The examination of substances which are insoluble in dilute acids and in aqua regia necessitates the use of special reagents which convert them into compounds which are soluble in the ordinary solvents (water or dilute acids). They can then be identified by the usual tests. In earlier texts it was recommended that the portion which remained after treatment with aqua regia should be fused with a mixture of sodium carbonate and potassium nitrate; the fused mass was extracted with water and filtered, and the solution was tested for anions such as sulphate, silicate, chromate, chloride or fluoride. The residue was dissolved in hydrochloric acid and the solution examined for metals such as calcium, strontium, barium, silver, aluminium and tin. Titanium compounds were fused with potassium hydrogen sulphate and the fused material was extracted with water to give a solution of titanium sulphate. In later schemes of analysis various preliminary tests were made on the insoluble portion. These included heating on charcoal, the microcosmic bead test, boiling with sodium hydroxide solution and the removal of lead and silver salts. The substance was then fused with sodium carbonate.

It was felt that a re-examination of the methods of treatment of insoluble substances was needed, because there did not appear to exist a systematic semi-micro scheme of analysis for these substances. In addition, little was known of the relative efficiency of the various fluxes commonly used in the fusion.

The present scheme attempts to remedy these deficiencies. It is a revision and extension of the method of treatment of insoluble substances at present included in the Advanced Scheme of the Midlands Association.

for Qualitative Analysis. The scheme covers only a selected number of insoluble substances, but in due course it is hoped to extend it to include others.

After the insoluble substance has been isolated, a number of preliminary tests, including an acid fusion, are carried out. Because a large number of such tests, including many spot methods, are used, it is recommended that they be done on the original material except where it is necessary to use the insoluble portion itself. The subsequent systematic analysis of the insoluble portion may then be completed with as little as 50 mg of the original material. The relatively large number of preliminary tests is justified because the insoluble material can often be quickly identified by these tests and confirmed in the later systematic analysis.

The preliminary tests are followed by the removal of silver salts, if these have been shown to be present, from the insoluble portion, which is then treated with sodium hydroxide solution. The residue is then boiled with sodium carbonate solution which converts a number of insoluble substances into metal carbonates. Substances which remain after this treatment are finally brought into solution by fusion with a mixture of sodium carbonate and sodium nitrate or by fusion with sodium peroxide.

The list of substances which were examined includes most of the well known "insolubles". These were:

- Insoluble silver salts: AgCl, AgBr, AgI, AgCNS;
- Insoluble sulphates: PbSO₄, BaSO₄, SrSO₄;
- Ignited oxides: Fe₂O₃, Al₂O₃, Cr₂O₃, SnO₂;
- Other oxides: WO₃, MoO₃, TiO₂, ZrO₂, SiO₂;
- Others: Cu₂Fe(CN)₆, CaF₂, Si, SiC, W, BN, fused PbCrO₄, and mineral silicates.

1. Isolation of the insoluble material

Procedure

Boil 50 mg of the original material with 2 ml of 4 N nitric acid for 2 minutes. Centrifuge

<table>
<thead>
<tr>
<th>Residue</th>
<th>Solution</th>
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<tbody>
<tr>
<td>Add 5 drops of aqua regia and boil for 1 minute*. Centrifuge</td>
<td>Evaporate half of the liquid to dryness. If there is a residue, dissolve in water containing 2 drops of 4 N nitric acid and examine for cations</td>
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</tbody>
</table>

* If black pyrolusite is present, it will only dissolve slowly in aqua regia. Continue boiling until all has dissolved, if necessary adding more aqua regia.