Synthesis and structure elucidation of N, N-diethyl-2-(2-methoxyphenyl)-2- oxoethaneselenoamide using modern Analytical techniques

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Abstract

In the present investigation of synthesized* N,N-diethyl-2-(2-methoxyphenyl)-2-oxoethaneselenoamide *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 NMR, Mass, IR, UV and Elemental analysis.

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

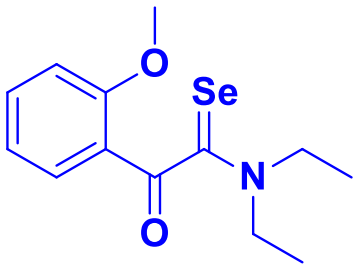
1. Introduction

Analysis of drugs in analytical chemistry makes it possible to separate chemical components obtained from both natural and artificial sources, estimate their concentrations, and quantify their quantities. Compounds of this kind often include one or more chemicals in addition to the other ingredients. The qualitative and quantitative analysis processes are the initial steps in the analytical chemistry process. When analysing a compound, quantitative analysis is being used to determine the total number of components present, whereas qualitative analysis is being used to estimate the number of samples that can be collected. As an example, the evaluation of a wide variety of substances or goods is crucial for the research of pharmaceuticals since it involves the analysis of the life of the substances or products. As seen by the flow of new medications into the market and the increase in their availability, drugs are in great demand. The newly created drugs are made up of either new types of current pharmaceuticals or modified versions of pharmaceuticals that are already in the market. These drugs are detailed in connection to pharmaceuticals that are now on the market as well as medications that are listed in the pharmacopoeia. In the drug development process, pharmacopoeia was utilised to report on the best therapeutic agents to remove from the market based on their efficacy. While a pharmaceutical compound is being developed, it is possible that certain pharmacopoeias will not contain its analytical profile of the medication. To this end, it is critical that the appropriate analytical techniques should be in place prior to beginning the development of innovative drugs.

With this selenium compound synthesis selective compound of selenium (N'N-Diethyl selenium compound) , we are going to synthesize selectively desired selenium compound and structure elucidation/analysis will be done by using modern analytical techniques with the graphical/spectroscopic representation.

All synthetic as well as analysis details are summarized;

1. Structure of N'N-Diethyl Selenium compound



Structure of N, N-diethyl-2-(2-methoxyphenyl)-2- oxoethaneselenoamide

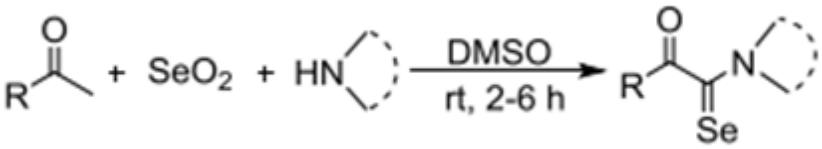
[a Selenium compound]

2. Experiment

As a part of our effort towards the synthetic application of selenium dioxide, in this chapter we report the synthesis of α-selenoamidation starting from aryl methyl ketones with secondary amines at room temperature without using any catalyst, acid or base

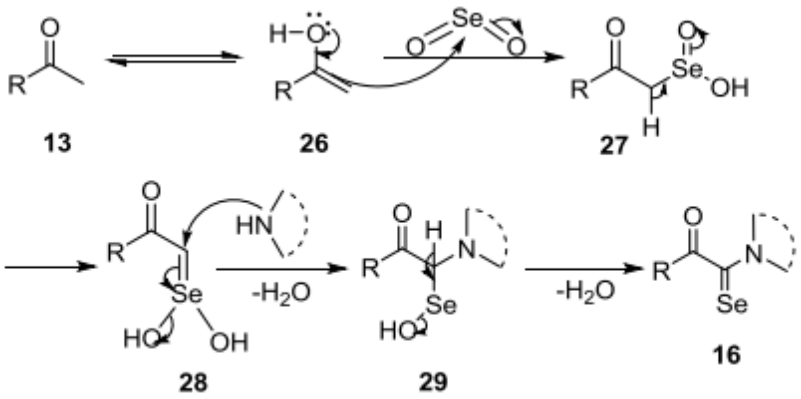
Below is the common route of synthesis of selenium compound.

2.1 General Route of Synthesis of Selenium compound



Scheme: General Synthesis route of selenium compound

2.2 Plausible mechanism of synthesized Selenium compound



Scheme: Plausible mechanism of Selenium compound

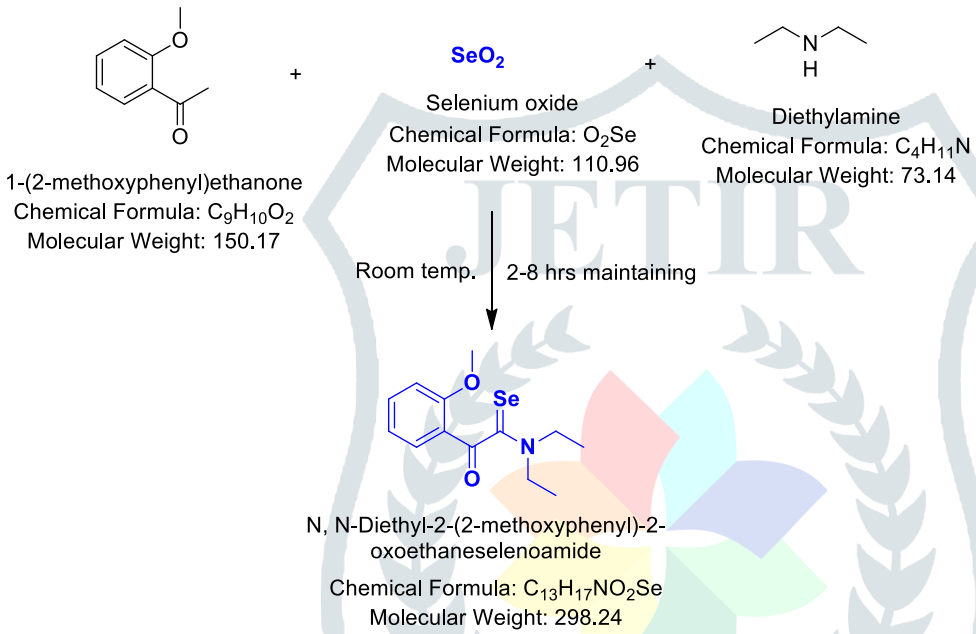
Above Route of synthesis is general scheme for synthesis of selenium compound, here R is variable. If we change R with other functionality, final product will also change. This scheme is very useful for synthesis of many different selenium derivatives.

For consideration of this general scheme we have synthesized specific selenium compound (N'N-Diethyl selenium compound).

After synthesis of N'N-Diethyl selenium compound, we have analyzed 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 N'N-Diethyl selenium compound

2.3 Route of synthesis of N'N-Diethyl selenium compound

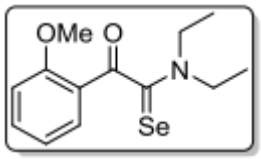


Synthesis scheme of N'N-Diethyl selenium compound

2.4 Synthesis procedure of N'N-Diethyl selenium compound

Initially, when 1-(2-Methoxyphenyl)ethanone-1-(2-methoxyphenyl)ethanone, (0.116 mL, 1.0 mmol, 1 equivalent) was treated with selenium dioxide, (110 mg, 1.0 mmol, 1 equivalent) and diethylamine, (0.107 mL, 1.0 mmol, 1 equivalent) at room temperature for 8H the reaction product N,N-Diethyl-2-(2-methoxyphenyl)-2-oxoethaneselenoamide was formed in 30% yield. Our efforts to optimize the reaction by varying the stoichiometry of the amine showed no improvement in the product yield (Table 5.1, entries 2-3). The optimized condition was achieved when the reaction was carried out using dimethyl sulfoxide as the solvent which resulted yield is 80% yield in 2 hr. Further attempts to improve the efficiency of the reaction by varying the amount of amine and using different solvents, provided no significant result.

2.5 Analysis data of N'N-Diethyl selenium compound



Description: Orange solid.

yield: 80%.

Melting point: 113˚C-115˚C

IR (KBr): 3076, 2982, 2940, 2838, 1637, 1597, 1537, 1437, 1383, 1297, 1281, 1112, 1015, 760 cm-^{1};

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 (dd, J = 2, 2 Hz, ^{1}H), 7.48-7.44 (m, ^{1}H), 7.03- 6.99 (m, ^{1}H), 6.88 (d, J = 8.4, ^{1}H), 4.02 (q, J = 6.8 Hz, 2H), 3.75 (s, 3H), 3.47 (q, J = 6.8 Hz, 2H) , 1.36 (t, J = 7.2 Hz, 3H), 1.24 (t, J = 6.8 Hz, 3H) ppm;

^{13}C NMR (100 MHz, $CDCl_3$) δ 201.4, 186.2, 157.7, 134.2, 130.7, 123.2, 120.2, 111.3, 54.5, 48.3, 46.3, 11.6, 9.9 ppm;

^{77}Se NMR (57.25 MHz, $CDCl_3$) δ 525.721;

MS (ES+) : Molecular formula $C_{13}H_{17}NO_2Se$, molecular weight- 299.04, found m/z 300.2 $[M + H]^+$.

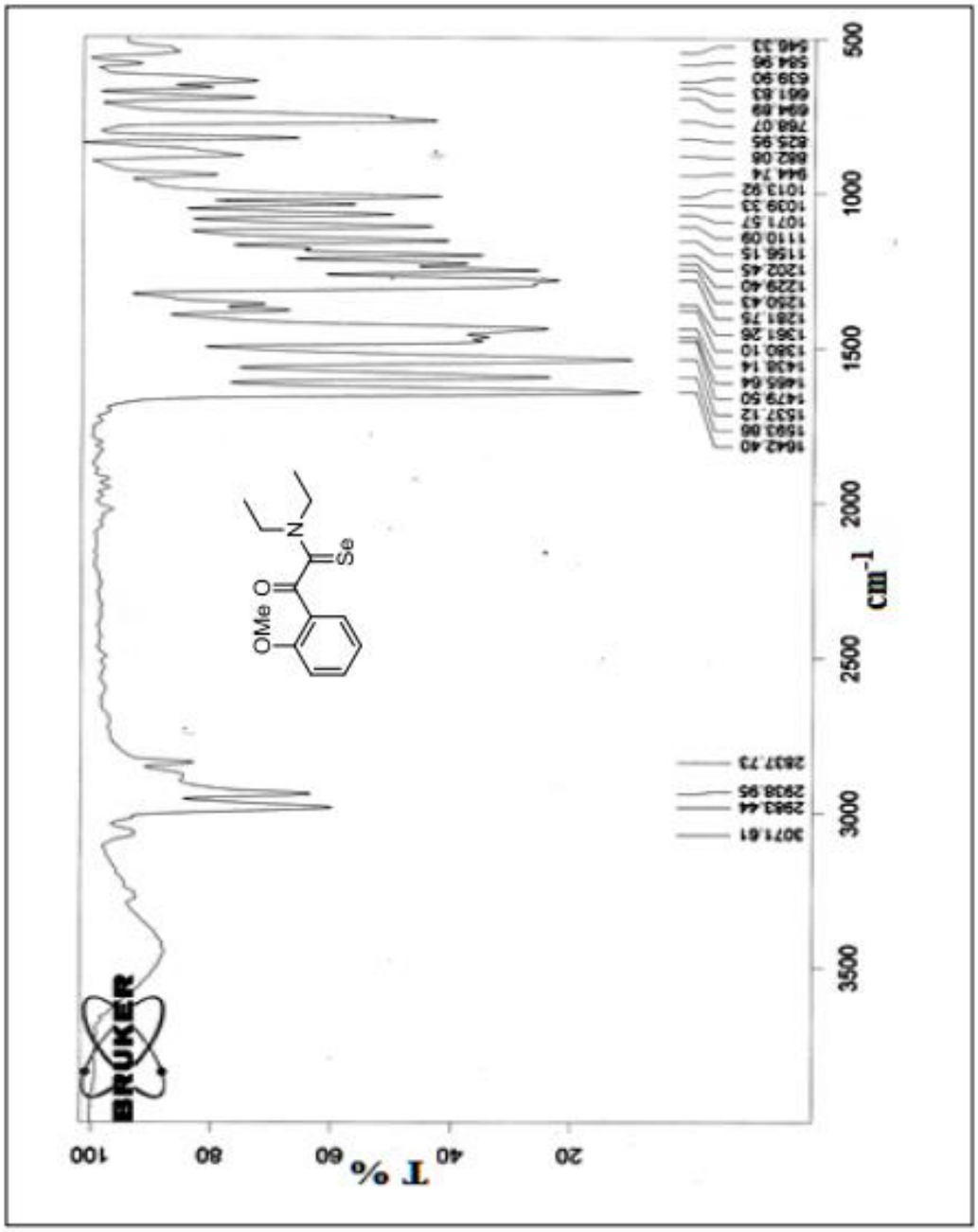


Figure 2.5.1 IR spectrum of N,N-diethyl-2-(2-methoxyphenyl)-2 oxoethaneselenoamide

IR (KBr): 3076, 2982, 2940, 2838, 1637, 1597, 1537, 1437, 1383, 1297, 1281, 1112, 1015, 760 cm-^{1};

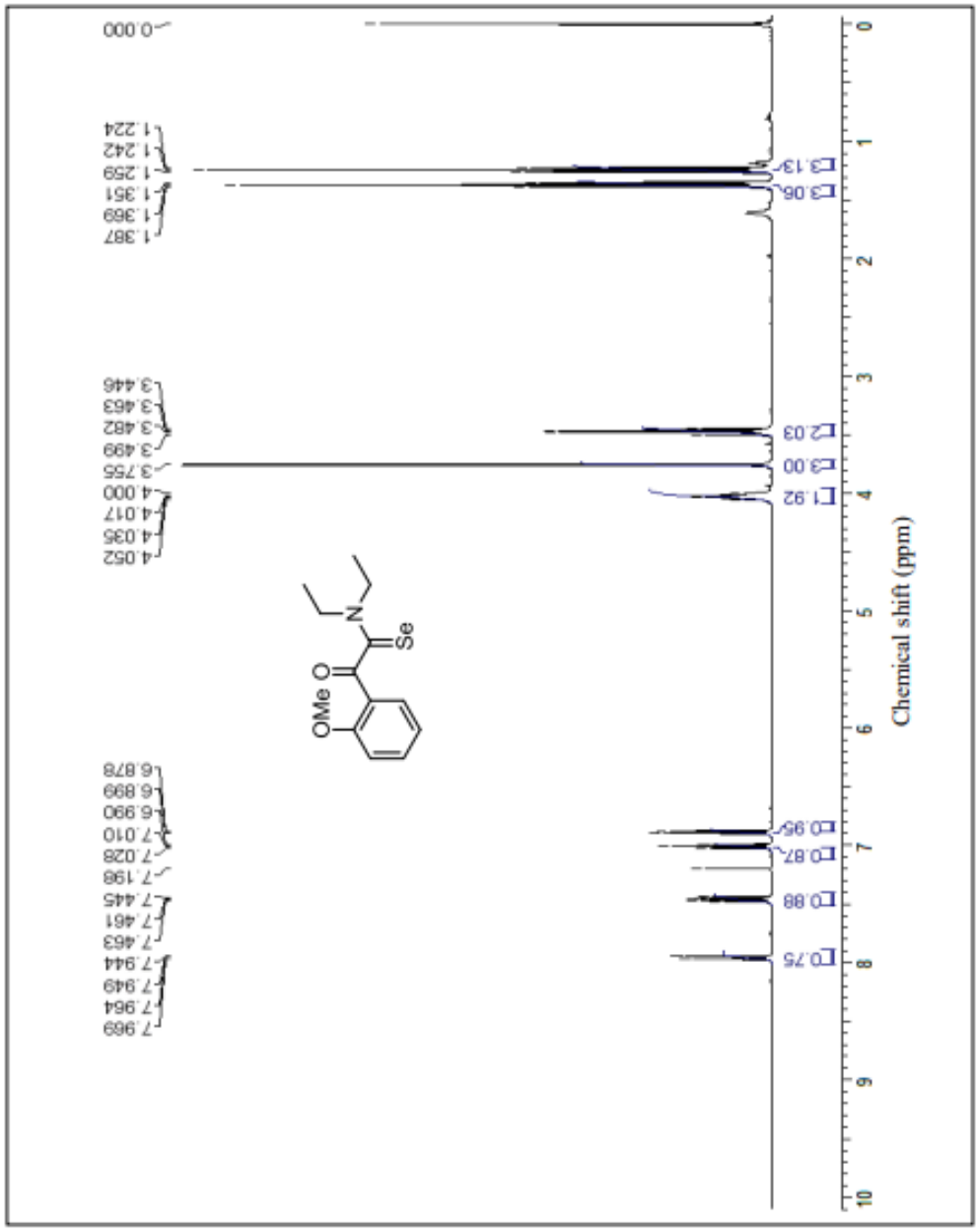


Figure 2.5.2 ^{1}H NMR (CDCl3, 400 MHz) spectrum of N,N-diethyl-2-(2- methoxyphenyl)-2-oxoethaneselenoamide

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 (dd, J = 2, 2 Hz, ^{1}H), 7.48-7.44 (m, ^{1}H), 7.03- 6.99 (m, ^{1}H), 6.88 (d, J = 8.4, ^{1}H), 4.02 (q, J = 6.8 Hz, 2H), 3.75 (s, 3H), 3.47 (q, J = 6.8 Hz, 2H) , 1.36 (t, J = 7.2 Hz, 3H), 1.24 (t, J = 6.8 Hz, 3H) ppm;

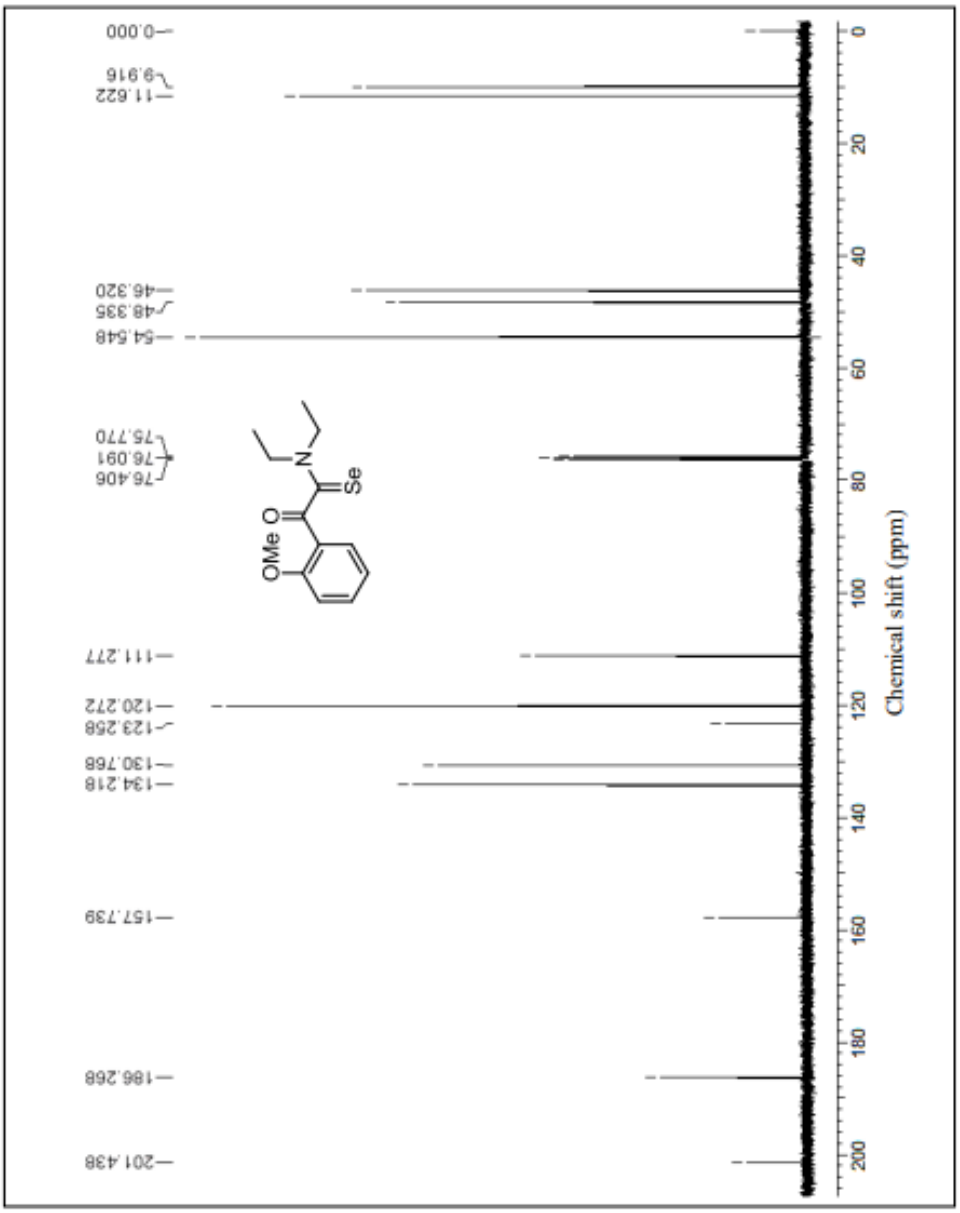


Figure 2.5.3 ^{13}C NMR (CDCl3, 100 MHz) spectrum of N,N-diethyl-2-(2- methoxyphenyl)-2-oxoethaneselenoamide

^{13}C NMR (100 MHz, $CDCl_3$) δ 201.4, 186.2, 157.7, 134.2, 130.7, 123.2, 120.2, 111.3, 54.5, 48.3, 46.3, 11.6, 9.9 ppm;

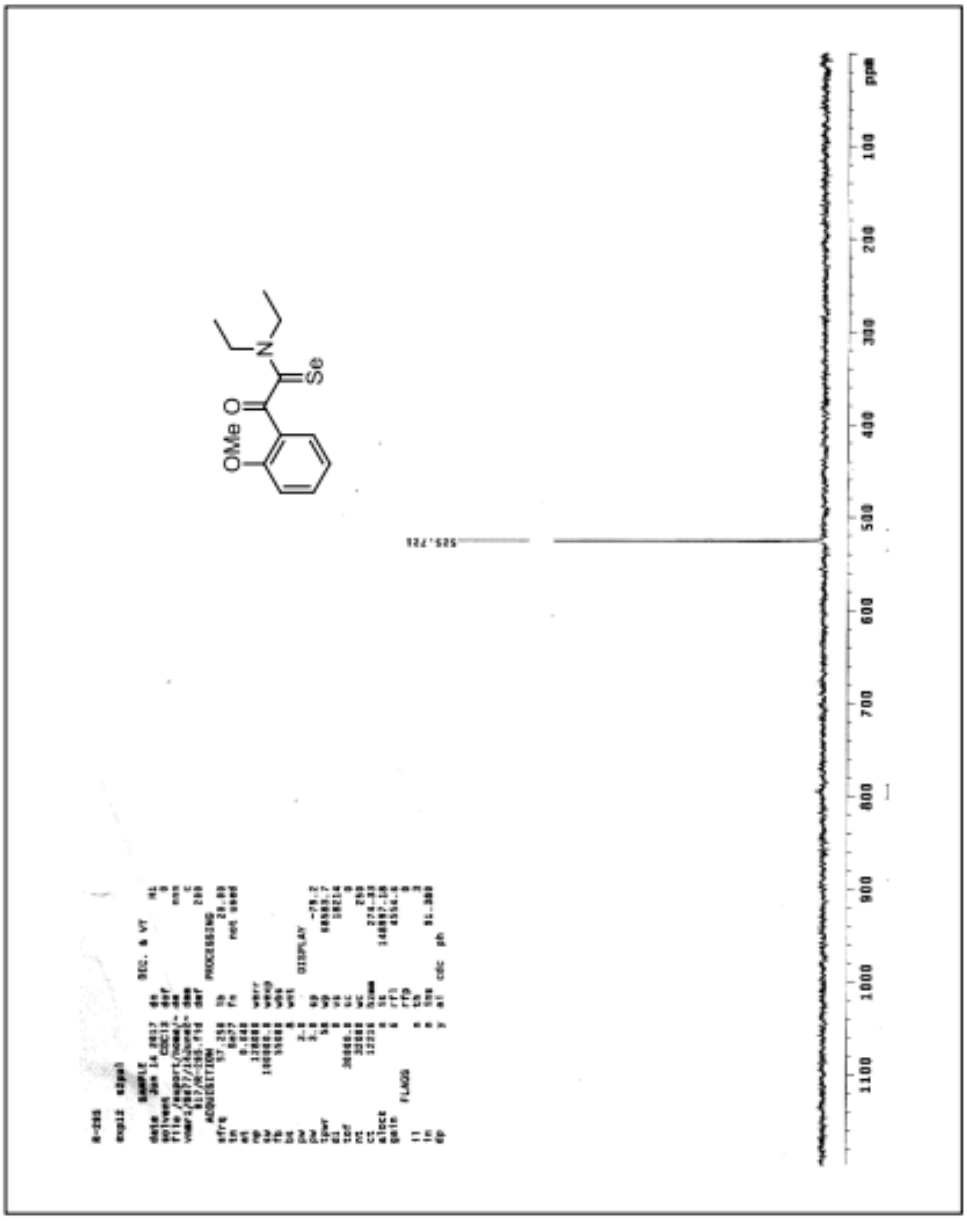


Figure 2.5.4 ^{77}Se NMR (CDCl3, 57.25 MHz) spectrum of N, N-diethyl-2-(2- methoxyphenyl)-2-oxoethaneselenoamide

^{77}Se NMR (57.25 MHz, $CDCl_3$) δ 525.721.

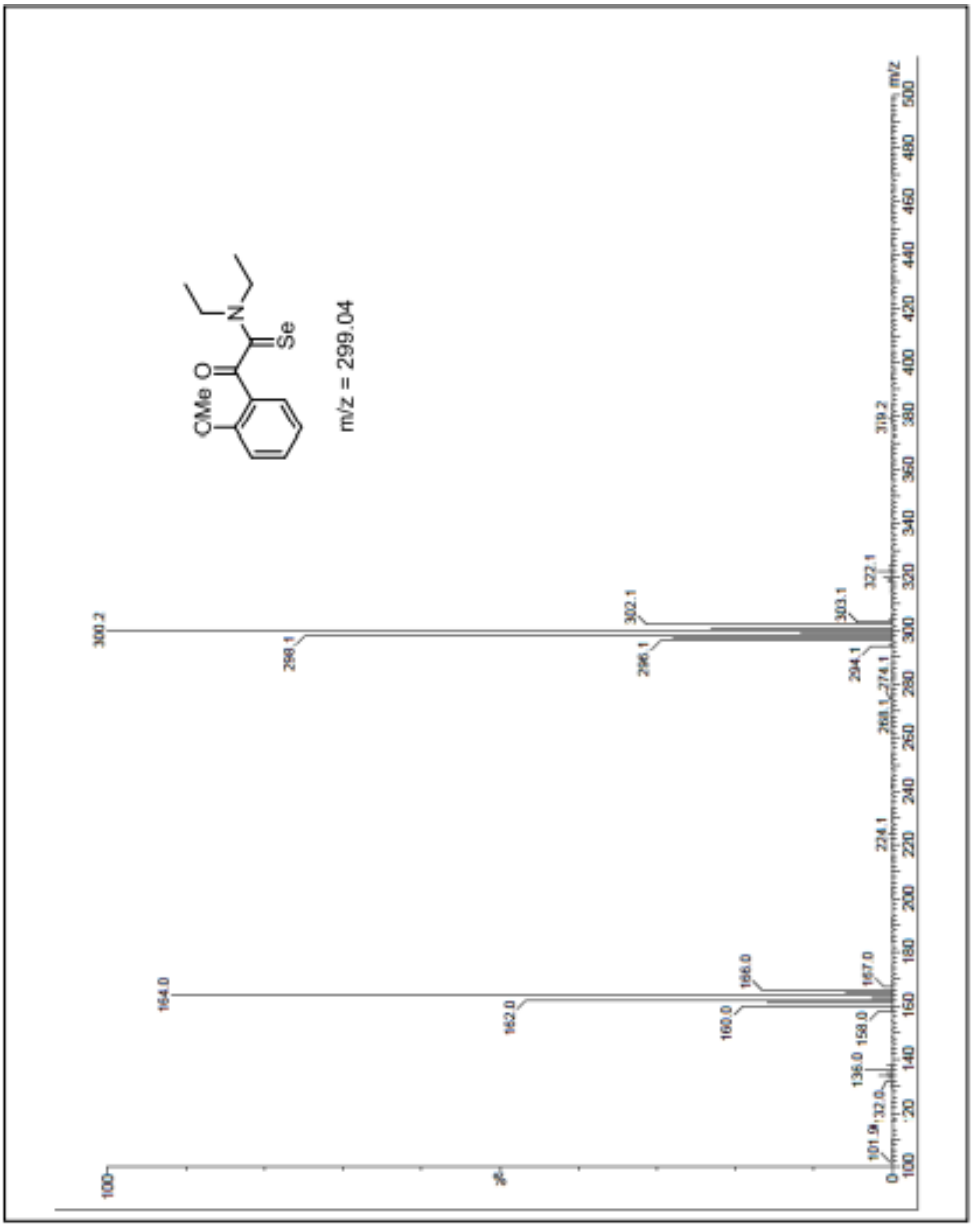


Figure 2.5.5 Mass spectrum of N, N-diethyl-2-(2-methoxyphenyl)-2- oxoethaneselenoamide

MS (ES+) : m/z 300.2 $[M + H]^+$.

3. Conclusion:

In continuation of 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.

In this study, we have synthesized N'N-Diethyl selenium compound without using catalyst and studied/optimized molar ratio of Selenium dioxide with respect to reaction temperature to achieve higher yield.

After synthesized of Selenium compound, it is well characterised/ elucidation using modern analytical techniques (XRD, NMR, Mass and IR).

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