PART – II
PART - II

SYNTHESES OF OLEOCHEMICALS

SYNTHESES OF 10-SUBSTITUTED ARYL UNDECANOIC ACIDS, OXADIAZOLE AND TRIAZOLE DERIVATIVES

INTRODUCTION

During the past decade production and utilization of oils and fats and their derivatives have grown both in size and diversity. In the industrial field there has been competition between oleochemicals and petrochemicals. The ever-increasing cost of petrochemicals has diverted the attention of chemists’ to synthesize oleochemicals derived from natural oils and fats. These fat derived chemicals are essential to a variety of oleochemical industries.

Interest in the biological and industrial potentialities of oleochemicals has resulted in the development of various synthetic procedures for the introduction of heterocyclic moiety into the hydrocarbon chain. Therefore, today oleochemicals have gained considerable momentum next only to petrochemicals in industry and technology of surfactants, lubricant additives, cosmetics, soaps, detergents, textiles, plastics, plasticizers, protective coatings, dispersants, intermediate chemicals, urethane derivatives, pharmaceuticals, organic pesticides, a variety of synthetic intermediates as stabilizers in plastic formulations, and the preparation of other long-chain compounds.
Oleochemistry deals with the synthesis of fatty acids derivatives and a special strength of oleochemical industry is that which generates the valuable products from non-edible oils or lower quality fats. The larger amounts of oils and fats are transformed by chemical reaction into basic fatty materials for use in important oil-based industries. The fundamental reactions are saponification, hydrolysis, polymerization, interesterification and hydrogenation. Many oleochemicals are manufactured starting with fatty acids and the most important being: (i) nitrogen derivatives, (ii) esters, (iii) metallic soaps, (iv) alcohols, (v) dimeric acids, (vi) ozonolysis products such as pelargonic and azelaic acids. In the world today nitrogen derivatives and esters are the two most important classes of derivatives consuming more than 50 % of fatty acids.

Reactions of double bond in fatty acid chain

A brief account of reactions in fatty acid chain has been described as follows.

Addition of Hydrogen cyanide and Nitriles

Ritter\textsuperscript{2-5} and others added hydrogen cyanide and a variety of nitriles to olefins in strong acid media to give substituted amides (1).

\[ \text{RCN} + \text{HC} = \text{C} \xrightarrow{\text{H}^+} \ \text{H}_2\text{O} \quad \text{RCONHCH-CH} \quad (1) \]
Roe and Swern\textsuperscript{6} applied this reaction to oleic acid in strong acid media yielded substituted amidostearic acids. In this reaction, sulphuric acid caused positional isomerisation through double bond migration. Under similar reaction conditions, different nitriles were successfully added to petroselinic acid\textsuperscript{7} and undecylenic acid\textsuperscript{8}.

**Addition of Thiocyanogen**

Kaufmann\textsuperscript{9} has reported that thiocyanogen adds quantitatively to the double bond of oleic acid, to one of the double bond of linoleic acid and to the two double bonds of linolenic acid to give the substituted thiocyanogen derivatives (2) of oleic, linoleic and linolenic fatty acids, respectively.

\[
\text{CH=CH-} + (\text{SCN})_2 \rightarrow \text{CH—CH—} \quad \begin{array}{c} \text{SCN} \\ \text{SCN} \end{array}
\]

**Addition of Phenols and Cresols**

Phenols and cresols react with oleic acid in sulphuric acid to give both positional isomers with respect to the site of the rearranged bond in the fatty chain, and also with respect to *ortho* and *para* positions of both groups on the aromatic ring. The product (3) is exceedingly complex.

\[
\text{CH}_2(\text{CH}_3)_2\text{CH=CH(CH}_3)_2\text{COOH} + \quad \text{CH}_2(\text{CH}_3)_2\text{CH=CH(CH}_3)_2\text{COOH} \quad \text{H}^+ \rightarrow \quad \text{CH}_2(\text{CH}_3)_2\text{CH=CH(CH}_3)_2\text{COOH} \quad \text{H}^+ \rightarrow \quad \text{CH}_2(\text{CH}_3)_2\text{CH=CH(CH}_3)_2\text{COOH} 
\]
Addition of Hydrogen sulphide and Mercaptans

Schwab, Gast, and Rohwedder\textsuperscript{10} were able to accomplish the nucleophilic addition of hydrogen sulphide to methyl oleate, methyl linoleate, and soyabean oil at $-70 \, ^\circ C$ to $+25 \, ^\circ C$ with boron trifluoride. With excess $H_2S$ and methyl oleate at $-70 \, ^\circ C$ the primary reaction products, as expected, is methyl 9-mercapto-stearate (4) and 10-mercapto-stearate (5). This has been applied for many industrial products; lubricants, synthetic rubbers, floatation collectors, and others.

![Chemical structure]

Sulphation and Sulphonation

The unsaturated acids that react readily at the double bond with concentrated sulphuric acid with the introduction of either the sulphate ($HSO_4^-$) group or sulphonate ($HSO_3^-$) group at elevated temperature. The hydroxyl groups containing seed oils such as castor oil is readily sulphated to form esters followed by neutralization which is carried out industrially and extensively in the preparation of so-called sulphonated oils for the textile industry.

The $\alpha$-sulphonation of saturated fatty acids is conveniently carried out with sulphur trioxide in dioxane. Stirton\textsuperscript{11} et al., have prepared a series of them in order
to evaluate the sodium salts as detergent materials. The preparation of sulphonated castor oil using sulphur trioxide\textsuperscript{12} apparently affords a product with a somewhat higher degree of sulphation and sulphonation than the conventional product (Turkey red oil) usually prepared with sulphuric acid.

It is possible to increase the hydroxyl group content of unsaturated oils, and other esters by partially sulphating the unsaturated bonds, followed by hydrolysis to remove the sulphate group for the replacement of hydroxyl group\textsuperscript{13}.

**Addition of Sulphur dichloride**

Sulphur dichloride (SC\textsubscript{12}), reacts readily with olefins. Grimm\textsuperscript{14} studied the addition of sulphur dichloride to various monoethenoid fatty materials (methyl oleate, methyl elaidate, oleonitrile) and ethyl linoleate and found that the products \(\beta,\beta'-\)dichlorosulphides (6), could be easily oxidized with per-acetic acid to the corresponding \(\beta,\beta'-\)dichlorosulphoxides (7), and \(\beta,\beta'-\)dichlorosulphones (8).

\[
\begin{align*}
2 \text{RCH}=\text{CHR}' & \quad + \quad \text{SCl}_2 \\
\text{CH}-\text{CH} & \quad \overset{(\text{O})}{\longrightarrow} \\
\text{CH}-\text{CH} & \quad \overset{(\text{O})}{\longrightarrow} \\
\text{CH}-\text{CH} & \quad \text{SO}_2
\end{align*}
\]

\(\text{R} = -(\text{CH}_2)_7\text{C}_3\text{H}_5\) or \(-(\text{CH}_2)_7\text{COOR}_3\) or \(-(\text{CH}_2)_7\text{CN}\)

Where \(\text{R}' = -(\text{CH}_2)_7\text{COOR}_3\) or \(-(\text{CH}_2)_7\text{CN}\) or \(-(\text{CH}_2)_7\text{C}_3\), respectively.
β, β'-Dichlorosulphides are reactive intermediates with labile chlorines that have been reacted with many nucleophiles to afford a series of potentially useful sulphur-containing fatty derivatives\textsuperscript{14}.

\[
\begin{align*}
\text{CH}_3\text{(CH}_2\text{)}_7\text{CH}—\text{CH}—\text{CH}_2\text{COOH} & + 2\text{Z}^- \rightarrow \text{CH}_3\text{(CH}_2\text{)}_7\text{CH}—\text{CH}_2\text{COOH} \\
\text{CH}_3\text{(CH}_2\text{)}_7\text{CH}—\text{CH}—\text{CH}_2\text{COOH} & + 2\text{Cl}^- + \text{positional isomers}
\end{align*}
\]

Where Z = OH, NH\textsubscript{2}, RNH, many others

**Addition of Maleic anhydride and other Dienophiles**

Fatty acids containing a conjugated diene system, in common with other dienes, react with dienophiles such as maleic anhydride and this reaction (*Diels-Alder*) is useful in the study of acids with conjugated unsaturation. Catalpic acid and octadeca-trans-9, trans-11-dienoic acid, for example, readily form maleic anhydride adducts (9 & 10) which are identical products (11) after hydrogenation. This confirms that catalpic acid contains a 9-trans, 11-trans-diene system.
Hydrogen and Reduction

In the presence of suitable catalyst, hydrogen adds to the double bonds of unsaturated fatty acids. The partial reduction of double bond may be achieved by the use of hydrazine and oxygen.

\[ -\text{CH}=\text{CH}- + \text{NH}_2\text{NH}_2 + \frac{1}{2}\text{O}_2 \rightarrow -\text{CH}_2\text{-CH}_2- + \text{N}_2 + \text{H}_2\text{O} \]

Halogenation

Chlorine, bromine, iodine monochloride and iodine monobromide are added to the double bonds of unsaturated acids and their derivatives.
Halogenated fatty compounds are being used in several novel applications, viz., as textile additives, as reactive intermediates and in dehalogenation processes to increase the degree of unsaturation.

**Addition of Carbon monoxide**

The reaction of carbon monoxide with the double bonds of fatty acids confirms that, at least three modes of addition:

1. **Hydroformylation**

   \[
   \text{Co}_2(CO)_8 \quad \xrightarrow{\text{CH} = \text{CH} - + \text{CO} + \text{H}_2} \quad \text{CHO} \quad \xrightarrow{} \quad \text{CHCH}_2_\text{CHO} \quad \xrightarrow{} \quad \text{CHCH}_2_\text{CH}_2\text{OH}
   \]

   The products of all three reactions are usually mixtures of many positional isomers.

2. **Koch reaction**

   \[
   \xrightarrow{\text{H}_2\text{SO}_4} \quad \text{CH} = \text{CH} - + \text{CO} + \text{ROH} \quad \xrightarrow{} \quad \text{COOR} \quad \text{R=} \quad \text{H}, \text{alkyl}
   \]

3. **Reppe reaction**

   \[
   \xrightarrow{\text{Ni(CO)}_4} \quad \text{CH} = \text{CH} - + \text{CO} + \text{ROH} \quad \xrightarrow{} \quad \text{COOR} \quad \text{R=} \quad \text{H}, \text{alkyl}
   \]
Reactions of carboxylic group in fatty acid chain

A brief account of reactions of carboxylic group in fatty acid chain has been described as follows.

General methods of synthesis of 1,3,4-oxadiazoles

In the five membered ring system the presence of two nitrogen and one oxygen heteroatoms defines an interesting class of compounds known as oxadiazoles. These may be of four types viz., 1,2,3-oxadiazole (12), or 1,2,4-oxadiazoles (13), 1,2,5-oxadiazole (14) and 1,3,4-oxadiazole (15).

\[
\begin{align*}
1,2,3-\text{oxadiazole} & & 1,2,4-\text{oxadiazole} \\
(12) & & (13)
\end{align*}
\]

\[
\begin{align*}
1,2,5-\text{oxadiazole} & & 1,3,4-\text{oxadiazole} \\
(14) & & (15)
\end{align*}
\]

Synthesis of oxadiazoles from thiosemicarbazides

Stolle and Gaertner\textsuperscript{15} synthesised 1,3,4-oxadiazoles (16) by cyclisation of thiosemicarbazides with PbO and NaN\textsubscript{3} in ethanol to give 2-substituted-5-aryl-1,3,4-oxadiazoles.
Hoggarth\textsuperscript{16} synthesised 2-amino-5-phenyl-1,3,4-oxadiazoles (17) by heating 1-benzoyl-s-methyl-isothiosemicarbazide for 10 minutes at 200 °C.

\begin{align*}
\text{RCONHNHCSNHR'} & \xrightarrow{\text{PbO/NaN}_3} \text{(16)} \\
\text{R=aryl R'=arylamino}
\end{align*}

Silberg and Cosma\textsuperscript{17} synthesised 1,3,4-oxadiazoles (18) by oxidative cyclisation of thiosemicarbazides with iodine in potassium iodide.

\begin{align*}
\text{RCONHNHCSNHC}_6\text{H}_5 & \xrightarrow{\text{I}_2 \text{ in KI}} \text{(18)} \\
\text{R=C}_6\text{H}_5, p(\text{Cl}) \text{C}_6\text{H}_4, p(\text{NO}_2) \text{C}_6\text{H}_4, o(\text{OH}) \text{C}_6\text{H}_4
\end{align*}

**Synthesis of oxadiazoles from hydrazines**

2,5-Diaryl-1,3,4-oxadiazoles (19) are prepared by the cyclisation of the corresponding 1,2-diaryldiydrazines in the presence of dehydrating agent, such as acetic anhydride\textsuperscript{18}.

\begin{align*}
\text{RCONHNH-CSNHR'} & \xrightarrow{\text{Ac}_2\text{O}} \text{(19)}
\end{align*}
1,3,4-Oxadiazoles (20) are also prepared by the condensation of an acid hydrazide of aromatic carboxylic or carbothionic acid with ortho ester such as ethyl ortho formate¹⁹.

\[
\begin{align*}
R\text{-C-NHNH}_2 + HC(OC_2H_5)_3 &\rightarrow R\text{-C}=O-N=N-R' \\
\text{Where } R=C_6H_5, \quad R'=H, \quad C_2H_5 \quad 4\text{-Pyridyl}
\end{align*}
\]

Konig et al.,²⁰ synthesized 2-hydroxy-5-(4-pyridyl)-1,3,4-oxadiazole (21) and 2-mercapto-5-(4-pyridyl)-1,3,4-oxadiazole (21) by reacting isonicotinic acid hydrazide with phosgene or thiophosgene.

\[
\begin{align*}
\text{Where, } X=O \text{ or } S \\
\text{Synthesis of oxadiazoles from semicarbazones}
\end{align*}
\]

2-Amino-5-phenyl-1,3,4-oxadiazoles (22) are prepared from benzaldehyde semicarbazone and sodium hypoiodide or hypobromite²¹.
Synthesis of oxadiazoles from Schiff's bases

Saikachi\textsuperscript{22} synthesized 5-substituted-2-(2-furyl)-1,3,4-oxadiazoles (23) by oxidative cyclisation of Schiff's bases by lead tetra acetate.

\[
\begin{array}{c}
\text{CONHN=CHR} \\
\text{Pb(OAc)}_4 \\
[O] \\
\end{array} \rightarrow \begin{array}{c}
\text{CON} \end{array}
\]

R = 2-furyl, 2-thienyl and substituted phenyl

However, 1,3,4-oxadiazoles (24) are also prepared by heating appropriate hydrazides with carbon disulphide and alcoholic alkali\textsuperscript{23}.

\[
\begin{array}{c}
R-\text{CONHNH}_2 \\
\text{CS}_2/\text{KOH} \\
\hline \\
R-\text{CONHNHC}_2 \text{S}_2 \text{K} \\
\hline \\
\text{R = Substituted phenyl} \\
\end{array}
\]

General methods of synthesis of 1,2,4-triazoles

In the five membered ring systems the presence of three nitrogen heteroatoms defines an interesting class of compounds known as triazoles. These may be of two types viz., 1,2,3-triazoles or \(v\)-triazoles (25) and 1,2,4- triazoles or \(s\)-triazoles (26).

\[
\begin{array}{c}
\text{HC} \\
\text{C} \\
\text{N} \\
\text{N} \\
\text{H} \\
\text{H} \\
\end{array} \quad \text{and} \quad \begin{array}{c}
\text{HC} \\
\text{C} \\
\text{N} \\
\text{N} \\
\text{H} \\
\text{H} \\
\end{array}
\]

(25) (26)
The widely applied methods for the synthesis of s-triazoles are the ring closure of acyl derivatives of aminoguanidines, semicarbazides and thiosemicarbazides in alkaline solutions.

**Synthesis of triazoles from formhydrazine and formamide**

The substituted triazoles can be (27) obtained by the fusion of N-formyl-N-alkyl or aryl hydrazine with formamide at 250 - 280 °C in poor yields. Because of difficulty in isolation of triazoles, this method has been modified by heating formamide with a substituted hydrazine hydrochloride and this general type of reaction is known as Pellizzari reaction24.

\[
\text{RNHNHCHO} + \text{HCONH}_2 \xrightarrow{\triangle} \text{HCONH}_2 + \text{RNHNH}_2\text{HCl}
\]

**Synthesis of 3-amino-1,2,4-triazoles (28) via formylamino guanidine**

The preparation of 3-amino triazoles via N- formylamino guanidine was really accomplished by heating a mixture of an amino guanidine salt (H$_2$CO$_3$, HCl, HNO$_3$, H$_2$SO$_4$) and formic acid in toluene25,26.
Synthesis of triazoles from aryl semicarbazides

The aryl semicarbazide on boiling with anhydrous formic acid yielded 3-hydroxy-1-aryl-1, 2, 4,1H-triazoles (29) which is on heating at 200 °C with P₂O₅ gave 80% of 1-aryl-1,2,4-1H-triazole(30).²⁷

\[
\text{R} = \text{C}_6\text{H}_5
\]

Synthesis of triazoles by cyclisation of acyl thiosemicarbazides

Hoggarth has reported the synthesis of 3-aryl-5-mercapto-1, 2, 4- triazole (31) by base catalyzed cyclisation of 4-acylthiosemicarbazides.²⁸

\[
\text{ArCONHNHCSNH}_2 \xrightarrow{\text{alkali}} \text{Ar} = \text{C}_6\text{H}_5, \text{N} - \text{C} - \text{N} - \text{C} - \text{SH} \quad (31)
\]

Synthesis of triazoles from s-triazines

The substituted 1,2,4-trizole i.e., 1-phenyl-1, 2, 4-1H-triazole (32) is obtained by the reaction of substituted hydrazine salt with s-triazine in 83% yield. When phenyl hydrazine hydrochloride is treated with s-triazine, which involves cleavage
of a molecule of s-triazine to yield a substituted formaimido hydrazone. This reacts immediately with another molecule of s-triazine to yield the substituted triazole\textsuperscript{29}. 

\[
\begin{array}{c}
\text{N} \quad \text{-} \quad \text{-} \quad \text{-} \quad \text{-} \quad \text{-} \quad \text{-} \quad \text{-} \quad \text{C} \\
\text{H} \\
\text{I} \\
\text{I} \\
\end{array} + \quad \begin{array}{c}
\text{3} \\
\text{NH}_2\text{NHPhHCl} \\
\end{array} \quad \begin{array}{c}
\text{HC} \\
\text{NNHR} \\
\text{NH}_2\text{HCl} \\
\end{array} \quad \begin{array}{c}
\text{N} \\
\text{N} \\
\text{N} \\
\end{array} \quad \begin{array}{c}
\text{N} \\
\text{C} \\
\text{H} \\
\text{C} \\
\text{N} \\
\text{N} \\
\text{Ph} \\
\end{array} \\
\text{3} \quad \text{NH}_4\text{Cl} \quad \begin{array}{c}
\text{N} \\
\text{N} \\
\text{C} \\
\text{H} \\
\text{C} \\
\text{N} \\
\text{N} \\
\text{Ph} \\
\end{array} \\
\text{(32)} \quad \begin{array}{c}
\text{N} \\
\text{N} \\
\text{C} \\
\text{H} \\
\text{C} \\
\text{N} \\
\text{N} \\
\text{Ph} \\
\end{array} + \quad \begin{array}{c}
\text{3} \\
\text{NH}_4\text{Cl} \\
\end{array}
\end{array}
\]
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PRESENT WORK

Interest in the biological and industrial potentialities has resulted in the development of various synthetic procedures for the production of oleochemicals. Therefore, oleochemicals have gained considerable momentum in industries of surfactants, lubricant additives, cosmetics, soaps, detergents, textiles, plastics, plasticizers, protective coatings, dispersants, intermediate chemicals, urethane derivatives, pharmaceuticals, organic pesticides and a variety of synthetic intermediates as stabilizers in plastic formulations.

The reactions between phenols and unsaturated compounds have been known for a long time. Roe and co-workers have reported the addition of phenols and phenyl ethers to oleic acid. Keeping these facts in view, the present investigation describes the novel type of acid-catalyzed addition of phenols and anilines such as phenol, anisole, m-cresol, o-chlorophenol, o-nitrophenol, aniline, o-toulidine, o-chloroaniline, m-chloroaniline and o-nitroaniline to the double bond of undec-10-enoic acid in the presence of 95% sulphuric acid to yield the 10-substituted aryl undecanoic acids (Scheme-1). The products formed on addition resulted in low yields. The chromatographic techniques have been used for the purification of compounds. The IR, $^1$H NMR, $^{13}$C NMR, MS and elemental analyses have characterized these newly synthesized compounds.
Scheme-1

(i) 95% $\text{H}_2\text{SO}_4$
(ii) Phenols & Anilines

\[
\text{CH}_2=\text{CH-(CH}_2)_8\text{-COOH} \quad \xrightarrow{\text{(ii)} \quad \text{A' 25°c}} \quad \text{CH}_3-\text{CH-(CH}_2)_8\text{-COOH}
\]

(iii) At 25° C
(iv) Hydrolysis

R =

(a) \[\text{OH}\]
(b) \[\text{OMe}\]
(c) \[\text{Cl OH}\]
(d) \[\text{CH}_3 \text{ OH}\]
(e) \[\text{NO}_2 \text{ OH}\]
(f) \[\text{NH}_2\]
(g) \[\text{NH}_2 \text{Cl}\]
(h) \[\text{Cl NH}_2\]
(i) \[\text{NH}_2 \text{CH}_3\]
(j) \[\text{NH}_2 \text{NO}_2\]
Further, the newly synthesized substituted arylundecanoic fatty acids were converted into their corresponding oxadiazoles and triazoles derivatives via hydrazides and thiosemicarbazides (Scheme-2). The IR, $^1$H NMR and elemental analyses characterized these compounds.
Scheme-2

R - COOC₂H₅ → R - CONHNH₂ (I)

KOH

R - CONHNH₂ (II)

KOH – pH 5.6

NaOH/KI

NC/CH₃OH/HCl

R - O - SH (thiol)

R - O - S (thione)

R - CONHNHCSNH₂ (IV)

R - CONHNHSNH₂ (V)

R =

(a) CH₃CH(CH₂)₈-

(b) CH₃CH(CH₂)₈-

(c) CH₃CH(CH₂)₈-

(d) CH₃CH(CH₂)₈-

(e) CH₃CH(CH₂)₈-

(f) CH₃CH(CH₂)₈-

(g) CH₃CH(CH₂)₈-

(h) CH₃CH(CH₂)₈-

(i) CH₃CH(CH₂)₈-

(j) CH₃CH(CH₂)₈-
EXPERIMENTAL

Preparation of 10-(p-hydroxyphenyl) undecanoic acid

A homogeneous mixture of undec-10-enoic acid (0.115 mole) and of phenol (0.45 mole) was prepared and was added by dropping funnel in 45 minutes to 95% sulphuric acid (0.99 mole) in a litre three-necked flask fitted with a thermometer and an efficient stirrer. The reaction temperature was maintained between 18-22 °C by external cooling. After complete addition, the mixture was poured into crushed ice and water and extracted with ether. The solvent was removed, and purified by preparative t.l.c.

Similarly, corresponding compounds were prepared by using anisole, m-cresol, o-chlorophenol, o-nitrophenol, aniline, o-touliidine, o-chloroaniline, m-chloroaniline and o-nitroaniline. The crude compounds were purified by preparative t.l.c.

Elemental analyses, yield and melting points of all the compounds are given in Table 1. Melting points were determined by open capillary method and are uncorrected.
TABLE - 1

<table>
<thead>
<tr>
<th>Sl. No.</th>
<th>Comp*, Molecular Formula</th>
<th>Yield %</th>
<th>M.P. °C</th>
<th>Carbon</th>
<th>Hydrogen</th>
<th>Nitrogen</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Cald*.</td>
<td>Found</td>
<td>Cald*.</td>
</tr>
<tr>
<td>1.</td>
<td>1a C$<em>{17}$H$</em>{26}$O$_3$</td>
<td>19.0</td>
<td>semisolid</td>
<td>73.38</td>
<td>73.25</td>
<td>9.35</td>
</tr>
<tr>
<td>2.</td>
<td>1b C$<em>{18}$H$</em>{28}$O$_3$</td>
<td>22.5</td>
<td>semisolid</td>
<td>73.97</td>
<td>73.86</td>
<td>9.58</td>
</tr>
<tr>
<td>3.</td>
<td>1c C$<em>{17}$H$</em>{25}$O$_2$Cl</td>
<td>22.0</td>
<td>semisolid</td>
<td>65.38</td>
<td>65.24</td>
<td>8.01</td>
</tr>
<tr>
<td>4.</td>
<td>1d C$<em>{18}$H$</em>{28}$O$_3$</td>
<td>17.5</td>
<td>semisolid</td>
<td>73.97</td>
<td>73.84</td>
<td>9.58</td>
</tr>
<tr>
<td>5.</td>
<td>1e C$<em>{17}$H$</em>{25}$NO$_3$</td>
<td>25.0</td>
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Comp* = Compound, Cald* = Calculated.
The newly synthesized 10-substituted aryl undecanoic acids, were further converted into oxadiazoles and triazoles derivatives.

**Preparation of hydrazides**

To a solution of an appropriate ester (0.1 mole) in 150 ethanol was added to 95% hydrazine hydrate (0.2 mole) and the mixture was heated under reflux for 4-5 hours. It was allowed to cool and the solid that separated was collected, washed with ethanol and dried. The crude compounds were recrystallised from ethanol.

**Preparation of 2-substituted-5-mercapto-1, 3, 4-oxadiazoles**

To a solution of an appropriate hydrazide (0.01 mole) in 10 ml of ethanol, a solution of CS$_2$ (2 ml) in 3 ml of water and 1 gm of potassium hydroxide was added and the mixture was refluxed for 8-10 hours until the H$_2$S is ceased. It was cooled and acidified with dilute hydrochloric acid. The solid mass that separated was collected. It was filtered, washed with distilled water and dried. The crude compounds were recrystallised from ethanol.

**Preparation of thiosemicarbazides**

To a solution of an appropriate hydrazide (0.02 mole) in 50 ml methanol, a solution of KCNS (0.03 mole) and 3 ml HCl was added with constant stirring. The mixture was immediately evaporated to dryness on a steam bath and heated for an additional hour with another 50 ml of methanol. The resulting solid was treated with distilled water and with little ethanol. The crude compound were recrystallised from ethanol.
Preparation of 3-substituted-5-mercapto 4H-1, 2, 4-triazoles

To a solution of an appropriate thiosemicarbazide (0.01 mole) in 15 ml of ethanol, a solution of 10.0% KOH (20 ml) was added and the reaction mixture was refluxed immediately for 8-10 hours on boiling water bath. It was cooled and acidified with dilute HCl at pH 5-6. The resulting solid is filtered off, washed with distilled water and dried. The crude compounds were recrystallised from ethanol.

Preparation of 3-substituted-5-amino-1, 3, 4-oxadiazoles

To a solution of an appropriate thiosemicarbazide (0.01 mole) in 15 ml of ethanol, a solution of 5N NaOH (5 ml) was added with cooling and stirring. To this clear solution, a solution of KI/I₂ was added till a permanent tinge colour of iodine persisted at room temperature. The mixture was immediately refluxed and more KI/I₂ solution was added till permanent tinge colour of iodine was remained. It was then cooled and poured to ice-cold water. The solid that separated was collected by filtration. Then it was washed with distilled water and with dilute thiosulphate solution and again with distilled water and was dried. The crude compounds were recrystallised from ethanol.

Elemental analyses, yield and melting points of all compounds are given in Table 2. Melting points were determined by open capillary method and are uncorrected.
**TABLE - 2**

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<th>M.P. °C</th>
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<th>Hydrogen</th>
<th>Nitrogen</th>
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Comp*. = Compound, Cald*. = Calculated.

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Comp\(^*\) = Compound, Cald\(^*\) = Calculated.
## Elemental analysis

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<tr>
<td>46. Vlf</td>
<td>C₁₈H₂₈N₃O₄</td>
<td>78.0</td>
<td>105-107</td>
<td>68.13</td>
<td>68.04</td>
<td>9.14</td>
<td>9.05</td>
<td>17.66</td>
<td>17.56</td>
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<tr>
<td>47. Vlg</td>
<td>C₁₈H₂₈N₄OCl</td>
<td>69.5</td>
<td>94-96</td>
<td>61.53</td>
<td>61.42</td>
<td>7.97</td>
<td>7.88</td>
<td>15.95</td>
<td>15.87</td>
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<tr>
<td>48. Vlh</td>
<td>C₁₈H₂₈N₄OCl</td>
<td>62.0</td>
<td>111-113</td>
<td>61.53</td>
<td>61.44</td>
<td>7.97</td>
<td>7.87</td>
<td>15.95</td>
<td>15.85</td>
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<tr>
<td>49. Vli</td>
<td>C₁₉H₁₃N₄O</td>
<td>68.0</td>
<td>87-89</td>
<td>68.67</td>
<td>68.58</td>
<td>9.33</td>
<td>9.22</td>
<td>16.86</td>
<td>16.78</td>
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<tr>
<td>50. Vlj</td>
<td>C₁₉H₂₈N₅O₃</td>
<td>69.5</td>
<td>103-105</td>
<td>59.66</td>
<td>59.58</td>
<td>7.73</td>
<td>7.64</td>
<td>19.33</td>
<td>19.24</td>
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</tbody>
</table>

Comp* = Compound, Cald* = Calculated.
References

1. Roe, E. T., Parker, W. E. and Swern, D.,

2. Kyame, L., Fisher, G. S. and Beckford, W. G.,

3. Hoggarth E.,

4. Boots, S. G. and Cheng, C. C.,

5. Hoggarth, E.,

6. Silberg, Al., and Cosma, N.,
RESULTS AND DISCUSSIONS

Spectral studies

10-substituted aryl undecanoic acids (Ia - Ij)

The structures of newly synthesized substituted 10-substituted aryl undecanoic acids, (Ia - Ij) were supported by IR, 'H NMR, 13C NMR, MS and elemental analyses.

IR spectra of all the compounds were taken on Fourier Transform Infrared (FTIR) Nicolet Impact-410 using KBr pallets and also as liquid films.

The compounds Ia, Ic, Id and Ie showed IR absorption band at 3400-3300 cm\(^{-1}\) for phenolic -OH group, which was merged with aromatic –CH stretching bands. And compounds If, Ig, Ih, Ii and Ij showed –NH stretching bands (doublet) at 3500-3300 cm\(^{-1}\) and –NH bending at 1630-1620 cm\(^{-1}\) were observed for the presence of –NH\(_2\) functional group, respectively. The carbonyl stretching at 1720-1700 cm\(^{-1}\) was observed for the saturated aliphatic carboxylic acid functional group.

The 'H NMR spectra of the 10-substituted aryl undecanoic acids (Ia - Ij) were recorded on Bruker Avanace-300 (300 MHz) model instrument using CDCl\(_3\) and DMSO as a solvent and TMSi as an internal standard.

The 'H NMR spectra of all the compounds (Ia - Ij) exhibited structure revealing proton signals at \(\delta\) 10.2 - 11.5 (broad singlet, 1H, -COOH, disappeared on addition of D\(_2\)O), 6.1 – 7.9 (aromatic protons), 2.7 - 3.1 (multiplet, 1H, -CH-),
2.1 - 2.3 (triplet, 2H, -CH$_2$-COOH), 1.0 - 2.0 (broad multiplet, shielded methylene protons, -CH$_2$) and 0.9-1.1 (doublet, 3H, -CH$_3$). The compounds (Ia), (Ic), (Id) and (Ie) showed proton signals at $\delta$ 5.0 - 6.0 (broad singlet, 1H, phenolic -OH, disappeared on D$_2$O addition). The compounds (If – Ij) showed proton signals at $\delta$ 4.0 - 6.0 (broad singlet, 2H, -NH$_2$, disappeared on D$_2$O addition). In compound (Ib) a singlet signal at $\delta$ 3.8 is also observed for –OCH$_3$.

The $^{13}$C NMR spectra of all the compounds (Ia - Ij) showed sharp singlet signals at $\delta$ 176 - 180 for carbonyl carbon and at $\delta$ 112-160 for aromatic carbons atoms and at $\delta$ 22 - 40 for saturated carbon atoms. The solvent CDCl$_3$ exhibited the signals at $\delta$ 78.

The mass spectra of all 10-substituted aryl undecanoic acids (Ia to Ij) were recorded on Auto Spec E1 mass spectrophotometry instrument.

The molecular ion peak of a straight chain monocarboxylic acid is weak but usually discernible. However, the molecular ion peaks of all the 10-substituted aryl undecanoic acids (Ia - Ij) were quite distinguishable. The most characteristic peaks at m/z 60 due to McLafferty rearrangements were observed in all the compounds for monocarboxylic acid.

The McLafferty rearrangement due to the carboxylic acid groups showed molecular ion peaks at m/z 60 in all the compounds.
Besides McLafferty rearrangement peaks, spectra of each compound resembles the series of hydrocarbon clusters at an interval of 14 mass units.

The details regarding each spectrum of the compounds (Ia-Ij) are given in Table-3.

The IR, $^1$H NMR, $^{13}$C NMR and MS of each compound (Ia – Ij) are enclosed as Spectrum No. 1- 40.
### TABLE – 3
Spectral Analyses

<table>
<thead>
<tr>
<th>Sl.No.</th>
<th>Compounds and Structures</th>
<th>Infrared cm^{-1} (KBr Pellets)</th>
<th>^{1}H Nuclear Magnetic Resonance Values in ppm</th>
<th>^{13}C NMR Values in ppm</th>
<th>Mass</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>CH₃-C-(CH₂)₆-COOH</td>
<td>3382 cm⁻¹ -OH stretching, for phenol.</td>
<td>11.2 (bs, H, -COOH, disappeared on D₂O addition)</td>
<td>177, (Carbonyl carbon)</td>
<td>278,261,233,219, 205,191,177,163, 149,135,121,107, 107,79,77,60&amp;51</td>
</tr>
<tr>
<td></td>
<td></td>
<td>3050 cm⁻¹ -C-H stretching, for aromatic hydrocarbon.</td>
<td>6.7&amp;6.9 (2d, 4H, aromatic protons)</td>
<td>155-114, (aromatic carbons)</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>2918&amp;2852 cm⁻¹ -C-H stretching, for chain hydrocarbon.</td>
<td>5.2 (bs, H, -OH₂, disappeared on D₂O addition)</td>
<td>78, (CDCl₃)</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>1705 cm⁻¹ -C=O stretching, for saturated aliphatic carboxylic acid.</td>
<td>2.9 (m, H, -CH-)</td>
<td>40-20, (Saturated carbons)</td>
<td></td>
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<tr>
<td></td>
<td></td>
<td></td>
<td>2.3 (t, 2H, -CH₂-COOH)</td>
<td></td>
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<td></td>
<td></td>
<td></td>
<td>1.6 (bm, 14H, Chain -CH₂-)</td>
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<td></td>
<td></td>
<td></td>
<td>1.1 (d, 3H, terminal -CH₃)</td>
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<tr>
<td>2</td>
<td>CH₃-C-(CH₂)₆-COOH</td>
<td>3050 cm⁻¹ -C-H stretching, for aromatic hydrocarbon.</td>
<td>10.5 (bs, H, -COOH, disappeared on D₂O addition)</td>
<td>178, (Carbonyl carbon)</td>
<td>292,275,247,233, 219,205,191,177,163, 163,143,77&amp;60</td>
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<tr>
<td></td>
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<td>2923&amp;2847 cm⁻¹ -C-H stretching, for chain hydrocarbon.</td>
<td>6.7&amp;6.9(2d, 4H, aromatic protons)</td>
<td>160-113, (aromatic carbons)</td>
<td></td>
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<tr>
<td></td>
<td></td>
<td>1705 cm⁻¹ -C=O stretching, for saturated aliphatic carboxylic acid.</td>
<td>3.8 (s, 3H, -OCH₃)</td>
<td>78, (CDCl₃)</td>
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<tr>
<td></td>
<td></td>
<td></td>
<td>2.8 (m, H, -CH-)</td>
<td>57, (carbon of -OCH₃)</td>
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<td>2.2 (t, 2H, -CH₂-COOH)</td>
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<td>1.4 (bm, 14H, Chain -CH₂-)</td>
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<td>0.9 (d, 3H, terminal -CH₃)</td>
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<td></td>
<td>1241 cm⁻¹ -C-O stretching, for anisole</td>
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<td>40-21, (Saturated carbons)</td>
<td>292,275,247,233, 219,205,191,177,163, 163,143,77&amp;60</td>
</tr>
<tr>
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</tr>
<tr>
<td>3.</td>
<td>CH₃-CH-(CH₂)n-COOH</td>
<td>3373 cm⁻¹ –OH stretching, for phenol superimposed on C-H stretching for aromatic hydrocarbon. 2919 &amp; 2851 cm⁻¹ –C-H stretching, for chain hydrocarbon, 1707 cm⁻¹ –C=O stretching, for saturated aliphatic carboxylic acid.</td>
<td>11.4 (bs, H, -COOH, disappeared on D₂O addition) 6.6, 6.8 &amp; 6.9 (2d &amp; 1s, 3H, aromatic protons) 5.9 (bs, H, -OH, disappeared on D₂O addition) 2.9 (m, H, -CH₃) 2.3 (t, 2H, -CH₃-COOH) 1.8 (bm, 14H, Chain -CH₂-) 1.1 (d, 3H, terminal-CH₃)</td>
<td>178, (Carboxyl carbon) 157-111, (aromatic carbons) 78, (CDCl₃) 40-22, (Saturated carbons)</td>
<td>312,295,266,253, 239,225,211,197, 183,169 &amp; 60</td>
</tr>
<tr>
<td>4.</td>
<td>CH₃-CH-(CH₂)n-COOH</td>
<td>3424 cm⁻¹ –OH stretching, for phenol superimposed on C-H stretching for aromatic hydrocarbon. 2925 &amp; 2852 cm⁻¹ –C-H stretching, for chain hydrocarbon, 1703 cm⁻¹ –C=O stretching, for saturated aliphatic carboxylic acid.</td>
<td>10.9 (bs, H, -COOH, disappeared on D₂O addition) 6.6, 6.7 &amp; 7.1 (2d &amp; 1s, 3H, aromatic protons) 5.4 (bs, H, -OH, disappeared on D₂O addition) 2.8 (m, H, -CH₃) 2.6 (s, 3H, o-CH₃) 2.3 (t, 2H, -CH₃-COOH) 1.7 (bm, 14H, Chain -CH₂-) 1.1 (d, 3H, terminal -CH₃)</td>
<td>177, (Carboxyl carbon) 155-115, (aromatic carbons) 78, (CDCl₃) 40-22, (Saturated carbons)</td>
<td>292,275,247,233, 219,205,191,177, 163,143 &amp; 60</td>
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</tr>
</tbody>
</table>
| 5. | CH₂(CH₃)₆-COOH | 3200 cm⁻¹ -OH stretching, for phenol superimposed upon -C-H stretching, for aromatic hydrocarbon  
|   |   | 2923 & 2852 cm⁻¹ -C=O stretching, for chain hydrocarbon  
|   |   | 1705 cm⁻¹ -C=O stretching, for saturated aliphatic carboxylic acid  
|   |   | 1530 cm⁻¹ -NO asymmetric stretching  
|   |   | 1377 cm⁻¹ - NO symmetric stretching.  
| 6. | CH₃CH-(CH₂)₆-COOH | 3437 & 3360 cm⁻¹ -NH stretching (doublet)  
|   |   | 3218 cm⁻¹ Fermi resonance band with overtone band at 1623 cm⁻¹  
|   |   | 3033 cm⁻¹ -C-H stretching, for aromatic hydrocarbon  
|   |   | 2923 & 2858 cm⁻¹ -C=O stretching, for chain hydrocarbon,  
|   |   | 1711 cm⁻¹ - C=O stretching, for saturated aliphatic carboxylic acid.  
|   |   | 1623 cm⁻¹ - N-H bending (def)  
|   |   | 1274 & 1175 cm⁻¹ - C-N stretching  
| 4. | 11.3 (bs, H, -COOH, disappeared on D₂O addition)  
|   |   | 7.1, 7.7 & 7.9 (2d & 1s, 3H, aromatic protons)  
|   |   | 5.9 (bs, H, -OH, disappeared on D₂O addition)  
|   |   | 2.9 (m, H, -CH₂-)  
|   |   | 2.2 (t, 2H, -CH₂-COOH)  
|   |   | 1.6 (bm, 14H, Chain -CH₂-)  
|   |   | 1.1 (d, 3H, terminal -CH₃)  
| 5. | 176, (Carbonyl carbon)  
|   |   | 150-116, (aromatic carbons)  
|   |   | 78, (CDCl₃)  
|   |   | 40-22, (Saturated carbons)  
| 6. | 323, 306, 278, 264, 250, 236, 222, 208, 194, 180, 166 & 60  
|   |   | 277, 260, 232, 218, 204, 190, 176, 162, 148, 134, 120 & 60  
|   |   | 178, (Carbonyl carbon)  
|   |   | 143-114, (aromatic carbons)  
|   |   | 78, (CDCl₃)  
|   |   | 40-22, (Saturated carbons)  
|   |   | 277, 260, 232, 218, 204, 190, 176, 162, 148, 134, 120 & 60
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<td>7.</td>
<td>CH₂CH-(CH₂)₉-COOH</td>
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<td>3470&amp;3374 cm⁻¹ – NH stretching(doublet).</td>
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<td>3218 cm⁻¹ Fermi resonance band with overtone band at 1622 cm⁻¹</td>
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<td>3054 cm⁻¹ –C-H stretching, for aromatic hydrocarbon.</td>
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<td>2930&amp;2856 cm⁻¹ –C-H stretching, for chain hydrocarbon.</td>
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<td>1715 cm⁻¹ - C=O stretching, for saturated aliphatic carboxylic acid.</td>
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<td>1622 cm⁻¹ – N-H bending (def)</td>
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<td>8.</td>
<td>CH₂CH-(CH₂)₉-COOH</td>
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<td>(lh)</td>
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<td>3472&amp;3371 cm⁻¹ – NH stretching(doublet).</td>
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<td>3220 cm⁻¹ Fermi resonance band with overtone band at 1622 cm⁻¹</td>
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<td>3054 cm⁻¹ –C-H stretching, for aromatic hydrocarbon.</td>
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<td>2930&amp;2860 cm⁻¹ –C-H stretching, for chain hydrocarbon.</td>
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<td>1720 cm⁻¹ - C=O stretching, for saturated aliphatic carboxylic acid.</td>
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<td>1625 cm⁻¹ – N-H bending (def)</td>
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</tbody>
</table>

- 177. (Carbonyl carbon)
- 144-115, (aromatic carbons)
- 78, (CDCl₃)
- 39-22, (Saturated carbons)

- 311,294,266, 252, 238,224, 210,196, 182, 169&60

- 6.4, 6.8 & 6.9 ( 2d & 1s, 3H, aromatic protons)
- 4.9 (bs, 2H, -NH₂, disappeared on D₂O addition)
- 2.8 (m, H, -CH₂-)
- 2.2 (t, 2H, -CH₂-COOH)
- 1.7 (bm, 14H, Chain -CH₂⁻)
- 1.1 (d, 3H, terminal -CH₃)

- 11.2 (bs, H, -COOH, disappeared on D₂O addition)
- 6.4, 6.8 & 6.9 ( 2d & 1s, 3H, aromatic protons)
- 4.9 (bs, 2H, -NH₂, disappeared on D₂O addition)
- 2.8 (m, H, -CH₂-)
- 2.2 (t, 2H, -CH₂-COOH)
- 1.7 (bm, 14H, Chain -CH₂⁻)
- 1.1 (d, 3H, terminal -CH₃)
<table>
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<tbody>
<tr>
<td>9.</td>
<td>CH$_2$-CH-(CH$_2$)$_6$-COOH</td>
<td><img src="image" alt="Chemical Structure" /></td>
<td><img src="image" alt="10.5 cm$^{-1}$ - NH stretching (doublet)." /></td>
<td><img src="image" alt="178, (Carbonyl carbon)" /></td>
<td><img src="image" alt="291, 274, 246, 232, 218, 204, 190, 176" /></td>
</tr>
<tr>
<td></td>
<td></td>
<td><img src="image" alt="3475 &amp; 3344 cm$^{-1}$ - NH stretching (doublet)." /></td>
<td><img src="image" alt="6.6, 7.4 &amp; 7.9 (C-H stretching, for chain hydrocarbon), 6.1, 6.7 &amp; 6.8 (aromatic protons)" /></td>
<td><img src="image" alt="144-115, (aromatic carbons)" /></td>
<td><img src="image" alt="40-22, (Saturated carbons)" /></td>
</tr>
<tr>
<td></td>
<td></td>
<td><img src="image" alt="3459 &amp; 3371 cm$^{-1}$ - NH stretching (doublet)." /></td>
<td><img src="image" alt="5.3 (bs, 2H, -NH$_2$, disappeared on D$_2$O addition)" /></td>
<td><img src="image" alt="178, (Carbonyl carbon)" /></td>
<td><img src="image" alt="291, 274, 246, 232, 218, 204, 190, 176" /></td>
</tr>
<tr>
<td>10.</td>
<td>CH$_2$-CH-(CH$_2$)$_6$-COOH</td>
<td><img src="image" alt="Chemical Structure" /></td>
<td><img src="image" alt="10.5 cm$^{-1}$ - NH stretching (doublet)." /></td>
<td><img src="image" alt="178, (Carbonyl carbon)" /></td>
<td><img src="image" alt="291, 274, 246, 232, 218, 204, 190, 176" /></td>
</tr>
<tr>
<td></td>
<td></td>
<td><img src="image" alt="3475 &amp; 3344 cm$^{-1}$ - NH stretching (doublet)." /></td>
<td><img src="image" alt="6.6, 7.4 &amp; 7.9 (C-H stretching, for chain hydrocarbon), 6.1, 6.7 &amp; 6.8 (aromatic protons)" /></td>
<td><img src="image" alt="144-115, (aromatic carbons)" /></td>
<td><img src="image" alt="40-22, (Saturated carbons)" /></td>
</tr>
</tbody>
</table>
Phenol

CH₂-CH-(CH₂)₆-COOH

Spectrum NO. 2

Current Data Parameters
NAME 0902-eavi-i
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date 20020902
Time 12.04
INSTRUM amx400
PRBS 5 mm Multinu
FUPNO
TD 12768
SOLVENT CDC13
NS 32
DS 0
SNR 10204.082 Hz
FIDRES 0.311404 Hz
AQ 160562 sec
DG 256
DN 49.000 usec
TE 300.0 K
HL 1 1 db
VL 1.00000000 sec
P1 5.60 usec
SP-1 400.1383603 MHz
NUCLEUS 1H

F2 - Processing parameters
SI 32768
SF 400.1383949 MHz
WDW EM
SSB 0
LB 0.30 Hz
PC 6.00

213
The diagram shows a spectrum with various wave numbers and peak frequencies. The chemical structure of CH₃-CH(CH₃)₂-COOH is indicated, along with annotations for specific wave numbers.
$\text{CH}_3\text{CH}-(\text{CH}_2)_2\text{COOH}$
Current Data Parameters
NAME 0902-savi-lh
EXPNO 9
PROCNO 1

F2 - Acquisition Parameters
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Time 11.09
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SNR 1.6056820 sec
RG 256
DW 49.000 usec
TE 100.0 K
HL1 1.00000000 sec
DI 5.60 usec
SPO1 400.1343949 MHz
NUCLEUS 1H

F2 - Processing parameters
SI 32768
SF 400.1343949 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 6.00
CH₂-CH-(CH₂)₈-COOH

NH₂

Cl

OCA
Current Data Parameters
NAME 0902-savi-lh
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
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Time 12.24
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PULPROG zg
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SOLVENT CDC13
NS 32
DS 0
SNR 10204.082 Hz
PFDRES 0.311404 Hz
AQ 1.6056820 sec
RG 256
DM 49.000 usec
DE 61.25 usec
TE 300.0 K
NL1 1 db
DI 1.0000000 sec
P1 5.60 usec
SVF1 400.1383603 MHz
NUCLEUS 1H

F3 - Processing parameters
SI 32768
DP 400.1343949 MHz
MDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 6.00

SPECTRUM NO. 26
CH₂CH(CH₂)₈COOH

SPECTRUM NO. 30
Current Data Parameters
NAME  0902-savi-lh
EXPNO 8
PROCNO 1

F2 - Acquisition Parameters
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Time  11:24
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PROBHD  5 mm Multinu
PULPROG  zg
TD  32768
SOLVENT  CDC13
NS  32
DS  0
SNR  10204.082 Hz
FIDRES  0.311404 Hz
AQ  1.6056820 sec
RG  256
DW  49.000 usec
DE  61.25 usec
TE  300.0 K
HL1  1 ds
DI  1.00000000 usec
P1  5.60 usec
SPO1  400.1383603 MHz
NUCLEUS  1H

F2 - Processing parameters
GI  32768
SP  400.1343949 MHz
WOW  EM
SSB  0
LB  0.30 Hz
GB  0
PC  6.00

SPECTRUM NO. 34
CH$_3$-CH-(CH$_2$)$_n$-COOH

SPECTRUM NO. 36
-CH-(CH2)8-COOH

\[ \text{CH}_3\text{-CH-(CH}_2\text{)}_8\text{-COOH} \]

**Spectrum No. 38**

**Current Data Parameters**
- **NAME**: 0902-savi-lh
- **EXPNO**: 4
- **PROCNO**: 1

**F2 - Acquisition Parameters**
- **Date**: 20020902
- **Time**: 12.42
- **INSTRUM**: amx400
- **PROBND**: 5 mm Multinu
- **PULPROG**: zg
- **TD**: 32768
- **SOXVENT**: CDCl3
- **NS**: 32
- **DS**: 0
- **SW**: 10204.082 Hz
- **FIDRES**: 0.311404 Hz
- **AQ**: 1.6056890 sec
- **RG**: 256
- **DM**: 49000 usec
- **DE**: 612.5 usec
- **TE**: 300.0 K
- **HL1**: 1 dB
- **D1**: 1.00000000 sec
- **F1**: 5.60 usec
- **SP1**: 400.1383603 MHz
- **NUCLEUS**: 1H

**F2 - Processing parameters**
- **SI**: 32768
- **SF**: 400.1383603 MHz
- **WDW**: EM
- **SSB**: 0
- **LB**: 0.30 Hz
- **PC**: 6.00
CH$_3$-CH-(CH$_2$)$_8$-COOH

SPECTRUM NO. 40
Hydrazides (IIa –IIj)

The IR spectra of all hydrazide derivatives (IIa - IIj) showed the absorption bands at 3176 - 3480 cm⁻¹ for the presence of -NH₂ functions merged with phenolic -OH and aromatic protons. The strong bands are at 1680 - 1711 cm⁻¹ due to carbonyl function attributed to carbonyl group.

¹H NMR spectra of hydrazide derivatives (IIa – IIj) showed sharp singlet signals were observed at δ 8.1 - 8.7 for -CO-NH-NH₂ protons, which are disappeared on D₂O addition. The singlet signals are at δ 4.2 – 4.5 due to the protons of -CO-NH-NH₂. The other usual signals were also observed.

The ¹³C NMR spectra of all the compounds (IIa - IIj) showed sharp singlet signals at δ 176-172 for carbonyl carbon of -CO-NH-NH₂. The solvent CDCl₃ exhibited the signals at δ 78. The other usual signals were also observed.

2-substituted-5-mercapto-1, 3, 4-oxadiazoles

The IR spectra of all 5-mercapto-oxadiazole derivatives (IIIa - IIIj) showed the characteristic absorption bands at 3100 - 3400 cm⁻¹ and at 1622 - 1650 cm⁻¹ for -NH stretching and -C=N stretching, respectively. A band around 1263-1279 cm⁻¹ and 1099 - 1127 cm⁻¹ were observed for -C=S stretching and =C-O-C= function, respectively.

¹H NMR spectra of 5-mercapto-oxadiazole derivatives (IIIa - IIIj) showed sharp singlet signals at δ 10.5 - 10.8 for -NH-C=S protons. The other usual signals were also observed.
The $^{13}$C NMR spectra of all the compounds (Ilia - Illj) showed sharp singlet signals at $\delta$ 155–152 & 150–146 for carbon atoms of oxadiazole ring, respectively. The solvent CDCl$_3$ exhibited the signals at $\delta$ 78. The other usual signals were also observed.

**Thiosemicarbazides**

The IR spectra of all the thiosemicarbazide derivatives (IVa - IVj) were observed the characteristic absorption bands at 3191 - 3288 cm$^{-1}$, 1690 - 1719 cm$^{-1}$ and 1160 - 1206 cm$^{-1}$ for $\text{-NH}_2$ stretching, for carbonyl function and for $\text{-C}=\text{S}$, respectively.

$^1$H NMR spectra of all the thiosemicarbazide derivatives (IVa - IVj) showed singlet signals at $\delta$ 5.8 – 6.5 for the protons of $\text{-CS-NH}_2$, which are disappeared on D$_2$O addition. The broad signals at 8.0 – 8.4 are due to $\text{-CO-NH-NH-}$ protons and The broad signals at 9.9 – 10.2 are due to $\text{-CO-NH-NH-}$ protons, which are disappeared on D$_2$O addition. The other usual signals were also observed.

The $^{13}$C NMR spectra of all the compounds (IVa - IVj) showed sharp singlet signals at $\delta$ 180 – 178 for $\text{-CS-}$ & 176 – 172 for carbonyl carbon. The solvent CDCl$_3$ exhibited the signals at $\delta$ 78. The other usual signals were also observed.
3-substituted-5-mercapto 4H-1, 2, 4-triazoles

The IR spectra of all the 5-mercapto-triazole derivatives (Va – Vj) showed the characteristic absorption bands at 3200 - 3300 cm\(^{-1}\), 1581 - 1607 cm\(^{-1}\) and 1240 - 1263 cm\(^{-1}\) for –NH\(_2\) stretching merged with phenolic –OH group and aromatic protons, for –C=\(\equiv\)N stretching and thione function, respectively.

\(^1\)H NMR spectra of all the 5-mercapto-triazole derivatives (Va – Vj) showed singlet signals at 6 9.7 - 10.2 for the protons of –NH–C=\(\equiv\)S- function. The other singlet signals were also observed at 6 5.5 - 6.0 for =C-NH-C=, which are disappeared on D\(_2\)O addition. The other usual signals were also observed.

The \(^{13}\)C NMR spectra of all the compounds (Va - Vj) showed sharp singlet signals at 6 142 - 138 and 138 - 136 for carbon atoms of triazole ring, respectively. The solvent CDCl\(_3\) exhibited the signals at 6 78. The other usual signals were also observed.

2-substituted-5-amino-1, 3, 4-oxadiazoles

The IR spectra of all the 5-amino-oxadiazole derivatives (Via - VIj) showed the characteristic absorption bands at 3178 – 3475 cm\(^{-1}\), 1610 – 1634 cm\(^{-1}\) and 1049 – 1116 cm\(^{-1}\) for –NH\(_2\) group merged with phenolic –OH function, aromatic –C-H stretching and –NH function, for –C=\(\equiv\)N function and for =C-O-C= function, respectively.
$^1$H NMR spectra of all the 5-amino-oxadiazole derivatives (VIA - VIj) showed sharp singlet signals at $\delta$ 7.4 - 7.8 for -C-NH$_2$ protons, which are disappeared on D$_2$O addition. The other usual signals were also observed.

The $^{13}$C NMR spectra of all the compounds (VIA - VIj) showed sharp singlet signals at $\delta$ 172 - 168 and 162 - 158 for carbon atoms of oxadiazole ring, respectively. The solvent CDCl$_3$ exhibited the signals at $\delta$ 78. The other usual signals were also observed.

The details regarding each spectrum of the compounds are given in Table-4. The spectra of IR, $^1$H NMR and $^{13}$C NMR of the compounds are enclosed as Spectrum No. 1-15.
TABLE - 4
Spectral Analyses

<table>
<thead>
<tr>
<th>SL.NO</th>
<th>COMPOUND STRUCTURE</th>
<th>IR</th>
<th>¹H NMR</th>
<th>¹C NMR</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>CH₃-CH-(CH₂)₂CONHNH₂</td>
<td>3176 &amp; 3273 cm⁻¹ (doublet) NH₂ merged with NH &amp; OH functions for hydrazide 1690 cm⁻¹ is attributed to carbonyl function</td>
<td>8.6 (s, H, [-CO-NH-NH₂], disappeared on D₂O addition) 4.4 (s, 2H, [-CO-NH-NH₂], disappeared on D₂O addition), Other usual signals were also observed.</td>
<td>174, (Carbonyl carbon) 154 - 115 (aromatic carbons) 40-20 (chain carbons)</td>
</tr>
<tr>
<td></td>
<td>(IIa)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2.</td>
<td>CH₃-CH-(CH₂)₂ =NH=S</td>
<td>3186 cm⁻¹ for –OH function 1650 cm⁻¹ –C=N stretching 1279 cm⁻¹ –C=S stretching 1192 cm⁻¹ =C-O-C= functional group</td>
<td>10.7 (s, H, [-NH-C=S-], disappeared on D₂O addition) (thione form is existed), Other usual signals were also observed.</td>
<td>154&amp;147 (oxadiazole ring carbons) 155-115 (aromatic carbons) 40-20 (chain carbons)</td>
</tr>
<tr>
<td></td>
<td>(IIIa)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>3.</td>
<td>CH₃-CH-(CH₂)₂CONHNHCSNH₂</td>
<td>3176 &amp; 3273 cm⁻¹ (doublet) NH₂ merged with -NH and OH functions. 1719 cm⁻¹ for –C=O group 1206 cm⁻¹ –C=S bending</td>
<td>8.3 (s, H, [-CO-NH-NH-CS-], disappeared on D₂O addition) (thione form) 6.2 (d, 2H, [-CS-NH₂], disappeared on D₂O addition) Other usual signals were also observed.</td>
<td>174, (Carbonyl carbon) 170, (-C=S) 154-115 (aromatic carbons) 40-20 (chain carbons)</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>3</td>
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<td>5</td>
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</tr>
</tbody>
</table>
| 4 | ![Formula](Va) | 3205 cm\(^{-1}\) - NH stretching merged with OH group  
1607 cm\(^{-1}\) - C=N stretching  
1284 cm\(^{-1}\) - C=S stretching | 10.1 (s, H, [-NH-C=S-], disappeared on D\(_2\)O addition)  
(thione form is existed)  
5.8 (s, H, [=C-NH-C=], disappeared on D\(_2\)O addition)  
Other usual signals were also observed. | 170 & 160 (triazole ring carbons)  
154-115 (aromatic carbons)  
41-20 (chain carbons) |
| 5 | ![Formula](Via) | 3178 & 3246 cm\(^{-1}\) (doublet) NH\(_2\) function.  
1610 cm\(^{-1}\) - C=N function.  
1049 cm\(^{-1}\) - C-O-C= function. | 7.4 (s, 2H, [=C-NH\(_2\)], disappeared on D\(_2\)O addition)  
(thione form)  
Other usual signals were also observed. | 170 & 160 (oxadiazole ring carbons)  
154-115 (aromatic carbons)  
41-20 (chain carbons) |
| 6 | ![Formula](Ilb) | 3186 & 3278 cm\(^{-1}\) (doublet) NH\(_2\) 1680 cm\(^{-1}\) is attributed to carbonyl function | 8.8 (s, H, [-CO-NH-NH\(_2\)], disappeared on D\(_2\)O addition)  
4.6(s, 2H, [-CO-NH-NH\(_2\)], disappeared on D\(_2\)O addition).  
Other usual signals were also observed. | 176, (Carbonyl carbon)  
154-116 (aromatic carbons)  
40-22 (chain carbons) |
<table>
<thead>
<tr>
<th>1</th>
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<th>3</th>
<th>4</th>
</tr>
</thead>
<tbody>
<tr>
<td>7</td>
<td><img src="image" alt="Molecular Structure" /></td>
<td>3186 cm⁻¹ for -OH function 1650 cm⁻¹ -C=N stretching 1279 cm⁻¹ -C=S stretching 1192 cm⁻¹ =C-O-C= functional group</td>
<td>10.5 (s, H, [-NH-C=S-], disappeared on D₂O addition), (thione form is existed) Other usual signals were also observed.</td>
</tr>
<tr>
<td>8</td>
<td><img src="image" alt="Molecular Structure" /></td>
<td>3176 &amp; 3273 cm⁻¹ (doublet) NH₂ 1719 cm⁻¹ for -C=O group 1206 cm⁻¹ -C=S bending</td>
<td>9.1 (s, H, [-CO-NH-NH-CS-], disappeared on D₂O addition), (thione form)</td>
</tr>
<tr>
<td>9</td>
<td><img src="image" alt="Molecular Structure" /></td>
<td>3205 cm⁻¹ NH stretching merged 1607 cm⁻¹ -C=N stretching 1284 cm⁻¹ -C=S stretching</td>
<td>10.1 (s, H, [-NH-C=S-], disappeared on D₂O addition), (thione form is existed)</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>143 &amp; 139 (triazole ring carbons) 157-115 (aromatic carbons) 42-21 (chain carbons)</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>3</td>
<td>4</td>
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<td>-------------------------------------------------------------------------------</td>
<td>-------------------------------------------------------------------</td>
</tr>
<tr>
<td>10</td>
<td>CH₃CH-(CH₂)₆-N⁺O⁻NH₂</td>
<td>3178 &amp; 3246 cm⁻¹ (doublet) NH₂</td>
<td>6.6 (s, 2H, [=C-NH₂⁻], disappeared on D₂O addition), (thione form)</td>
</tr>
<tr>
<td></td>
<td>(Vlb)</td>
<td>1610 cm⁻¹ =C=N function.</td>
<td>Other usual signals were also observed.</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1049 cm⁻¹ =C-O-C= function.</td>
<td></td>
</tr>
<tr>
<td>11</td>
<td>CH₃CH-(CH₂)₆-COHNH₂</td>
<td>3181 &amp; 3293 cm⁻¹ (doublet) NH₂ merged with NH and OH functions for hydrazide</td>
<td>8.8 (s, H, [-CO-NH-NH₂⁻], disappeared on D₂O addition)</td>
</tr>
<tr>
<td></td>
<td>(Ilc)</td>
<td>1673 cm⁻¹ is attributed to carbonyl function</td>
<td>4.3 (s, 2H, [-CO-NH-NH₂⁻], disappeared on D₂O addition), Other usual</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>signals were also observed.</td>
</tr>
<tr>
<td>12</td>
<td>CH₃CH-(CH₂)₆-N⁺O⁻S⁻SH</td>
<td>3196 cm⁻¹ for –OH function</td>
<td>10.5 (s, H, [-NH-C=S⁻⁻], disappeared on D₂O addition) (thione form is</td>
</tr>
<tr>
<td></td>
<td>(Ille)</td>
<td>1656 cm⁻¹ =C=N stretching</td>
<td>existed), Other usual signals were also observed.</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1272 cm⁻¹ =C=S stretching</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>1182 cm⁻¹ =C-O-C= functional group</td>
<td></td>
</tr>
</tbody>
</table>

Carbons:
- 172 & 165 (oxadiazole ring carbons)
- 157 - 115 (aromatic carbons)
- 41 - 21 (chain carbons)
<table>
<thead>
<tr>
<th>1</th>
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</thead>
<tbody>
<tr>
<td>13</td>
<td>CH₃-CH(CH₂)₄-CONHNHCSNH₂</td>
<td>3186 &amp; 3284 cm⁻¹ (doublet) NH₂ merged with -NH and OH functions for hydrazide</td>
<td>9.6 (s, H, [-CO-NH-NH-CS-], disappeared on D₂O addition), (thione form)</td>
<td>176, (Carbonyl carbon) 172, (-C=S)</td>
</tr>
<tr>
<td></td>
<td>(IVc)</td>
<td>1722 cm⁻¹ for -C=O group</td>
<td>7.7 (s, H, [-CO-NH-NH-CS-], disappeared on D₂O addition), (thione form)</td>
<td>157-115 (aromatic carbons) 43-20 (chain carbons)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1216 cm⁻¹ -C=S bending</td>
<td>7.4 (d, 2H, [-CS-NH₂], disappeared on D₂O addition), Other usual signals were also observed.</td>
<td></td>
</tr>
<tr>
<td>14</td>
<td>CH₃-CH(CH₂)₄-CONHNHCSNH₂</td>
<td>3205 cm⁻¹ -NH stretching merged with OH group</td>
<td>9.9 (s, H, [-NH-C=S-], disappeared on D₂O addition) (thione form is existed)</td>
<td>141&amp;139 (triazole ring carbons) 154-117 (aromatic carbons)</td>
</tr>
<tr>
<td></td>
<td>(Vc)</td>
<td>1617 cm⁻¹ -C=N stretching</td>
<td>5.5 (s, H, [-C-N=C=], disappeared on D₂O addition), Other usual signals were also observed.</td>
<td>41-21 (chain carbons)</td>
</tr>
<tr>
<td>15</td>
<td>CH₃-CH(CH₂)₄-CONHNHCSNH₂</td>
<td>3188&amp;3256 cm⁻¹ (doublet) NH₂ merged with OH function.</td>
<td>6.5 (s, 2H, [-C-NH₂-], disappeared on D₂O addition), (thione form) Other usual signals were also observed.</td>
<td>171 &amp; 162 (oxadiazole ring carbons) 154-118 (aromatic carbons)</td>
</tr>
<tr>
<td></td>
<td>(VIc)</td>
<td>1619 cm⁻¹ -C=N function.</td>
<td></td>
<td>41-20 (chain carbons)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1046 cm⁻¹ -C-O-C= function.</td>
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</tbody>
</table>

Merged with -NH and OH functions for hydrazide.
<table>
<thead>
<tr>
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</thead>
<tbody>
<tr>
<td>16</td>
<td>CH$_3$-CH-(CH$_2$)$_6$-CONHNH$_2$</td>
<td>3179 &amp; 3283 cm$^{-1}$ (doublet) NH$_3$ merged with NH&amp; OH</td>
<td>8.9 (s, H, [-CO-NH-NH$_2$], disappeared on D$_2$O addition)</td>
<td>174, (Carbonyl carbon)</td>
</tr>
<tr>
<td></td>
<td>(Ild)</td>
<td>functions.</td>
<td></td>
<td>154-115 (aromatic carbons)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1719 cm$^{-1}$ is attributed to carbonyl function</td>
<td></td>
<td>40-20 (chain carbons)</td>
</tr>
<tr>
<td>17</td>
<td>CH$_3$-CH-(CH$_2$)$_6$-O-SH</td>
<td>3187 cm$^{-1}$ for –OH function</td>
<td>10.6 (s, H, [-NH-C=S-], disappeared on D$_2$O addition) (thione form is existed), Other usual signals were also observed.</td>
<td>158&amp;147 (oxadiazole ring carbons)</td>
</tr>
<tr>
<td></td>
<td>(IIId)</td>
<td>1661 cm$^{-1}$ –C=N stretching</td>
<td></td>
<td>151-115 (aromatic carbons)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1274 cm$^{-1}$ –C=S stretching</td>
<td></td>
<td>40-21 (chain carbons)</td>
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<tr>
<td></td>
<td></td>
<td>1198 cm$^{-1}$ =C-O-C= functional group</td>
<td></td>
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<tr>
<td>18</td>
<td>CH$_3$-CH-(CH$_2$)$_6$-CONHNHCNSH$_2$</td>
<td>3170 &amp; 3280 cm$^{-1}$ (doublet) NH$_3$ merged with NH and OH</td>
<td>9.0 (s, 2H, 2 x [-CO-NH-NH-C S-], disappeared on D$_2$O addition), (thione form)</td>
<td>176, (Carbonyl carbon)</td>
</tr>
<tr>
<td></td>
<td>(IVd)</td>
<td>functions</td>
<td></td>
<td>171, (-C=S)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1729 cm$^{-1}$ for –C=O group</td>
<td></td>
<td>154-119 (aromatic carbons)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1216 cm$^{-1}$ –C=S bending</td>
<td></td>
<td>41-21 (chain carbons)</td>
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<tr>
<td>19</td>
<td><img src="image" alt="Structure" /></td>
<td>3213 cm⁻¹ NH stretching merged with OH group</td>
<td>9.7 (s, H, [-NH-C=S-], disappeared on D₂O addition)</td>
<td>144&amp;138 (triazole ring carbons)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1589 cm⁻¹ C=N stretching</td>
<td>(thione form is existed)</td>
<td>154-114 (aromatic carbons)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1266 cm⁻¹ C=S stretching</td>
<td>5.8 (s, H, [=C-NH-C=], disappeared on D₂O addition), Other usual signals were also observed.</td>
<td>43-20 (chain carbons)</td>
</tr>
<tr>
<td>20</td>
<td><img src="image" alt="Structure" /></td>
<td>3171&amp;3246 cm⁻¹ (doublet) NH₂ merged with OH function.</td>
<td>6.5 (s, 2H, [=C-NH₂], disappeared on D₂O addition), (thione form) Other usual signals were also observed.</td>
<td>174 &amp; 160 (oxadiazole ring carbons)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1617 cm⁻¹ C=N function.</td>
<td>154-114 (aromatic carbons)</td>
<td>41-20 (chain carbons)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1040 cm⁻¹ =C=O-C= function.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>21</td>
<td><img src="image" alt="Structure" /></td>
<td>3178 &amp; 3273 cm⁻¹ (doublet) NH₂ merged with NH₂ &amp; OH functions for hydrazide</td>
<td>8.8 (s, H, [-CO-NH-NH₂], disappeared on D₂O addition)</td>
<td>174, (Carbonyl carbon)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1712 cm⁻¹ is attributed to carbonyl function</td>
<td>4.5 (s, 2H, [-CO-NH-NH₂], disappeared on D₂O addition), Other usual signals were also observed.</td>
<td>154-115 (aromatic carbons)</td>
</tr>
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<td></td>
<td>40-20 (chain carbons)</td>
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<td>------------------------------------------------------------------</td>
<td>------------------------------------------------------------------</td>
<td>------------------------------------------------------------------</td>
</tr>
<tr>
<td>22</td>
<td>(\text{CH}<em>3\cdot\text{CH-(CH}<em>2\text{)}</em>{18}\text{SH}</em>{\text{O}})</td>
<td>3188 cm(^{-1}) for –OH function [OH\text{ function}]</td>
<td>10.6 (s, H, (-\text{NH}-\text{C}=\text{S}-), disappeared on D(_2)O addition) (thione form is existed), Other usual signals were also observed.</td>
<td>153 &amp; 146 (oxadiazole ring carbons)</td>
</tr>
<tr>
<td></td>
<td>(\text{NO}_{2})</td>
<td>1651 cm(^{-1}) -C=\text{N} stretching [\text{C}=\text{N}\text{ stretching}]</td>
<td></td>
<td>157-115 (aromatic carbons)</td>
</tr>
<tr>
<td></td>
<td>(\text{OH})</td>
<td>1276 cm(^{-1}) -C=S stretching [\text{C}=\text{S}\text{ stretching}]</td>
<td></td>
<td>40-21 (chain carbons)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1191 cm(^{-1}) =C-O-C= functional group [\text{C}=\text{O}\text{-C= functional group}]</td>
<td></td>
<td></td>
</tr>
<tr>
<td>23</td>
<td>(\text{CH}_3\cdot\text{CH-(CH}<em>2\text{)}</em>{18}\text{CONHNHCSNH}_2)</td>
<td>3186 &amp; 3270 cm(^{-1}) (doublet) NH(_2) merged with -NH and OH functions [\text{C}=\text{O}\text{ group}]</td>
<td>9.4 (s, H, (-\text{CO}-\text{NH}-\text{NH-CS-}), disappeared on D(_2)O addition) (thione form)</td>
<td>173, (Carbonyl carbon) [\text{C}=\text{S}\text{ }\text{ring carbons}]</td>
</tr>
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<td></td>
<td>(\text{NO}_{2})</td>
<td>1716 cm(^{-1}) for -C=O group [\text{C}=\text{O}\text{ group}]</td>
<td></td>
<td>169, (-\text{C}=\text{S})</td>
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<td></td>
<td>(\text{OH})</td>
<td>1216 cm(^{-1}) -C=S bending [\text{C}=\text{S}\text{ bending}]</td>
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<td>154-115 (aromatic carbons)</td>
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<td>40-20 (chain carbons)</td>
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<td>24</td>
<td>(\text{CH}<em>3\cdot\text{CH-(CH}<em>2\text{)}</em>{18}\text{SH}</em>{\text{O}})</td>
<td>3201 cm(^{-1}) -NH stretching merged with OH group [\text{C}=\text{N}\text{ stretching}]</td>
<td>9.7 (s, H, (-\text{NH}-\text{C}=\text{S}-), disappeared on D(_2)O addition) (thione form is existed)</td>
<td>141&amp;139 (triazole ring carbons)</td>
</tr>
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<td></td>
<td>(\text{NO}_{2})</td>
<td>1607 cm(^{-1}) -C=N stretching [\text{C}=\text{N}\text{ stretching}]</td>
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<td>154-115 (aromatic carbons)</td>
</tr>
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<td></td>
<td>(\text{OH})</td>
<td>1288 cm(^{-1}) -C=S stretching [\text{C}=\text{S}\text{ stretching}]</td>
<td></td>
<td>41-21 (chain carbons)</td>
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<td>25</td>
<td>CH₃CH₂CH₂NO₂</td>
<td>NH₂</td>
<td>3176 &amp; 3248 cm⁻¹ (doublet) NH₂ merged with -NH and OH functions for hydrazide</td>
<td>6.3 (s, 2H, [=C-NH₂⁻], disappeared on D₂O addition), (thione form) Other usual signals were also observed.</td>
</tr>
<tr>
<td></td>
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<td>(Vle)</td>
<td>1610 cm⁻¹ -C=N function. 1052 cm⁻¹ =C=O-C= function.</td>
<td>154-115 (aromatic carbons)</td>
</tr>
<tr>
<td>26</td>
<td>CH₃CH₂CH₂CONNH₂</td>
<td>NH₂</td>
<td>3178 &amp; 3279 cm⁻¹ (doublet) NH₂ merged with NH &amp; OH functions for hydrazide 1718 cm⁻¹ is attributed to carbonyl function</td>
<td>8.7 (s, H, [-CO-NH-NH₂⁻], disappeared on D₂O addition) 4.6 (s, 2H, [-CO-NH-NH₂⁻], disappeared on D₂O addition), Other usual signals were also observed.</td>
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<td>(III f)</td>
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<td>154-115 (aromatic carbons)</td>
</tr>
<tr>
<td>27</td>
<td>CH₃CH₂CH₂SH</td>
<td>NH₂</td>
<td>3192 cm⁻¹ for -NH₂ function 1652 cm⁻¹ -C=N stretching 1271 cm⁻¹ -C=S stretching 1198 cm⁻¹ =C=O-C= functional group</td>
<td>10.5 (s, H, [-NH-C=S⁻], disappeared on D₂O addition) (thione form is existed), Other usual signals were also observed.</td>
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<td>(IIIf)</td>
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<td>155-115 (aromatic carbons)</td>
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<tr>
<td>28</td>
<td>CH$_3$CH$_2$(CH$_2$)$_6$CONH-NH$_2$</td>
<td>3181 &amp; 3278 cm$^{-1}$ (doublet) NH$_2$ merged with -NH stretching. 1722 cm$^{-1}$ for $-\text{C}=\text{O}$ group 1211 cm$^{-1}$ $-\text{C}=\text{S}$ bending.</td>
<td>9.4 (s, H, [-CO-NH-NH-CS-], disappeared on D$_2$O addition), (thione form) 7.7 (s, H, [-CO-NH-NH-CS-], disappeared on D$_2$O addition), (thione form) 7.1 (d, 2H, [-CS-NH$_2$], disappeared on D$_2$O addition). Other usual signals were also observed.</td>
<td>176, (Carbonyl carbon) 174, (-C=S) 154-118 (aromatic carbons) 40-22 (chain carbons)</td>
</tr>
<tr>
<td>29</td>
<td>CH$_3$CH$_2$(CH$_2$)$_6$N=N=SH</td>
<td>3215 cm$^{-1}$ -NH stretching merged with OH group 1608 cm$^{-1}$ $-\text{C}=\text{N}$ stretching 1288 cm$^{-1}$ $-\text{C}=\text{S}$ stretching</td>
<td>10.1 (s, H, [-NH-C=S-], disappeared on D$_2$O addition) (thione form is existed) 6.1 (s, H, [=C-NH-C=], disappeared on D$_2$O addition). Other usual signals were also observed.</td>
<td>141&amp;138 (oxadiazole ring carbons) 154-115 (aromatic carbons) 41-20 (chain carbons)</td>
</tr>
<tr>
<td>30</td>
<td>CH$_3$CH$_2$(CH$_2$)$_6$CON=NH$_2$</td>
<td>3181 &amp; 3256 cm$^{-1}$ (doublet) NH$_2$ merged with -NH stretching. 1611 cm$^{-1}$ $-\text{C}=\text{N}$ function. 1049 cm$^{-1}$ $=\text{C}-\text{O}-\text{C}$= function.</td>
<td>6.5 (s, 2H, [=C-NH$_2$], disappeared on D$_2$O addition), (thione form) Other usual signals were also observed.</td>
<td>173 &amp; 160 (oxadiazole ring carbons) 154-115 (aromatic carbons) 41-20 (chain carbons)</td>
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<tr>
<td>31</td>
<td>CH₂CH₂(CH₂)₆CONHNH₂</td>
<td>CH₂CH₂CH₂CH₂CH₂CO₂H</td>
<td>3182 &amp; 3289 cm⁻¹ (doublet) NH₂ merged with NH functions for hydrazide</td>
<td>8.9 (s, H, [-CO-NH-NH₂], disappeared on D₂O addition)</td>
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<td>(IIg)</td>
<td>(H)</td>
<td>1689 cm⁻¹ is attributed to carbonyl function</td>
<td>4.4 (s, 2H, [-CO-NH-NH₂], disappeared on D₂O addition), Other usual signals were also observed.</td>
</tr>
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<td>(IIIg)</td>
<td>3186 cm⁻¹ for -OH function</td>
<td>10.6 (s, H, [-NH-C=S-], disappeared on D₂O addition) (thione form is existed), Other usual signals were also observed.</td>
</tr>
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<td>(IVg)</td>
<td>1650 cm⁻¹ -C=N stretching</td>
<td>9.3 (s, H, [-CO-NH-NH-CS-], disappeared on D₂O addition), (thione form)</td>
</tr>
<tr>
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<td></td>
<td>1279 cm⁻¹ -C=S stretching</td>
<td>7.8 (s, H, [-CO-NH-NH-CS-], disappeared on D₂O addition), (thione form)</td>
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<td>1192 cm⁻¹ =C-O-C= functional group</td>
<td>7.2 (d, 2H, [-CS-NH₂], disappeared on D₂O addition), Other usual signals were also observed.</td>
</tr>
</tbody>
</table>

**Notes:**
- 3182 & 3289 cm⁻¹ (doublet) NH₂ merged with NH functions for hydrazide.
- 1689 cm⁻¹ is attributed to carbonyl function.
- 3186 cm⁻¹ for -OH function.
- 1650 cm⁻¹ -C=N stretching.
- 1279 cm⁻¹ -C=S stretching.
- 1192 cm⁻¹ =C-O-C= functional group.
- 266
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<td>34</td>
<td><img src="image1" alt="Chemical Structure 1" /></td>
<td>3205 cm(^{-1}) -NH stretching merged with NH(_2) group</td>
<td>9.8 (s, H, [-NH-C=S-], disappeared on D(_2)O addition)</td>
<td>141&amp;138 (triazole ring carbons)</td>
</tr>
<tr>
<td>35</td>
<td><img src="image2" alt="Chemical Structure 2" /></td>
<td>1607 cm(^{-1}) -C=N stretching</td>
<td>(thione form is existed)</td>
<td>155-115 (aromatic carbons)</td>
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<td>36</td>
<td><img src="image3" alt="Chemical Structure 3" /></td>
<td>1284 cm(^{-1}) -C=S stretching</td>
<td>5.9 (s, H, [-C-NH-C=], disappeared on D(_2)O addition), Other usual signals were also observed.</td>
<td>40-20 (chain carbons)</td>
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Other usual signals were also observed.
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<td>37</td>
<td><img src="image" alt="Structure" /></td>
<td>3191 cm⁻¹ for –NH₂ function</td>
<td>10.6 (s, H, [–NH–C=S–], disappeared on D₂O addition) (thione form is existed), Other usual signals were also observed.</td>
<td>154&amp;148 (oxadiazole ring carbons)</td>
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<td>1655 cm⁻¹ –C=N stretching</td>
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<td>155-115 (aromatic carbons)</td>
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<td>1279 cm⁻¹ –C=S stretching</td>
<td></td>
<td>40-22 (chain carbons)</td>
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<td>1192 cm⁻¹ =C–O–C= functional group</td>
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<tr>
<td>38</td>
<td><img src="image" alt="Structure" /></td>
<td>3179 &amp; 3283 cm⁻¹ (doublet) NH₂ merged with -NH functions.</td>
<td>9.3 (s, H, [–CO–NH–NH–CS–], disappeared on D₂O addition), (thione form)</td>
<td>174, (Carbonyl carbon)</td>
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<td></td>
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<td>1710 cm⁻¹ for –C=O group</td>
<td>7.7(s, H, [–CO–NH–NH–CS–], disappeared on D₂O addition), (thione form)</td>
<td>170, (-C=S)</td>
</tr>
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<td>1208 cm⁻¹ –C=S bending</td>
<td>7.2 (d, 2H, [–CS–NH₂], disappeared on D₂O addition), (thione form)</td>
<td>155-115 (aromatic carbons)</td>
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<td>Other usual signals were also observed.</td>
<td>40-20 (chain carbons)</td>
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<tr>
<td>39</td>
<td><img src="image" alt="Structure" /></td>
<td>3225 cm⁻¹-NH stretching merged with NH₂ group</td>
<td>9.8 (s, H, [–NH–C=S–], disappeared on D₂O addition) (thione form is existed)</td>
<td>141&amp;138 (triazole ring carbons)</td>
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<td>1604 cm⁻¹ –C=N stretching</td>
<td>(thione form is existed)</td>
<td>154-115 (aromatic carbons)</td>
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<td>1285 cm⁻¹ –C=S stretching</td>
<td>5.8 (s, H, [=C–NH–C=], disappeared on D₂O addition), Other usual signals were also observed.</td>
<td>41-21 (chain carbons)</td>
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<tr>
<td>40</td>
<td>CH$_3$-CH-(CH$_2$)$_5$-CONHNH$_2$</td>
<td>3170 &amp; 3249 cm$^{-1}$ (doublet) NH$_2$ merged with -NH functions</td>
<td>6.5 (s, 2H, [=C-NH$_2$-], disappeared on D$_2$O addition), (thione form) Other usual signals were also observed.</td>
<td>170 &amp; 165 (oxadiazole ring carbons)</td>
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<td>(VIIh)</td>
<td>1610 cm$^{-1}$ -C=N function.</td>
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<td>1043 cm$^{-1}$ =C-O-C= function.</td>
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<td>41</td>
<td>CH$_3$-CH-(CH$_2$)$_5$-CONHNH$_2$</td>
<td>3184 &amp; 3280 cm$^{-1}$ (doublet) NH$_2$ merged with NH functions.</td>
<td>8.8 (s, H, [-CO-NH-NH$_2$], disappeared on D$_2$O addition)</td>
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<td>(iii)</td>
<td>1715 cm$^{-1}$ is attributed to carbonyl function</td>
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<td>4.5 (s, 2H, [-CO-NH-NH$_2$], disappeared on D$_2$O addition), Other usual signals were also observed.</td>
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<td>42</td>
<td>CH$_3$-CH-(CH$_2$)$_5$-CONHNH$_2$</td>
<td>3184 cm$^{-1}$ for –NH$_2$ function</td>
<td>10.5 (s, H, [-NH-C=S-], disappeared on D$_2$O addition) (thione form is existed), Other usual signals were also observed.</td>
<td>156 &amp; 147 (oxadiazole ring carbons)</td>
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<td>(IIIi)</td>
<td>1655 cm$^{-1}$ -C=N stretching</td>
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<td>1270 cm$^{-1}$ -C=S stretching</td>
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<td>1194 cm$^{-1}$ =C-O-C= functional group</td>
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<td>43</td>
<td>CH₃-CH-(CH₂)₆-CO-NHCSNH₂ (Vii)</td>
<td>3176 &amp; 3273 cm⁻¹ (doublet) NH₂ merged with -NH functions</td>
<td>9.3 (s, H, [CO-NH-C-S-], disappeared on D₂O addition), (thione form)</td>
<td>174, (Carbonyl carbon)</td>
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<td>1719 cm⁻¹ for -C=O group</td>
<td>170, (-C=S)</td>
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<td>1206 cm⁻¹ -C=S bending</td>
<td>154-115 (aromatic carbons)</td>
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<td>140 &amp; 137 (triazole ring carbons)</td>
<td>43-20 (chain carbons)</td>
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<tr>
<td>44</td>
<td>CH₃-CH-(CH₂)₆-NH₂ (V)</td>
<td>3215 cm⁻¹ NH stretching merged with NH₂ group</td>
<td>9.7 (s, H, [NH=C=S-], disappeared on D₂O addition)</td>
<td>140 &amp; 137 (triazole ring carbons)</td>
</tr>
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<td>1607 cm⁻¹ -C=N stretching</td>
<td>154-118 (aromatic carbons)</td>
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<td>1284 cm⁻¹ -C=S stretching</td>
<td>41-20 (chain carbons)</td>
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<td>45</td>
<td>CH₃-CH-(CH₂)₆-O-NH₂ (V)</td>
<td>3178 &amp; 3246 cm⁻¹ (doublet) NH₂ merged with -NH functions.</td>
<td>6.6 (s, 2H, [C=N-H₃-], disappeared on D₂O addition), (thione form)</td>
<td>170 &amp; 160 (oxadiazole ring carbons)</td>
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<td>1610 cm⁻¹ -C=N function.</td>
<td>154-115 (aromatic carbons)</td>
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<td>1049 cm⁻¹ =C-O-C= function.</td>
<td>41-21 (chain carbons)</td>
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| 46 | \[
\text{CH}_3\text{-CH-(CH}_2\text{)_5-CO-NNH}_2
\]

(II)

| 3170 & 3269 cm\(^{-1}\) (doublet) NH\(_2\) merged with NH functions. |
| 1708 cm\(^{-1}\) is attributed to carbonyl function |
| 8.8 (s, H, [-CO-NH-NH\(_2\)], disappeared on D\(_2\)O addition) |
| 4.1 (s, 2H, [-CO-NH-NH\(_2\)], disappeared on D\(_2\)O addition), Other usual signals were also observed. |
| 173, (Carbonyl carbon) |
| 154-114 (aromatic carbons) |
| 43-21 (chain carbons) |
| 47 | \[
\text{CH}_3\text{-CH-(CH}_2\text{)_5-CO-SH}
\]

(III)

| 3185 & 3241 cm\(^{-1}\) (doublet) NH\(_2\) stretching |
| 1652 cm\(^{-1}\) -C=N stretching |
| 1270 cm\(^{-1}\) -C=S stretching |
| 1190 cm\(^{-1}\) =C-O-C= functional group |
| 10.8 (s, H, [-NH-C=S-], disappeared on D\(_2\)O addition) (thione form is existed), Other usual signals were also observed. |
| 174 & 147 (oxadiazole ring carbons) |
| 154-115 (aromatic carbons) |
| 41-20 (chain carbons) |
| 48 | \[
\text{CH}_3\text{-CH-(CH}_2\text{)_5-CO-NNHSNH}_2
\]

(IV)

<p>| 3176 &amp; 3273 cm(^{-1}) (doublet) NH(_2) merged with -NH functions |
| 1719 cm(^{-1}) for -C=O group |
| 1206 cm(^{-1}) -C=S bending |
| 9.2 (s, 2H, 2 x [-CO-NH-NH-CS-], disappeared on D(_2)O addition), (thione form) |
| 7.9 (s, 2H, 2 x [-CO-NH-NH-CS-], disappeared on D(_2)O addition), (thione form) |
| 7.1 (d, 4H, 2 x [-CS-NH(_2)], disappeared on D(_2)O addition), Other usual signals were also observed. |
| 174, (Carbonyl carbon) |
| 170, (-C=S) |
| 154-115 (aromatic carbons) |
| 41-20 (chain carbons) |</p>
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<td>49</td>
<td><img src="" alt="Molecule 1" /></td>
<td>3180 &amp; 3241 cm⁻¹ (doublet) NH₂ stretching. 1606 cm⁻¹ -C=N stretching 1285 cm⁻¹ -C=S stretching</td>
<td>9.7 (s, H, [-NH-C=S-], disappeared on D₂O addition) (thione form is existed) 5.8 (s, H, [-C-NH-C=], disappeared on D₂O addition), Other usual signals were also observed.</td>
<td>145 &amp; 138 (triazole ring carbons) 154-116 (aromatic carbons) 41-20 (chain carbons)</td>
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<td><img src="" alt="Molecule 2" /></td>
<td>3170 &amp; 3241 cm⁻¹ (doublet) NH₂ stretching. 1619 cm⁻¹ -C=N function. 1040 cm⁻¹ =C-O-C= function.</td>
<td>6.4 (s, 2H, [=C-NH₂-], disappeared on D₂O addition), (thione form) Other usual signals were also observed.</td>
<td>171 &amp; 163 (oxadiazole ring carbons) 154-115 (aromatic carbons) 41-20 (chain carbons)</td>
</tr>
</tbody>
</table>
CH₂CH(CH₂)₆CONH₂

SPECTRUM NO. 2
SPECTRUM NO. 3

\[ CH_2-CH_2-CO-NH_2 \]

[Chemical structure diagram]
Phenol

Current Data Parameters
NAME 0902-savi-1h
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20000902
Time 12:04
INSTRUM amx400
PROBHD 5 mm Multinu
PULPROG XP
TD 12768
SOLVENT CDC13
NS 32
DS 0
SNH 10204.082 Hz
FIDRES 0.311404 Hz
AQ 1.6056820 sec
RG 256
DW 49.000 usec
DS 61.25 usec
TE 100.0 K
ML1 1 dB
D1 1.0000000 sec
P1 5.60 usec
SFO1 400.1383603 MHz
NUCLEUS 1H

F2 - Processing parameters
GI 12768
SF 400.1343949 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 6.00

SPECTRUM NO. 5
CH₃CH₂(CH₂)₆CONHNHCN₃H₂

SPECTRUM NO. 8

Phenol 3
Current Data Parameters
NAME 0902-Ravi-1h
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date 20020902
Time 12:10
INSTRUM amx400
PROBHD 5 mm Multinu
PULPROG zg
TD 12768
SOLVENT CDC13
NS 32
DS 0
SNR 10204.082 Hz
FIDRES 0.311404 Hz
AQ 1.605682 sec
RG 256
DW 49.000 usec
DE 61.25 usec
TE 300.0 K
HL1 1 dB
DI 1.0000000 sec
F1 5.60 usec
SFO1 400.1383603 MHz
NUCLEUS 1H

F2 - Processing parameters
SI 12768
SF 400.1383603 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 6.00
Current Data Parameters
NAME  0902-savi-lh
EXPNO  1
PROCNO  1

F2 - Acquisition Parameters
Date_  20020902
Time  12.04
INSTRUM  amx400
PROBHD  5 mm Multinu
PULPROG  zg
TD  32768
SOLVENT  CDCl3
NS  32
DS  0
SNH  10204.082 Hz
FIDRES  0.311404 Hz
AQ  1.6056820 sec
RG  256
DW  49.000 usec
DE  61.25 usec
TE  300.0 K
ML1  1 dB
D1  1.0000000000 sec
P1  5.60 usec
SPOL  400.1383603 MHz
NUCLEUS  1H

F2 - Processing parameters
SI  32768
SF  400.1383603 MHz
MCN  0
SGB  0
LB  0.30 Hz
GBC  0
PC  6.00

SPECTRUM NO. 11
SPECTRUM NO. 14
SPECTRUM NO. 15