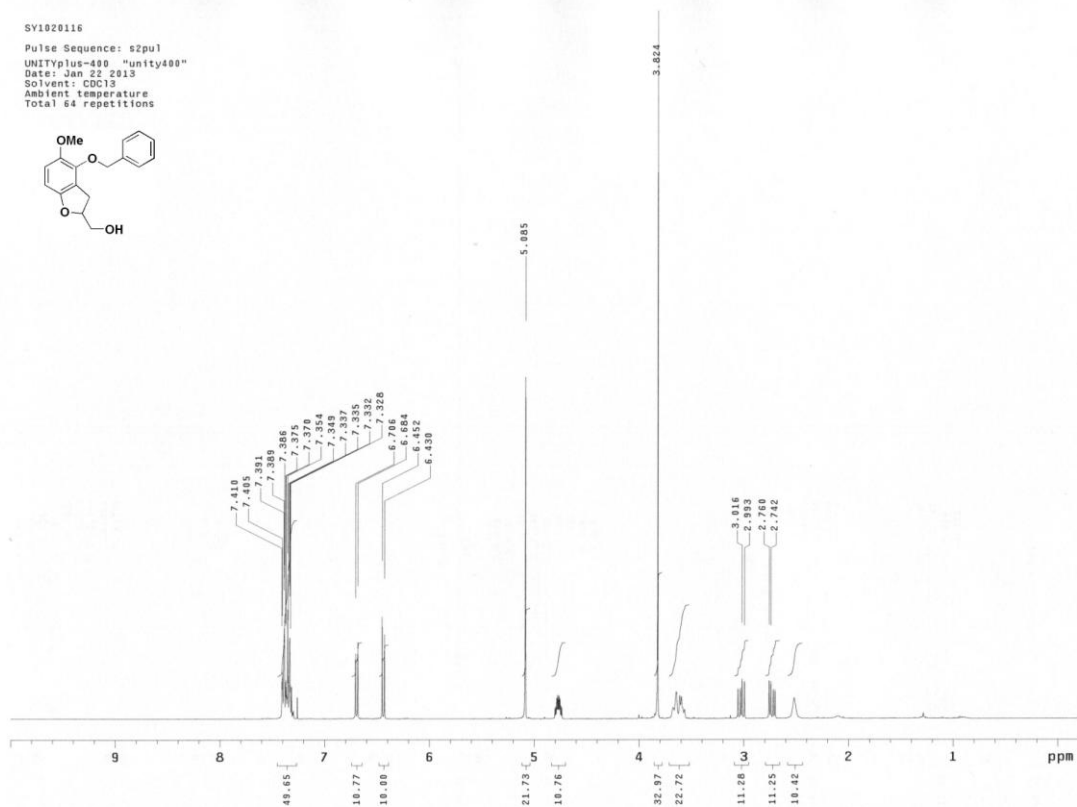
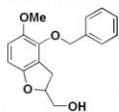
SV1020109
Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Jan 14 2013
Solvent: CDCl3
Ambient temperature
Total 512 repetitions



Compound 1d

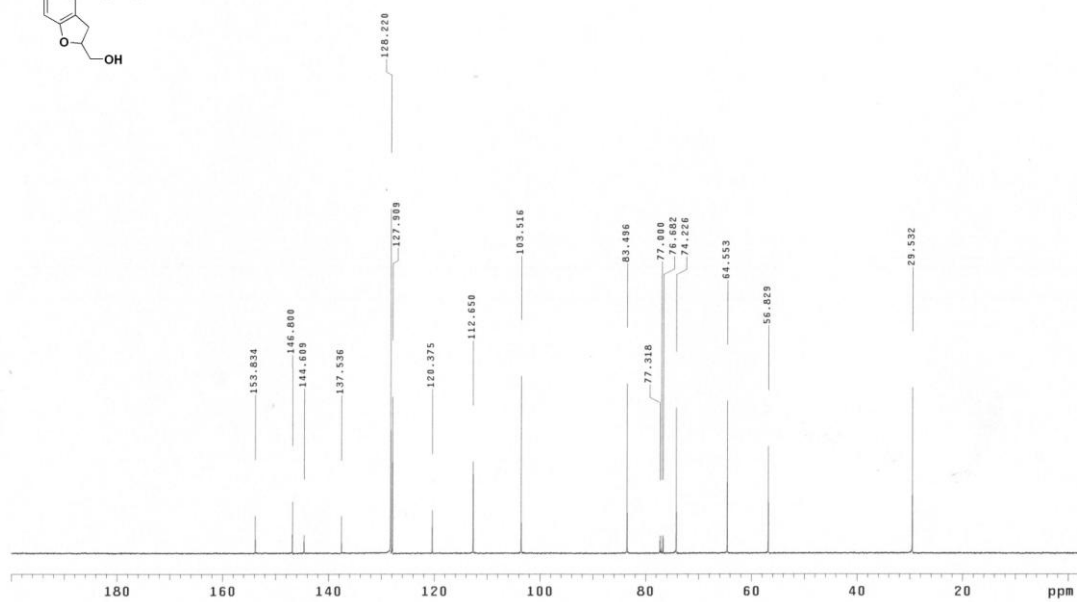
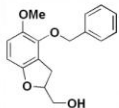
SV1020116

Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Jan 22 2013
Solvent: CDCl3
Ambient temperature
Total 64 repetitions



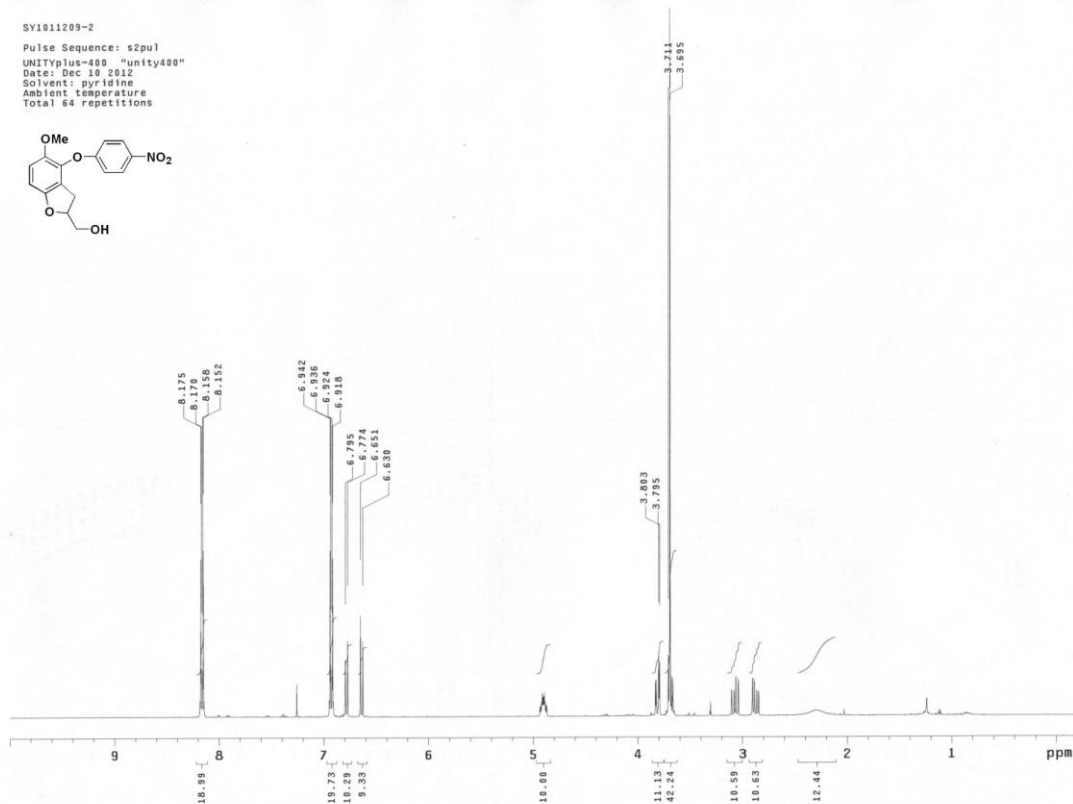
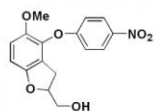
SV1020116

Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Jan 22 2013
Solvent: CDCl3
Ambient temperature
Total 512 repetitions

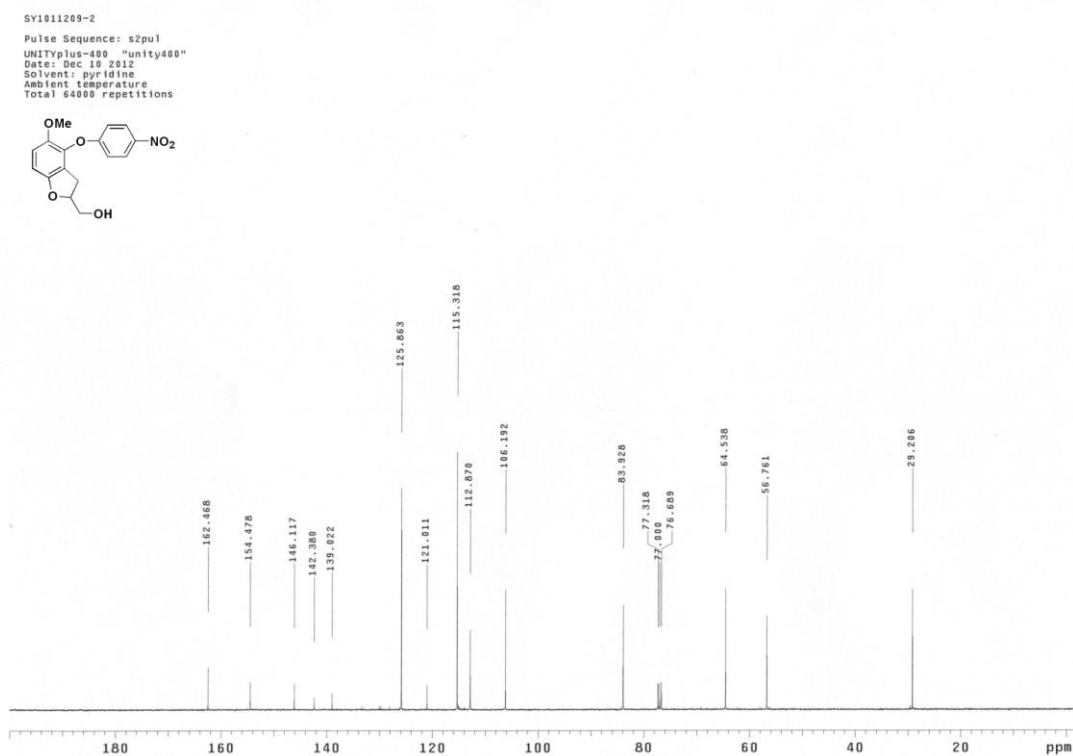
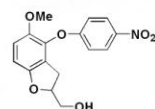


Compound 1e

SV1011209-2
Pulse Sequence: s2pul
UNITYplus-400 "unity400"
Date: Dec 10 2012
Solvent: pyridine
Ambient temperature
Total 64 repetitions

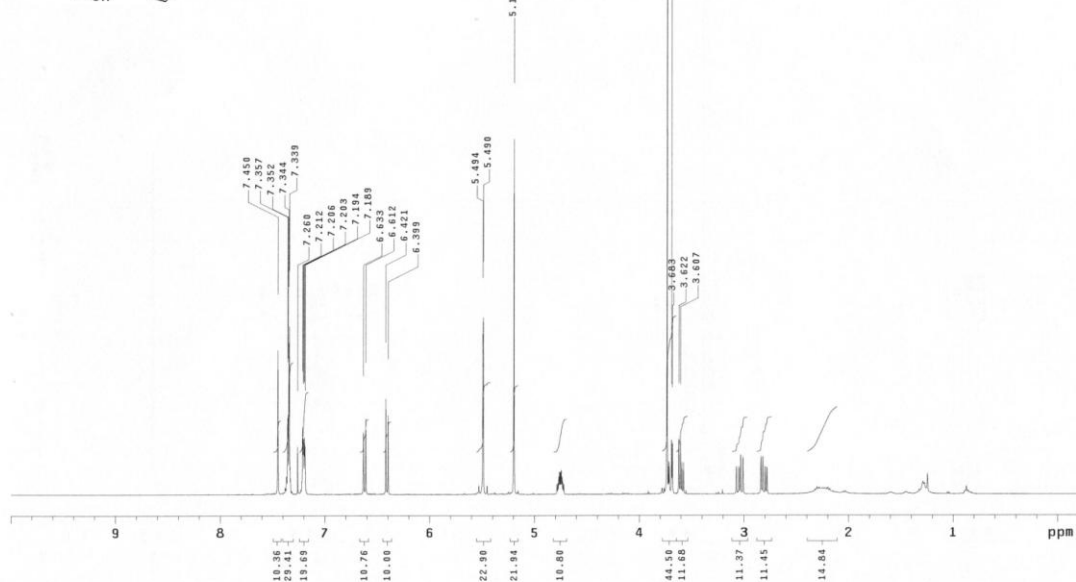
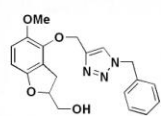


SV1011209-2
Pulse Sequence: s2pul
UNITYplus-400 "unity400"
Date: Dec 10 2012
Solvent: pyridine
Ambient temperature
Total 64000 repetitions

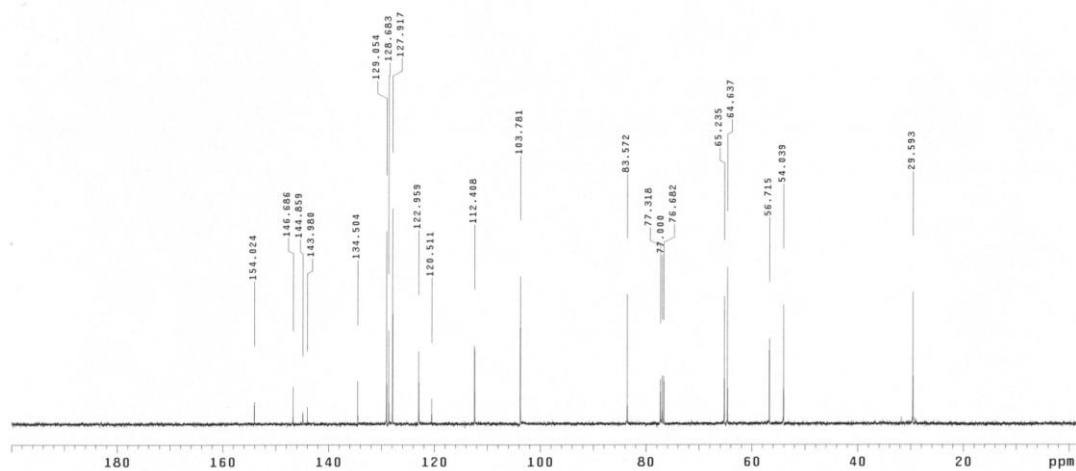
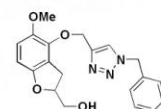


Compound 1f

SY1020131
 Pulse Sequence: s2pu1
 UNITYplus-400 "unity400"
 Date: Jan 31 2013
 Solvent: CDCl3
 Ambient temperature
 Total 32 repetitions

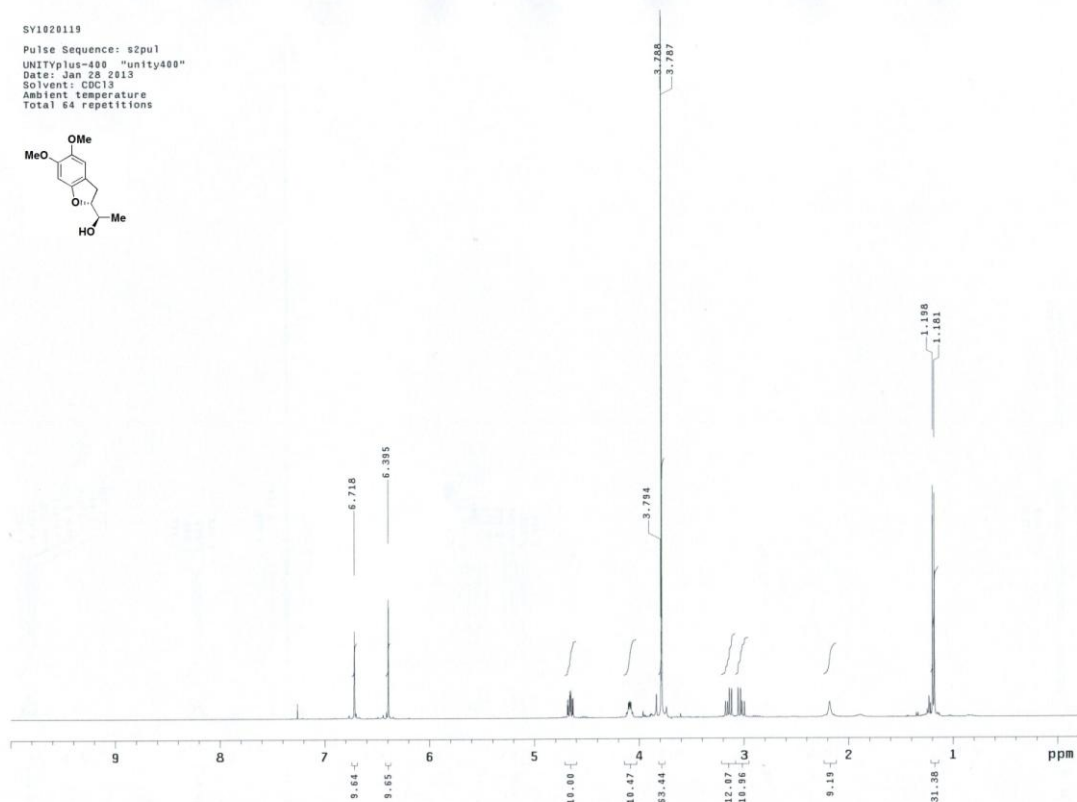
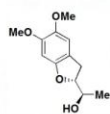


SY1020131
 Pulse Sequence: s2pu1
 UNITYplus-400 "unity400"
 Date: Jan 31 2013
 Solvent: CDCl3
 Ambient temperature
 Total 1008 repetitions

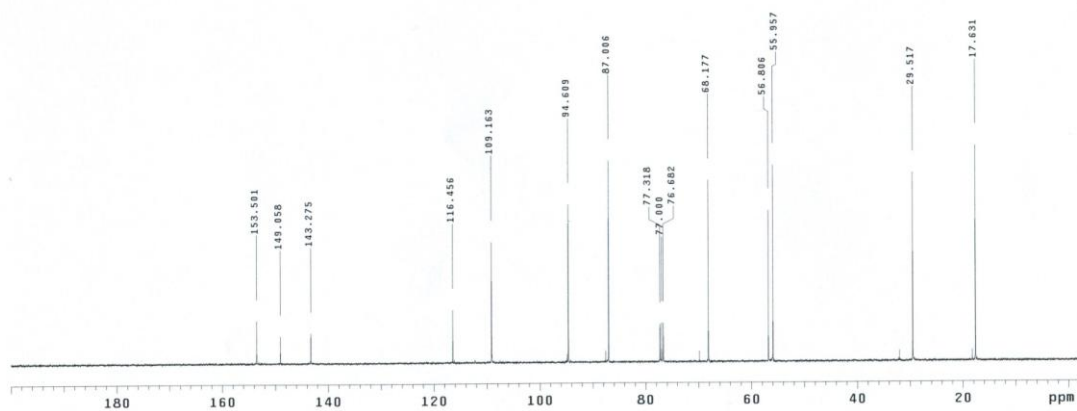
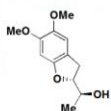


Compound 1g

SV1020119
Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Jan 28 2013
Solvent: CDCl3
Ambient temperature
Total 64 repetitions

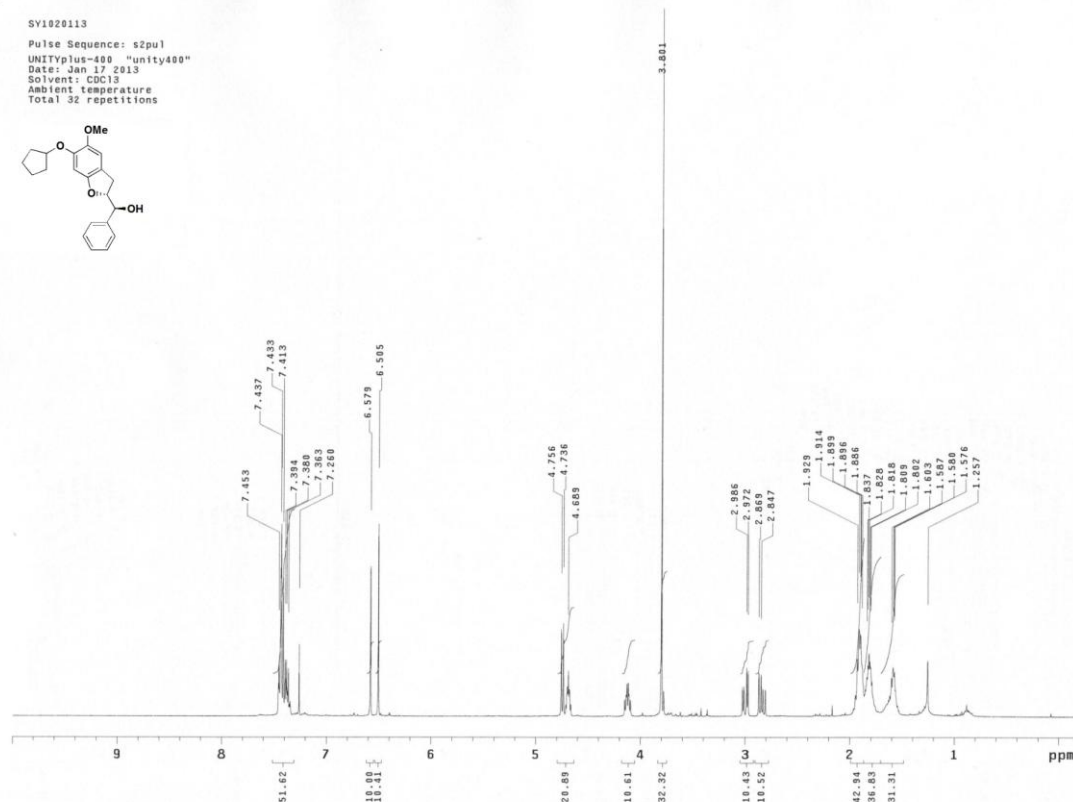
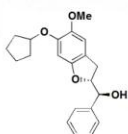


SK1020118
Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Jan 28 2013
Solvent: CDCl3
Ambient temperature
Total 1104 repetitions

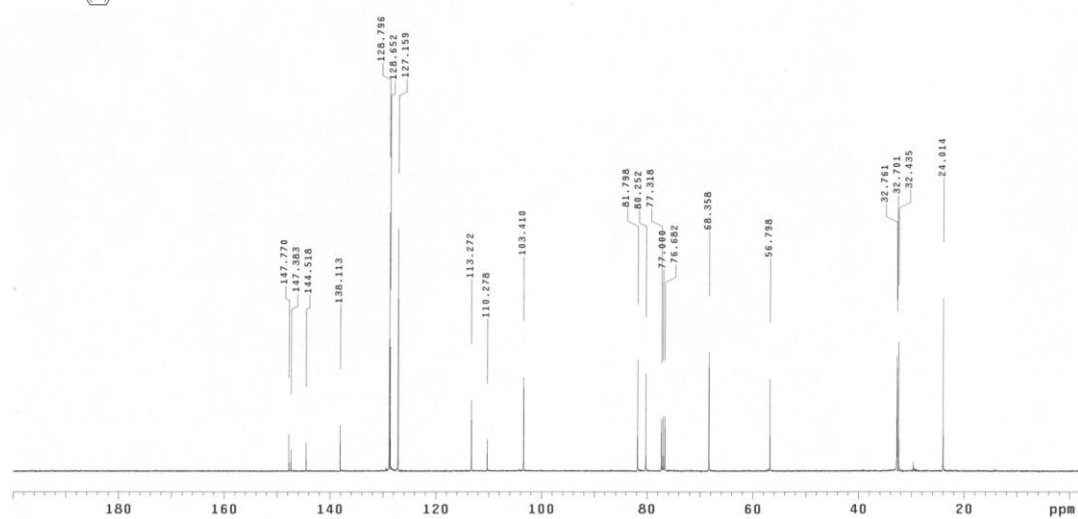
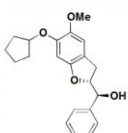


Compound 1h

SV1020113
Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Jan 17 2013
Solvent: CDCl3
Ambient temperature
Total 32 repetitions

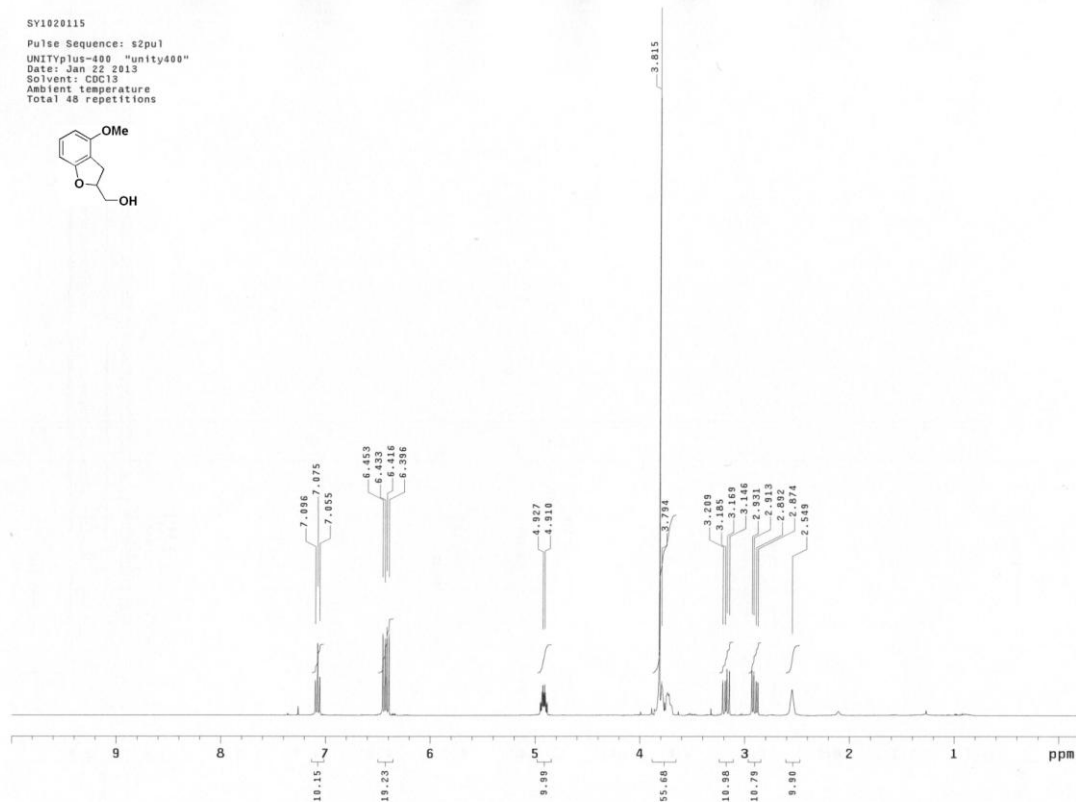


SV1020113
Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Jan 17 2013
Solvent: CDCl3
Ambient temperature
Total 10704 repetitions

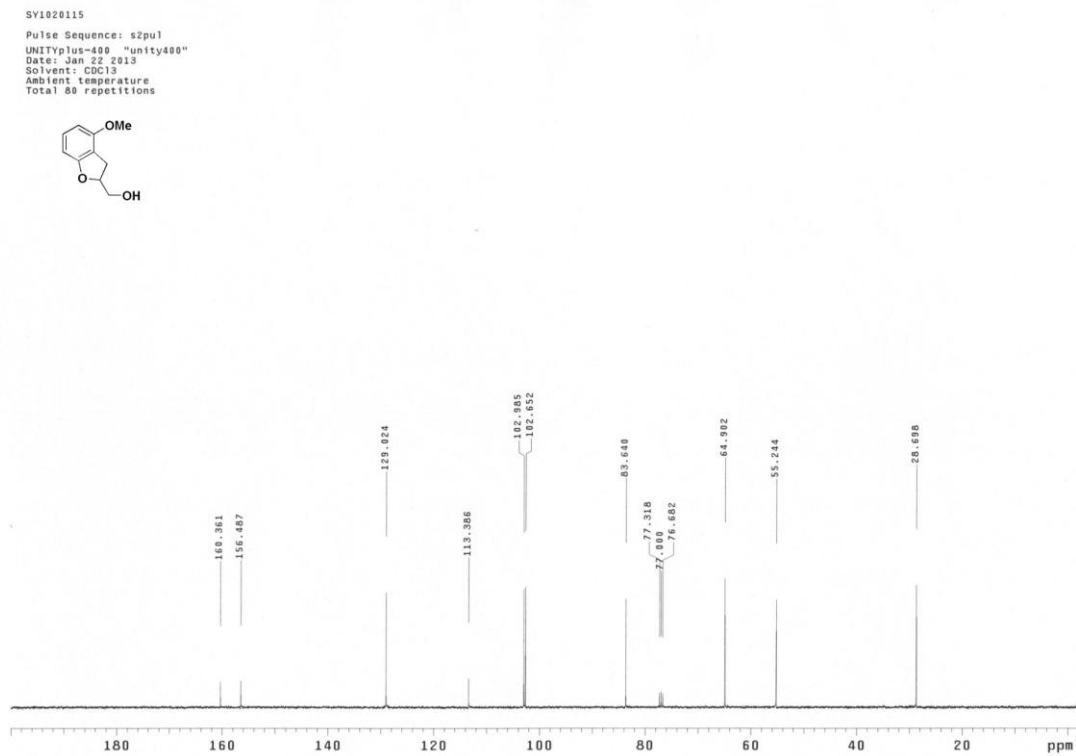


Compound 1i

SY1020115
Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Jan 22 2013
Solvent: CDCl3
Ambient temperature
Total 48 repetitions

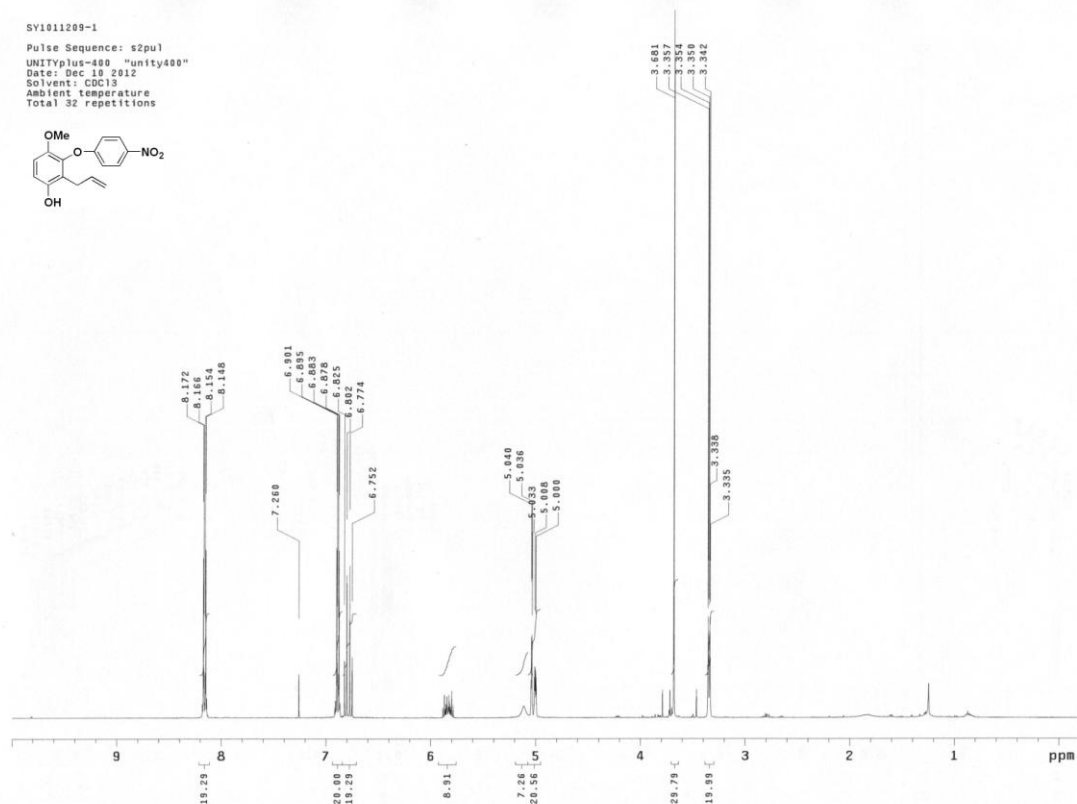
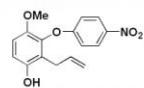


SY1020115
Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Jan 22 2013
Solvent: CDCl3
Ambient temperature
Total 88 repetitions

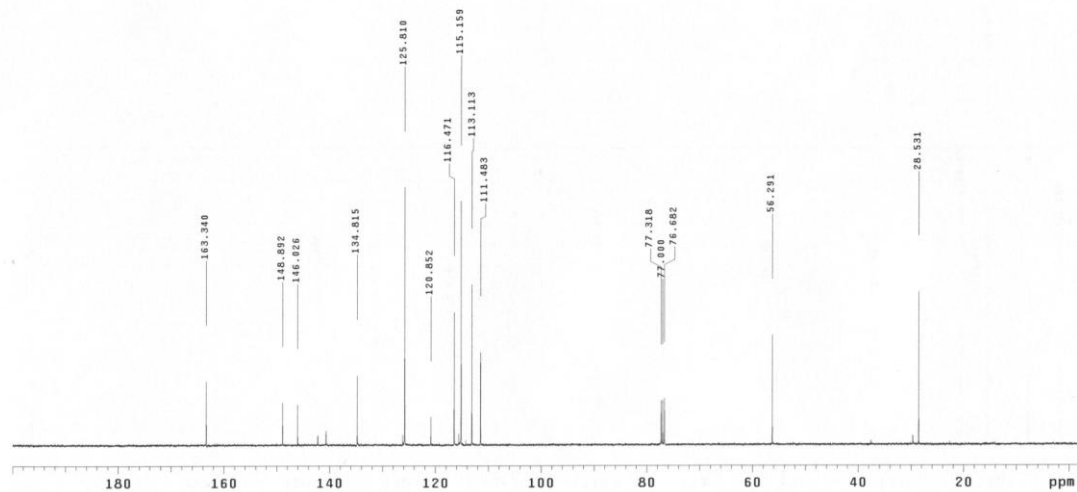
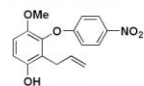


Compound 2a

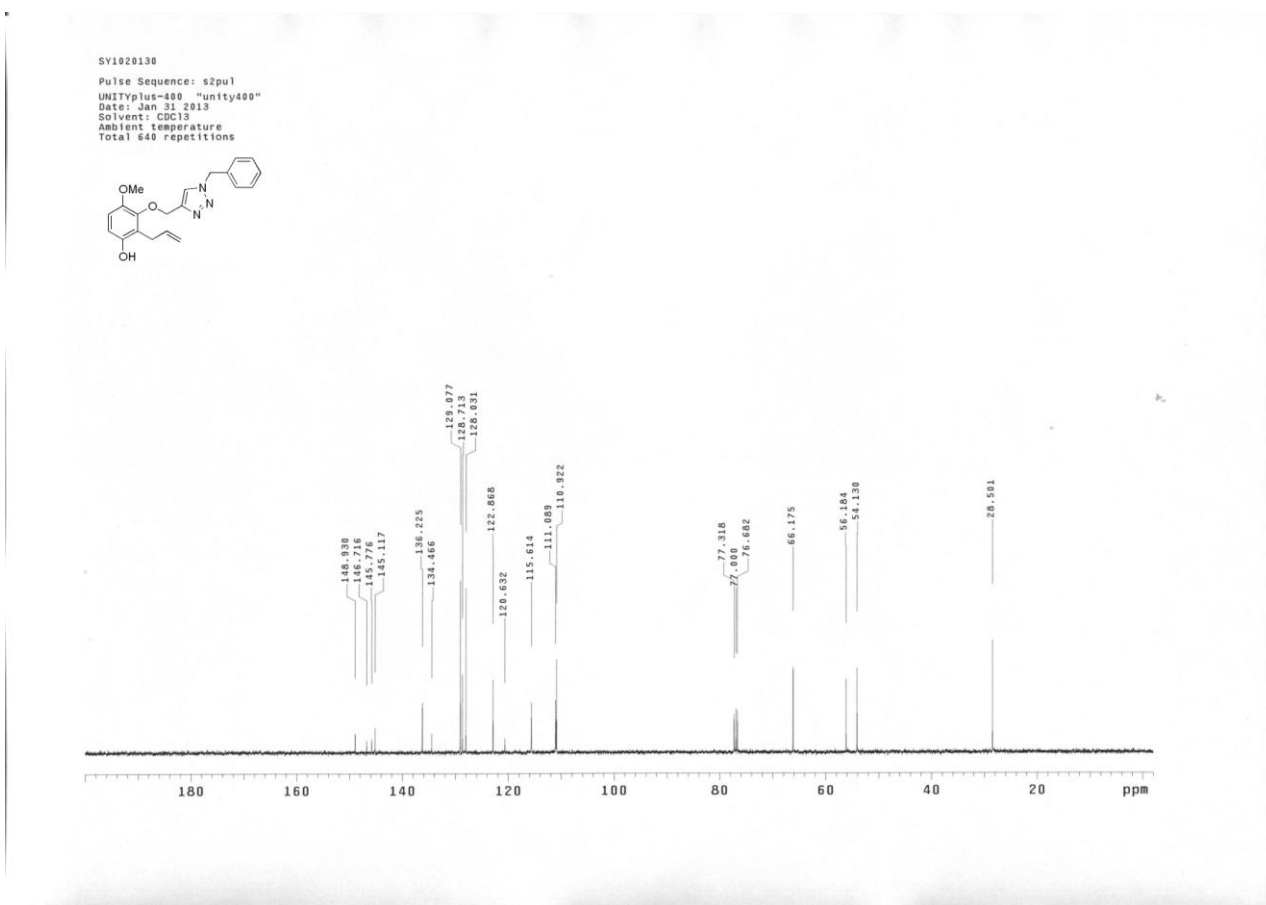
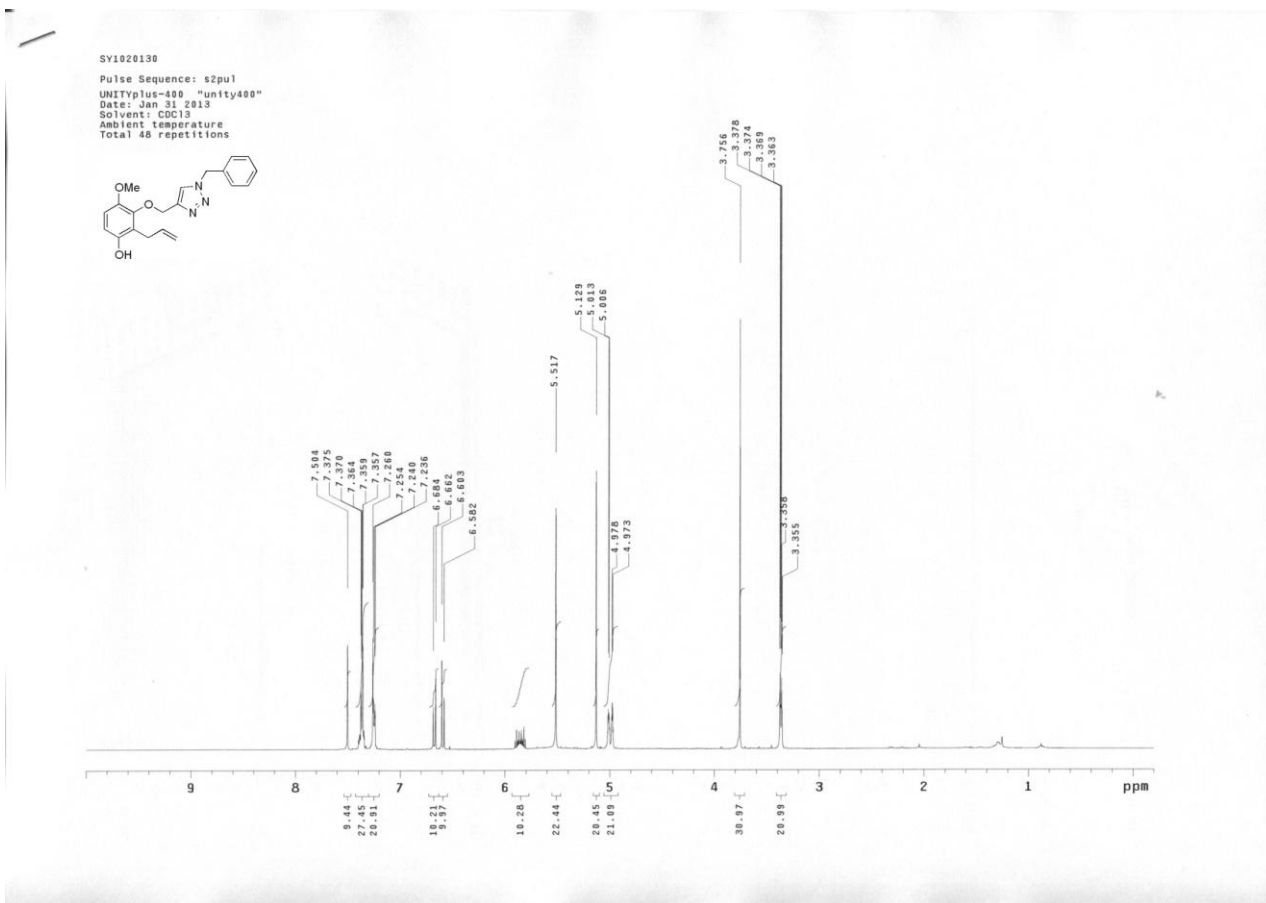
SV1011209-1
Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Dec 10 2012
Solvent: CDCl3
Ambient temperature
Total 32 repetitions



SV1011209-1
Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Dec 10 2012
Solvent: CDCl3
Ambient temperature
Total 2880 repetitions



Compound 2b



X-ray crystal data of compound 2b

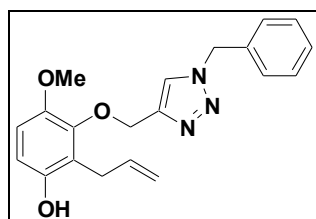


Table 1. Crystal data and structure refinement for mo_140319lt_0m_a.

Identification code	mo_140319lt_0m_a	
Empirical formula	C ₂₀ H ₂₁ N ₃ O ₃	
Formula weight	351.40	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	C 2/c	
Unit cell dimensions	a = 30.00(4) Å	α = 90°.
	b = 6.608(8) Å	β = 91.67(2)°.
	c = 18.39(2) Å	γ = 90°.
Volume	3643(8) Å ³	
Z	8	
Density (calculated)	1.281 Mg/m ³	
Absorption coefficient	0.088 mm ⁻¹	
F(000)	1488	
Crystal size	0.15 x 0.05 x 0.05 mm ³	
Theta range for data collection	1.358 to 27.376°.	
Index ranges	-37 ≤ h ≤ 38, -8 ≤ k ≤ 8, -22 ≤ l ≤ 23	
Reflections collected	15674	
Independent reflections	3955 [R(int) = 0.2217]	
Completeness to theta = 25.242°	100.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9485 and 0.4774	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	3955 / 0 / 238	
Goodness-of-fit on F ²	0.965	
Final R indices [I > 2σ(I)]	R1 = 0.1200, wR2 = 0.2820	
R indices (all data)	R1 = 0.2467, wR2 = 0.3585	
Extinction coefficient	0.024(3)	
Largest diff. peak and hole	0.579 and -0.411 e.Å ⁻³	

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for mo_140319lt_0m_a. $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	2165(2)	-1270(11)	8359(3)	44(2)
C(2)	2292(2)	-2383(11)	7748(3)	42(2)
C(3)	2041(2)	-2236(10)	7088(3)	40(2)
C(4)	1668(2)	-989(11)	7044(3)	45(2)
C(5)	1527(2)	118(11)	7661(3)	44(2)
C(6)	1781(2)	-36(11)	8307(3)	44(2)
C(7)	2756(2)	-2641(11)	9102(3)	45(2)
C(8)	1100(2)	1374(13)	7612(4)	56(2)
C(9)	1150(2)	3532(14)	7413(3)	60(2)
C(10)	933(3)	5101(14)	7701(5)	73(3)
C(11)	1909(2)	2637(11)	9178(3)	54(2)
C(12)	1624(2)	4028(12)	9602(3)	45(2)
C(13)	1447(2)	5842(12)	9402(3)	49(2)
C(14)	957(2)	8411(11)	10061(3)	49(2)
C(15)	542(2)	8426(13)	9558(3)	51(2)
C(16)	294(2)	6625(12)	9419(3)	52(2)
C(17)	-84(2)	6732(16)	8971(4)	73(3)
C(18)	-210(3)	8533(17)	8666(4)	79(3)
C(19)	31(3)	10301(17)	8783(4)	84(3)
C(20)	410(2)	10240(12)	9237(3)	55(2)
N(1)	1500(2)	3608(10)	10307(2)	55(2)
N(2)	1247(2)	5177(10)	10531(2)	54(2)
N(3)	1219(2)	6518(10)	9988(2)	49(2)
O(1)	2381(1)	-1325(7)	9032(2)	47(1)
O(2)	1627(1)	967(8)	8919(2)	51(1)
O(3)	1413(1)	-797(8)	6409(2)	52(1)

Table 3. Bond lengths [Å] and angles [°] for mo_140319lt_0m_a.

C(1)-O(1)	1.380(6)
C(1)-C(2)	1.406(8)
C(1)-C(6)	1.412(9)
C(2)-C(3)	1.412(7)
C(2)-H(2)	0.9500
C(3)-C(4)	1.388(8)
C(3)-H(3A)	0.9500
C(4)-O(3)	1.385(6)
C(4)-C(5)	1.425(9)
C(5)-C(6)	1.395(7)
C(5)-C(8)	1.528(9)
C(6)-O(2)	1.395(7)
C(7)-O(1)	1.424(7)
C(7)-H(7A)	0.9800
C(7)-H(7B)	0.9800
C(7)-H(7C)	0.9800
C(8)-C(9)	1.481(10)
C(8)-H(8A)	0.9900
C(8)-H(8B)	0.9900
C(9)-C(10)	1.341(11)
C(9)-H(9)	0.9500
C(10)-H(10A)	0.9500
C(10)-H(10B)	0.9500
C(11)-O(2)	1.462(8)
C(11)-C(12)	1.492(9)
C(11)-H(11A)	0.9900
C(11)-H(11B)	0.9900
C(12)-C(13)	1.357(10)
C(12)-N(1)	1.387(7)
C(13)-N(3)	1.369(7)
C(13)-H(13)	0.9500
C(14)-N(3)	1.486(9)
C(14)-C(15)	1.530(8)
C(14)-H(14A)	0.9900
C(14)-H(14B)	0.9900
C(15)-C(20)	1.388(10)
C(15)-C(16)	1.422(10)

C(16)-C(17)	1.384(9)
C(16)-H(16)	0.9500
C(17)-C(18)	1.365(12)
C(17)-H(17)	0.9500
C(18)-C(19)	1.387(13)
C(18)-H(18)	0.9500
C(19)-C(20)	1.391(11)
C(19)-H(19)	0.9500
C(20)-H(20)	0.9500
N(1)-N(2)	1.356(8)
N(2)-N(3)	1.336(7)
O(3)-H(3)	0.8400
O(1)-C(1)-C(2)	124.8(5)
O(1)-C(1)-C(6)	116.0(5)
C(2)-C(1)-C(6)	119.1(5)
C(1)-C(2)-C(3)	120.0(6)
C(1)-C(2)-H(2)	120.0
C(3)-C(2)-H(2)	120.0
C(4)-C(3)-C(2)	120.0(5)
C(4)-C(3)-H(3A)	120.0
C(2)-C(3)-H(3A)	120.0
O(3)-C(4)-C(3)	121.9(5)
O(3)-C(4)-C(5)	117.0(5)
C(3)-C(4)-C(5)	121.0(5)
C(6)-C(5)-C(4)	118.2(5)
C(6)-C(5)-C(8)	121.7(6)
C(4)-C(5)-C(8)	120.1(5)
C(5)-C(6)-O(2)	117.9(5)
C(5)-C(6)-C(1)	121.6(5)
O(2)-C(6)-C(1)	120.4(5)
O(1)-C(7)-H(7A)	109.5
O(1)-C(7)-H(7B)	109.5
H(7A)-C(7)-H(7B)	109.5
O(1)-C(7)-H(7C)	109.5
H(7A)-C(7)-H(7C)	109.5
H(7B)-C(7)-H(7C)	109.5
C(9)-C(8)-C(5)	116.5(6)
C(9)-C(8)-H(8A)	108.2

C(5)-C(8)-H(8A)	108.2
C(9)-C(8)-H(8B)	108.2
C(5)-C(8)-H(8B)	108.2
H(8A)-C(8)-H(8B)	107.3
C(10)-C(9)-C(8)	126.4(7)
C(10)-C(9)-H(9)	116.8
C(8)-C(9)-H(9)	116.8
C(9)-C(10)-H(10A)	120.0
C(9)-C(10)-H(10B)	120.0
H(10A)-C(10)-H(10B)	120.0
O(2)-C(11)-C(12)	107.5(5)
O(2)-C(11)-H(11A)	110.2
C(12)-C(11)-H(11A)	110.2
O(2)-C(11)-H(11B)	110.2
C(12)-C(11)-H(11B)	110.2
H(11A)-C(11)-H(11B)	108.5
C(13)-C(12)-N(1)	108.5(6)
C(13)-C(12)-C(11)	128.9(5)
N(1)-C(12)-C(11)	122.6(7)
C(12)-C(13)-N(3)	105.9(5)
C(12)-C(13)-H(13)	127.0
N(3)-C(13)-H(13)	127.0
N(3)-C(14)-C(15)	112.0(5)
N(3)-C(14)-H(14A)	109.2
C(15)-C(14)-H(14A)	109.2
N(3)-C(14)-H(14B)	109.2
C(15)-C(14)-H(14B)	109.2
H(14A)-C(14)-H(14B)	107.9
C(20)-C(15)-C(16)	120.4(6)
C(20)-C(15)-C(14)	118.6(7)
C(16)-C(15)-C(14)	120.9(7)
C(17)-C(16)-C(15)	118.5(8)
C(17)-C(16)-H(16)	120.7
C(15)-C(16)-H(16)	120.7
C(18)-C(17)-C(16)	120.2(9)
C(18)-C(17)-H(17)	119.9
C(16)-C(17)-H(17)	119.9
C(17)-C(18)-C(19)	122.2(8)
C(17)-C(18)-H(18)	118.9

C(19)-C(18)-H(18)	118.9
C(18)-C(19)-C(20)	118.8(8)
C(18)-C(19)-H(19)	120.6
C(20)-C(19)-H(19)	120.6
C(15)-C(20)-C(19)	119.8(8)
C(15)-C(20)-H(20)	120.1
C(19)-C(20)-H(20)	120.1
N(2)-N(1)-C(12)	107.4(6)
N(3)-N(2)-N(1)	107.7(5)
N(2)-N(3)-C(13)	110.5(6)
N(2)-N(3)-C(14)	120.9(5)
C(13)-N(3)-C(14)	128.6(5)
C(1)-O(1)-C(7)	116.5(4)
C(6)-O(2)-C(11)	114.8(4)
C(4)-O(3)-H(3)	109.5

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for mo_140319lt_0m_a. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	U ¹¹	U ²²	U ³³	U ²³	U ¹³	U ¹²
C(1)	24(3)	88(6)	18(2)	-1(3)	-5(2)	-2(3)
C(2)	17(3)	92(6)	18(2)	3(3)	-3(2)	-4(3)
C(3)	20(3)	79(5)	22(2)	1(3)	-7(2)	2(3)
C(4)	24(3)	88(6)	23(3)	0(3)	-13(2)	-1(3)
C(5)	16(3)	76(5)	41(3)	5(3)	-8(2)	-4(3)
C(6)	22(3)	89(6)	22(3)	-7(3)	-1(2)	-5(3)
C(7)	24(3)	86(6)	24(3)	0(3)	-8(2)	6(3)
C(8)	16(3)	97(7)	53(4)	6(4)	-13(3)	5(4)
C(9)	38(4)	102(7)	39(3)	14(4)	7(3)	15(5)
C(10)	40(4)	94(7)	85(6)	3(5)	-15(4)	-1(5)
C(11)	29(3)	93(6)	39(3)	-10(3)	-3(3)	-1(4)
C(12)	25(3)	85(6)	25(3)	-6(3)	-7(2)	-7(4)
C(13)	26(3)	101(6)	20(3)	2(3)	1(2)	-5(4)
C(14)	40(4)	71(5)	36(3)	-10(3)	-4(3)	-7(4)
C(15)	33(3)	96(6)	25(3)	-3(3)	6(3)	8(4)
C(16)	38(4)	78(6)	39(3)	-5(3)	-7(3)	2(4)
C(17)	25(3)	137(9)	56(4)	-7(5)	-11(3)	7(4)
C(18)	39(4)	139(10)	59(5)	23(5)	-9(4)	22(6)
C(19)	45(5)	136(10)	70(5)	34(5)	13(4)	21(6)
C(20)	41(4)	75(6)	51(4)	6(4)	11(3)	16(4)
N(1)	29(3)	110(6)	26(2)	8(3)	-12(2)	-1(3)
N(2)	36(3)	102(5)	24(2)	2(3)	-5(2)	-4(3)
N(3)	29(3)	102(5)	17(2)	5(3)	-7(2)	-2(3)
O(1)	23(2)	99(4)	18(2)	-1(2)	-6(2)	3(2)
O(2)	23(2)	92(4)	39(2)	-6(2)	1(2)	-10(2)
O(3)	29(2)	91(4)	34(2)	-2(2)	-20(2)	3(2)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^{-3}$) for mo_140319lt_0m_a.

	x	y	z	U(eq)
H(2)	2547	-3234	7778	51
H(3A)	2127	-2989	6675	48
H(7A)	2660	-4038	9012	67
H(7B)	2887	-2533	9595	67
H(7C)	2979	-2256	8748	67
H(8A)	894	732	7250	67
H(8B)	956	1309	8089	67
H(9)	1355	3833	7045	71
H(10A)	724	4878	8070	88
H(10B)	989	6437	7535	88
H(11A)	2036	3364	8760	64
H(11B)	2158	2116	9489	64
H(13)	1476	6505	8947	59
H(14A)	1147	9583	9944	59
H(14B)	865	8556	10571	59
H(16)	387	5375	9630	62
H(17)	-256	5549	8875	87
H(18)	-472	8578	8363	95
H(19)	-61	11531	8558	100
H(20)	578	11437	9327	66
H(3)	1501	-1629	6099	78