PART II

GENERAL INTRODUCTION
GENERAL INTRODUCTION

In 1928, Feigl (21) used 5-(p-dimethylaminobenzylidene) rhodanine for the detection of silver. Since that time rhodanine derivatives have been used as reagents for qualitative and quantitative determination of various ions (55, 56). Rhodanine β-substituted propionic acid has been found to be an insecticide (27).

3-Allylrhodanine and 5-isonitroso-3-allyl rhodanine possess anthelmintic activity in vitro against T. aceti (44). The latter paralysed ascaris but other 5-benzylidene derivatives were lethal to Fasciola hepatica (45). Rhodanine derivatives, including metallic derivatives, benzylidene derivatives and compounds containing phenothiazine residues, have been tested for their anthelmintic activity against Ascaris lumbricoides and F. hepatica (46).

5-(p-Dimethylaminobenzylidene) rhodanine has been investigated with regard to its effect on Mycobacterium tuberculosis, Trichophyton gypseum, Torulopsis minor, S. aureus and S. paratyphi (57).

Rhodanine when alkylated in the methylene carbon atom, has been shown by Leonard (40) to possess pharmacological properties of the same type as the barbituric acid derivatives.
5,5-Dimethylrhodanine has been found to possess anticonvulsant activity in rabbits, against Metrazol shock (16).

5,5-Dimethyl and 5,5-diethylrhodanines have been found to possess sedative action (19, 20, 40).

The antithyroid activity of rhodanine has been compared with that of thiouracil (14).

Rhodanines contain in their structure the grouping,

\[
-N-C-S-
\]

present in many plant fungicides like tetramethylthiouram-disulphide and the salts of dithiocarbamic acid, and possess also a carbonyl group conjugated with an ethylenic linkage found in another type of fungicides (24). Rhodanines with acidic substituents attached to N and also their condensation products with aldehydes and ketones possess appreciable fungicidal activity, when tested by the method of Montgomery and Moore (49), the percentage of inhibition being cent per cent (52).

Many derivatives of rhodanine have shown fungitoxic or bacteriostatic activity (7)(9). 5-(1-Methylalkylidene) rhodanines and 5-(1-methyl-2-alkylthioethylidene) rhodanines have been tested for activity against A. niger (8). With this organism, the former series shows a peak of activity, where R is ethyl or n-propyl, while maximum activity in the latter series occurs, when R is methyl. The type of linkage of
sulphur to carbon may affect the microbiological activity of the molecule. In addition to sulphur in the thiazolidine ring, rhodanine contains a thione group which may tautomerize to a sulphhydryl group. Replacement of the thione group by a carbonyl group, as in 2,4-thiazolidinedione, gives a molecule in which less tendency for tautomerization is present.

In the rhodanine derivatives, the most effective compounds are those in which the alkyl group has two or three carbon atoms (5).

Some 5-substituted rhodanines exhibit mildew preventing activity (10).

3-(p-Chlorophenyl)-5-methylrhodanine N, 244 and 3-(p-chlorophenyl)-5-ethylrhodanine are useful as effective insecticides, fungicides and nematocides (4). They also reduce the nematode population without harming the plants or imparting an off-flavour to the fruit (62). Aqueous emulsion of N, 244 completely controls the growth of the parasite-Meloidogyne incognita (63).

Allan et al., (1) considered the sodium salt of 2-(3-ethyl-5-rhodaninylidene methyl) benzenesulphonate as fungicide while the salts of the transition metals as possible analytical reagents. Raval and Trivedi (53) have prepared some 3-benzyl-5-alkyl/aryl rhodanines.
Brown et al., (12) converted 3-substituted rhodanines to their 5-isonitroso derivatives by reaction with isopropyl nitrite and hydrochloric acid. The 3-benzyl-5-isonitroso-rhodanine containing a chlorine substituent in the para position of the benzyl group, shows considerable increase in the fungistatic activity over the corresponding 3-chlorinated benzylrhodanine, while the accompanying loss in the bacteriostatic activity is relatively slight. Such enhancement is not produced by the presence of 5-ethoxymethylene or 5-dimethylaminomethylene substituents in 3-(p-chlorobenzyl)rhodanine.

\[
\begin{align*}
\text{S} & \quad \text{N} \quad \text{CH}_2 \\
\text{S=O} & \quad \text{N} \quad \text{CH}_2
\end{align*}
\]

Allan et al., (2) condensed rhodanine and its derivatives with aromatic aldehydes containing iodine and reported the products as potential fungicides and germicides.

Garraway (23) reported the fungicidal activity of some 3-substituted rhodanines and their thiazine analogues.
Kharidia and Trivedi (32) have prepared 3-(substituted benzyl)-5-alkyl rhodanines. Shroff and Trivedi (59) have synthesized 3-(aryloxyalkyl)rhodanines.

3-Aminorhodanine and its derivatives are useful as effective tuberculostatic and fungistatic agents (67).

5-Ethylidenerhodanine (47), 3-(α-carboxy-Y-methyl-butyl)rhodanine (35), substituted 5-(5-phenyl-2-furfurylidene)rhodanines (37), 3-furfurylrhodanine and its 5-arylidene derivatives (64), α-(di-(N-rhodanyl)caproic acid (33), 3-(α-carboxypropyl)rhodanine and its 5-arylidene derivatives (39), 3-β-carboxyethylrhodanine and their derivatives of oxo compounds not belonging to aromatic aldehydes (66), 3-(α-Carboxy-3-indolyl)ethylrhodanine (34), 3-γ-carboxypropylrhodanine (31), 3-(p-hydroxyphenyl)rhodanine (38) have been synthesized.

5-(5-Nitrofurfurylidene)-3-ethyl-4-oxo-2-thioxothiazolidine possesses antibacterial activity (50).

3-(3,4-Dichlorophenyl)-5-alkylrhodanine has been found to possess excellent ascaricidal or molluscacidal properties (17). 3-Phenylrhodanines are useful as antimalarial agents (68). 3-(4-Alkoxyphenyl)-5-alkylrhodanines have been found to possess insecticidal properties (13).
3-(3,4-Dichlorophenyl)-5-methylrhodanines exhibit anthelmintic and molluscicidal effects (29).

5-(5-nitro-2-thienylidene)rhodanine and 5-[5-nitro-furyl)] acrylidene rhodanine have been shown to possess biological activity (65).

3-Aryl-5-(2-methylbutyl)-2-mercapto-4-thiazolidinones exhibit anticonvulsant activity (58).

3-Arylaminorhodanines possess antimicrobial properties (61).

Substituted 5-(phenyl-2-furfurylidene)rhodanines have been found to be physiological active (36).
PRESENT WORK

Very few bis rhodaninyls seem to have been prepared. It was therefore of considerable interest to study bis rhodaninyls. With this in view bis rhodaninyls have been prepared by condensation of various dithiocarbamates with \( \alpha,\beta \)-dibromosuccinic acid.

\[ (A) \text{Bis} \left[ 3\text{-arylrhodaninyl-(5)} \right]; \]

\[
\begin{align*}
X, \quad \text{C}_6\text{H}_4\text{NH} & \quad \text{HOO} \quad \text{COOH} & \quad \text{HN} \cdot \text{C}_6\text{H}_4 \cdot \text{X} \\
\text{S} = \text{C} & \quad \text{CH} & \quad \text{CH} & \quad \text{Br} & \quad \text{Br} & \quad \text{H}_4\text{NS} \\
\text{S} \quad \text{NH}_4 & \quad \text{Br} & \quad \text{Br} & \quad \text{H}_4\text{NS}
\end{align*}
\]

Where \( X = \text{H}; \quad \text{o-m-p-CH}_3; \quad \text{o-m-p-Cl}; \quad \text{p-Br}; \quad \text{o-p-CH}_3 \)
\( \text{p-OC}_2\text{H}_5; \quad \text{p-OC}_3\text{H}_7; \quad \text{p-OC}_4\text{H}_9; \quad 2,6-(\text{CH}_3)_2 \)

\[ (B) \text{Bis} \left[ 3\text{-benzylrhodaninyl-(5)} \right]; \]

\[
\begin{align*}
X, \quad \text{C}_6\text{H}_4\cdot \text{CH}_2\cdot \text{NH} & \quad \text{HOO} \quad \text{COOH} & \quad \text{HN} \cdot \text{C}_6\text{H}_4 \cdot \text{X} \\
\text{S} = \text{C} & \quad \text{CH} & \quad \text{CH} & \quad \text{C} = \text{S} \\
\text{S} \quad \text{NH}_4 & \quad \text{Br} & \quad \text{Br} & \quad \text{H}_4\text{NS}
\end{align*}
\]
Where $X=H; \, o-Cl; \, o-m-p-\text{CH}_3; \, p-\text{OCH}_3; \, 3,4-(\text{CH}_3)_2$

(C) Bis[3-α/β-aralkylrhodaninyl-(5)]:

(i) $\text{C}_6\text{H}_5\cdot\text{CH}_2\cdot\text{NH} + \text{HOOC} \rightarrow \text{C}_6\text{H}_4\cdot\text{X} \rightarrow \text{C}_6\text{H}_4\cdot\text{X}$

(ii) $\text{C}_6\text{H}_5\cdot\text{CH}_2\cdot\text{CH}_2\cdot\text{NH} + \text{HOOC} \rightarrow \text{C}_6\text{H}_4\cdot\text{X} \rightarrow \text{C}_6\text{H}_4\cdot\text{X}$
PART-II

THEORETICAL

***************
A few important methods for the synthesis of rhodanines are described below:

(A) The most general synthesis of rhodanine involves the reaction of an α-halo acid or ester or acid halide with the salt of a dithiocarbamic acid, the latter being prepared from amine, carbon disulphide and ammonia (3, 6, 20, 28).

\[
\text{R-NH}_2 + \text{CS}_2 + \text{NH}_3 \rightarrow \text{R-NH-} \mid \text{SNH}_4 \\
\text{R-NH-C-SNH}_4 + \text{R'}\text{-CH-COOH} \rightarrow \text{R-N-} \mid \text{CH-COOH}
\]

Ammonium dithiocarbamate

Rhodanine itself is prepared in 44-65 per cent yield from monochloroacetic acid and ammonium dithiocarbamate (Campbell, N., (15); Julian, P.L., (30). Several 3-aryl and 3-benzylrhodanines have been prepared in a similar way by Brown et al., (11).

Bashour (4) has used the above method to prepare 3-(p-chlorophenyl)-5-methyl/ethylrhodanines by the condensation of ammonium dithiocarbamate of p-chloroaniline with α-bromopropionic/n-butyric acids.
5-Carbaloxyrhodanines have been prepared by the condensation of α-bromodialkyl malonate with ammonium dithiocarbamate (18).

Rhodanine-N-salicylic acid and rhodanine-N-benzoic acid have been prepared by the condensation of monochloroacetic acid with the ammonium dithiocarbamate of p-aminosalicylic acid and anthranilic acid respectively (52, 54).
(B) α-Mercapto acids react with organic isothiocyanate to yield N-substituted rhodanines (3).

\[
R\text{-NCS} + \text{HSCH}_2\text{COOH} \rightarrow S=C
\]

(C) α-Thiocyanate acids or their esters yield rhodanine derivatives on treatment with hydrogen sulphide (48) or thiolacetic acid (69).

\[
\text{CH}_2\text{COOC}_2\text{H}_5 + \text{H}_2\text{S} \rightarrow S=C
\]

(D) 2-Imino-4-thiazolidinone i.e. pseudothiohydantoin is converted into rhodanine by the action of carbon disulphide in alcohol at 160°C (48).

(E) Condensation of primary amines with dicarboxymethyl trithiocarbonate yields N-substituted rhodanines (28).

\[
R\text{-NH}_2 + S=C \rightarrow S=C + \text{SHCH}_2\text{COOH}
\]
The free amine or its hydrochloride with sufficient base for neutralisation is refluxed with dicarboxymethyl trithiocarbonate. Ring closure is affected during the same operation.

Several benzothiazolyi rhodanines have been prepared by the condensation of appropriately substituted amino benzothiazoles and dicarboxymethyl trithiocarbonate (70).

Rhodanine has been prepared by the reaction of mono-chloroacetic acid with ammonium thiocyanate (51), by the action of ethyl chloroacetate upon ammonium dithiocarbamate in the presence of alcohol and hydrogen chloride (48), by saturating solution of thioglycolic acid and potassium thiocyanate in absolute alcohol with hydrogen chloride (22), and by ring closure of thiocarbamyl-thioglycolic acid in various ways (25, 28, 30).

Homologues of rhodanine such as 5-phenyl and 3,5-dimethyl-rhodanines have been prepared similarly (20), while N-alkyl/aryl derivatives i.e 3-methyl and 3-phenylrhodanines have been synthesized by heating mercaptoacetic acid with methyl and phenyl isothiocyranates respectively.

On account of the presence of reactive methylene group, the rhodanines have been condensed with various aldehydes to yield corresponding benzal derivatives (26) or with ketones (8).
Rhodanine has been condensed with ethyl orthoformate in presence of acetic anhydride giving 5-ethoxymethylene rhodanine, which condenses with primary and secondary amines giving the corresponding 5-aminomethylrhodanines (41,42,43).

Rhodanine-β-substituted propionic acid has been prepared by the condensation of rhodanine and β-propiolactone (27).

\[
\text{HOOC-CH}_{2}-\text{CH}_{2}-\text{S-C} \quad \text{NH} \quad \text{CO}
\]

Bis [3-arylrhodaninyl-(5)], bis [3-benzylrhodaninyl-(5)] and bis [3-α/β-aralkylrhodaninyl-(5)] have been prepared by the condensation of appropriate ammonium dithiocarbamate with α,β-dibromosuccinic acid.

Where: \(X=H; \ o-m-p-\text{CH}_3; \ o-m-p-\text{Cl}; \ p-\text{Br}; \ o-p-\text{OCH}_3; \ p-\text{OCH}_2\text{H}_5; \ p-\text{OCH}_3\text{H}_7; \ p-\text{OCH}_4\text{H}_9; \ 2,6-(\text{CH}_3)_2\)
Where: $X=H; \text{o-Cl}; \text{o-m-p-CH}_3; \text{p-OCH}_3; 3,4-(\text{CH}_3)_2$

(iii) (a)
Compounds prepared are shown in the tables.
<table>
<thead>
<tr>
<th>No.</th>
<th>X</th>
<th>M.P. °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>H</td>
<td>145</td>
</tr>
<tr>
<td>2.</td>
<td>o-CH₃</td>
<td>172</td>
</tr>
<tr>
<td>3.</td>
<td>m-CH₃</td>
<td>117</td>
</tr>
<tr>
<td>4.</td>
<td>p-CH₃</td>
<td>182</td>
</tr>
<tr>
<td>5.</td>
<td>o-Cl</td>
<td>121</td>
</tr>
<tr>
<td>6.</td>
<td>m-Cl</td>
<td>118</td>
</tr>
<tr>
<td>7.</td>
<td>p-Cl</td>
<td>178</td>
</tr>
<tr>
<td>8.</td>
<td>p-Br</td>
<td>164</td>
</tr>
<tr>
<td>9.</td>
<td>o-OCH₃</td>
<td>198d</td>
</tr>
<tr>
<td>10.</td>
<td>p-OCH₃</td>
<td>186</td>
</tr>
<tr>
<td>11.</td>
<td>p-O₂C₂H₅</td>
<td>172</td>
</tr>
<tr>
<td>12.</td>
<td>p-n-O₂C₃H₇</td>
<td>137</td>
</tr>
<tr>
<td>13.</td>
<td>p-n-O₂C₄H₉</td>
<td>168</td>
</tr>
<tr>
<td>14.</td>
<td>2,6- (CH₃)₂</td>
<td>212</td>
</tr>
</tbody>
</table>
TABLE 2.2

BIS [3-BENZYLRODINYL]-(5):

```

<table>
<thead>
<tr>
<th>No.</th>
<th>X</th>
<th>M.P. °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>H</td>
<td>146</td>
</tr>
<tr>
<td>2.</td>
<td>o-Cl</td>
<td>205d</td>
</tr>
<tr>
<td>3.</td>
<td>o-CH₃</td>
<td>178</td>
</tr>
<tr>
<td>4.</td>
<td>m-CH₃</td>
<td>126</td>
</tr>
<tr>
<td>5.</td>
<td>p-CH₃</td>
<td>180</td>
</tr>
<tr>
<td>6.</td>
<td>p-OCH₃</td>
<td>192</td>
</tr>
<tr>
<td>7.</td>
<td>3,4-(CH₃)₂</td>
<td>220</td>
</tr>
</tbody>
</table>
```

Diagram of the structure: [Chemical structure image]
### TABLE Z.3

(a) **BIS 3-α-PHENYLETHYL RHODAMINE B-5**:  

<table>
<thead>
<tr>
<th>No.</th>
<th>M.P. °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>245</td>
</tr>
</tbody>
</table>

(b) **BIS 3-β-PHENYLETHYL RHODAMINE B-5**:  

<table>
<thead>
<tr>
<th>No.</th>
<th>M.P. °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>117</td>
</tr>
</tbody>
</table>
PART - II

EXPERIMENTAL

*******************************

#
EXPERIMENTAL

p-n-Alkoxyanilines, benzyl amines, α-phenylethyl amine and α,β-dibromosuccinic acid required for the synthesis of bis rhodaninyls were prepared as described in Experimental Part-I.

β-phenylethyl amine was of Eastman practical grade.

GENERAL METHOD FOR THE PREPARATION OF:

(A) BIS [3-ARYL RHODANINYL-(5)] .
(B) BIS [3-BENZYL RHODANINYL-(5)] .
(C) BIS [3-α/β-ARALKYL RHODANINYL-(5)] .

Carbon disulphide (10.0 g., 0.13 mole) and the appropriate amine (0.1 mole) were added within fifteen minutes to liquor ammonia (16.0 ml.) placed in a conical flask cooled in an ice bath. The ammonium dithiocarbamate formed was filtered and washed with cold ether.

α,β-Dibromosuccinic acid (0.05 mole) was dissolved in sodium hydroxide solution (10.0 ml., 40 per cent) and to this, sodium carbonate was added till bastic. The ammonium dithiocarbamate, as prepared above, was added during ten minutes with constant stirring to the solution of sodium salt of α,β-dibromosuccinic acid, which was cooled to 5-10°C. Stirring was continued, while the flask was allowed to attain room temperature. Concentrated hydrochloric acid (34.0 ml.) was added and the mixture was heated at 85-90°C
for thirty minutes. A yellow oil was obtained, which solidified on cooling. These bis rhodaninyls (yield about 50-60 per cent) were crystallized from alcohol. (Brown, F. C., Bradsher, C. K., Morgan, E. C., Tetenbaum, M., and Wilder, F., J. Am. Chem. Soc., 78, 384, 1956).

The compounds prepared are shown in the tables.
TABLE - 2.4

BIS 3-ARYL RHODANINYL-(5)

\[
\begin{align*}
\text{No.} & : \quad \text{Compounds} \quad (X-) & : \quad \text{M.P.} \quad \text{M.F.} & : \quad \text{Molecular formula} & : \quad \text{Per cent Sulphur} \\
1. & : \quad H & : \quad 145 & : \quad C_{18}H_{12}N_2O_2S_4 & : \quad 30.7 & : \quad 30.8 \\
2. & : \quad o-CH_3 & : \quad 172 & : \quad C_{20}H_{16}N_2O_2S_4 & : \quad 28.8 & : \quad 28.8 \\
3. & : \quad m-CH_3 & : \quad 117 & : \quad C_{20}H_{16}N_2O_2S_4 & : \quad 28.6 & : \quad 28.8 \\
4. & : \quad p-CH_3 & : \quad 182 & : \quad C_{20}H_{16}N_2O_2S_4 & : \quad 28.7 & : \quad 28.8 \\
5. & : \quad o-Cl & : \quad 121 & : \quad C_{18}H_{10}N_2O_2S_4Cl_2 & : \quad 26.3 & : \quad 26.4 \\
6. & : \quad m-Cl & : \quad 118 & : \quad C_{18}H_{10}N_2O_2S_4Cl_2 & : \quad 26.3 & : \quad 26.4
\end{align*}
\]
<table>
<thead>
<tr>
<th>No.</th>
<th>Compounds (X=)</th>
<th>M.P.</th>
<th>Molecular formula</th>
<th>Found</th>
<th>Required</th>
</tr>
</thead>
<tbody>
<tr>
<td>7.</td>
<td>p-Cl</td>
<td>178</td>
<td>C_{18}H_{10}N_{2}O_{2}S_{4}Cl_{2}</td>
<td>26.4</td>
<td>26.4</td>
</tr>
<tr>
<td>8.</td>
<td>p-Br</td>
<td>164</td>
<td>C_{18}H_{10}N_{2}O_{2}S_{4}Br_{2}</td>
<td>26.4</td>
<td>26.4</td>
</tr>
<tr>
<td>9.</td>
<td>o-CH_{3}</td>
<td>198d</td>
<td>C_{20}H_{16}N_{2}O_{4}S_{4}</td>
<td>26.8</td>
<td>26.8</td>
</tr>
<tr>
<td>10.</td>
<td>p-CH_{3}</td>
<td>186</td>
<td>C_{20}H_{16}N_{2}O_{4}S_{4}</td>
<td>26.8</td>
<td>26.8</td>
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<tr>
<td>11.</td>
<td>p-OCH_{3}</td>
<td>172</td>
<td>C_{20}H_{16}N_{2}O_{4}S_{4}</td>
<td>25.3</td>
<td>25.4</td>
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<td>12.</td>
<td>p-OCH_{2}H_{5}</td>
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<td>C_{22}H_{24}N_{2}O_{4}S_{4}</td>
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<td>13.</td>
<td>p-OCH_{2}H_{9}</td>
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<td>C_{24}H_{28}N_{2}O_{4}S_{4}</td>
<td>22.7</td>
<td>22.9</td>
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<td>14.</td>
<td>2,6-(CH_{3})<em>{2}C</em>{6}H_{5}</td>
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<td>C_{22}H_{24}N_{2}O_{4}S_{4}</td>
<td>27.0</td>
<td>27.1</td>
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<tr>
<td>No.</td>
<td>Compounds</td>
<td>M.P. (°C)</td>
<td>Molecular formula</td>
<td>Per cent Sulphur</td>
<td></td>
</tr>
<tr>
<td>-----</td>
<td>-------------------</td>
<td>-----------</td>
<td>-------------------</td>
<td>------------------</td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>H</td>
<td>146</td>
<td>C₂₀H₁₆N₂O₂S₄</td>
<td>28.7</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>o-Cl</td>
<td>205d</td>
<td>C₂₀H₁₄N₂O₂S₄Cl₂</td>
<td>24.9</td>
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</tr>
<tr>
<td>3</td>
<td>p-CH₃</td>
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<td>27.0</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>m-CH₃</td>
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</tr>
<tr>
<td>5</td>
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<td></td>
</tr>
<tr>
<td>6</td>
<td>p-OCH₃</td>
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</tr>
<tr>
<td>7</td>
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<td>C₂₄H₂₄N₂O₂S₄</td>
<td>25.5</td>
<td></td>
</tr>
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</table>
### Table 2.6

**A** BIS [3-α-Phenylethyl RHODANTHYL-(5)]

<table>
<thead>
<tr>
<th>No.</th>
<th>M.P.</th>
<th>Molecular formula</th>
<th>Per cent Sulphur</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>°C</td>
<td></td>
<td>Found</td>
</tr>
<tr>
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**B** BIS [3-α-Phenylethyl RHODANTHYL-(5)]

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<th>M.P.</th>
<th>Molecular formula</th>
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* PART - II *
* REFERENCES *
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REFERENCES:


3. Andreasch, R., and Zipser, A., Monatsh. Chem., 24, 499 (1903); ibid., 25, 159 (1904); ibid., 26, 1191 (1905); ibid., 29, 399 (1908); ibid., 31, 785 (1910); ibid., 38, 121 (1917); ibid., 39, 419 (1918).


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