PART-II
Section A

Synthesis of 3-aryl-4-aryl sulphonyl-5-aryl/hetaryl isoxazolines
INTRODUCTION

A good deal of importance is being given to isoxazoline derivatives due to their wide use in medicinal chemistry. The isoxazole ring system, as its name indicates, is one containing a nitrogen and an oxygen atom adjacent to each other in the five membered cycle.

The structure can be represented as follows:

\[
\begin{array}{c}
\text{N} \\
\text{O} \\
\text{5} \\
\text{4} \\
\text{3}
\end{array}
\]

Although isoxazole derivatives have known for more than eighty years, the investigation of their chemistry commenced rather slowly. Cresol obtained the isoxazole in the year 1884 by the action of hydroxyl amine hydrochloride (NH₂OH HCl) on benzoylaceton. Dihydroisoxazoles or isoxazolines are biologically important compounds. Theoretically, three isoxazolines are possible depending on the location of a double bond. i) 2-Isozazoline, ii) 3-isoxazoline, iii) 4-isoxazoline, as shown below:

\[
\begin{array}{c}
\text{(i)} \\
\text{(ii)} \\
\text{(iii)}
\end{array}
\]
2-Isoxazolines are comparatively more stable and much important.

Until recently, no natural product containing the isoxazole ring was known, except the glycosides, hipilagin isolated in 1920 from Malphighiacea hipatogo madobalata\(^2\). In the year 1935, Bula et al\(^3\) isolated the antibiotic, cycloserin or oxamycin from streptomycyes or orchideneus, a simple derivative of 4-aminoisoxazolidone. It is a well known broad spectrum antibiotic and is found useful in the treatment of tuberculosis\(^4\) and leprosy\(^5\). Similarly, a number of isoxazole derivatives have been shown to possess potential, antibacterial, antitubercular, antifungal and anti-viral activities\(^6\). 3,4-Dimethyl-5-sulphanilamido isoxazole\(^7\) (Gastrin) was found to be therapeutically active and is used as drug even today. Anilidoisoxazolines synthesised by Khalil and others\(^8\) were found to possess remarkable bactericidal activity against some gram +ve and gram -ve bacteria. A number of isoxazoline derivatives have been used as photosensitizer and supersensitizer\(^9,10\). Recently, it was observed by Donald\(^11\) that pregnann 16, 17 d-2-isoxazolines were useful for fertility control and pregnancy maintenance. Some substituted isoxazolines have been reported as herbicides and plant growth regulants\(^12\). Many of them are used for the control of plant phytopathogens\(^13\). Antimicrobial and antifungal activities have been reported.
in 3-methyl-4-(4'-bromo-2'-methyl benzene azo)-5-isoxazolines by Mittal and others\textsuperscript{14}. A number of isoxazoline-3-one derivatives have been synthesized as anti-inflammants\textsuperscript{15} and analgesics\textsuperscript{16}.

Keeping this in view the attempt was made to synthesize some new 4-aryl sulphonyl isoxazolines which may have significant chemotherapeutic activities.

**Methods for the preparation of isoxazolines**

A number of methods can be used for the preparation of isoxazolines. The most widely used methods are discussed here.

a) **From Isoxazoles**

Isoxazoles may be converted to 5-hydroxy isoxazolines. This transformation is essentially the formation of pseudo base\textsuperscript{17}. The reaction can be represented in Fig. II\textsubscript{A}-1.

b) **From Nitro compounds**

Isoxazoline oxides can be prepared from properly substituted nitro compound as shown in Fig. II\textsubscript{A}-2.

c) **From \(\beta\)-haloketones**

When a \(\beta\)-haloketone\textsuperscript{19} is treated with hydroxyl amine in alkaline solution, the isoxazoline is obtained by the displacement of halocatom by an anion of the normal ketoxime.
**METHOD - I**

\[
\begin{align*}
CH_3 & \quad \text{H} & \quad \text{C} & \quad \text{H}_5 \\
\text{H}_5 & \quad \text{C} & \quad \text{H}_5 & \quad \text{N} \\
\text{H}_5 & \quad \text{C} & \quad \text{O} & \quad \text{H}_5 \\
\text{H}_5 & \quad \text{C} & \quad \text{H}_5 & \quad \text{N} \\
\text{H}_5 & \quad \text{C} & \quad \text{H}_5 & \quad \text{N} \\
\text{H}_5 & \quad \text{C} & \quad \text{H}_5 & \quad \text{N} \\
\text{H}_5 & \quad \text{C} & \quad \text{H}_5 & \quad \text{N} \\
(\text{Fig IIA-1})
\end{align*}
\]

**METHOD - II**

\[
\begin{align*}
\text{H}_5 & \quad \text{C} & \quad \text{CH} = \text{CH} & \quad \text{CO} & \quad \text{H}_5 \\
\text{H}_5 & \quad \text{C} & \quad \text{CH} = \text{CH} & \quad \text{CO} & \quad \text{H}_5 \\
\text{H}_5 & \quad \text{C} & \quad \text{CO} & \quad \text{H}_5 \\
\text{H}_5 & \quad \text{C} & \quad \text{CO} & \quad \text{H}_5 \\
\text{H}_5 & \quad \text{C} & \quad \text{CO} & \quad \text{H}_5 \\
\text{H}_5 & \quad \text{C} & \quad \text{CO} & \quad \text{H}_5 \\
\text{H}_5 & \quad \text{C} & \quad \text{CO} & \quad \text{H}_5 \\
(\text{Fig IIA-2})
\end{align*}
\]

**METHOD - III**

\[
\begin{align*}
\text{CH}_3 & \quad \text{N} & \quad \text{CH}_2 & \quad \text{CH}_2 & \quad \text{CH} & \quad \text{C} & \quad \text{CH}_5 \\
\text{CH}_3 & \quad \text{N} & \quad \text{CH}_2 & \quad \text{CH}_2 & \quad \text{CH} & \quad \text{C} & \quad \text{CH}_5 \\
\text{CH}_3 & \quad \text{N} & \quad \text{CH}_2 & \quad \text{CH}_2 & \quad \text{CH} & \quad \text{C} & \quad \text{CH}_5 \\
\text{CH}_3 & \quad \text{N} & \quad \text{CH}_2 & \quad \text{CH}_2 & \quad \text{CH} & \quad \text{C} & \quad \text{CH}_5 \\
\text{CH}_3 & \quad \text{N} & \quad \text{CH}_2 & \quad \text{CH}_2 & \quad \text{CH} & \quad \text{C} & \quad \text{CH}_5 \\
\text{CH}_3 & \quad \text{N} & \quad \text{CH}_2 & \quad \text{CH}_2 & \quad \text{CH} & \quad \text{C} & \quad \text{CH}_5 \\
(\text{Fig IIA-3})
\end{align*}
\]
A similar reaction takes place when the oxime of mannich base is treated with aqueous alcoholic sodium hydroxide. In this case trialkylamine is displaced instead of halogen. The reaction can be written as shown in Fig. II-A-3.

d) From Olefins

The reaction of olefins with nitrile oxide is a general method for preparing isoxazolines. This synthesis is successful with benzonitrile oxide and varied olefins such as allyl alcohol, vinyl chloride, styrene, stilbene and eugenol (Fig. II-A-4).

e) From $\alpha,\beta$-unsaturated ketones

Generally isoxazolines are prepared by this method. It involves the reaction of an $\alpha,\beta$-unsaturated ketone with hydroxylamine in basic medium. The mechanism is not definitely established. Both 1,4- and 1,2 addition of hydroxylamine may take place depending on the experimental conditions. Van Auwers et al. have reported that the isoxazolines are not directly formed by cyclisation of unsaturated ketoximes. The theory of 1,4-addition of hydroxylamine to $\alpha,\beta$-unsaturated ketone was formerly accepted by Barnes and coworkers. But recently, they have proposed the 1,2 addition mechanism for the formation of 2-isoxazolines. Intermediate products (addition) formed by 1,2-addition of hydroxylamine to unsaturated ketones are decomposed to have isoxazolines. The structural and
METHOD - IV

\[
\begin{align*}
\text{HO} & \text{N} \equiv \text{C} \equiv \text{N} \equiv \text{O} \\
+ \\
\text{HO} & \text{C} \equiv \text{C} \equiv \text{CH}_2 \equiv \text{OH} \\
\rightarrow \\
\text{HO} & \text{C} \equiv \text{CH}_2 \equiv \text{CH}_2 \equiv \text{OH}
\end{align*}
\]

\[
(Fig \text{IIA-4})
\]

\[
\begin{align*}
\text{R} & \text{C} \equiv \text{CH} \equiv \text{CH} \equiv \text{Ar} + \text{H}_2 \text{NOH} \text{ HCl} \\
\text{H}_2 \text{ addition} \\
\text{R} & \text{C} \equiv \text{CH} \equiv \text{CH} \equiv \text{Ar} \quad \text{1:4 addition}
\end{align*}
\]

\[
\begin{align*}
\text{R} & \text{C} \equiv \text{CH} \equiv \text{CH} \equiv \text{Ar} \\
\text{R} & \text{C} \equiv \text{CH} \equiv \text{CH} \equiv \text{Ar} \\
\text{R} & \text{C} \equiv \text{CH} \equiv \text{CH} \equiv \text{Ar}
\end{align*}
\]

\[
(Fig \text{IIA-5})
\]
spectral evidences have also supported the 1,2 addition of hydroxylamine. A mechanism involving 1,4 addition was shown to be inconsistent with the experimental results by Blatt. The isoxazoline formation in basic or neutral medium is reported. Some workers have reported the synthesis of isoxazolines in presence of strong mineral acids. Recently Aziz claimed the formation of 2-isoza-
zolines from $\alpha,\beta$-unsaturated ketone by 1,2 addition mechanism in basic medium (Fig. II A-5).

**PRESENT WORK**

Few 2-isoazolines containing heterocyclic substituents have been studied recently by Aziz. Some 2-isoazolines have been reported by Padhye. But there is no report on the synthesis of 2-isoazolines having sulphonyl moiety at 4-position. Therefore, it was thought worthwhile to synthesize some new isoxazolines because of their use in medicinal chemistry.

In the present work 3-aryl-4-aryl sulphonyl-5-aryl- isoazolines were synthesized by the interaction of $\alpha,\beta$-unsaturated ketones (described in Section B of Part-I) and hydroxylamine in presence of sodium acetate. This method is easy to carry out, the yields are better and the $\alpha,\beta$-unsaturated ketones were readily available.
**Present Work**

\[
\begin{align*}
R &- \text{C} & -\text{SO}_2 - R' \\
\text{NH}_2\text{OH} & \rightarrow & \text{NaCOOCH}_3 \\
& \downarrow & \\
R &- \text{C} & -\text{SO}_2 - \text{H} & - R' \\
\end{align*}
\]

Where,

\[R = \text{H, } \text{CH}_3, \text{ Cl}\]

\[R' = \text{H, } \text{CH}_3, \text{ Cl}\]

\[R'^* = \text{Phenyl, Hetaryl}\]

(Fig II.6)
**General method for the synthesis of 3-(substituted phenyl)-4-(aryl sulphonyl)-5-aryl isoxazolines**

A mixture of 1-aryl-3-aryl-2-aryl sulfonyl-2-propene-1-one (0.01 mol) and hydroxylamine hydrochloride (0.015 mol) and sodium acetate (0.02 mol) was refluxed in ethylalcohol for 5-6 hrs. After cooling, the reaction mixture was poured into ice-cold water. The solid thus obtained was filtered, washed with water and crystallised from proper solvent. Purity of the product was checked by TLC. The structures of the compounds were established by IR, UV and elemental analysis. The M.p's, yields, solvent for crystallisation and nitrogen analysis are given in Table II_A-1.
Discussion of IR spectra

The IR of some of the isoxazolines were scanned on Perkin-Elmer infracord in nujol mull. Following frequencies are observed for different groups.

The absorption bands at 1590-1610 cm\(^{-1}\) is due to \(\text{-C} = \text{N}\) of isoxazolines. The \(\text{-SO}_2\) group shows absorption bands at 1320-1360 cm\(^{-1}\) and 1160-1190 cm\(^{-1}\) due to asymmetric and symmetric stretching. All the compounds showed characteristic bands of isoxazoline ring at 1460, 1250 are due to \(\text{N} = \text{C}\). (IR. SP. No. II A-1)

Discussion of U.V.

The ultra violet spectra of the some isoxazolines were taken in CHCl\(_3\).

The bands at 260 nm indicate the conversion of chalcones into isoxazolines.
Discussion of NMR

The NMR spectra of few representative compounds were studied in CDCl₃ on varian FT-80A spectrophotometer using TMS as internal standard. The compound with spectrum No. 4 was assigned the structure 3-(4'-methyl phenyl)-4-sulfonyl phenyl-5-thienyl-isoxazoline from the following PMR data. Chemical shift in δ scale (ppm) 2.5 (S-3H-CH₃), 4.7 (2H CH-CH of isoxazoline ring), 7-8 (m 12H - aromatic protons). (NMR SP NO 1A-1)
Table II\textsubscript{A\textsuperscript{-1}}

3-(Substituted phenyl)-4-(aryl sulphenyl)-5-aryl-isoazolines (Fig. II\textsubscript{A\textsuperscript{-6}})

<table>
<thead>
<tr>
<th>R</th>
<th>R'</th>
<th>R''</th>
<th>M.P. (°C)</th>
<th>Yield (%)</th>
<th>Percentage of Nitrogen</th>
<th>Cal.</th>
<th>Found</th>
</tr>
</thead>
<tbody>
<tr>
<td>H</td>
<td>H</td>
<td>2-Hydroxy phenyl</td>
<td>130</td>
<td>40</td>
<td>3.69</td>
<td>3.47</td>
<td></td>
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<tr>
<td>H</td>
<td>H</td>
<td>4-Chloro phenyl</td>
<td>127</td>
<td>52</td>
<td>3.52</td>
<td>3.51</td>
<td></td>
</tr>
<tr>
<td>H</td>
<td>H</td>
<td>4-Methoxy phenyl</td>
<td>112</td>
<td>44</td>
<td>3.57</td>
<td>3.32</td>
<td></td>
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<tr>
<td>H</td>
<td>H</td>
<td>Phenyl</td>
<td>125</td>
<td>52</td>
<td>3.85</td>
<td>3.55</td>
<td></td>
</tr>
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<td>H</td>
<td>H</td>
<td>4-Pyridyl</td>
<td>125</td>
<td>47</td>
<td>7.67</td>
<td>7.28</td>
<td></td>
</tr>
<tr>
<td>H</td>
<td>H</td>
<td>2-Thienyl</td>
<td>143</td>
<td>52</td>
<td>3.79</td>
<td>3.85</td>
<td></td>
</tr>
<tr>
<td>CH\textsubscript{3}</td>
<td>H</td>
<td>Phenyl</td>
<td>151</td>
<td>56</td>
<td>3.71</td>
<td>3.65</td>
<td></td>
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<tr>
<td>CH\textsubscript{3}</td>
<td>H</td>
<td>2-Hydroxy phenyl</td>
<td>95</td>
<td>61</td>
<td>3.55</td>
<td>3.43</td>
<td></td>
</tr>
<tr>
<td>CH\textsubscript{3}</td>
<td>H</td>
<td>4-Chloro phenyl</td>
<td>150</td>
<td>63</td>
<td>3.40</td>
<td>3.49</td>
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<table>
<thead>
<tr>
<th>R</th>
<th>R'</th>
<th>R&quot;</th>
<th>M.P. (°C)</th>
<th>Yield (%)</th>
<th>Percentage of Nitrogen</th>
</tr>
</thead>
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<tr>
<td></td>
<td></td>
<td></td>
<td></td>
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<td>Cal.</td>
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<tr>
<td>CH₃</td>
<td>H</td>
<td>4-Methoxy phenyl</td>
<td>150</td>
<td>47</td>
<td>4.54</td>
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<td>CH₃</td>
<td>H</td>
<td>2-Chloro phenyl</td>
<td>145</td>
<td>58</td>
<td>3.40</td>
</tr>
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<td>CH₃</td>
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<td>2-Thienyl</td>
<td>162</td>
<td>55</td>
<td>3.66</td>
</tr>
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<td>CH₃</td>
<td>CH₃</td>
<td>Phenyl</td>
<td>110</td>
<td>66</td>
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<td>CH₃</td>
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<td>73</td>
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<td>CH₃</td>
<td>CH₃</td>
<td>2-Furyl</td>
<td>140</td>
<td>80</td>
<td>3.67</td>
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<tr>
<td>CH₃</td>
<td>CH₃</td>
<td>4-Pyridyl</td>
<td>160</td>
<td>64</td>
<td>6.90</td>
</tr>
<tr>
<td>CH₃</td>
<td>CH₃</td>
<td>2-Thienyl</td>
<td>162</td>
<td>62</td>
<td>3.54</td>
</tr>
</tbody>
</table>

All above compounds are crystallised from EtOH
EXPERIMENTAL

Preparation of 3-(4'-methyl phenyl)-4-(4''-methyl-phenyl sulphonyl)-5-phenylisoxazoline

A mixture of 1-(4 methyl phenyl)-3phenyl2-(4''-methyl phenyl sulphonyl)-2-propene-1-one (0.01 mol, 3.6 gms), hydroxylamine (1.03 gm, 0.015 mol) and sodium acetate (0.02 mol, 1.64 gms) in 25 ml of ethanol was refluxed for 5 to 6 hours. The reaction mixture was cooled and poured into ice water. Solid obtained was filtered, washed and crystallized from ethanol.

M.P. 110°C, yield 66 %.

Similarly other compounds of the series were prepared (Table II_A-1).
Section B

Synthesis of N-phenyl-3-(aryl/hetaryl)-4-aryl-sulphonyl-3-aryl pyrazolines
INTRODUCTION

A large number of substituted 1,3,5-triphenyl-2-pyrazolines and some related compounds have been synthesized. Their derivatives are fluorescent compounds which are widely used for the whitening of textile fibers, plastics and paper. Donald E Rivett\(^1\) described the preparation, absorption and fluorescence properties of a number of 1,3,5-triphenyl-2-pyrazolines. Biological activities, medicinal importance and methods of preparation of pyrazolines are discussed in detail in Part I (Section C).

PRESENT WORK

Survey of literature revealed that attempts were not made to synthesize aryl sulphonyl pyrazolines. It was thought likely that its association with sulfonyl moiety and additional phenyl ring would impart some changes in the activities of the present nucleus. Here we report synthesis of \(N\)-phenyl-5-(aryl/heteraryl)-4-aryl sulfonyl-3-aryl pyrazolines. In the present work the reaction of chalcones (Section B of Part I) with phenyl hydrazine was carried out under acidic conditions.
PRESENT WORK

\[
\begin{align*}
R & \quad \text{C} \quad \text{C} \quad \text{SO}_2 \quad \text{R} \\
\text{ACOH} & \quad \text{Ph-NH-NH}_2 \\
\downarrow & \\
\begin{array}{c}
R \quad \text{C} \quad \text{C} \quad \text{SO}_2 \\
\text{R} \quad \text{N} \quad \text{N} \quad \text{R} \\
\text{R} \quad \text{C} \quad \text{C} \quad \text{SO}_2 \\
\text{R} \quad \text{R} \end{array}
\end{align*}
\]

Where,

\[
\begin{align*}
R & = H, CH_3 \\
R' & = H, CH_3, Cl. \\
R'' & = Phenyl, Hetaryl
\end{align*}
\]

(Fig.IIB-1)
General procedure for the preparation of N-phenyl-5-(aryl/heteraryl)-4-aryl sulphonyl-3-aryl pyrazoline

0.5 moles of chalcone, 10 ml of glacial acetic acid, 2 moles of phenyl hydrazin in 25 ml alcohol were heated under reflux for 4 to 6 hours. The solution was diluted with water after cooling. The solid was filtered, washed and crystallized from a proper solvent (Fig. II_B-1).

The physical constants, crystallisation solvents, % yields and the result of elemental analysis are tabulated in Table II_B-1. These compounds gave negative test for chalcones and did not give any precipitate with 2,4-dinitrophenyl hydrazine indicating that the reaction has taken place at $\text{C} - \text{CH} = \text{CH}-$ grouping. The structures were further confirmed by IR, UV and TLC.
Discussion of IR spectra

The IR spectra of some of the representative compounds from the series were recorded on Perkin-Elmer infracord in mull.

Sharp bands at 1690 to 1610 indicate the presence of C=N. The absorption due to $-\text{SO}_2-$ group is observed from 1300 to 1390 cm$^{-1}$ and 1090 to 1150 cm$^{-1}$ for asymmetric and symmetric stretching vibrations. All the compounds showed characteristic bands of pyrazoline ring. The absorption bands at 1210 to 1270 cm$^{-1}$ are due to N=N=C of pyrazoline. (IR SP No.-II3-1)

Discussion of U.V.

The ultraviolet spectra of some of the compounds under study were taken in CHCl$_3$.

Bands at 245 nm, 290 nm and 345 nm were correlated with chalcones. The bands indicate cyclisation of chalcones into pyrazoline.
Table II$_B$-1

N-Phenyl-5-(aryl/hetaryl)-4-aryl sulphonyl-3-aryl pyrazolines (Fig. II$_B$-1)

<table>
<thead>
<tr>
<th>R</th>
<th>R'</th>
<th>R''</th>
<th>M.P. (°C)</th>
<th>Yield (%)</th>
<th>Percentage of Nitrogen</th>
<th>Cal.</th>
<th>Found</th>
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<tbody>
<tr>
<td>H</td>
<td>H</td>
<td>Phenyl</td>
<td>165</td>
<td>70</td>
<td>6.39</td>
<td>6.24</td>
<td></td>
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<tr>
<td>H</td>
<td>H</td>
<td>2-Hydroxy phenyl</td>
<td>171</td>
<td>66</td>
<td>6.16</td>
<td>6.09</td>
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<th>R''</th>
<th>M.P. (°C)</th>
<th>Yield (%)</th>
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</tr>
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<td>4-Chloro phenyl</td>
<td>165</td>
<td>57.8</td>
<td>5.76</td>
</tr>
<tr>
<td>H</td>
<td>CH3</td>
<td>4-Pyridyl</td>
<td>183</td>
<td>62.5</td>
<td>9.33</td>
</tr>
<tr>
<td>H</td>
<td>CH3</td>
<td>2-Thienyl</td>
<td>172</td>
<td>60</td>
<td>6.11</td>
</tr>
</tbody>
</table>
EXPERIMENTAL

Synthesis of 3-phenyl-4-phenylsulfonyl-5-(4'-methyl phenyl) pyrazoline

A mixture of 1-(4'-methyl phenyl)-3-phenyl2-phenyl sulfenyl-2-propene-1-one (0.01 mol, 1.7 gms) and phenyl hydrazine (0.015 mol, 0.8 gm) in ethanol (25 ml) and acetic acid (10 ml) was treated under reflux for 5-6 hours. The reaction mixture was concentrated, cooled and poured into ice cold water. The solid separated was filtered, washed with water and crystallised from ethanol.

M.P. 145°C, yield 58 %.

Other compounds of this series were prepared by following the above procedure (Table IIa-1).
REFERENCES


22. D' Aleontres and Grunanger, Gazz. Chim. et al. 80, 741 (1950); 80, 831 (1900).

23. von Auwers and Muller, J. Prack. Chem., 137, 102 (1933).


