Supplementary Material

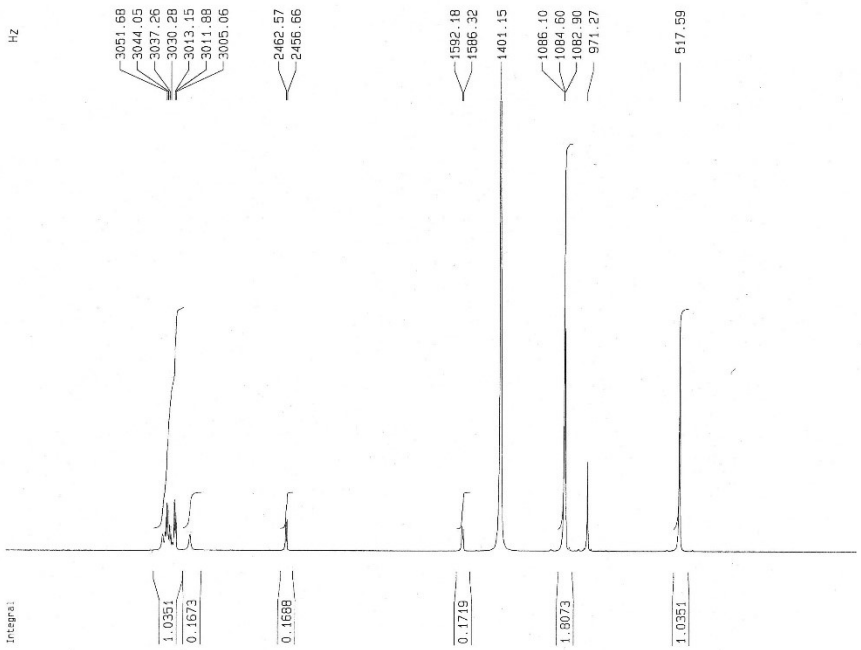
Regio- and diastereoselective synthesis of *trans*-dihydrofuran-3-carboxamides by radical addition of 1,3-dicarbonyl compounds to acrylamides using manganese(III) acetate and determination of exact configuration by X-ray crystallography

Mehmet Yilmaz,[a*] Aslı Ustalar,[a] Bedriye Uçan,[b] and A. Tarık Pekel[b]

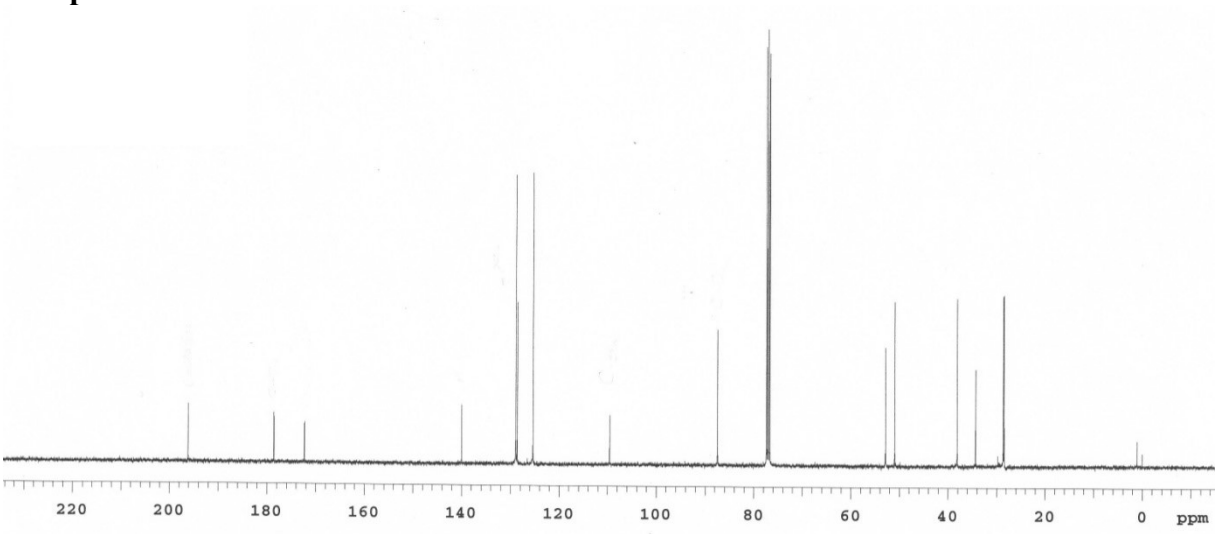
[1]Department of Chemistry, Faculty of Arts and Sciences, Kocaeli University, 41380 Umuttepe,Kocaeli, Turkey
[2]Department of Chemistry, Faculty of Science, Ankara University, 06100 Tandogan, Ankara, Turkey
E-mail: mehmet.yilmaz@kocaeli.edu.tr

Contents

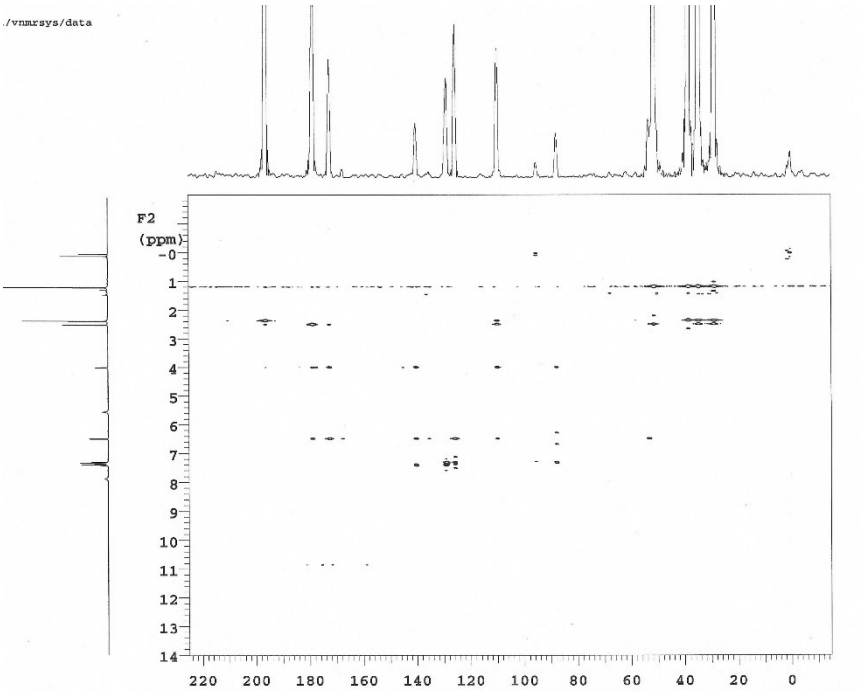
Compound 7a ^{1}H NMR



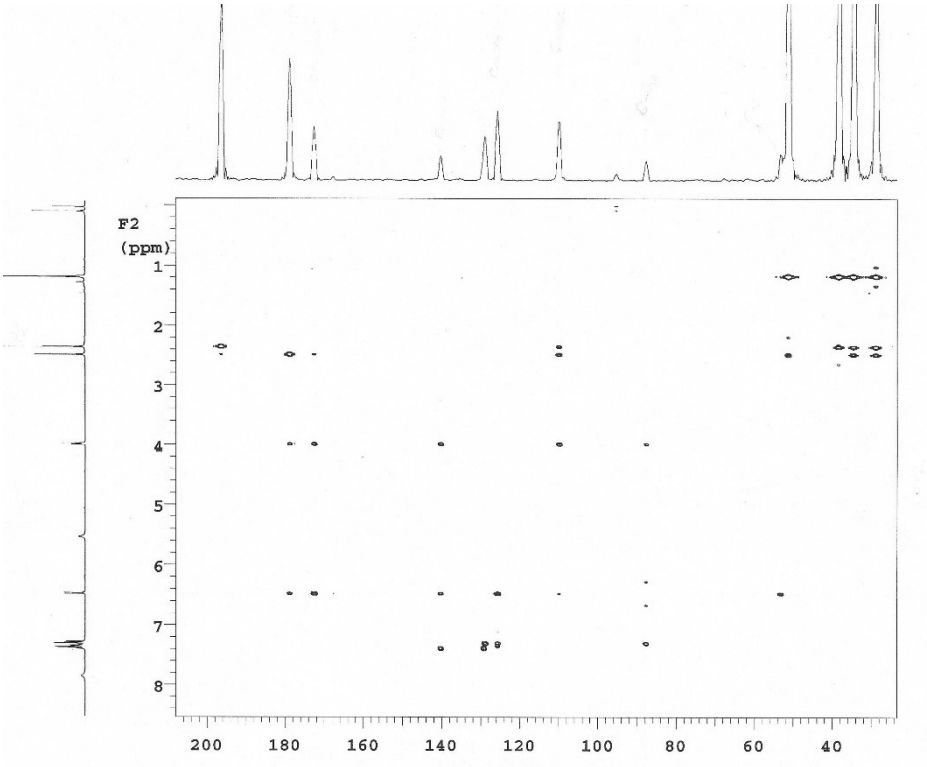
Compound 7a ^{13}C NMR



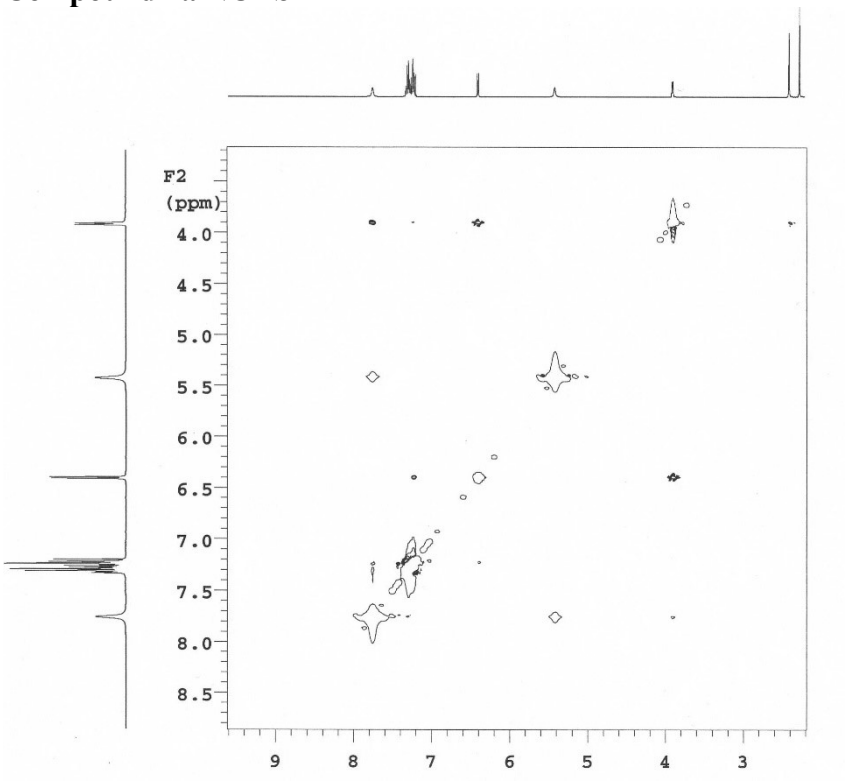
Compound 7a HMBC



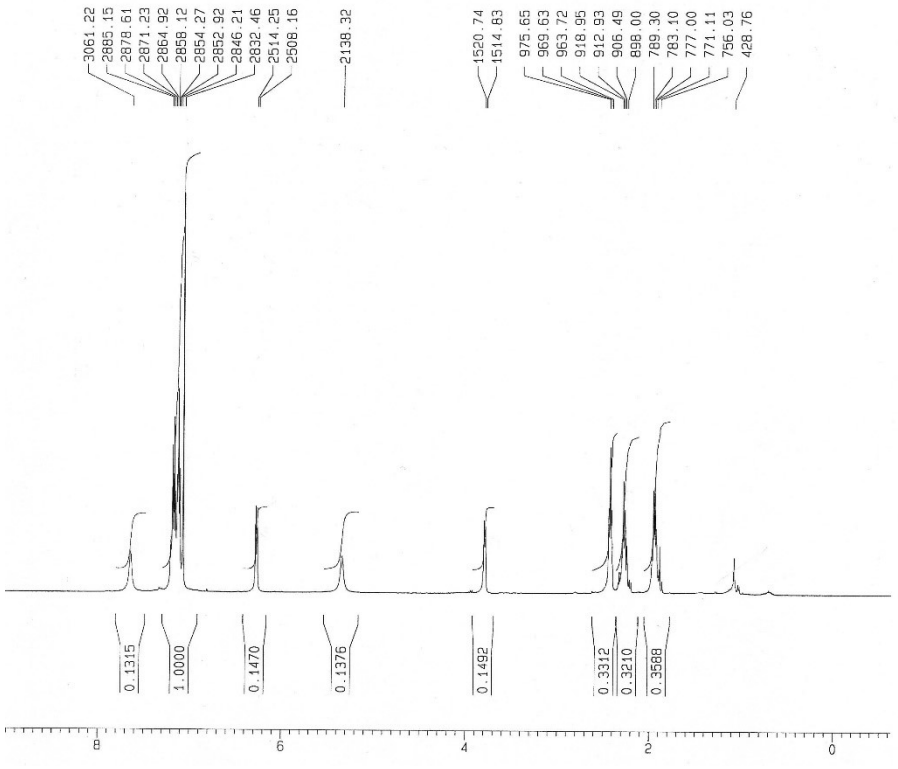
Compound 7a HMBC Expended



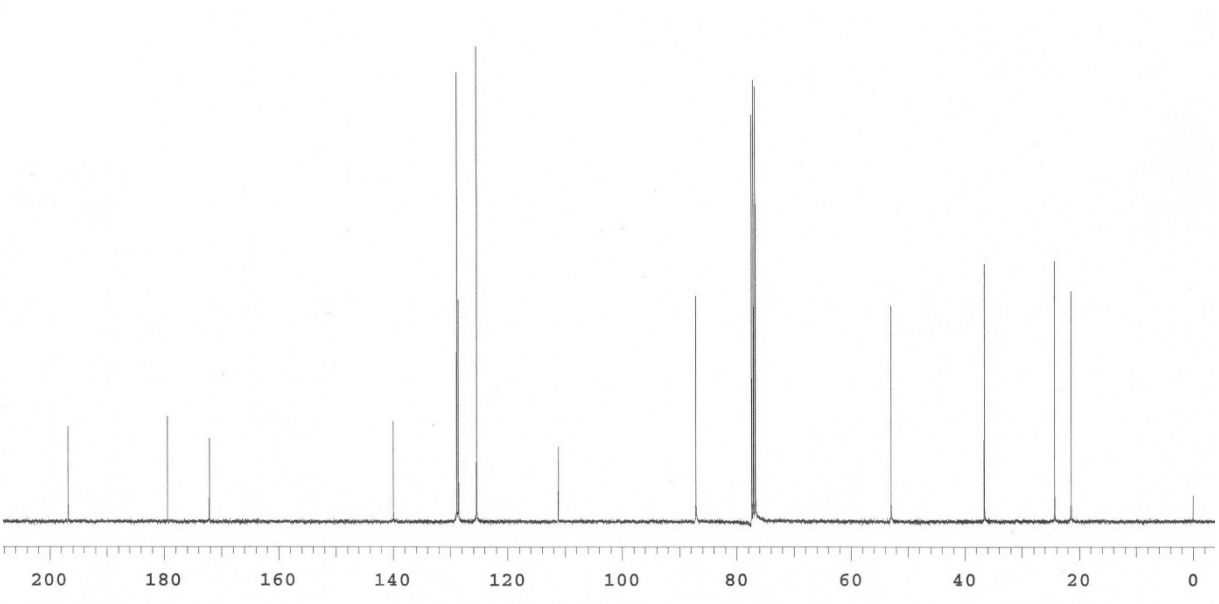
Compound 7a NOESY



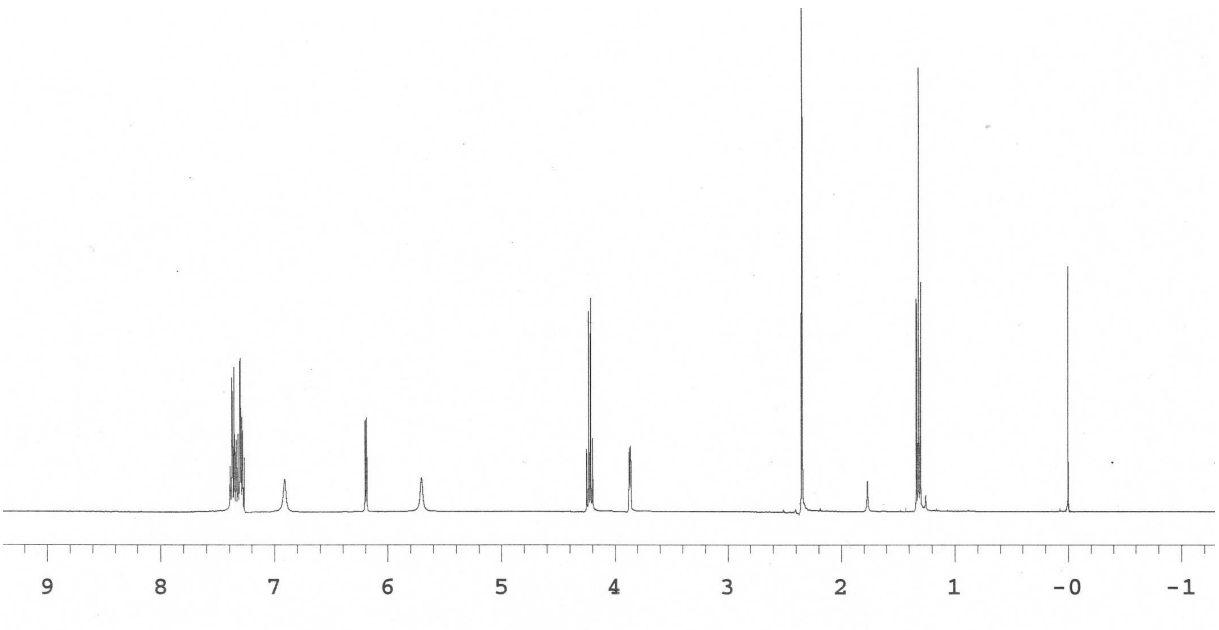
Compound 7b ^{1}H NMR



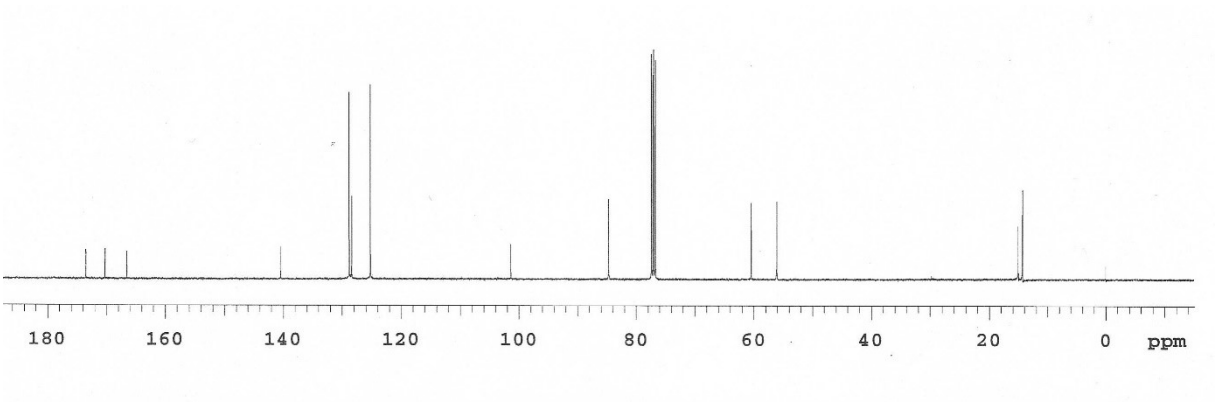
Compound 7b ^{13}C NMR



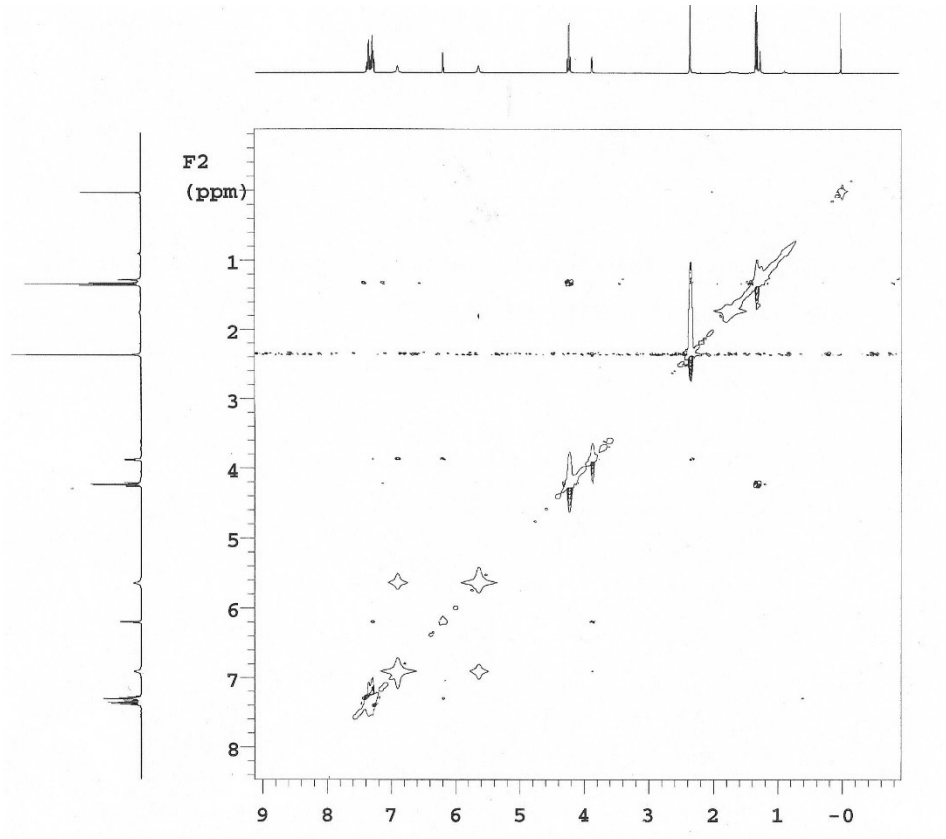
Compound 7c ^{1}H NMR



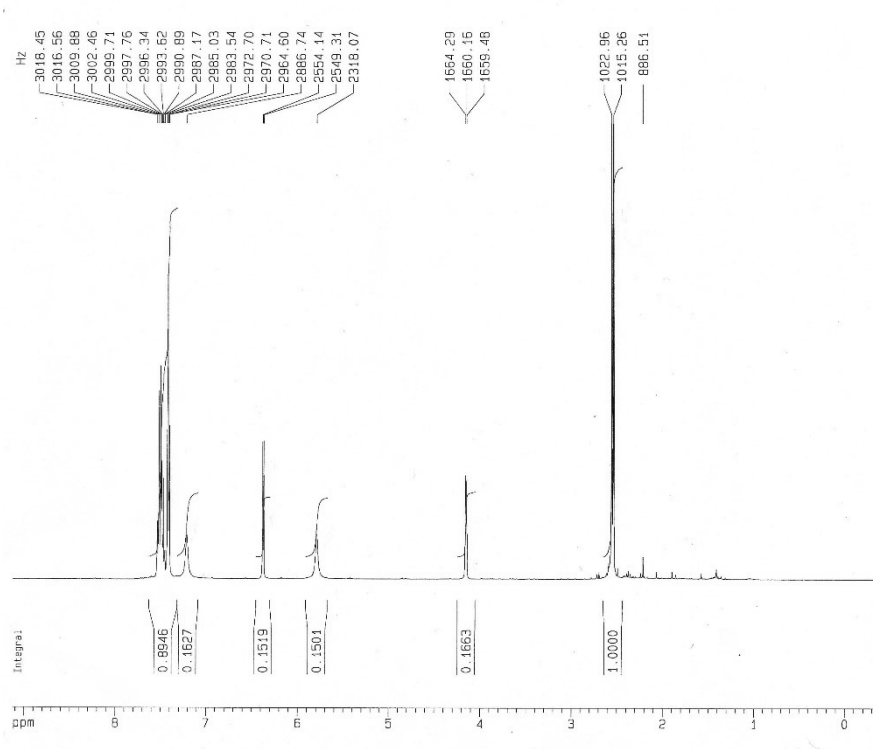
Compound 7c ^{13}C NMR



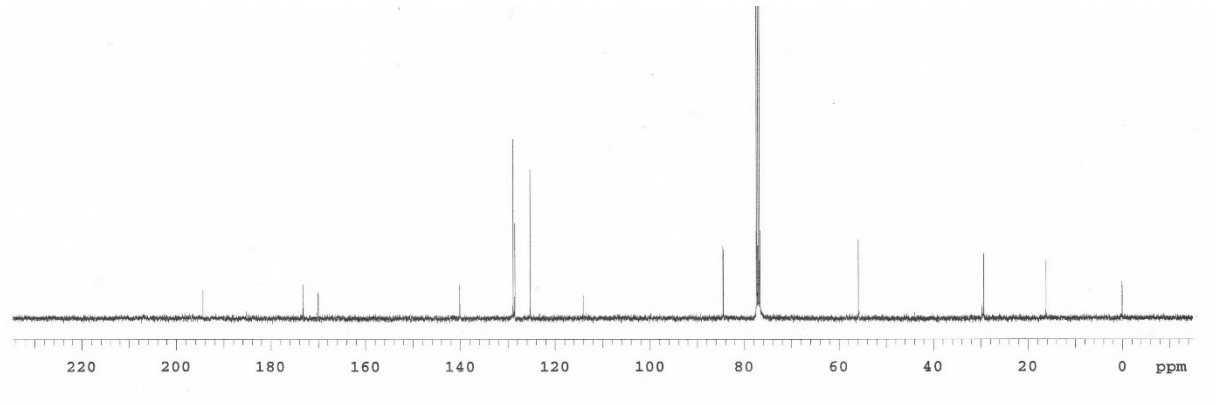
Compound 7c NOESY



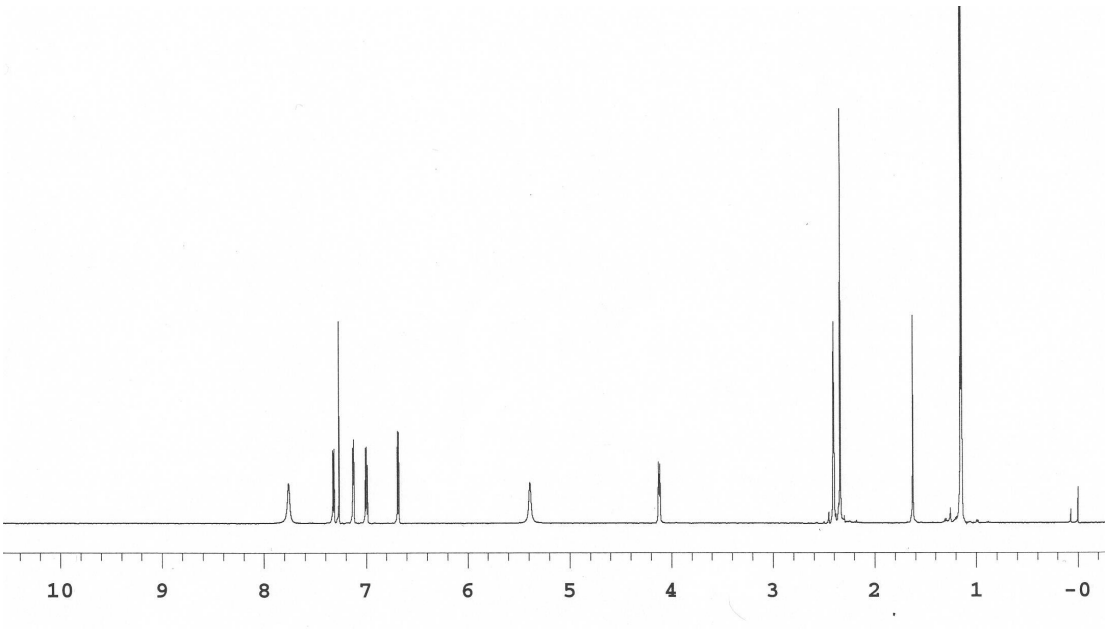
Compound 7d ^{1}H NMR



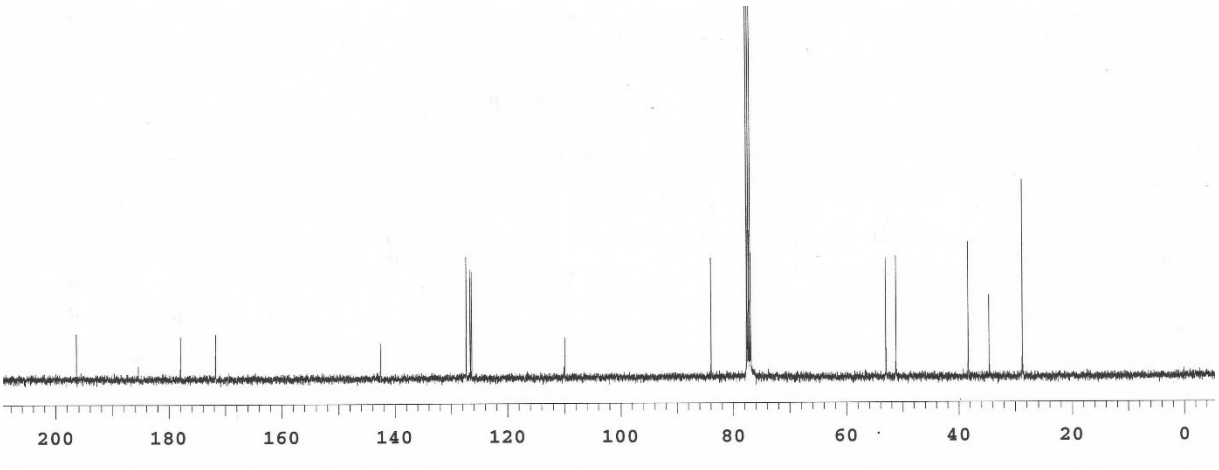
Compound 7d ^{13}C NMR



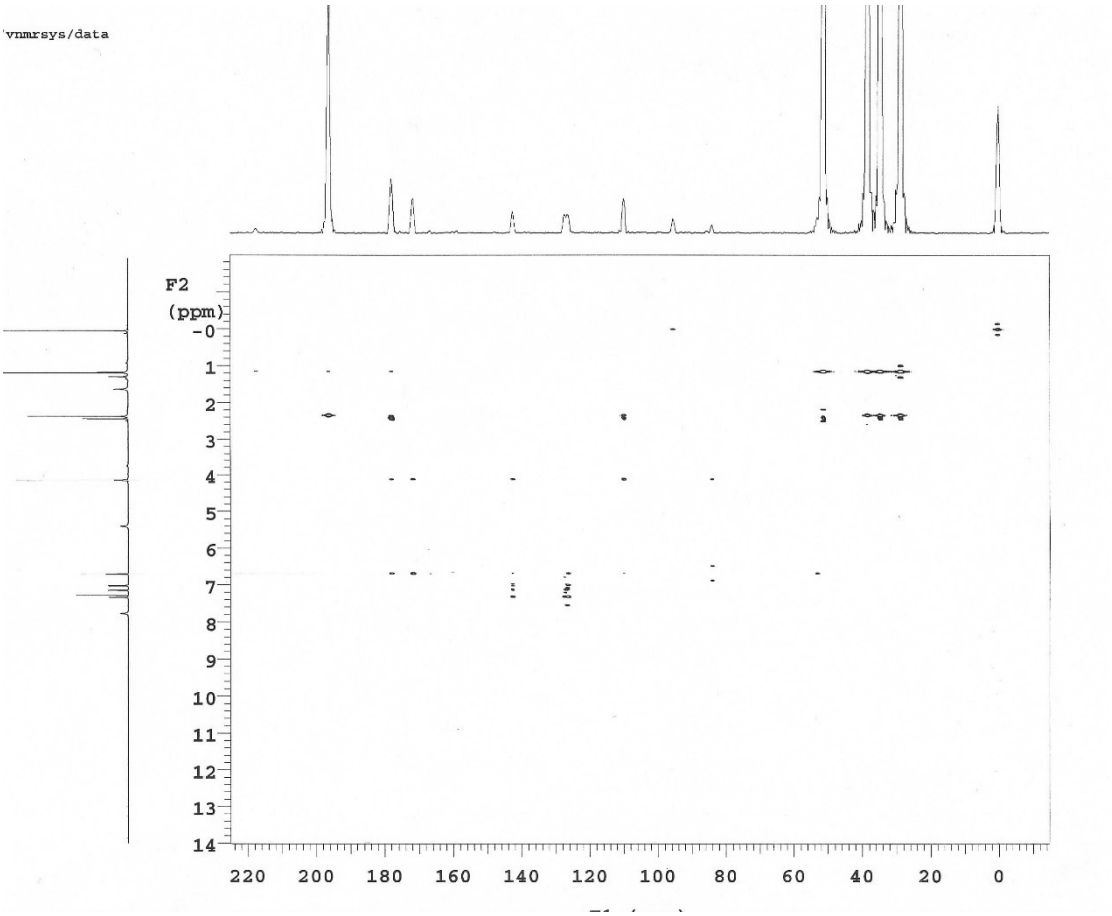
Compound 7e ^{1}H NMR



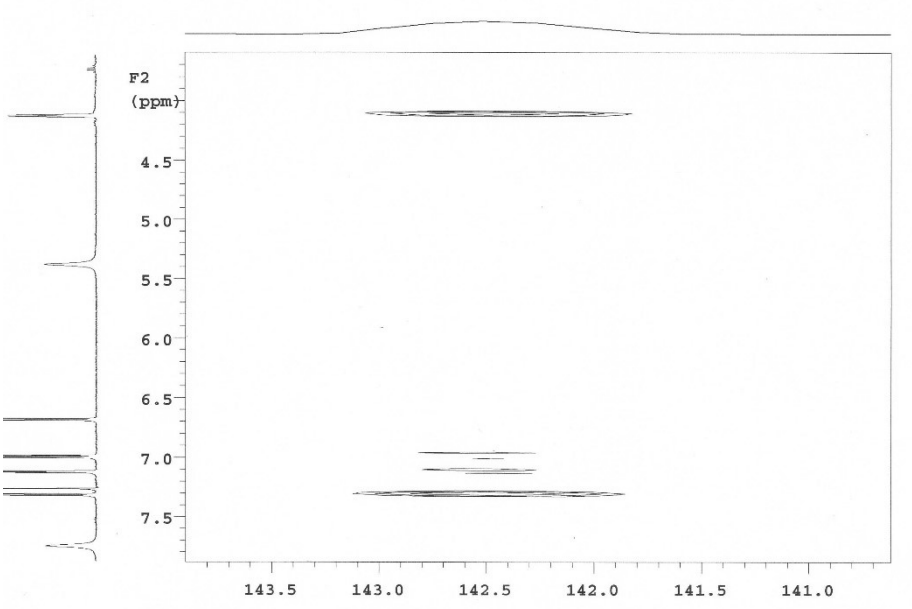
Compound 7e ^{13}C NMR



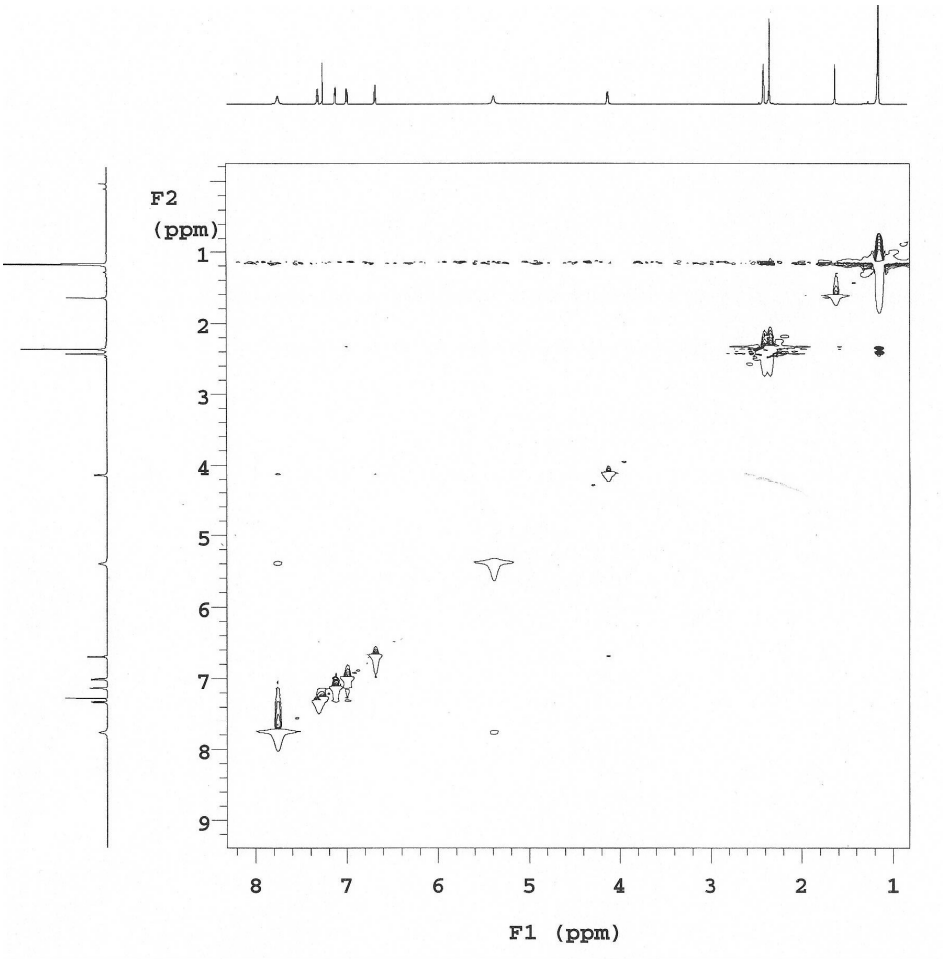
Compound 7e HMBC



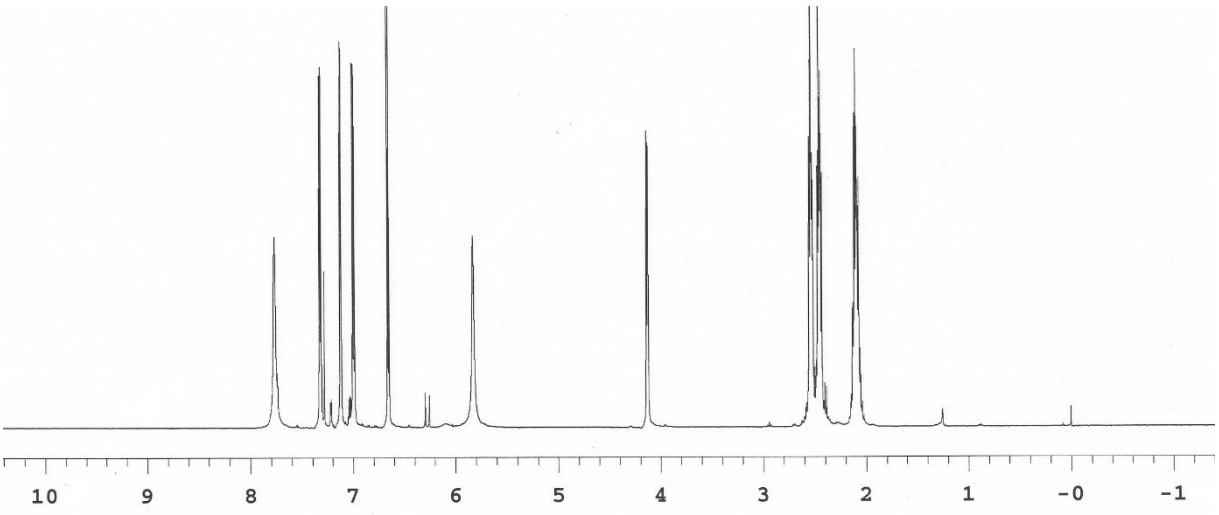
Compound 7e HMBC Expended



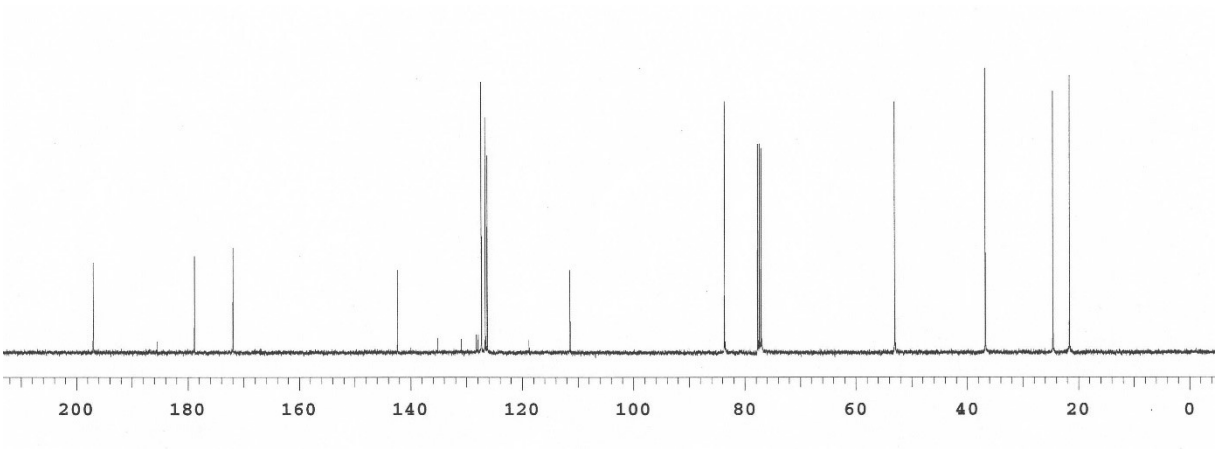
Compound 7e NOESY



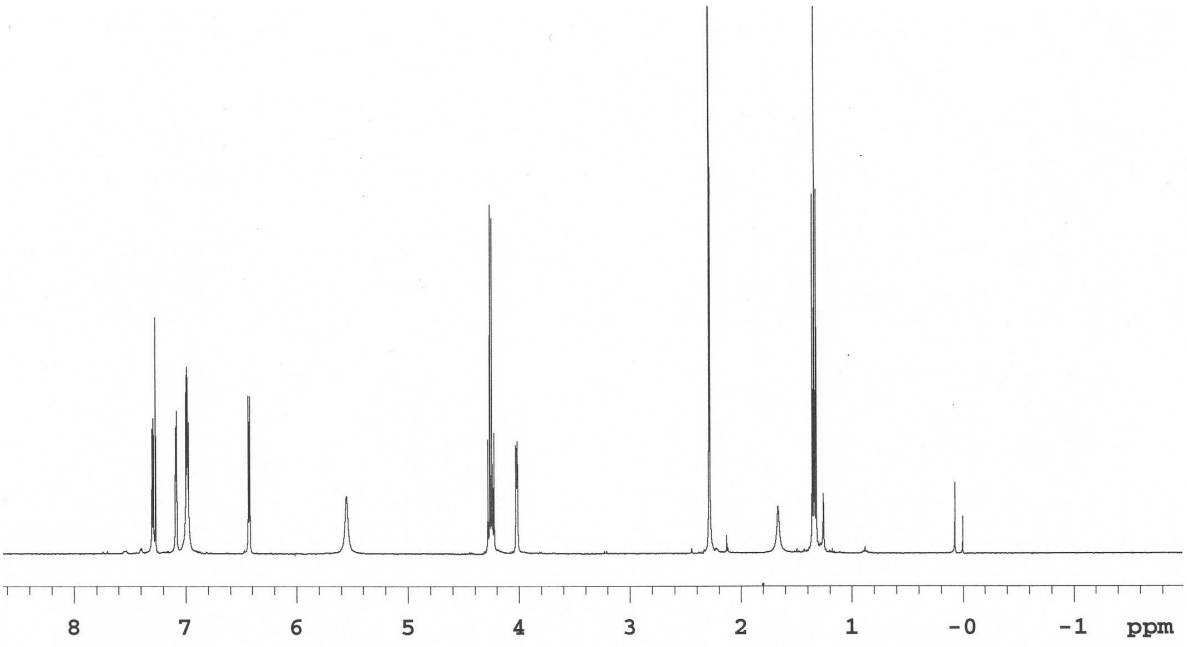
Compound 7f ^{1}H NMR



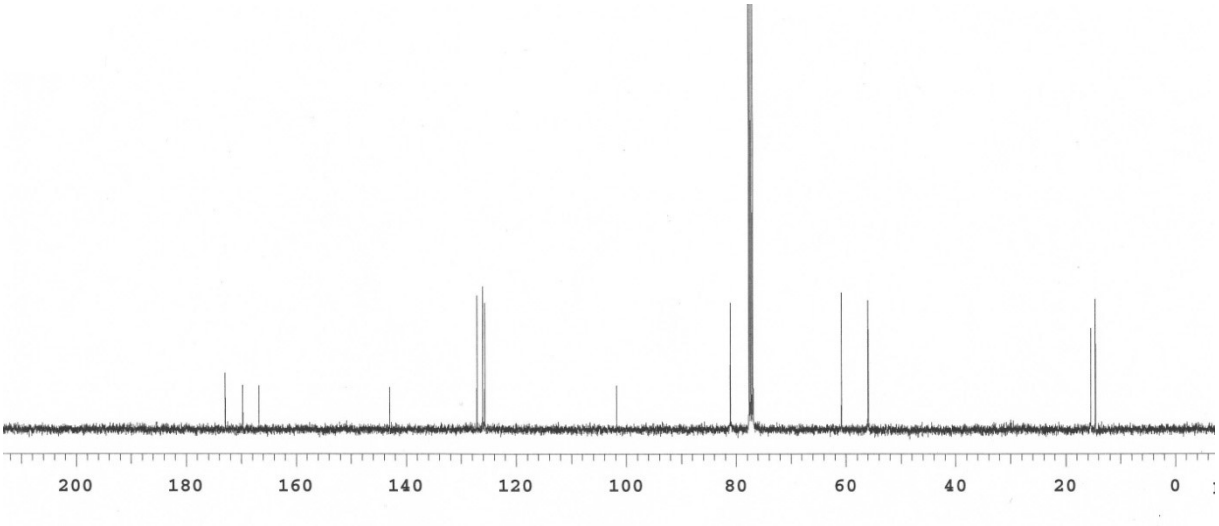
Compound 7f ^{13}C NMR



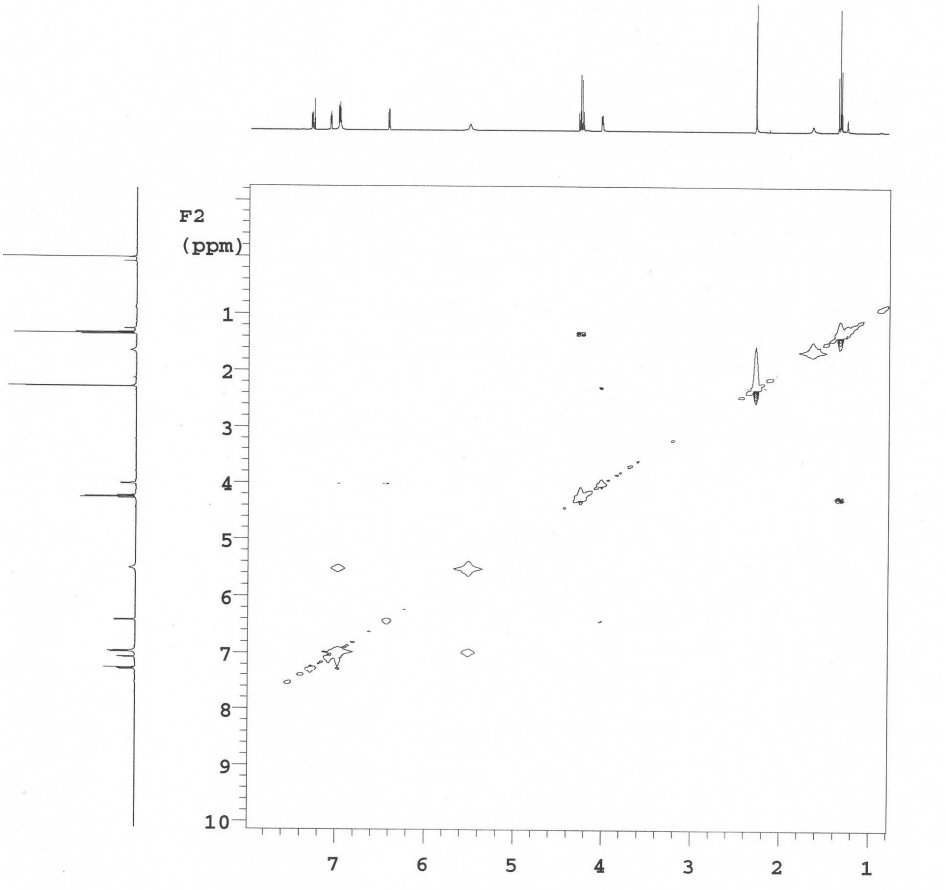
Compound 7g ^{1}H NMR



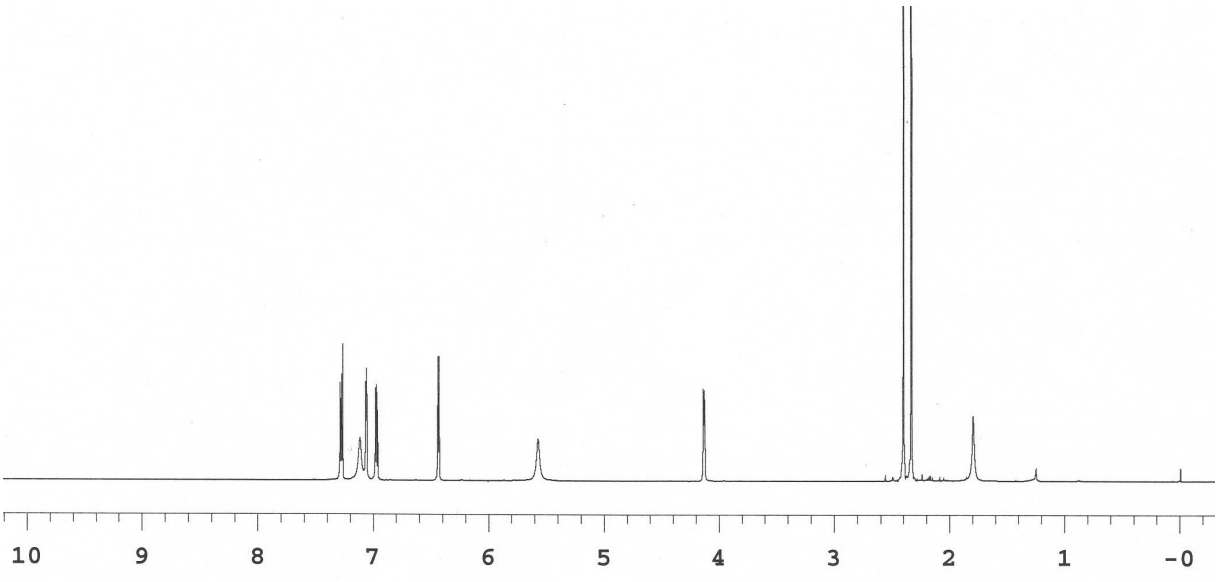
Compound 7g ^{13}C NMR



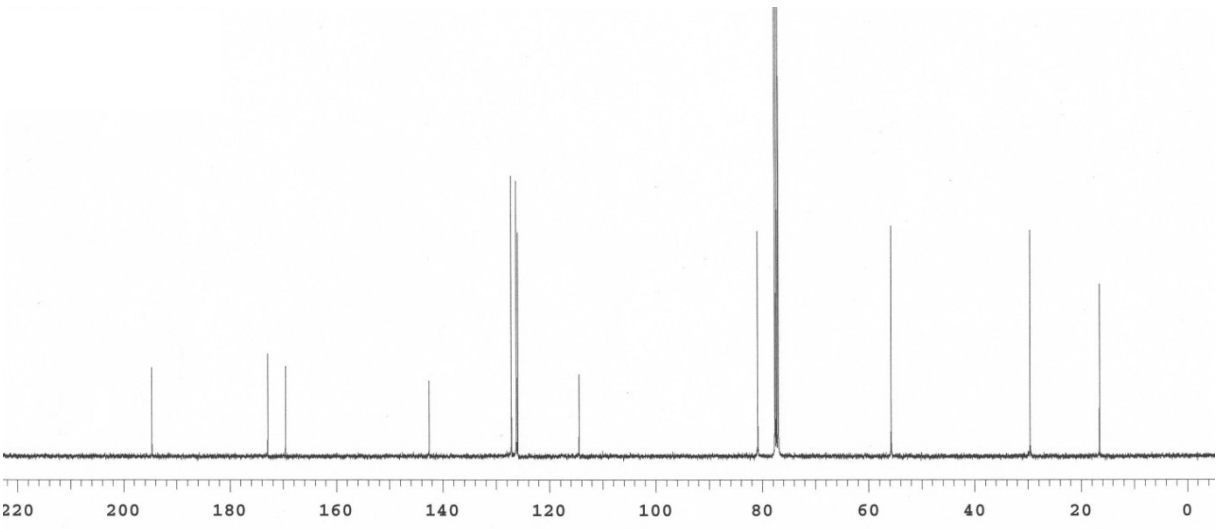
Compound 7g NOESY



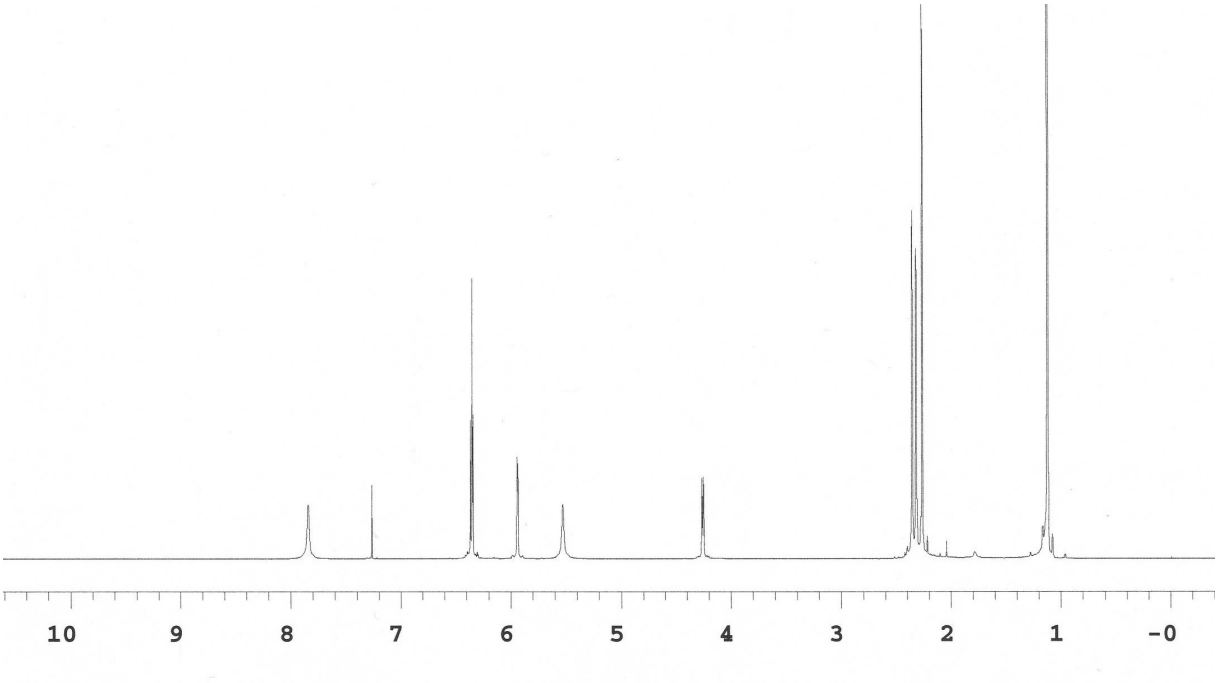
Compound 7h ^{1}H NMR



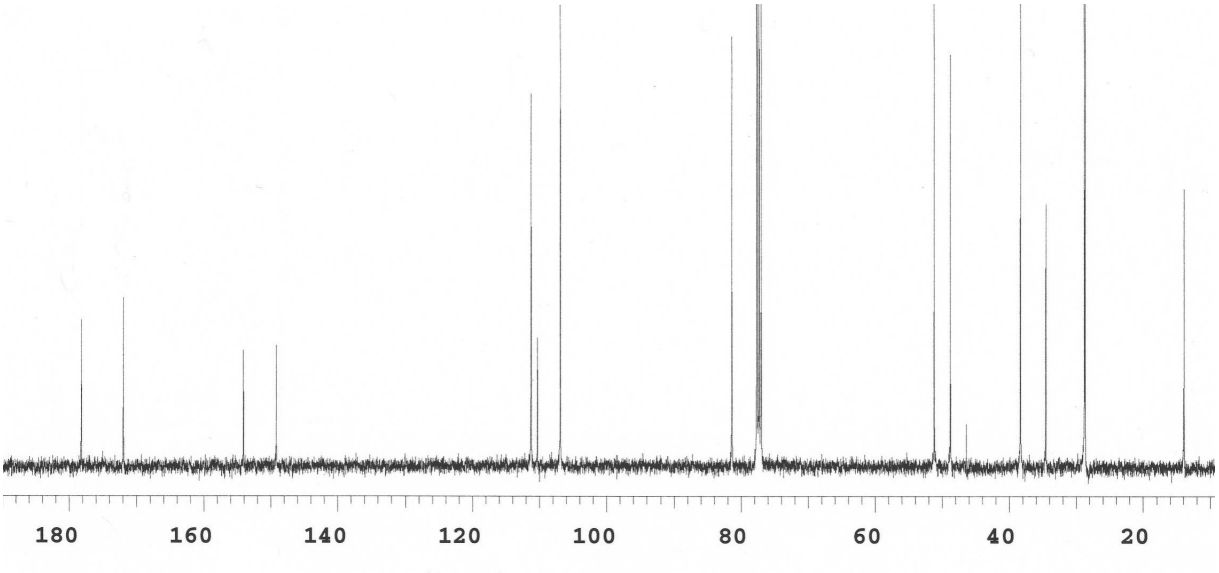
Compound 7h ^{13}C NMR



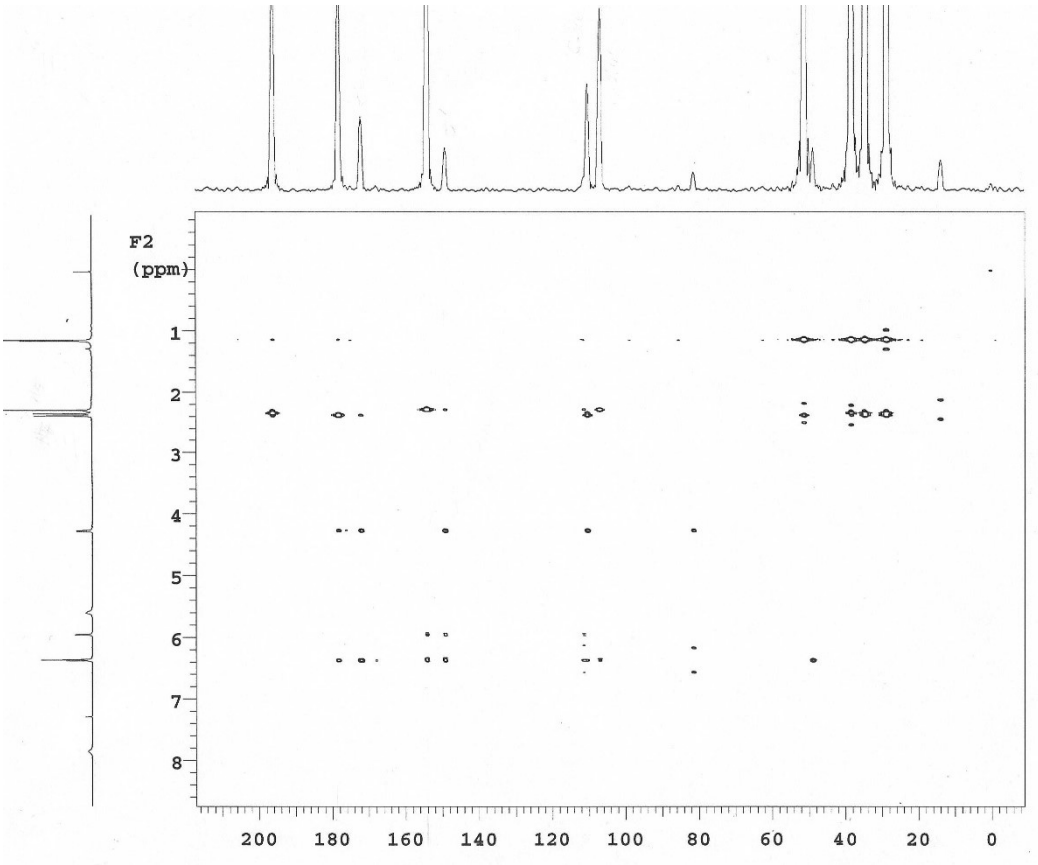
Compound 7i ^{1}H NMR



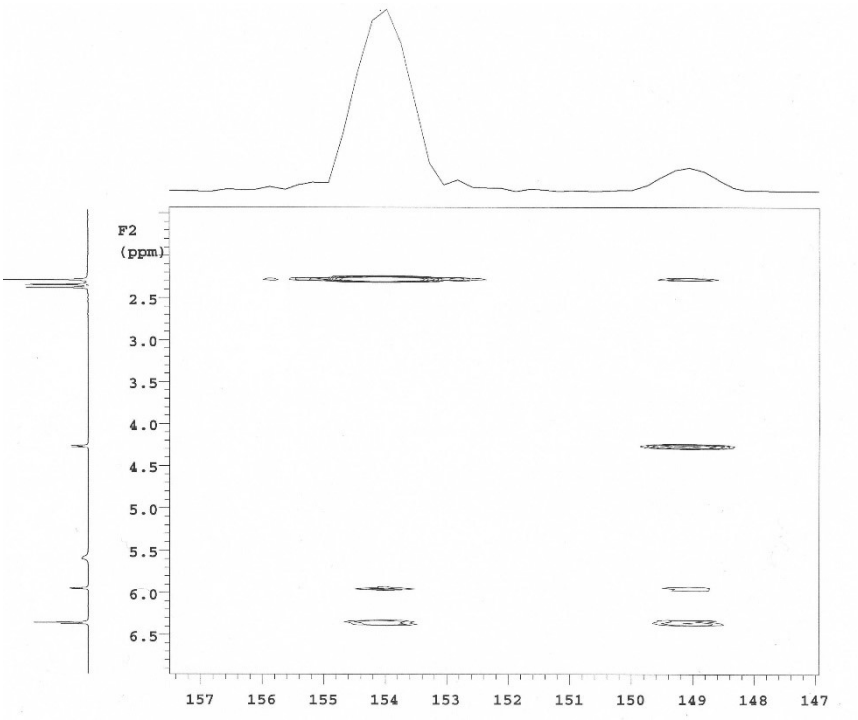
Compound 7i ^{13}C NMR



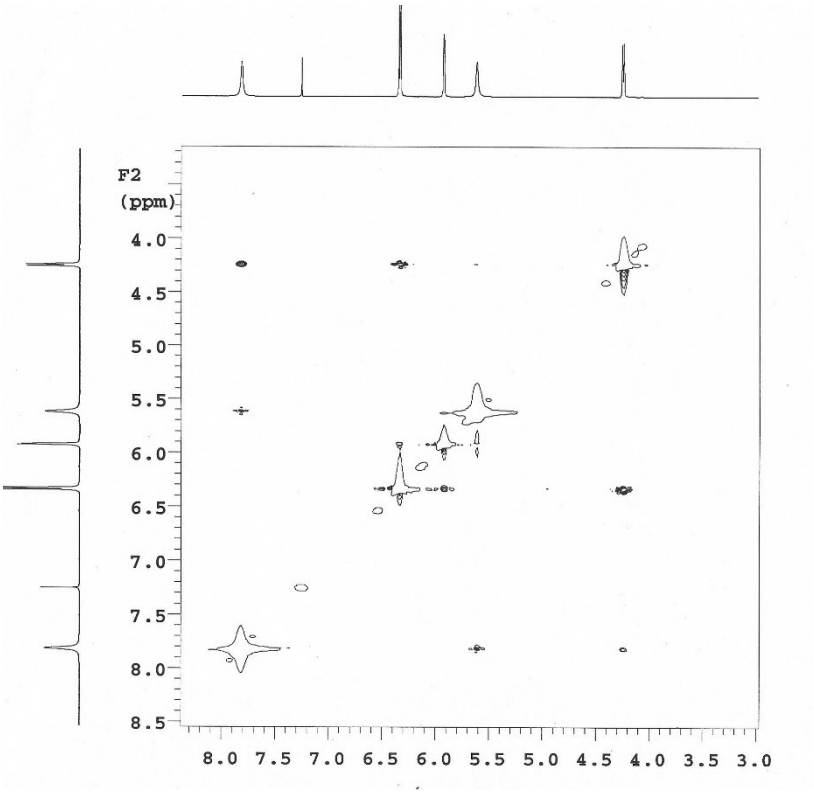
Compound 7i HMBC



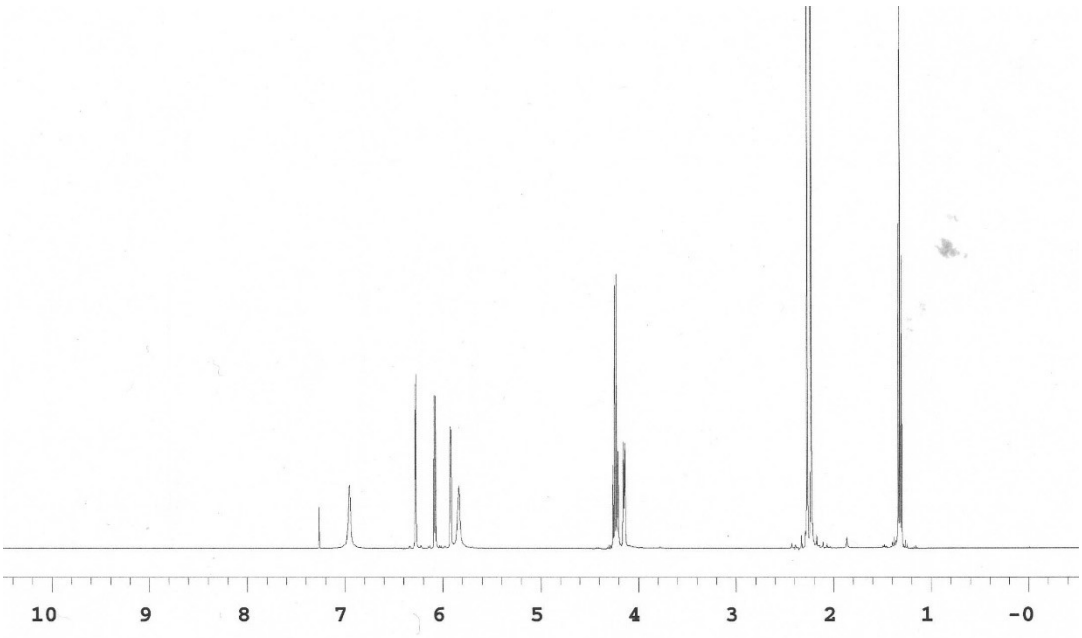
Compound 7i HMBC Expended



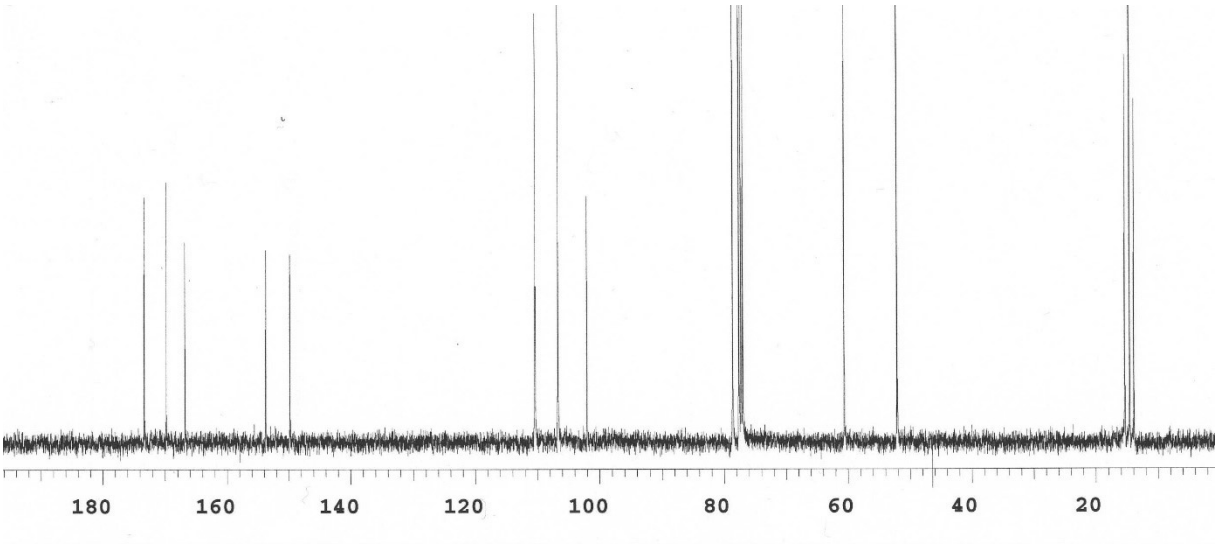
Compound 7i NOESY



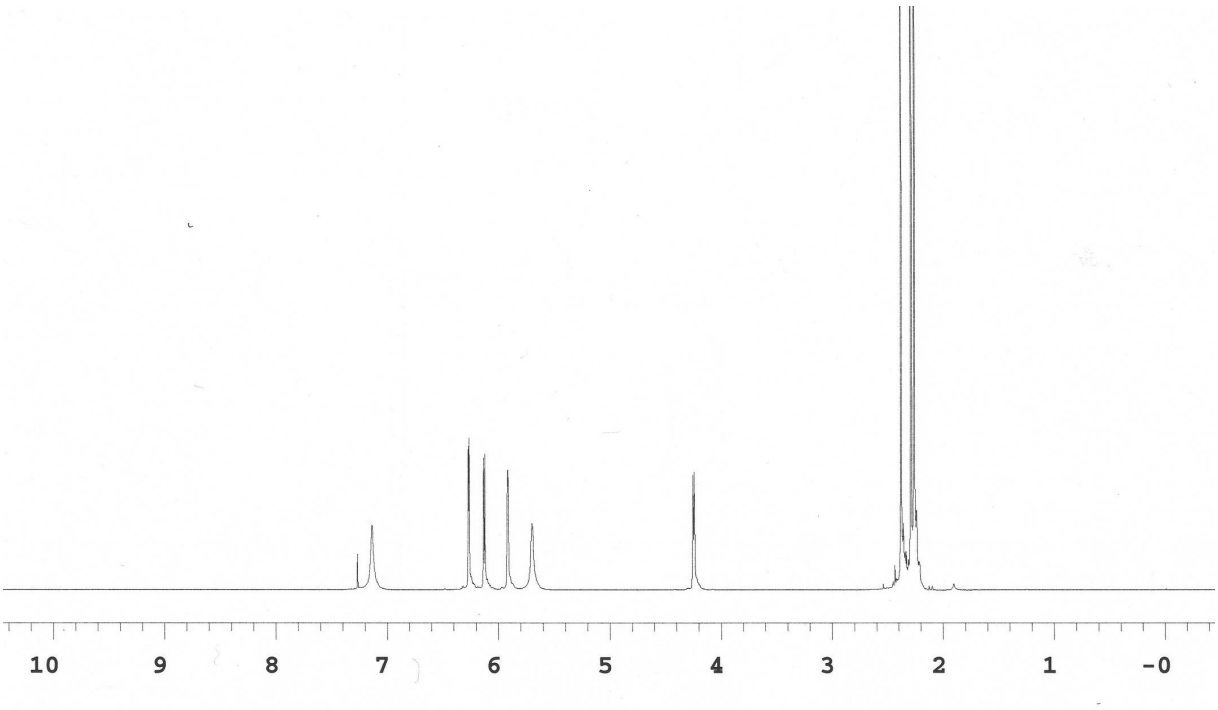
Compound 7j ^{1}H NMR



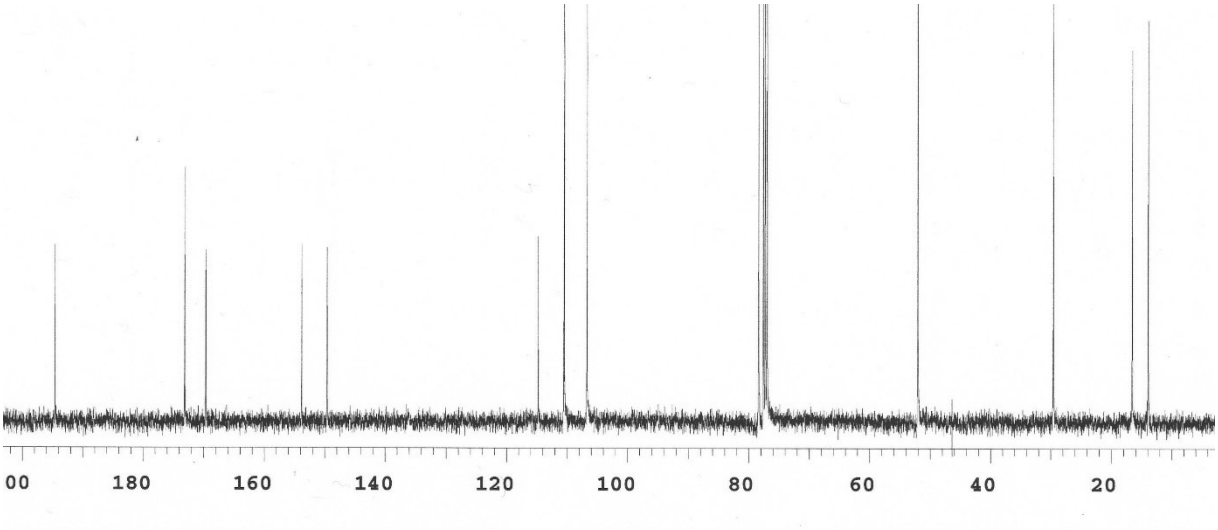
Compound 7j ^{13}C NMR



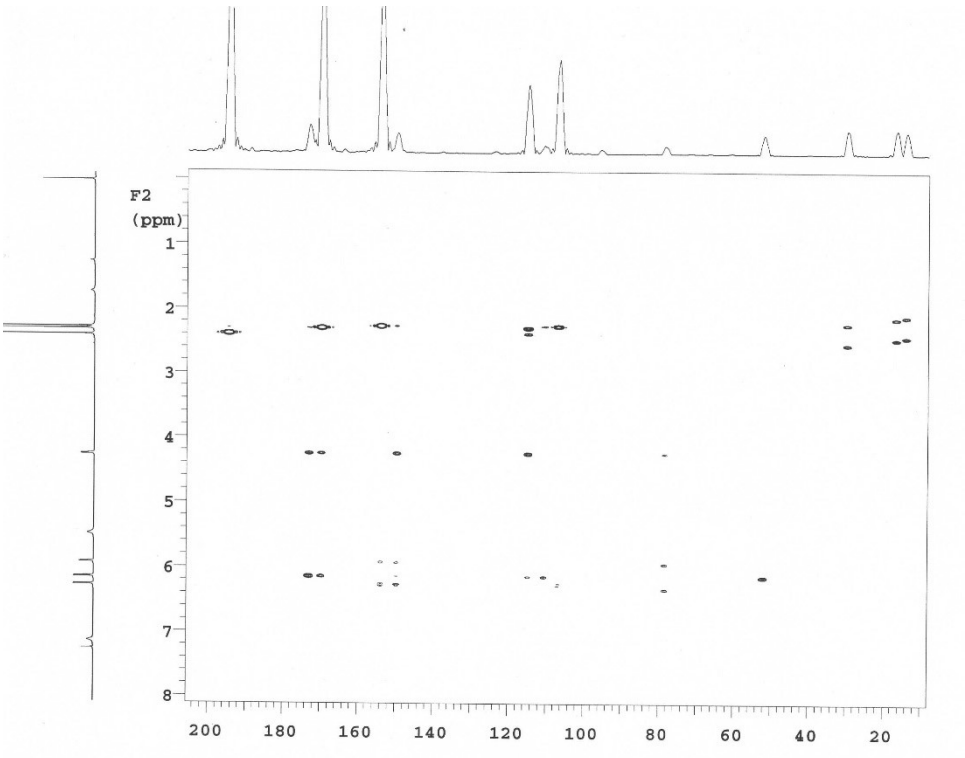
Compound 7k ^{1}H NMR



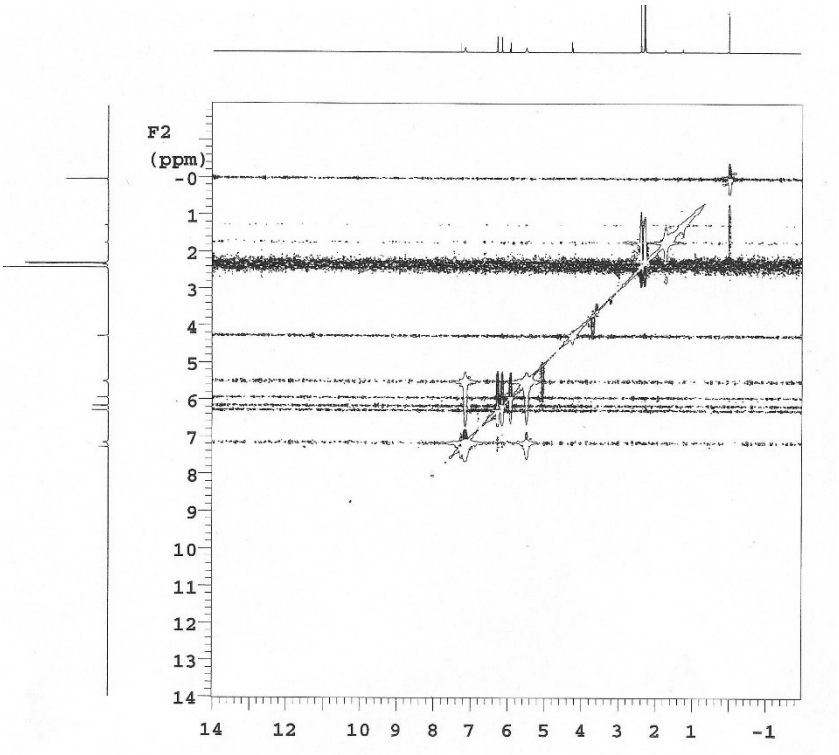
Compound 7k ^{13}C NMR



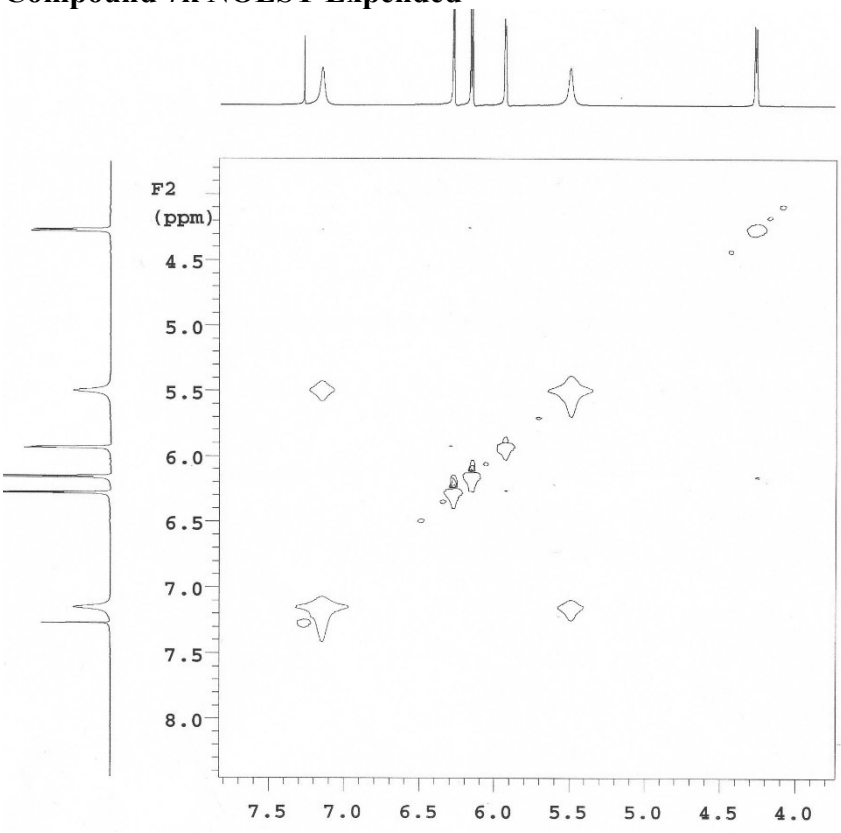
Compound 7k HMBC



Compound 7k NOESY



Compound 7k NOESY Expended



X-Ray Crystallography Information

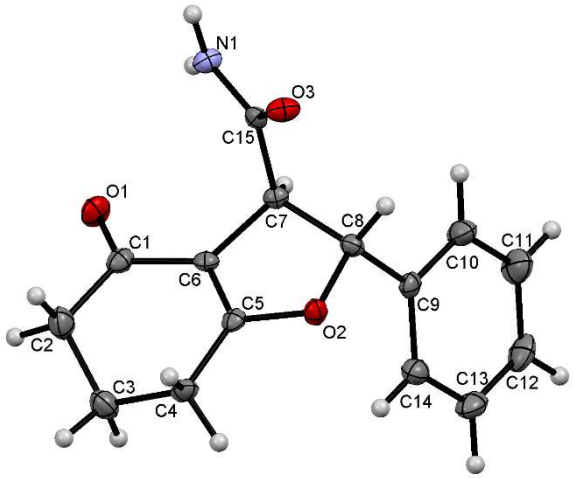
X-ray data collection and structure refinement

Unit cell measurements and intensity data collection was performed on an Bruker APEX II QUAZAR three-circle diffractometer using monochromatized Mo Kα microfocus sealed-tube (λ = 0.71073 Å) using φ and ω technique at 120 K. Data integration and reduction were carried out with Bruker SAINT [1] software package using a wide-frame algorithm. Absorption correction was performed using the multi-scan method (SADABS) [1]. Space groups were determined using XPREP implemented in APEX2 [1]. Structures were solved and refined using Bruker SHELXTL software package [2]. All non-hydrogen atoms were refined anisotropically using all reflections with $I > 2\sigma(I)$. Aromatic and aliphatic C-bound H atoms were positioned geometrically and refined using a riding mode and Uiso(H) = 1.2Ueq(C). Crystallographic data and refinement details of **1-5** are summarized in Table 1. The molecular drawings were carried out with and Mercury program [3].

1. Bruker APEX2 (Version 2014.1-1), SAINT (Version 8.34A) and SADABS (Version 2012/1). Bruker AXS Inc., Madison, Wisconsin, USA.
2. Bruker *SHELXTL* (Version 6.14). Bruker AXS Inc., Madison, Wisconsin, USA.
3. C. F. Macrae, P. R. Edgington, P. McCabe, E. Pidcock, G. P. Shields, R. Taylor, M. Towler, J. van de Streek, J. Appl. Cryst. 39 (2006) 453-457.

Crystallographic Data for 7b

CCDC 997073



A clear colourless block-like specimen of $C_{15}H_{15}NO_3$, approximate dimensions 0.140 mm x 0.229 mm x 0.316 mm, was used for the X-ray crystallographic analysis. The X-ray intensity data were measured.

A total of 1039 frames were collected. The total exposure time was 5.77 hours. The frames were integrated with the Bruker SAINT software package using a wide-frame algorithm. The integration of the data using amonoclinic unit cell yielded a total of 39061 reflections to a maximum θ angle of 25.50° (0.83 Å resolution), of which 7044 were independent (average redundancy 5.545, completeness = 97.9%, R_{int} = 3.46%, R_{sig} =3.12%) and 5739 (81.47%) were greater than $2\sigma(F^2)$. The final cell constants of a = 10.1561(2) Å, b = 41.9865(8) Å, c = 9.1009(2) Å, β = 92.9801(10)°, volume = 3875.55(14) $Å^3$, are based upon the refinement of the XYZ-centroids of 9874 reflections above 20 σ(I) with 4.585° < 2θ < 54.83°. Data were corrected for absorption effects using the multi-scan method (SADABS). The ratio of minimum to maximum apparent transmission was 0.922. The calculated minimum and maximum transmission coefficients (based on crystal size) are 0.9710 and 0.9870.

The structure was solved and refined using the Bruker SHELXTL Software Package, using the space group P 1 21/c 1, with Z = 12 for the formula unit, $C_{15}H_{15}NO_3$. The final anisotropic full-matrix least-squares refinement on F^2 with 532 variables converged at R1 = 4.79%, for the observed data and wR2 = 10.82% for all data. The goodness-of-fit was 1.047. The largest peak in the final difference electron density synthesis was0.848 $e^-/Å^3$ and the largest hole was -0.456 $e^-/Å^3$ with an RMS deviation of 0.049 $e^-/Å^3$. On the basis of the final model, the calculated density was 1.323 g/cm^3 and F(000), 1632 e^-.

Table 1. Sample and crystal data for 7b.

Identification code	CCDC 997073	
Chemical formula	$C_{15}H_{15}NO_3$	
Formula weight	257.28	
Temperature	120(2) K	
Wavelength	0.71073 Å	
Crystal size	0.140 x 0.229 x 0.316 mm	
Crystal habit	clear colourless block	
Crystal system	monoclinic	
Space group	P 1 21/c 1	
Unit cell dimensions	a = 10.1561(2) Å	α = 90°
	b = 41.9865(8) Å	β = 92.9801(10)°
	c = 9.1009(2) Å	γ = 90°

Volume	3875.55(14) $Å^3$
Z	12
Density (calculated)	1.323 g/cm^3
Absorption coefficient	0.093 mm^{-1}
F(000)	1632

Table 2. Data collection and structure refinement for 7b

Theta range for data collection	0.97 to 25.50°	
Index ranges	-12<=h<=12, -50<=k<=48, -10<=l<=10	
Reflections collected	39061	
Independent reflections	7044 [R(int) = 0.0346]	
Coverage of independent reflections	97.9%	
Absorption correction	multi-scan	
Max. and min. transmission	0.9870 and 0.9710	
Structure solution technique	direct methods	
Structure solution program	SHELXS-2013 (Sheldrick, 2013)	
Refinement method	Full-matrix least-squares on F^2	
Refinement program	SHELXL-2013 (Sheldrick, 2013)	
Function minimized	$\Sigma w(F_o^2 - F_c^2)^2$	
Data / restraints / parameters	7044 / 7 / 532	
Goodness-of-fit on F^2	1.047	
Final R indices	5739 data; I>2σ(I)	R1 = 0.0479, wR2 = 0.1015
	all data	R1 = 0.0623, wR2 = 0.1082
Weighting scheme	$w=1/[\sigma^2(F_o^2)+(0.0301P)^2+4.2836P]$ where $P=(F_o^2+2F_c^2)/3$	
Largest diff. peak and hole	0.848 and -0.456 $eÅ^{-3}$	
R.M.S. deviation from mean	0.049 $eÅ^{-3}$	

Table 3. Atomic coordinates and equivalent isotropic atomic displacement parameters (Å^2) for 7b.

U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x/a	y/b	z/c	U(eq)
C1	0.58258(19)	0.32431(5)	0.1708(2)	0.0177(4)
C2	0.6387(2)	0.34729(5)	0.2860(2)	0.0253(5)
C3	0.5413(2)	0.35726(5)	0.3990(2)	0.0250(5)
C4	0.4721(2)	0.32921(5)	0.4685(2)	0.0186(4)
C5	0.43646(18)	0.30501(4)	0.3550(2)	0.0145(4)
C6	0.48499(18)	0.30234(4)	0.2216(2)	0.0142(4)
C7	0.42944(18)	0.27244(4)	0.1484(2)	0.0138(4)
C8	0.31626(19)	0.26468(4)	0.2496(2)	0.0156(4)
C9	0.18032(19)	0.27370(4)	0.1878(2)	0.0160(4)
C10	0.1235(2)	0.25576(5)	0.0726(2)	0.0253(5)
C11	0.9984(2)	0.26287(6)	0.0141(3)	0.0323(6)
C12	0.9277(2)	0.28773(5)	0.0712(3)	0.0293(5)
C13	0.9826(2)	0.30529(5)	0.1865(3)	0.0272(5)
C14	0.1089(2)	0.29856(5)	0.2445(2)	0.0218(5)
C15	0.53559(18)	0.24655(4)	0.1558(2)	0.0142(4)
C16	0.0815(2)	0.05919(5)	0.5388(2)	0.0182(4)
C17	0.0577(2)	0.09204(5)	0.4752(2)	0.0260(5)
C18	0.0796(2)	0.11883(5)	0.5871(2)	0.0274(5)
C19	0.2149(2)	0.11697(5)	0.6688(2)	0.0243(5)
C20	0.24137(19)	0.08354(5)	0.7133(2)	0.0182(4)
C21	0.17771(19)	0.05753(5)	0.6609(2)	0.0159(4)
C22	0.23055(19)	0.02870(5)	0.7435(2)	0.0159(4)
C23	0.35671(19)	0.04278(5)	0.8209(2)	0.0178(4)
C24	0.48173(19)	0.03385(5)	0.7485(2)	0.0184(4)
C25	0.5379(2)	0.00426(5)	0.7790(2)	0.0239(5)
C26	0.6523(2)	0.99488(6)	0.7150(3)	0.0301(5)
C27	0.7124(2)	0.01517(6)	0.6194(2)	0.0305(5)

	x/a	y/b	z/c	U(eq)
C28	0.6568(2)	0.04452(6)	0.5869(3)	0.0325(6)
C29	0.5420(2)	0.05387(5)	0.6511(2)	0.0263(5)
C30	0.13149(19)	0.01737(4)	0.8533(2)	0.0162(4)
C31	0.16097(19)	0.14276(5)	0.1516(2)	0.0177(4)
C32	0.2285(2)	0.11456(5)	0.1284(2)	0.0219(5)
C33	0.3486(2)	0.10885(5)	0.2043(3)	0.0279(5)
C34	0.4023(2)	0.13091(6)	0.3023(2)	0.0285(5)
C35	0.3357(2)	0.15904(6)	0.3257(2)	0.0280(5)
C36	0.2154(2)	0.16487(5)	0.2510(2)	0.0228(5)
C37	0.02714(19)	0.14995(5)	0.0780(2)	0.0184(4)
C38	0.91486(18)	0.14778(4)	0.1851(2)	0.0142(4)
C39	0.81517(19)	0.17506(4)	0.1676(2)	0.0148(4)
C40	0.8685(2)	0.07666(6)	0.9356(3)	0.0481(8)
C41	0.9039(2)	0.10661(5)	0.0153(3)	0.0258(5)
C42	0.85432(19)	0.11627(5)	0.1412(2)	0.0195(4)
C43	0.7640(2)	0.09709(5)	0.2217(3)	0.0293(6)
C44	0.7323(3)	0.06490(6)	0.1578(4)	0.0522(8)
C45	0.7424(3)	0.06285(6)	0.9973(4)	0.0572(8)
N1	0.61888(17)	0.24615(4)	0.04874(19)	0.0179(4)
N2	0.03055(17)	0.00049(4)	0.79667(19)	0.0203(4)
N3	0.72658(17)	0.17582(4)	0.26936(19)	0.0192(4)
O1	0.62052(14)	0.32368(3)	0.04521(15)	0.0240(3)
O2	0.34647(13)	0.28269(3)	0.38602(14)	0.0171(3)
O3	0.54274(14)	0.22784(3)	0.26154(15)	0.0199(3)
O4	0.02648(14)	0.03539(3)	0.48607(16)	0.0247(3)
O5	0.33893(13)	0.07753(3)	0.81551(15)	0.0208(3)
O6	0.14576(14)	0.02437(3)	0.98493(15)	0.0237(3)
O7	0.72161(15)	0.10539(4)	0.3394(2)	0.0347(4)
O8	0.99613(14)	0.12625(4)	0.96287(15)	0.0270(4)
O9	0.81805(14)	0.19415(3)	0.06449(16)	0.0235(3)

Table 4. Bond lengths (Å) for 7b.

C1-O1	1.225(2)	C1-C6	1.447(3)

C1-C2	1.515(3)	C2-C3	1.522(3)
C3-C4	1.525(3)	C4-C5	1.480(3)
C5-C6	1.338(3)	C5-O2	1.349(2)
C6-C7	1.516(3)	C7-C15	1.530(3)
C7-C8	1.545(3)	C8-O2	1.472(2)
C8-C9	1.512(3)	C9-C14	1.386(3)
C9-C10	1.392(3)	C10-C11	1.384(3)
C11-C12	1.384(3)	C12-C13	1.377(3)
C13-C14	1.390(3)	C15-O3	1.241(2)
C15-N1	1.323(3)	C16-O4	1.230(2)
C16-C21	1.442(3)	C16-C17	1.510(3)
C17-C18	1.526(3)	C18-C19	1.530(3)
C19-C20	1.482(3)	C20-C21	1.344(3)
C20-O5	1.347(2)	C21-C22	1.509(3)
C22-C30	1.530(3)	C22-C23	1.547(3)
C23-O5	1.471(2)	C23-C24	1.508(3)
C24-C29	1.386(3)	C24-C25	1.389(3)
C25-C26	1.384(3)	C26-C27	1.382(3)
C27-C28	1.381(3)	C28-C29	1.389(3)
C30-O6	1.235(2)	C30-N2	1.328(3)
C31-C32	1.390(3)	C31-C36	1.390(3)
C31-C37	1.514(3)	C32-C33	1.391(3)
C33-C34	1.379(3)	C34-C35	1.383(3)
C35-C36	1.389(3)	C37-O8	1.468(2)
C37-C38	1.541(3)	C38-C42	1.504(3)
C38-C39	1.532(3)	C39-O9	1.236(2)
C39-N3	1.325(3)	C40-C41	1.487(3)
C40-C45	1.538(4)	C41-C42	1.339(3)
C41-O8	1.353(3)	C42-C43	1.448(3)
C43-O7	1.225(3)	C43-C44	1.500(3)
C44-C45	1.472(4)		

Table 5. Bond angles (°) for 7b.

O1-C1-C6	122.57(18)	O1-C1-C2	122.29(18)
C6-C1-C2	115.09(17)	C1-C2-C3	114.10(17)

C2-C3-C4	113.40(17)	C5-C4-C3	110.00(17)
C6-C5-O2	114.63(17)	C6-C5-C4	127.01(18)
O2-C5-C4	118.35(16)	C5-C6-C1	121.81(18)
C5-C6-C7	108.78(16)	C1-C6-C7	129.23(17)
C6-C7-C15	108.88(15)	C6-C7-C8	100.59(15)
C15-C7-C8	111.62(15)	O2-C8-C9	109.36(15)
O2-C8-C7	105.40(14)	C9-C8-C7	114.75(16)
C14-C9-C10	118.92(19)	C14-C9-C8	122.37(18)
C10-C9-C8	118.68(17)	C11-C10-C9	120.6(2)
C10-C11-C12	120.2(2)	C13-C12-C11	119.5(2)
C12-C13-C14	120.7(2)	C9-C14-C13	120.1(2)
O3-C15-N1	123.28(18)	O3-C15-C7	119.77(17)
N1-C15-C7	116.91(17)	O4-C16-C21	122.48(18)
O4-C16-C17	122.12(18)	C21-C16-C17	115.33(18)
C16-C17-C18	113.73(17)	C17-C18-C19	112.52(18)
C20-C19-C18	109.09(17)	C21-C20-O5	114.59(18)
C21-C20-C19	126.53(19)	O5-C20-C19	118.87(17)
C20-C21-C16	121.91(18)	C20-C21-C22	108.82(17)
C16-C21-C22	129.17(17)	C21-C22-C30	110.24(15)
C21-C22-C23	100.66(15)	C30-C22-C23	112.26(16)
O5-C23-C24	109.69(16)	O5-C23-C22	105.46(15)
C24-C23-C22	113.71(16)	C29-C24-C25	118.68(19)
C29-C24-C23	122.54(19)	C25-C24-C23	118.77(18)
C26-C25-C24	121.0(2)	C27-C26-C25	119.8(2)
C28-C27-C26	119.7(2)	C27-C28-C29	120.3(2)
C24-C29-C28	120.4(2)	O6-C30-N2	123.54(18)
O6-C30-C22	120.77(17)	N2-C30-C22	115.67(17)
C32-C31-C36	119.02(19)	C32-C31-C37	122.77(18)

C36-C31-C37	118.15(18)	C31-C32-C33	119.9(2)
C34-C33-C32	120.8(2)	C33-C34-C35	119.6(2)
C34-C35-C36	120.0(2)	C35-C36-C31	120.7(2)
O8-C37-C31	109.57(16)	O8-C37-C38	105.85(15)
C31-C37-C38	112.66(16)	C42-C38-C39	111.80(15)
C42-C38-C37	100.95(15)	C39-C38-C37	113.38(16)
O9-C39-N3	123.99(18)	O9-C39-C38	121.51(17)
N3-C39-C38	114.50(17)	C41-C40-C45	109.0(2)
C42-C41-O8	114.55(18)	C42-C41-C40	125.6(2)
O8-C41-C40	119.9(2)	C41-C42-C43	122.8(2)
C41-C42-C38	109.09(18)	C43-C42-C38	128.01(19)
O7-C43-C42	123.0(2)	O7-C43-C44	121.2(2)
C42-C43-C44	115.7(2)	C45-C44-C43	114.4(2)
C44-C45-C40	116.3(2)	C5-O2-C8	107.12(14)
C20-O5-C23	107.07(14)	C41-O8-C37	106.75(15)

Table 6. Torsion angles (°) for 7b.

O1-C1-C2-C3	149.5(2)	C6-C1-C2-C3	-32.9(3)
C1-C2-C3-C4	50.6(3)	C2-C3-C4-C5	-40.9(2)
C3-C4-C5-C6	17.3(3)	C3-C4-C5-O2	-163.32(17)
O2-C5-C6-C1	-179.93(16)	C4-C5-C6-C1	-0.5(3)
O2-C5-C6-C7	-4.4(2)	C4-C5-C6-C7	174.96(18)
O1-C1-C6-C5	-174.28(19)	C2-C1-C6-C5	8.1(3)
O1-C1-C6-C7	11.2(3)	C2-C1-C6-C7	-166.35(18)
C5-C6-C7-C15	-103.38(18)	C1-C6-C7-C15	71.7(2)
C5-C6-C7-C8	14.02(19)	C1-C6-C7-C8	-170.93(19)
C6-C7-C8-O2	-18.03(18)	C15-C7-C8-O2	97.33(17)
C6-C7-C8-C9	102.35(18)	C15-C7-C8-C9	-142.29(16)
O2-C8-C9-C14	8.4(2)	C7-C8-C9-C14	-109.8(2)
O2-C8-C9-C10	-169.63(17)	C7-C8-C9-C10	72.2(2)
C14-C9-C10-C11	0.9(3)	C8-C9-C10-C11	179.0(2)
C9-C10-C11-C12	-0.9(4)	C10-C11-C12-C13	0.0(4)
C11-C12-C13-C14	0.9(3)	C10-C9-C14-C13	0.0(3)
C8-C9-C14-C13	-178.06(19)	C12-C13-C14-C9	-0.9(3)
C6-C7-C15-O3	91.2(2)	C8-C7-C15-O3	-18.9(2)
C6-C7-C15-N1	-86.6(2)	C8-C7-C15-N1	163.21(16)

O4-C16-C17-C18	153.3(2)	C21-C16-C17-C18	-29.5(3)
C16-C17-C18-C19	53.0(3)	C17-C18-C19-C20	-44.5(2)
C18-C19-C20-C21	16.2(3)	C18-C19-C20-O5	-164.38(17)
O5-C20-C21-C16	-172.47(17)	C19-C20-C21-C16	7.0(3)
O5-C20-C21-C22	4.2(2)	C19-C20-C21-C22	-176.40(19)
O4-C16-C21-C20	177.03(19)	C17-C16-C21-C20	-0.1(3)
O4-C16-C21-C22	1.1(3)	C17-C16-C21-C22	-176.05(19)
C20-C21-C22-C30	104.98(19)	C16-C21-C22-C30	-78.7(2)
C20-C21-C22-C23	-13.7(2)	C16-C21-C22-C23	162.62(19)
C21-C22-C23-O5	17.83(18)	C30-C22-C23-O5	-99.39(17)
C21-C22-C23-C24	-102.39(18)	C30-C22-C23-C24	140.39(17)
O5-C23-C24-C29	-19.8(3)	C22-C23-C24-C29	98.0(2)
O5-C23-C24-C25	160.97(17)	C22-C23-C24-C25	-81.2(2)
C29-C24-C25-C26	0.6(3)	C23-C24-C25-C26	179.80(19)
C24-C25-C26-C27	0.1(3)	C25-C26-C27-C28	-0.8(3)
C26-C27-C28-C29	0.8(3)	C25-C24-C29-C28	-0.6(3)
C23-C24-C29-C28	-179.8(2)	C27-C28-C29-C24	-0.1(3)
C21-C22-C30-O6	-99.7(2)	C23-C22-C30-O6	11.6(3)
C21-C22-C30-N2	78.9(2)	C23-C22-C30-N2	-169.74(17)
C36-C31-C32-C33	0.1(3)	C37-C31-C32-C33	-177.23(19)
C31-C32-C33-C34	-0.3(3)	C32-C33-C34-C35	0.2(3)
C33-C34-C35-C36	0.2(3)	C34-C35-C36-C31	-0.4(3)
C32-C31-C36-C35	0.2(3)	C37-C31-C36-C35	177.73(19)
C32-C31-C37-O8	-10.7(3)	C36-C31-C37-O8	171.92(17)
C32-C31-C37-C38	106.8(2)	C36-C31-C37-C38	-70.5(2)
O8-C37-C38-C42	16.06(19)	C31-C37-C38-C42	-103.67(18)
O8-C37-C38-C39	-103.65(18)	C31-C37-C38-C39	136.62(17)
C42-C38-C39-O9	-104.7(2)	C37-C38-C39-O9	8.7(3)
C42-C38-C39-N3	74.2(2)	C37-C38-C39-N3	-172.50(17)
C45-C40-C41-C42	14.2(3)	C45-C40-C41-O8	-167.6(2)
O8-C41-C42-C43	-174.03(18)	C40-C41-C42-C43	4.2(3)
O8-C41-C42-C38	2.1(2)	C40-C41-C42-C38	-179.7(2)
C39-C38-C42-C41	109.46(19)	C37-C38-C42-C41	-11.4(2)
C39-C38-C42-C43	-74.7(3)	C37-C38-C42-C43	164.44(19)
C41-C42-C43-O7	177.6(2)	C38-C42-C43-O7	2.3(3)
C41-C42-C43-C44	2.0(3)	C38-C42-C43-C44	-173.3(2)
O7-C43-C44-C45	156.7(2)	C42-C43-C44-C45	-27.6(3)

C43-C44-C45-C40	48.0(3)	C41-C40-C45-C44	-39.9(3)
C6-C5-O2-C8	-8.1(2)	C4-C5-O2-C8	172.46(16)
C9-C8-O2-C5	-107.17(16)	C7-C8-O2-C5	16.69(18)
C21-C20-O5-C23	8.2(2)	C19-C20-O5-C23	-171.26(17)
C24-C23-O5-C20	106.24(17)	C22-C23-O5-C20	-16.58(19)
C42-C41-O8-C37	9.0(2)	C40-C41-O8-C37	-169.4(2)
C31-C37-O8-C41	105.93(18)	C38-C37-O8-C41	-15.8(2)

Table 7. Anisotropic atomic displacement parameters (Å^2) for 7b.

The anisotropic atomic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2} U_{11} + ... + 2 h k a^* b^* U_{12}]$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C1	0.0165(10)	0.0179(10)	0.0188(11)	0.0034(8)	0.0027(9)	0.0033(8)
C2	0.0249(12)	0.0237(11)	0.0276(12)	0.0001(9)	0.0037(10)	-0.0074(9)
C3	0.0291(12)	0.0213(11)	0.0248(12)	-0.0048(9)	0.0014(10)	-0.0044(9)
C4	0.0177(10)	0.0217(11)	0.0167(11)	-0.0051(8)	0.0023(8)	0.0022(8)
C5	0.0117(9)	0.0140(9)	0.0178(11)	0.0028(8)	0.0010(8)	0.0030(8)
C6	0.0145(10)	0.0125(9)	0.0155(10)	-0.0007(8)	0.0011(8)	0.0032(8)
C7	0.0151(10)	0.0151(10)	0.0113(10)	0.0003(7)	0.0005(8)	0.0013(8)
C8	0.0180(10)	0.0136(9)	0.0152(10)	-0.0021(8)	0.0016(8)	-0.0001(8)
C9	0.0147(10)	0.0157(10)	0.0181(11)	0.0027(8)	0.0046(8)	-0.0020(8)
C10	0.0180(11)	0.0295(12)	0.0285(12)	-0.0092(10)	0.0014(9)	0.0030(9)
C11	0.0194(12)	0.0426(14)	0.0343(14)	-0.0102(11)	-0.0050(10)	-0.0009(10)
C12	0.0135(11)	0.0367(13)	0.0374(14)	0.0068(11)	-0.0009(10)	0.0013(10)
C13	0.0233(12)	0.0215(11)	0.0374(14)	0.0021(10)	0.0086(10)	0.0066(9)
C14	0.0228(11)	0.0173(10)	0.0255(12)	-0.0007(9)	0.0048(9)	0.0006(9)
C15	0.0143(10)	0.0160(10)	0.0121(10)	-0.0039(8)	-0.0009(8)	-0.0015(8)
C16	0.0190(11)	0.0208(10)	0.0151(10)	0.0005(8)	0.0040(8)	0.0004(8)
C17	0.0335(13)	0.0251(12)	0.0190(11)	0.0052(9)	-0.0030(10)	0.0030(10)
C18	0.0364(13)	0.0198(11)	0.0258(12)	0.0035(9)	0.0006(10)	0.0051(10)
C19	0.0310(12)	0.0184(11)	0.0235(12)	-0.0012(9)	0.0026(10)	-0.0018(9)
C20	0.0181(10)	0.0219(11)	0.0149(10)	-0.0005(8)	0.0044(9)	0.0000(8)
C21	0.0173(10)	0.0167(10)	0.0140(10)	-0.0001(8)	0.0032(8)	-0.0006(8)
C22	0.0161(10)	0.0175(10)	0.0142(10)	0.0005(8)	0.0012(8)	-0.0002(8)
C23	0.0180(10)	0.0188(10)	0.0164(11)	0.0013(8)	0.0003(8)	-0.0012(8)

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C24	0.0146(10)	0.0243(11)	0.0160(10)	-0.0024(8)	-0.0030(8)	-0.0022(8)
C25	0.0203(11)	0.0221(11)	0.0290(12)	-0.0004(9)	-0.0014(9)	-0.0022(9)
C26	0.0230(12)	0.0296(12)	0.0370(14)	-0.0085(10)	-0.0045(10)	0.0055(10)
C27	0.0182(11)	0.0485(15)	0.0248(13)	-0.0108(11)	0.0010(10)	0.0018(10)
C28	0.0264(13)	0.0479(15)	0.0239(13)	0.0040(11)	0.0070(10)	-0.0043(11)
C29	0.0233(12)	0.0318(12)	0.0240(12)	0.0067(10)	0.0032(10)	0.0022(10)
C30	0.0165(10)	0.0152(10)	0.0172(11)	0.0036(8)	0.0030(8)	0.0042(8)
C31	0.0142(10)	0.0229(11)	0.0165(11)	0.0029(8)	0.0069(8)	-0.0004(8)
C32	0.0198(11)	0.0226(11)	0.0238(12)	0.0009(9)	0.0069(9)	-0.0002(9)
C33	0.0207(11)	0.0293(12)	0.0344(13)	0.0088(10)	0.0094(10)	0.0088(9)
C34	0.0150(11)	0.0429(14)	0.0277(13)	0.0122(11)	0.0030(9)	0.0009(10)
C35	0.0216(12)	0.0365(13)	0.0259(13)	-0.0002(10)	0.0005(10)	-0.0069(10)
C36	0.0198(11)	0.0224(11)	0.0267(12)	-0.0006(9)	0.0053(9)	-0.0010(9)
C37	0.0182(10)	0.0205(10)	0.0166(11)	-0.0018(8)	0.0028(8)	0.0009(8)
C38	0.0132(10)	0.0170(10)	0.0126(10)	0.0020(8)	0.0012(8)	0.0001(8)
C39	0.0148(10)	0.0161(10)	0.0135(10)	-0.0024(8)	-0.0005(8)	-0.0010(8)
C40	0.0287(14)	0.0481(16)	0.0657(19)	-0.0385(15)	-0.0135(13)	0.0139(12)
C41	0.0166(11)	0.0258(11)	0.0342(13)	-0.0085(10)	-0.0057(10)	0.0053(9)
C42	0.0135(10)	0.0150(10)	0.0296(12)	-0.0011(9)	-0.0020(9)	0.0045(8)
C43	0.0138(11)	0.0171(11)	0.0568(17)	0.0063(11)	-0.0002(11)	0.0041(9)
C44	0.0329(15)	0.0227(13)	0.102(3)	-0.0102(14)	0.0118(16)	-0.0036(11)
C45	0.067(2)	0.0287(14)	0.073(2)	-0.0072(14)	-0.0301(17)	-0.0117(14)
N1	0.0177(9)	0.0196(9)	0.0166(9)	0.0006(7)	0.0033(8)	0.0031(7)
N2	0.0177(9)	0.0266(10)	0.0166(9)	-0.0004(8)	0.0018(7)	-0.0046(7)
N3	0.0204(9)	0.0213(9)	0.0166(9)	0.0014(7)	0.0056(8)	0.0065(7)
O1	0.0264(8)	0.0282(8)	0.0183(8)	0.0019(6)	0.0084(7)	-0.0033(6)
O2	0.0173(7)	0.0205(7)	0.0138(7)	-0.0014(6)	0.0027(6)	-0.0040(6)
O3	0.0245(8)	0.0203(7)	0.0150(7)	0.0016(6)	0.0027(6)	0.0075(6)
O4	0.0261(8)	0.0256(8)	0.0217(8)	-0.0028(6)	-0.0038(6)	-0.0031(7)
O5	0.0204(8)	0.0187(7)	0.0228(8)	-0.0045(6)	-0.0027(6)	-0.0004(6)
O6	0.0231(8)	0.0327(8)	0.0154(8)	-0.0012(6)	0.0034(6)	-0.0067(6)
O7	0.0230(8)	0.0302(9)	0.0521(11)	0.0174(8)	0.0138(8)	0.0044(7)
O8	0.0221(8)	0.0406(9)	0.0183(8)	-0.0106(7)	0.0021(6)	0.0033(7)
O9	0.0256(8)	0.0216(8)	0.0242(8)	0.0078(6)	0.0081(6)	0.0065(6)

Table 8. Hydrogen atomic coordinates and isotropic atomic displacement parameters (Å^2) for 7b.

	x/a	y/b	z/c	U(eq)
H2A	0.6700	0.3666	0.2360	0.03
H2B	0.7160	0.3373	0.3381	0.03
H3A	0.4739	0.3714	0.3508	0.03
H3B	0.5887	0.3697	0.4776	0.03
H4A	0.5312	0.3196	0.5463	0.022
H4B	0.3916	0.3367	0.5144	0.022
H7	0.3959	0.2766	0.0450	0.017
H8	0.3178	0.2414	0.2725	0.019
H10	0.1710	0.2385	0.0337	0.03
H11	-0.0390	0.2506	-0.0654	0.039
H12	-0.1582	0.2926	0.0311	0.035
H13	-0.0662	0.3222	0.2269	0.033
H14	0.1464	0.3110	0.3232	0.026
H17A	-0.0341	0.0932	0.4332	0.031
H17B	0.1172	0.0953	0.3938	0.031
H18A	0.0102	0.1178	0.6594	0.033
H18B	0.0713	0.1396	0.5357	0.033
H19A	0.2839	0.1245	0.6040	0.029
H19B	0.2165	0.1308	0.7569	0.029
H22	0.2526	0.0112	0.6743	0.019
H23	0.3629	0.0357	0.9259	0.021
H25	0.4971	-0.0098	0.8448	0.029
H26	0.6895	-0.0254	0.7368	0.036
H27	0.7916	0.0089	0.5762	0.037
H28	0.6974	0.0584	0.5202	0.039
H29	0.5044	0.0741	0.6283	0.032
H32	1.1928	0.0992	0.0607	0.026
H33	1.3942	0.0895	0.1884	0.033
H34	1.4844	0.1268	0.3535	0.034
H35	1.3723	0.1744	0.3928	0.034
H36	1.1698	0.1842	0.2679	0.027
H37	1.0281	0.1717	0.0333	0.022
H38	0.9521	0.1468	0.2889	0.017

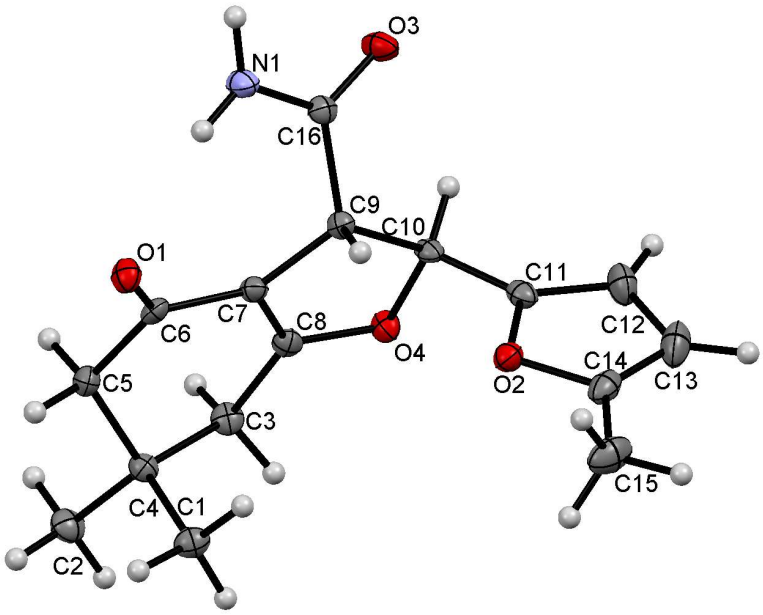
	x/a	y/b	z/c	U(eq)
H40A	0.9414	0.0611	-0.0516	0.058
H40B	0.8536	0.0811	-0.1708	0.058
H44A	0.7929	0.0491	0.2053	0.063
H44B	0.6415	0.0591	0.1820	0.063
H45A	0.6657	0.0740	-0.0503	0.069
H45B	0.7358	0.0402	-0.0316	0.069
H1N	0.606(3)	0.2586(7)	-0.031(3)	0.069
H2N	0.685(2)	0.2317(6)	0.054(3)	0.069
H3N	0.026(3)	-0.0048(7)	0.701(2)	0.069
H4N	-0.032(3)	-0.0064(7)	0.858(3)	0.069
H5N	0.729(3)	0.1603(6)	0.337(3)	0.069
H6N	0.667(3)	0.1918(6)	0.269(3)	0.069

Table 10. Hydrogen bond distances (Å) and angles (°) for 7b.

	Donor-H	Acceptor-H	Donor-Acceptor	Angle
N1-H2N···O9	0.906(18)	2.074(18)	2.975(2)	172.0
N2-H3N···O4	0.893(18)	2.18(2)	3.012(2)	155.0
N2-H3N···O4	0.893(18)	2.59(3)	3.182(2)	125.0
N1-H1N···O3	0.895(18)	2.05(2)	2.900(2)	159.0
N3-H6N···O3	0.905(18)	1.967(18)	2.872(2)	177.0
N3-H5N···O7	0.897(18)	2.31(3)	3.026(2)	137.0
N3-H5N···O1	0.897(18)	2.34(3)	2.783(2)	110.0
N2-H4N···O6	0.911(18)	2.033(19)	2.935(2)	171.0
C40-H40A···O6	0.99	2.59	3.581(3)	175.2
C17-H17A···O7	0.99	2.63	3.613(3)	171.0
C2-H2B···O9	0.99	2.61	3.505(3)	149.8

Crystallographic Data for 7i

CCDC 997070



A clear colourless block-like specimen of $C_{16}H_{19}NO_4$, approximate dimensions 0.080 mm x 0.120 mm x 0.180 mm, was used for the X-ray crystallographic analysis. The X-ray intensity data were measured.

The total exposure time was 7.66 hours. The frames were integrated with the Bruker SAINT software package using a wide-frame algorithm. The integration of the data using an orthorhombic unit cell yielded a total of23828 reflections to a maximum θ angle of 27.47° (0.77 Å resolution), of which 3257 were independent (average redundancy 7.316, completeness = 99.9%, R_{int} = 4.53%, R_{sig} = 3.06%) and 2982 (91.56%) were greater than $2\sigma(F^2)$. The final cell constants of a = 22.1303(4) Å, b = 8.7685(2) Å, c = 7.40320(10) Å, volume = 1436.59(5) $Å^3$, are based upon the refinement of the XYZ-centroids of 6155 reflections above 20 σ(I) with 4.997° < 2θ < 54.82°. Data were corrected for absorption effects using the multi-scan method (SADABS). The ratio of minimum to maximum apparent transmission was 0.939. The calculated minimum and maximum transmission coefficients (based on crystal size) are 0.9830 and 0.9920.

The structure was solved and refined using the Bruker SHELXTL Software Package, using the space group P n a 21, with Z = 4 for the formula unit, $C_{16}H_{19}NO_4$. The final anisotropic full-matrix least-squares refinement on F^2 with 199 variables converged at R1 = 3.65%, for the observed data and wR2 = 8.90% for all data. The goodness-of-fit was 1.078. The largest peak in the final difference electron density synthesis was 0.191 $e^-/Å^3$and the largest hole was -0.171 $e^-/Å^3$ with an RMS deviation of 0.042 $e^-/Å^3$. On the basis of the final model, the calculated density was 1.338 g/cm^3 and F(000), 616 e^-.

Table 1. Sample and crystal data for 7i.

Identification code	14gyte02_MEL14	
Chemical formula	$C_{16}H_{19}NO_4$	
Formula weight	289.32	
Temperature	120(2) K	
Wavelength	0.71073 Å	
Crystal size	0.080 x 0.120 x 0.180 mm	
Crystal habit	clear colourless block	
Crystal system	orthorhombic	
Space group	P n a 21	
Unit cell dimensions	a = 22.1303(4) Å	α = 90°
	b = 8.7685(2) Å	β = 90°
	c = 7.40320(10) Å	γ = 90°
Volume	1436.59(5) $Å^3$	
Z	4	
Density (calculated)	1.338 g/cm^3	
Absorption coefficient	0.096 mm^{-1}	
F(000)	616	

Table 2. Data collection and structure refinement for 7i.

Theta range for data collection	1.84 to 27.47°
Index ranges	-28<=h<=28, -11<=k<=11, -9<=l<=9
Reflections collected	23828
Independent reflections	3257 [R(int) = 0.0453]
Coverage of independent reflections	99.9%
Absorption correction	multi-scan
Max. and min. transmission	0.9920 and 0.9830
Structure solution technique	direct methods
Structure solution program	SHELXS-2013 (Sheldrick, 2013)
Refinement method	Full-matrix least-squares on F^2
Refinement program	SHELXL-2013 (Sheldrick, 2013)
Function minimized	$\Sigma w(F_o^2 - F_c^2)^2$

Data / restraints / parameters	3257 / 3 / 199	
Goodness-of-fit on F^2	1.078	
Final R indices	2982 data; $I>2\sigma(I)$	R1 = 0.0365, wR2 = 0.0858
	all data	R1 = 0.0424, wR2 = 0.0890
Weighting scheme	$w=1/[\sigma^2(F_o^2)+(0.0394P)^2+0.4673P]$ where $P=(F_o^2+2F_c^2)/3$	
Absolute structure parameter	0.2(4)	
Largest diff. peak and hole	0.191 and -0.171 eÅ^{-3}	
R.M.S. deviation from mean	0.042 eÅ^{-3}	

Table 3. Atomic coordinates and equivalent isotropic atomic displacement parameters (Å^2) for 7i.

U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x/a	y/b	z/c	U(eq)
C1	0.30481(11)	0.7536(3)	0.5203(4)	0.0192(5)
C2	0.34802(11)	0.0109(3)	0.5820(4)	0.0210(5)
C3	0.23878(10)	0.9610(3)	0.6488(3)	0.0167(5)
C4	0.29166(10)	0.9254(3)	0.5203(3)	0.0149(5)
C5	0.27415(10)	0.9799(3)	0.3294(3)	0.0160(5)
C6	0.21544(10)	0.9142(2)	0.2586(3)	0.0136(5)
C7	0.16845(10)	0.8949(2)	0.3914(3)	0.0136(5)
C8	0.18062(10)	0.9094(2)	0.5691(3)	0.0151(5)
C9	0.10526(10)	0.8301(3)	0.3690(3)	0.0132(5)
C10	0.08402(9)	0.8151(2)	0.5677(3)	0.0137(5)
C11	0.06656(10)	0.6593(3)	0.6224(3)	0.0165(5)
C12	0.02384(12)	0.6056(3)	0.7344(4)	0.0246(6)
C13	0.02856(13)	0.4435(3)	0.7316(4)	0.0288(6)
C14	0.07365(12)	0.4080(3)	0.6179(4)	0.0214(5)
C15	0.10050(13)	0.2620(3)	0.5548(4)	0.0292(6)
C16	0.06110(10)	0.9211(3)	0.2526(3)	0.0146(5)
N1	0.07916(10)	0.9472(2)	0.0834(3)	0.0204(5)
O1	0.20922(8)	0.88082(19)	0.0973(2)	0.0185(4)
O2	0.09826(7)	0.53974(18)	0.5480(2)	0.0169(4)
O3	0.01130(8)	0.9609(2)	0.3095(3)	0.0216(4)

	x/a	y/b	z/c	U(eq)
O4	0.13590(7)	0.86770(18)	0.6806(3)	0.0159(4)

Table 4. Bond lengths (Å) for 7i.

C1-C4	1.534(3)	C2-C4	1.525(3)
C3-C8	1.486(3)	C3-C4	1.540(3)
C4-C5	1.541(4)	C5-C6	1.515(3)
C6-O1	1.237(3)	C6-C7	1.441(3)
C7-C8	1.349(4)	C7-C9	1.519(3)
C8-O4	1.339(3)	C9-C16	1.528(3)
C9-C10	1.550(3)	C10-C11	1.477(3)
C10-O4	1.493(3)	C11-C12	1.343(3)
C11-O2	1.377(3)	C12-C13	1.425(4)
C13-C14	1.342(4)	C14-O2	1.378(3)
C14-C15	1.486(4)	C16-O3	1.230(3)
C16-N1	1.335(3)		

Table 5. Bond angles (°) for 7i.

C8-C3-C4	110.6(2)	C2-C4-C1	109.11(19)
C2-C4-C3	109.7(2)	C1-C4-C3	110.04(19)
C2-C4-C5	109.2(2)	C1-C4-C5	110.6(2)
C3-C4-C5	108.22(19)	C6-C5-C4	114.53(19)
O1-C6-C7	123.4(2)	O1-C6-C5	121.3(2)
C7-C6-C5	115.3(2)	C8-C7-C6	120.7(2)
C8-C7-C9	109.0(2)	C6-C7-C9	129.3(2)
O4-C8-C7	115.3(2)	O4-C8-C3	118.6(2)
C7-C8-C3	126.0(2)	C7-C9-C16	117.06(18)
C7-C9-C10	101.95(18)	C16-C9-C10	112.68(18)
C11-C10-O4	109.48(18)	C11-C10-C9	114.70(19)
O4-C10-C9	105.79(17)	C12-C11-O2	109.8(2)
C12-C11-C10	132.7(2)	O2-C11-C10	117.5(2)
C11-C12-C13	106.8(2)	C14-C13-C12	107.1(2)

C13-C14-O2	109.6(2)	C13-C14-C15	133.9(2)
O2-C14-C15	116.5(2)	O3-C16-N1	122.8(2)
O3-C16-C9	121.8(2)	N1-C16-C9	115.3(2)
C11-O2-C14	106.7(2)	C8-O4-C10	107.91(19)

Table 6. Torsion angles (°) for 7i.

C8-C3-C4-C2	166.8(2)	C8-C3-C4-C1	-73.1(2)
C8-C3-C4-C5	47.8(3)	C2-C4-C5-C6	-174.7(2)
C1-C4-C5-C6	65.2(3)	C3-C4-C5-C6	-55.4(3)
C4-C5-C6-O1	-143.4(2)	C4-C5-C6-C7	36.9(3)
O1-C6-C7-C8	169.1(2)	C5-C6-C7-C8	-11.2(3)
O1-C6-C7-C9	1.7(4)	C5-C6-C7-C9	-178.6(2)
C6-C7-C8-O4	-170.78(19)	C9-C7-C8-O4	-1.1(3)
C6-C7-C8-C3	6.9(4)	C9-C7-C8-C3	176.6(2)
C4-C3-C8-O4	150.97(19)	C4-C3-C8-C7	-26.6(3)
C8-C7-C9-C16	124.8(2)	C6-C7-C9-C16	-66.7(3)
C8-C7-C9-C10	1.4(2)	C6-C7-C9-C10	169.9(2)
C7-C9-C10-C11	-122.0(2)	C16-C9-C10-C11	111.7(2)
C7-C9-C10-O4	-1.2(2)	C16-C9-C10-O4	-127.56(19)
O4-C10-C11-C12	96.5(3)	C9-C10-C11-C12	-144.8(3)
O4-C10-C11-O2	-84.0(2)	C9-C10-C11-O2	34.7(3)
O2-C11-C12-C13	0.1(3)	C10-C11-C12-C13	179.6(3)
C11-C12-C13-C14	-0.2(3)	C12-C13-C14-O2	0.2(3)
C12-C13-C14-C15	-179.2(3)	C7-C9-C16-O3	-125.7(2)

C10-C9-C16-O3	-7.9(3)	C7-C9-C16-N1	57.6(3)
C10-C9-C16-N1	175.3(2)	C12-C11-O2-C14	0.0(3)
C10-C11-O2-C14	-179.6(2)	C13-C14-O2-C11	-0.2(3)
C15-C14-O2-C11	179.4(2)	C7-C8-O4-C10	0.2(3)
C3-C8-O4-C10	-177.66(19)	C11-C10-O4-C8	124.82(19)
C9-C10-O4-C8	0.7(2)		

Table 7. Anisotropic atomic displacement parameters (Å^2) for 7i.

The anisotropic atomic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2} U_{11} + ... + 2 h k a^* b^* U_{12}]$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C1	0.0219(12)	0.0170(11)	0.0188(12)	-0.0013(10)	-0.0021(10)	0.0018(9)
C2	0.0183(11)	0.0210(12)	0.0237(14)	-0.0015(11)	-0.0045(11)	-0.0034(9)
C3	0.0186(12)	0.0177(11)	0.0136(13)	-0.0033(9)	-0.0016(9)	-0.0014(9)
C4	0.0146(11)	0.0152(11)	0.0148(13)	-0.0012(9)	-0.0006(9)	-0.0004(8)
C5	0.0155(11)	0.0170(11)	0.0154(12)	0.0030(9)	0.0012(10)	-0.0010(9)
C6	0.0170(11)	0.0105(10)	0.0133(12)	0.0030(9)	-0.0007(9)	0.0017(8)
C7	0.0131(11)	0.0136(10)	0.0140(11)	0.0006(10)	-0.0005(9)	0.0020(9)
C8	0.0160(10)	0.0130(10)	0.0164(12)	-0.0007(10)	0.0013(10)	0.0022(8)
C9	0.0140(11)	0.0133(10)	0.0124(11)	-0.0008(9)	0.0004(9)	0.0008(8)
C10	0.0115(10)	0.0201(11)	0.0096(11)	0.0008(9)	-0.0017(9)	0.0016(8)
C11	0.0125(10)	0.0227(12)	0.0144(12)	-0.0005(9)	-0.0017(9)	0.0013(9)
C12	0.0222(12)	0.0300(14)	0.0215(14)	0.0007(11)	0.0064(11)	-0.0053(10)
C13	0.0319(15)	0.0314(15)	0.0230(15)	0.0052(12)	0.0015(12)	-0.0142(11)
C14	0.0255(13)	0.0192(11)	0.0195(13)	0.0047(10)	-0.0078(11)	-0.0064(10)
C15	0.0385(15)	0.0163(12)	0.0328(17)	0.0016(12)	-0.0108(13)	-0.0024(11)
C16	0.0149(10)	0.0130(10)	0.0160(12)	-0.0013(9)	-0.0029(10)	-0.0017(8)
N1	0.0162(10)	0.0295(11)	0.0156(12)	0.0057(9)	0.0010(9)	0.0055(8)

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O1	0.0199(8)	0.0219(9)	0.0137(9)	0.0002(7)	0.0011(7)	-0.0026(7)
O2	0.0180(8)	0.0163(8)	0.0164(9)	0.0019(7)	-0.0012(7)	-0.0003(6)
O3	0.0174(8)	0.0305(10)	0.0169(9)	0.0005(8)	0.0014(7)	0.0080(7)
O4	0.0164(8)	0.0205(8)	0.0109(8)	-0.0017(7)	0.0007(7)	-0.0009(6)

Table 8. Hydrogen atomic coordinates and isotropic atomic displacement parameters (Å^2) for 7i.

	x/a	y/b	z/c	U(eq)
H1A	0.3373	0.7315	0.4342	0.029
H1B	0.2683	0.6979	0.4851	0.029
H1C	0.3173	0.7218	0.6416	0.029
H2A	0.3820	0.9844	0.5032	0.032
H2B	0.3576	0.9823	0.7067	0.032
H2C	0.3406	1.1210	0.5756	0.032
H3A	0.2454	0.9088	0.7657	0.02
H3B	0.2371	1.0721	0.6717	0.02
H5A	0.2709	1.0925	0.3305	0.019
H5B	0.3071	0.9524	0.2448	0.019
H9	0.1088	0.7253	0.3168	0.016
H10	0.0490	0.8851	0.5878	0.016
H12	-0.0042	0.6643	0.8024	0.03
H13	0.0043	0.3736	0.7978	0.035
H15A	0.0810	0.1766	0.6173	0.044
H15B	0.1439	0.2615	0.5812	0.044
H15C	0.0943	0.2517	0.4243	0.044
H1	0.1153(10)	0.918(3)	0.047(4)	0.019
H2	0.0522(12)	0.988(3)	0.007(4)	0.019

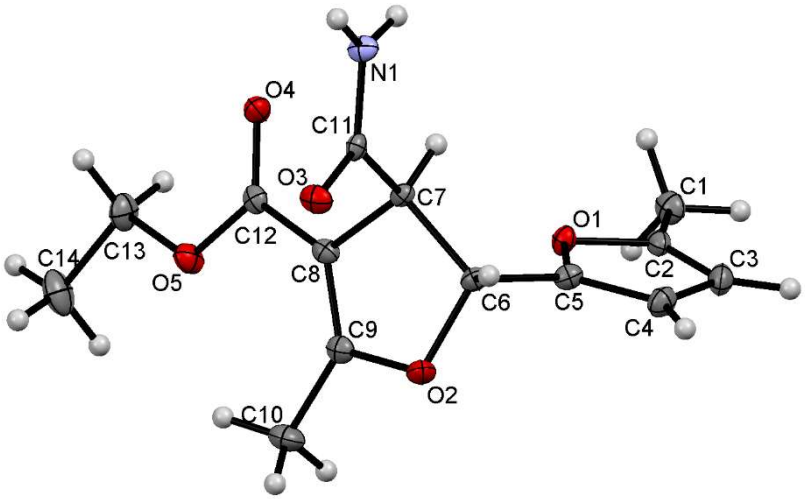
Table 9. Hydrogen bond distances (Å) and angles (°) for 7i.

	Donor-H	Acceptor-H	Donor-Acceptor	Angle
C9-H9···O2	1.0	2.37	2.874(3)	110.1

	Donor-H	Acceptor-H	Donor-Acceptor	Angle
N1-H1···O1	0.88(2)	2.14(2)	2.938(3)	151.0
C3-H3A···O1	0.99	2.59	3.457(3)	145.7
C10-H10···O3	1.0	2.51	3.393(3)	147.1
N1-H2···O3	0.89(2)	2.08(2)	2.961(3)	169.0

Crystallographic Data for 7j

CCDC 997071



A clear colourless block-like specimen of $C_{14}H_{17}NO_5$, approximate dimensions 0.278 mm x 0.437 mm x 0.528 mm, was used for the X-ray crystallographic analysis. The X-ray intensity data were measured.

The total exposure time was 3.83 hours. The frames were integrated with the Bruker SAINT software package using a wide-frame algorithm. The integration of the data using a monoclinic unit cell yielded a total of 23416reflections to a maximum θ angle of 27.50° (0.77 Å resolution), of which 3174 were independent (average redundancy 7.377, completeness = 99.7%, R_{int} = 2.52%, R_{sig} = 1.42%) and 2970 (93.57%) were greater than $2\sigma(F^2)$. The final cell constants of a = 8.8418(3) Å, b = 20.5412(7) Å, c = 8.1757(3) Å, β = 111.9312(13)°, volume = 1377.42(8) $Å^3$, are based upon the refinement of the XYZ-centroids

of 9872 reflections above 20 σ(I) with 4.966° < 2θ < 55.00°. Data were corrected for absorption effects using the multi-scan method (SADABS). The ratio of minimum to maximum apparent transmission was 0.890. The calculated minimum and maximum transmission coefficients (based on crystal size) are 0.9480 and 0.9720.

The structure was solved and refined using the Bruker SHELXTL Software Package, using the space group P 1 21/c 1, with Z = 4 for the formula unit, $C_{14}H_{17}NO_5$. The final anisotropic full-matrix least-squares refinement on F^2 with 190 variables converged at R1 = 3.62%, for the observed data and wR2 = 8.63% for all data. The goodness-of-fit was 1.066. The largest peak in the final difference electron density synthesis was 0.399 $e^-/Å^3$and the largest hole was -0.211 $e^-/Å^3$ with an RMS deviation of 0.045 $e^-/Å^3$. On the basis of the final model, the calculated density was 1.347 g/cm^3 and F(000), 592 e^-.

Table 1. Sample and crystal data for 7j.

Identification code	14gyte03_MEL16	
Chemical formula	$C_{14}H_{17}NO_5$	
Formula weight	279.28	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal size	0.278 x 0.437 x 0.528 mm	
Crystal habit	clear colourless block	
Crystal system	monoclinic	
Space group	P 1 21/c 1	
Unit cell dimensions	a = 8.8418(3) Å	α = 90°
	b = 20.5412(7) Å	β = 111.9312(13)°
	c = 8.1757(3) Å	γ = 90°
Volume	1377.42(8) $Å^3$	
Z	4	
Density (calculated)	1.347 g/cm^3	
Absorption coefficient	0.103 mm^{-1}	
F(000)	592	

Table 2. Data collection and structure refinement for 7j.

Theta range for data collection	1.98 to 27.50°
Index ranges	-10<=h<=11, -26<=k<=26, -10<=l<=10
Reflections collected	23416
Independent reflections	3174 [R(int) = 0.0252]
Coverage of independent reflections	99.7%
Absorption correction	multi-scan
Max. and min. transmission	0.9720 and 0.9480

Structure solution technique	direct methods	
Structure solution program	SHELXS-2013 (Sheldrick, 2013)	
Refinement method	Full-matrix least-squares on F^2	
Refinement program	SHELXL-2013 (Sheldrick, 2013)	
Function minimized	$\Sigma\ w(F_o^2 - F_c^2)^2$	
Data / restraints / parameters	3174 / 2 / 190	
Goodness-of-fit on F^2	1.066	
Final R indices	2970 data; $I>2\sigma(I)$	R1 = 0.0362, wR2 = 0.0848
	all data	R1 = 0.0386, wR2 = 0.0863
Weighting scheme	$w=1/[\sigma^2(F_o^2)+(0.0286P)^2+0.9456P]$ where $P=(F_o^2+2F_c^2)/3$	
Largest diff. peak and hole	0.399 and -0.211 eÅ^{-3}	
R.M.S. deviation from mean	0.045 eÅ^{-3}	

Table 3. Atomic coordinates and equivalent isotropic atomic displacement parameters (Å^2) for 7j.

U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x/a	y/b	z/c	U(eq)
C1	0.08462(16)	0.33535(6)	0.93801(16)	0.0183(2)
C2	0.12435(14)	0.33110(6)	0.77718(15)	0.0141(2)
C3	0.26029(15)	0.31760(6)	0.74555(16)	0.0178(2)
C4	0.21791(15)	0.32281(6)	0.55890(16)	0.0171(2)
C5	0.05860(14)	0.33885(6)	0.48933(15)	0.0127(2)
C6	0.94216(14)	0.34993(6)	0.30661(15)	0.0124(2)
C7	0.76538(13)	0.32950(5)	0.27730(14)	0.0106(2)
C8	0.68436(14)	0.39511(5)	0.26645(14)	0.0121(2)
C9	0.78562(14)	0.44243(6)	0.25505(15)	0.0141(2)

	x/a	y/b	z/c	U(eq)
C10	0.76843(17)	0.51427(6)	0.23479(18)	0.0214(3)
C11	0.68741(13)	0.29136(5)	0.10544(14)	0.0112(2)
C12	0.52603(14)	0.40059(6)	0.28081(14)	0.0124(2)
C13	0.31733(15)	0.47102(6)	0.29426(17)	0.0202(3)
C14	0.27641(18)	0.54244(7)	0.26711(19)	0.0271(3)
N1	0.62114(12)	0.23447(5)	0.11843(13)	0.0140(2)
O1	0.99741(10)	0.34457(4)	0.62097(10)	0.01311(17)
O2	0.93334(10)	0.42024(4)	0.26547(11)	0.01573(18)
O3	0.68846(11)	0.31408(4)	0.96607(11)	0.01649(18)
O4	0.45495(10)	0.35429(4)	0.31298(11)	0.01528(18)
O5	0.46610(10)	0.46135(4)	0.26019(12)	0.01765(19)

Table 4. Bond lengths (Å) for 7j.

C1-C2	1.4849(16)	C2-C3	1.3493(17)
C2-O1	1.3772(13)	C3-C4	1.4330(17)
C4-C5	1.3482(16)	C5-O1	1.3784(13)
C5-C6	1.4813(16)	C6-O2	1.4783(14)
C6-C7	1.5481(15)	C7-C8	1.5133(15)
C7-C11	1.5297(15)	C8-C9	1.3481(16)
C8-C12	1.4522(16)	C9-O2	1.3557(14)
C9-C10	1.4864(16)	C11-O3	1.2345(14)
C11-N1	1.3292(15)	C12-O4	1.2217(14)
C12-O5	1.3418(14)	C13-O5	1.4563(14)
C13-C14	1.5074(18)		

Table 5. Bond angles (°) for 7j.

C3-C2-O1	109.84(10)	C3-C2-C1	134.75(11)
O1-C2-C1	115.40(10)	C2-C3-C4	106.94(10)
C5-C4-C3	106.50(11)	C4-C5-O1	110.14(10)
C4-C5-C6	133.32(11)	O1-C5-C6	116.53(10)
O2-C6-C5	109.71(9)	O2-C6-C7	105.38(9)
C5-C6-C7	113.31(9)	C8-C7-C11	111.18(9)
C8-C7-C6	101.31(9)	C11-C7-C6	111.32(9)

C9-C8-C12	129.41(11)	C9-C8-C7	109.52(10)
C12-C8-C7	120.91(10)	C8-C9-O2	113.72(10)
C8-C9-C10	132.89(11)	O2-C9-C10	113.39(10)
O3-C11-N1	124.17(10)	O3-C11-C7	119.87(10)
N1-C11-C7	115.96(9)	O4-C12-O5	122.72(10)
O4-C12-C8	123.10(10)	O5-C12-C8	114.16(10)
O5-C13-C14	106.79(11)	C2-O1-C5	106.58(9)
C9-O2-C6	108.10(8)	C12-O5-C13	116.45(9)

Table 6. Torsion angles (°) for 7j.

O1-C2-C3-C4	0.07(14)	C1-C2-C3-C4	-178.84(13)
C2-C3-C4-C5	-0.28(14)	C3-C4-C5-O1	0.39(14)
C3-C4-C5-C6	-178.52(12)	C4-C5-C6-O2	-95.15(15)
O1-C5-C6-O2	85.99(11)	C4-C5-C6-C7	147.40(13)
O1-C5-C6-C7	-31.47(14)	O2-C6-C7-C8	-13.58(10)
C5-C6-C7-C8	106.37(10)	O2-C6-C7-C11	104.68(10)
C5-C6-C7-C11	-135.37(10)	C11-C7-C8-C9	-107.77(11)
C6-C7-C8-C9	10.60(11)	C11-C7-C8-C12	76.34(12)
C6-C7-C8-C12	-165.29(10)	C12-C8-C9-O2	172.17(11)
C7-C8-C9-O2	-3.27(13)	C12-C8-C9-C10	-7.2(2)
C7-C8-C9-C10	177.33(12)	C8-C7-C11-O3	60.01(13)
C6-C7-C11-O3	-52.13(14)	C8-C7-C11-N1	-119.78(11)
C6-C7-C11-N1	128.07(10)	C9-C8-C12-O4	-170.27(12)
C7-C8-C12-O4	4.72(17)	C9-C8-C12-O5	8.09(17)
C7-C8-C12-O5	-176.92(9)	C3-C2-O1-C5	0.17(13)
C1-C2-O1-C5	179.30(10)	C4-C5-O1-C2	-0.35(13)
C6-C5-O1-C2	178.77(9)	C8-C9-O2-C6	-6.23(13)

C10-C9-O2-C6	173.29(9)	C5-C6-O2-C9	-109.64(10)
C7-C6-O2-C9	12.67(11)	O4-C12-O5-C13	5.53(16)
C8-C12-O5-C13	-172.84(10)	C14-C13-O5-C12	178.68(10)

Table 7. Anisotropic atomic displacement parameters (Å^2) for 7j.

The anisotropic atomic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2} U_{11} + ... + 2 h k a^* b^* U_{12}]$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C1	0.0209(6)	0.0195(6)	0.0131(5)	-0.0005(4)	0.0045(5)	-0.0002(5)
C2	0.0141(5)	0.0120(5)	0.0127(5)	0.0007(4)	0.0010(4)	-0.0007(4)
C3	0.0132(5)	0.0184(6)	0.0182(6)	0.0008(4)	0.0019(4)	0.0020(4)
C4	0.0133(5)	0.0187(6)	0.0203(6)	-0.0013(5)	0.0074(5)	0.0006(4)
C5	0.0132(5)	0.0134(5)	0.0129(5)	-0.0023(4)	0.0065(4)	-0.0017(4)
C6	0.0122(5)	0.0134(5)	0.0126(5)	-0.0012(4)	0.0057(4)	-0.0017(4)
C7	0.0100(5)	0.0121(5)	0.0099(5)	-0.0005(4)	0.0041(4)	-0.0006(4)
C8	0.0151(5)	0.0107(5)	0.0108(5)	-0.0008(4)	0.0051(4)	0.0001(4)
C9	0.0167(5)	0.0146(5)	0.0112(5)	-0.0003(4)	0.0052(4)	-0.0009(4)
C10	0.0260(7)	0.0136(6)	0.0254(7)	0.0019(5)	0.0105(5)	-0.0033(5)
C11	0.0085(5)	0.0132(5)	0.0117(5)	-0.0013(4)	0.0037(4)	0.0019(4)
C12	0.0148(5)	0.0133(5)	0.0083(5)	-0.0007(4)	0.0034(4)	0.0012(4)
C13	0.0171(6)	0.0220(6)	0.0235(6)	0.0000(5)	0.0099(5)	0.0069(5)
C14	0.0310(7)	0.0247(7)	0.0254(7)	0.0012(5)	0.0101(6)	0.0147(6)
N1	0.0169(5)	0.0146(5)	0.0106(4)	-0.0023(4)	0.0052(4)	-0.0034(4)
O1	0.0111(4)	0.0173(4)	0.0106(4)	-0.0009(3)	0.0037(3)	0.0004(3)
O2	0.0141(4)	0.0156(4)	0.0180(4)	0.0027(3)	0.0064(3)	-0.0026(3)

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O3	0.0222(4)	0.0168(4)	0.0110(4)	-0.0003(3)	0.0067(3)	-0.0026(3)
O4	0.0135(4)	0.0157(4)	0.0179(4)	0.0025(3)	0.0072(3)	0.0009(3)
O5	0.0184(4)	0.0137(4)	0.0238(5)	0.0014(3)	0.0114(4)	0.0042(3)

Table 8. Hydrogen atomic coordinates and isotropic atomic displacement parameters (Å^2) for 7j.

	x/a	y/b	z/c	U(eq)
H1A	1.1812	0.3241	1.0416	0.028
H1B	0.9961	0.3050	0.9279	0.028
H1C	1.0503	0.3798	0.9507	0.028
H3	1.3643	0.3067	0.8308	0.021
H4	1.2882	0.3162	0.4965	0.021
H6	0.9796	0.3257	0.2225	0.015
H7	0.7621	0.3039	0.3799	0.013
H10A	0.6542	0.5264	0.2058	0.032
H10B	0.8032	0.5284	0.1398	0.032
H10C	0.8366	0.5353	0.3454	0.032
H13A	0.3345	0.4581	0.4165	0.024
H13B	0.2275	0.4445	0.2121	0.024
H14A	0.3683	0.5681	0.3454	0.041
H14B	0.1794	0.5515	0.2941	0.041
H14C	0.2549	0.5541	0.1442	0.041
H1	0.6299(19)	0.2197(8)	0.2241(18)	0.021
H2	0.5749(19)	0.2113(7)	0.0211(18)	0.021

Table 9. Hydrogen bond distances (Å) and angles (°) for 7j.

	Donor-H	Acceptor-H	Donor-Acceptor	Angle
C4-H4···O4	0.95	2.59	3.4642(15)	153.9
C10-H10A···O5	0.98	2.3	2.9639(16)	124.0

	Donor-H	Acceptor-H	Donor-Acceptor	Angle
N1-H1···O3	0.892(13)	1.974(13)	2.8571(13)	170.4
N1-H2···O4	0.886(13)	2.118(13)	2.9964(13)	171.3

