Synthesis and structure elucidation of 2, 6-dimethyl-3, 5-diphenyl-2H-1, 4-selenazine using modern sophisticated Analytical techniques

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***Abstract*: In the present investigation of 2, 6-dimethyl-3, 5-diphenyl-2H-1, 4-selenazine involving one stage in which attacking of Selenium into carbonyl group followed by condensation reaction with amine and removal of hydroxyl group from desired Selenium compound. This Selenium compound is further confirmed by ^{1}H NMR, ^{13}C NMR, ^{77}Se NMR, Mass and IR.**

***Keywords*: Organic ligand containing N'N-Diethyl selenium compound, IR, ^{1}H NMR, ^{13}C NMR, ^{77}Se NMR and Mass spectrum.**

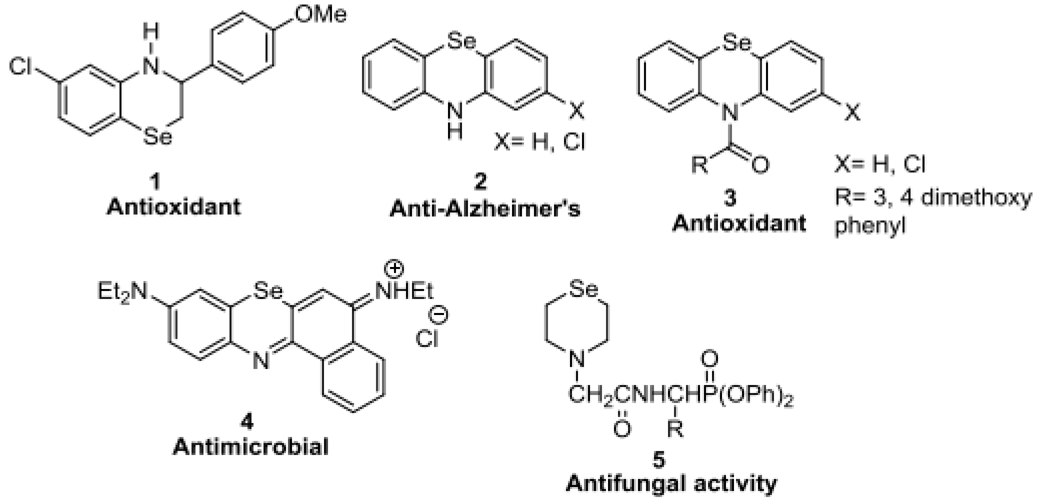
1. Introduction

Heterocycles containing selenium atoms occupy a special place among organoselenium compounds. Selenium-containing heterocycles represent an interesting class of compounds in the field of medicinal chemistry as well as in materials sciences. Based on the advantages related to the presence of selenium in the heterocyclic ring and its biological importance, The selenium-containing scaffolds like ebselen, selenadiazoles, selenochromenes, selenium-embedded polysaccharide-protein complexes and benzoselenophene fused imidazopyridines have received much attention for their unique pharmacological properties.

Drug Innovators synthesise many compounds over the process of drug development to identify imperfections in a pharmaceutical compound. The structure and pharmacological behaviour of these compounds can be studied with appropriate analytical technology. 1, 4-Selenazine is a class of selenium containing heterocycles that constitutes an important intermediate in organic synthesis and also exhibit potential for various biological properties such as antifungal activity, antimicrobial photosensitizer, antioxidant and anti-Alzheimer's

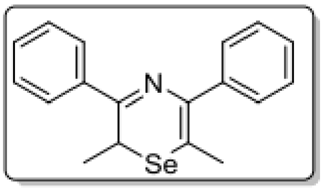
Figure 1.1 represents some of the biologically active molecules containing the 1,4-selenazine moiety. 1,4-selenazine is also used as a chromophore for photosensitization of ruthenium nitrosyls complexes. In spite of their importance, however, relatively few studies have been reported on the synthesis of 1, 4- selenazine.

Figure 1.1



All synthetic as well as analysis details are summarized below.

2. Structure of 2, 6-dimethyl-3, 5-diphenyl-2H-1, 4-selenazine



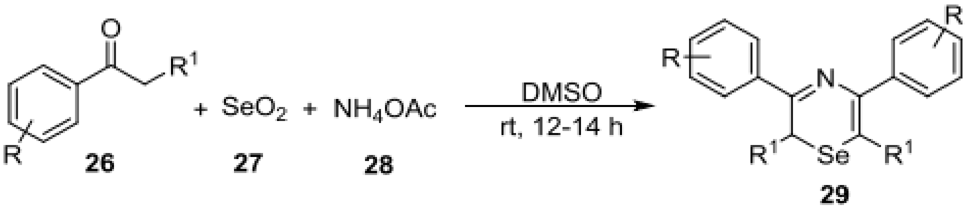
Structure of **2, 6-dimethyl-3, 5-diphenyl-2H-1, 4-selenazine**
[a ,1 4-Selenazine compound]

3. Experiment

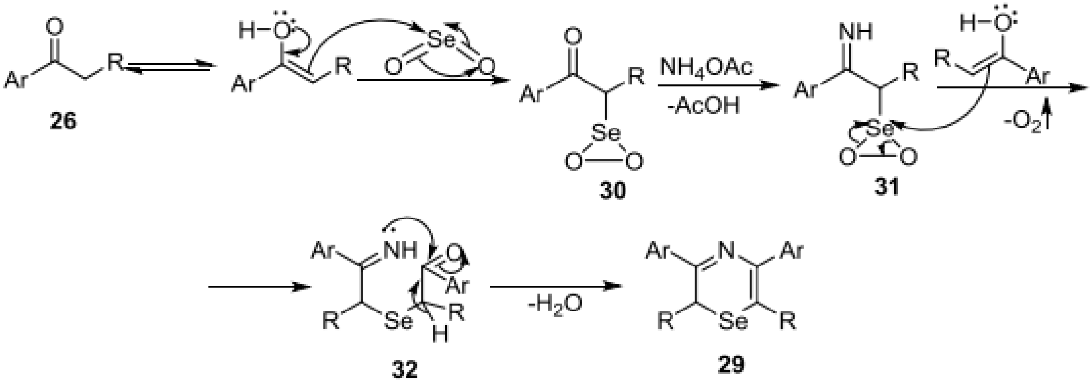
As a part of our effort towards the synthetic application of selenium dioxide, in our ongoing study on the synthetic utility of selenium dioxide for C-Se bond formation, we have developed a method for the synthesis of 3,5-diphenyl-2H-1,4- selenazine and its derivatives by three-component condensation of aryl alkyl ketones (26), selenium dioxide (27) and ammonium acetate (28) at room temperature in DMSO as a solvent
Below is the common route of synthesis of selenium compound.

3.1 General Route of Synthesis of Selenium compound

Scheme: General Synthesis route of selenium compound

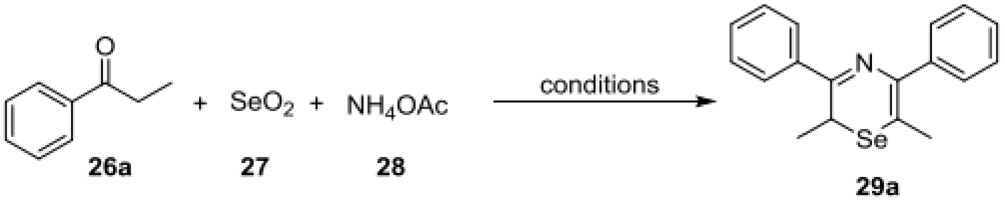


3.2 Plausible mechanism of synthesized Selenium compound

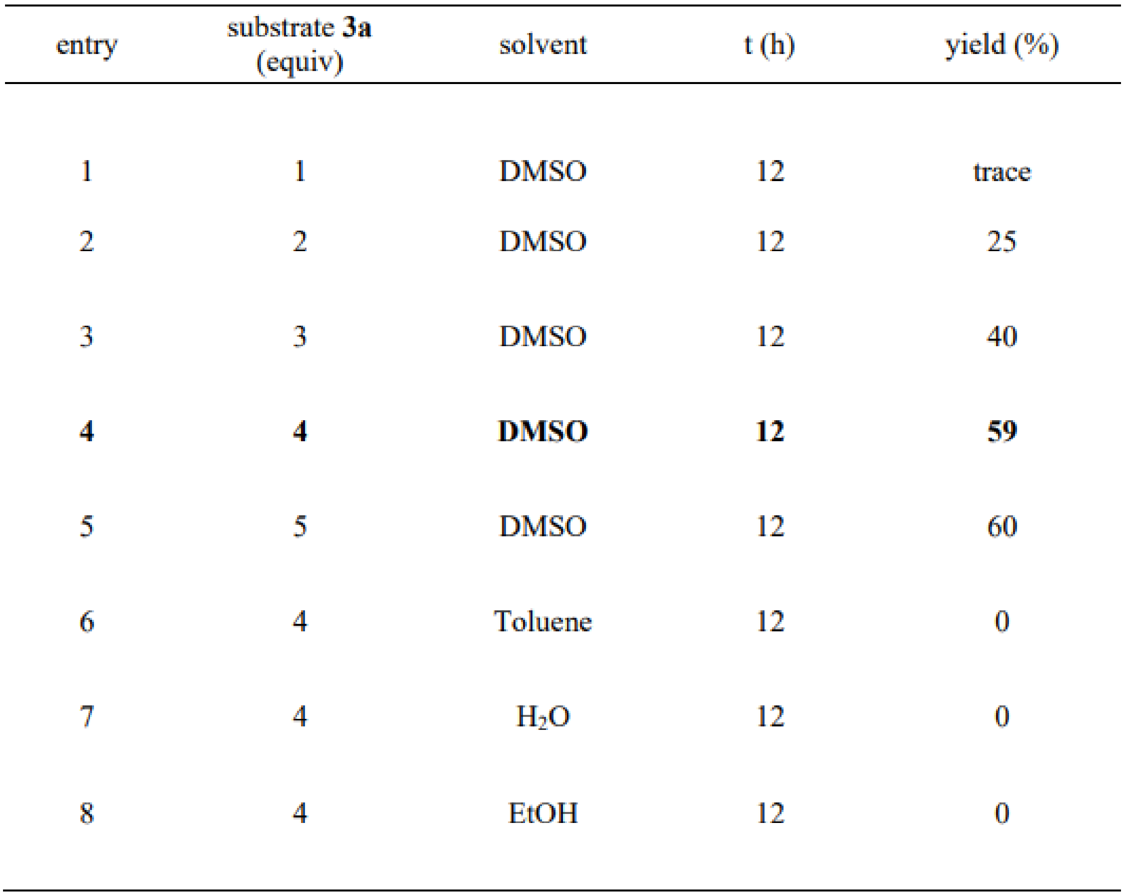


Above Route of synthesis is general scheme for synthesis of selenium compound, here R is variable. If we change R with other functionality, final product will also change. This scheme is very useful for synthesis of many different selenium derivatives.

3.3 Optimization of the reaction conditions



To initiate our investigation, we choose the readily available propiophenone (26a) as a model substrate for the proposed reaction cascade.
Our initial effort in reacting propiophenone (26a) (1.0 mmol, 1 equiv), SeO2 (27) (0.5 mmol, 0.5 equiv) and NH4OAc (28) (1.0 mmol, 1.0 equiv) in DMSO (1 mL) at room temperature for 12 h, the corresponding product 29a was obtained only in trace amount. Different experiments performed for reaction conditions are summarized in table **3.3.1**



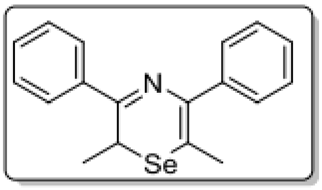
entry	substrate **3a** (equiv)	solvent	t (h)	yield (%)
1	1	DMSO	12	trace
2	2	DMSO	12	25
3	3	DMSO	12	40
4	**4**	**DMSO**	**12**	**59**
5	5	DMSO	12	60
6	4	Toluene	12	0
7	4	H_2O	12	0
8	4	EtOH	12	0

[a]Reaction conditions: ketone (26) (1.0 mmol), SeO2 (0.5mmol), solvent (1 mL), room temperature.

After optimizing reaction, we have synthesized specific selenium compound (**2, 6-dimethyl-3, 5-diphenyl-2H-1, 4-selenazine**).

After synthesis, we have analysed the compound using various analytical techniques like ^{1}H NMR, ^{13}C NMR, ^{77}Se NMR, Mass spectra and IR. Below is the summarized study data of **2, 6-dimethyl-3, 5-diphenyl-2H-1, 4-selenazine.**

3.4 Analysis data of 2, 6-dimethyl-3, 5-diphenyl-2H-1, 4-selenazine



Description/Appearance: Oil
Yield: 59%
IR (KBr): 3057, 3024, 2953, 2905, 2849, 1563, 1441, 1330, 765, 698 cm^{-1}
^{1}H NMR (400 MHz, $CDCl_3$) : δ 7.96-7.94 (m, 2H), 7.58 (d, J = 7.2 Hz, 2H), 7.39-7.32 (m, 5H), 7.24 (t, J = 7.6, 7.2 Hz, 1H), 4.14 (q, J = 7.2, 6.8 Hz, 1H), 2.24 (s, 3H), 1.51 (d, J = 6.8 Hz, 3H) ppm.
^{13}C NMR (100 MHz, $CDCl_3$): δ 150.6, 142.2, 138.4, 137.0, 129.9, 128.8, 128.5, 128.0, 127.3, 127.0, 110.7, 25.6, 21.3, 16.6 ppm.
^{77}Se NMR (57.25 MHz, $CDCl_3$) : δ 276.219.
HRMS (ESI) calcd for $C_{18}H_{17}NSe$ 327.0526, found m/z 328.0596 $[M + H]^+$.

Figure 3.4.1: IR spectrum of 2,6-dimethyl-3,5-diphenyl-2H-1,4-selenazine (29a)

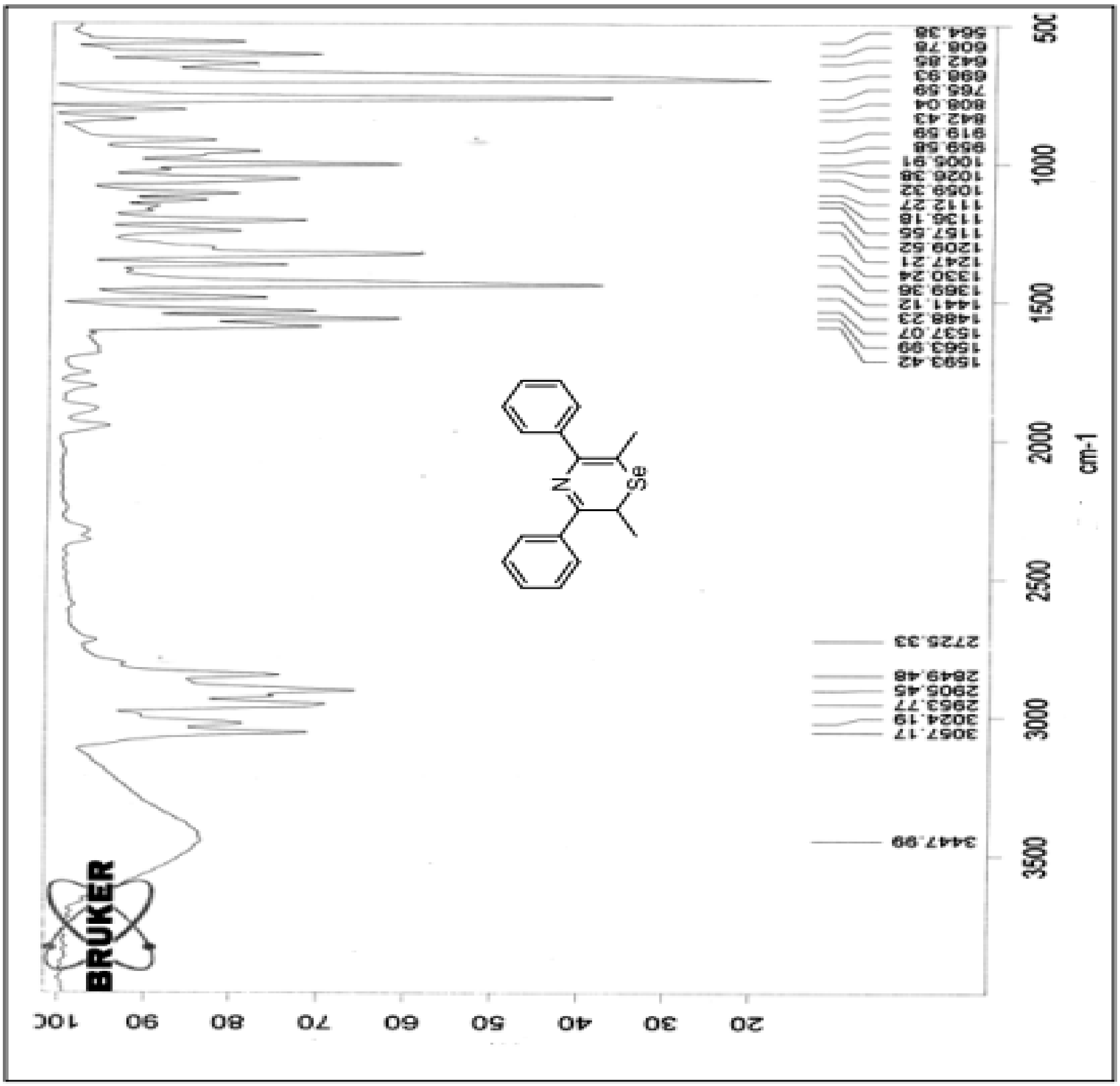


Figure 3.4.2 : 1H NMR ($CDCl_3$, 400 MHz) spectrum of 2,6-dimethyl-3,5-diphenyl-2H-1,4- selenazine (29a)

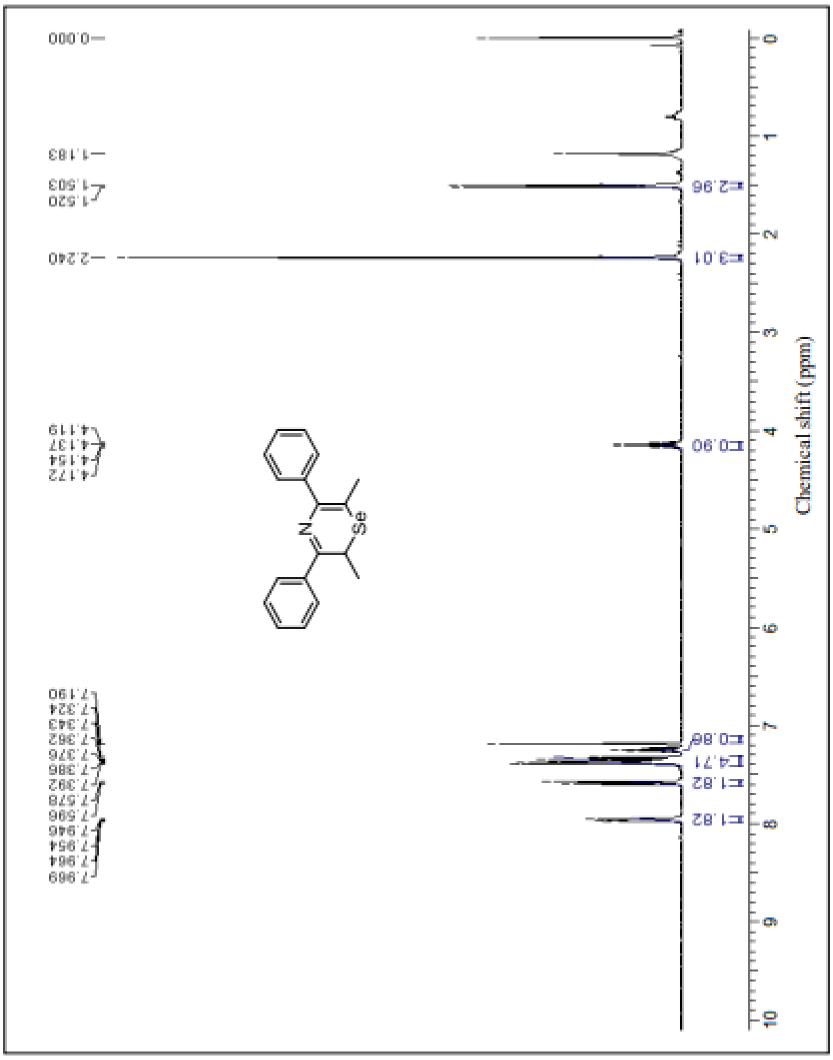


Figure 3.4.3: ^{13}C NMR (CDCl3, 100 MHz) spectrum of 2,6-dimethyl-3,5-diphenyl-2H1,4-selenazine (29a)

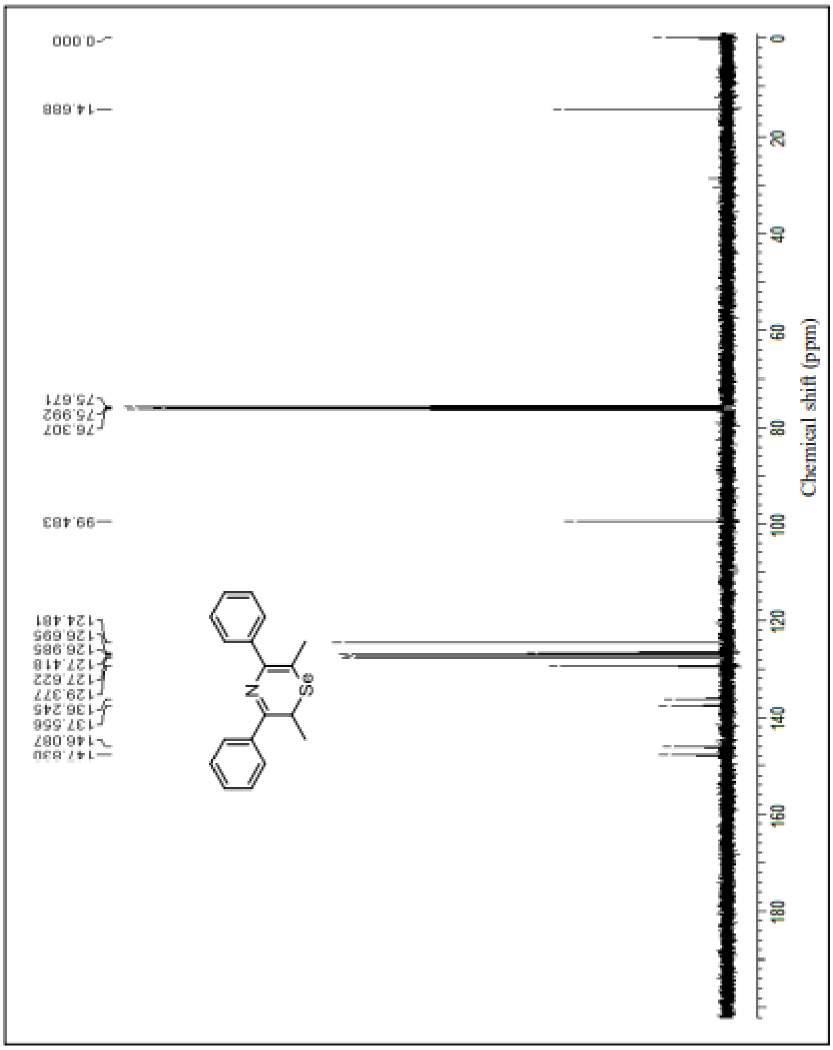


Figure 3.4.4: ^{77}Se NMR spectrum ($CDCl_3$, 57.25 MHz) of 2,6-dimethyl-3,5-diphenyl-2H1,4-selenazine (29a)

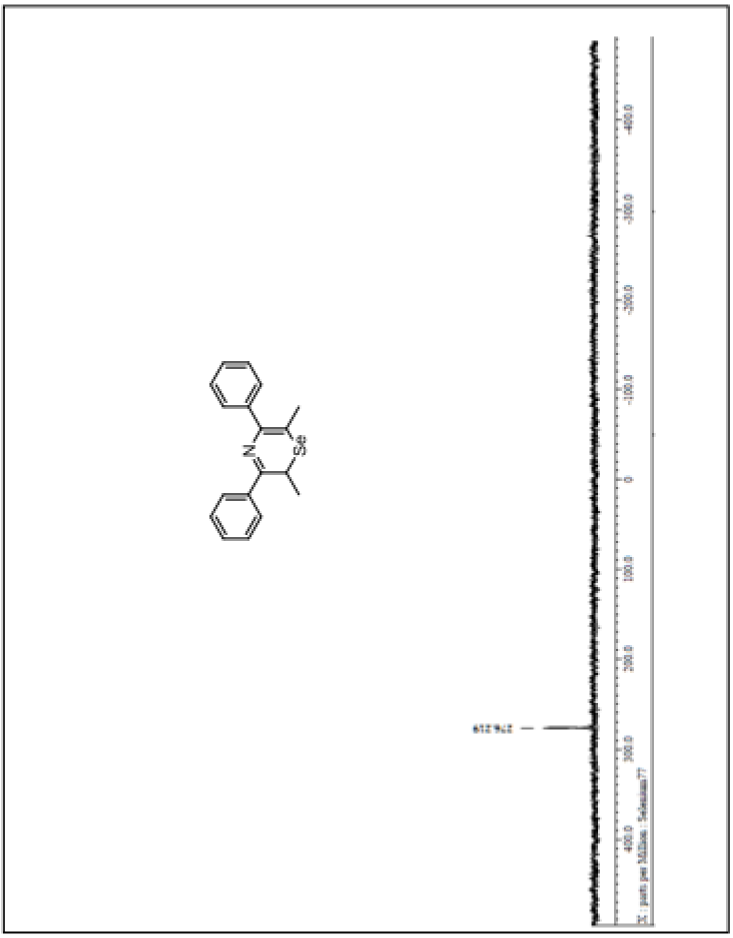
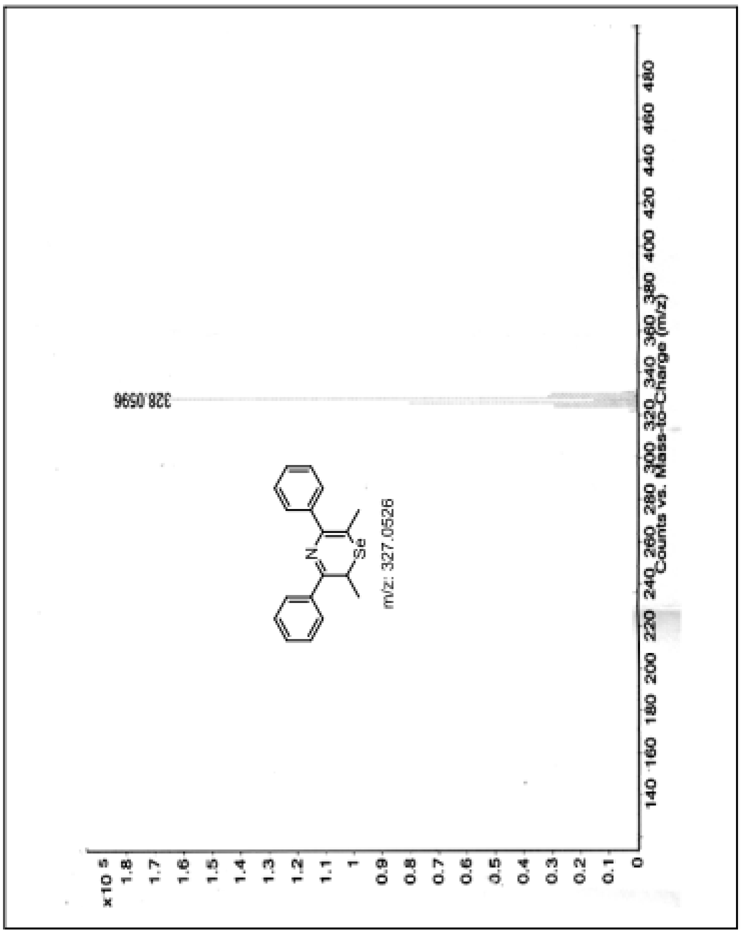


Figure 3.4.5: Mass spectrum of 2,6-dimethyl-3,5-diphenyl-2H-1,4-selenazine (29a)



Conclusion:

In continuation of our work on the synthetic usefulness of selenium dioxide, we shall have described a direct technique for the selenoamidation of aryl methyl ketones. The selenylating substance used in this procedure is readily accessible selenium dioxide. This approach has the advantage of not requiring a catalyst, acid, or base, and proceeding under moderate reaction conditions. To the best of our knowledge, this is the first approach for the synthesis of so yet unreported α-oxo-N-alkyl selenoamides. The current approach and the characteristics of the unreported compounds will be further researched and examined in the near future. We will have developed a methodology for the synthesis of novel selenium-containing heterocycles, namely 1, 4-selenazine, as part of our research into the creation of new techniques. The approach presents a succession of 1, 4-selenazine and its derivatives

After synthesized of Selenium compound, it is well characterised/ elucidation using modern analytical techniques (^{1}H NMR, ^{13}C NMR, $^{77\text{Se}}$ NMR, Mass spectra and IR).

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