

Fabrication of magnetically separable ruthenium nanoparticles decorated on channelled silica microspheres: Efficient catalysts for chemoselective hydrogenation of nitroarenes

Gauravjyoti D. Kalita^a, Manash Das,^b Pankaj Das^{a}*

^aDepartment of Chemistry, Dibrugarh University, Dibrugarh, Assam, India, 786004.

^bMaterials Science Division, CSIR-North East Institute of Science and Technology, Jorhat-785006, India

Supporting Information

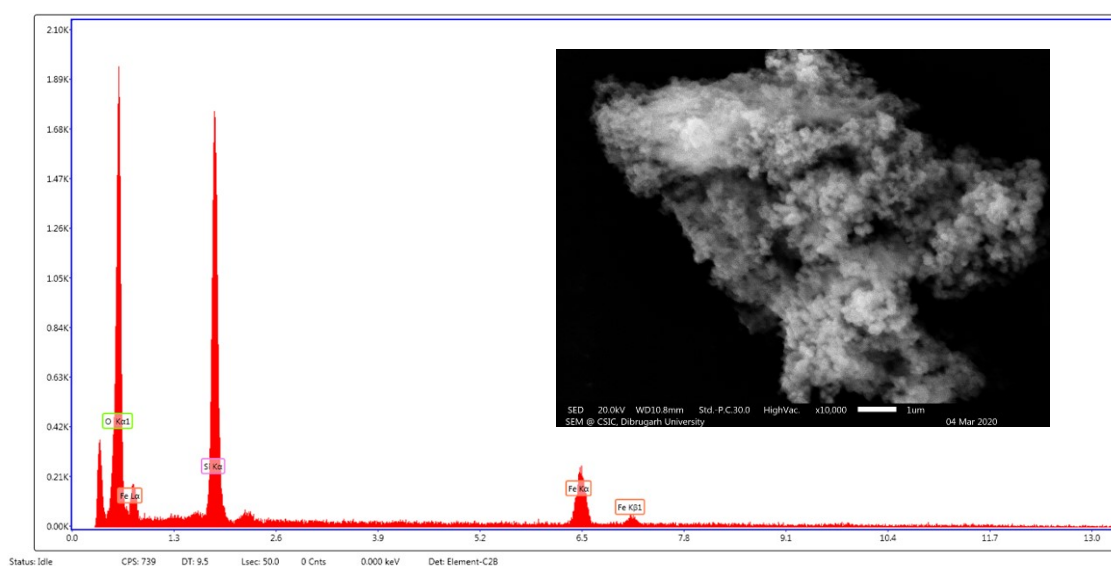


Fig. S1 EDX spectra and corresponding SEM micrograph (inset) of Fe₃O₄/SiO₂

eZAF Smart Quant Results

Element	O K	Si K	Fe K
Weight %	33.98	31.23	24.79
Atomic %	55.04	28.82	16.14
Net Int.	113.46	181.51	65.01
Error %	8.54	5.17	4.95
K ratio	0.1566	0.2161	0.3104
Z	1.1102	1.0151	0.8596
R	0.9375	0.9901	1.0534
A	0.4152	0.6781	1.0012
F	1.0000	1.0051	1.0369

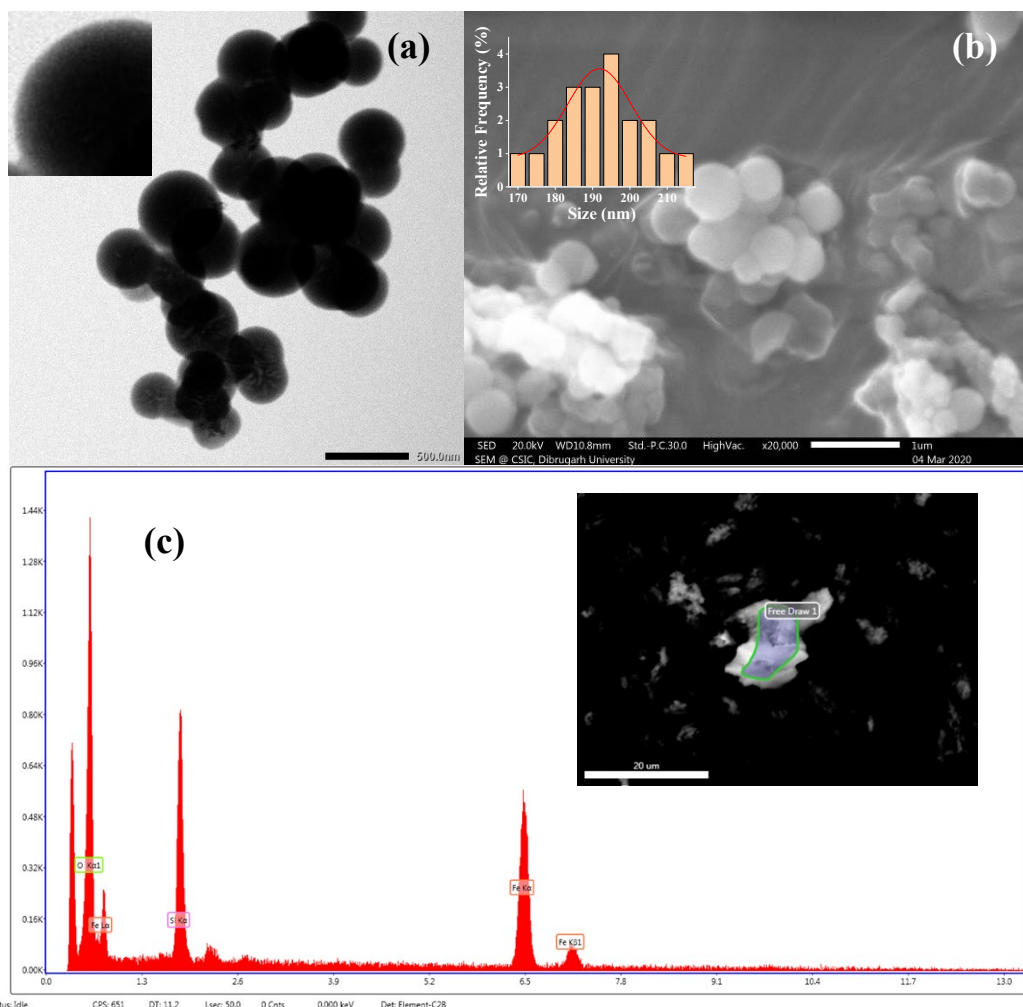


Fig. S2 (a) TEM-HAADF micrograph with magnified cross-section (inset) **(b)** SEM image with corresponding particles size distribution (inset) and **(c)** EDX spectra of Fe₃O₄-CSM

eZAF Smart Quant Results

Element	O K	Si K	Fe K
Weight %	16.76	14.13	29.11
Atomic %	37.57	18.05	24.38
Net Int.	72.97	81.13	124.45
Error %	8.52	7.02	3.10
K ratio	0.0901	0.0864	0.6595
Z	1.1874	1.0893	0.9279
R	0.8978	0.9542	1.0278
A	0.4526	0.5569	1.0008
F	1.0000	1.0074	1.0276

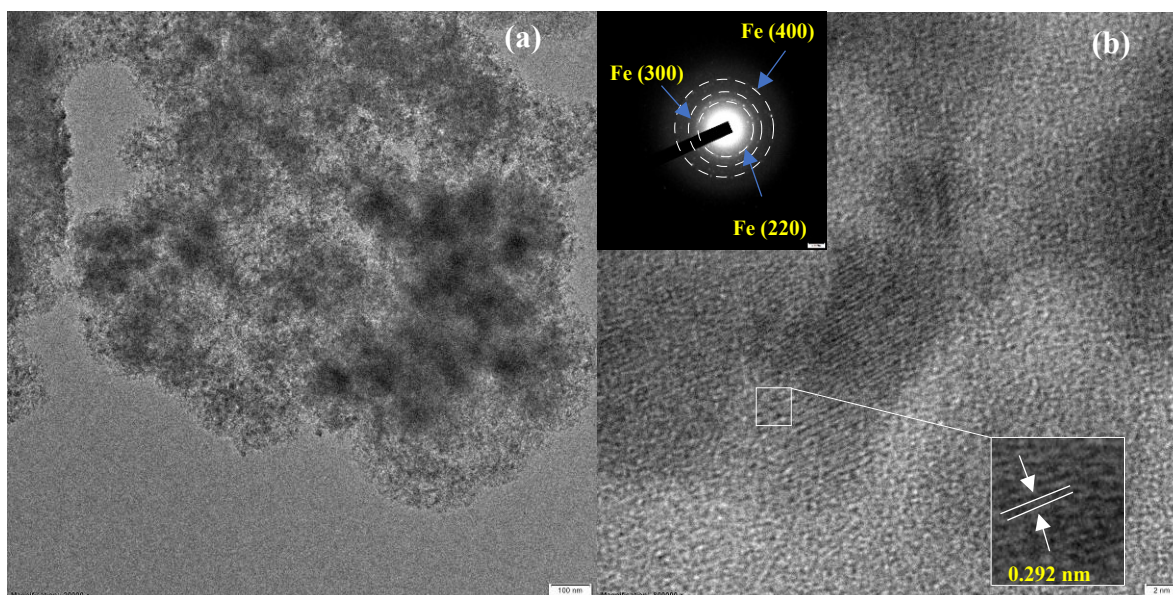


Fig. S3 (a) TEM micrograph and (b) HRTEM micrograph of Ru@Fe₃O₄/SiO₂ with corresponding SAED pattern (inset) and magnified lattice cross-section (inset)

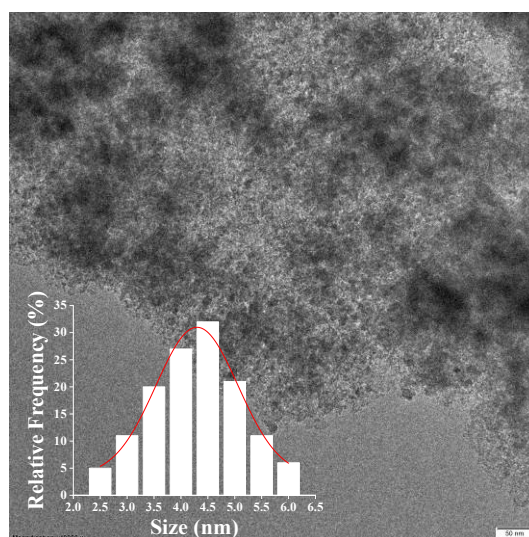


Fig. S4 TEM micrograph of Ru@Fe₃O₄/SiO₂ depicting ultrasmall Ru nanoparticles supported onto Fe₃O₄/SiO₂ with corresponding size distribution histogram (inset)

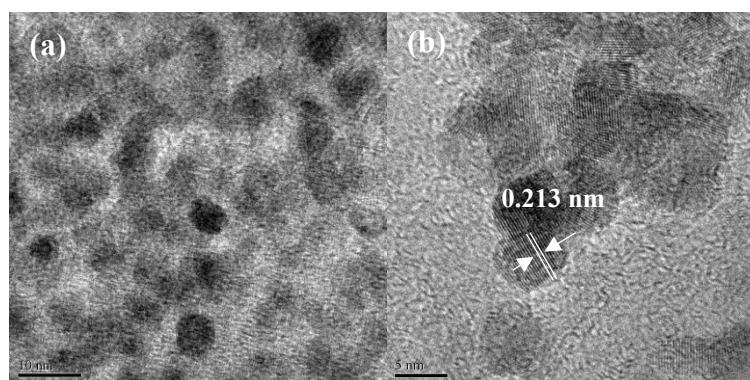


Fig. S5 (a) TEM micrograph of Ru@Fe₃O₄/SiO₂ depicting ultrasmall Ru nanoparticles and **(b)** HRTEM image of a lattice cross-section with corresponding interplanar fringe spacing (inset)

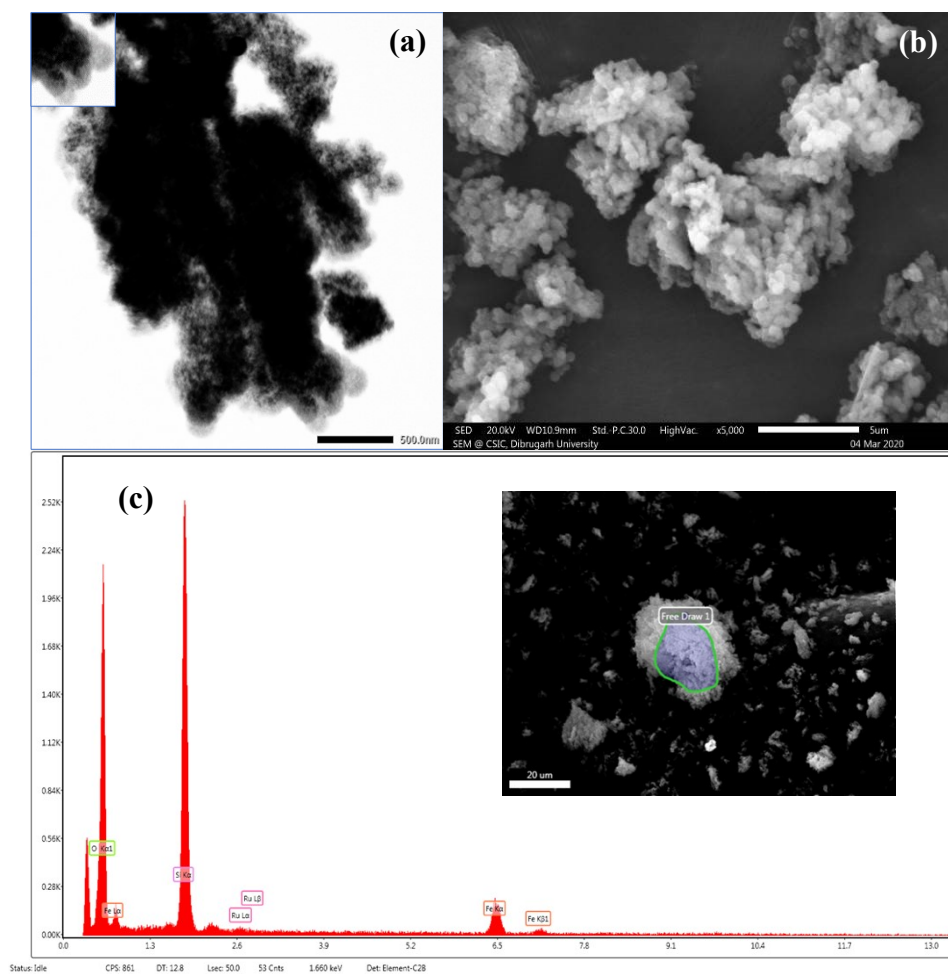


Fig. S6 (a) TEM-HAADF micrograph with magnified cross-section (inset) **(b)** SEM image and **(c)** EDX spectra of Ru@Fe₃O₄-CSM

eZAF Smart Quant Results

Element	O K	Si K	Ru L	Fe K
Weight %	37.47	37.09	0.87	24.57
Atomic %	56.97	32.12	0.21	10.70
Net Int.	136.61	269.25	2.75	54.00
Error %	8.72	4.56	28.41	5.57
K ratio	0.1583	0.2692	0.0060	0.2165
Z	1.0935	0.9987	0.7560	0.8446
R	0.9466	0.9980	1.2041	1.0588
A	0.3864	0.7233	0.8938	1.0000
F	1.0000	1.0044	1.0250	1.0434

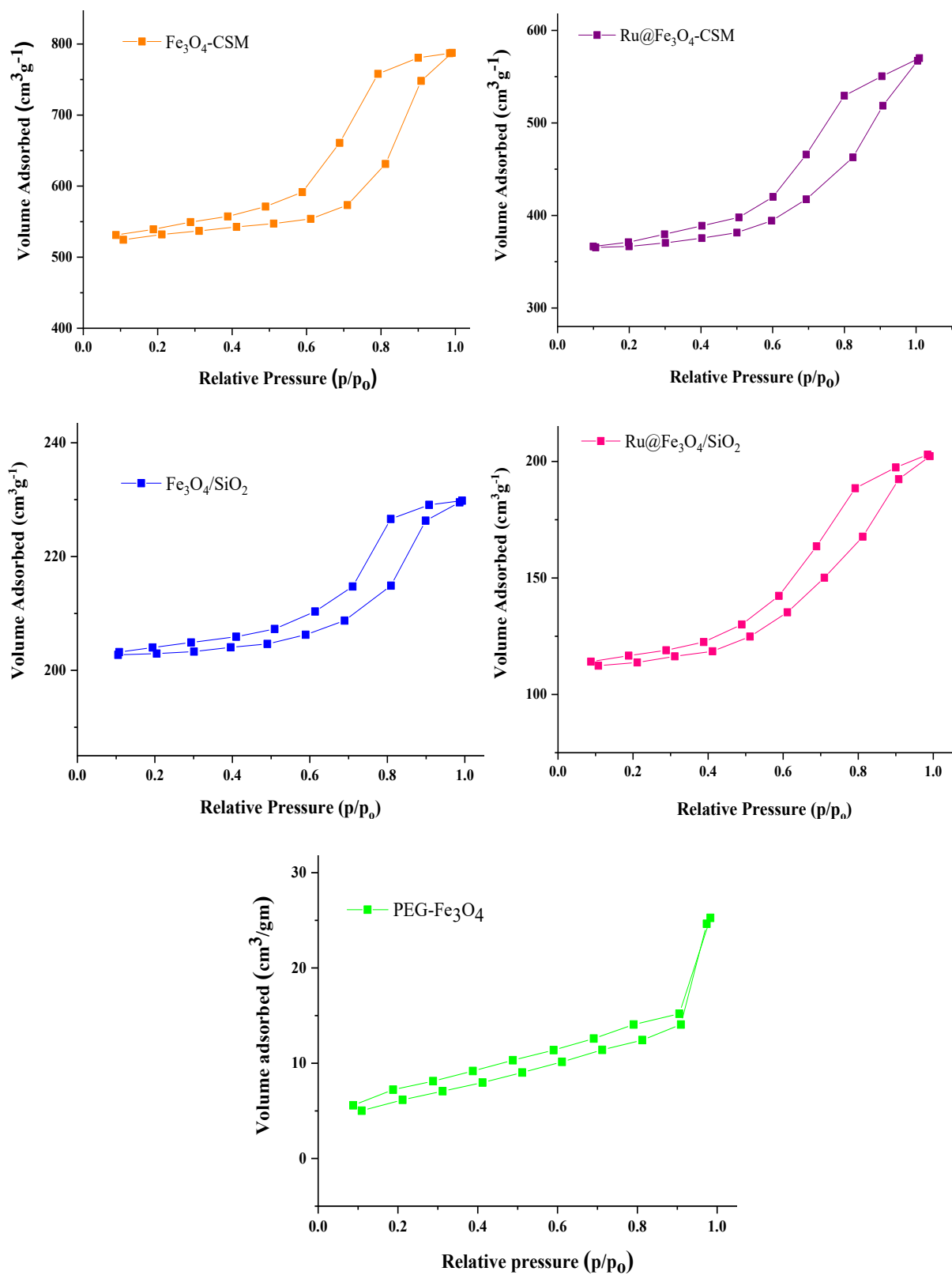


Fig. S7 N₂-sorption isotherms for the Fe₃O₄-based materials: Fe₃O₄/SiO₂, Fe₃O₄-CSM, Ru@Fe₃O₄/SiO₂ and Ru@Fe₃O₄-CSM

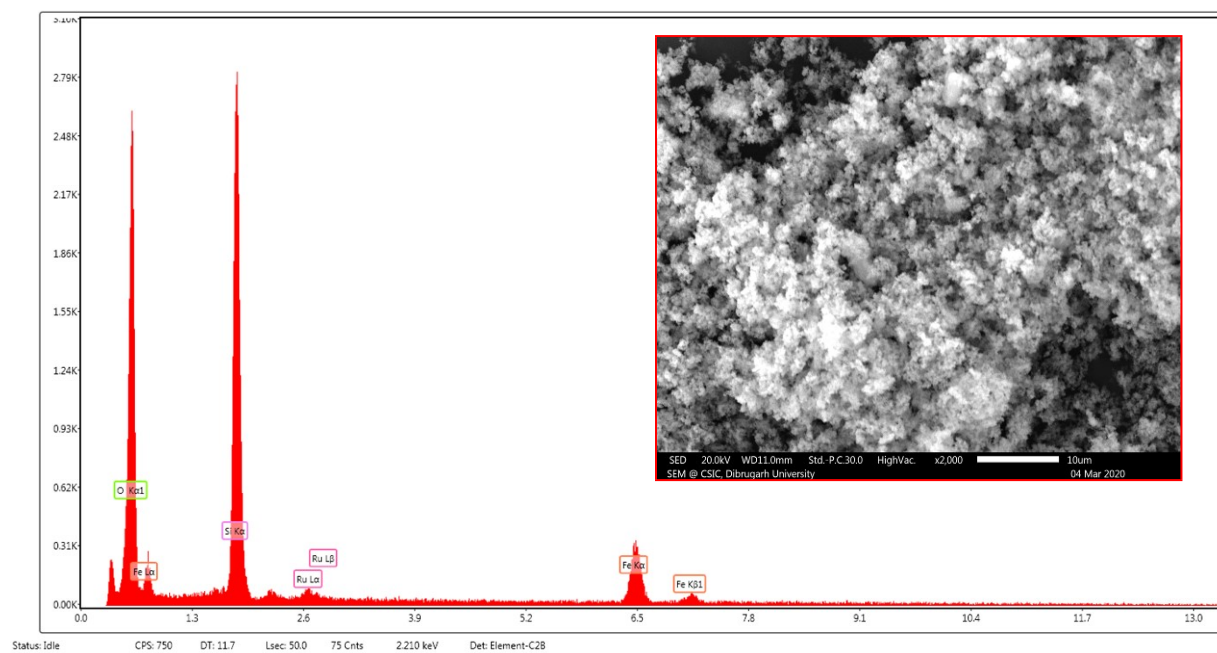


Fig. S8 EDX spectra and corresponding SEM micrograph of the catalyst, Ru@Fe₃O₄-CSM recovered after recyclability tests performed up to 5th run

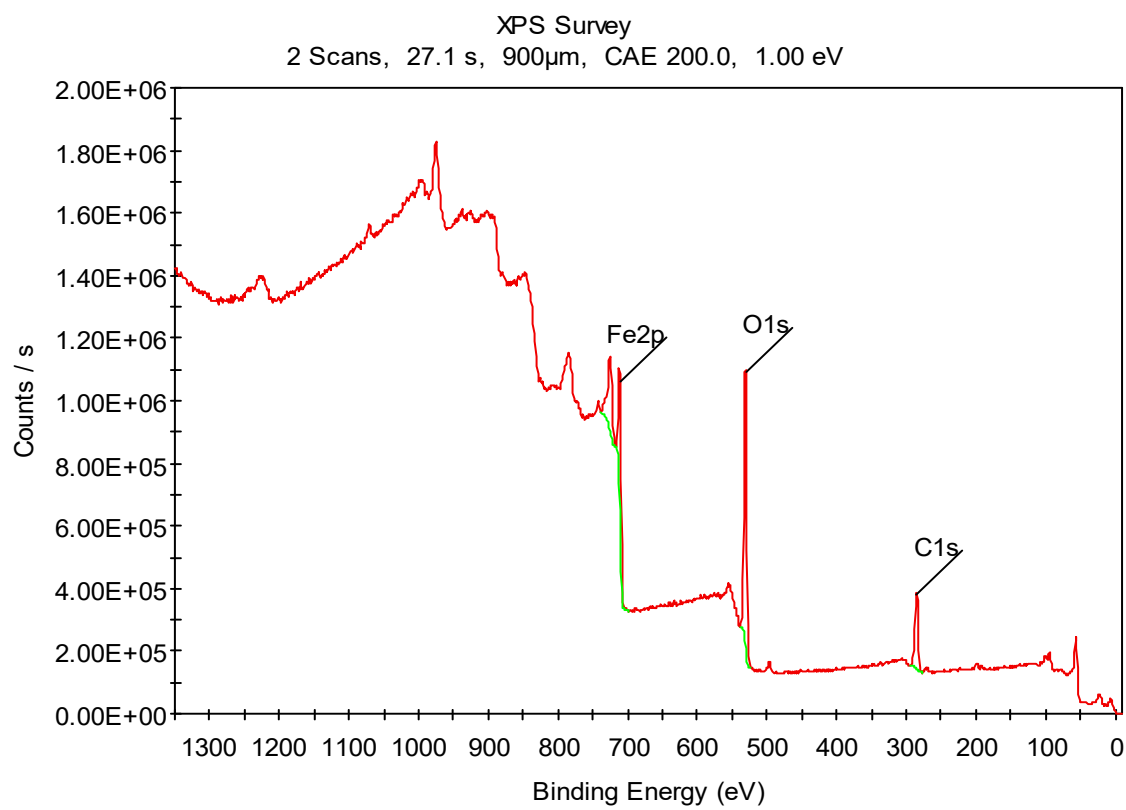


Fig. S9 XPS survey spectrum of the as prepared of $\text{Fe}_3\text{O}_4/\text{SiO}_2$

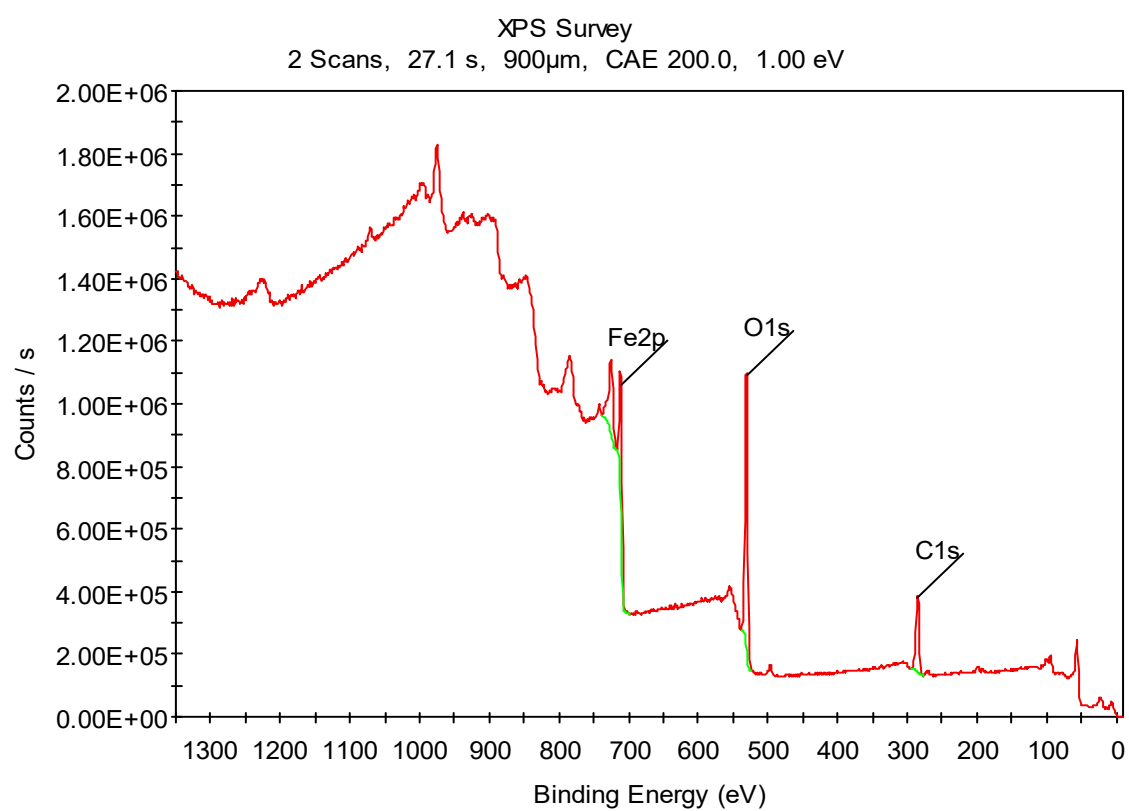


Fig. S10 XPS survey spectrum of the as prepared of Fe_3O_4 -CSM

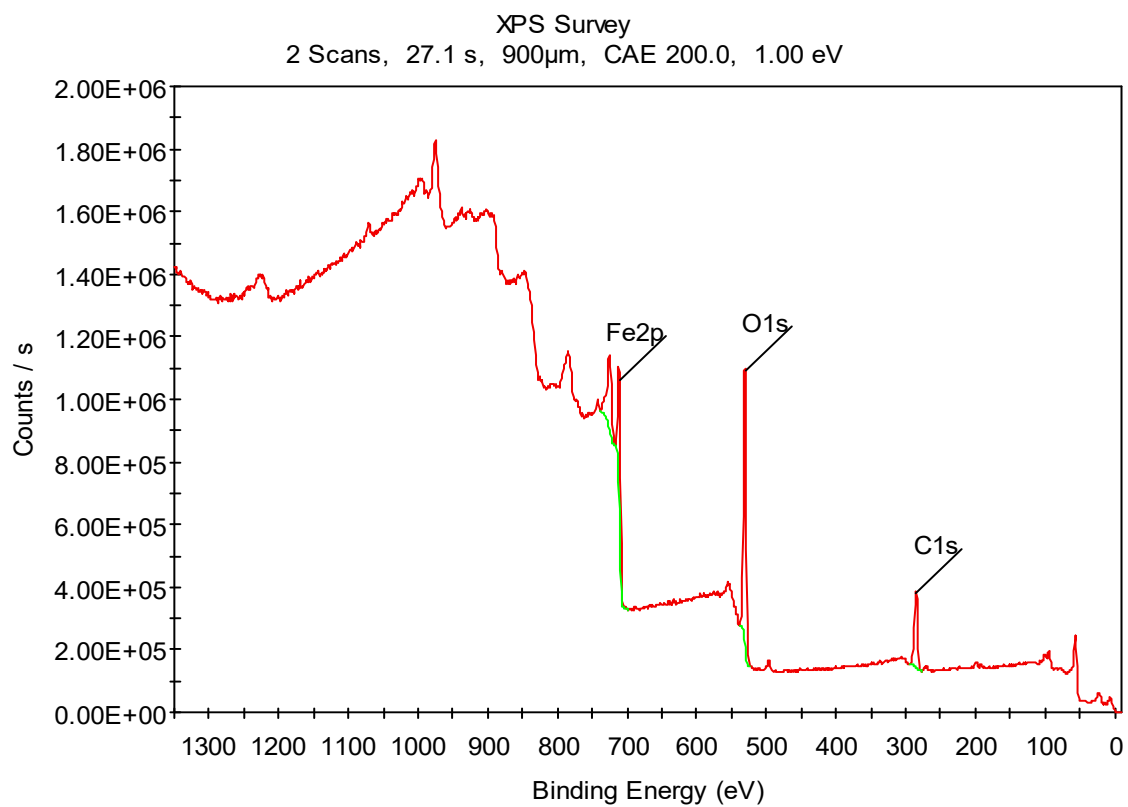


Fig. S11 XPS survey spectrum of the as prepared of Ru@Fe₃O₄-CSM

Magnetic measurements

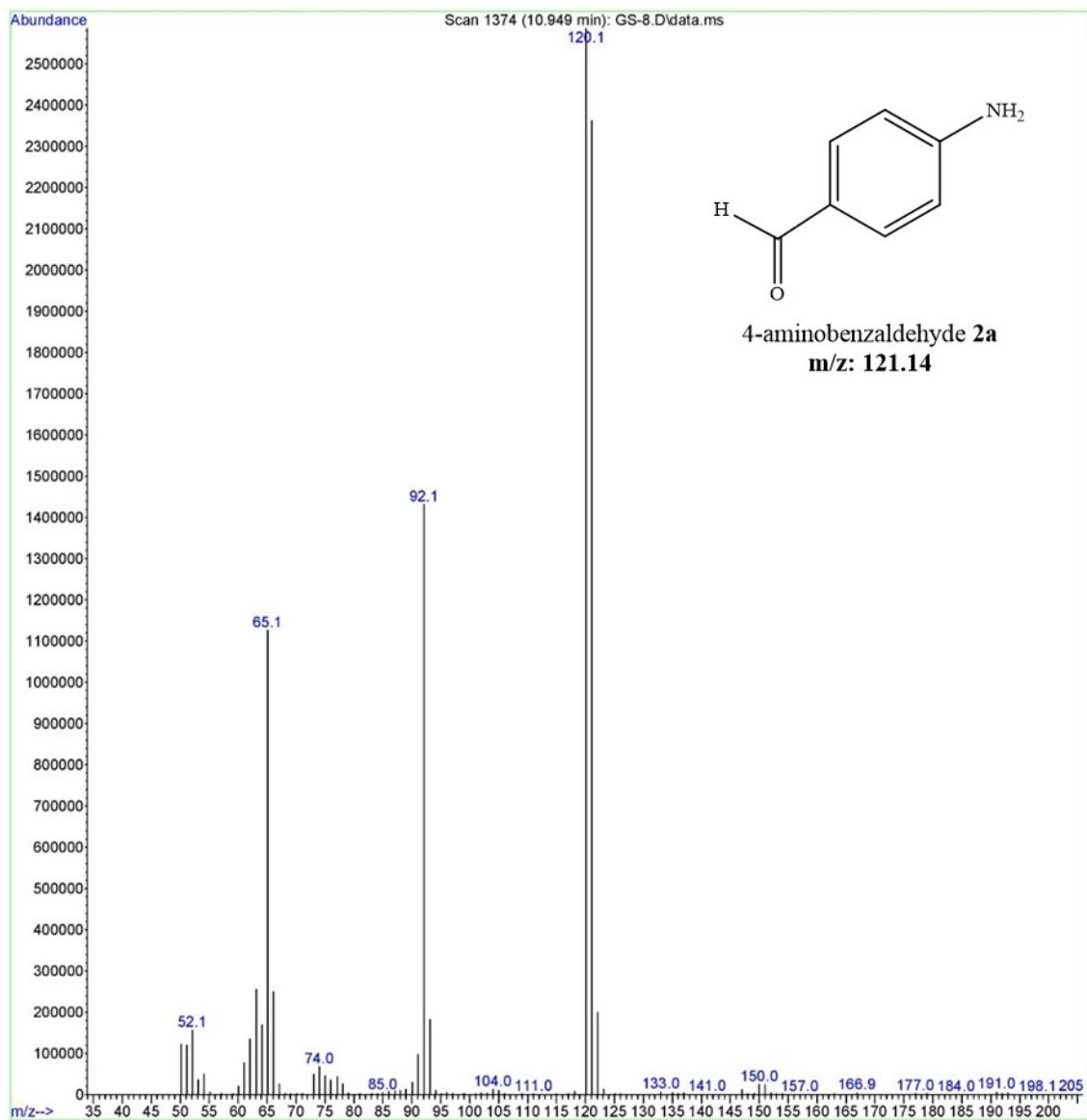
Tables S1 Numerical values for VSM analysis of Ru@Fe ₃ O ₄ /SiO ₂			
Hysteresis	Upward Part	Downward Part	Average
Hc offset Oe	-749.95	-749.44	-749.69
S	0.635	0.636	0.636
Hc Oe	-749.95	-749.44	-0.26
Ms emu/g	43.146E + 0	42.814E + 0	42.980E + 0
M at H emu/g	43.146E + 0	42.814E + 0	42.980E + 0
S	0.55	0.55	0.55
Mr emu/g	23.702E + 0	23.699E + 0	-1.482E - 3
H1 Oe	-553.20	-553.30	-553.25
H2 Oe	-946.20	-945.83	-946.01

Tables S2 Numerical values for VSM analysis of Ru@Fe ₃ O ₄ -CSM			
Hysteresis	Upward Part	Downward Part	Average
Hc offset Oe	-748.04	-752.26	-750.15
S	0.556	0.562	0.559
Hc Oe	-748.04	-752.26	2.11
Ms emu/g	19.312E + 0	-19.170E + 0	19.241E + 0
M at H emu/g	19.312E + 0	-19.170E + 0	19.241E + 0
S	0.49	0.49	0.49
Mr emu/g	9.429E + 0	9.454E + 0	12.422E - 3
H1 Oe	-519.05	-522.66	-520.86
H2 Oe	-977.58	-981.05	979.32

GC-Mass spectra of Products

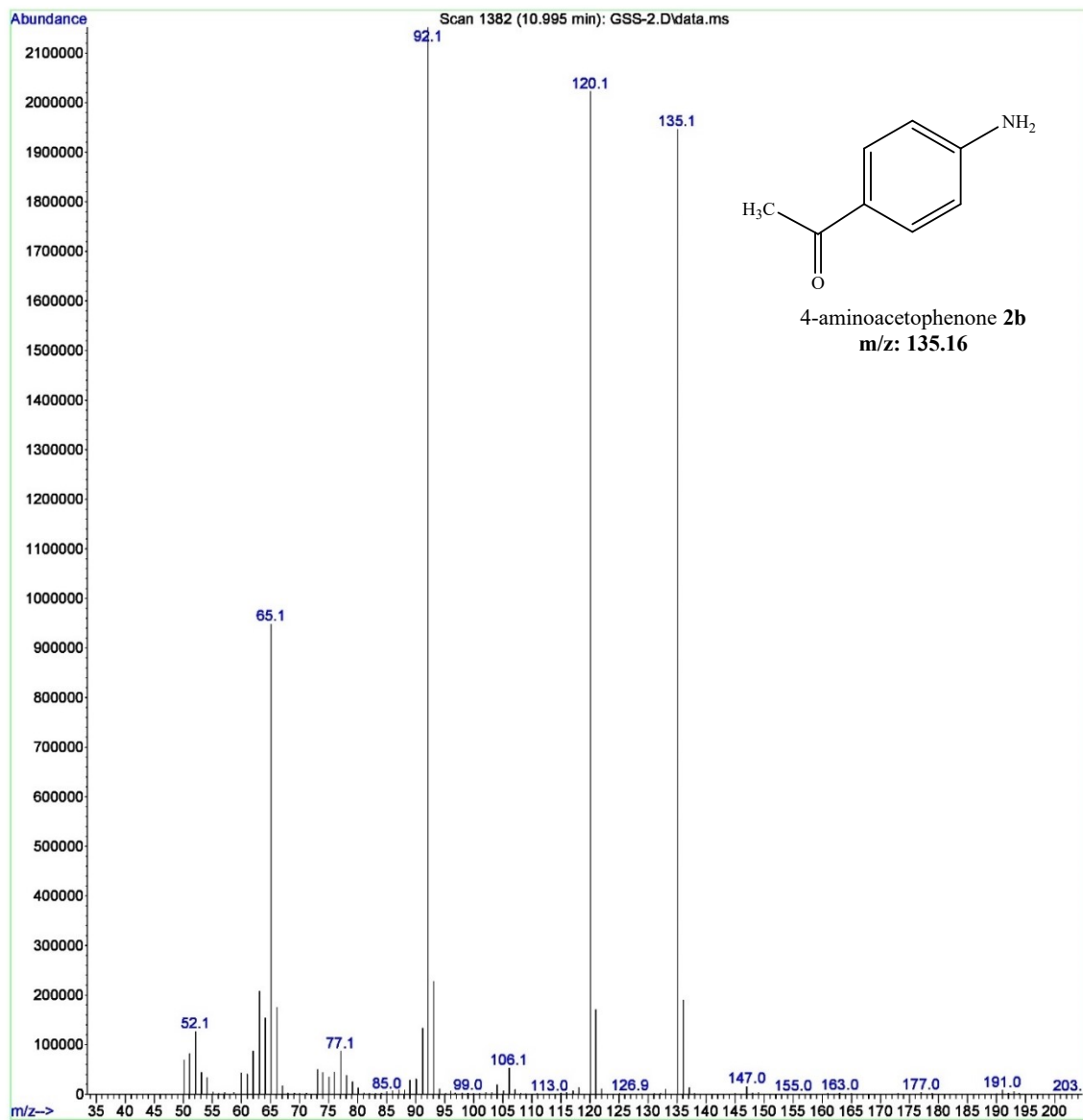
4-aminobenzaldehyde (S1)

Instrument : GCMSD
Sample Name: 4-Nitrobenzaldehyde
Misc Info :
Vial Number: 8



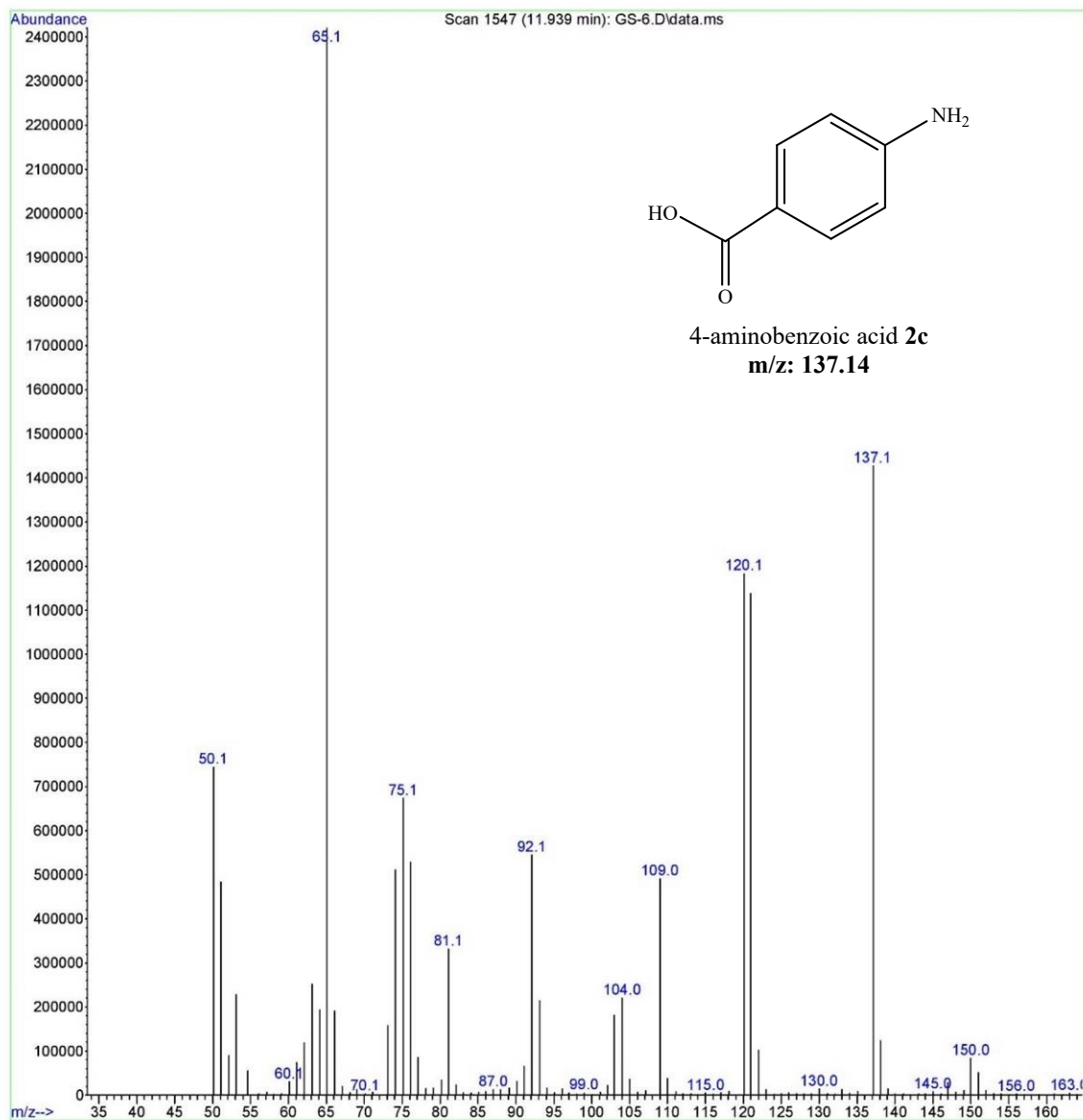
4-aminoacetophenone (S2)

Instrument : GCMSD
Sample Name: 4-Nitroacetophenon
Misc Info :
Vial Number: 2



4-aminobenzoic acid (S3)

Instrument : GCMSD
Sample Name: 4-nitrobenzoic acid
Misc Info :
Vial Number: 6



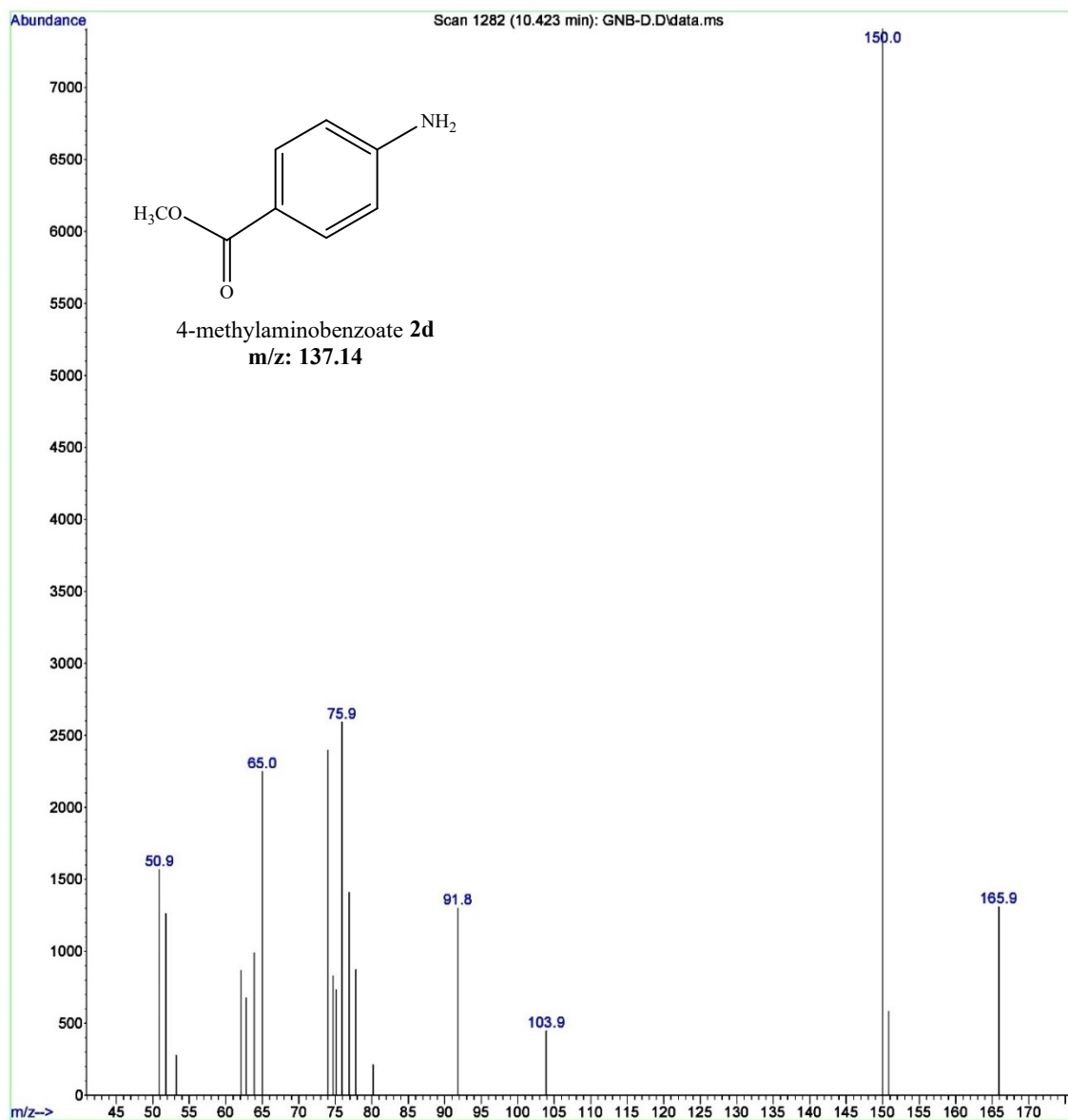
4-methylaminobenzoate (methylantranilate) (S4)

Instrument : GCMSD

Sample Name: GNB-D

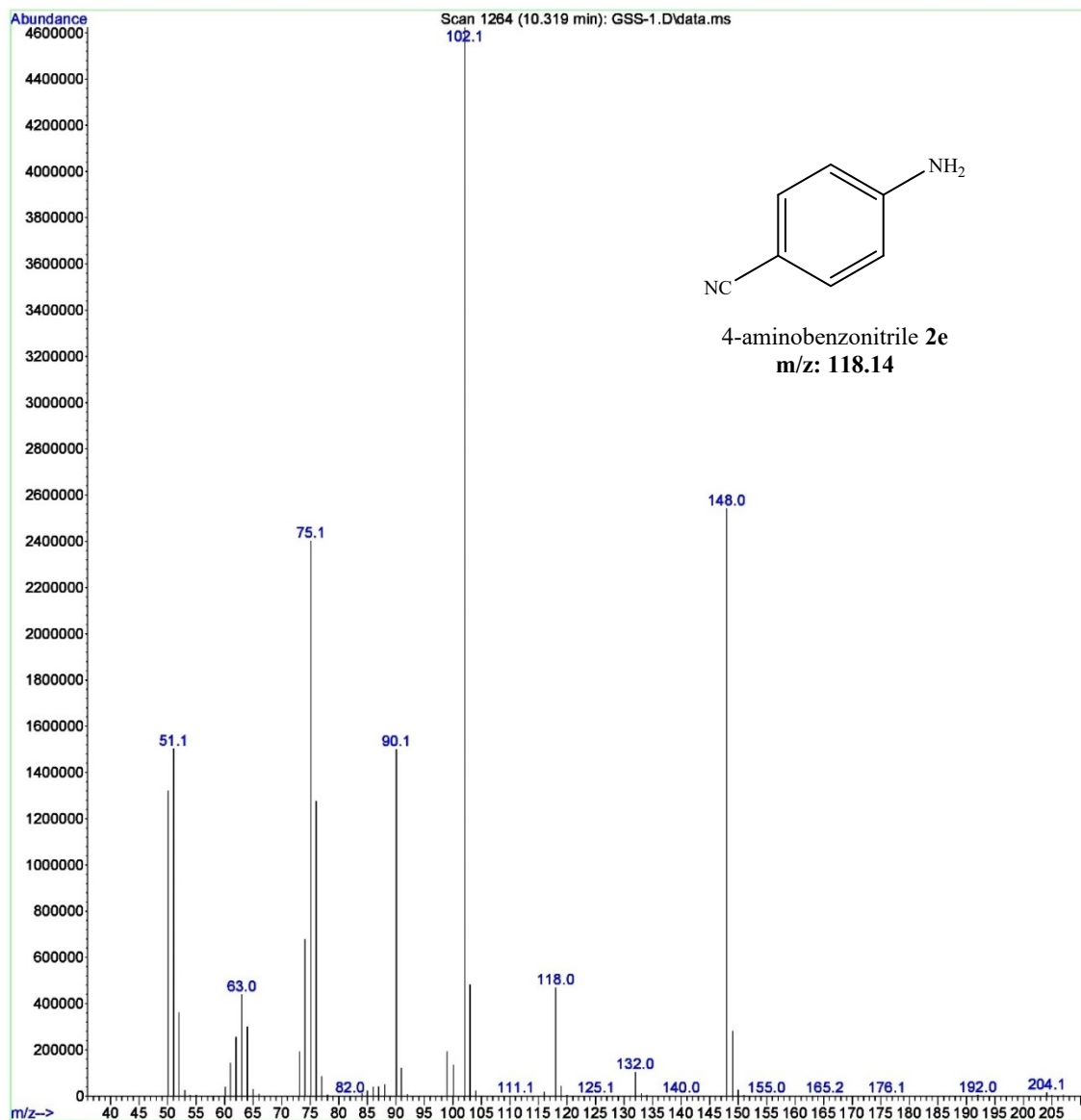
Misc Info :

Vial Number: 1



4-aminobenzonitrile (S5)

Instrument : GCMSD
Sample Name: 4-Nitrobenzonitrile
Misc Info :
Vial Number: 1



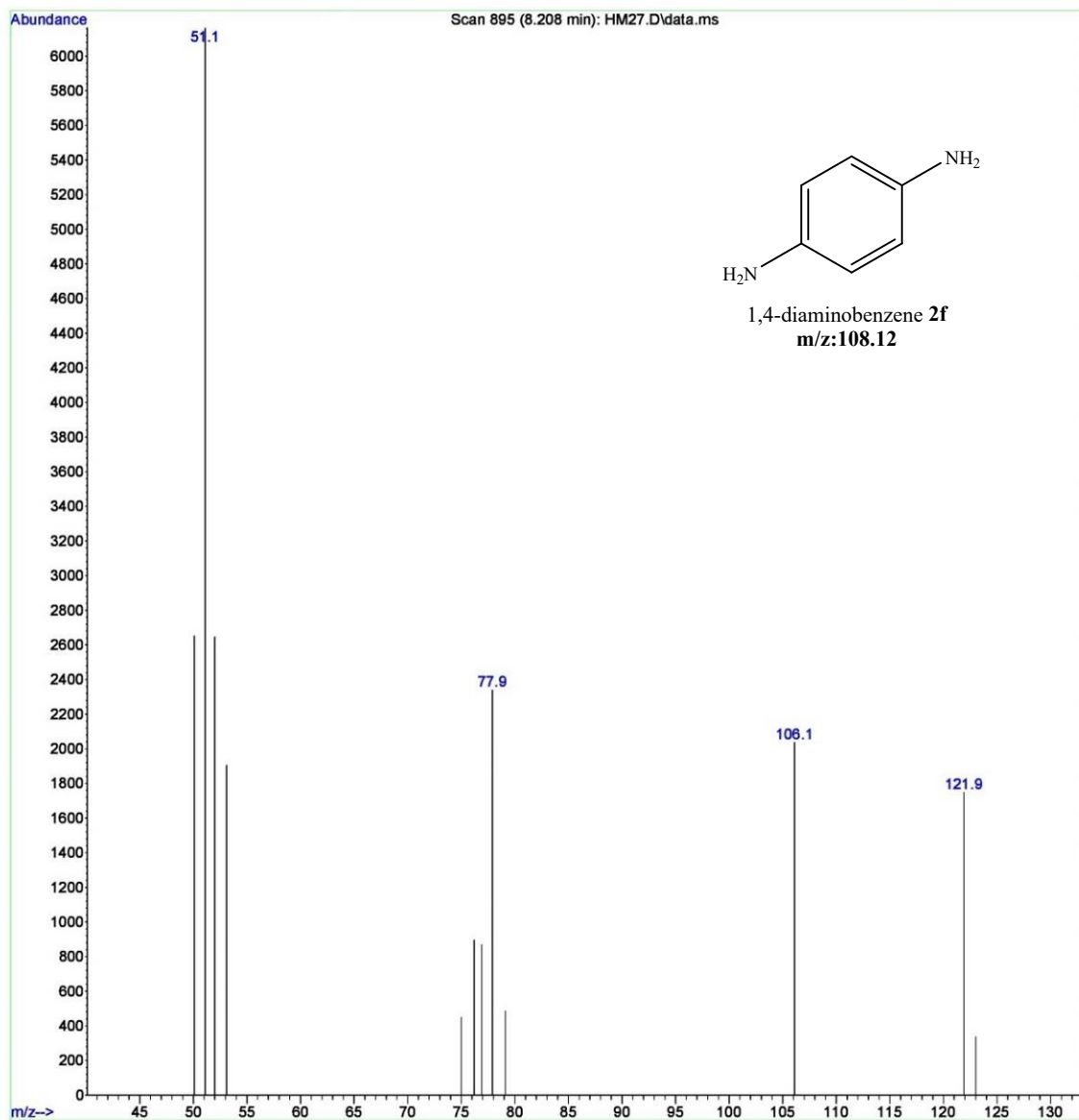
1,4-diaminobenzene (p-phenylenediamine) (S6)

Instrument : GCMSD

Sample Name: HM27

Misc Info :

Vial Number: 1



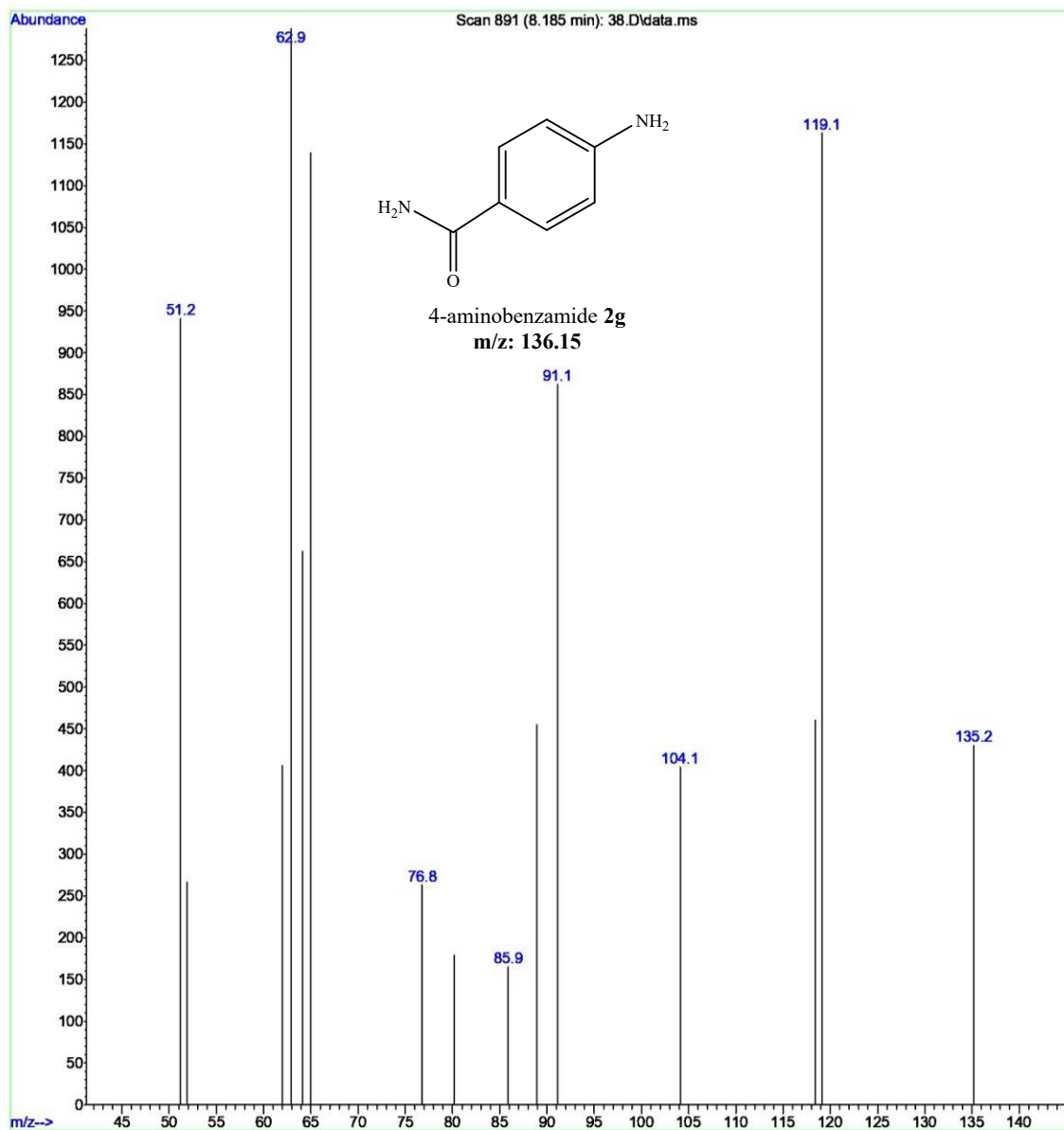
4-aminobenzamide (p-phenylenediamine) (S7)

Instrument : GCMSD

Sample Name: 38

Misc Info :

Vial Number: 1



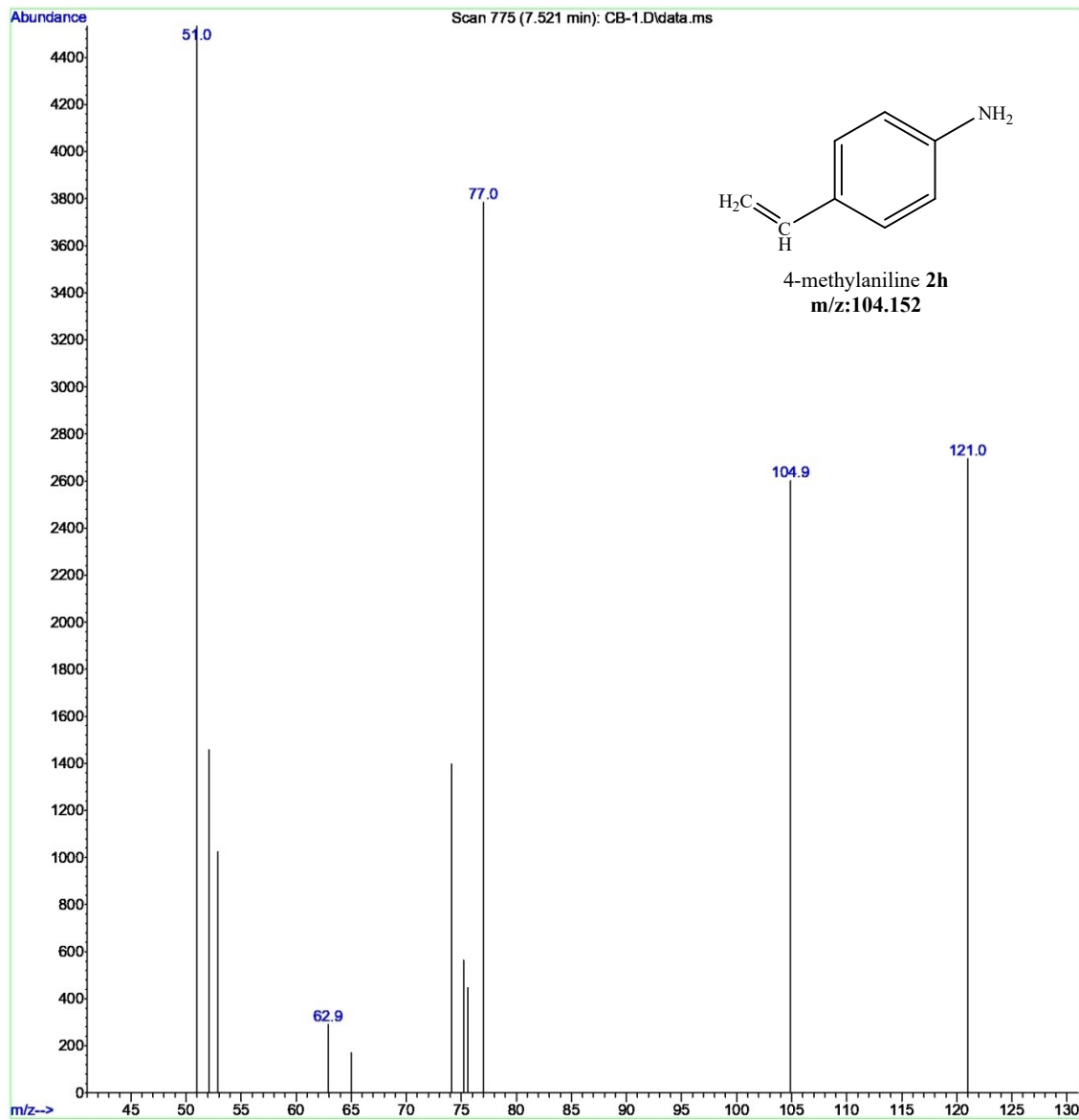
Styrene (S8)

Instrument : GCMSD

Sample Name: CB-1

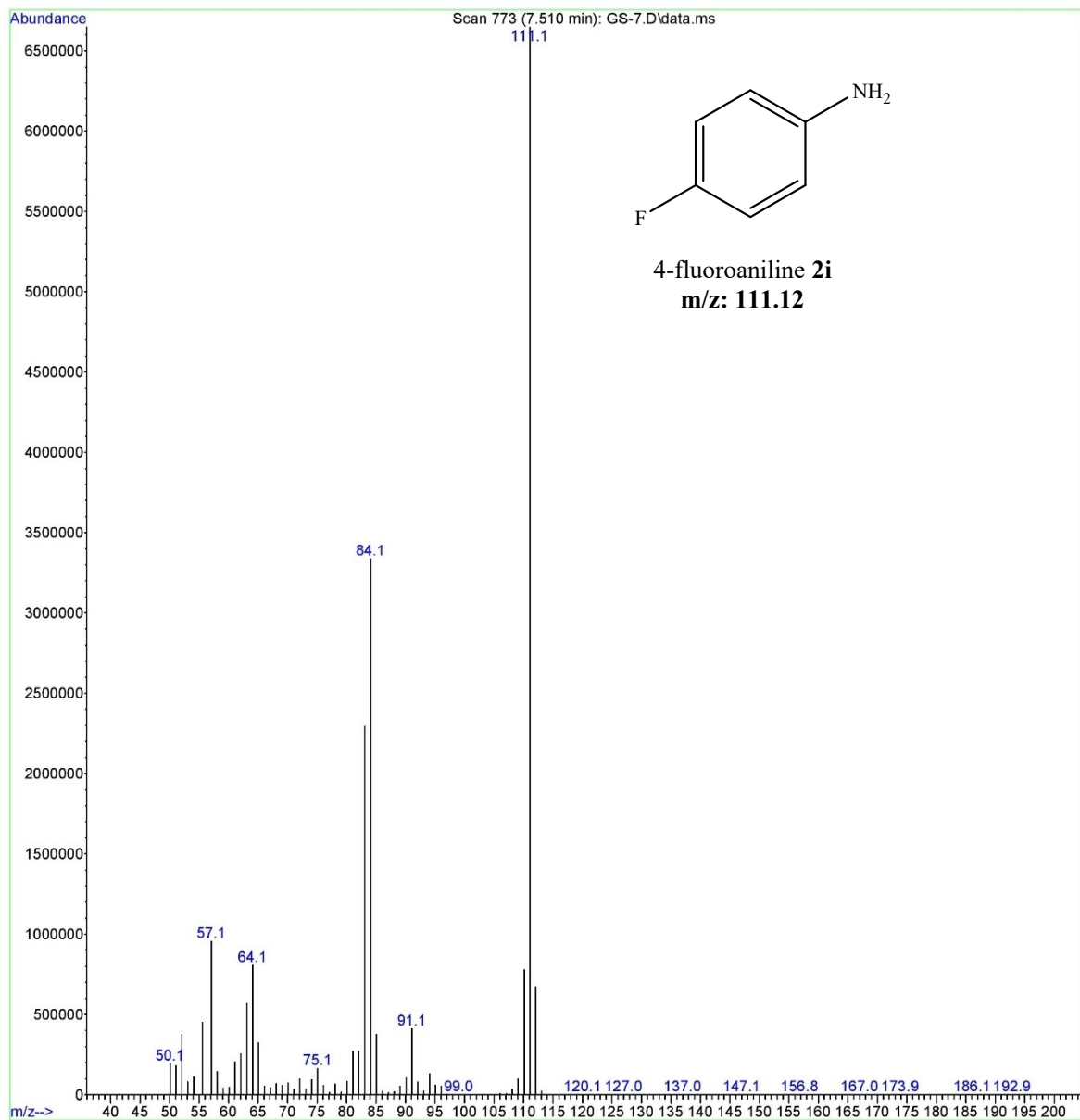
Misc Info :

Vial Number: 1



4-fluoroaniline (S9)

Instrument : GCMSD
Sample Name: 1-Fluoro-4-Nitrobenzene
Misc Info :
Vial Number: 7



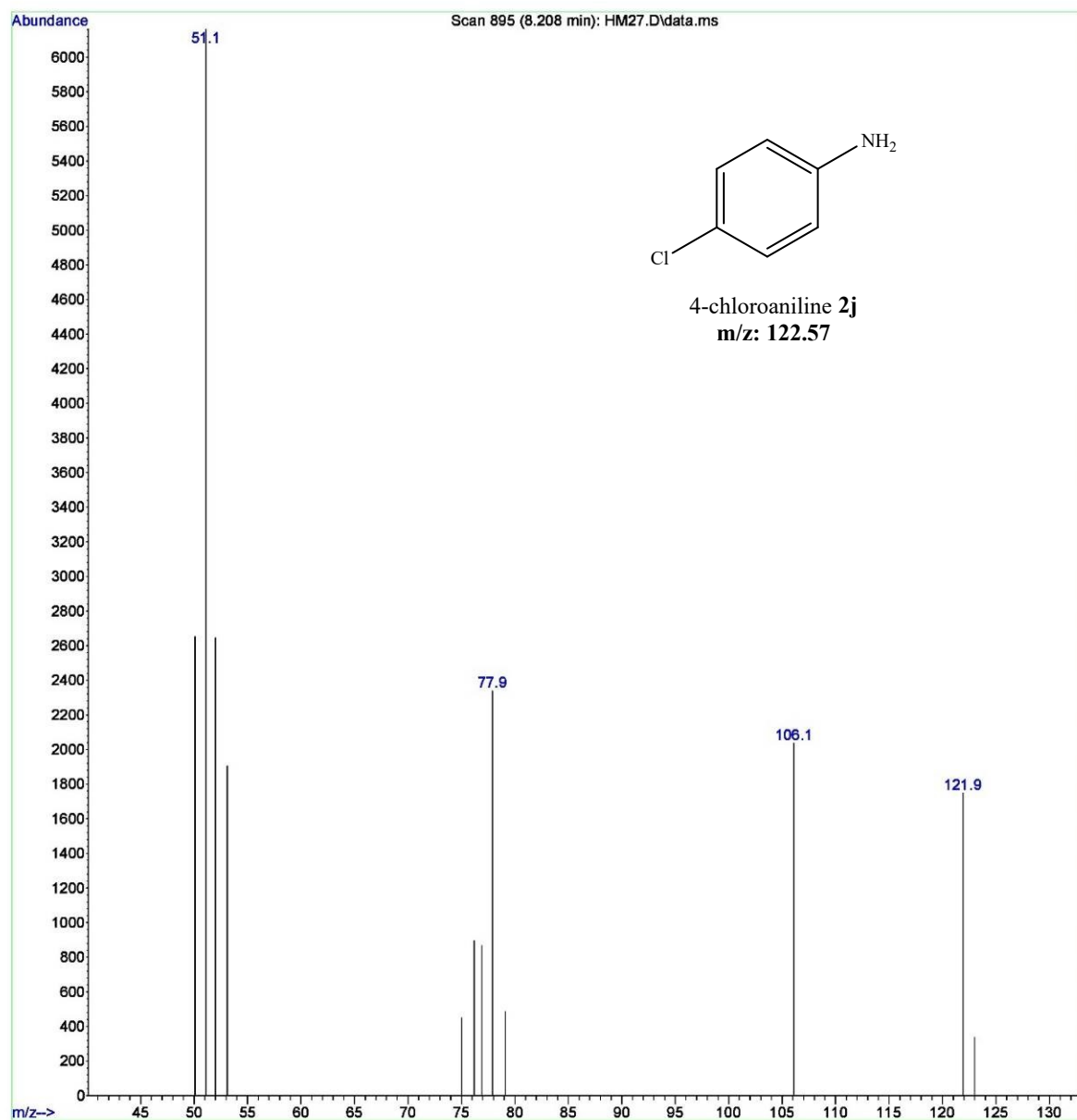
4-chloroaniline (S10)

Instrument : GCMSD

Sample Name: HM27

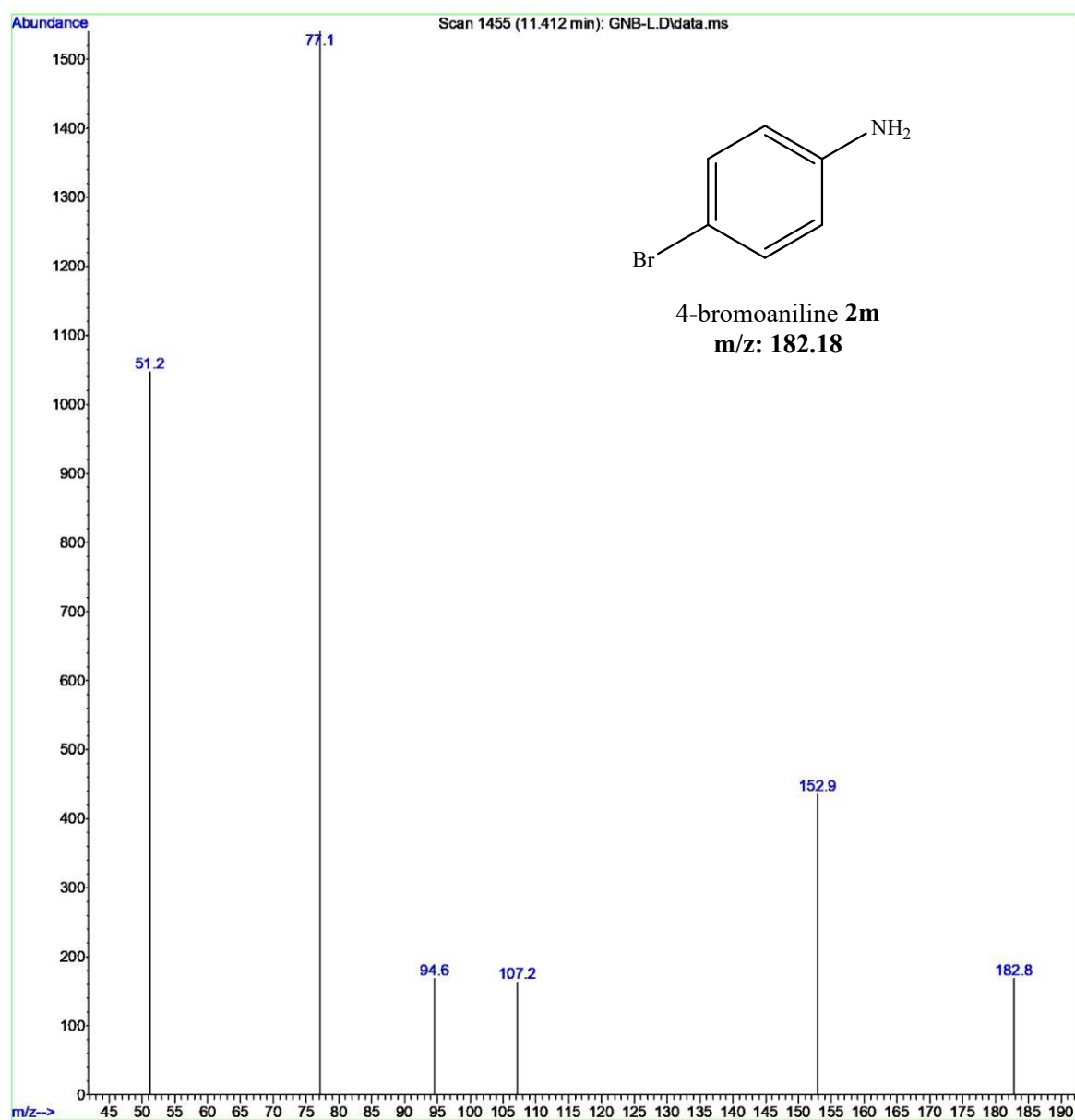
Misc Info :

Vial Number: 1



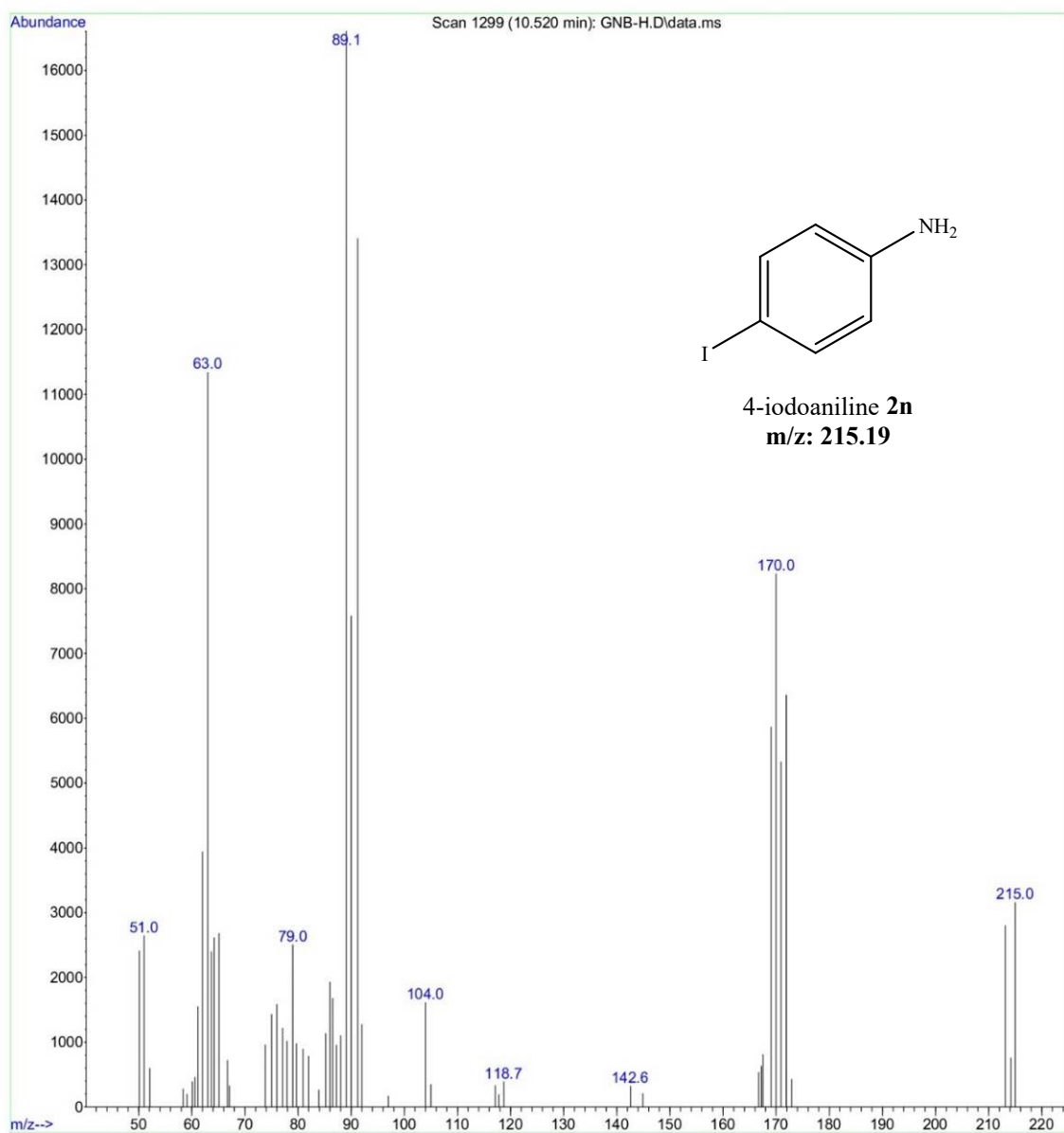
4-bromoaniline (S11)

Instrument : GCMSD
Sample Name: GNB-L
Misc Info :
Vial Number: 1



4-iodoaniline (S12)

Instrument : GCMSD
Sample Name: GNB-H
Misc Info :
Vial Number: 1



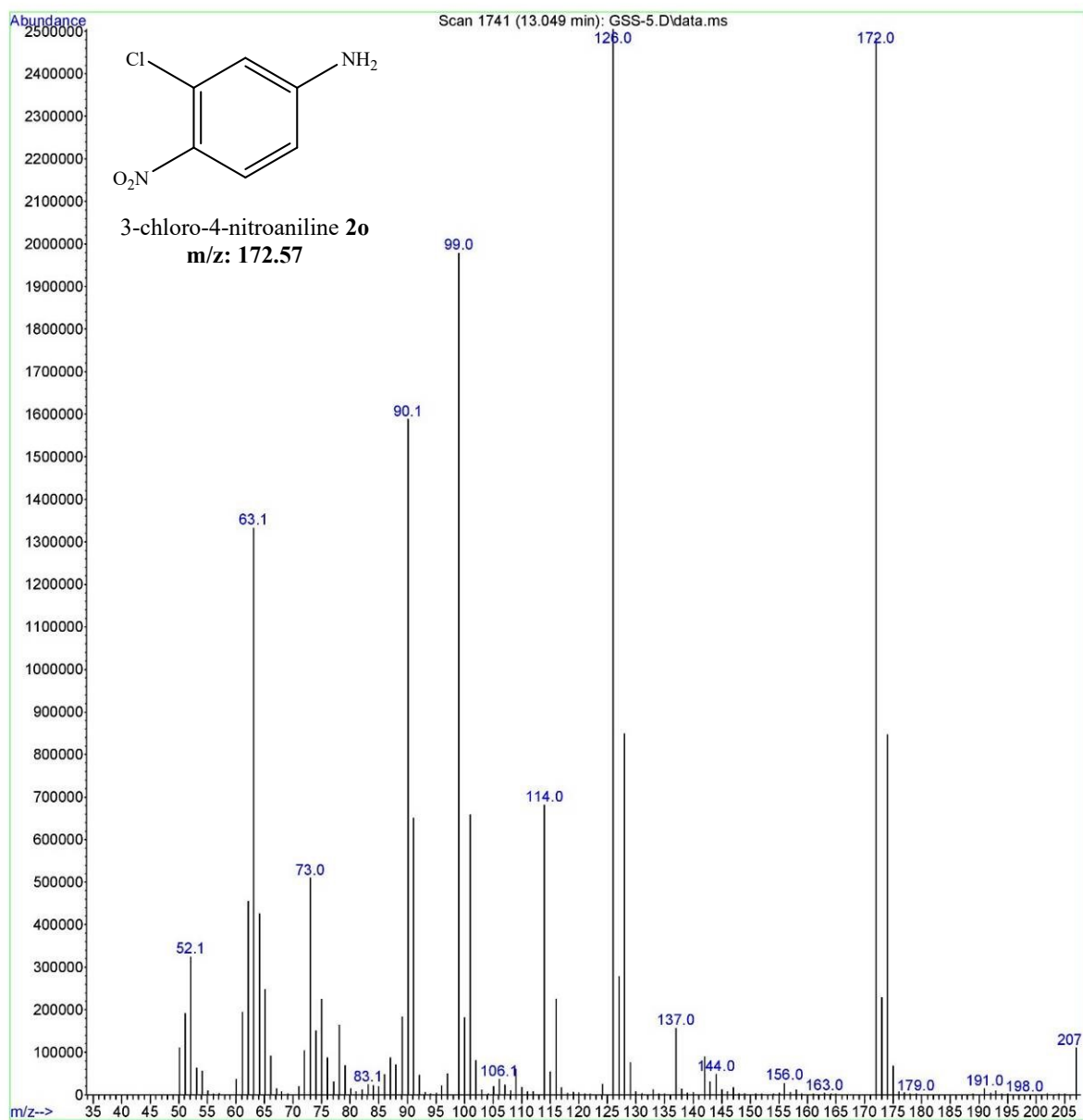
3-chloro-4-nitroaniline (S13)

Instrument : GCMSD

Sample Name: 1-Chloro-2,5-Dinitrobenzene

Misc Info :

Vial Number: 5



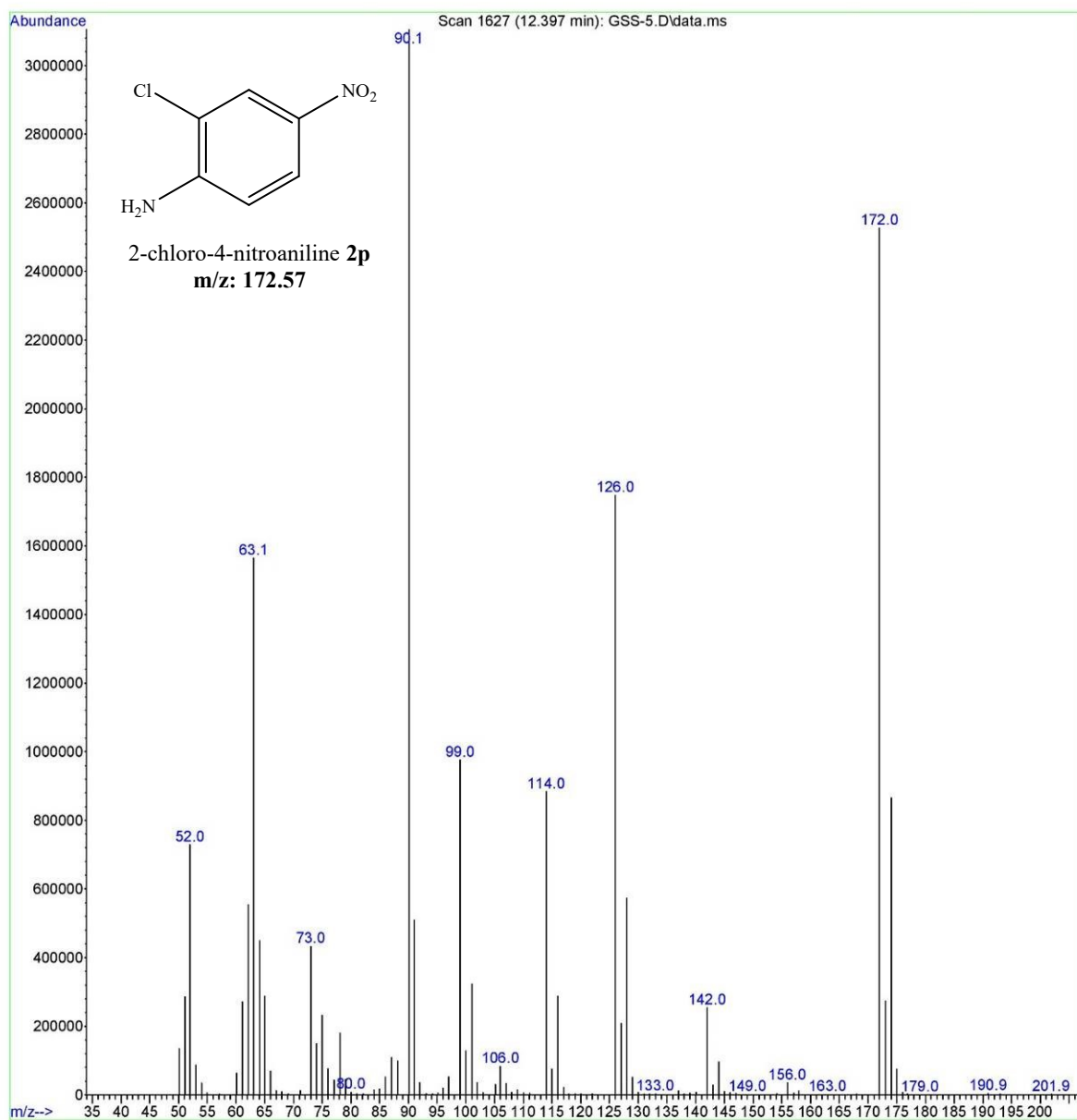
2-chloro-4-nitroaniline (S14)

Instrument : GCMSD

Sample Name: 1-Chloro-2,5-Dinitrobenzene

Misc Info :

Vial Number: 5



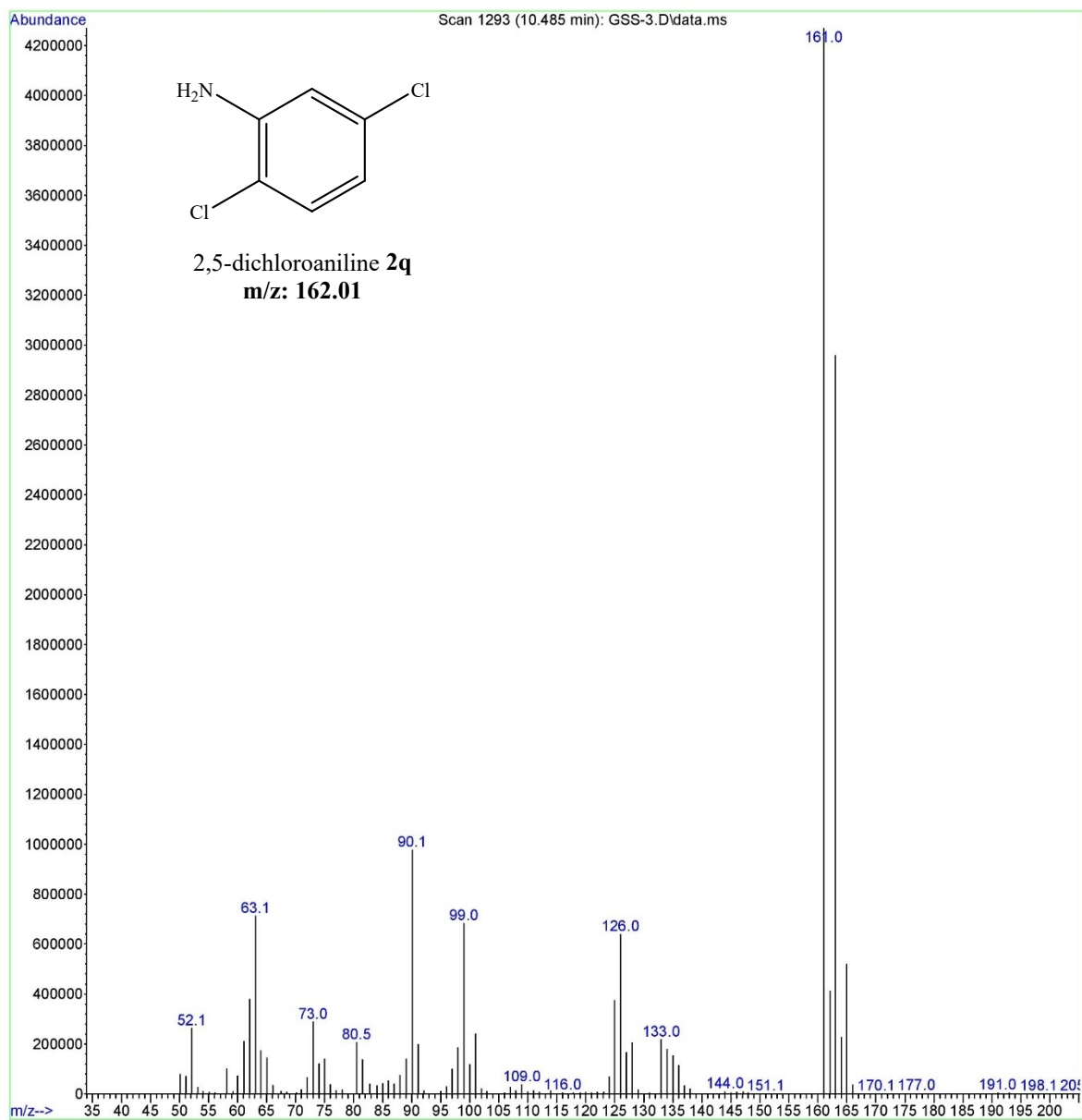
2,5-dichloroaniline (S15)

Instrument : GCMSD

Sample Name: 2,5-Dichloro-1-Nitrobenzene

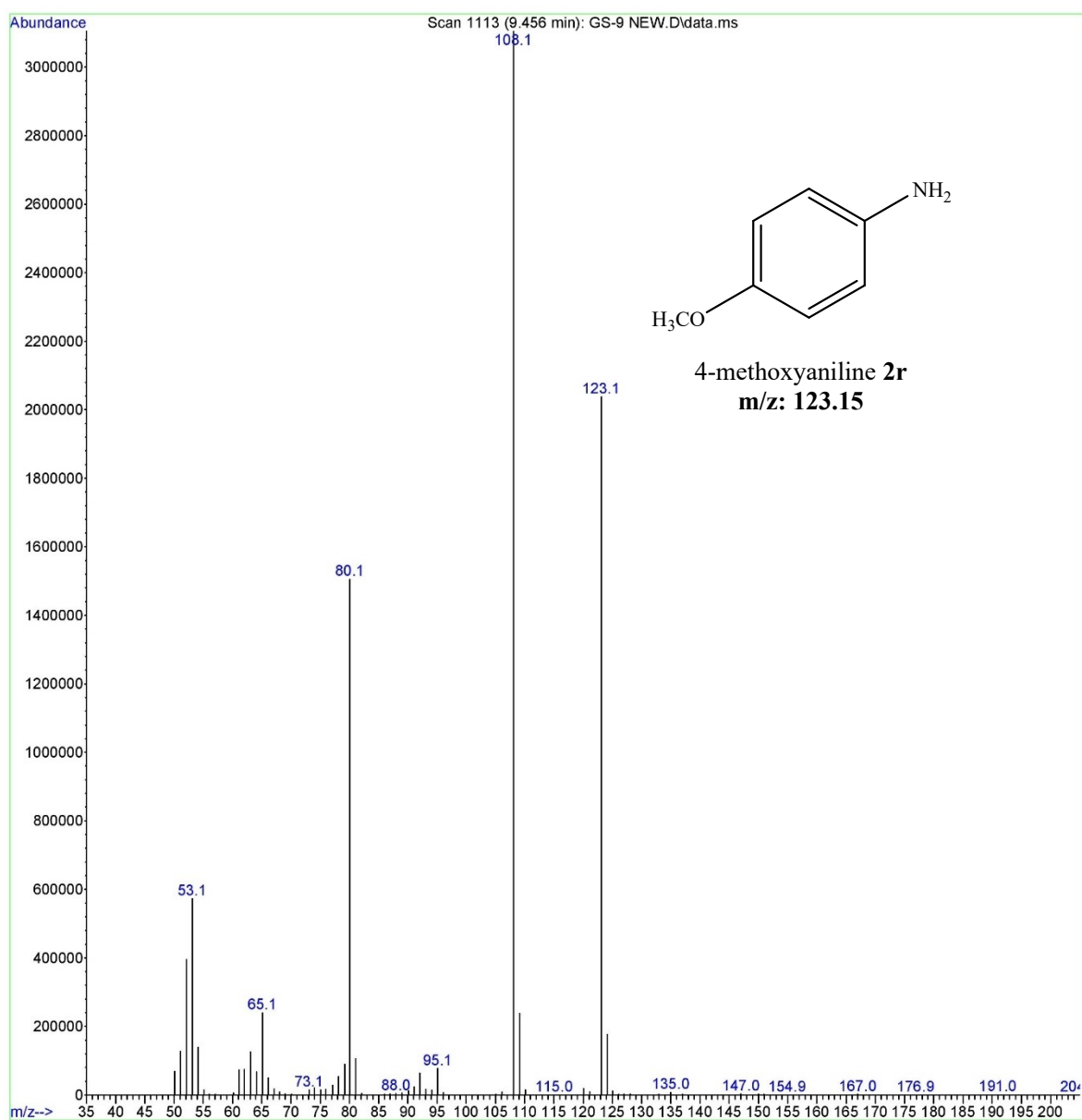
Misc Info :

Vial Number: 3



4-methoxyaniline (S16)

Instrument : GCMSD
Sample Name: 4-Nitroanisoie
Misc Info :
Vial Number: 9



4-methylaniline (S17)

File : C:\Users\Admin\Desktop\GAURAV\GSS-4.D
Operator :
Acquired : 17 Sep 2019 14:34 using AcqMethod Diganta.M
Instrument : GCMSD
Sample Name: 4-Nitrotoluene
Misc Info :
Vial Number: 4

