Levene and Senior\textsuperscript{1} give a method used by them for the preparation of large quantities of guanidine. Their process, which is not new, seems satisfactory but the purity of the product when tested is not over 95 per cent.

For the past year we have been using a large amount of guanidine in various forms, and being unable to obtain any of the salts which usually come from Germany, we tried some of the various methods of preparation given in Beilstein’s \textit{Handbuch}, and adopted the following which not only gives a good yield but also a product of great purity. The method seems to have been first suggested by Delitsch and Volhard,\textsuperscript{2} but they give no details. Guanidine may be obtained by heating dry ammonium thiocyanate in a short-necked Jena flask for 20 hours at a temperature from 190–200°C. For this purpose an iron or copper oil bath is necessary, using fairly heavy oil so as to ensure a temperature within the range stated.

\textit{Method.}

About 100 gm. of dry ammonium thiocyanate are placed in the flask and immersed in the oil, which should be above the level of the crystals. The heat is then raised gently until the required point is reached. When the 20 hours have expired, the mass is allowed to cool, extracted with hot water, and filtered. The filtrate, which contains the guanidine thiocyanate, is evaporated

\textsuperscript{*} The expenses connected with this work were defrayed by a grant from the Medical Research Committee.


Fig. 2. Crystals of guanidine carbonate, produced by the process described. Camera lucida drawing. X 20.
to dryness and weighed, and for every one part of the thiocyanate present, 0.58 part of potassium carbonate is added, with sufficient water for solution. The whole is then evaporated once more to dryness. The potassium thiocyanate is then removed by repeated treatment with 90 per cent ethyl alcohol, decanting off after each addition, until a drop of the supernatant liquid fails to give a red color with perchloride of iron. Crude guanidine carbonate is obtained by this means, as it is insoluble in alcohol. It can be further purified by dissolving in a small quantity of water and adding 90 per cent alcohol to about five or six times the original volume, and allowing to settle, when pure guanidine carbonate crystallizes out.

Free guanidine may be obtained from this salt by treatment with the theoretical quantity of dilute sulfuric acid and barium hydroxide, but for convenience in use and preservation, the carbonate is the best salt.

Yield.—100 gm. of ammonium thiocyanate yielded 15 to 20 gm. of pure guanidine carbonate.

**Tests of Purity.**

1. Precipitation with sodium picrate: 0.1 gm. of prepared salt gave 0.158 gm. of picrate; theoretical 0.160 gm.
2. Nitrogen content of guanidine picrate formed.

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<thead>
<tr>
<th></th>
<th>Calculated for</th>
<th>Found:</th>
</tr>
</thead>
<tbody>
<tr>
<td>CH₅N₅O₃Na</td>
<td>a.</td>
<td>b.</td>
</tr>
<tr>
<td>N</td>
<td>29.11</td>
<td>29.50</td>
</tr>
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</table>

3. Titration with 0.1 N sulfuric acid: 1 cc. 0.1 N H₂SO₄ = 0.009 gm. guanidine carbonate. 0.18 gm. taken (indicator, methyl orange) required 20.01 cc. of acid; i.e., 20.01 X 0.009 = 0.18009 gm. guanidine carbonate.
THE PREPARATION OF GUANIDINE
J. Smith Sharpe


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