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Preparation And Identification Of New Organoselenium Compounds Based On N-Phenyl-2 Selenocyanatoacetamide

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Abstract

The current study focuses on the three-step process for creating new organoselenium compounds , potassium selenocyanate was produced by reacting selenium metal (se) with potassium cyanide (KCN) in DMSO dimethyl sulphoxide as a solvent in the presence of the aragon gas of interest ,N-phenyl-2-selenocyanatoacetamide was produced in good quantities by reacting potassium selenocyanatewith 2-chloro-N-phenylacetamide , the last step Includestrihalide derivatives were obtained by reacted N-phenyl-2-selenocyanatoacetamide with bromine, thionyl chloride and iodine. All compounds were characterized by IR, H-NMR and mass-spectrometry.

Keywords : organoselenium , selenium metal and N-phenyl-2-selenocyanatoacetamide

Introduction

Berzelius discovered se in sludg of the lead chambers of sulphuric acid(H_2SO_4) chamber of a plant at Gripsholmsweden in 1817[1][2].Selenium belongs to the Periodic Table Sixteengroup ,the atom is two electrons short of the next noble gas's configuration and the element has a nonmetallic covalent-nature [3] , electronic configuration of selenium is $[Ar]3d^{10}4s^{2}4p^{4}$[4].

By generating chalconide ions, they can complete the noble gas configuration. Se^{-2},only the salts of the most electropositive elements contain these ions, and they are not stable. [4].

oxidation statesof Se form compounds are -1, -2, +1, +2, +4 and +6, +2 and +4 are most stable oxidation states. The isotopes and natural abundance of Se atom are 9.19% 82Se,49.82% 80Se, 23.52% 78Se ,7.58% 77Se, 9.02%76Se and 0.87% 74Se. The electronegativity of se is 2.55 on Pauling scale and atomic radius is 120pm.

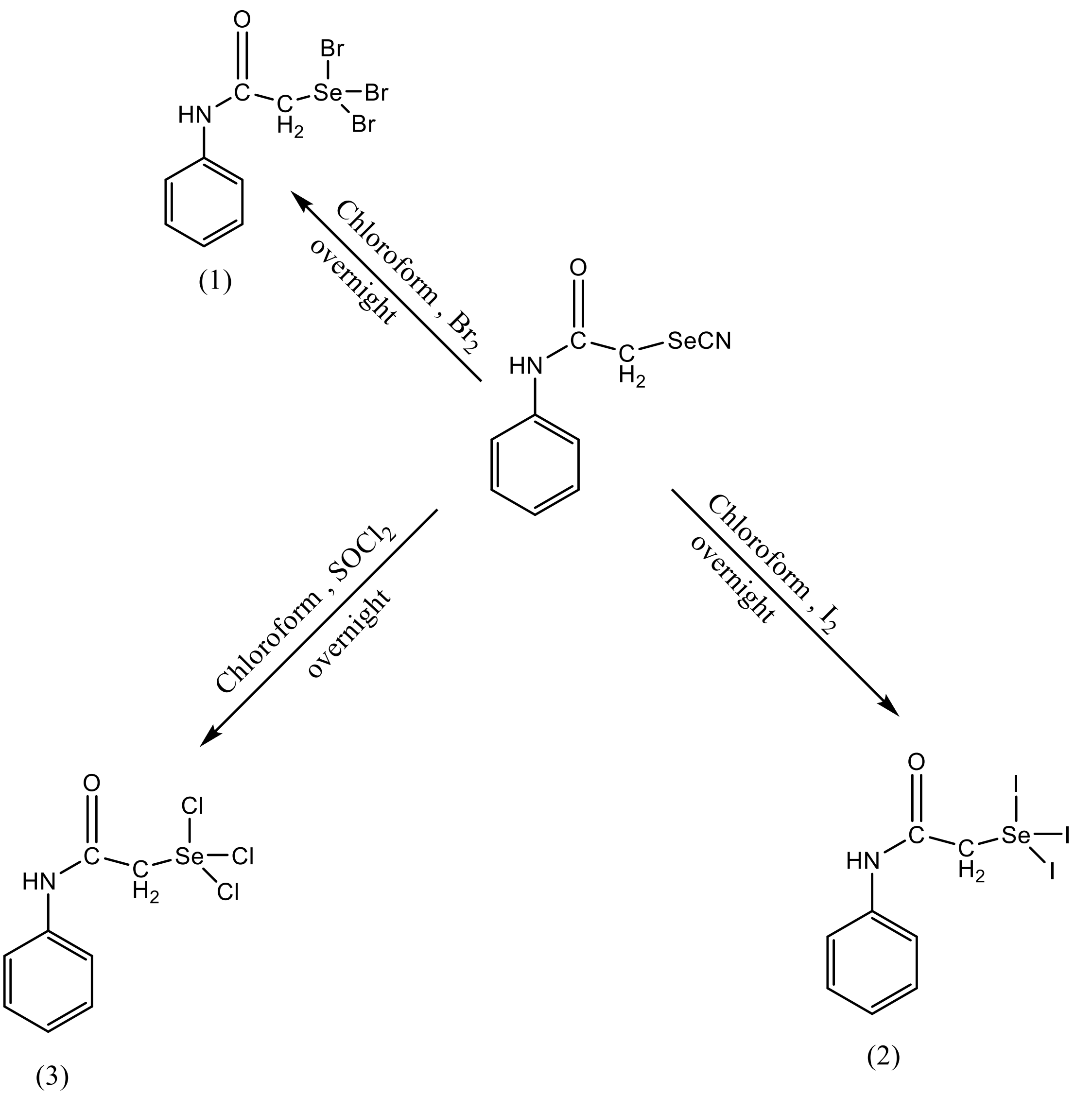
2. Experimental

2.1: Chemicals and Apparatus

Chemicals Fluka, σ-Aldrich and British Drug House(BDH)were used. Using an open hairliketube dissolving point mechanicalassembly, the liquefyingpoint was determined. Proton nuclear magnetic resonance(1HNMR) spectra wasrecorded on Bruker DRX-System al500MHzwith TMS as aninner reference utilizing DMSO-d6 dis-solvable. Infra-red spectra (IR) were record with KBr circles utilizing a Fourier transform infrared spectrophotometer (FT- IR)bruker model 8400S in4000-500 cm^{-1}.

2.2 Preparation of N-phenyl-2-(tribromo- λ^4-selaneyl) acetamide (1) , N-phenyl-2-(triiodo-λ^4-selaneyl) acetamide(2) and N-phenyl-2-(trichloro-λ^4-selaneyl) acetamide(3)

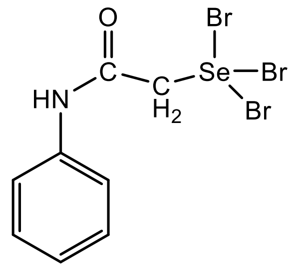
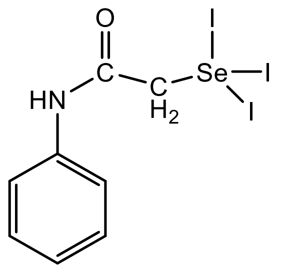
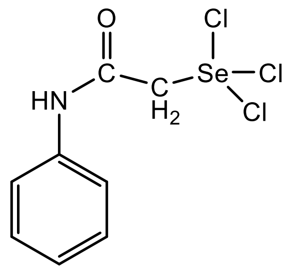
0.002 mole of N-phenyl-2-selenocyanatoacetamidedissolved in10ml of chloroform , carefully added to it a few drop of halogens dissolved in 5ml of chloroform with continued stirring, consisting of crystal leave solution to stagnate[5], filter, wash sediment with chloroform and dry , asshown in scheme (1)



Scheme (1): Preparation of N-phenyl-2-(tribromo- λ^4-selaneyl) acetamide (1) , N-phenyl-2-(triiodo-λ^4-selaneyl) acetamide(2) and N-phenyl-2-(trichloro-λ^4-selaneyl) acetamide(3)

Table 1. Physical data of N-phenyl-2-(tribromo- λ^4-selaneyl) acetamide (1) , N-phenyl-2-(triiodo-λ^4-selaneyl) acetamide(2) and N-phenyl-2-(trichloro-λ^4-selaneyl) acetamide(3)

Symbol	Structureformula Name	Molecular Wight	M.P/C°	Yield %	Color	Reaction time

(1)	 *N*-phenyl-2-(tribromo-λ^4-selaneyl) acetamide	452.83	138-140	33	Brown	24h
(2)	 *N*-phenyl-2-(triiodo-λ^4-selaneyl) acetamide	593.83	150-152	25	Brown	24h
(3)	 *N*-phenyl-2-(trichloro-λ^4-selaneyl) acetamide	319.47	121-123	31	Brown	24h

3. Results and Discussion

Threeorganoselenium compounds, N-phenyl-2-(tribromo- λ^4-selaneyl) acetamide (1) , N-phenyl-2-(triiodo-λ^4-selaneyl) acetamide(2) and N-phenyl-2-(trichloro-λ^4-selaneyl) acetamide(3) are produced by react N-phenyl-2-selenocyanatoacetamidewith halogens, as shown in Scheme (1)

Infrared(IR) spectra confirm the suggested structure of compounds , IR spectroscopy of the prepared compounds is shown in Figure (1),(2)and (3)and Table (2)[7][6][8]

(1) : (IR/cm^{-1}): ((-C=O1665))((-NH- st-3287))((-NH ben-1514)) ((-C-N1183)) ((C-Caliph ≈ 980)) ((C=Car≈1600))((C-Haliph- ≈3000))((C-Hst-ar- ≈3100))((C-Hben-ar- 736)).

(2) : (IR/cm^{-1}): ((-C=O1654)) ((-NH st- 3291)) ((-NH ben- 1524)) ((-C-N1238)) ((C-Caliph-932)) ((C=Car-1589)) ((C-Haliph- ≈3000)) ((C-Hst-ar-≈3100)) ((C-Hben-ar-748)).

(3) : (IR/cm^{-1}): ((-C=O1657)) ((-NH st-3294)) ((-NH ben-1527)) ((-C-N1309)) ((C-Caliph-1030)) ((C=Car-1594)) ((C-Haliph-2924)) ((C-Hst-ar-3024)) ((C-Hben-ar-748)).

Table 2. The Infraredbeams of prepared compounds /cm^{-1}.

	C=O	N-Hst-	N-H ben-	C-N	C-C aliph	C=C ar	C-H aliph	C-H st-ar	C-H ben/ar
(1)	1665	3287	1514	1183	≈980	≈1600	≈3000	≈3100	736
(2)	1654	3291	1524	1238	932	1589	≈3000	≈3100	748
(3)	1657	3294	1527	1309	1030	1594	2924	3024	748

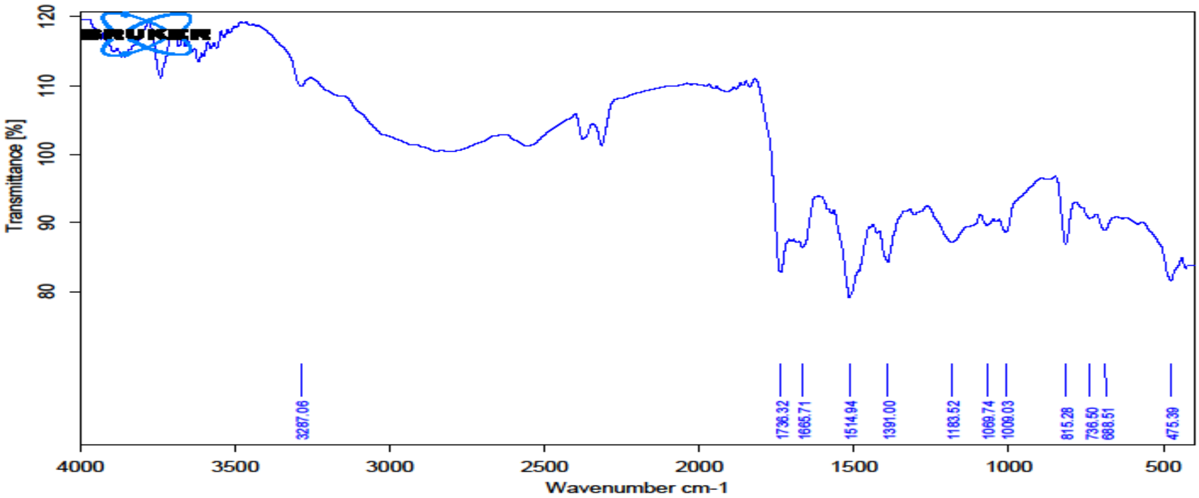


Figure 1. InfraredIR-spectra of N-phenyl-2-(tribromo-λ^4-selaneyl)acetamide (1) .

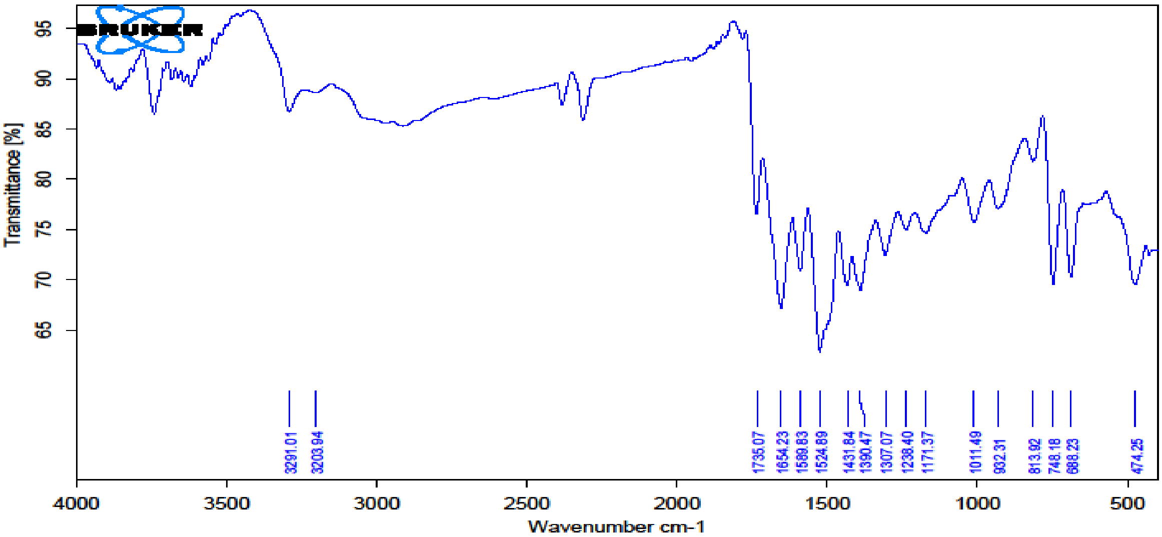


Figure 2. InfraredIR-spectra of N-phenyl-2-(triiodo-λ^4-selaneyl)acetamide (2) .

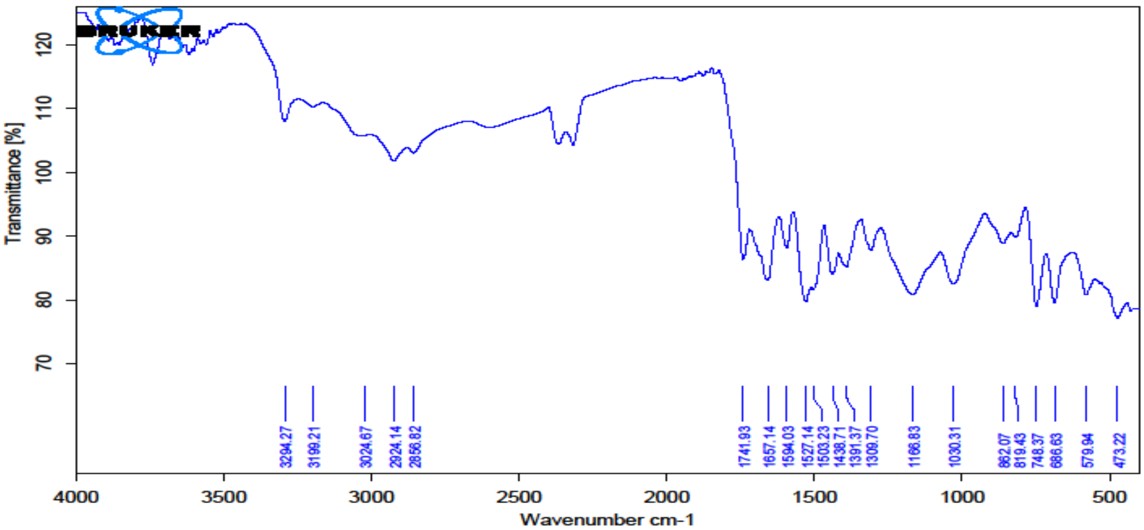


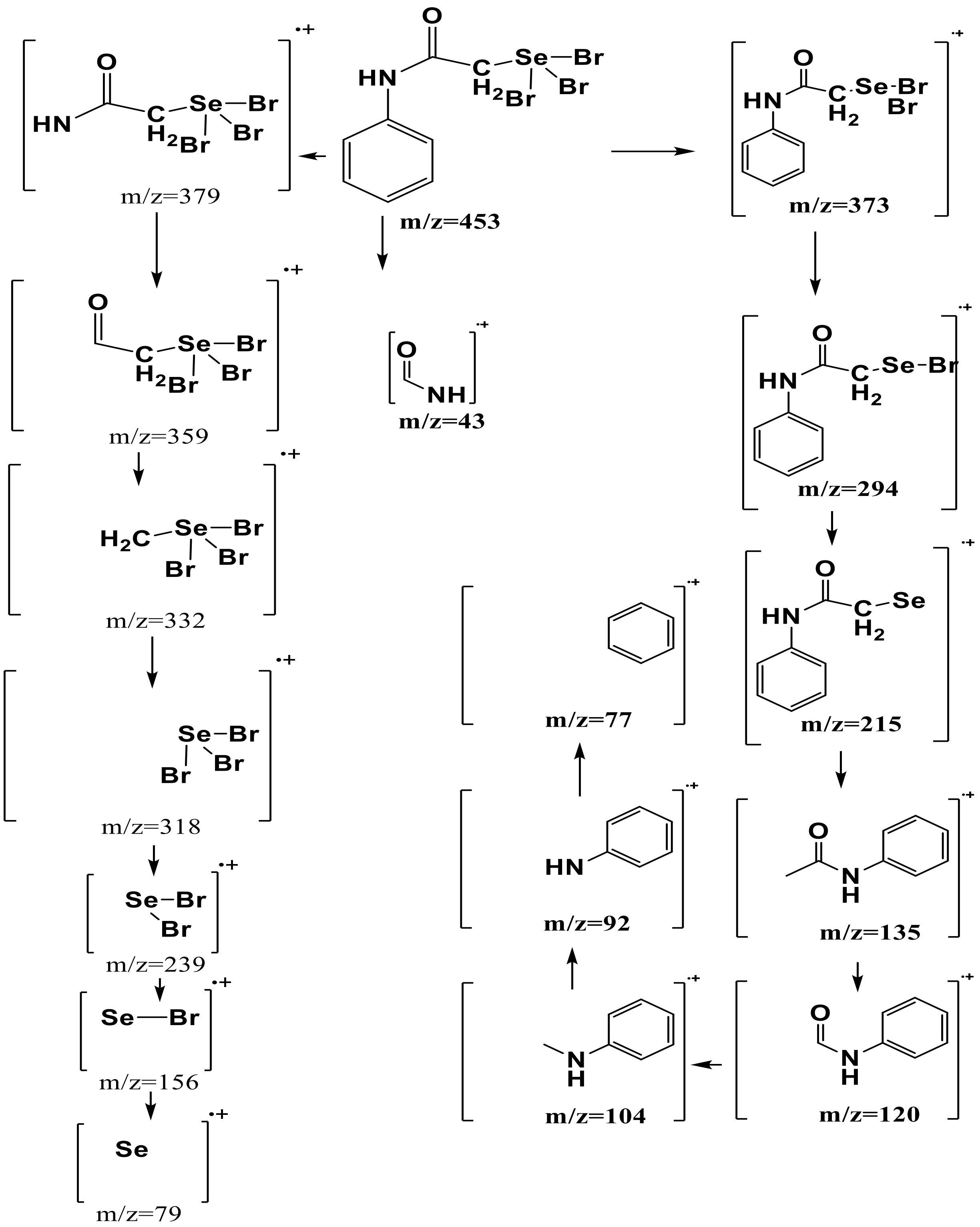
Figure 3. InfraredIR-spectra of N-phenyl-2-(trichloro-λ^4-selaneyl)acetamide(3) .

Mass-spectrum ofN-phenyl-2-(tribromo-λ^4-selaneyl)acetamide (1) show parent beams ion at 453 m/z[8] which represent the M-Wt of compound .Addition other packs shown in table (3) Figure (4) and Scheme (2).

Table 3. Important peaks of N-phenyl-2-(tribromo-λ^4-selaneyl)acetamide (1).

Molecular formula	m/z	Molecular formula	m/z
$C_8H_8Br_3NOSe$	453	CH_2Br_3Se	332
$C_8H_8Br_2NOSe$	373	Br_3Se	318
$C_8H_8BrNOSe$	294	Br_2Se	239
C_8H_8NOSe	215	BrSe	156
$C_2H_3Br_3NOSe$	379	Se	79
$C_2H_2Br_3OSe$	259	CH_2NO	43
C_6H_5	77	C_8H_8NO	135
C_7H_6N	104	C_6H_6N	92

Scheme 2. Mechanism of fragmentation ofN-phenyl-2-(tribromo-λ^4-selaneyl)acetamide (1).


m/z=453
m/z=379
m/z=373
m/z=359
m/z=43
m/z=294
m/z=332
m/z=215
m/z=77
m/z=318
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m/z=156
m/z=104
m/z=120
m/z=79

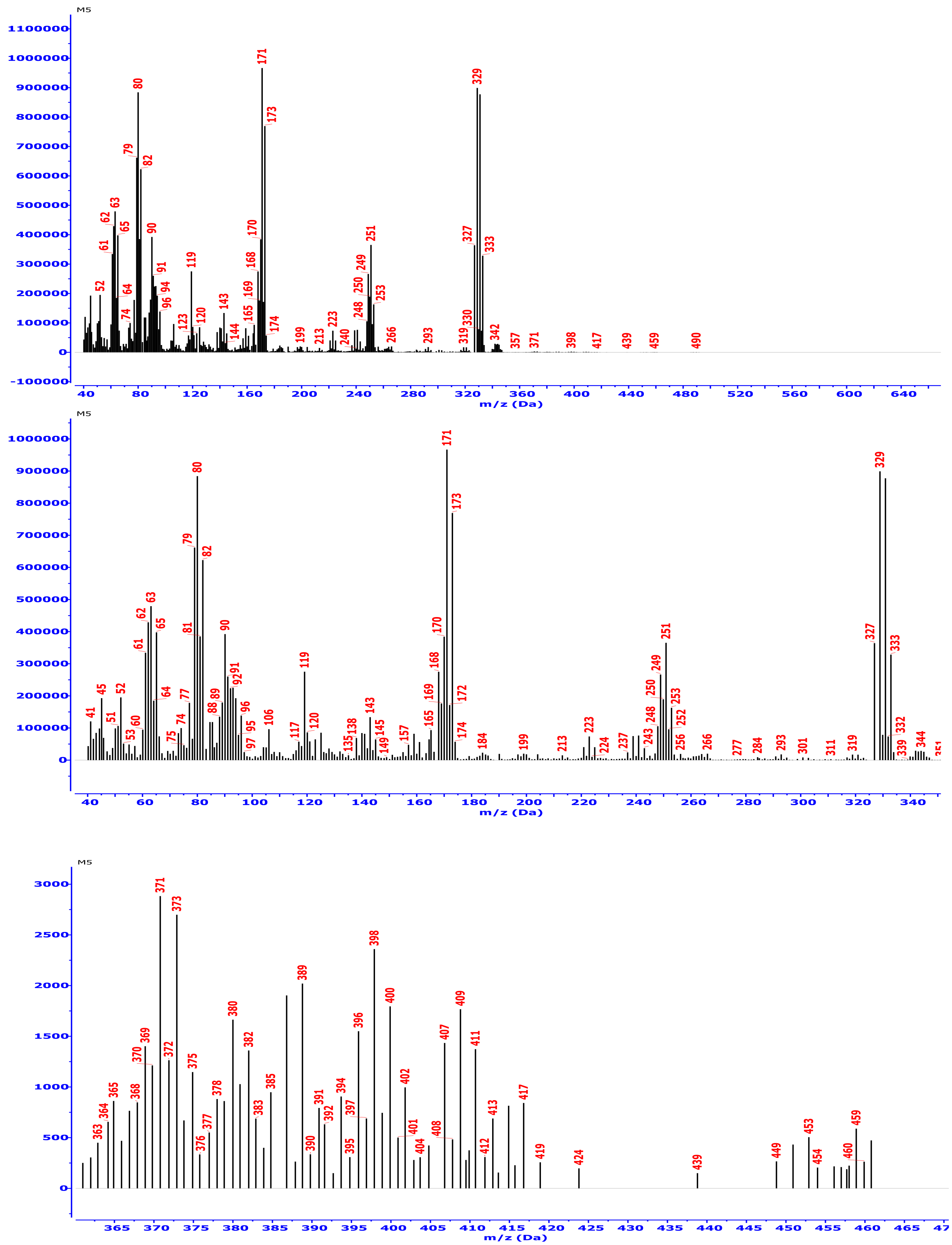


Figure 4. Mass-spectra ofN-phenyl-2-(tribromo-λ^4-selaneyl)acetamide (1).

Mass-spectrum of N-phenyl-2-(triiodo-λ^4-selaneyl)acetamide (2) show parent beams ion at 598 m/z[8] which represent the M-Wt of compound .Addition other packages shown in table (4) Figure (5) and Scheme (3).

Table 4. Important peaks of N-phenyl-2-(triiodo-λ^4-selaneyl)acetamide (2).

Molecular formula	m/z	Molecular formula	m/z
$C_8H_8I_3NOSe$	598	C_8H_8NO	135
CH_2I_3Se	474	I_3Se	460
$C_8H_8I_2NOSe$	467	I_2Se	343
C_8H_8NOSe	215	CH_2NO	43
$C_2H_3Br_3NOSe$	379	C_6H_6N	92
$C_2H_2Br_3OSe$	259		
C_6H_5	77		
C_7H_6N	104		

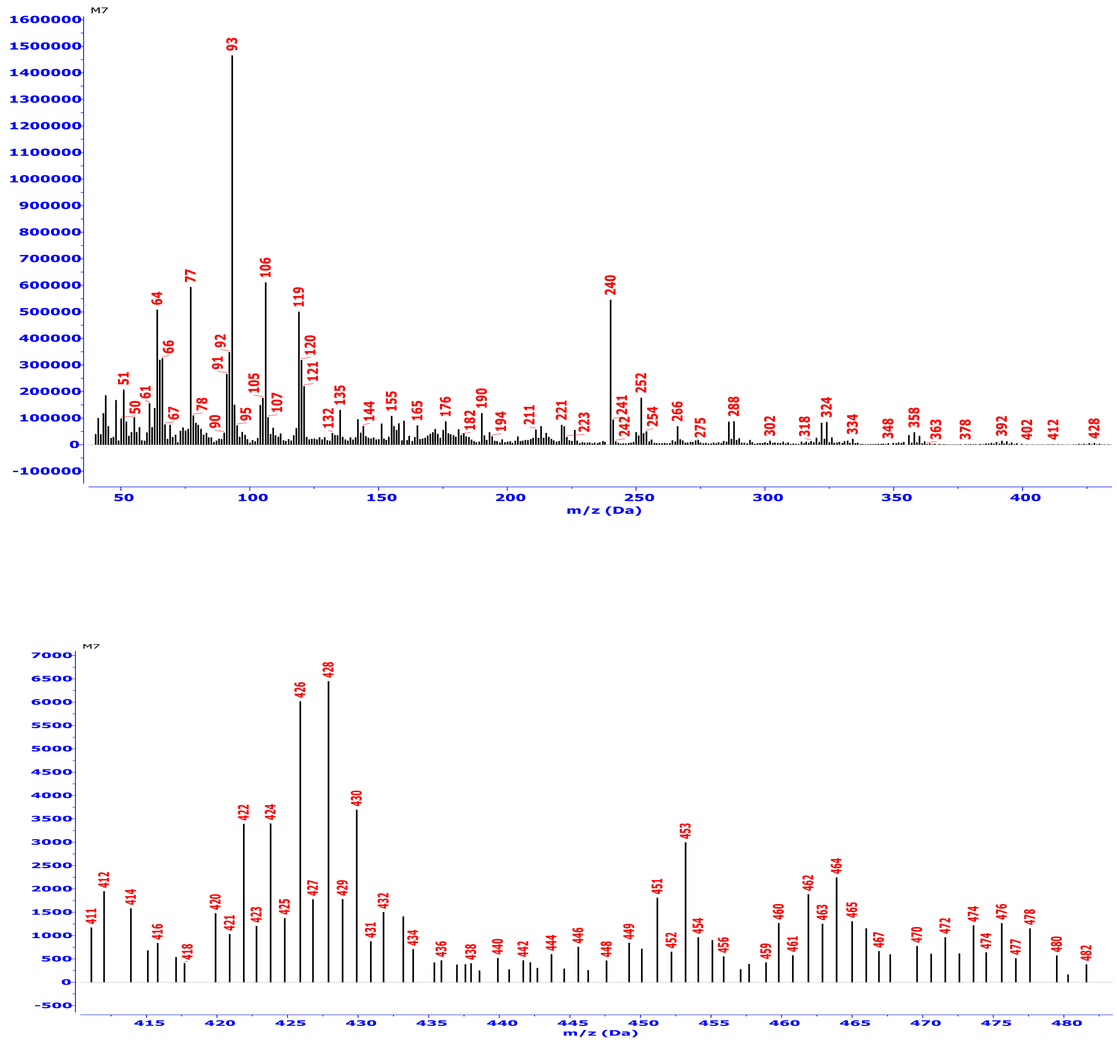
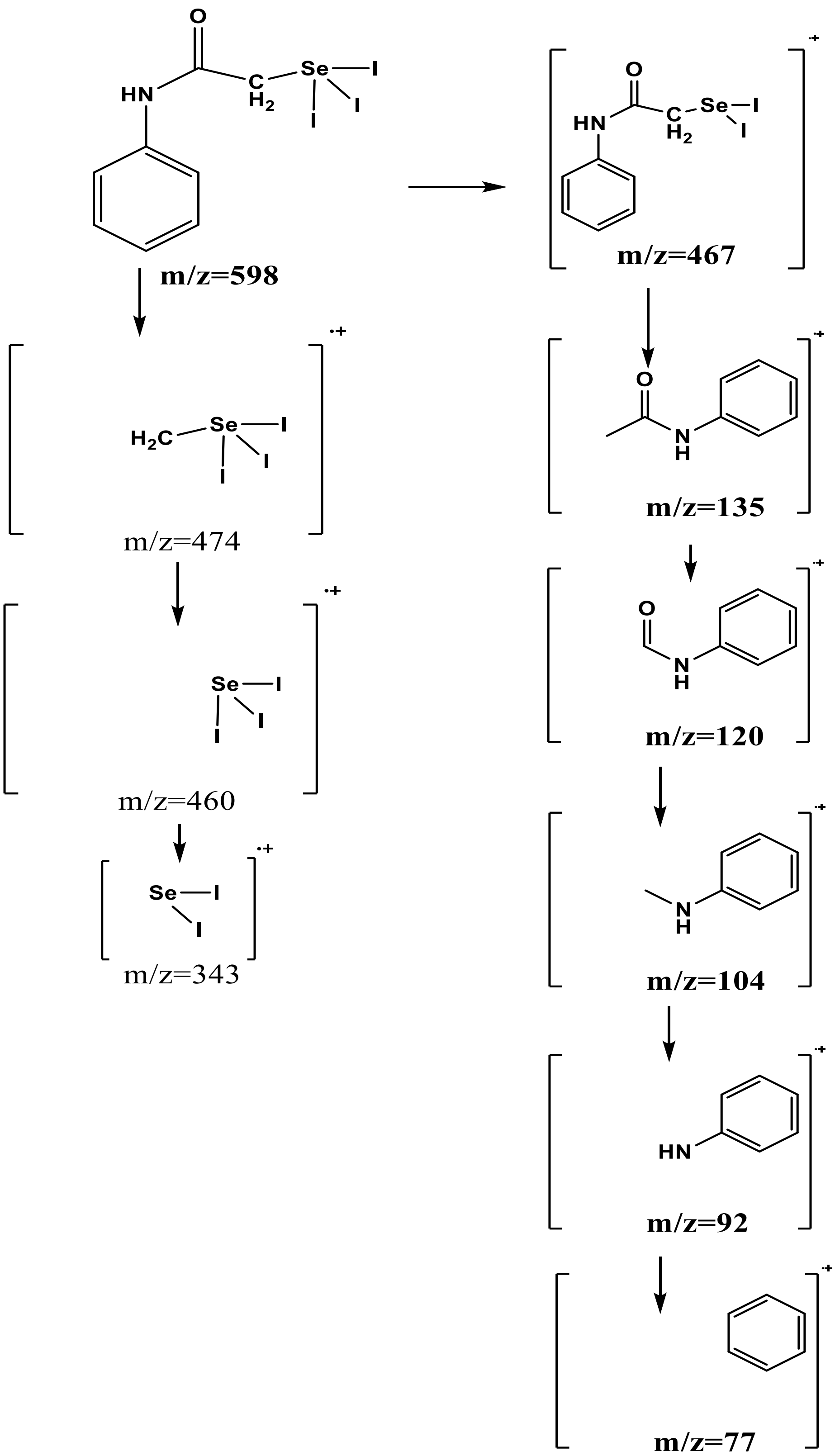


Figure 5. Mass-spectra of N-phenyl-2-(triiodo-λ[4]-selaneyl)acetamide (2).

Mass-spectrum of N-phenyl-2-(trichloro-λ[4]-selaneyl)acetamide (3) show parent beams ion at 320 m/z[9][10][8] , representM-Wt of compound .Aadditionother packsshown in table (5) Figure (6) and Scheme (4).

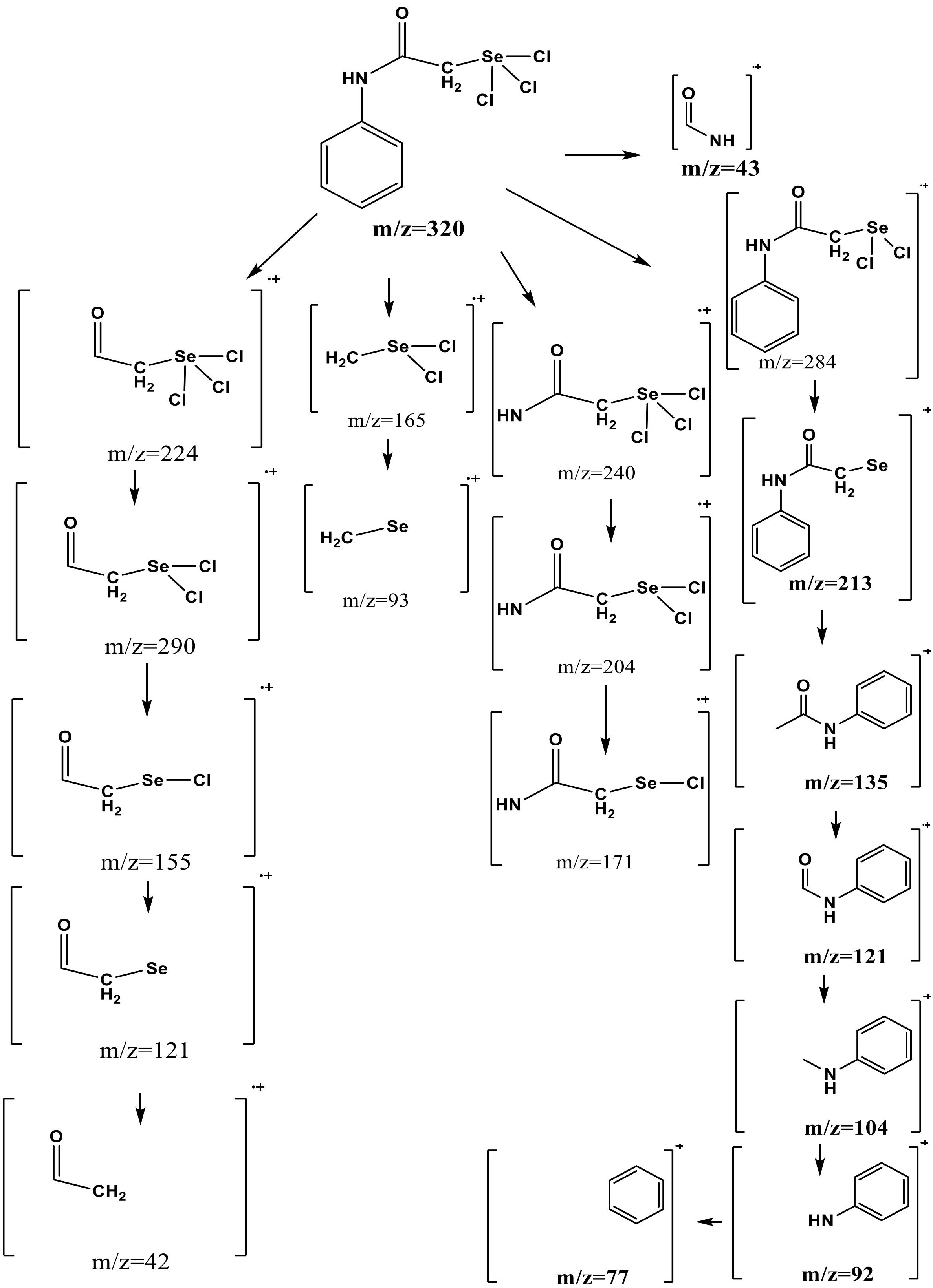


Scheme 3. Mechanism of fragmentation of N-phenyl-2-(triiodo-λ^4-selaneyl)acetamide (2)

Table 5. Important peaks of N-phenyl-2-(trichloro-λ^4-selaneyl)acetamide (3).

Molecular formula	m/z	Molecular formula	m/z
$C_8H_8Cl_3NOSe$	320	$C_2H_3Cl_2NOSe$	204
$C_8H_8Cl_2NOSe$	284	CH_2Br_3Te	382
C_8H_8NOSe	213	C_2H_2OTe	121
C_8H_8NO	135	C_2H_2ClOSe	155
C_7H_6NO	121	$C_2H_3ClNOSe$	171
C_7H_6N	104	CH_2NO	43
C_6H_5	77	$C_2H_2Cl_3OSe$	224
C_6H_6N	92	CNSe	93
CH_2Cl_2Se	165	CH_2BrTe	222
$C_2H_2Cl_2OSe$	190	C_2H_2O	42
$C_2H_3Cl_3NOSe$	240		171

Scheme 4. Mechanism of fragmentation of N-phenyl-2-(trichloro-λ^4-selaneyl)acetamide (3).


m/z=320
m/z=43
m/z=284
m/z=224
m/z=165
m/z=240
m/z=213
m/z=290
m/z=93
m/z=204
m/z=135
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m/z=171
m/z=121
m/z=121
m/z=104
m/z=42
m/z=77
m/z=92

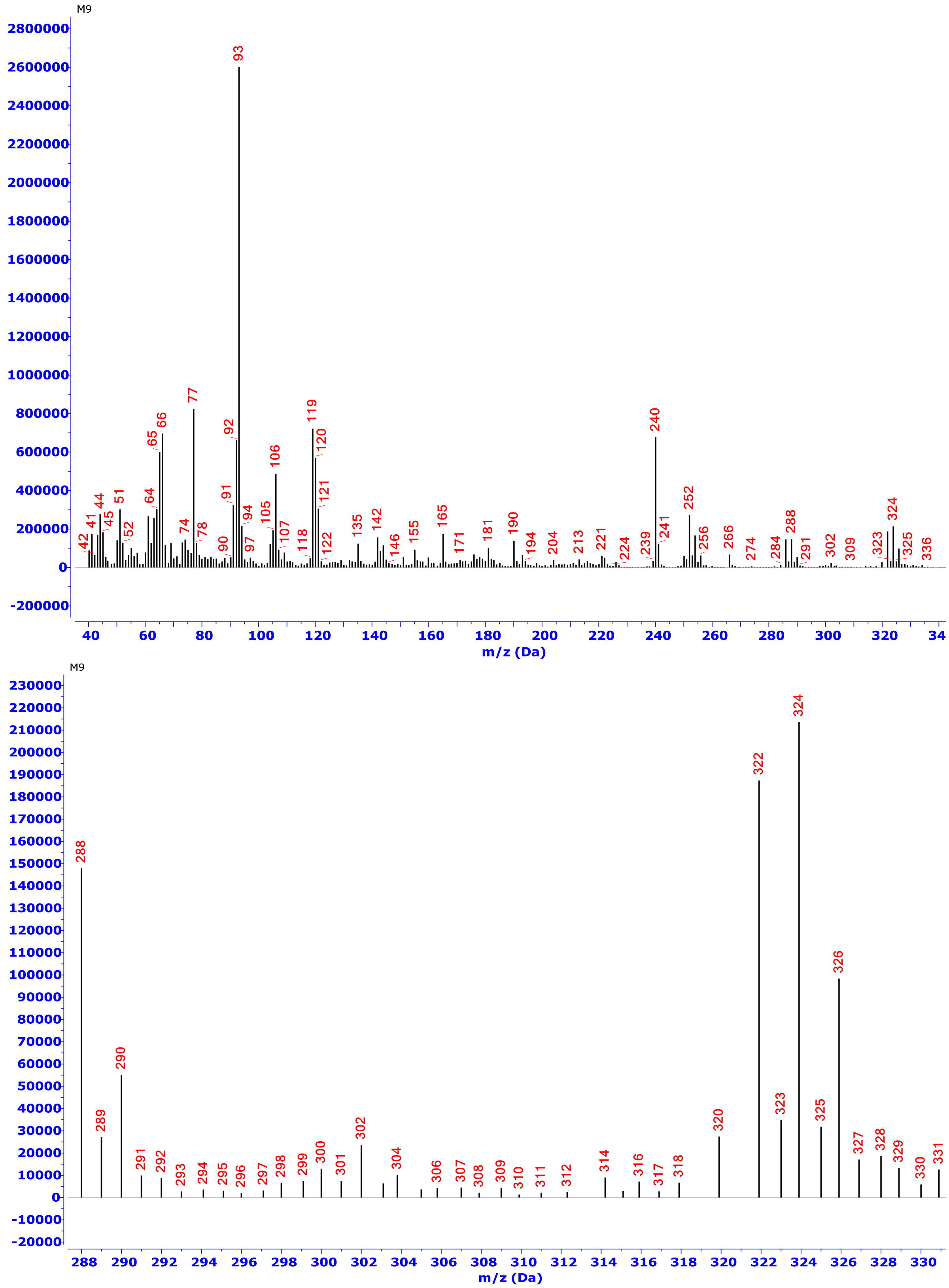


Figure 6. Mass-spectra of N-phenyl-2-(trichloro-λ^4-selaneyl)acetamide(3)

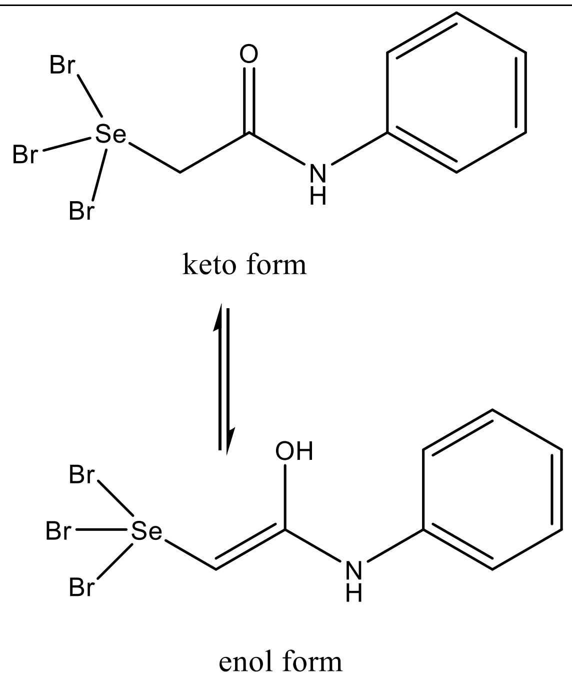
Nuclear Magnetic Resonance spectra 1HNMR

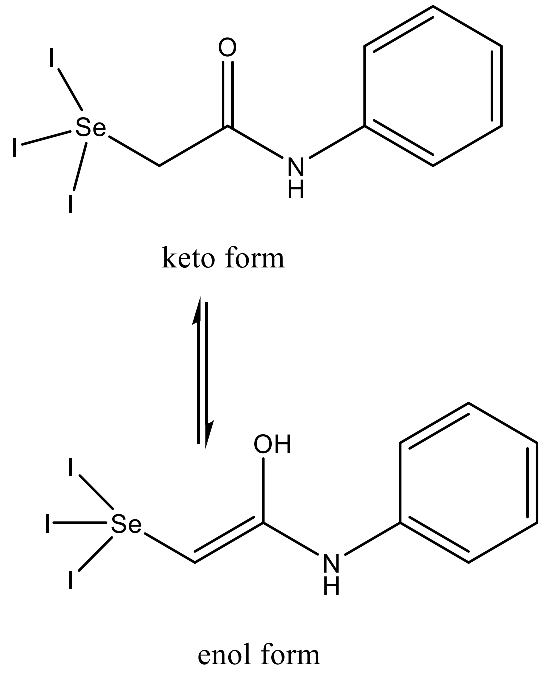
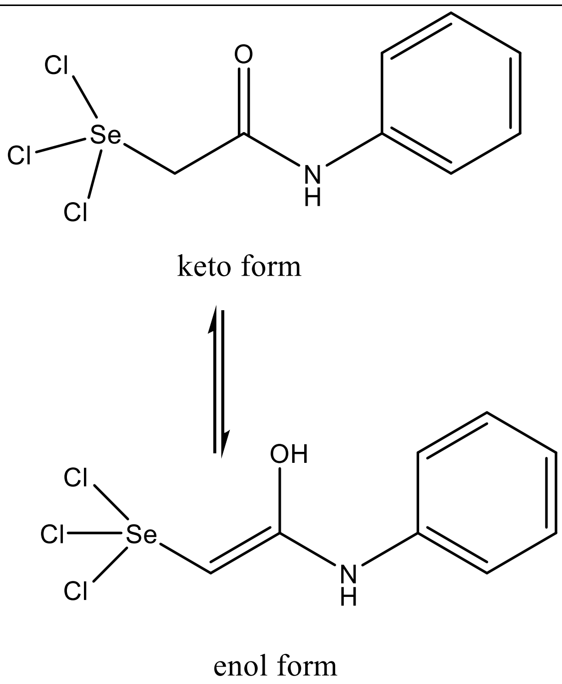
1HNMR-spectrum of N-phenyl-2-(tribromo-λ4-selaneyl)acetamide (1) showed 3-single beams at 3.9 ppmrefer to aliph-H (keto-form) and 4.6ppm toaliph-H (enol-form) , 10.96ppm to N-H proton . The H of Ar- region appeare as multiple beam withadisplacement of 6.87-7.85 ppm with complement five-H , shown in Figure (7) , (6) and Table (6)[6][8] .

1HNMR-spectrum of N-phenyl-2-(triiodo-λ4-selaneyl)acetamide (2) show single-beams at 2.98 ppmrefer to aliph-H (keto-form) , 3.45ppm toaliph-H (enol-form) , 10.03ppm to N-H proton and 10.82 ppm to O-H proton (keto-form) . protons of Ar- region appearemultiple beam within adisplacement of 6.99-8.28 ppm with complement five protons, shown in Figure (8) and Table (6)[6][8] .

1HNMR-spectrum of N-phenyl-2-(trichloro-λ4-selaneyl)acetamide (3) show single beams at 3.92 ppmrefer to aliph-H (keto-form) , 4.43ppm toaliph-H (enol-form) , 10.07ppm to N-H proton and 10.83 ppm to O-H proton (keto-form) . H of ar-region appearemultiple beam within displacement of 7.03-7.88 ppm with complement five-H , shown in Figure (9) and Table (6)[6][8] .

Table 6.HNMR-spectra of N-phenyl-2-(tribromo- λ^4-selaneyl) acetamide (1) , N-phenyl-2-(triiodo-λ^4-selaneyl) acetamide(2) and N-phenyl-2-(trichloro-λ^4-selaneyl) acetamide(3)

Compound	Structure	H-NMR
1		6.87-7.85 (5H,m) Ar-proton , 3.9 (2H,s) Aliph- proton(keto-form) , 4.6(1H,s) Aliph-proton(enol-form) , 10.96(1H,s) N-H proton .

2		6.99-8.28 (5H,m) ar-proton , 2.98 (2H,s) aliph- proton(keto-form) , 3.45(1H,s) Aliph-proton(enol-form) , 10.03(1H,s) NH proton , 10.82 (1H,s) O-H proton (keto-form)
3		7.03-7.88 (5H,m) ar-proton , 3.92 (2H,s) aliph- proton(keto-form) , 4.43(1H,s) aliph-proton(enol-form) , 10.07(1H,s) N-H proton , 10.83 (1H,s) O-H proton (keto-form)

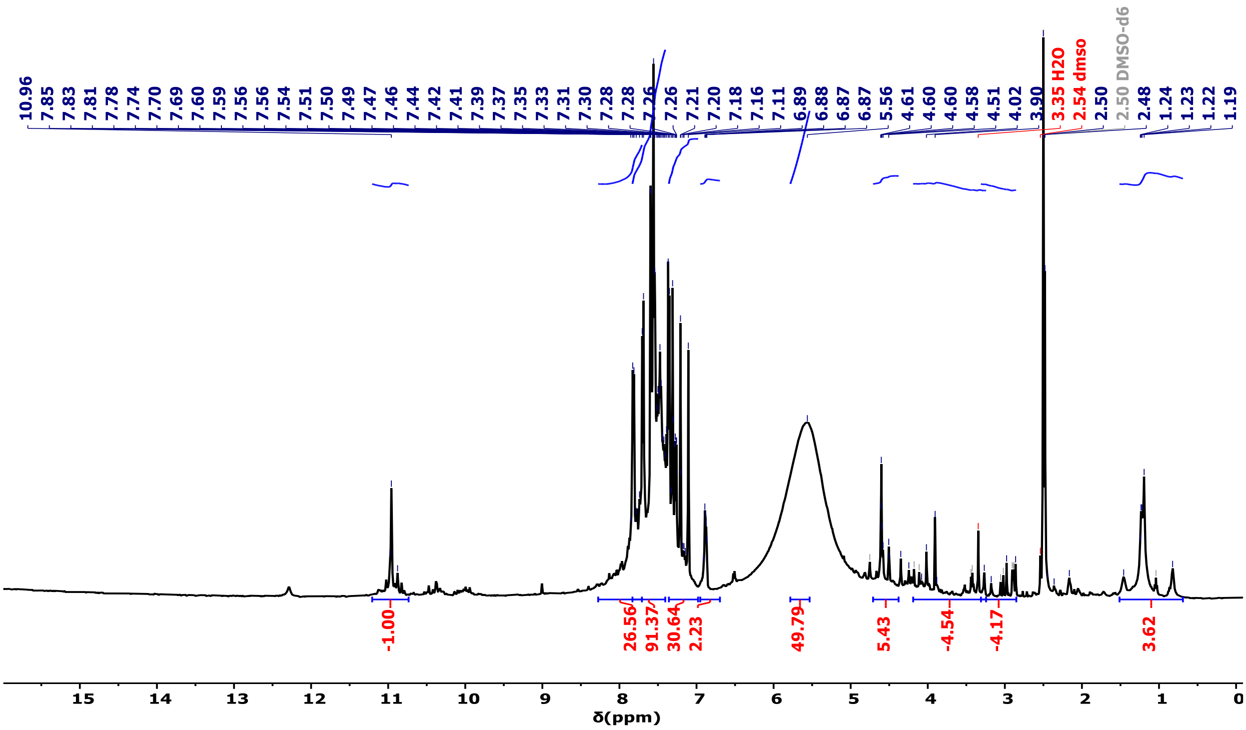


Figure 7.1H-NMR-spectra of N-phenyl-2-(tribromo-λ^4-selaneyl) acetamide (1)

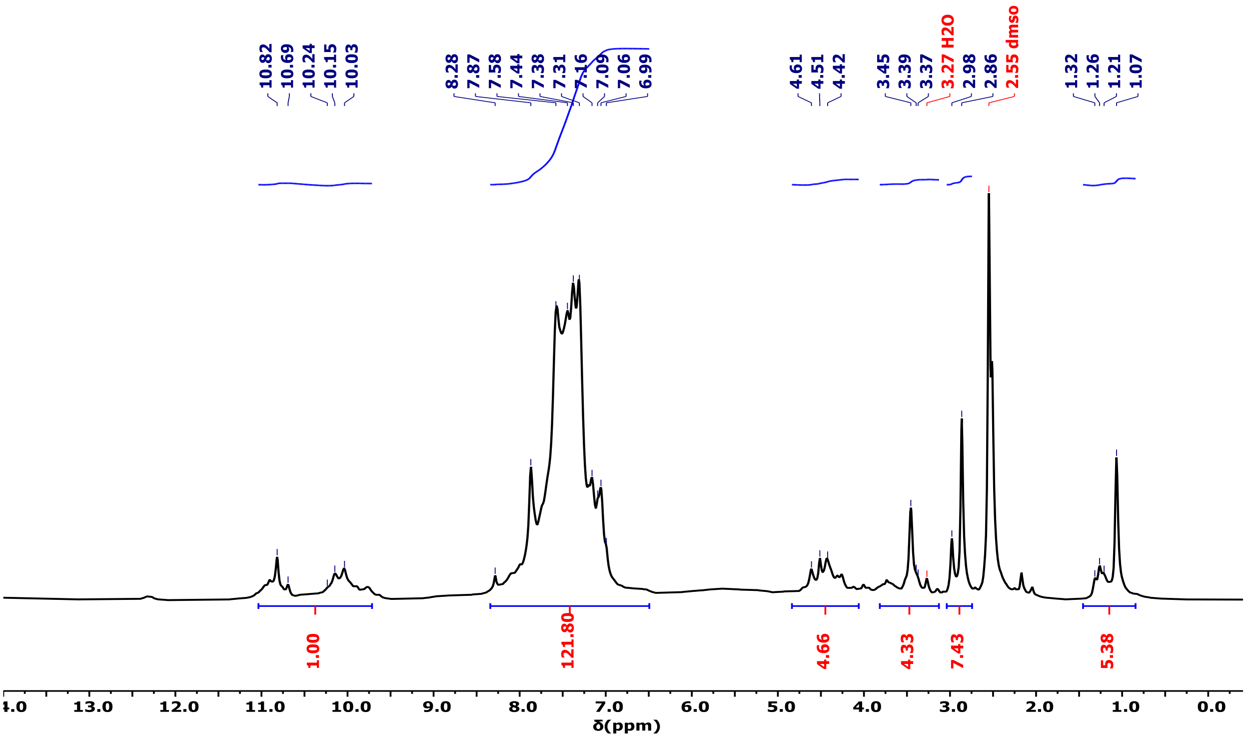


Figure 8.1H-NMR-spectra of N-phenyl-2-(triiodo-λ^4-selaneyl) acetamide (2).

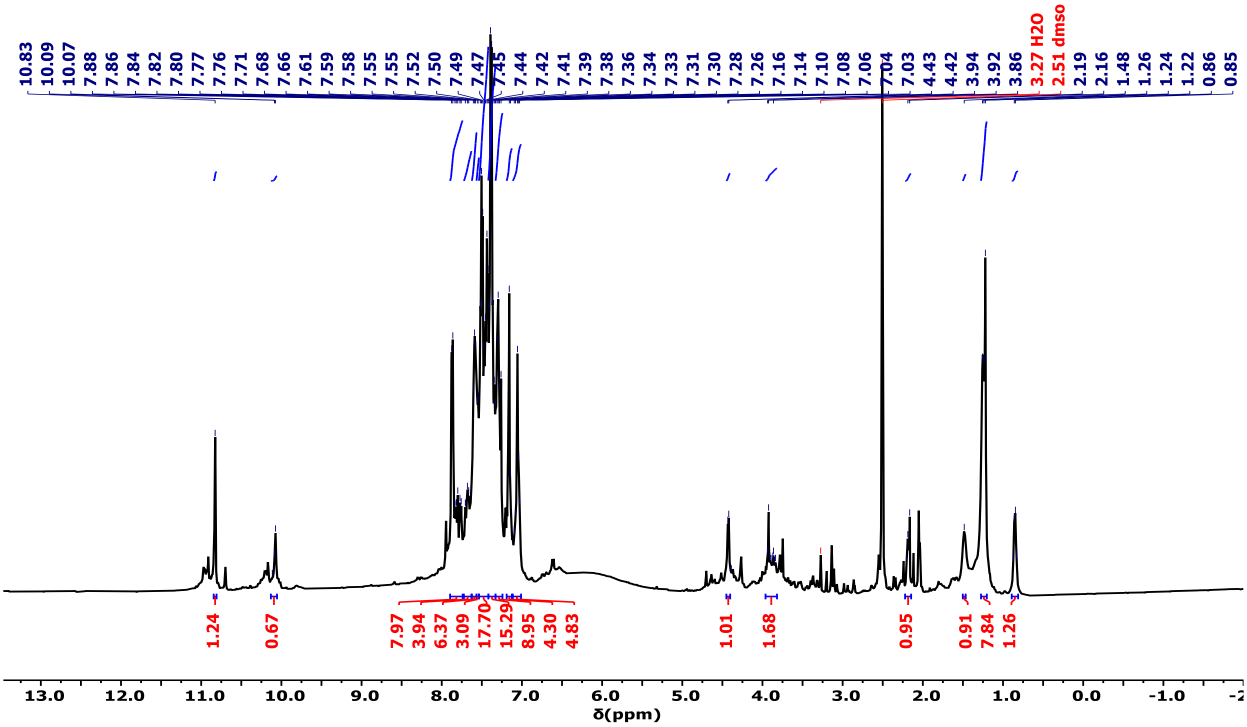


Figure 9.1HNMR-spectra of N-phenyl-2-(trichloro-λ4-selaneyl) acetamide (3)

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