DETERMINATION OF THIOUREA AND ITS DERIVATIVES WITH BROMINE MONO-CHLORIDE REAGENT BY MICRO-METHOD

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Abstract- A survey of literature reveals that bromine mono-chloride has not been used for the determination of these compounds on micro scale. More a method has been developed for the determination of thio-urea with the use of bromine mono-chloride (BrCl) reagent.

KEYWORDS: Thio-urea, Bromine monochloride, Micro scale method.

I. INTRODUCTION-
Thio-urea are generally used as preservatives, insecticides, rodenticides and in the pharmaceutical preparation. Many Thiourea derivatives posses antibacterial and antipyretics properties as well. They are of grate value in the characterization of Organic Compounds and are used in dyes, photographic film, plastics and textile industry and for the manufacture of chemical deposited radiation – detector materials. In these operation it is often necessary know the purity of particular thioureas derivatives sample and therefore, a suitable method for their easy would of grate value. A number of methods have been proposed for the determination of Thioureas. Yadav and Jain performed coulometric estimation of Thiourea. Joshi demonstrated that tetravalent selenium can be used as quantitative oxidant for Thiourea.

PRESENT WORK
A survey of literature reveals that bromine mono-chloride has not been used for the determination of these compounds on micro scale. More a method has been developed for the determination of thiourea with the use of bromine mono-chloride (BrCl) reagent. Most of the methods described above need sophisticated instruments and drastic reaction conditions; therefore, it was thought to develop a simple, quick and convenient method for the determination of the some Thio-urea and its derivatives.

Using Thiourea as the test sample, the effect of temperature was observed that the best recovery of the sample was obtained at a temperature of boiled water bath give best result of 40 °C (Table-I). 40 °C temperature for their complete oxidation and Reaction time 15 Minutes and the concentration was varied from 0.10N to 0.40 N and the Recovery of the sample was calculated. It was found that the best recovery was obtained by using 0.3 N concentration of the reagent.

RESULT AND DISCUSSION:
As is evident from the table the recommended procedure micro determination of Thiourea, recovery of the Sample is...
accurate up to 10 mg. of the sample size but for the general procedure 1-5 mg. sample rise is sufficient. Thiourea was taken as the representative compound to study the effect of various variables on the reaction.

POSSIBLE COURSE OF REACTION

While studying the reaction time, it was observed that Thiourea derivatives containing electron with drawing group like Naphthyl, Phenyl etc. are readily oxidized due to weakening of C=S bond by these groups thus Naphthyl Thiourea is oxidised in the following manner like phenyl Thiourea.

\[
\text{Naphthyl Thiourea} + 8 \text{BrCl} \rightarrow \text{Naphthyl Urea} + 8 \text{H}_2\text{O} + 8 \text{Cl}^-
\]

Similarly, Ally thiourea reacts with eight equivalent of Bromine mono chloride giving rise to corresponding urea and Sulphuric acid.

\[
\text{Ally thiourea} + 8 \text{BrCl} \rightarrow \text{Ally Urea} + 8 \text{H}_2\text{S}_4\text{O}_4
\]

Based on above observation of a general reaction for the oxidation of all these Thiourea compound may be written in the following form.

\[
\text{R - NH - C - NH}_2 + 8 \text{BrCl} \rightarrow \text{R - NH - C - NH}_2 + 8 \text{H}_2\text{O} + 8 \text{Cl}^-
\]

Where \( R = \text{H, C6 H5, CH2=CH- CH2-} \) Groups

As indicated in stiochiometry table o- ditolyl Thiourea consume 16 equivalent of bromine mono chloride (BrCl) it may passing is oxidised in the following manner.

\[
\text{O} \quad \text{BrCl} \quad 8 \text{O} \quad \text{N} \quad \text{BrCl} \quad 8 \text{Naphtyl thiourea} \quad \text{Ally thiourea} \quad \text{Naphtyl urea} \quad \text{Ally urea} \quad \text{Sulphuric acid}
\]

It was observe that the process of Urea and it derivatives due inform in the determination.

GENREL PROCEDURE

Aliquots containing 1-5 mg of the sample were taken in a 100-ml. Erlenmeyer flask followed by the addition of 6 ml of 0.3 N Bromine mono chloride reagents and 5 ml. of 10% Sulphuric acid. The reaction contents were shaken gently and kept on a boiling water bath for 20 minutes. After the reaction was over, the reaction mixture was cooled to room temperature. The unconsumed bromine mono chloride reagent was titrated against 0.025 N ferrous ammonium sulphate using N-Naphthyl anthranilic acid as an indicators. A blank experiment was also run under identical condition using all the reagent except the sample. Discovery of the sample was calculated by following expression.

\[
\text{Mg. of the Sample} = \frac{M \times N \times (B - S)}{n}
\]

Where -\( M = \text{Molecular weight of the sample} \)
-\( N = \text{Molarity of Ferrous Ammonium Sulphate} \)
-\( B = \text{Volume of Ferrous Ammonium Sulphate} \)
-\( S = \text{Volume of Ferrous Ammonium Sulphate} \)

\( \text{consume to titrate the blank Experiment.} \)

\( \text{S} = \text{Volume of Ferrous Ammonium Sulphate} \)

\( \text{consume to titrate the sample Experiment.} \)
N = Number of mole of Bromine mono chloride reagent consumed per mole of the sample.

TABLE - 1: Micro determination of thiourea and its some derivatives with recommended procedure with 0.3N Bromine Mono Chloride reagent

<table>
<thead>
<tr>
<th>Aliquots taken (ml.)</th>
<th>Amount present(mg.)</th>
<th>Reaction time (Min)</th>
<th>Amount Recover(mg.)</th>
<th>Morality</th>
<th>Error %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thiourea</td>
<td>1.0000</td>
<td></td>
<td>0.9975</td>
<td>8</td>
<td>-0.25</td>
</tr>
<tr>
<td></td>
<td>3.0000</td>
<td>15</td>
<td>3.0066</td>
<td>5.0120</td>
<td>+0.22</td>
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<tr>
<td></td>
<td>5.0000</td>
<td></td>
<td>5.0045</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Phenyl Thiourea</td>
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<td></td>
<td>1.0042</td>
<td>8</td>
<td>+0.42</td>
</tr>
<tr>
<td></td>
<td>3.0000</td>
<td>15</td>
<td>2.0046</td>
<td>2.9900</td>
<td>-0.10</td>
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<tr>
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<td></td>
<td>4.9900</td>
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<tr>
<td>Á-Naphthyl thiourea</td>
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<td></td>
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<tr>
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<td>3.0000</td>
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<td>4.9900</td>
<td>-0.45</td>
</tr>
<tr>
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<td>5.0000</td>
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<td>-0.20</td>
</tr>
<tr>
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<td>1.0120</td>
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<tr>
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<td>20</td>
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<td>5.0335</td>
<td>+0.40</td>
</tr>
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<td>+0.42</td>
</tr>
<tr>
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<td></td>
<td>+0.24</td>
</tr>
<tr>
<td>õ-di–tolyl thiourea</td>
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<td></td>
<td>0.9975</td>
<td>16</td>
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<tr>
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<td></td>
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<td></td>
<td></td>
<td></td>
<td>-0.20</td>
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</tbody>
</table>

REFERENCE-

2. Erik Wernersson, Björn Stenqvist, Mikael Lund Cellulose 2015 Article ASAP.