Electronic Supplementary Material (ESI) for Dalton Transactions.

Supporting Information

2-Phenylbenzothiazole Conjugated with Cyclopentadienyl Tricarbonyl [$CpM(CO)_3$] (M = Re, ^{99m}Tc) Complexes as Potential Imaging Probes for β-Amyloid Plaques

Jianhua Jia[†], Mengchao Cui[,†], Jiapei Dai[‡], Boli Liu[†]*

Contents:

1. Purity and retention time of key target compounds

Table S1. Purity and retention time of key target compounds

Compd	Flow rate (mL/min)	Mobile phase (ACN%)	Agela Technologies, 5 μm	Retention time (RT, min)	Purity (%)
20	1	80	4.6 × 250 mm	8.07	98.22
[^{99m}Tc]20	1	80	4.6 × 250 mm	9.08	99.75
21	1	80	4.6 × 250 mm	10.09	90.48
[^{99m}Tc]21	1	80	4.6 × 250 mm	11.50	99.27
22	1	80	4.6 × 250 mm	12.09	98.26
[^{99m}Tc]22	1	80	4.6 × 250 mm	13.45	99.43
23	1	80	4.6 × 250 mm	12.96	97.55
[^{99m}Tc]23	1	80	4.6 × 250 mm	14.47	98.03

2. *In vitro* autoradiography on brain sections of normal mice for complexes [^{99m}Tc]**20** - **23**

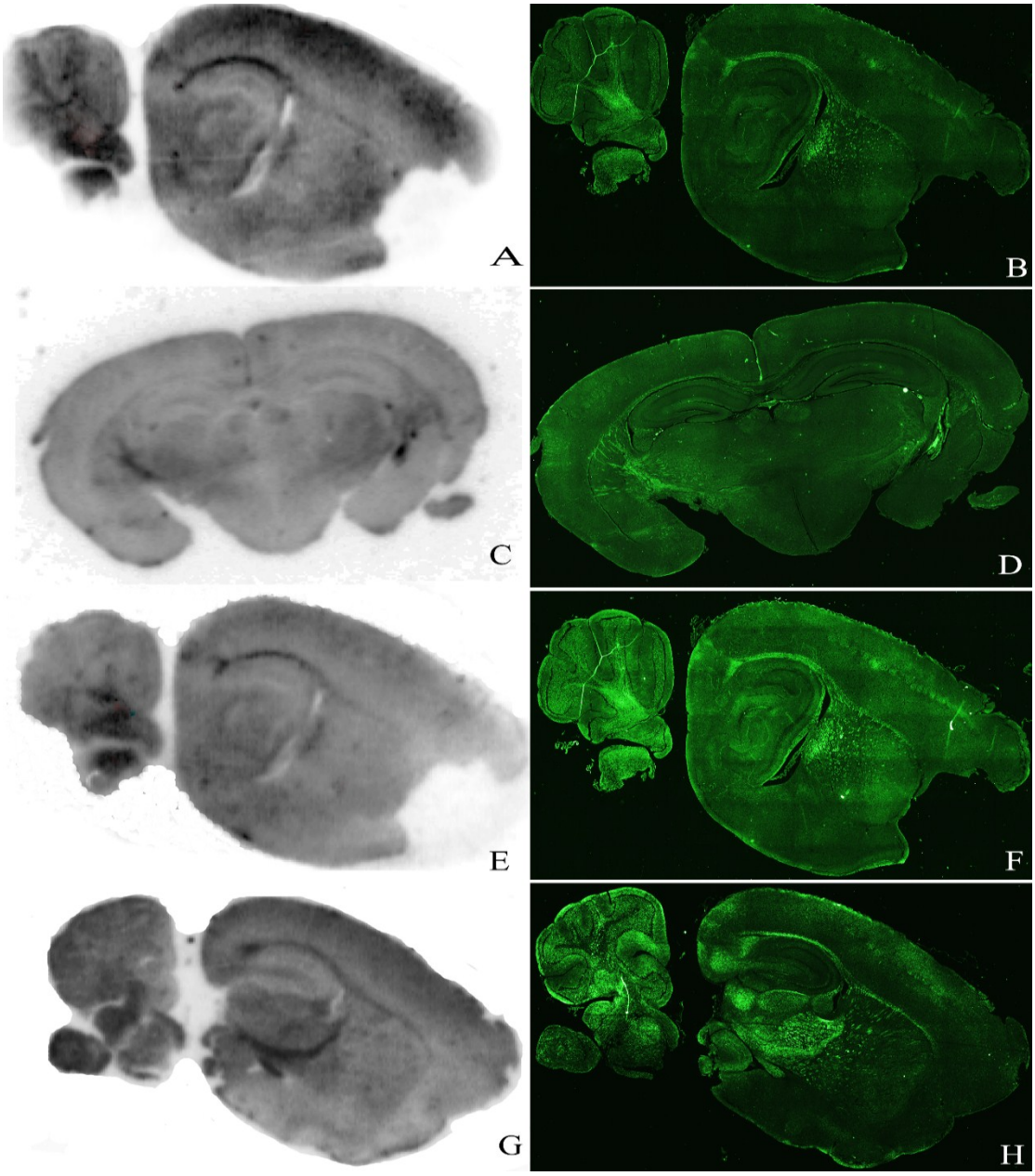


Figure S1. *In vitro* autoradiography of [^{99m}Tc]**20** (A), [^{99m}Tc]**21** (C), [^{99m}Tc]**22** (E) and [^{99m}Tc]**23** (G) on brain sections of wild-type (C57BL6, 12-month-old, female). The same sections were confirmed by fluorescence staining using Thioflavin-S (B, D, F and H).

3. *In vitro* autoradiography on brain sections of AD patient for complex [^{99m}Tc]**22**

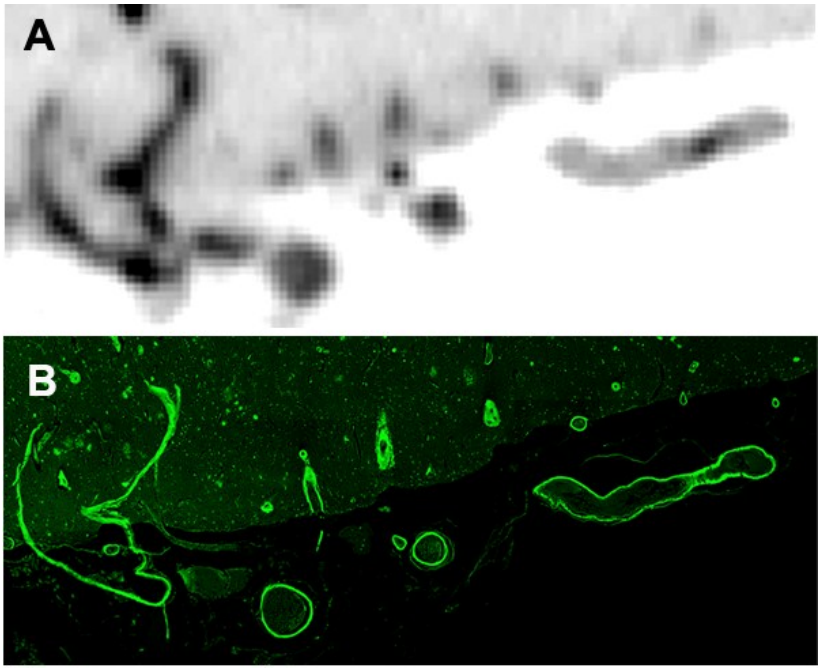


Figure S2. *In vitro* autoradiography of [^{99m}Tc]**22** (A) on brain sections of an AD patient (68-year-old, female, frontal lobe). The presence and distribution of cerebrovascular amyloids in the section were confirmed by fluorescence staining using Thioflavin-S (B).

4. Biodistribution experiments with normal mice of [^{99m}Tc]**20 - 23**[a]

Table S2. Biodistribution in normal mice (ICR, 5 weeks, 22 - 25 g, male) after i.v. injection of [^{99m}Tc]**20 - 23**[a]

Organ	2 min	10 min	30 min	60 min
		[^{99m}Tc]**20**		
Blood	2.96 ± 0.85	1.20 ± 0.17	0.66 ± 0.16	0.56 ± 0.12
Brain	0.50 ± 0.10	0.48 ± 0.09	0.28 ± 0.10	0.18 ± 0.07
Heart	10.03 ± 2.11	5.41 ± 0.83	2.30 ± 0.68	1.07 ± 0.36
Liver	33.61 ± 2.77	47.37 ± 5.54	45.58 ± 11.16	40.37 ± 7.78
Spleen	11.00 ± 2.56	11.52 ± 1.69	4.86 ± 0.60	2.44 ± 0.56
Lung	65.84 ± 12.78	53.52 ± 13.83	27.78 ± 5.78	15.58 ± 4.73
Kidney	9.38 ± 1.01	9.08 ± 0.96	5.84 ± 1.33	4.04 ± 0.64
Pancreas	2.27 ± 0.96	2.74 ± 0.43	1.31 ± 0.33	0.76 ± 0.22
muscle	1.52 ± 0.21	0.38 ± 0.03	0.26 ± 0.06	0.19 ± 0.04
Stomach [b]	0.63 ± 0.09	1.42 ± 0.76	3.04 ± 1.99	4.75 ± 3.20
Intestine [b]	2.26 ± 0.12	26.51 ± 2.93	49.06 ± 3.41	48.62 ± 5.09
		[^{99m}Tc]**21**		
Blood	5.84 ± 0.75	2.19 ± 0.63	1.23 ± 0.27	0.76 ± 0.10
Brain	0.36 ± 0.07	0.34 ± 0.10	0.29 ± 0.08	0.19 ± 0.02
Heart	17.65 ± 3.20	15.15 ± 2.76	5.78 ± 1.07	2.42 ± 0.49
Liver	53.56 ± 6.60	70.67 ± 10.20	67.61 ± 15.64	53.02 ± 10.28
Spleen	13.68 ± 2.91	13.85 ± 3.39	10.82 ± 2.84	2.21 ± 0.17
Lung	14.39 ± 3.40	6.30 ± 0.77	3.42 ± 1.14	1.41 ± 0.24
Kidney	13.04 ± 1.85	10.94 ± 1.61	6.36 ± 1.01	3.52 ± 0.46
Pancreas	4.21 ± 0.63	3.53 ± 1.08	2.41 ± 0.68	1.17 ± 0.32
muscle	2.81 ± 0.36	2.93 ± 0.61	1.79 ± 0.45	1.20 ± 0.21
Stomach [b]	0.88 ± 0.19	1.40 ± 0.30	1.66 ± 0.28	1.52 ± 0.43
Intestine [b]	3.55 ± 0.47	6.18 ± 1.63	11.02 ± 1.24	12.29 ± 2.57
		[^{99m}Tc]**22**		
Blood	3.28 ± 0.28	0.75 ± 0.08	0.54 ± 0.06	0.51 ± 0.07
Brain	0.26 ± 0.04	0.17 ± 0.02	0.15 ± 0.01	0.11 ± 0.02
Heart	14.96 ± 2.51	5.16 ± 0.77	2.15 ± 0.32	1.19 ± 0.11
Liver	46.18 ± 3.47	40.82 ± 6.26	48.37 ± 4.19	53.51 ± 2.41

Spleen	10.79 ± 2.00	3.02 ± 0.37	1.68 ± 0.21	1.23 ± 0.37
Lung	11.96 ± 2.73	2.22 ± 0.50	1.10 ± 0.22	0.85 ± 0.06
Kidney	14.46 ± 2.34	8.74 ± 1.41	4.78 ± 0.57	3.78 ± 0.41
Pancreas	3.99 ± 0.67	2.20 ± 0.28	0.95 ± 0.45	0.69 ± 0.09
muscle	2.76 ± 0.37	1.61 ± 0.19	1.18 ± 0.22	1.03 ± 0.15
Stomach [b]	0.81 ± 0.12	0.73 ± 0.14	0.90 ± 0.09	1.46 ± 0.27
Intestine [b]	3.69 ± 0.43	5.06 ± 1.19	11.61 ± 1.54	19.80 ± 2.37
		[^{99m}Tc]**23**		
Blood	9.56 ± 1.52	1.27 ± 0.14	0.68 ± 0.28	1.00 ± 0.12
Brain	0.37 ± 0.08	0.12 ± 0.01	0.11 ± 0.02	0.14 ± 0.03
Heart	19.25 ± 3.91	11.98 ± 1.72	6.80 ± 1.38	4.68 ± 0.73
Liver	53.74 ± 6.25	59.09 ± 7.59	57.52 ± 8.62	73.62 ± 6.65
Spleen	15.62 ± 2.57	5.90 ± 0.72	3.22 ± 0.60	2.20 ± 0.37
Lung	28.30 ± 5.09	4.35 ± 0.76	1.94 ± 0.24	1.74 ± 0.25
Kidney	17.79 ± 3.07	11.86 ± 1.33	7.29 ± 2.06	5.91 ± 0.96
Pancreas	3.25 ± 0.49	2.77 ± 0.57	1.57 ± 0.56	1.43 ± 0.18
muscle	2.78 ± 0.81	2.16 ± 0.33	1.76 ± 0.29	1.70 ± 0.30
Stomach [b]	1.26 ± 0.41	1.56 ± 0.39	2.01 ± 0.32	3.20 ± 0.83
Intestine [b]	4.26 ± 0.63	5.59 ± 0.64	10.02 ± 2.92	23.38 ± 5.44

[a] Expressed as % injected dose per gram. Average for 5 mice ± standard deviation.

[b] Expressed as % injected dose per organ.

5. The absorption and fluorescence spectra of rhenium complexes **20** - **23**

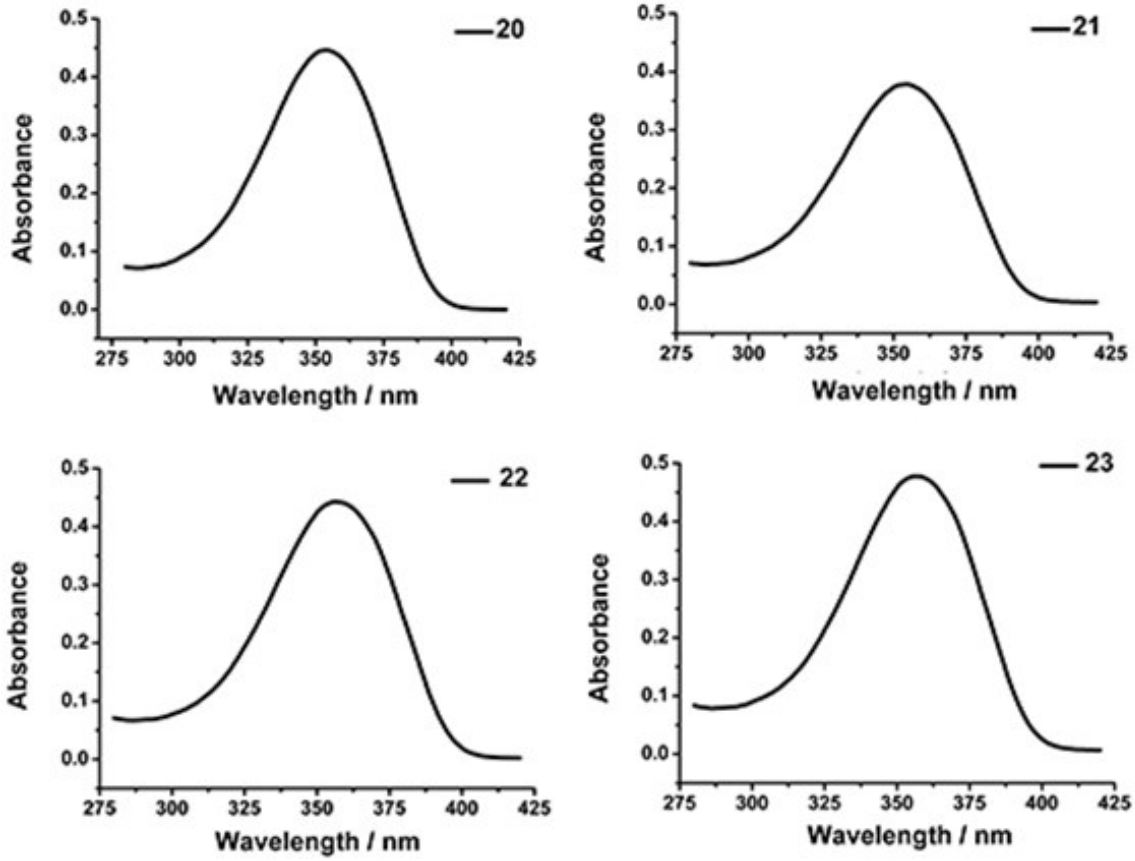


Figure S3. Absorption spectra of rhenium complexes **20 -23** (10 μM) in ethanol**.**

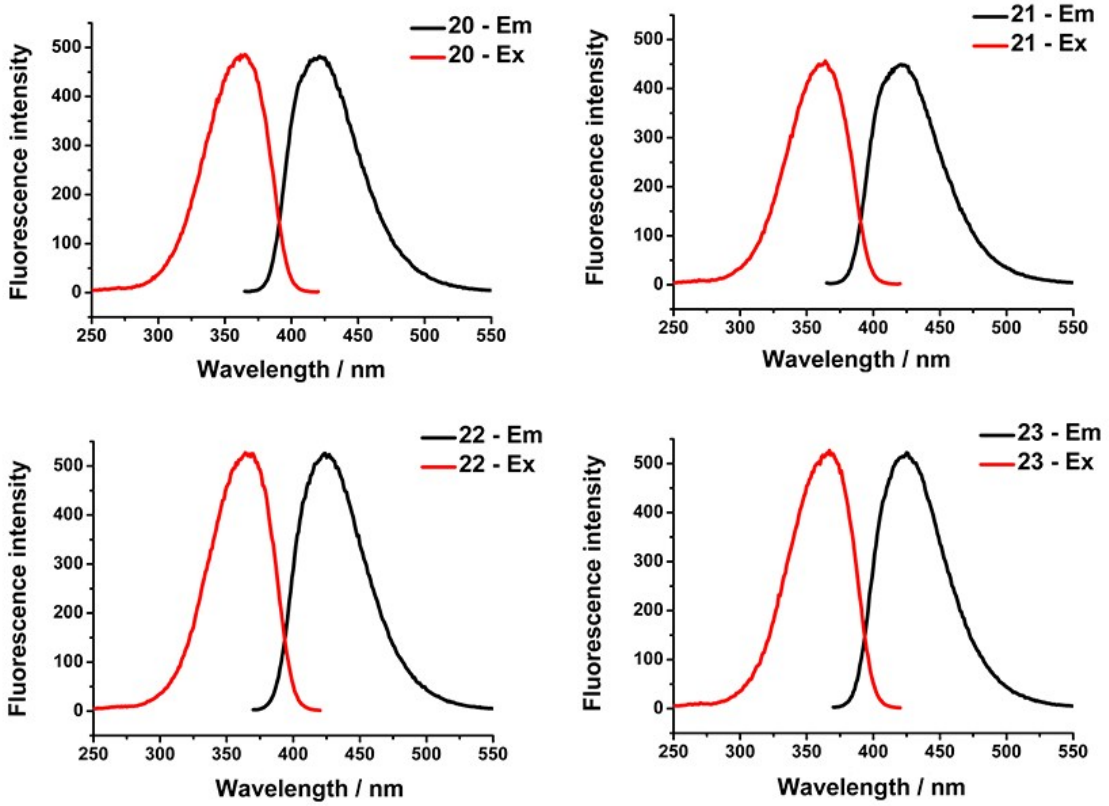


Figure S4. The excitation and emission spectra of rhenium complexes **20 -23** (10 μM) in ethanol.

6. Crystal data

Atomic parameters						
Atom	**Ox.**	**Wyck.**	**Site**	**S.O.F.**	**x/a**	**y/b**	**z/c**	**U [Å²]**
Re1		1a	1		0.92660(2)	0.27630(1)	-0.02164(1)	
S1		1a	1		1.00842(14)	0.83614(8)	0.56351(6)	
N1		1a	1		0.5616(4)	0.1577(3)	0.1324(2)	
H1N		1a	1		0.643(6)	0.168(4)	0.155(3)	0.011(11)
N2		1a	1		1.0992(5)	0.8214(3)	0.7253(2)	
N3		1a	1		1.3668(5)	1.3275(3)	0.7724(3)	
O1		1a	1		0.4266(4)	0.1150(3)	-0.0044(2)	
O2		1a	1		0.7268(4)	0.4378(2)	0.5423(2)	
O3		1a	1		1.2608(5)	0.4043(3)	0.0823(3)	
O4		1a	1		0.9834(6)	0.4070(3)	-0.1705(3)	
O5		1a	1		0.6978(6)	0.4178(3)	0.0774(3)	
C1		1a	1		0.8970(5)	0.1283(3)	0.0401(3)	
H1		1a	1		0.92250	0.13250	0.10220	0.0250
C2		1a	1		1.0171(5)	0.1201(3)	-0.0303(3)	
H2		1a	1		1.13680	0.11770	-0.02310	0.0300
C3		1a	1		0.9279(5)	0.1163(3)	-0.1123(3)	
H3		1a	1		0.97720	0.11120	-0.16980	0.0300
C4		1a	1		0.7507(5)	0.1214(3)	-0.0940(3)	
H4		1a	1		0.66120	0.12000	-0.13700	0.0260
C5		1a	1		0.7322(5)	0.1289(3)	0.0004(2)	
C6		1a	1		0.5606(5)	0.1337(3)	0.0429(3)	
C7		1a	1		0.3989(5)	0.1638(3)	0.1786(3)	
H7A		1a	1		0.31360	0.09680	0.16040	0.0290
H7B		1a	1		0.34860	0.22010	0.16050	0.0290
C8		1a	1		0.4302(5)	0.1861(3)	0.2798(3)	
H8A		1a	1		0.49930	0.13710	0.29630	0.0290
H8B		1a	1		0.31630	0.17280	0.30850	0.0290
C9		1a	1		0.5260(6)	0.2981(3)	0.3169(3)	
H9A		1a	1		0.44900	0.34670	0.30910	0.0290
H9B		1a	1		0.63100	0.31520	0.28180	0.0290
C10		1a	1		0.5813(6)	0.3150(3)	0.4161(3)	
H10A		1a	1		0.47670	0.29890	0.45180	0.0290
H10B		1a	1		0.65860	0.26670	0.42450	0.0290
C11		1a	1		0.6763(6)	0.4268(3)	0.4494(3)	
H11A		1a	1		0.78110	0.44360	0.41400	0.0290
H11B		1a	1		0.59900	0.47550	0.44240	0.0290
C12		1a	1		0.9507(5)	0.7145(3)	0.5938(3)	

C13	1a	1	0.8572(5)	0.6210(3)	0.5428(3)	
H13	1a	1	0.82000	0.61710	0.48230	0.0270
C14	1a	1	0.8206(5)	0.5339(3)	0.5839(3)	
C15	1a	1	0.8785(6)	0.5400(3)	0.6730(3)	
H15	1a	1	0.85300	0.47940	0.69970	0.0320
C16	1a	1	0.9721(6)	0.6332(3)	0.7222(3)	
H16	1a	1	1.01040	0.63670	0.78250	0.0310
C17	1a	1	1.0099(5)	0.7216(3)	0.6831(3)	
C18	1a	1	1.1059(5)	0.8891(3)	0.6720(3)	
C19	1a	1	1.1803(5)	1.0015(3)	0.6952(3)	
C20	1a	1	1.2760(5)	1.0451(3)	0.7763(3)	
H20	1a	1	1.29820	1.00010	0.81510	0.0240
C21	1a	1	1.3384(5)	1.1510(3)	0.8012(3)	
H21	1a	1	1.40430	1.17740	0.85630	0.0240
C22	1a	1	1.3071(5)	1.2217(3)	0.7467(3)	
C23	1a	1	1.2114(6)	1.1783(3)	0.6645(3)	
H23	1a	1	1.18920	1.22310	0.62540	0.0350
C24	1a	1	1.1498(6)	1.0708(3)	0.6405(3)	
H24	1a	1	1.08500	1.04350	0.58510	0.0330
C25	1a	1	1.3503(8)	1.3978(4)	0.7110(4)	
H25A	1a	1	1.41080	1.37830	0.65590	0.0480
H25B	1a	1	1.40270	1.47000	0.73950	0.0480
H25C	1a	1	1.22650	1.39240	0.69610	0.0480
C26	1a	1	1.4719(6)	1.3689(4)	0.8549(3)	
H26A	1a	1	1.40660	1.34670	0.90560	0.0410
H26B	1a	1	1.50020	1.44560	0.86410	0.0410
H26C	1a	1	1.58020	1.34240	0.85070	0.0410
C27	1a	1	1.1338(6)	0.3602(4)	0.0423(3)	
C28	1a	1	0.9637(6)	0.3598(4)	-0.1141(3)	
C29	1a	1	0.7884(7)	0.3687(4)	0.0402(4)	
Cl1	1a	1	0.3167(2)	-0.01040(13)	0.41464(14)	
Cl2	1a	1	0.2387(2)	-0.22911(15)	0.32531(14)	
C59	1a	1	0.3730(9)	-0.1328(5)	0.4025(5)	
H59A	1a	1	0.49510	-0.12620	0.38340	0.0670
H59B	1a	1	0.36780	-0.15490	0.46170	0.0670

Anisotropic displacement parameters, in Å²

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Re1	0.01791(9)	0.01831(9)	0.02425(9)	0.00111(5)	0.00001(6)	0.00432(6)
S1	0.0310(5)	0.0201(4)	0.0183(4)	0.0022(4)	-0.0035(4)	0.0042(3)
N1	0.0161(15)	0.0238(16)	0.0230(16)	0.0019(12)	-0.0009(12)	0.0019(13)
N2	0.0255(16)	0.0196(15)	0.0181(14)	0.0025(12)	-0.0020(12)	0.0019(12)

N3	0.037(2)	0.0187(16)	0.0329(19)	0.0013(14)	-0.0064(16)	0.0058(14)
O1	0.0165(13)	0.0431(18)	0.0245(14)	0.0052(12)	-0.0013(11)	0.0062(13)
O2	0.0356(17)	0.0213(14)	0.0230(14)	-0.0005(12)	-0.0038(12)	0.0023(11)
O3	0.0341(19)	0.050(2)	0.057(2)	-0.0122(17)	-0.0085(17)	0.002(2)
O4	0.056(3)	0.046(2)	0.047(2)	0.0013(19)	-0.0011(19)	0.0255(19)
O5	0.047(2)	0.037(2)	0.086(3)	0.0190(19)	0.018(2)	-0.005(2)
C1	0.0187(16)	0.0207(17)	0.0228(18)	0.0034(13)	-0.0028(14)	0.0036(14)
C2	0.0173(17)	0.0231(19)	0.033(2)	0.0032(14)	0.0005(15)	0.0038(16)
C3	0.0207(18)	0.0243(19)	0.028(2)	0.0047(15)	0.0060(15)	0.0026(16)
C4	0.0175(16)	0.0239(18)	0.0224(18)	0.0045(14)	0.0002(13)	0.0014(14)
C5	0.0171(15)	0.0162(16)	0.0190(16)	0.0021(12)	-0.0018(13)	-0.0009(13)
C6	0.0157(16)	0.0186(16)	0.0252(18)	0.0025(13)	0.0003(13)	0.0077(14)
C7	0.0187(17)	0.0267(19)	0.0233(18)	0.0011(15)	-0.0006(14)	0.0005(15)
C8	0.0225(18)	0.0246(19)	0.0230(18)	0.0010(15)	0.0017(14)	0.0016(15)
C9	0.0266(19)	0.0206(18)	0.0237(18)	0.0036(15)	-0.0001(15)	0.0014(15)
C10	0.0266(19)	0.0192(17)	0.0246(19)	0.0035(15)	-0.0004(15)	-0.0002(15)
C11	0.0261(19)	0.0221(18)	0.0213(18)	0.0025(15)	-0.0009(15)	0.0002(15)
C12	0.0226(17)	0.0215(17)	0.0177(16)	0.0050(14)	0.0007(13)	0.0026(14)
C13	0.0249(18)	0.0215(18)	0.0215(18)	0.0041(15)	-0.0019(14)	0.0032(14)
C14	0.0252(19)	0.0205(18)	0.0218(18)	0.0024(15)	0.0006(14)	0.0001(14)
C15	0.030(2)	0.026(2)	0.0234(19)	0.0008(16)	-0.0001(16)	0.0088(16)
C16	0.029(2)	0.0238(19)	0.0221(18)	0.0001(16)	-0.0041(15)	0.0058(15)
C17	0.0192(16)	0.0209(17)	0.0198(17)	0.0036(14)	-0.0005(13)	0.0007(14)
C18	0.0190(16)	0.0223(18)	0.0190(16)	0.0043(14)	-0.0002(13)	0.0030(14)
C19	0.0222(17)	0.0219(18)	0.0179(16)	0.0034(14)	0.0023(13)	0.0044(14)
C20	0.0187(16)	0.0238(18)	0.0191(16)	0.0056(14)	0.0004(13)	0.0074(14)
C21	0.0173(16)	0.0221(18)	0.0206(17)	0.0025(13)	-0.0021(13)	0.0025(14)
C22	0.0231(18)	0.0211(18)	0.0226(18)	0.0038(14)	0.0021(14)	0.0038(15)
C23	0.038(2)	0.025(2)	0.026(2)	0.0034(17)	-0.0051(17)	0.0093(16)
C24	0.033(2)	0.0235(19)	0.0232(19)	0.0011(16)	-0.0072(16)	0.0036(15)
C25	0.055(3)	0.022(2)	0.043(3)	0.000(2)	-0.004(2)	0.012(2)
C26	0.034(2)	0.024(2)	0.039(2)	0.0013(18)	-0.0084(19)	-0.0048(18)
C27	0.0228(19)	0.028(2)	0.031(2)	-0.0070(16)	-0.0039(16)	0.0003(17)
C28	0.034(2)	0.026(2)	0.030(2)	0.0001(17)	-0.0009(18)	0.0110(17)
C29	0.034(2)	0.023(2)	0.047(3)	0.0047(18)	0.002(2)	0.002(2)
Cl1	0.0626(10)	0.0425(8)	0.0921(13)	0.0059(7)	0.0068(9)	0.0218(9)
Cl2	0.0548(10)	0.0539(9)	0.0861(13)	0.0060(8)	0.0077(9)	-0.0013(9)
C59	0.052(4)	0.056(4)	0.060(4)	0.019(3)	0.007(3)	0.009(3)

Selected geometric informations

Atoms 1,2	d 1,2 [?]	Atoms 1,2	d 1,2 [?]
Re1—C27	1.915(4)	C8—H8B	0.9900

Re1—C29	1.919(5)	C9—C10	1.525(6)
Re1—C28	1.924(4)	C9—H9A	0.9900
Re1—C1	2.294(4)	C9—H9B	0.9900
Re1—C5	2.296(4)	C10—C11	1.510(5)
Re1—C2	2.305(4)	C10—H10A	0.9900
Re1—C4	2.310(4)	C10—H10B	0.9900
Re1—C3	2.311(4)	C11—H11A	0.9900
S1—C12	1.729(4)	C11—H11B	0.9900
S1—C18	1.768(4)	C12—C13	1.395(5)
N1—C6	1.334(5)	C12—C17	1.408(5)
N1—C7	1.464(5)	C13—C14	1.389(6)
N1—H1N	0.70(5)	C13—H13	0.9500
N2—C18	1.305(5)	C14—C15	1.404(6)
N2—C17	1.401(5)	C15—C16	1.383(6)
N3—C22	1.369(5)	C15—H15	0.9500
N3—C26	1.448(6)	C16—C17	1.387(6)
N3—C25	1.454(6)	C16—H16	0.9500
O1—C6	1.224(4)	C18—C19	1.463(5)
O2—C14	1.372(5)	C19—C24	1.398(6)
O2—C11	1.433(5)	C19—C20	1.400(5)
O3—C27	1.152(5)	C20—C21	1.371(5)
O4—C28	1.140(6)	C20—H20	0.9500
O5—C29	1.141(6)	C21—C22	1.411(5)
C1—C5	1.424(5)	C21—H21	0.9500
C1—C2	1.427(6)	C22—C23	1.414(6)
C1—H1	0.9500	C23—C24	1.388(6)
C2—C3	1.411(6)	C23—H23	0.9500
C2—H2	0.9500	C24—H24	0.9500
C3—C4	1.428(5)	C25—H25A	0.9800
C3—H3	0.9500	C25—H25B	0.9800
C4—C5	1.424(5)	C25—H25C	0.9800
C4—H4	0.9500	C26—H26A	0.9800
C5—C6	1.498(5)	C26—H26B	0.9800
C7—C8	1.517(6)	C26—H26C	0.9800
C7—H7A	0.9900	Cl1—C59	1.746(7)
C7—H7B	0.9900	Cl2—C59	1.733(7)
C8—C9	1.523(6)	C59—H59A	0.9900
C8—H8A	0.9900	C59—H59B	0.9900

Atoms 1,2,3	Angle 1,2,3 [iã]	Atoms 1,2,3	Angle 1,2,3 [iã]
C27—Re1—C29	90.8(2)	C9—C8—H8B	108.900

C27—Re1—C28	89.9(2)	H8A—C8—H8B	107.700
C29—Re1—C28	89.6(2)	C8—C9—C10	112.9(3)
C27—Re1—C1	100.48(17)	C8—C9—H9A	109.000
C29—Re1—C1	110.31(19)	C10—C9—H9A	109.000
C28—Re1—C1	157.18(18)	C8—C9—H9B	109.000
C27—Re1—C5	133.85(17)	C10—C9—H9B	109.000
C29—Re1—C5	92.92(18)	H9A—C9—H9B	107.800
C28—Re1—C5	136.11(16)	C11—C10—C9	110.8(3)
C1—Re1—C5	36.14(13)	C11—C10—H10A	109.500
C27—Re1—C2	94.79(18)	C9—C10—H10A	109.500
C29—Re1—C2	146.4(2)	C11—C10—H10B	109.500
C28—Re1—C2	123.38(19)	C9—C10—H10B	109.500
C1—Re1—C2	36.14(15)	H10A—C10—H10B	108.100
C5—Re1—C2	60.03(14)	O2—C11—C10	108.4(3)
C27—Re1—C4	154.54(18)	O2—C11—H11A	110.000
C29—Re1—C4	110.77(18)	C10—C11—H11A	110.000
C28—Re1—C4	103.22(17)	O2—C11—H11B	110.000
C1—Re1—C4	60.11(14)	C10—C11—H11B	110.000
C5—Re1—C4	36.03(13)	H11A—C11—H11B	108.400
C2—Re1—C4	59.77(14)	C13—C12—C17	122.2(4)
C27—Re1—C3	121.57(18)	C13—C12—S1	128.4(3)
C29—Re1—C3	146.75(18)	C17—C12—S1	109.4(3)
C28—Re1—C3	97.32(18)	C14—C13—C12	117.6(4)
C1—Re1—C3	59.93(15)	C14—C13—H13	121.200
C5—Re1—C3	59.98(14)	C12—C13—H13	121.200
C2—Re1—C3	35.58(15)	O2—C14—C13	123.7(4)
C4—Re1—C3	35.99(14)	O2—C14—C15	115.5(4)
C12—S1—C18	89.74(18)	C13—C14—C15	120.8(4)
C6—N1—C7	120.5(3)	C16—C15—C14	120.8(4)
C6—N1—H1N	117.(4)	C16—C15—H15	119.600
C7—N1—H1N	123.(4)	C14—C15—H15	119.600
C18—N2—C17	111.1(3)	C15—C16—C17	119.7(4)
C22—N3—C26	120.0(4)	C15—C16—H16	120.200
C22—N3—C25	120.4(4)	C17—C16—H16	120.200
C26—N3—C25	118.8(4)	C16—C17—N2	125.9(3)
C14—O2—C11	116.9(3)	C16—C17—C12	119.0(4)
C5—C1—C2	107.7(3)	N2—C17—C12	115.0(4)
C5—C1—Re1	72.0(2)	N2—C18—C19	126.1(3)
C2—C1—Re1	72.3(2)	N2—C18—S1	114.7(3)
C5—C1—H1	126.200	C19—C18—S1	119.2(3)
C2—C1—H1	126.200	C24—C19—C20	117.1(4)

Re1—C1—H1	121.300	C24—C19—C18	121.6(3)
C3—C2—C1	108.3(3)	C20—C19—C18	121.2(3)
C3—C2—Re1	72.4(2)	C21—C20—C19	121.8(4)
C1—C2—Re1	71.5(2)	C21—C20—H20	119.100
C3—C2—H2	125.800	C19—C20—H20	119.100
C1—C2—H2	125.800	C20—C21—C22	121.5(3)
Re1—C2—H2	121.900	C20—C21—H21	119.300
C2—C3—C4	108.2(4)	C22—C21—H21	119.300
C2—C3—Re1	72.0(2)	N3—C22—C21	121.4(4)
C4—C3—Re1	72.0(2)	N3—C22—C23	121.5(4)
C2—C3—H3	125.900	C21—C22—C23	117.1(4)
C4—C3—H3	125.900	C24—C23—C22	120.5(4)
Re1—C3—H3	121.900	C24—C23—H23	119.700
C5—C4—C3	107.7(3)	C22—C23—H23	119.700
C5—C4—Re1	71.4(2)	C23—C24—C19	122.0(4)
C3—C4—Re1	72.1(2)	C23—C24—H24	119.000
C5—C4—H4	126.200	C19—C24—H24	119.000
C3—C4—H4	126.200	N3—C25—H25A	109.500
Re1—C4—H4	122.100	N3—C25—H25B	109.500
C1—C5—C4	108.1(3)	H25A—C25—H25B	109.500
C1—C5—C6	130.0(3)	N3—C25—H25C	109.500
C4—C5—C6	121.8(3)	H25A—C25—H25C	109.500
C1—C5—Re1	71.9(2)	H25B—C25—H25C	109.500
C4—C5—Re1	72.5(2)	N3—C26—H26A	109.500
C6—C5—Re1	122.4(3)	N3—C26—H26B	109.500
O1—C6—N1	122.7(4)	H26A—C26—H26B	109.500
O1—C6—C5	119.9(4)	N3—C26—H26C	109.500
N1—C6—C5	117.4(3)	H26A—C26—H26C	109.500
N1—C7—C8	111.1(3)	H26B—C26—H26C	109.500
N1—C7—H7A	109.400	O3—C27—Re1	175.2(5)
C8—C7—H7A	109.400	O4—C28—Re1	178.1(4)
N1—C7—H7B	109.400	O5—C29—Re1	175.4(5)
C8—C7—H7B	109.400	Cl2—C59—Cl1	113.5(4)
H7A—C7—H7B	108.000	Cl2—C59—H59A	108.900
C7—C8—C9	113.4(4)	Cl1—C59—H59A	108.900
C7—C8—H8A	108.900	Cl2—C59—H59B	108.900
C9—C8—H8A	108.900	Cl1—C59—H59B	108.900
C7—C8—H8B	108.900	H59A—C59—H59B	107.700

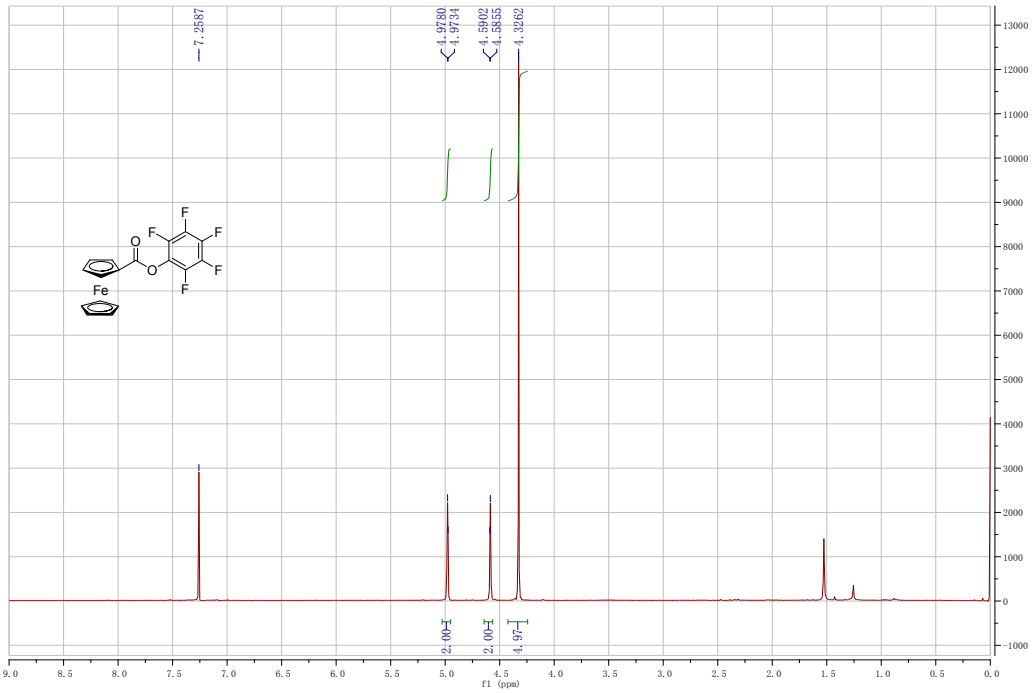
Atoms 1,2,3,4	Tors. an. 1,2,3,4 [iã]	Atoms 1,2,3,4	Tors. an. 1,2,3,4 [iã]
C27—Re1—C1—C5	160.3(2)	C28—Re1—C5—C6	-87.4(4)

C29—Re1—C1—C5	65.5(3)	C1—Re1—C5—C6	126.4(4)
C28—Re1—C1—C5	-84.1(5)	C2—Re1—C5—C6	164.2(3)
C2—Re1—C1—C5	-116.0(3)	C4—Re1—C5—C6	-117.1(4)
C4—Re1—C1—C5	-37.4(2)	C3—Re1—C5—C6	-154.5(3)
C3—Re1—C1—C5	-79.2(2)	C7—N1—C6—O1	-0.7(6)
C27—Re1—C1—C2	-83.7(3)	C7—N1—C6—C5	180.0(3)
C29—Re1—C1—C2	-178.5(3)	C1—C5—C6—O1	-167.1(4)
C28—Re1—C1—C2	31.9(5)	C4—C5—C6—O1	11.3(6)
C5—Re1—C1—C2	116.0(3)	Re1—C5—C6—O1	99.9(4)
C4—Re1—C1—C2	78.6(2)	C1—C5—C6—N1	12.2(6)
C3—Re1—C1—C2	36.8(2)	C4—C5—C6—N1	-169.4(4)
C5—C1—C2—C3	0.3(5)	Re1—C5—C6—N1	-80.8(4)
Re1—C1—C2—C3	-63.5(3)	C6—N1—C7—C8	176.8(4)
C5—C1—C2—Re1	63.8(3)	N1—C7—C8—C9	72.3(5)
C27—Re1—C2—C3	-141.8(3)	C7—C8—C9—C10	-171.2(4)
C29—Re1—C2—C3	119.5(4)	C8—C9—C10—C11	179.7(4)
C28—Re1—C2—C3	-48.8(3)	C14—O2—C11—C10	177.8(4)
C1—Re1—C2—C3	117.0(3)	C9—C10—C11—O2	-179.4(3)
C5—Re1—C2—C3	79.3(2)	C18—S1—C12—C13	178.1(4)
C4—Re1—C2—C3	37.4(2)	C18—S1—C12—C17	-0.5(3)
C27—Re1—C2—C1	101.2(3)	C17—C12—C13—C14	1.1(6)
C29—Re1—C2—C1	2.6(4)	S1—C12—C13—C14	-177.3(3)
C28—Re1—C2—C1	-165.8(2)	C11—O2—C14—C13	4.8(6)
C5—Re1—C2—C1	-37.7(2)	C11—O2—C14—C15	-175.7(4)
C4—Re1—C2—C1	-79.6(2)	C12—C13—C14—O2	178.5(4)
C3—Re1—C2—C1	-117.0(3)	C12—C13—C14—C15	-1.0(6)
C1—C2—C3—C4	-0.3(5)	O2—C14—C15—C16	-179.0(4)
Re1—C2—C3—C4	-63.3(3)	C13—C14—C15—C16	0.5(7)
C1—C2—C3—Re1	62.9(3)	C14—C15—C16—C17	-0.2(7)
C27—Re1—C3—C2	46.3(3)	C15—C16—C17—N2	178.3(4)
C29—Re1—C3—C2	-118.7(4)	C15—C16—C17—C12	0.4(6)
C28—Re1—C3—C2	140.7(3)	C18—N2—C17—C16	-176.7(4)
C1—Re1—C3—C2	-37.4(2)	C18—N2—C17—C12	1.3(5)
C5—Re1—C3—C2	-79.4(3)	C13—C12—C17—C16	-0.8(6)
C4—Re1—C3—C2	-116.9(4)	S1—C12—C17—C16	177.8(3)
C27—Re1—C3—C4	163.2(3)	C13—C12—C17—N2	-179.0(4)
C29—Re1—C3—C4	-1.8(5)	S1—C12—C17—N2	-0.3(4)
C28—Re1—C3—C4	-102.4(3)	C17—N2—C18—C19	176.4(4)
C1—Re1—C3—C4	79.5(3)	C17—N2—C18—S1	-1.7(4)
C5—Re1—C3—C4	37.5(2)	C12—S1—C18—N2	1.3(3)
C2—Re1—C3—C4	116.9(4)	C12—S1—C18—C19	-176.9(3)

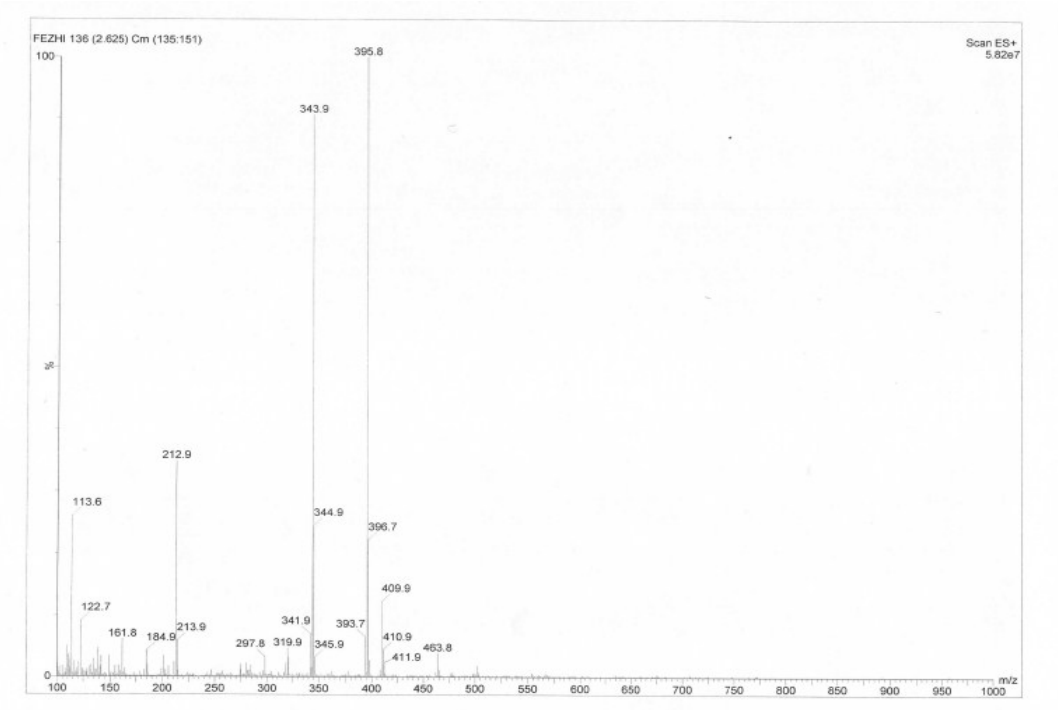
C2—C3—C4—C5	0.3(5)	N2—C18—C19—C24	-166.6(4)
Re1—C3—C4—C5	-63.0(3)	S1—C18—C19—C24	11.4(6)
C2—C3—C4—Re1	63.3(3)	N2—C18—C19—C20	9.3(6)
C27—Re1—C4—C5	81.5(5)	S1—C18—C19—C20	-172.7(3)
C29—Re1—C4—C5	-64.6(3)	C24—C19—C20—C21	-0.4(6)
C28—Re1—C4—C5	-159.3(2)	C18—C19—C20—C21	-176.5(4)
C1—Re1—C4—C5	37.5(2)	C19—C20—C21—C22	0.9(6)
C2—Re1—C4—C5	79.5(2)	C26—N3—C22—C21	3.1(7)
C3—Re1—C4—C5	116.4(3)	C25—N3—C22—C21	173.0(4)
C27—Re1—C4—C3	-35.0(5)	C26—N3—C22—C23	-176.8(4)
C29—Re1—C4—C3	178.9(3)	C25—N3—C22—C23	-7.0(7)
C28—Re1—C4—C3	84.2(3)	C20—C21—C22—N3	178.8(4)
C1—Re1—C4—C3	-78.9(3)	C20—C21—C22—C23	-1.3(6)
C5—Re1—C4—C3	-116.4(3)	N3—C22—C23—C24	-179.0(4)
C2—Re1—C4—C3	-36.9(2)	C21—C22—C23—C24	1.1(7)
C2—C1—C5—C4	-0.1(4)	C22—C23—C24—C19	-0.5(7)
Re1—C1—C5—C4	63.9(3)	C20—C19—C24—C23	0.2(7)
C2—C1—C5—C6	178.5(4)	C18—C19—C24—C23	176.2(4)
Re1—C1—C5—C6	-117.5(4)	C29—Re1—C27—O3	129.(5)
C2—C1—C5—Re1	-64.0(3)	C28—Re1—C27—O3	-141.(5)
C3—C4—C5—C1	-0.1(4)	C1—Re1—C27—O3	18.(5)
Re1—C4—C5—C1	-63.5(3)	C5—Re1—C27—O3	34.(6)
C3—C4—C5—C6	-178.8(3)	C2—Re1—C27—O3	-18.(5)
Re1—C4—C5—C6	117.8(3)	C4—Re1—C27—O3	-20.(6)
C3—C4—C5—Re1	63.4(3)	C3—Re1—C27—O3	-43.(6)
C27—Re1—C5—C1	-27.4(3)	C27—Re1—C28—O4	152.(15)
C29—Re1—C5—C1	-121.3(3)	C29—Re1—C28—O4	-117.(15)
C28—Re1—C5—C1	146.2(3)	C1—Re1—C28—O4	34.(15)
C2—Re1—C5—C1	37.7(2)	C5—Re1—C28—O4	-23.(15)
C4—Re1—C5—C1	116.5(3)	C2—Re1—C28—O4	56.(15)
C3—Re1—C5—C1	79.0(2)	C4—Re1—C28—O4	-6.(15)
C27—Re1—C5—C4	-143.9(3)	C3—Re1—C28—O4	30.(15)
C29—Re1—C5—C4	122.2(3)	C27—Re1—C29—O5	-140.(7)
C28—Re1—C5—C4	29.7(4)	C28—Re1—C29—O5	130.(7)
C1—Re1—C5—C4	-116.5(3)	C1—Re1—C29—O5	-39.(7)
C2—Re1—C5—C4	-78.7(2)	C5—Re1—C29—O5	-6.(7)
C3—Re1—C5—C4	-37.4(2)	C2—Re1—C29—O5	-41.(7)
C27—Re1—C5—C6	99.0(3)	C4—Re1—C29—O5	26.(7)
C29—Re1—C5—C6	5.2(3)	C3—Re1—C29—O5	27.(7)

7. ^{1}H-NMR, ^{13}C-NMR, MS and HRMS data of synthesized compounds

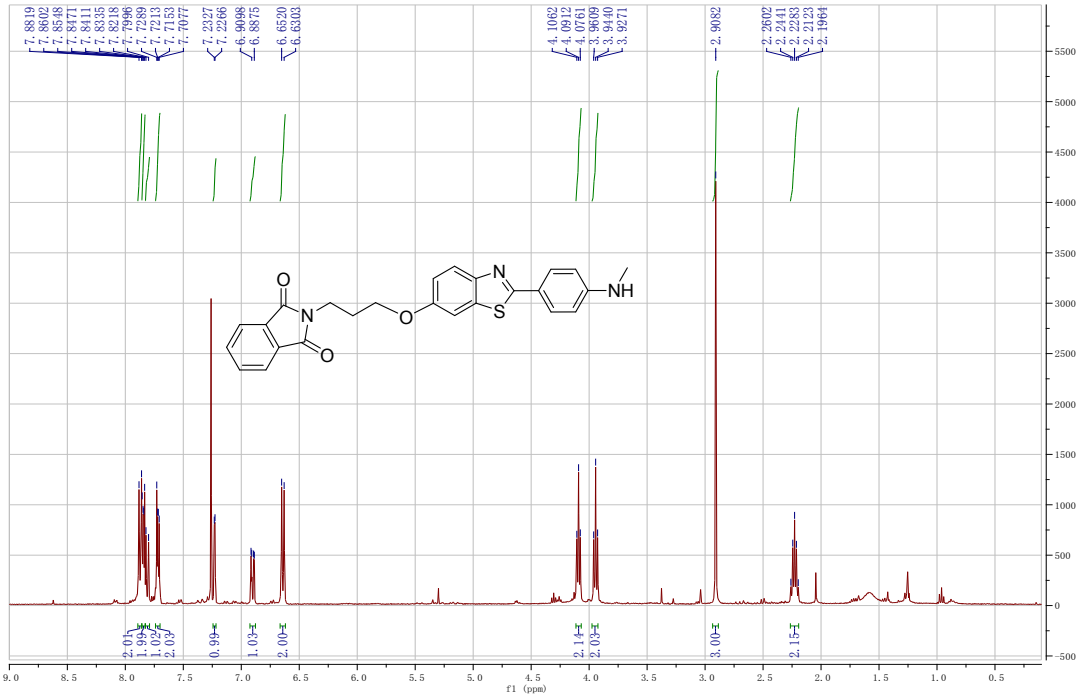
^{1}H-NMR for compound **1**



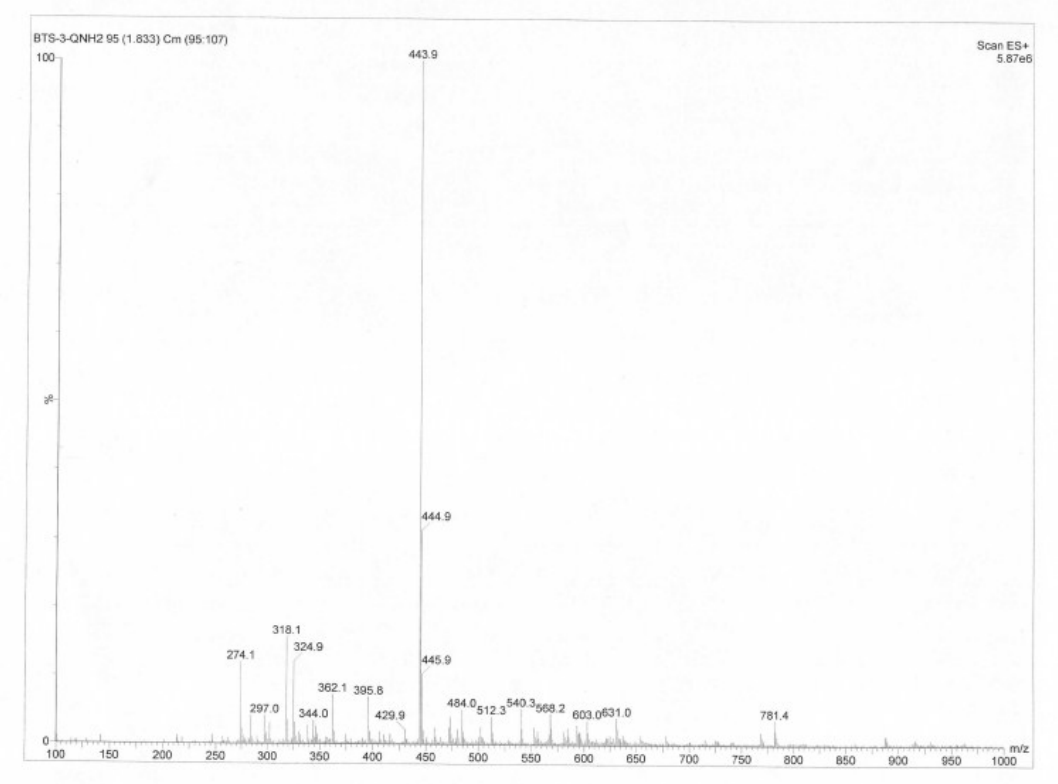
MS for compound **1**



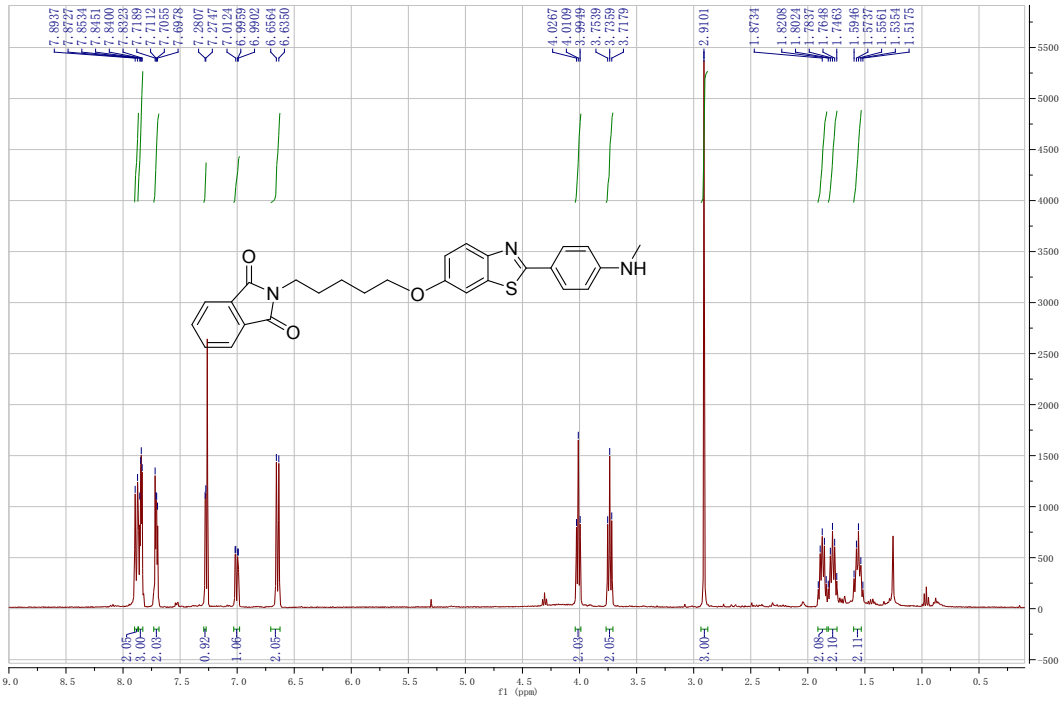
^{1}H-NMR for compound **8**



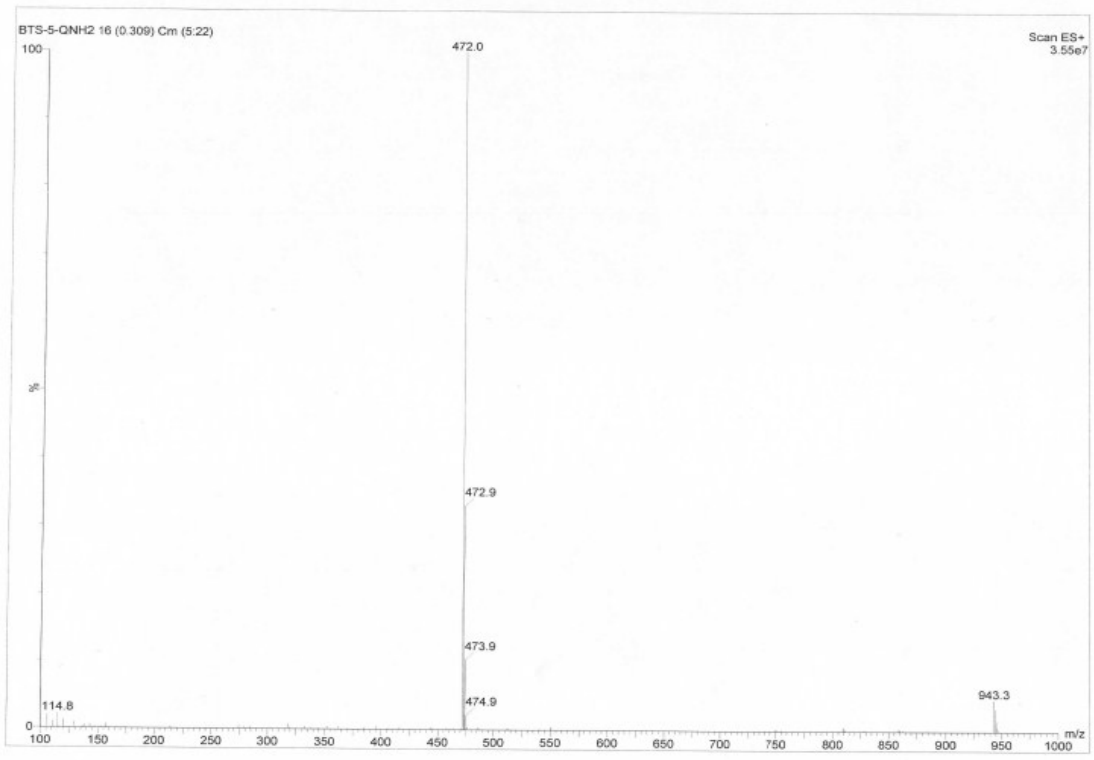
MS for compound **8**



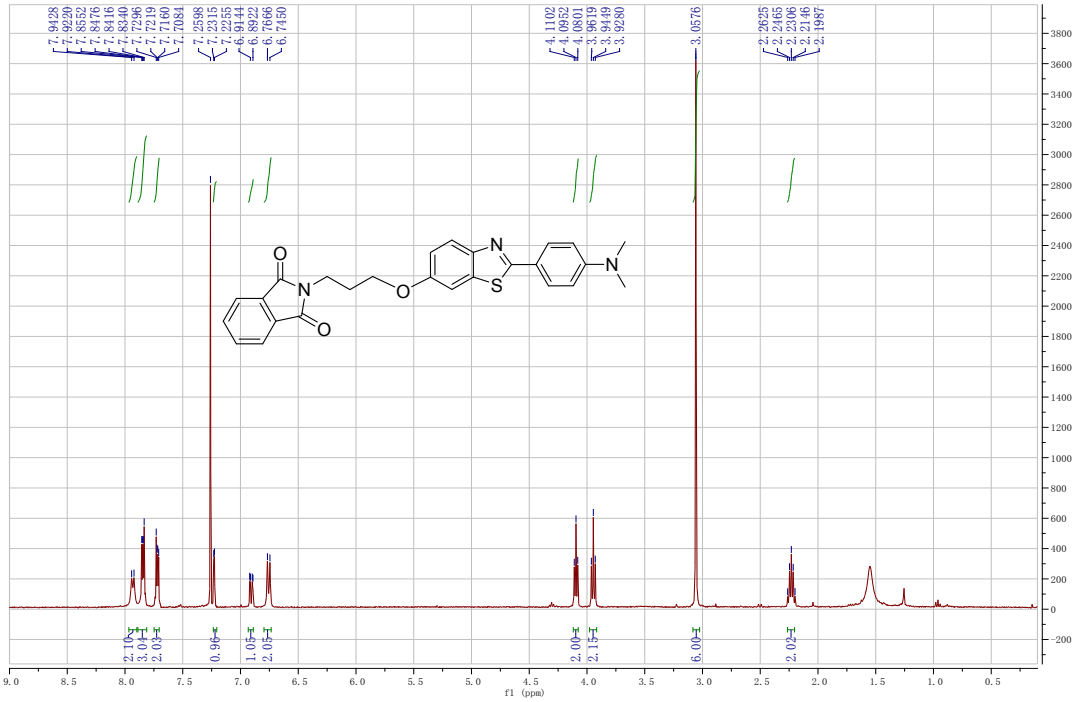
^{1}H-NMR for compound **9**



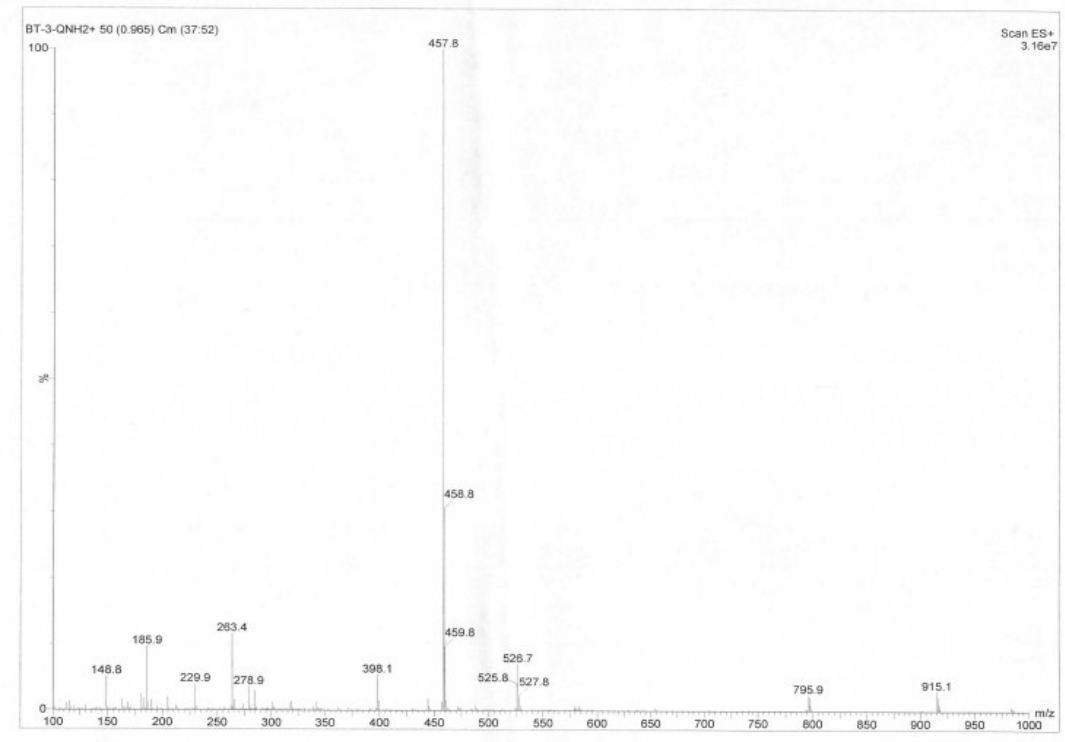
MS for compound **9**



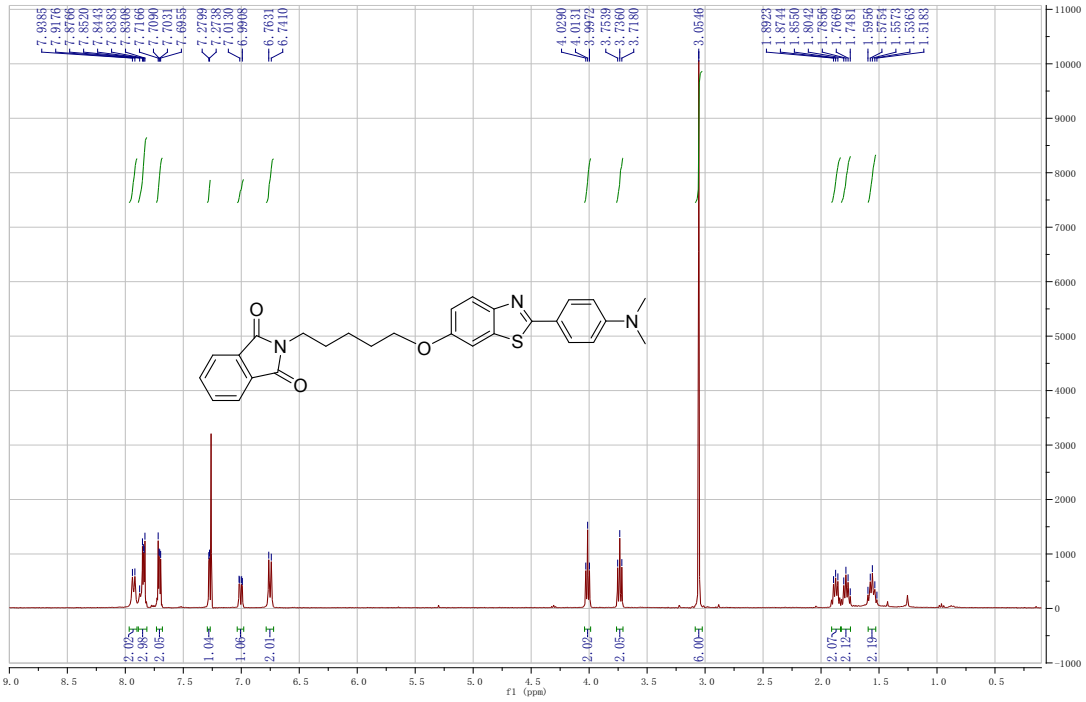
^{1}H-NMR for compound **10**



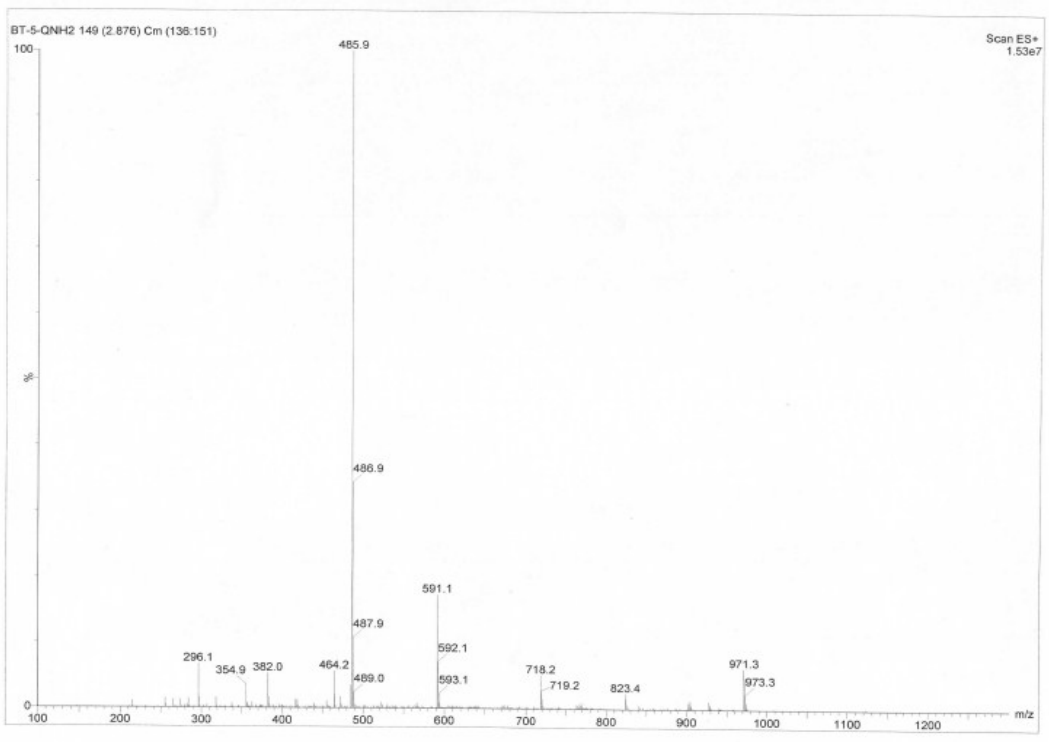
MS for compound **10**



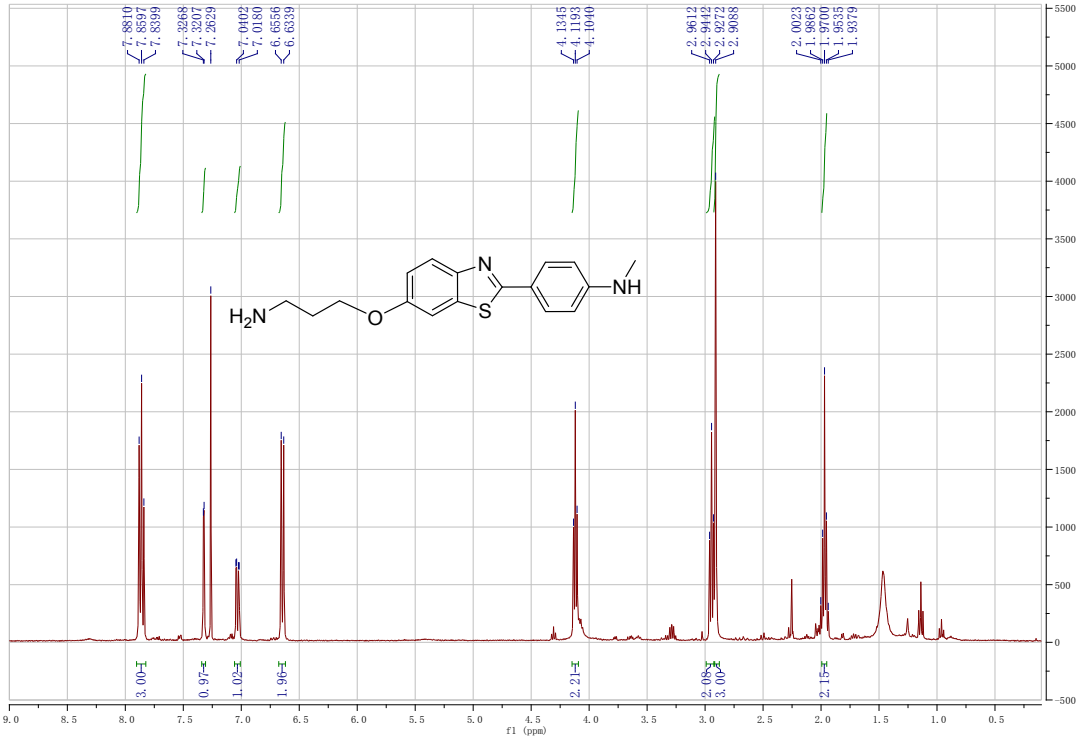
^{1}H-NMR for compound **11**



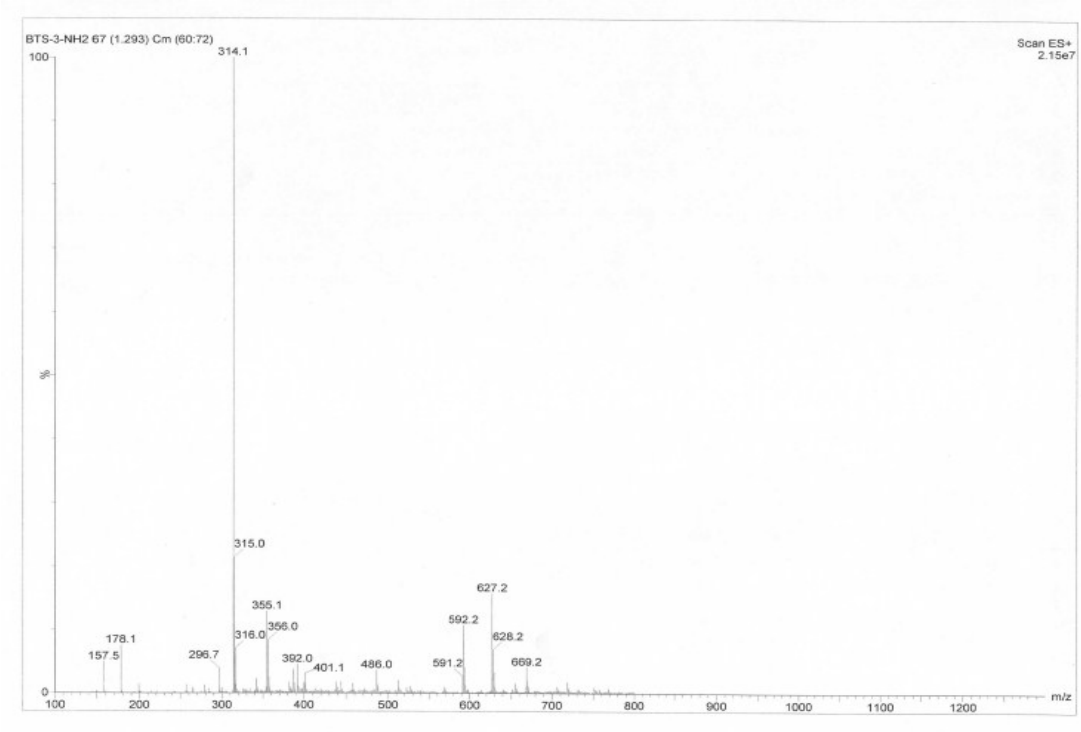
MS for compound **11**



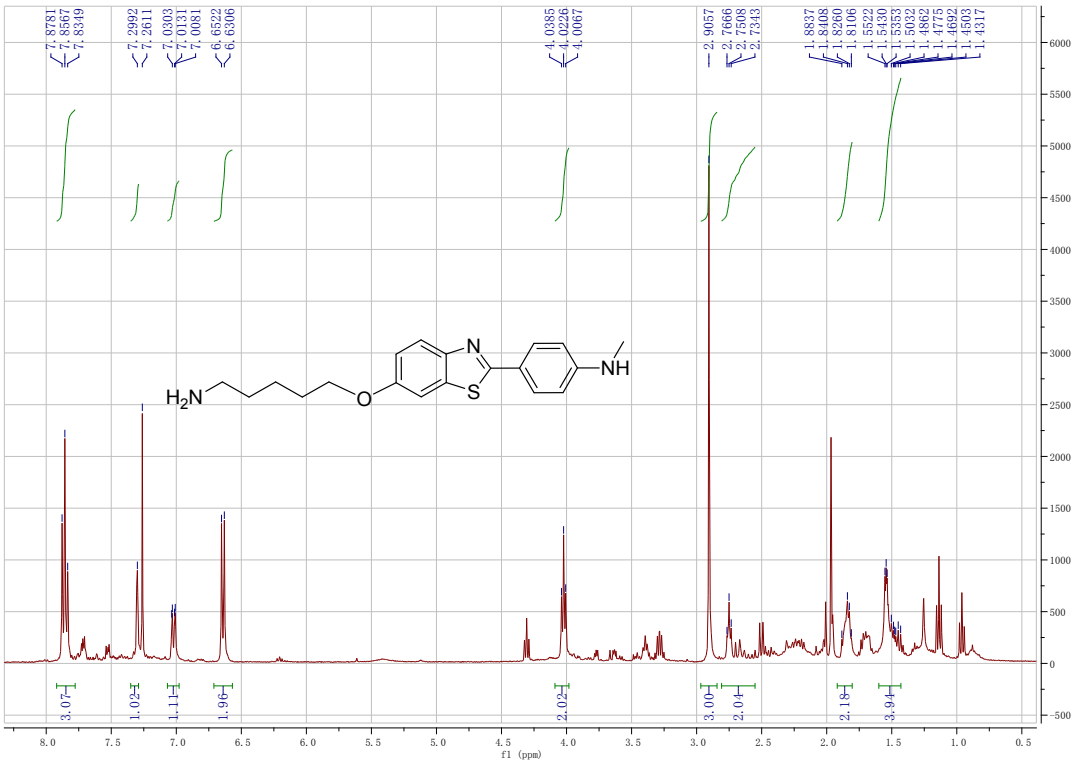
^{1}H-NMR for compound **12**



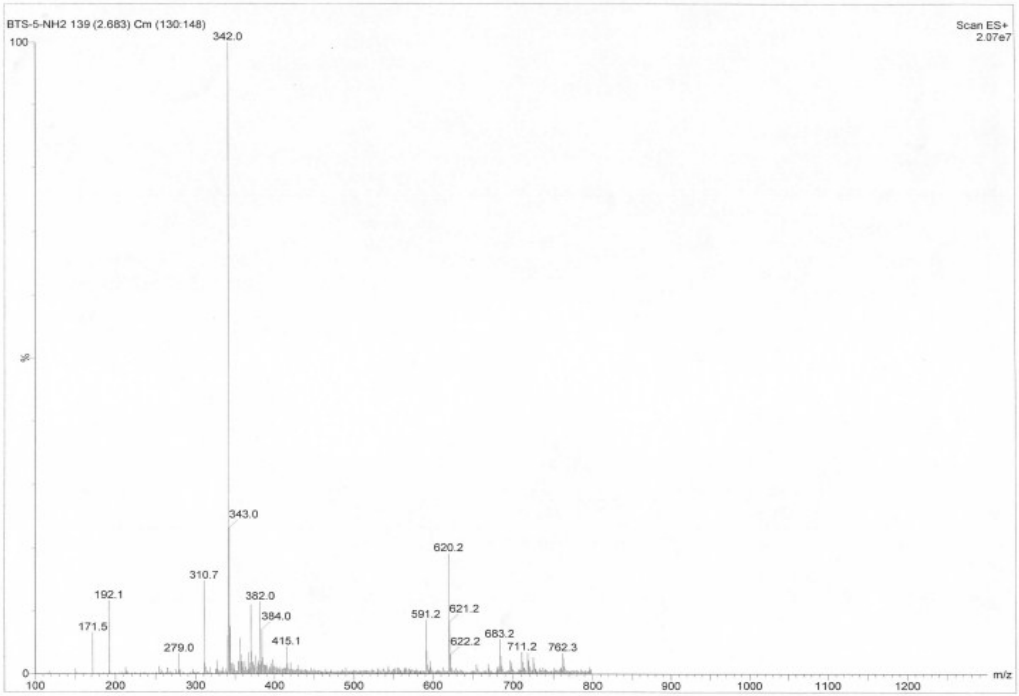
MS for compound **12**



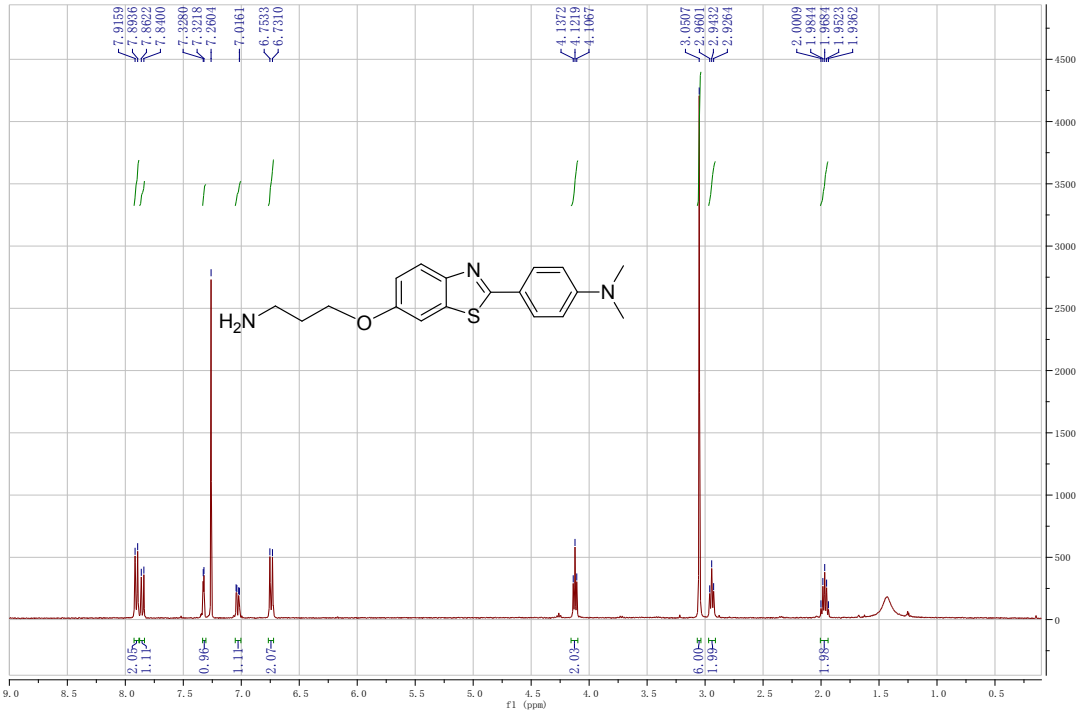
^{1}H-NMR for compound **13**



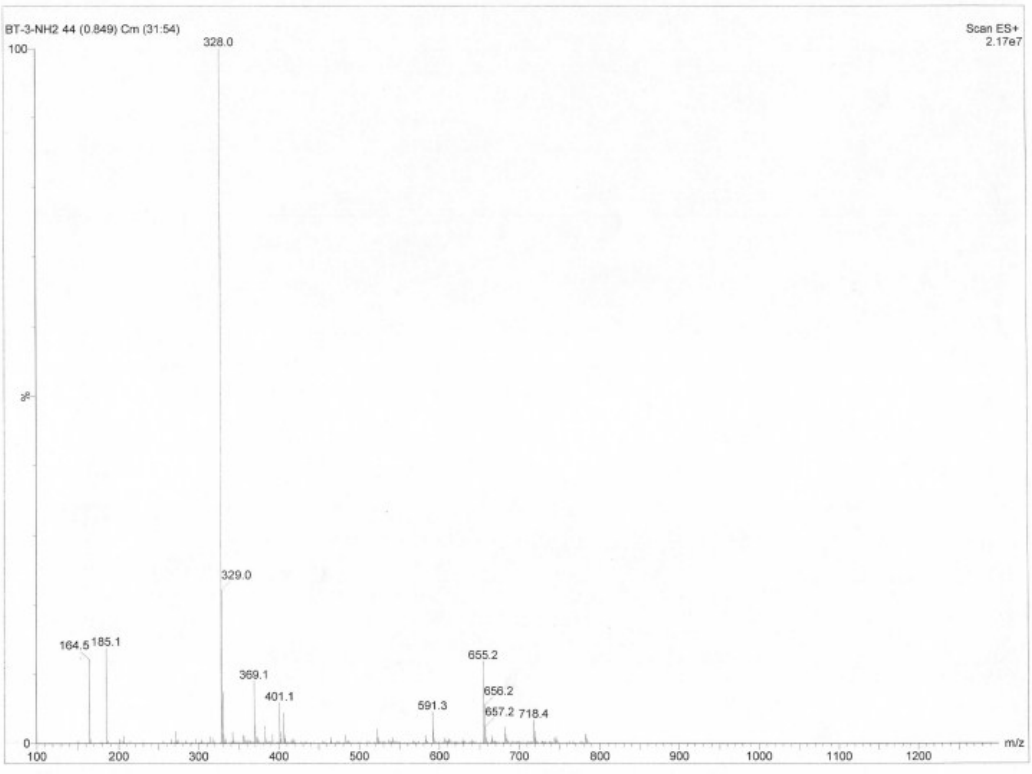
MS for compound **13**



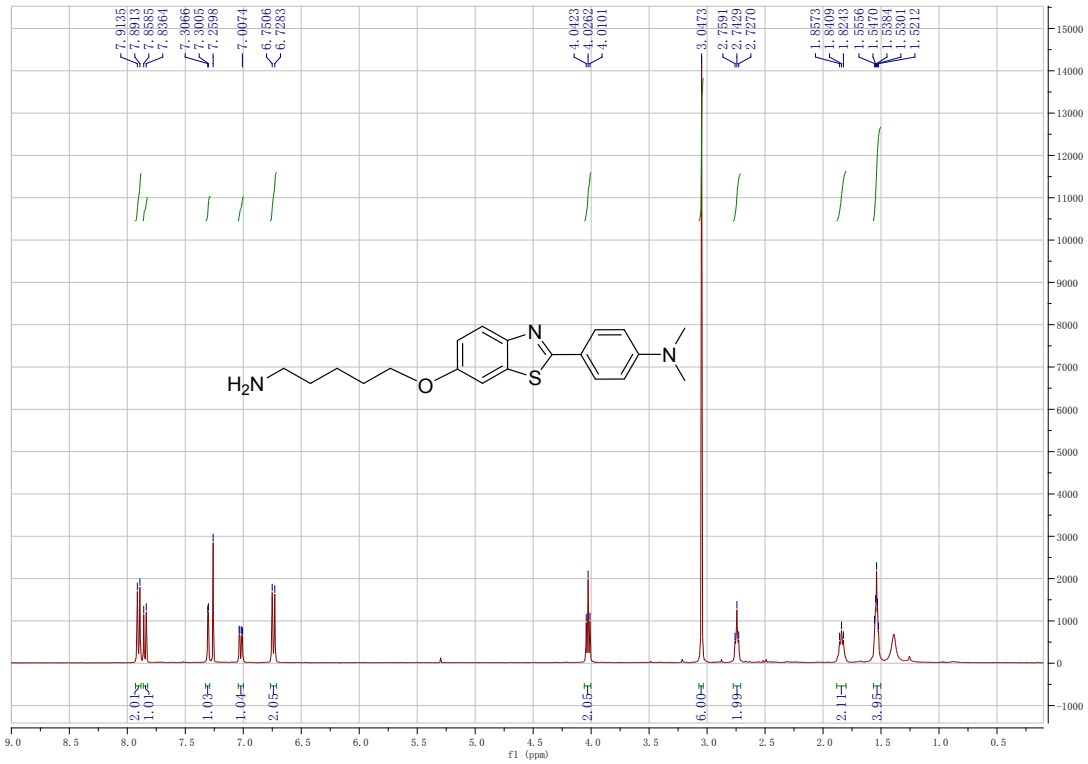
^{1}H-NMR for compound **14**



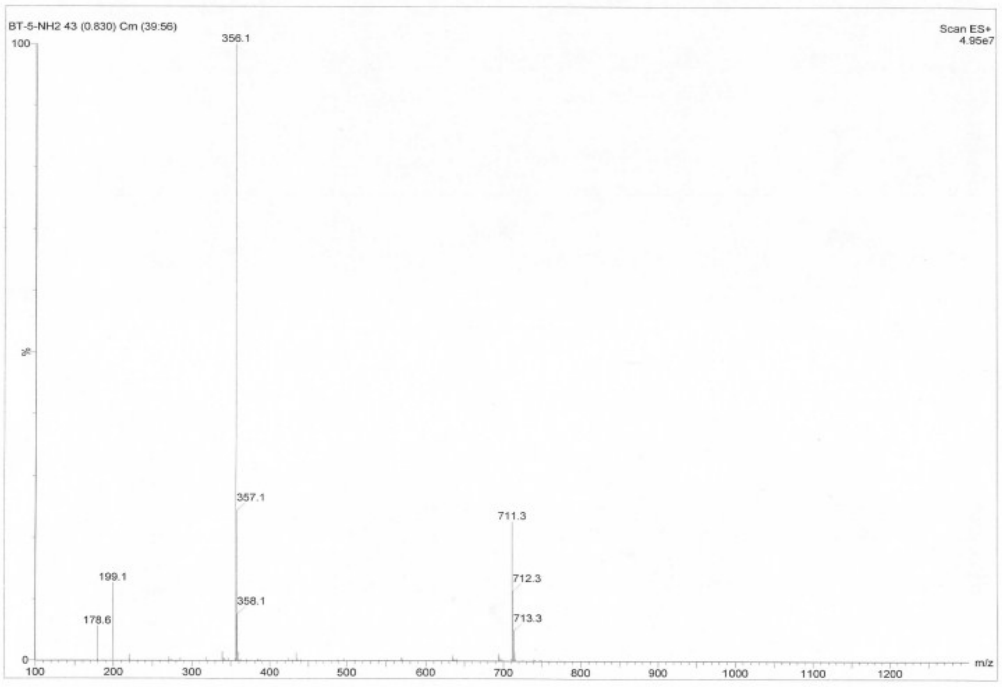
MS for compound **14**



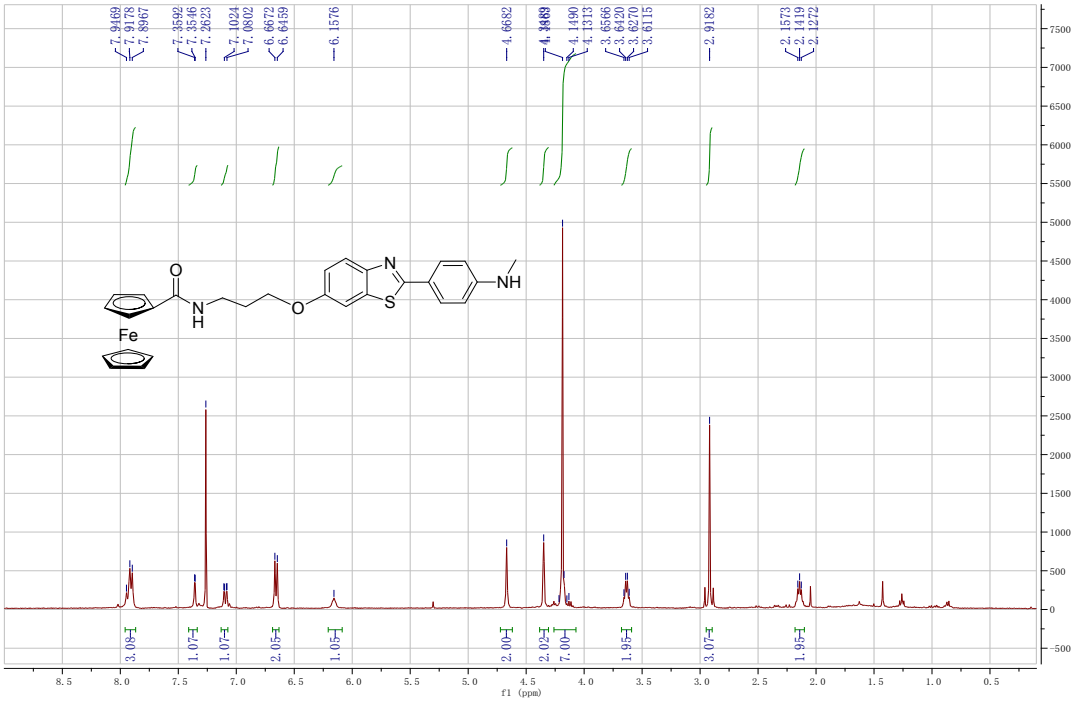
^{1}H-NMR for compound **15**



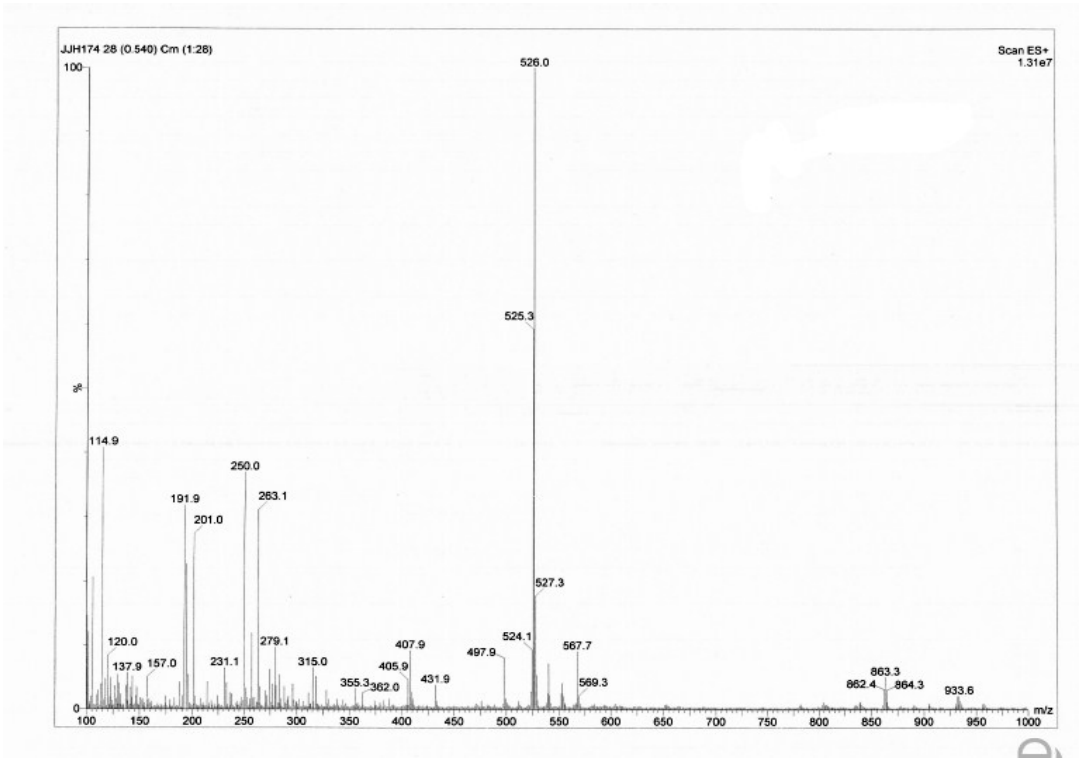
MS for compound **15**



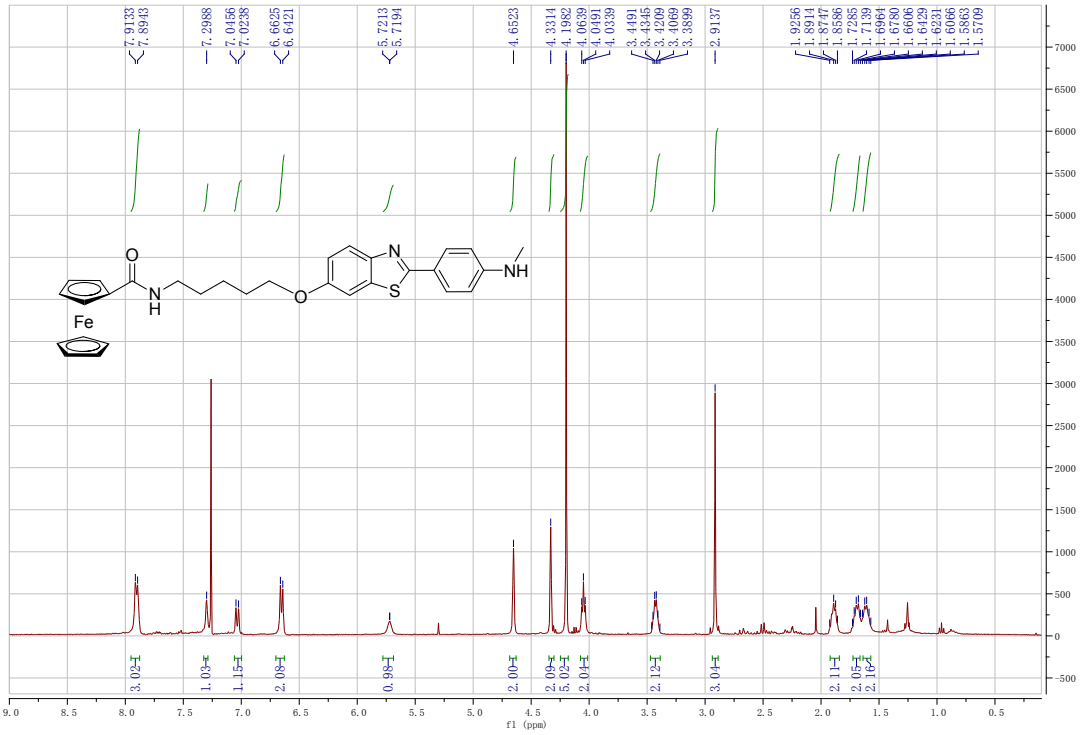
[1]H-NMR for compound **16**



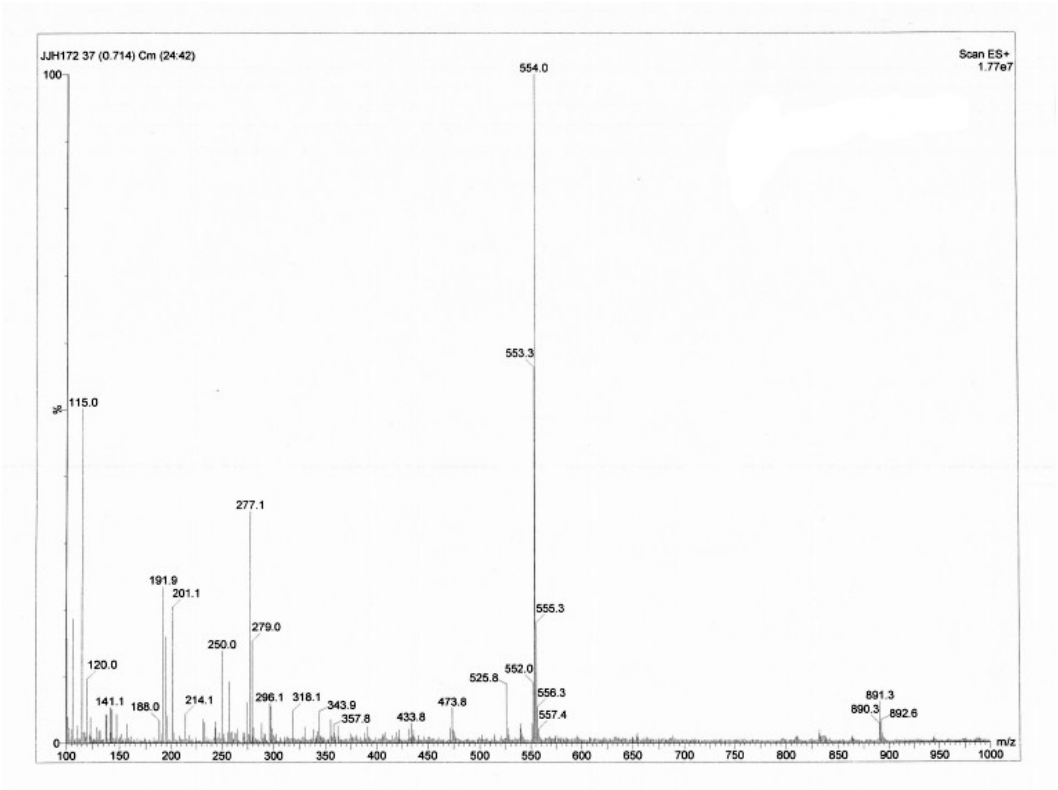
HRMS for compound **16**



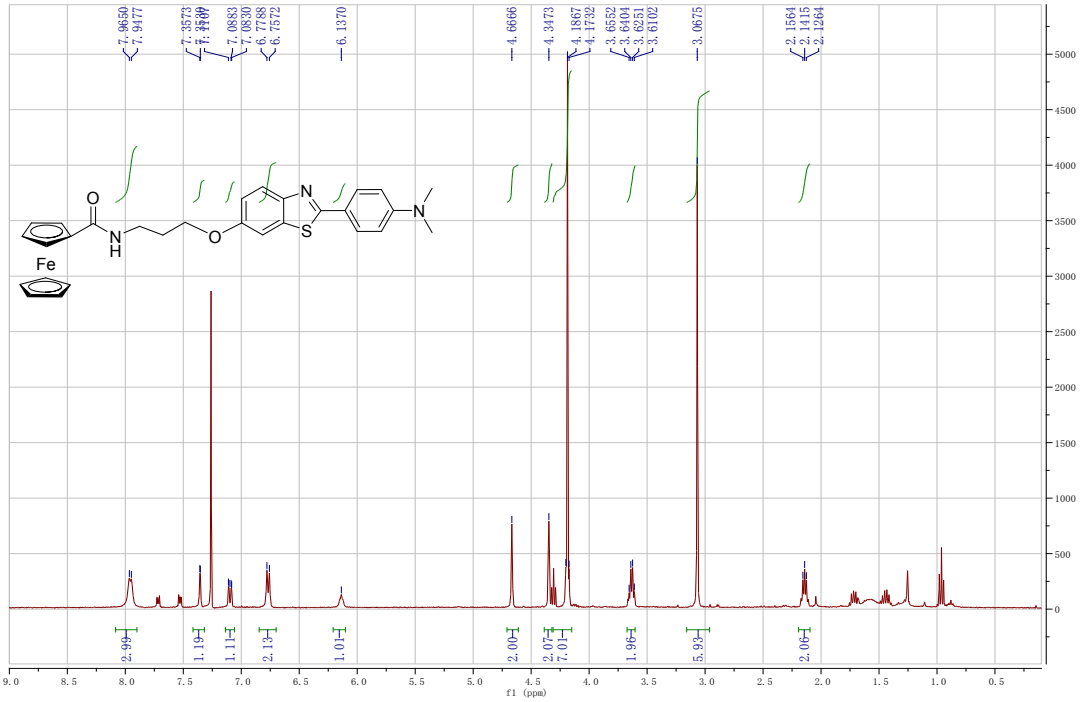
^{1}H-NMR for compound **17**



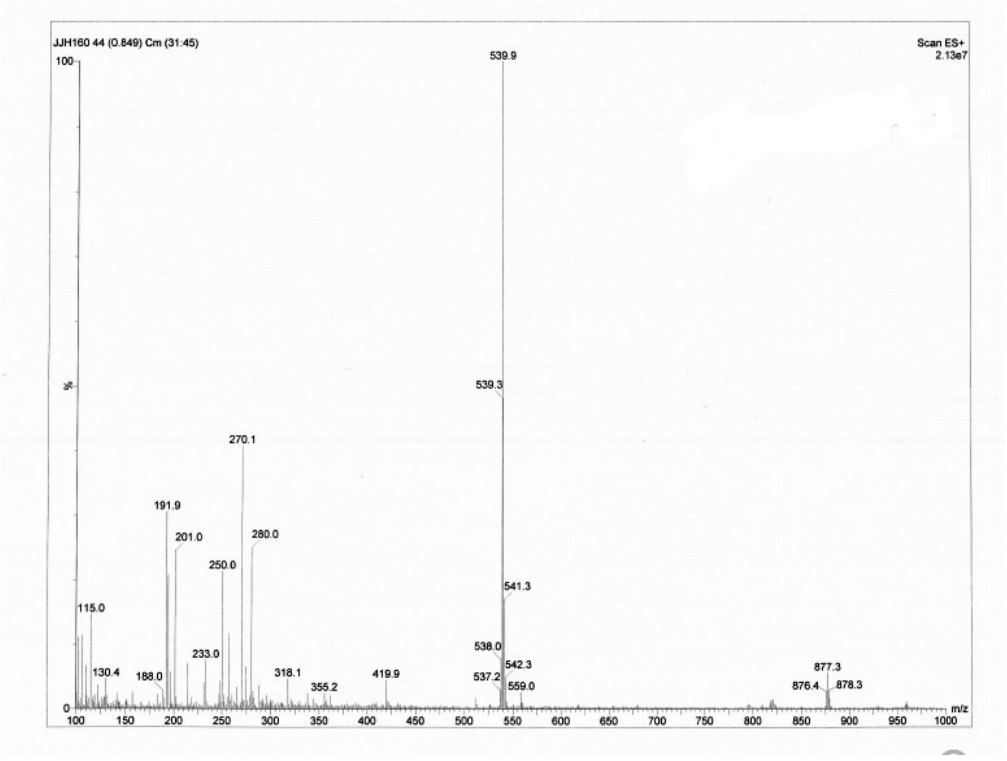
HRMS for compound **17**



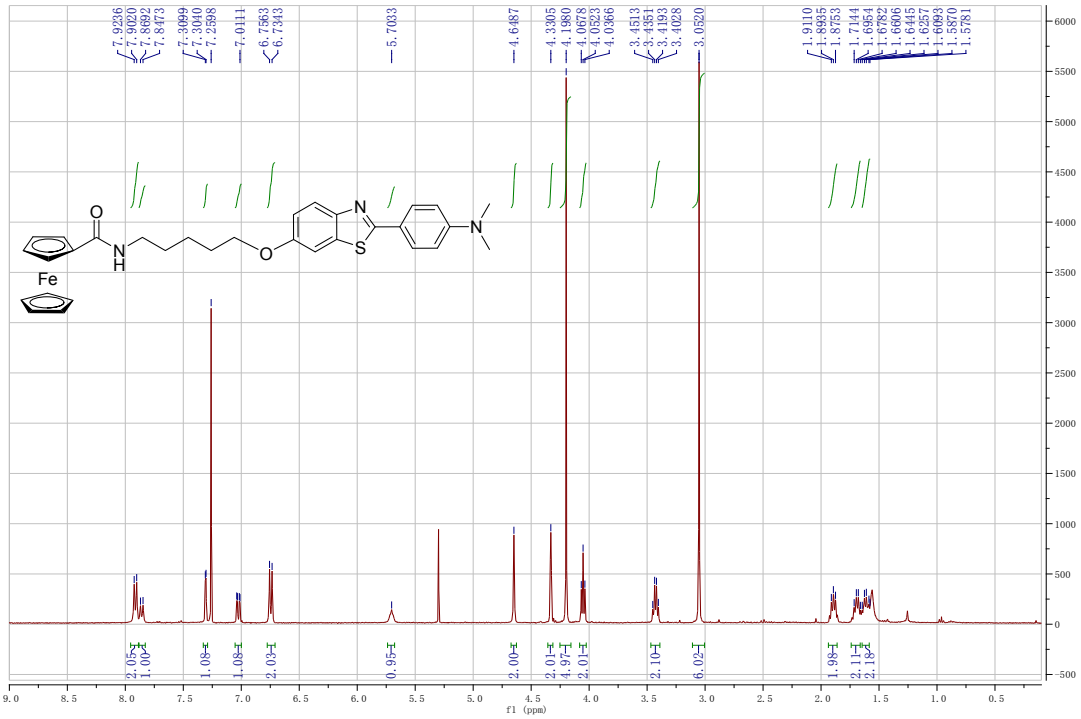
^{1}H-NMR for compound **18**



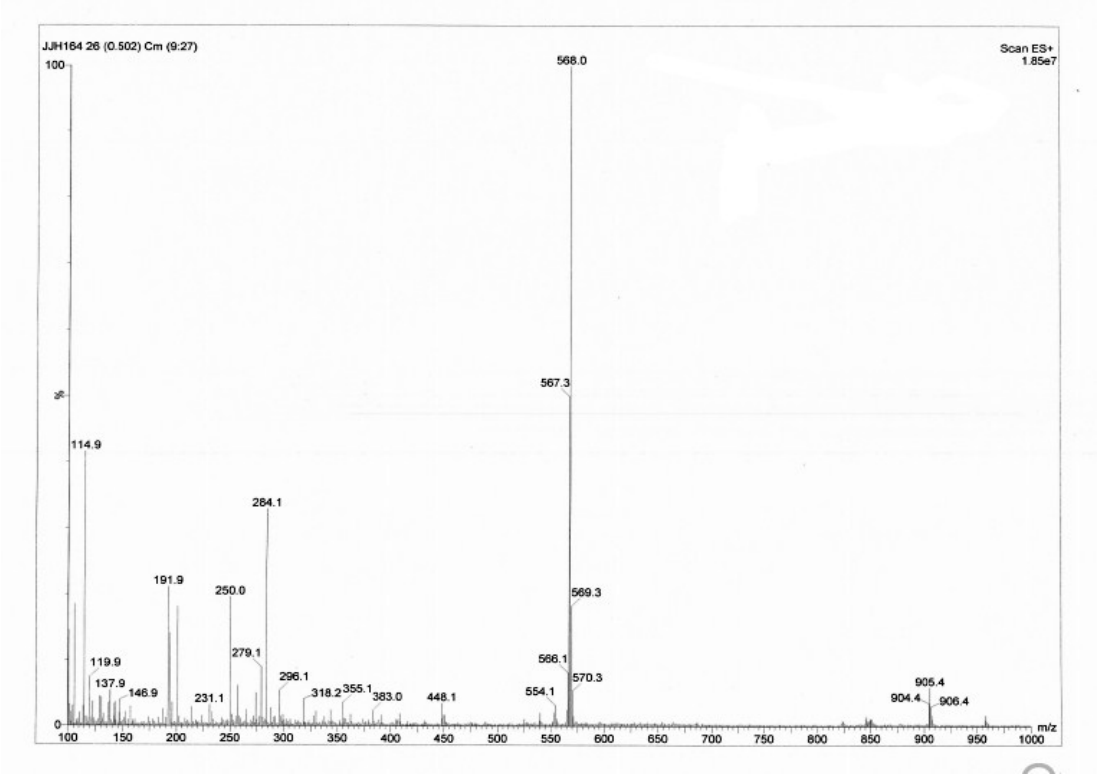
MS for compound **18**



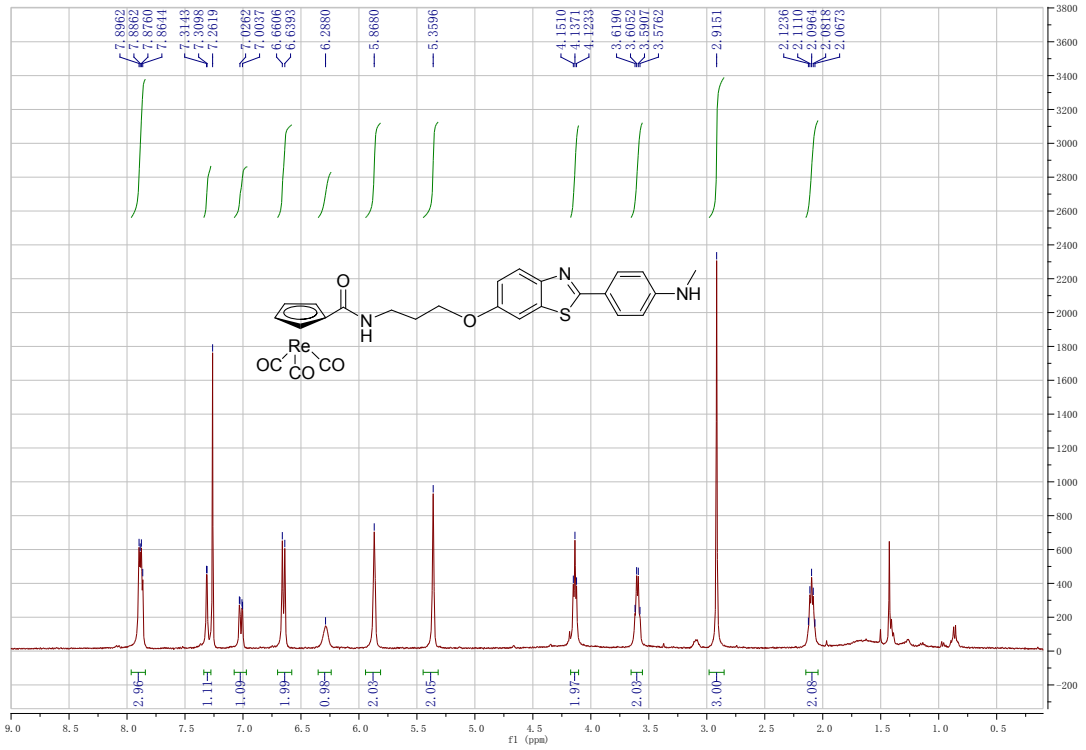
^{1}H-NMR for compound **19**



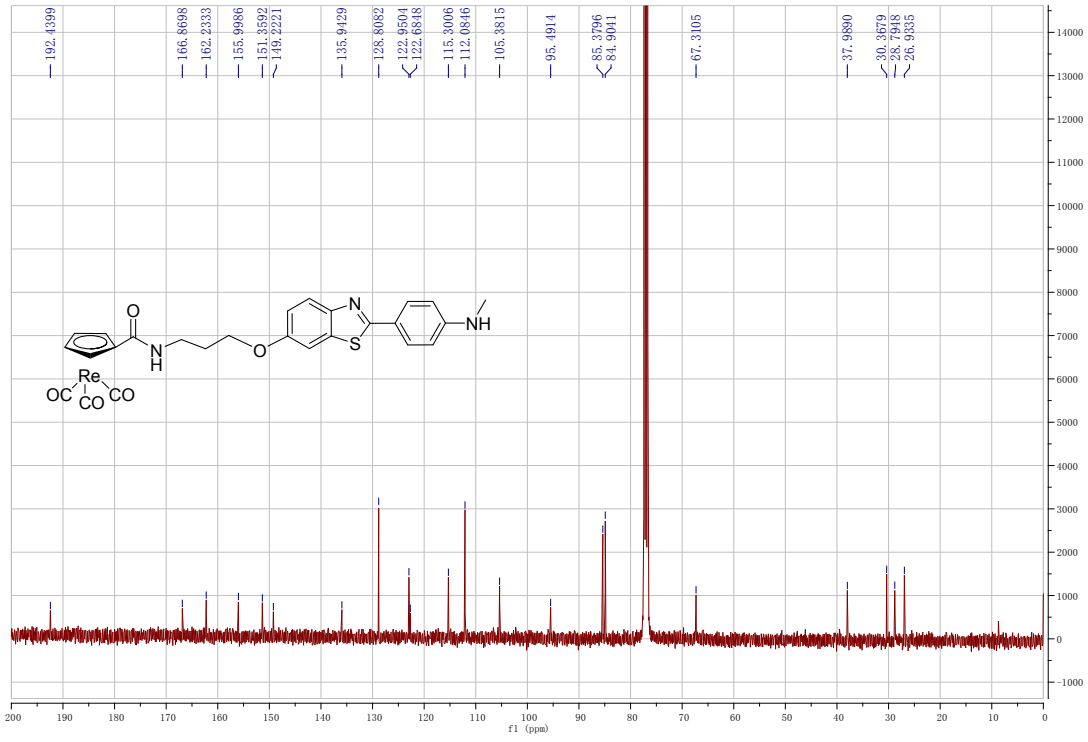
MS for compound **19**



^{1}H-NMR for compound **20**



^{13}C-NMR for compound **20**



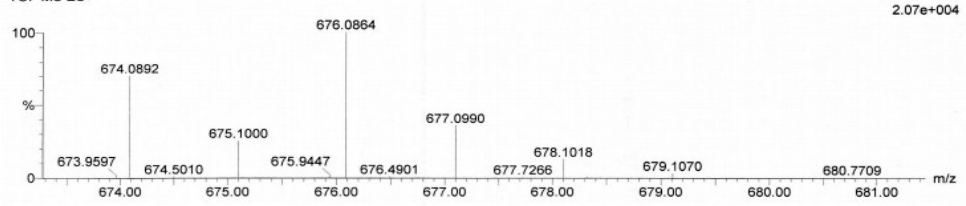
HRMS for compound **20**

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 3.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

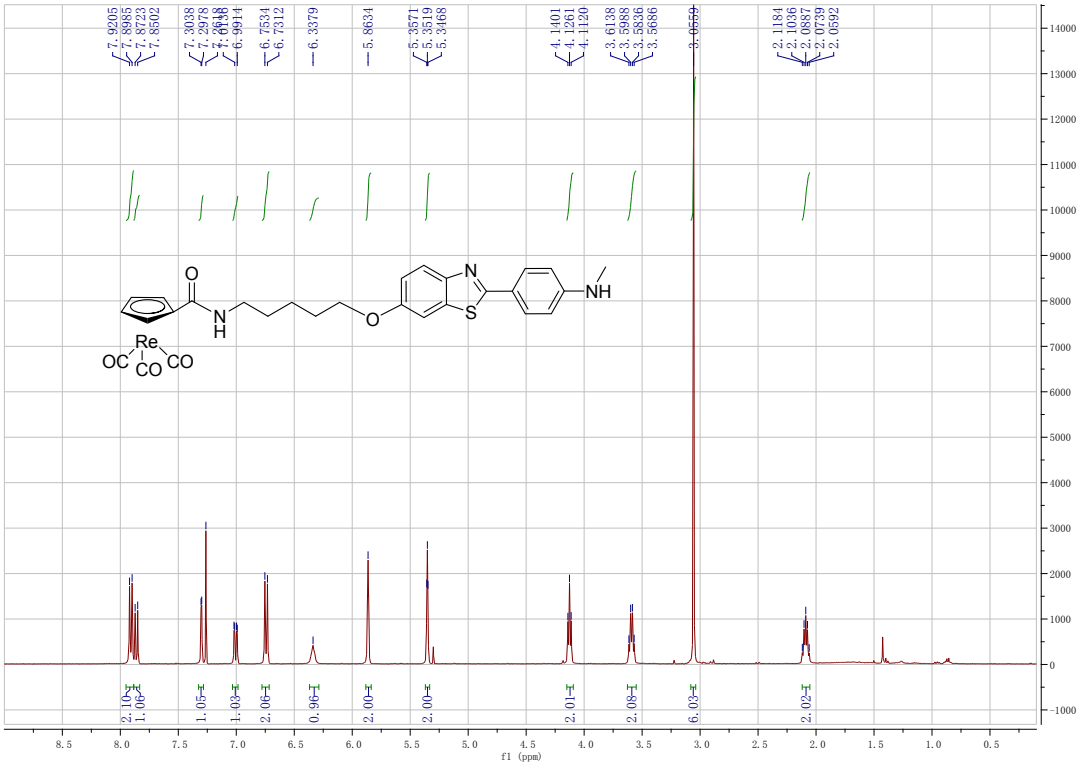
Monoisotopic Mass, Odd Electron Ions
3731 formula(e) evaluated with 12 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 0-45 H: 0-70 N: 0-10 O: 0-10 S: 0-2 185Re: 0-1

JJH 179 6 (0.111)
TOF MS ES+ 2.07e+004

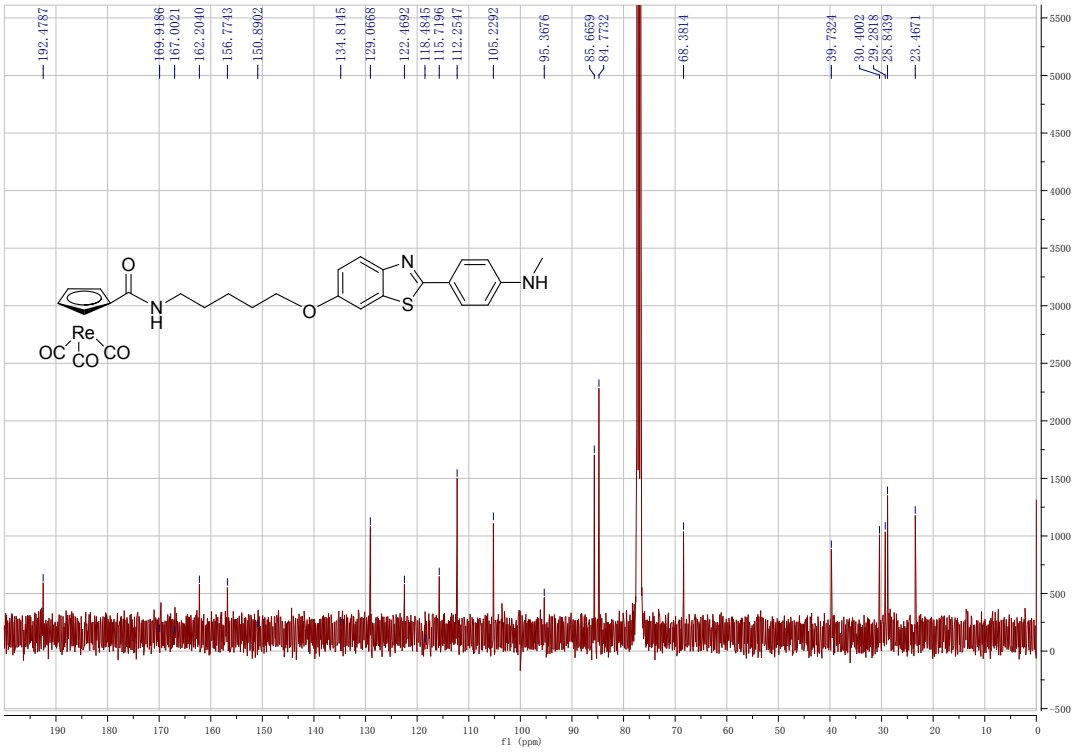


Minimum:				-1.5		
Maximum:		5.0	3.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
674.0892	674.0894	-0.2	-0.3	29.0	4.9	C30 H14 N10 O10
	674.0890	0.2	0.3	23.0	6.0	C30 H22 N6 O9 S2
	674.0895	-0.3	-0.4	13.0	160.0	C19 H23 N9 O3 S2 185Re
	674.0895	-0.3	-0.4	26.0	9.8	C34 H19 N O3 185Re
	674.0888	0.4	0.6	17.0	39.0	C26 H23 N3 O5 S 185Re ✓
	674.0896	-0.4	-0.6	32.0	102.4	C38 H18 N4 O7 S
	674.0886	0.6	0.9	14.0	262.7	C18 H19 N9 O8 185Re
	674.0882	1.0	1.5	8.0	254.5	C18 H27 N5 O7 S2 185Re
	674.0902	-1.0	-1.5	22.0	11.0	C27 H19 N7 O S 185Re
	674.0903	-1.1	-1.6	28.0	23.8	C31 H18 N10 O5 S2
	674.0876	1.6	2.4	42.0	226.1	C42 H10 N8 O3
	674.0910	-1.8	-2.7	37.0	157.2	C39 H14 N8 O3 S

^{1}H-NMR for compound **21**



^{13}C-NMR for compound **21**



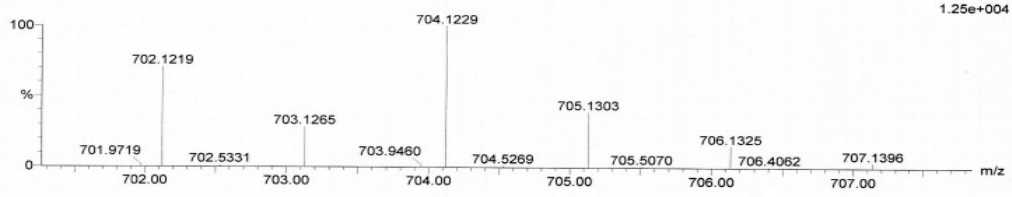
HRMS for compound **21**

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 3.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

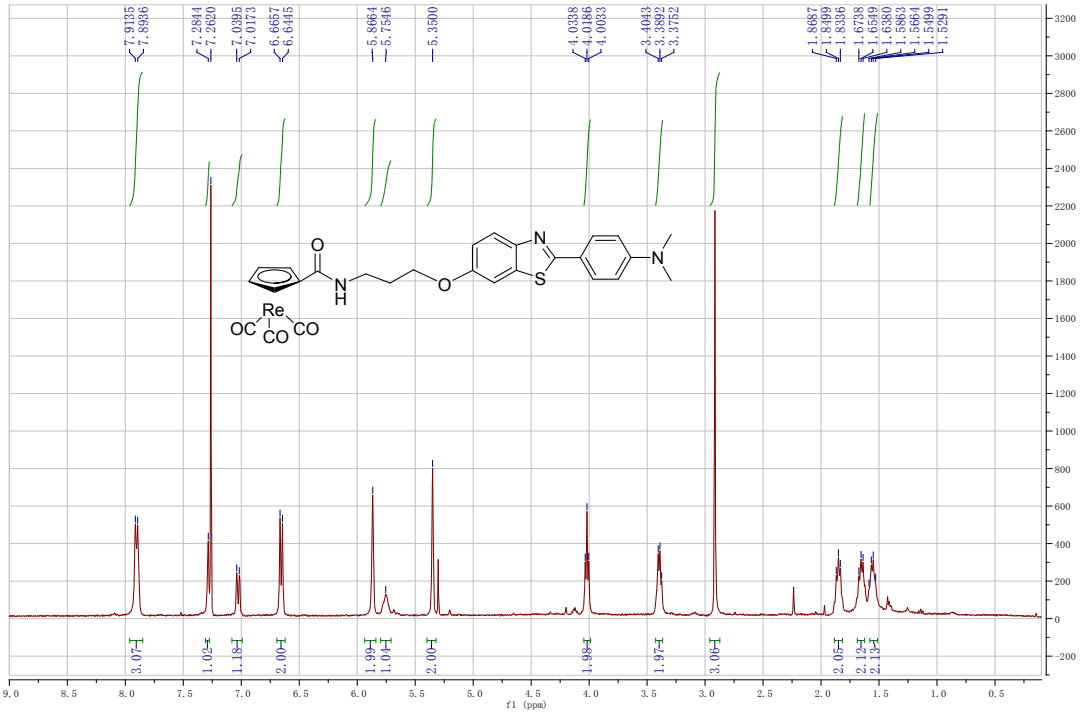
Monoisotopic Mass, Odd Electron Ions
3646 formula(e) evaluated with 14 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 0-45 H: 0-70 N: 0-10 O: 0-10 S: 0-2 185Re: 0-1

JJH 177 32 (0.592)
TOF MS ES+

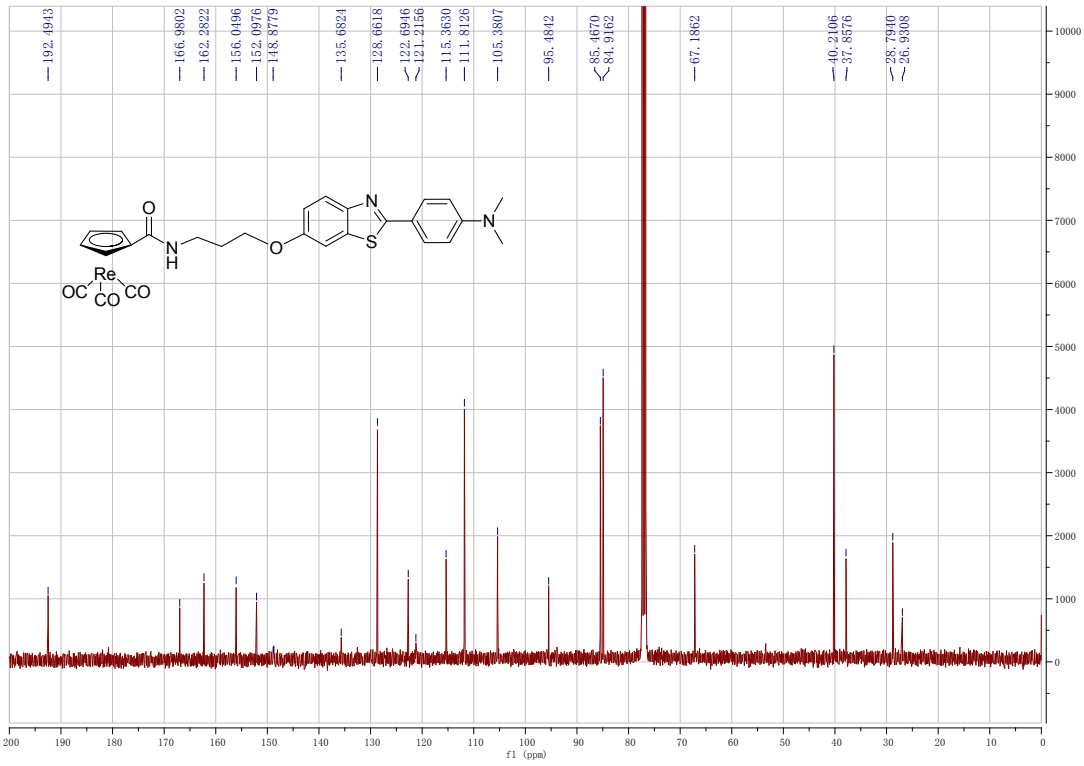


Minimum:				-1.5		
Maximum:		5.0	3.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
702.1219	702.1216	0.3	0.4	28.0	5.8	C33 H22 N10 O5 S2
	702.1215	0.4	0.6	22.0	11.9	C29 H23 N7 O S 185Re
	702.1223	-0.4	-0.6	37.0	67.8	C41 H18 N8 O3 S
	702.1226	-0.7	-1.0	13.0	114.7	C24 H27 N3 O10 185Re
	702.1209	1.0	1.4	32.0	41.1	C40 H22 N4 O7 S
	702.1208	1.1	1.6	26.0	1.1	C36 H23 N O3 185Re
	702.1208	1.1	1.6	13.0	110.9	C21 H27 N9 O3 S2 185Re
	702.1207	1.2	1.7	29.0	0.2	C32 H18 N10 O10
	702.1233	-1.4	-2.0	9.0	249.9	C17 H27 N9 O8 S 185Re
	702.1203	1.6	2.3	23.0	0.4	C32 H26 N6 O9 S2
	702.1235	-1.6	-2.3	12.0	66.2	C25 H31 N3 O5 S2 185Re
	702.1201	1.8	2.6	17.0	32.7	C28 H27 N3 O5 S 185Re ✓
	702.1199	2.0	2.8	14.0	174.0	C20 H23 N9 O8 185Re
	702.1240	-2.1	-3.0	18.0	69.3	C25 H23 N7 O6 185Re

^{1}H-NMR for compound **22**



^{13}C-NMR for compound **22**



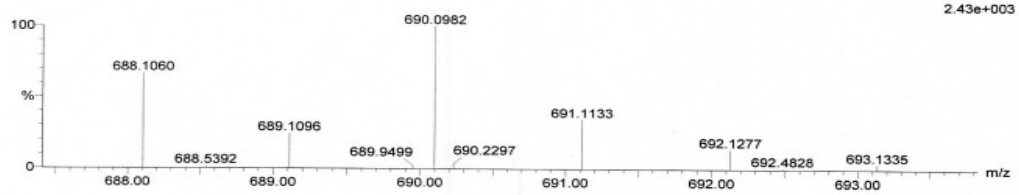
HRMS for compound **22**

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 3.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

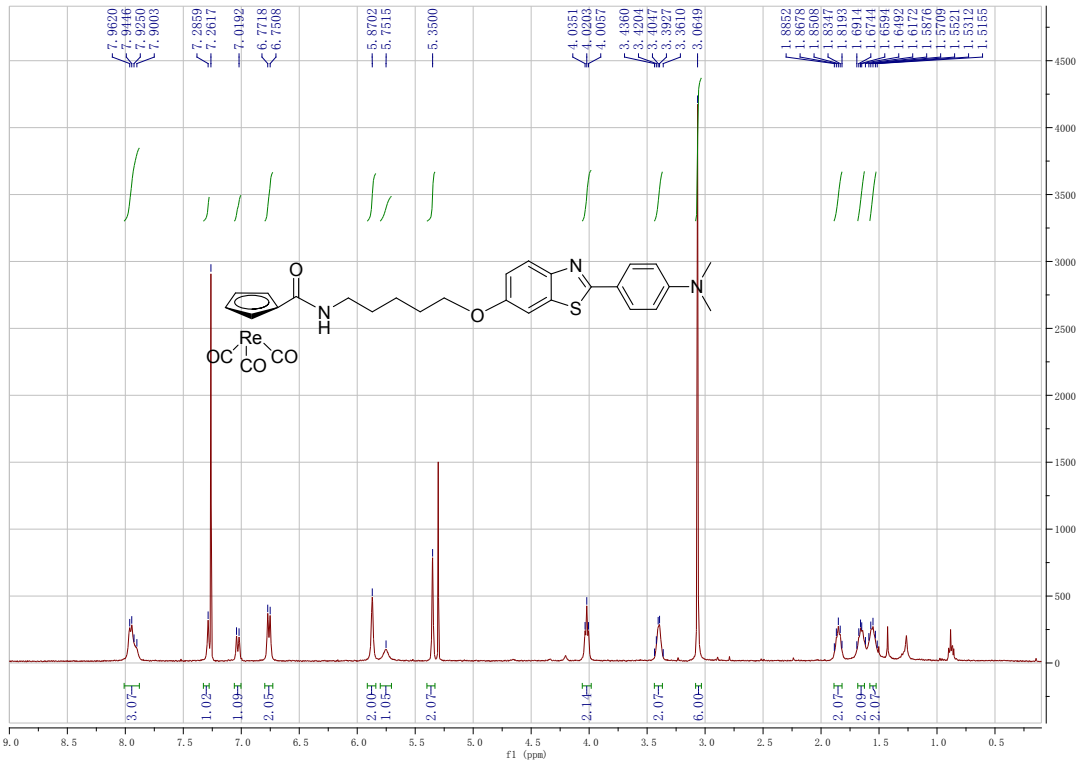
Monoisotopic Mass, Odd Electron Ions
3701 formula(e) evaluated with 13 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 0-45 H: 0-70 N: 0-10 O: 0-10 S: 0-2 185Re: 0-1

JJH 176 8 (0.148)
TOF MS ES+ 2.43e+003

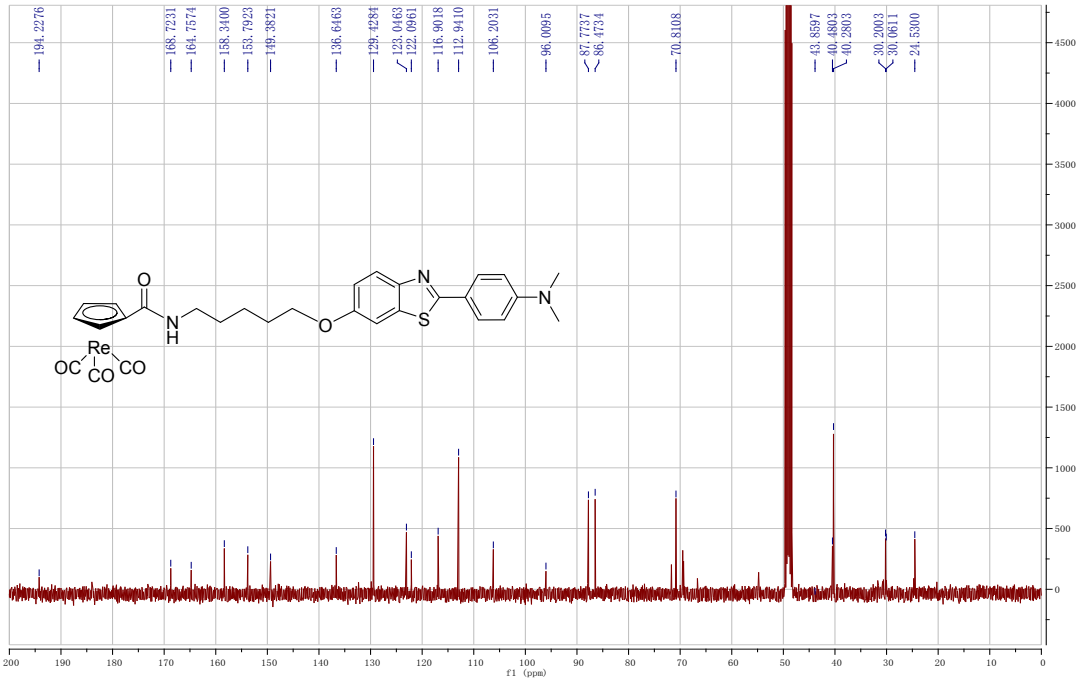


Minimum:				-1.5		
Maximum:		5.0	3.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
688.1060	688.1060	0.0	0.0	28.0	3.8	C32 H20 N10 O5 S2
	688.1058	0.2	0.3	22.0	0.4	C28 H21 N7 O S 185Re
	688.1066	-0.6	-0.9	37.0	20.3	C40 H16 N8 O3 S
	688.1053	0.7	1.0	32.0	13.8	C39 H20 N4 O7 S
	688.1052	0.8	1.2	13.0	13.9	C20 H25 N9 O3 S2 185Re
	688.1051	0.9	1.3	26.0	1.8	C35 H21 N O3 185Re
	688.1051	0.9	1.3	29.0	1.0	C31 H16 N10 O10
	688.1070	-1.0	-1.5	13.0	14.5	C23 H25 N3 O10 185Re
	688.1046	1.4	2.0	23.0	1.1	C31 H24 N6 O9 S2
	688.1045	1.5	2.2	17.0	2.5	C27 H25 N3 O5 S 185Re ✓
	688.1043	1.7	2.5	14.0	24.0	C19 H21 N9 O8 185Re
	688.1077	-1.7	-2.5	9.0	36.8	C16 H25 N9 O8 S 185Re
	688.1078	-1.8	-2.6	12.0	7.1	C24 H29 N3 O5 S2 185Re

^{1}H-NMR for compound **23**



^{13}C-NMR for compound **23**



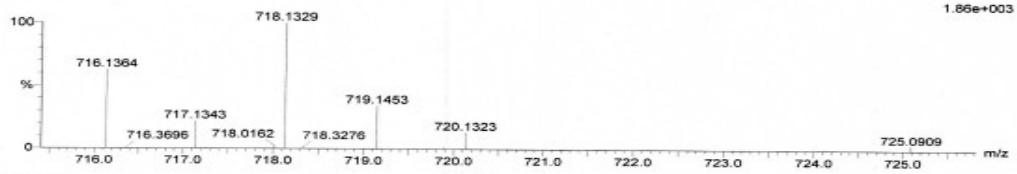
HRMS for compound **23**

Elemental Composition Report Page 1

Single Mass Analysis
Tolerance = 3.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Odd and Even Electron Ions
3585 formula(e) evaluated with 27 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 0-45 H: 0-70 N: 0-10 O: 0-10 S: 0-2 185Re: 0-1

JJH 173 23 (0.426)
TOF MS ES+ 1.86e+003



Minimum:				-1.5		
Maximum:		5.0	3.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
716.1364	716.1364	0.0	0.0	29.0	3.9	C33 H20 N10 O10
	716.1364	0.0	0.0	26.0	5.2	C37 H25 N O3 185Re
	716.1365	-0.1	-0.1	7.5	5.6	C23 H35 N2 O8 S2 185Re
	716.1365	-0.1	-0.1	13.0	3.7	C22 H29 N9 O3 S2 185Re
	716.1366	-0.2	-0.3	32.0	17.7	C41 H24 N4 O7 S
	716.1359	0.5	0.7	23.0	4.2	C33 H28 N6 O9 S2
	716.1369	-0.5	-0.7	13.5	6.0	C23 H27 N6 O9 185Re
	716.1358	0.6	0.8	17.0	0.1	C29 H29 N3 O5 S 185Re
	716.1358	0.6	0.8	22.5	0.1	C28 H23 N10 S 185Re
	716.1371	-0.7	-1.0	22.0	0.5	C30 H25 N7 O S 185Re
	716.1371	-0.7	-1.0	16.5	0.2	C31 H31 O6 S 185Re
	716.1356	0.8	1.1	14.0	8.4	C21 H25 N9 O8 185Re
	716.1373	-0.9	-1.3	22.5	5.9	C35 H30 N3 O10 S2
	716.1373	-0.9	-1.3	28.0	7.7	C34 H24 N10 O5 S2
	716.1352	1.2	1.7	32.5	14.9	C39 H22 N7 O6 S
	716.1351	1.3	1.8	8.0	8.0	C21 H33 N5 O7 S2 185Re
	716.1351	1.3	1.8	26.5	3.6	C35 H23 N4 O2 185Re
	716.1378	-1.4	-2.0	12.5	2.3	C24 H31 N6 O4 S2 185Re
	716.1379	-1.5	-2.1	31.5	20.6	C43 H26 N O8 S
	716.1379	-1.5	-2.1	37.0	23.6	C42 H20 N8 O3 S
	716.1346	1.8	2.5	23.5	2.8	C31 H26 N9 O8 S2
	716.1346	1.8	2.5	20.5	3.9	C35 H31 O S2 185Re
	716.1383	-1.9	-2.7	18.5	2.5	C24 H23 N10 O5 185Re
	716.1345	1.9	2.7	42.0	30.2	C45 H16 N8 O3
	716.1383	-1.9	-2.7	13.0	4.0	C25 H29 N3 O10 185Re
	716.1344	2.0	2.8	17.5	0.4	C27 H27 N6 O4 S 185Re
	716.1385	-2.1	-2.9	21.5	1.3	C32 H27 N4 O2 S 185Re