
The compounds of Series-8 have been synthesized from following three steps of Scheme-3.

Step-1: Synthesis of ethyl (isonicotinamid-4-yl) acetate, Compound-1.

**Conventional method:** Equimolar solution of isonicotinamide (30 g, 0.24 mol), anhydrous potassium carbonate (33.95 g, 0.24 mol) and ethyl chloroacetate (30.10 g, 0.24 mol) in methanol (200 ml) was stirred for about 5 hours. The solvent was removed in vacuo and the residue thus obtained was purified over the column of silica gel, eluted with chloroform and recrystallized from methanol to furnish compound 1.

\[
\text{Isonicotinamide} \xrightarrow{\text{ClCH}_2\text{COOC}_2\text{H}_5} \text{Compound - 1}
\]

**Proposed mechanism for step – 1**

\[
\begin{align*}
\text{N} & \xrightarrow{-\text{H}^+} \text{deprotonation} \\
\text{CONH}_2 & \xrightarrow{\text{ClCH}_2\text{COOC}_2\text{H}_5} \text{CONHCH}_2\text{COOC}_2\text{H}_5
\end{align*}
\]

Apoorva Upadhyay  |  Dr. H.S. Gour University (A Central University), Sagar
The characterization data of the **compound-1** is given herein:

**Molecular formula**: $\text{C}_{10}\text{H}_{12}\text{N}_{2}\text{O}_{3}$

**Elemental analysis**

<table>
<thead>
<tr>
<th></th>
<th>Found (%)</th>
<th>Calculated (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>57.63</td>
<td>57.66</td>
</tr>
<tr>
<td>H</td>
<td>5.72</td>
<td>5.76</td>
</tr>
<tr>
<td>N</td>
<td>13.41</td>
<td>13.45</td>
</tr>
</tbody>
</table>

**TLC**: Solvent systems $R_f$ values

- CHCl$_3$ : CH$_3$OH (9:1 v/v) : 0.78
- CHCl$_3$ : CH$_3$OH (8:2 v/v) : 0.86

**M.p. ($^\circ$C)**: 130-131

**Yield (%)**: 71

**IR**: 3330 (NH str. in CONH), 3047, 1552, 1485, 1125 and 742 (aromatic ring), 2960, 2930, 2870, 2855, 1460, 1455, 1381 and 1370 (CH$_3$ and CH$_2$), 2932, 1462 and 1435 (N-CH$_2$), 1725 (>C=O, ester), 1638 (>C=O, amide), 1600 (>C=N), 1220 (C-O str.) and 1212 (C-N str.).

**$^1$HNMR**: 8.01 (t, 1H, J=6Hz, CONH), 7.26-8.20 (m, 4H, Ar-H), 4.21 (q, 2H, J = 7 Hz, COOCH$_2$CH$_3$), 2.47 (s, 2H, N-CH$_2$) and 1.23 (t, 3H, J = 7Hz, COOCH$_2$CH$_3$).

**$^{13}$CNMR**: 168.5 (>C=O, ester), 167.32 (>C=O, amide), 150.68, 150.18 (C near N of pyridine ring), 121.80, 121.62 and 121.30 (C of pyridine ring), 61.1 (COOCH$_2$CH$_3$), 39.26 (N-CH$_2$) and 14.5 (COOCH$_2$CH$_3$).

**Mass**: 208 [M$^+$], 163, 135, 130, 121, 106, 102, 87, 78 and 73.
**Microwave method:** A mixture of isonicotinamide (30 g, 0.24 mol), anhydrous potassium carbonate (33.95 g, 0.24 mol) and ethyl chloroacetate (30.10 g, 0.24 mol) was taken in a 250 ml beaker, mixed well and irradiated in a microwave oven at 800W for 2 minutes. The completion of the reaction was monitored by TLC. After completion of the reaction the beaker was removed from the oven and the mixture was allowed to cool at room temperature. The product was recrystallized from methanol to furnish **compound 1**, yield 85%, m.p.130-131ºC. Spectral and analytical data were found to be similar as reported for conventional method (on page 160).

**Step-2: Synthesis of 2-(isonicotinamid-4-yl) acetohydrazide, Compound-2.**

**Conventional method:** Equimolar solution of the **compound 1** (27 g, 0.13 mol) and hydrazine hydrate (6.49 g, 0.13 mol) in methanol (120 ml) was stirred for about 8 hours. The solvent was removed in vacuo and the residue thus obtained was purified over the column of silica gel, eluted with chloroform and recrystallized from methanol to furnish **compound 2**.

\[
\begin{align*}
\text{CONHCH}_2\text{COOC}_2\text{H}_5 &\xrightarrow{\text{NH}_2\text{NH}_2\cdot\text{H}_2\text{O}} \text{CONHCH}_2\text{CONHNNH}_2 \\
\text{CONHCH}_2\text{COOC}_2\text{H}_5 &\xrightarrow{\text{NH}_2\text{NH}_2\cdot\text{H}_2\text{O}} \text{CONHCH}_2\text{CONHNNH}_2
\end{align*}
\]

**Compound - 1**

**Compound - 2**

**Proposed mechanism for step – 2**

\[
\begin{align*}
\text{NH}_2\text{NH}_2\cdot\text{H}_2\text{O} &\xrightarrow{\text{(dehydration of water from hydrate)}} \text{NH}_2\text{NH}_2 \xrightarrow{-\text{H}^\ominus} \text{NNNH}_2 \\
\text{CONHCH}_2\text{COOC}_2\text{H}_5 &\xrightarrow{\text{NNNH}_2} \text{CONHCH}_2\text{CONHNNH}_2
\end{align*}
\]
The characterization data of the compound-2 is given herein:

**Molecular formula** : \( \text{C}_8\text{H}_{10}\text{N}_4\text{O}_2 \)

**Elemental analysis** :

<table>
<thead>
<tr>
<th></th>
<th>Found (%)</th>
<th>Calculated (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>49.42</td>
<td>49.45</td>
</tr>
<tr>
<td>H</td>
<td>5.13</td>
<td>5.15</td>
</tr>
<tr>
<td>N</td>
<td>28.81</td>
<td>28.85</td>
</tr>
</tbody>
</table>

**TLC** :

<table>
<thead>
<tr>
<th>Solvent systems</th>
<th>R\text{f} values</th>
</tr>
</thead>
<tbody>
<tr>
<td>CHCl\text{3} : CH\text{3}OH (9:1 v/v)</td>
<td>0.74</td>
</tr>
<tr>
<td>CHCl\text{3} : CH\text{3}OH (8:2 v/v)</td>
<td>0.82</td>
</tr>
</tbody>
</table>

**M.p. (\text{oC})** : 125-127

**Yield (%)** : 74

**IR** :

3313, 3277, 3260 and 1050 (-NHNH\text{2}), 3049, 1555, 1483, 1127 and 741 (aromatic ring), 2935, 1464 and 1437 (N-CH\text{2}), 1662, 1640 (>C=O, amide), 1602 (>C=N) and 1218 (C-N str.).

**\textsuperscript{1}HNMR** :

8.50 (s, 1H, CONH), 8.02 (t, 1H, J=6Hz, CONH), 7.28-8.22 (m, 4H, Ar-H), 4.45 (s, 2H, NH\text{2}) and 2.49 (s, 2H, N-CH\text{2}).

**\textsuperscript{13}CNMR** :

170.10, 167.34 (CONH), 150.70, 150.19 (C near N of pyridine ring), 121.81, 121.62 and 121.32 (C of pyridine ring) and 39.28 (N-CH\text{2}).

**Mass** :

194 [M\textsuperscript{+}], 178, 163, 135, 121, 116, 106, 88, 78 and 73.
Microwave method: A mixture of compound 1 (27 g, 0.13 mol) and hydrazine hydrate (6.49 g, 0.13 mol) was taken in a 250 ml beaker, mixed well and irradiated in a microwave oven at 800W for 3 minutes and worked up as usual (on page 161). The product was recrystallized from methanol to furnish compound 2, yield 89%, m.p. 125-127ºC. Spectral and analytical data were found to similar as reported for conventional method (on page 162).

Step-3: Synthesis of 2-(isonicotinamid-4-yl) acetylhydrazino benzylidene, Compound AU-36.

Conventional method: Equimolar solution of the compound 2 (1.5 g, 0.008 mol) and benzaldehyde (0.82 g, 0.008 mol) in methanol (25 ml) was stirred for about 3 hours. The solvent was removed in vacuo and the residue thus obtained was purified over the column of silica gel, eluted with chloroform and recrystallized from chloroform to furnish compound AU-36.

Similarly other compounds AU-37 to AU-40 were synthesized by treating the compound-2 with selected aromatic aldehydes (Table 4.1.1). The characterization data of the compounds AU-36 to AU-40 are given in Section-4.2.

Microwave method: A mixture of the compound 2 (1.5 g, 0.008 mol) and benzaldehyde (0.82 g, 0.008 mol) was taken in a 250 ml beaker, mixed well and irradiated in a microwave oven at 800W for 2 minutes and worked up as usual (on page 161). The product was recrystallized from chloroform to furnish compound AU-36.

Similarly other compounds AU-37 to AU-40 were synthesized by treating the compound-2 with selected aromatic aldehydes (Table 4.1.1). The characterization data of the compounds AU-36 to AU-40 were found to similar as reported for conventional method and are given in Section-4.2.
Proposed mechanism for step – 3

Table 4.1.1: Quantity of the selected aromatic aldehydes taken for step-3.

<table>
<thead>
<tr>
<th>S. No.</th>
<th>Name of the aromatic aldehydes</th>
<th>Molecular weight</th>
<th>Quantity (in gram)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Benzaldehyde</td>
<td>106.12</td>
<td>0.82</td>
</tr>
<tr>
<td>2</td>
<td>4-Chlorobenzaldehyde</td>
<td>140.57</td>
<td>1.09</td>
</tr>
<tr>
<td>3</td>
<td>3-Bromobenzaldehyde</td>
<td>185.02</td>
<td>1.43</td>
</tr>
<tr>
<td>4</td>
<td>3-Nitrobenzaldehyde</td>
<td>151.00</td>
<td>1.17</td>
</tr>
<tr>
<td>5</td>
<td>4-Methylbenzaldehyde</td>
<td>120.15</td>
<td>0.93</td>
</tr>
</tbody>
</table>

Compound AU-36

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Molecular formula</strong></td>
<td>C_{15}H_{14}N_{4}O_{2}</td>
</tr>
<tr>
<td><strong>Elemental analysis</strong></td>
<td></td>
</tr>
<tr>
<td>Found (%)</td>
<td>63.76, 4.91, 19.80</td>
</tr>
<tr>
<td>Calculated (%)</td>
<td>63.80, 4.96, 19.85</td>
</tr>
<tr>
<td><strong>TLC</strong></td>
<td></td>
</tr>
<tr>
<td>Solvent systems</td>
<td>R_f values</td>
</tr>
<tr>
<td>CHCl_3 : CH_3COCH_3 (9:1 v/v)</td>
<td>0.71</td>
</tr>
<tr>
<td>CHCl_3 : CH_3OH (8:2 v/v)</td>
<td>0.77</td>
</tr>
<tr>
<td><strong>M.p. (°C)</strong></td>
<td>40-142</td>
</tr>
<tr>
<td><strong>Recrystallization solvent</strong></td>
<td>Chloroform</td>
</tr>
<tr>
<td><strong>IR</strong></td>
<td>3318 (NH str. in CONH), 3051, 1557, 1481, 1129 and 744 (aromatic ring), 2936, 1461 and 1436 (N-CH_2), 1664, 1639 (&gt;C=O, amide), 1622 (N=CH-Ar), 1604 (&gt;C=N), 1225 (C- N str.) and 1042 (N-N str.).</td>
</tr>
<tr>
<td><strong>^1H NMR</strong></td>
<td>8.80 (s, 1H, N=CH-), 8.52 (s, 1H, CONH), 8.03 (t, 1H, J=6Hz, CONH), 7.27-8.21 (m, 9H, Ar-H) and 2.48 (s, 2H, N-CH_2).</td>
</tr>
<tr>
<td><strong>^13C NMR</strong></td>
<td>170.12, 167.33 (CONH), 150.69, 150.20 (C near N of pyridine ring), 142.8 (N=CH-Ar), 128.04-132.45 (C of aromatic ring), 121.83, 121.60 and 121.31 (C of pyridine ring) and 39.27 (N-CH_2)</td>
</tr>
<tr>
<td><strong>Mass</strong></td>
<td>282 [M^+], 204, 178, 176, 163, 161, 147, 135, 121, 119, 106, 104 and 78.</td>
</tr>
<tr>
<td><strong>Chemical name</strong></td>
<td>2-(Isonicotinamid-4-yl) acetylhydrazino benzylidene.</td>
</tr>
</tbody>
</table>
Compound AU-37

Molecular formula : $C_{15}H_{13}N_4O_2Cl$

Elemental analysis :  

<table>
<thead>
<tr>
<th>Element</th>
<th>Found (%)</th>
<th>Calculated (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>56.82</td>
<td>56.85</td>
</tr>
<tr>
<td>H</td>
<td>4.06</td>
<td>4.10</td>
</tr>
<tr>
<td>N</td>
<td>17.65</td>
<td>17.69</td>
</tr>
</tbody>
</table>

TLC :  

<table>
<thead>
<tr>
<th>Solvent systems</th>
<th>$R_f$ values</th>
</tr>
</thead>
<tbody>
<tr>
<td>CHCl$_3$ : CH$_3$OH (9:1 v/v)</td>
<td>0.71</td>
</tr>
<tr>
<td>CHCl$_3$ : CH$_3$OH (8:2 v/v)</td>
<td>0.76</td>
</tr>
</tbody>
</table>

M.p. ($^{\circ}$C) : 168-169

Recrystallization solvent : Methanol

IR : 3320 (NH str. in CONH), 3052, 1559, 1483, 1125 and 747 (aromatic ring), 2931, 1463 and 1440 (N-CH$_2$), 1665, 1642 (>C=O, amide), 1623 (N=CH-Ar), 1603 (>C=N), 1228 (C-N str.), 1040 (N-N str.) and 748 (Ar-Cl).

$^1$HNMR : 8.83 (s, 1H, N=CH-), 8.53 (s, 1H, CONH), 8.04 (t, 1H, CONH), 7.30- 8.24 (m, 8H, Ar-H) and 2.51 (s, 2H, N-CH$_2$).

$^{13}$CNMR : 170.11, 167.36 (CONH), 150.72, 150.21 (C near N of pyridine ring), 143.1 (N=CH-Ar), 135.6 (>C=N), 133.1 (C-Cl, aromatic), 128.05-132.46 (C of aromatic ring), 121.84, 121.55 and 121.33 (C of pyridine ring) and 39.30 (N-CH$_2$).

Mass : 317 [M]$^+$, 239, 211, 196, 182, 178, 163, 154, 139, 135, 121, 106 and 78.

Chemical name : 2-(Isonicotinamid-4-yl)acetylhydrazino-4-chlorobenzylidene.
Compound AU-38

**Molecular formula** : \( \text{C}_{15}\text{H}_{13}\text{N}_{4}\text{O}_{2}\text{Br} \)

**Elemental analysis** : 

<table>
<thead>
<tr>
<th></th>
<th>C</th>
<th>H</th>
<th>N</th>
</tr>
</thead>
<tbody>
<tr>
<td>Found</td>
<td>49.80</td>
<td>3.57</td>
<td>15.47</td>
</tr>
<tr>
<td>Calculated</td>
<td>49.84</td>
<td>3.60</td>
<td>15.51</td>
</tr>
</tbody>
</table>

**TLC** : Solvent systems Rf values
- \( \text{CHCl}_3 : \text{CH}_3\text{COCH}_3 \) (9:1 v/v) 0.71
- \( \text{CHCl}_3 : \text{CH}_3\text{OH} \) (8:2 v/v) 0.77

**M.p. (°C)** : 153-154

**Recrystallization solvent** : Acetone

**IR** : 3324 (NH str. in CONH), 3053, 1560, 1482, 1127 and 746 (aromatic ring), 2933, 1464 and 1441 (N-CH\(_2\)), 1668, 1643 (>C=O, amide), 1625 (N=CH-Ar), 1600 (>C=N), 1226 (C-N str.), 1036 (N-N str.) and 613 (Ar-Br).

**\(^1\)HNMR** : 8.84 (s, 1H, N=CH-), 8.56 (s, 1H, CONH), 8.08 (t, 1H, CONH), 7.33-8.27 (m, 8H, Ar-H) and 2.52 (s, 2H, N-CH\(_2\)).

**\(^{13}\)CNMR** : 170.15, 167.37 (CONH), 150.76, 150.24 (C near N of pyridine ring), 143.2 (N=CH-Ar), 135.7 (>C=N), 128.08-132.49 (C of aromatic ring), 121.88, 121.60 and 121.36 (C of pyridine ring), 118.8 (C-Br, aromatic) and 39.31 (N-CH\(_2\)).


**Chemical name** : 2-(Isonicotinamid-4-yl)acetylhydrazino-3- bromobenzylidine.
Compound AU-39

Molecular formula : \( \text{C}_{15}\text{H}_{13}\text{N}_{5}\text{O}_{4} \)

Elemental analysis : 
\[
\begin{array}{ccc}
\text{C} & \text{H} & \text{N} \\
\text{Found} (%) & 54.99 & 3.94 & 21.37 \\
\text{Calculated} (%) & 55.02 & 3.97 & 21.40 \\
\end{array}
\]

TLC : 
- Solvent systems
  - \( \text{CHCl}_{3} : \text{CHOH} \) (9:1 v/v) \( R_f \) value 0.81
  - \( \text{CHCl}_{3} : \text{CH}_{3}\text{OH} \) (8:2 v/v) \( R_f \) value 0.88

M.p. (\( ^{\circ}\text{C} \)) : 159-160

Recrystallization solvent : Methanol

IR : 3325 (NH str. in CONH), 3055, 1558, 1485, 1126 and 748 (aromatic ring), 2932, 1466 and 1442 (N-CH\(_2\)), 1669, 1648 (>C=O, amide), 1630 (N=CH-Ar), 1607 (>C=N), 1529, 1342 (Ar-NO\(_2\)), 1229 (C-N str.) and 1033 (N-N str.).

\(^1\text{HNMR} \): 8.86 (s, 1H, N = CH-), 8.57 (s, 1H, CONH), 8.09 (t, 1H, CONH), 7.32-8.26 (m, 8H, Ar-H) and 2.55 (s, 2H, N-CH\(_2\)).

\(^{13}\text{CNMR} \): 170.18, 167.40 (CONH), 150.75, 150.25 (C near N of pyridine ring), 143.6 (N=CH-Ar), 140.3 (C-NO\(_2\), aromatic), 128.10-132.51 (C of aromatic ring), 121.87, 121.66 and 121.39 (C of pyridine ring) and 39.35 (N-CH\(_2\)).

Mass : 327 [M]\(^+\), 249, 221, 206, 192, 178, 164, 163, 149, 135, 121, 106 and 78.

Chemical name : 2-(Isonicotinamid-4-yl)acetylhydrazino-3-nitrobenzylidene.
Compound AU-40

Molecular formula : \( \text{C}_{16}\text{H}_{16}\text{N}_{4}\text{O}_{2} \)

Elemental analysis :

<table>
<thead>
<tr>
<th>Element</th>
<th>Found (%)</th>
<th>Calculated (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>64.81</td>
<td>64.84</td>
</tr>
<tr>
<td>H</td>
<td>5.36</td>
<td>5.40</td>
</tr>
<tr>
<td>N</td>
<td>18.88</td>
<td>18.91</td>
</tr>
</tbody>
</table>

TLC :

<table>
<thead>
<tr>
<th>Solvent systems</th>
<th>( R_f ) values</th>
</tr>
</thead>
<tbody>
<tr>
<td>( \text{CHCl}_3 : \text{CH}_3\text{OH} ) (9:1 v/v)</td>
<td>0.75</td>
</tr>
<tr>
<td>( \text{CHCl}_3 : \text{CH}_3\text{OH} ) (8:2 v/v)</td>
<td>0.82</td>
</tr>
</tbody>
</table>

M.p. (°C) : 135-136

Recrystallization solvent : Acetone

IR :

3327 (NH str. in CONH), 3054, 1561, 1484, 1129 and 749 (aromatic ring), 2934, 1465 and 1445 (N-CH\(_2\)), 2920 (Ar-CH\(_3\)), 1671, 1645 (>C=O, amide), 1629 (N=CH-Ar), 1611 (>C=N), 1227 (C-N str.) and 1034 (N-N str.).

\(^1\)HNMR :

8.87 (s, 1H, N=CH-), 8.60 (s, 1H, CONH), 8.10 (t, 1H, CONH), 7.37-8.31 (m, 8H, Ar-H), 2.58 (s, 2H, N-CH\(_2\)) and 2.27 (s, 3H, Ar-CH\(_3\)).

\(^1^3\)CNMR :

170.19, 167.41 (CONH), 150.78, 150.28 (C near N of pyridine ring), 143.7 (N=CH-Ar), 128.11-132.52 (C of aromatic ring), 121.90, 121.68 and 121.40 (C of pyridine ring), 39.36 (N-CH\(_2\)) and 22.6 (CH\(_3\)).

Mass :

296 [M]^+ , 218, 190, 178, 175, 163, 161, 135, 121, 118, 106 and 78.

Chemical name : 2-(Isonicotinamid-4-yl)acetylhydrazino-4- methylbenzylidene.

The compounds of Series-9 (AU-41 to AU-45) have been synthesized from compounds of Series-8 (AU-36 to AU-40) as precursors, step-4 (Scheme-3).

Step-4: Synthesis of 4-phenyl-3-chloro-1-[(isonicotinamid-4-yl)acetamido]-2-oxo-azetidine, Compound AU-41.

Conventional Method: Equimolar solution of the compound AU-36 (1 g, 0.003 mol) and chloroacetyl chloride (0.40 g, 0.003 mol) with triethylamine (0.36 g, 0.003 mol) in acetone (25 ml) was stirred for about 5 hours. The solvent was removed in vacuo and the residue thus obtained was purified over the column of silica gel, eluted with chloroform and recrystallized from methanol to furnish compound AU-41.

Other compounds AU-42 to AU-45 were synthesized in a similar manner using AU-37 to AU-40 with chloroacetyl chloride and triethylamine (Table 4.3.1) respectively. Characterization data are given in Section 4.4.

Microwave method: A mixture of the compound AU-36 (1 g, 0.003 mol) and chloroacetyl chloride (0.40 g, 0.003 mol) with triethylamine (0.36 g, 0.003 mol) was taken in a 250 ml beaker, mixed well and irradiated in a microwave oven at 800W for 4 minutes and worked up as usual (on page 161). The product was recrystallized from methanol to furnish compound AU-41, yield 84%, m.p. 135-137ºC.

Other compounds AU-42 to AU-45 were synthesized in a similar manner using AU-37 to AU-40 with chloroacetyl chloride and triethylamine (Table 4.3.1). The characterization data of the compounds AU-41 to AU-45 were found to similar as reported for conventional method and are given in Section-4.4.
AU-36 to AU-40
(Series - 8)

AU-41 to AU-45
(Series - 9)

Ar= Various substituted aryl groups

Proposed mechanism for step – 4
Table 4.3.1: Quantity of various benzylidenes, chloroacetyl chloride and triethylamine taken for step-4.

<table>
<thead>
<tr>
<th>Compds</th>
<th>Name of the benzylidenes</th>
<th>Mol. wt.</th>
<th>Quantity of benzylidenes (in gram)</th>
<th>Quantity of chloroacetyl chloride (in gram)</th>
<th>Quantity of triethylamine (in gram)</th>
</tr>
</thead>
<tbody>
<tr>
<td>AU-36</td>
<td>2-(Isonicotinamid-4-yl) acetylhydrazino-benzylidene</td>
<td>282</td>
<td>1.00</td>
<td>0.40</td>
<td>0.36</td>
</tr>
<tr>
<td>AU-37</td>
<td>2-(Isonicotinamid-4-yl) acetylhydrazino-4-chlorobenzylidene</td>
<td>316.5</td>
<td>1.26</td>
<td>0.45</td>
<td>0.40</td>
</tr>
<tr>
<td>AU-38</td>
<td>2-(Isonicotinamid-4-yl) acetylhydrazino-3-bromobenzylidene</td>
<td>361</td>
<td>1.15</td>
<td>0.36</td>
<td>0.32</td>
</tr>
<tr>
<td>AU-39</td>
<td>2-(Isonicotinamid-4-yl) acetylhydrazino-3-nitrobenzylidene</td>
<td>327</td>
<td>1.35</td>
<td>0.47</td>
<td>0.42</td>
</tr>
<tr>
<td>AU-40</td>
<td>2-(Isonicotinamid-4-yl) acetylhydrazino-4-methylbenzylidene</td>
<td>296</td>
<td>1.10</td>
<td>0.42</td>
<td>0.38</td>
</tr>
</tbody>
</table>

Compound AU-41

Molecular formula : \( \text{C}_{17}\text{H}_{15}\text{N}_{4}\text{O}_{3}\text{Cl} \)

Elemental analysis :

<table>
<thead>
<tr>
<th>Element</th>
<th>Found (%)</th>
<th>Calculated (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>56.84</td>
<td>56.88</td>
</tr>
<tr>
<td>H</td>
<td>4.16</td>
<td>4.18</td>
</tr>
<tr>
<td>N</td>
<td>15.57</td>
<td>15.61</td>
</tr>
</tbody>
</table>

TLC: Solvent systems \( R_f \) values

- \( \text{CHCl}_3 : \text{CH}_3\text{COCH}_3 \) (9:1 v/v) : 0.72
- \( \text{CHCl}_3 : \text{CH}_3\text{OH} \) (9:1 v/v) : 0.79

M.p. \( (^\circ\text{C}) \) : 135-137

Recrystallization solvent : Methanol

IR:

- 3340 (NH str. in CONH), 3060, 1556, 1489, 1130 and 740 (aromatic ring), 2940, 1461 and 1449 (N-CH\(_2\)), 1720 (>C=O, cyclic), 1675, 1653 (>C=O, amide), 1613 (>C=N), 1224 (C-N str.), 1040 (N-N str.) and 740 (CH-Cl).

\(^1\text{HNMR} \):

- 8.63 (s, 1H, CONH), 8.16 (t, 1H, J=6Hz, CONH), 7.43-8.35 (m, 9H, Ar-H), 5.10 (d, 1H, J=5Hz, CHCl), 4.20 (d, 1H, J=5Hz, -N-CH-) and 2.60 (s, 2H, N-CH\(_2\)).

\(^{13}\text{CNMR} \):

- 172.0 (>C=O, cyclic), 170.23, 167.47 (CONH), 150.81, 150.32 (C near N of pyridine ring), 133.5 (CH-Cl), 128.14-132.55 (C of aromatic ring), 121.92, 121.73 and 121.42 (C of pyridine ring), 41.1 (N-CH\(_{-}\)) and 39.40 (N-CH\(_2\)).

Mass : 359 [M\(^+\)], 331, 281, 253, 238, 225, 224, 210, 196, 181, 178, 168, 163, 153, 135, 121, 106 and 78 (Chart 4).

Chemical name : 4-Phenyl-3-chloro-1-[(isonicotinamid-4-yl) acetamido]-2-oxo-azetidine.
Compound AU-42

Molecular formula : C_{17}H_{14}N_{4}O_{3}Cl_{2}

Elemental analysis : C  H  N
Functional analysis: C  H  N

Found (%) : 51.86  3.53  14.20
Calculated (%) : 51.89  3.56  14.24

TLC : Solvent systems  R_f  values
CHCl_3 : CH_3OH (9:1 v/v)  0.68
CHCl_3 : CH_3OH (8:2 v/v)  0.78

M.p. (°C) : 171-172
Recrystallization solvent : Methanol

IR : 3335 (NH str. in CONH), 3061, 1560, 1485, 1452 (N-CH_2),
1721 (>C=O, cyclic), 1678, 1658
(>C=O, amide), 1618 (>C=N), 1227
(C-N str.), 1037 (N-N str.), 745 (Ar-
Cl) and 741 (CH-Cl).

^1HNMR : 8.64 (s, 1H, CONH), 8.17 (t, 1H,
J = 6Hz, CONH), 7.46-8.38 (m, 9H,
Ar-H), 5.11 (d, 1H, J = 5Hz, CHCl),
4.21 (d, 1H, J = 5Hz, -N-CH-) and
2.61 (s, 2H, N-CH_2).

^{13}CNMR : 172.4 (>C=O, cyclic), 170.26,
167.50 (CONH), 150.84, 150.33 (C
near N of pyridine ring), 133.9 (C-
Cl, aromatic), 132.8 (CH-Cl),
128.15-132.57 (C of aromatic
ring), 121.93, 121.76 and 121.44 (C
of pyridine ring), 41.2 (N-CH-) and
39.44 (N-CH_2).

Mass : 394 [M'], 366, 316, 288, 273, 260,
259, 245, 231, 216, 203, 188, 178,
163, 135, 121, 106 and 78.

Chemical name : 4-(4-Chlorophenyl)-3-chloro-1-
[(isonicotinamid-4-yl) acetamido]-2-
oxo-azetidine.
Compound AU-43

Molecular formula : C_{17}H_{14}N_{4}O_{3}ClBr

Elemental analysis : \[\begin{array}{ccc}
C & H & N \\
\text{Found} (%) & 46.57 & 3.16 & 12.77 \\
\text{Calculated} (%) & 46.61 & 3.20 & 12.80 \\
\end{array}\]

TLC : Solvent systems \(R_f\) values
- CHCl\(_3\) : CH\(_3\)OH (9:1 v/v) \(0.69\)
- CHCl\(_3\) : CH\(_3\)OH (8:2 v/v) \(0.75\)

M.p. (\(^{\circ}\)C) : 149-150

Recrystallization solvent : Ethanol

IR : 3332 (NH str. in CONH), 3065, 1562, 1483, 1125 and 749 (aromatic ring), 2937, 1466 and 1455 (N-CH\(_2\)), 1724 (>C=O, cyclic), 1679, 1659 (>C=O, amide), 1621 (>C=N), 1228 (C-N str.), 1035 (N-N str.), 744 (CH-Cl) and 616 (Ar- Br).

\(^1\)HNMR : 8.68 (s, 1H, CONH), 8.20 (t, 1H, J = 6Hz, CONH), 7.47-8.40 (m, 9H, Ar-H), 5.14 (d, 1H, J = 5Hz, CHCl), 4.24 (d, 1H, J=5Hz, -N-CH-) and 2.64 (s, 2H, N-CH\(_2\)).

\(^13\)CNMR : 172.5 (>C=O, cyclic), 170.29, 167.51 (CONH), 150.86, 150.36 (C near N of pyridine ring), 132.9 (CH-Cl), 128.18-132.58 (C of aromatic ring), 121.98, 121.69 and 121.48 (C of pyridine ring), 118.2 (C-Br, aromatic), 41.5 (N-CH-) and 39.47 (N-CH\(_2\)).


Chemical name : 4-(3-Bromophenyl)-3-chloro-1-[(isonicotinamid-4-yl)acetamido]-2-oxo-azetidine.
Compound AU-44

Molecular formula : \( C_{17}H_{14}N_{5}O_{5}Cl \)

Elemental analysis :

\[
\begin{array}{ccc}
\text{Found (\%)} & \text{C} & \text{H} & \text{N} \\
50.51 & 3.44 & 17.31 \\
\end{array}
\]

\[
\begin{array}{ccc}
\text{Calculated (\%)} & \text{C} & \text{H} & \text{N} \\
50.54 & 3.47 & 17.34 \\
\end{array}
\]

TLC : Solvent systems \( R_f \) values

- \( \text{CHCl}_3 : \text{CH}_3\text{OH} (9:1 \text{v/v}) \) : 0.72
- \( \text{CHCl}_3 : \text{CH}_3\text{OH} (8:2 \text{v/v}) \) : 0.78

M.p. (\( ^{\circ}\text{C} \)) : 140-141

Recrystallization solvent : Methanol

IR : 3336 (NH str. in CONH), 3066, 1564, 1486, 1126 and 748 (aromatic ring), 2939, 1465 and 1456 (N-CH\(_2\)), 1725 (>C=O, cyclic), 1682, 1662 (>C=O, amide), 1624 (>C=N), 1526, 1340 (Ar-NO\(_2\)), 1230 (C-N str.), 1038 (N-N str.) and 745 (CH-Cl).

\(^1\text{HNMR} \) : 8.69 (s, 1H, CONH), 8.23 (t, 1H, J=6Hz, CONH), 7.50-8.41 (m, 9H, Ar-H), 5.15 (d, 1H, J = 5Hz, CHCl), 4.25 (d, 1H, J=5Hz, -N-CH-) and 2.65 (s, 2H, N-CH\(_2\)).

\(^{13}\text{CNMR} \) : 172.8 (>C=O, cyclic), 170.30, 167.55 (CONH), 150.88, 150.37 (C near N of pyridine ring), 140.1 (C-NO\(_2\), aromatic), 133.3 (CH-Cl), 128.14-132.55 (C of aromatic ring), 121.97, 121.71 and 121.52 (C of pyridine ring), 41.8 (N-CH-) and 39.48 (N-CH\(_2\)).


Chemical name : 4-(3-Nitrophenyl)-3-chloro-1-[[isonicotinamid-4-yl] acetamido]-2-oxo-azetidine.
## Compound AU-45

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Molecular formula</td>
<td>( C_{18}H_{17}N_4O_3Cl )</td>
</tr>
<tr>
<td>Elemental analysis</td>
<td></td>
</tr>
<tr>
<td>Found (%)</td>
<td>( C: 57.93 \quad H: 4.52 \quad N: 15.01 )</td>
</tr>
<tr>
<td>Calculated (%)</td>
<td>( C: 57.97 \quad H: 4.56 \quad N: 15.03 )</td>
</tr>
<tr>
<td>TLC</td>
<td>Solvent systems ( \text{R}_f ) values</td>
</tr>
<tr>
<td>( \text{CHCl}_3 : \text{CH}_3\text{OH (9:1 v/v)} )</td>
<td>0.71</td>
</tr>
<tr>
<td>( \text{CHCl}_3 : \text{CH}_3\text{OH (8:2 v/v)} )</td>
<td>0.79</td>
</tr>
<tr>
<td>M.p. (°C)</td>
<td>132-133</td>
</tr>
<tr>
<td>Recrystallization solvent</td>
<td>Chloroform</td>
</tr>
<tr>
<td>IR</td>
<td>3334 (NH str. in CONH), 3068, 1563, 1488, 1131 and 750 (aromatic ring), 2938, 1468 and 1459 (N-CH(_2)), 2922 (Ar-CH(_3)), 1728 (&gt;C=O, cyclic), 1683, 1663 (&gt;C=O, amide), 1625 (&gt;C=N), 1226 (C-N str.), 1036 (N-N str.) and 748 (CH-Cl).</td>
</tr>
<tr>
<td>(^{1}\text{HNMR})</td>
<td>8.71 (s, 1H, CONH), 8.24 (t, 1H, J = 6Hz, CONH), 7.52-8.43 (m, 9H, Ar-H), 5.18 (d, 1H, J = 5Hz, CHCl), 4.28 (d, 1H, J=5Hz, -N-CH-), 2.68 (s, 2H, N-CH(_2)) and 2.21 (s, 3H, Ar-CH(_3)).</td>
</tr>
<tr>
<td>(^{13}\text{CNMR})</td>
<td>172.9 (&gt;C=O, cyclic), 170.32, 167.56 (CONH), 150.89, 150.40 (C near N of pyridine ring), 133.4 (CH-Cl), 128.25-132.64 (C of aromatic ring), 122.13, 121.83 and 121.53 (C of pyridine ring), 41.9 (N-CH-), 39.50 (N-CH(_2)) and 22.3 (Ar-CH(_3)).</td>
</tr>
<tr>
<td>Mass</td>
<td>373 [M(^+)], 345, 295, 267, 252, 239, 238, 224, 210, 195, 182, 178, 167, 163, 135, 121, 106 and 78.</td>
</tr>
<tr>
<td>Chemical name</td>
<td>4-(4-Methylphenyl)-3-chloro-1-[(isonicotinamid-4-yl) acetamido]-2-oxo-azetidine.</td>
</tr>
</tbody>
</table>
SECTION-4.5: CONVENTIONAL AND MICROWAVE ASSISTED SYNTHESIS OF THE COMPOUNDS OF SERIES-10 (AU-46 TO AU-50) OF SCHEME-3.

The compounds of Series-10 (AU-46 to AU-50) have been synthesized from compounds of Series-8 (AU-36 to AU-40) as precursors, step-5 (Scheme-3).

Step-5: Synthesis of 2-phenyl-3-[(isonicotinamid-4-yl) acetamido]-4-oxo-1,3-thiazolidine, Compound AU-46.

Conventional method: Equimolar solution of the compound AU-36 (1.2 g, 0.004 mol) and thioglycolic acid (0.39 g, 0.004 mol) with a pinch of anhydrous ZnCl$_2$ in methanol (20 ml) was stirred for about 4 hours. The solvent was removed in vacuo and the residue thus obtained was purified over the column of silica gel, eluted with chloroform and recrystallized from acetone to furnish compound AU-46.

Other compounds AU-47 to AU-50 were synthesized in a similar manner using AU-37 to AU-40 with thioglycolic acid (Table 4.5.1) respectively. The characterization data of the compounds AU-46 to AU-50 are given in Section-4.6.

Microwave method: A mixture of the compound AU-36 (1.2 g, 0.004 mol) and thioglycolic acid (0.39 g, 0.004 mol) with a pinch of anhydrous ZnCl$_2$ was taken in a 250 ml beaker, mixed well and irradiated in a microwave oven at 800W for 3 minutes and worked up as usual (on page 161). The product was recrystallized from acetone to furnish compound AU-46, yield 88%, m.p. 138-139ºC.

Other compounds AU-47 to AU-50 were synthesized in a similar manner using AU-37 to AU-40 with thioglycolic acid (Table 4.5.1) respectively. The characterization data of the compounds AU-46 to AU-50 were found to similar as reported for conventional method and are given in Section-4.6.
AU-36 to AU-40
(Series - 8)

AU-46 to AU-50
(Series - 10)

Ar= Various substituted aryl groups

Proposed mechanism for step – 5
Table 4.5.1: Quantity of various benzylidenes and thioglycolic acid taken for step-5.

<table>
<thead>
<tr>
<th>Compds</th>
<th>Name of the benzylidenes</th>
<th>Mol. wt.</th>
<th>Quantity of benzylidenes (in gram)</th>
<th>Quantity of thioglycolic acid (in gram)</th>
</tr>
</thead>
<tbody>
<tr>
<td>AU-36</td>
<td>2-(Isonicotinamid-4-yl) acetylhydrazino benzylidene</td>
<td>282</td>
<td>1.20</td>
<td>0.39</td>
</tr>
<tr>
<td>AU-37</td>
<td>2-(Isonicotinamid-4-yl) acetylhydrazino-4-chlorobenzylidene</td>
<td>316.5</td>
<td>1.30</td>
<td>0.38</td>
</tr>
<tr>
<td>AU-38</td>
<td>2-(Isonicotinamid-4-yl) acetylhydrazino-3-bromobenzylidene</td>
<td>361</td>
<td>1.24</td>
<td>0.32</td>
</tr>
<tr>
<td>AU-39</td>
<td>2-(Isonicotinamid-4-yl) acetylhydrazino-3-nitrobenzylidene</td>
<td>327</td>
<td>1.40</td>
<td>0.29</td>
</tr>
<tr>
<td>AU-40</td>
<td>2-(Isonicotinamid-4-yl) acetylhydrazino-4-methylbenzylidene</td>
<td>296</td>
<td>1.12</td>
<td>0.35</td>
</tr>
</tbody>
</table>
SECTION-4.6: CHARACTERIZATION OF THE COMPOUNDS OF SERIES-6 (AU-46 TO AU-50) OF SCHEME-3.

Compound AU-46

Molecular formula : \( \text{C}_{17}\text{H}_{16}\text{N}_{4}\text{O}_{3}\text{S} \)

Elemental analysis : 

<table>
<thead>
<tr>
<th></th>
<th>Found (%)</th>
<th>Calculated (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>57.26</td>
<td>57.28</td>
</tr>
<tr>
<td>H</td>
<td>4.47</td>
<td>4.49</td>
</tr>
<tr>
<td>N</td>
<td>15.68</td>
<td>15.72</td>
</tr>
</tbody>
</table>

TLC : 

<table>
<thead>
<tr>
<th>Solvent systems</th>
<th>Rf values</th>
</tr>
</thead>
<tbody>
<tr>
<td>( \text{CHCl}<em>{3} : \text{CH}</em>{3}	ext{OH} ) (9:1 v/v)</td>
<td>0.71</td>
</tr>
<tr>
<td>( \text{CHCl}<em>{3} : \text{CH}</em>{3}	ext{OH} ) (8:2 v/v)</td>
<td>0.79</td>
</tr>
</tbody>
</table>

M.p. (°C) : 138-139

Recrystallization solvent : Acetone

IR : 

<table>
<thead>
<tr>
<th>Wave numbers (cm(^{-1}))</th>
</tr>
</thead>
<tbody>
<tr>
<td>NH str. in CONH : 3322</td>
</tr>
<tr>
<td>3058, 1551, 1485, 1131 and 746 (aromatic ring), 2980 (N-CH-S), 2941, 1470 (S-CH(_2)), 2937, 1466 and 1448 (N-CH(_2)), 1710 (&gt;C=O, cyclic), 1673, 1650 (&gt;C=O, amide), 1612 (&gt;C=N), 1227 (C-N str.), 1046 (N-N str.) and 690 (C-S-C).</td>
</tr>
</tbody>
</table>

\(^{1}\)HNMR : 

<table>
<thead>
<tr>
<th>Chemical shifts (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>8.61(^{-}) (s, 1H, CONH), 8.14 (t, 1H, CONH), 7.40-8.34 (m, 9H, Ar-H), 4.10 (s, 1H, -N-CH(_2)-), 3.11 (s, 2H, COCH(_2)S) and 2.59 (s, 2H, N-CH(_2)).</td>
</tr>
</tbody>
</table>

\(^{13}\)CNMR : 

<table>
<thead>
<tr>
<th>Chemical shifts (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>172.1 (&gt;C=O, cyclic), 170.22, 167.44 (CONH), 150.79, 150.29 (C near N of pyridine ring), 128.15-132.56 (C of aromatic ring), 121.93, 121.75 and 121.43 (C of pyridine ring), 43.1 (COCH(_2)S), 41.1 (NCHS) and 39.39 (N-CH(_2)).</td>
</tr>
</tbody>
</table>

Mass : 

<table>
<thead>
<tr>
<th>Mass values</th>
</tr>
</thead>
<tbody>
<tr>
<td>356 [M](^{+}), 328, 314, 278, 250, 236, 235, 221, 208, 193, 179, 178, 163, 151, 150, 136, 135, 121, 106 and 78.</td>
</tr>
</tbody>
</table>

Chemical name : 2-Phenyl-3-[(isonicotinamid-4-yl)acetamido]-4-oxo-1,3-thiazolidine.
Compound AU-47

Molecular formula : \( C_{17}H_{15}N_4O_3S\text{Cl} \)

Elemental analysis : 

<table>
<thead>
<tr>
<th></th>
<th>Found (%)</th>
<th>Calculated (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>52.18</td>
<td>52.22</td>
</tr>
<tr>
<td>H</td>
<td>3.80</td>
<td>3.84</td>
</tr>
<tr>
<td>N</td>
<td>14.32</td>
<td>14.33</td>
</tr>
</tbody>
</table>

TLC

Solvent systems \( R_f \) values
- \( \text{CHCl}_3 : \text{CH}_3\text{OH} \) (9:1 v/v) : 0.75
- \( \text{CHCl}_3 : \text{CH}_3\text{OH} \) (8:2 v/v) : 0.81

M.p. (°C) : 152-153

Recrystallization solvent : Chloroform

IR : 1525 (NH str. in CONH), 1483, 1278 and 741 (aromatic ring), 2986 (N-CH-S), 2934, 1463 and 1450 (N-CH), 1713 (>C=O, cyclic), 1676, 1651 (>C=O, amide), 1614 (>C=N), 1223 (C-N str.), 1048 (N-N str.), 749 (Ar-Cl) and 691 (C-S-C).

\(^1\text{HNMR} : 8.62 (s, 1H, CONH), 8.15 (t, 1H, CONH), 7.41-8.35 (m, 8H, Ar-H), 4.14 (s, 2H, COCH\text{\_2}S) and 2.61 (s, 2H, N-CH\text{\_2}).

\(^{13}\text{CNMR} : 172.2 (>\text{C}=\text{O}, \text{cyclic}), 170.23, 167.45 (\text{CONH}), 150.81, 15034 (\text{C near } N \text{ of pyridine ring}), 133.6 (\text{C-Cl, aromatic}), 128.16-132.57 (\text{C of aromatic ring}), 121.94, 121.61 and 121.44 (\text{C of pyridine ring}), 43.2 (COCH\text{\_2}S), 41.2 (NCHS) and 39.40 (N-CH\text{\_2}).


Chemical name : 2-(4-Chlorophenyl)-3-[(isonicotinamid-4-yl) acetamido]-4-oxo-1,3-thiazolidine.
Compounds AU-48

Molecular formula : \( C_{17}H_{15}N_4O_3SBr \)
Elemental analysis : \[ \begin{array}{ccc}
\text{C} & \text{H} & \text{N} \\
\text{Found} & 46.84 & 3.43 & 12.84 \\
\text{Calculated} & 46.88 & 3.45 & 12.87
\end{array} \]
TLC : Solvent systems
- \( \text{CHCl}_3: \text{CH}_3\text{OH} \ (9:1 \text{ v/v}) \) 0.71
- \( \text{CHCl}_3: \text{CH}_3\text{OH} \ (8:2 \text{ v/v}) \) 0.78
M.p. (\( \circ \text{C} \)) : 136-137
Recrystallization solvent : Ethanol
IR : 3328 (NH str. in CONH), 3063, 1555, 1486, 1129 and 743 (aromatic ring), 2985 (N-CH-S), 2937, 1467 (S-CH\(_2\)), 2932, 1462 and 1451 (N-CH\(_2\)), 1712 (>C=O, cyclic), 1677, 1654 (>C=O, amide), 1617 (>C=N), 1225 (C-N str.), 1042 (N-N str.), 693 (C-S-C) and 614 (Ar-Br).
\(^1\)HNMR : 8.65 (s, 1H, CONH), 8.18 (t, 1H, CONH), 7.44-8.38 (m, 8H, Ar-H), 4.15 (s, 1H, -N-CH-S) and 2.64 (s, 2H, N-CH\(_2\)).
\(^13\)CNMR : 172.4 (>C=O, cyclic), 170.24, 167.47 (CONH), 150.83, 150.33 (C near N of pyridine ring), 128.20-132.61 (C of aromatic ring), 121.98, 121.76 and 121.45 (C of pyridine ring), 118.7 (C-Br, aromatic), 43.6 (COCH\(_2\)S), 41.5 (NCHS) and 39.41 (N-CH\(_2\)).
Chemical name : 2-(3-Bromophenyl)-3-[(isonicotinamid-4-yl)acetamido]-4-oxo-1,3-thiazolidine.
Compound AU-49

Molecular formula : \(\text{C}_{17}\text{H}_{15}\text{N}_{5}\text{O}_{5}\text{S}\)

Elemental analysis : \begin{align*}
\text{C} & : 50.86 \\
\text{H} & : 3.74 \\
\text{N} & : 17.45 \\
\end{align*}

Found (%) : \begin{align*}
\text{C} & : 50.84 \\
\text{H} & : 3.72 \\
\text{N} & : 17.42 \\
\end{align*}

Calculated (%) : \begin{align*}
\text{C} & : 50.86 \\
\text{H} & : 3.74 \\
\text{N} & : 17.45 \\
\end{align*}

TLC : Solvent systems \(R_f\) values
- \(\text{CHCl}_3 : \text{CH}_3\text{OH} (9:1 \text{ v/v})\) : 0.81
- \(\text{CHCl}_3 : \text{CH}_3\text{OH} (8:2 \text{ v/v})\) : 0.89

M.p. \(\text{C}^\circ\) : 144-145

Recrystallization solvent : Chloroform

IR : \begin{align*}
3326 \text{ (NH str. in CONH), } 3065, \\
1554, 1489, 1126 \text{ and } 742 \text{ (aromatic ring), } 2988 \text{ (N-CH-S), } 2939, 1469 \\
(\text{S-CH}_2), 2935, 1464 \text{ and } 1455 \text{ (N-CH}_2), 1716 \text{ (>C=O, cyclic), } 1680, \\
1655 \text{ (>C=O, amide), } 1616 \text{ (>C=N), } 1526, 1343 \text{ (Ar-NO}_2), 1228 \text{ (C-N str.), } 1041 \text{ (N-N str.) and } 697 \\
(\text{C-S-C}).
\end{align*}

\(^1\text{HNMR}\) : \begin{align*}
8.66 \text{ (s, 1H, CONH), } 8.19 \text{ (t, 1H, CONH), } 7.48-8.42 \text{ (m, 8H, Ar-H), } \\
4.19 \text{ (s, 1H, -N-CH-), } 3.17 \text{ (s, 2H, COCH}_2\text{S) and } 2.65 \text{ (s, 2H, N-CH}_2). \\
\end{align*}

\(^{13}\text{CNMR}\) : \begin{align*}
172.7 \text{ (>C=O, cyclic), } 170.29, \\
167.51 \text{ (CONH), } 150.88, 150.36 \text{ (C near N of pyridine ring), } 140.6 \text{ (C-NO}_2, \text{ aromatic), } 128.19-132.60 \text{ (C of aromatic ring), } 121.97, 121.71 \text{ and } \\
121.48 \text{ (C of pyridine ring), } 43.7 \text{ (COCH}_2\text{S), } 41.8 \text{ (NCHS) and } 39.44 \\
(\text{N-CH}_2). \\
\end{align*}

Mass : \begin{align*}
401 \text{ [M]^+}, 373, 359, 323, 295, 281, \\
280, 266, 253, 238, 224, 196, 195, \\
181, 178, 163, 135, 121, 106 \text{ and } 78. \\
\end{align*}

Chemical name : \begin{align*}
2-(3-\text{Nitrophenyl})-3-\text{[(isonicotinamid-4-yl)acetamido]}-4-\text{oxo-1,3-thiazolidine.}
\end{align*}
Compound AU-50

Molecular formula : $\text{C}_{18}\text{H}_{18}\text{N}_{4}\text{O}_{3}\text{S}$

Elemental analysis :
- Found (%) : C 58.33, H 4.82, N 15.10
- Calculated (%) : C 58.36, H 4.86, N 15.13

TLC :
- Solvent systems
  - $\text{CHCl}_3 : \text{CH}_3\text{OH} (9:1 \text{v/v})$ : $R_f$ 0.72
  - $\text{CHCl}_3 : \text{CH}_3\text{OH} (8:2 \text{v/v})$ : $R_f$ 0.78

M.p. ($^\circ\text{C}$) : 115-116

Recrystallization solvent : Chloroform

IR :
- 3329 (NH str. in CONH), 3066, 1557, 1490, 1128 and 744 (aromatic ring), 2989 (N-CH-S), 2940, 1468 (S-CH$_2$), 2936, 1465 and 1454 (N-CH$_2$), 2923 (Ar-CH$_3$), 1718 (>C=O, cyclic), 1681, 1658 (>C=O, amide), 1620 (>C=N), 1230 (C-N str.), 1044 (N-N str.) and 698 (C-S-C).

$^1$HNMR :
- 8.69 (s, 1H, CONH), 8.20 (t, 1H, CONH), 7.46-8.40 (m, 8H, Ar-H), 4.20 (s, 1H, -N-CH-), 3.18 (s, 2H, COCH$_2$S), 2.66 (s, 2H, N-CH$_2$) and 2.26 (s, 3H, Ar-CH$_3$).

$^{13}$CNMR :
- 172.8 (>C=O, cyclic), 170.28, 167.52 (CONH), 150.87, 150.37 (C near N of pyridine ring), 128.22-132.63 (C of aromatic ring), 122.10, 121.80 and 121.52 (C of pyridine ring), 43.9 (COCH$_2$S), 41.9 (NCHS), 39.45 (N-CH$_2$) and 22.7 (Ar-CH$_3$).

Mass :

Chemical name : 2-(4-Methylphenyl)-3-[[isonicotinamid-4-yl] acetamido]-4-oxo-1,3-thiazolidine.

The compounds of Series-11 (AU-51 to AU-55) have been synthesized from compounds of Series-10 (AU-46 to AU-50) as precursors, step-6 (Scheme-3).

Step-6: Synthesis of 5-benzylidene-2-phenyl-3-[(isonicotinamid-4-yl)acetamido]-4-oxo-1, 3-thiazolidine, Compound AU-51.

**Conventional method:** Equimolar solution of the compound AU-46 (1.2 g, 0.003 mol) and thioglycolic acid (0.30 g, 0.003 mol) in methanol (20 ml) in the presence of sodium ethoxide was stirred for about 2 hours. The solvent was removed in vacuo and the residue thus obtained was purified over the column of silica gel, eluted with chloroform and recrystallized from acetone to furnish compound AU-51.

Similarly other compounds AU-52 to AU-55 were synthesized in a similar manner using compounds AU-47 to AU-50 (Table 4.7.1) and various selected aromatic aldehydes (Table 4.7.2). The characterization data of the compounds AU-51 to AU-55 are given in Section-4.8.

**Microwave method:** A mixture of the compound AU-46 (1 g, 0.003 mol) and benzaldehyde (0.30 g, 0.003 mol) in the presence of sodium ethoxide was taken in a 250 ml beaker, mixed well and irradiated in a microwave oven at 800W for 2 minutes and worked up as usual (on page 161). The product was recrystallized from acetone to furnish compound AU-51.

Similarly other compounds AU-52 to AU-55 were synthesized in a similar manner using compounds AU-47 to AU-50 (Table 4.7.1) and various selected aromatic aldehydes (Table 4.7.2). The characterization data of the compounds AU-51 to AU-55 were found to similar as reported for conventional method and are given in Section-4.8.
AU-46 to AU-50
(Series - 10)

AU-51 to AU-55
(Series - 11)

Ar = Ar₁ = Various substituted aryl groups

Proposed mechanism for step – 6

\[ \text{C}_2\text{H}_3\text{ONa} \rightarrow \text{C}_2\text{H}_3\text{O}^- + \text{Na}^+ \]

\[ \text{Ar} - \text{C} - \text{H} \rightarrow \text{Ar} - \text{C} - \text{H} \]

\[ \text{NCONHCH}_2\text{CONHN}_{\text{H}}\text{Ar} + \text{C}_2\text{H}_3\text{O}^- \rightarrow \text{NCONHCH}_2\text{CONHN}_{\text{H}}\text{Ar} \]

\[ \text{NCONHCH}_2\text{CONHN}_{\text{H}}\text{Ar} + \text{Ar} - \text{C} - \text{H} \rightarrow \text{NCONHCH}_2\text{CONHN}_{\text{H}}\text{Ar} \]

\[ + \text{H}^+ \text{ Protonation } \rightarrow \text{NCONHCH}_2\text{CONHN}_{\text{H}}\text{Ar} \]

\[ - \text{H}_2\text{O} \rightarrow \text{NCONHCH}_2\text{CONHN}_{\text{H}}\text{Ar} \]
### Table 4.7.1: Quantity of various thiazolidin-4-ones taken for step-6.

<table>
<thead>
<tr>
<th>Compounds</th>
<th>Name of the thiazolidinones</th>
<th>Molecular weight</th>
<th>Quantity (in gram)</th>
</tr>
</thead>
<tbody>
<tr>
<td>AU-46</td>
<td>2-Phenyl-3-[(isonicotinamid-4-yl) acetamido]-4-oxo-1, 3-thiazolidine</td>
<td>356</td>
<td>1.00</td>
</tr>
<tr>
<td>AU-47</td>
<td>2-(4-Chlorophenyl)-3-[(isonicotinamid-4-yl) acetamido]-4-oxo-1,3-thiazolidine</td>
<td>390.5</td>
<td>1.12</td>
</tr>
<tr>
<td>AU-48</td>
<td>2-(3-Bromophenyl)-3-[(isonicotinamid-4-yl) acetamido]-4-oxo-1,3-thiazolidine</td>
<td>435</td>
<td>1.10</td>
</tr>
<tr>
<td>AU-49</td>
<td>2-(3-Nitrophenyl)-3-[(isonicotinamid-4-yl) acetamido]-4-oxo-1,3-thiazolidine</td>
<td>401</td>
<td>1.18</td>
</tr>
<tr>
<td>AU-50</td>
<td>2-(4-Methylphenyl)-3-[(isonicotinamid-4-yl) acetamido]-4-oxo-1,3-thiazolidine</td>
<td>370</td>
<td>1.00</td>
</tr>
</tbody>
</table>

### Table 4.7.2: Quantity of the selected aromatic aldehydes taken for step-6.

<table>
<thead>
<tr>
<th>S. No.</th>
<th>Name of the aromatic aldehydes</th>
<th>Molecular weight</th>
<th>Quantity (in gram)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Benzaldehyde</td>
<td>106.12</td>
<td>0.30</td>
</tr>
<tr>
<td>2</td>
<td>2-Chlorobenzaldehyde</td>
<td>140.57</td>
<td>0.40</td>
</tr>
<tr>
<td>3</td>
<td>2-Bromobenzaldehyde</td>
<td>185.02</td>
<td>0.47</td>
</tr>
<tr>
<td>4</td>
<td>2-Nitrobenzaldehyde</td>
<td>151.00</td>
<td>0.44</td>
</tr>
<tr>
<td>5</td>
<td>2-Methoxybenzaldehyde</td>
<td>136.15</td>
<td>0.32</td>
</tr>
</tbody>
</table>
SECTION-4.8: CHARACTERIZATION OF THE COMPOUNDS OF SERIES-11 (AU-51 TO AU-55) OF SCHEME-3.

Compound AU-51

Molecular formula: $\text{C}_{24}\text{H}_{20}\text{N}_{4}\text{O}_{3}\text{S}$

Elemental analysis:

<table>
<thead>
<tr>
<th>Found (%)</th>
<th>Calculated (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>64.80</td>
</tr>
<tr>
<td>H</td>
<td>4.47</td>
</tr>
<tr>
<td>N</td>
<td>12.59</td>
</tr>
</tbody>
</table>

TLC:

<table>
<thead>
<tr>
<th>Solvent systems</th>
<th>$R_f$ values</th>
</tr>
</thead>
<tbody>
<tr>
<td>CHCl$_3$: CH$_3$OH (9:1 v/v)</td>
<td>0.70</td>
</tr>
<tr>
<td>CHCl$_3$: CH$_3$OH (8:2 v/v)</td>
<td>0.76</td>
</tr>
</tbody>
</table>

M.p. ($^\circ$C): 132-134

Recrystallization solvent: Acetone

IR: $3330$ (NH str. in CONH), 1552, 1486, 1127 and 743 (aromatic ring), $2987$ (N-CH-S), $3065$, $1552$, $1486$, and $1450$ (N-CH$_2$), $1713$ (>C=O, cyclic), $1680$, $1655$ (>C=O, amide), $1630$ (>C=CH-Ar), $1610$ (>C=N), $1228$ (C-N str.), $1037$ (N-N str.) and $692$ (C-S-C).

$^1$HNMR: $8.63$ (s, 1H, CONH), $8.15$ (t, 1H, CONH), $7.45$-8.36 (m, 9H, Ar-H), $5.12$ (s, 1H, >C=CH-Ar), $4.14$ (s, 1H, -N-CH$_2$) and $2.60$ (s, 2H, N-CH$_2$).

$^{13}$CNMR: $172.2$ (>C=O, cyclic), $170.20$, $167.40$ (CONH), $150.90$, $150.41$ (C near N of pyridine ring), $129.1$, $125.2$ (>C=CH-Ar), $128.20$-132.61 (C of aromatic ring), $121.85$, $121.60$ and $121.40$ (C of pyridine ring), $41.5$ (NCHS) and $39.40$ (N-CH$_2$).


Chemical name: 5-Benzylidene-2-phenyl-3-[(isonicotinamid-4-yl) acetamido]-4-oxo-1,3-thiazolidine.
Compound AU-52

Molecular formula : $\text{C}_{24}\text{H}_{18}\text{N}_{4}\text{O}_{3}\text{S}\text{Cl}_{2}$

Elemental analysis : 

<table>
<thead>
<tr>
<th>Found (%)</th>
<th>Calculated (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>H</td>
</tr>
<tr>
<td>56.10</td>
<td>3.47</td>
</tr>
<tr>
<td>56.13</td>
<td>3.51</td>
</tr>
</tbody>
</table>

TLC : Solvent systems & $R_f$ values

<table>
<thead>
<tr>
<th>Solvent systems</th>
<th>$R_f$ values</th>
</tr>
</thead>
<tbody>
<tr>
<td>CHCl$_3$ : CH$_3$OH (9:1 v/v)</td>
<td>0.81</td>
</tr>
<tr>
<td>CHCl$_3$ : CH$_3$OH (8:2 v/v)</td>
<td>0.88</td>
</tr>
</tbody>
</table>

M.p. (°C) : 153-154

Recrystallization solvent : Chloroform

IR : 

- 3328 (NH str. in CONH), 3061, 1555, 1488, 1129 and 745 (aromatic ring), 2988 (N-CH-S), 2927, 1460 and 1445 (N- CH$_2$), 1714 (>C=O, cyclic), 1679, 1658 (>C=O, amide), 1631 (>C=CH-Ar), 1611 (>C=N), 1226 (C-N str.), 1039 (N-N str.), 744 (Ar-Cl) and 691 (C-S-C).

$^1$HNMR : 

- 8.64 (s, 1H, CONH), 8.16 (t, 1H, CONH), 7.46-8.39 (m, 8H, Ar-H), 5.13 (s, 1H, >C=CH-Ar), 4.15 (s, 1H, -N-CH-) and 2.61 (s, 2H, N-CH$_2$).

$^{13}$CNMR : 

- 172.3 (>C=O, cyclic), 170.21, 167.43 (CONH), 150.92, 150.42 (C near N of pyridine ring), 133.8, 133.2 (C-Cl, aromatic), 129.2, 125.7 (>C=CH-Ar), 128.21-132.64 (C of aromatic ring), 121.88, 121.67 and 121.45 (C of pyridine ring), 41.6 (NCHS) and 39.45 (N-CH$_2$).

Mass : 


Chemical name : 5-(4-Chlorobenzylidene)-2-(4-chlorophenyl)-3-[(isonicotinamid-4-yl) acetamido]-4-oxo-1,3-thiazolidine.
Compound AU-53

**Molecular formula**: $\text{C}_{24}\text{H}_{18}\text{N}_4\text{O}_3\text{SBr}_2$

**Elemental analysis**

<table>
<thead>
<tr>
<th>Element</th>
<th>Found (%)</th>
<th>Calculated (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>47.80</td>
<td>47.83</td>
</tr>
<tr>
<td>H</td>
<td>2.94</td>
<td>2.99</td>
</tr>
<tr>
<td>N</td>
<td>9.27</td>
<td>9.30</td>
</tr>
</tbody>
</table>

**TLC**

- Solvent systems: $\text{CHCl}_3 : \text{CH}_3\text{OH}$ (9:1 v/v) $R_f = 0.86$
- Solvent systems: $\text{CHCl}_3 : \text{CH}_3\text{OH}$ (8:2 v/v) $R_f = 0.92$

**M.p. ($^\circ\text{C}$)**

- 144-145

**Recrystallization solvent**

- Methanol

**IR**

- 3332 (NH str. in CONH), 3064, 1553, 1487, 1128 and 746 (aromatic ring), 2990 (N-CH-S), 2926, 1456 and 1443 (N-CH$_2$), 1717 (>C=O, cyclic), 1683, 1659 (>C=O, amide), 1636 (>C=CH-Ar), 1615 (>C=N), 1227 (C-N str.), 1038 (N-N str.), 694 (C-S-C) and 618 (Ar-Br).

**$^1$HNMR**

- 8.67 (s, 1H, CONH), 8.18 (t, 1H, CONH), 7.41-8.40 (m, 8H, Ar-H), 5.15 (s, 1H, >C=CH-Ar), 4.18 (s, 1H, -N-CH-) and 2.64 (s, 2H, N-CH$_2$).

**$^{13}$CNMR**

- 172.5 (>C=O, cyclic), 170.24, 167.45 (CONH), 150.93, 150.42 (C near N of pyridine ring), 129.5, 125.4 (>C=CH-Ar), 128.25-132.65 (C of aromatic ring), 121.87, 121.59 and 121.43 (C of pyridine ring), 118.4, 118.1 (C-Br, aromatic), 41.9 (NCHS) and 39.46 (N-CH$_2$).

**Mass**

- 602 [M$^+$], 574, 524, 496, 481, 467,439, 424, 396, 393, 315, 287, 272, 258, 230, 215, 178, 163, 135, 121, 106 and 78.

**Chemical name**

- 5-((3-Bromobenzylidene)-2-(3-bromophenyl)-3-[[isonicotinamid-4-yl] acetamido]-4-oxo-1,3-thiazolidine.
Compound AU-54

Molecular formula : $\text{C}_{24}\text{H}_{18}\text{N}_{6}\text{O}_{7}\text{S}$

Elemental analysis :

<table>
<thead>
<tr>
<th>Elemental analysis</th>
<th>Found (%)</th>
<th>Calculated (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>53.87</td>
<td>53.92</td>
</tr>
<tr>
<td>H</td>
<td>3.33</td>
<td>3.37</td>
</tr>
<tr>
<td>N</td>
<td>15.68</td>
<td>15.73</td>
</tr>
</tbody>
</table>

TLC:

Solvent systems | $R_f$ values |
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>CHCl$_3$ : CH$_3$OH (9:1 v/v)</td>
<td>0.82</td>
</tr>
<tr>
<td>CHCl$_3$ : CH$_3$OH (8:2 v/v)</td>
<td>0.89</td>
</tr>
</tbody>
</table>

M.p. (°C) : 147-148

Recrystallization solvent : Acetone

IR:

3331 (NH str. in CONH), 3067, 1556, 1489, 1130 and 752 (aromatic ring), 2995 (N-CH-S), 2923, 1459 and 1442 (N-CH$_2$), 1718 (>C=O, cyclic), 1685, 1662 (>C=O, amide), 1633 (>C=CH-Ar), 1618 (>C=N), 1525, 1340 (Ar-NO$_2$), 1225 (C-N str.), 1041 (N-N str.) and 698 (C-S-C).

$^1$HNMR:

8.68 (s, 1H, CONH), 8.21 (t, 1H, CONH), 7.43-8.37 (m, 8H, Ar-H), 5.17 (s, 1H, >C=CH-Ar), 4.19 (s, 1H, -N-CH-) and 2.66 (s, 2H, N-CH$_2$).

$^{13}$CNMR:

172.4 (>C=O, cyclic), 170.28, 167.46 (CONH), 150.93, 150.47 (C near N of pyridine ring), 140.5, 140.1 (C-NO$_2$, aromatic), 129.6, 125.7 (>C=CH-Ar), 128.22-132.66 (C of aromatic ring), 121.91, 121.70 and 121.42 (C of pyridine ring), 41.8 (NCHS) and 39.52 (N-CH$_2$).

Mass:

534 [M$^+$], 506, 456, 428, 413, 399, 371, 359, 356, 328, 281, 253, 238, 224, 196, 181, 178, 163, 135, 121, 106 and 78.

Chemical name:

5-(3-Nitrobenzylidene)-2-(3-nitrophenyl)-3-[(isonicotinamid-4-yl)acetamido]-4-oxo-1,3-thiazolidine.
Compound AU-55

Molecular formula : \( \text{C}_{26}\text{H}_{24}\text{N}_{4}\text{O}_{3}\text{S} \)

Elemental analysis : 

\[
\begin{array}{ccc}
\text{C} & \text{H} & \text{N} \\
\text{Found} (%) & 66.02 & 5.04 & 11.83 \\
\text{Calculated} (%) & 66.08 & 5.08 & 11.86 \\
\end{array}
\]

TLC : Solvent systems

\[
\begin{array}{l}
\text{CHCl}_{3} : \text{CH}_{3}\text{OH} (9:1 \text{ v/v}) & 0.84 \\
\text{CHCl}_{3} : \text{CH}_{3}\text{OH} (8:2 \text{ v/v}) & 0.90 \\
\end{array}
\]

M.p. (\(\degree\text{C}\)) : 190-192

Recrystallization solvent : Chloroform

IR : 3335 (NH str. in CONH), 3069, 1558, 1491, 1132 and 750 (aromatic ring), 2996 (N-CH-S), 2935 (Ar-CH\(_3\)), 2920, 1458 and 1437 (N-CH\(_2\)), 1721 (>C=O, cyclic), 1680, 1663 (>C=O, amide), 1637 (>C=CH-Ar), 1619 (>C=N), 1224 (C-N str.), 1043 (N-N str.) and 690 (C-S-C).

\(^1\text{HNMR} : 8.70 (s, 1H, CONH), 8.18 (t, 1H, CONH), 7.45-8.39 (m, 8H, Ar-H), 5.20 (s, 1H, >C=CH-Ar), 4.20 (s, 1H, N-CH\(_{2}\)), 2.67 (s, 2H, N-CH\(_{2}\)) and 2.25 (s, 6H, Ar-CH\(_{3}\)).

\(^{13}\text{CNMR} : 172.5 (>\text{C}=\text{O}, \text{cyclic}), 170.29, 167.49 (\text{CONH}), 150.94, 150.44 (\text{C near N of pyridine ring}), 129.6, 125.4 (>\text{C}=\text{CH}-\text{Ar}), 128.26-132.68 (\text{C of aromatic ring}), 121.94, 121.70 and 121.51 (\text{C of pyridine ring}), 41.7 (\text{NCHS}), 39.53 (\text{N-CH}_{2}) and 22.9, 22.4 (\text{Ar-CH}_{3}).


Chemical name : 5-(4-Methylbenzylidene)-2-(4-methylphenyl)-3-[(isonicotinamid-4-yl) acetamido]-4-oxo-1,3-thiazolidine.

It is interesting to mention that all the reactions under microwave irradiation were completed within 1-4 minutes giving excellent yields whereas similar reactions under conventional heating (steam bath) at reflux for 1.5-5 hours gave lower yields as compared to microwave method. The impact of microwave irradiation and conventional heating for the synthesis of compounds AU-36 to AU-55 have been compared (Table 4.9.1).

Table 4.9.1: Comparison of conventional and microwave synthesis for AU-36 to AU-55.

<table>
<thead>
<tr>
<th>Compounds</th>
<th>Conventional</th>
<th>Microwave*</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Yield (%)</td>
<td>Time (hours)</td>
</tr>
<tr>
<td>AU-36</td>
<td>71</td>
<td>3</td>
</tr>
<tr>
<td>AU-37</td>
<td>67</td>
<td>2.5</td>
</tr>
<tr>
<td>AU-38</td>
<td>70</td>
<td>2</td>
</tr>
<tr>
<td>AU-39</td>
<td>72</td>
<td>2.5</td>
</tr>
<tr>
<td>AU-40</td>
<td>62</td>
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<tr>
<td>AU-41</td>
<td>69</td>
<td>5</td>
</tr>
<tr>
<td>AU-42</td>
<td>70</td>
<td>3</td>
</tr>
<tr>
<td>AU-43</td>
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<td>4.5</td>
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<td>AU-44</td>
<td>75</td>
<td>2.5</td>
</tr>
<tr>
<td>AU-45</td>
<td>68</td>
<td>3</td>
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<tr>
<td>AU-46</td>
<td>68</td>
<td>4</td>
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<tr>
<td>AU-47</td>
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<td>3</td>
</tr>
<tr>
<td>AU-48</td>
<td>70</td>
<td>2.5</td>
</tr>
<tr>
<td>AU-49</td>
<td>73</td>
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<td>64</td>
<td>4</td>
</tr>
<tr>
<td>AU-51</td>
<td>69</td>
<td>2</td>
</tr>
<tr>
<td>AU-52</td>
<td>68</td>
<td>2</td>
</tr>
<tr>
<td>AU-53</td>
<td>71</td>
<td>2</td>
</tr>
<tr>
<td>AU-54</td>
<td>72</td>
<td>1.5</td>
</tr>
<tr>
<td>AU-55</td>
<td>66</td>
<td>2.5</td>
</tr>
</tbody>
</table>

* Microwave irradiation power = 800W