Solid Sample Insertion Systems and L'vov Platform Effect*

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Verschiedene Probenträger werden unter diesen Gesichtspunkten miteinander verglichen. Alle Forderungen an ein solches System können nur erfüllt werden, wenn die Abmessungen des Graphitrohrs ausreichend groß sind, vor allem, um das kritische „handling“ der Feststoffprobe sicher zu ermöglichen.

Summary. A sample insertion system for solids designed for an electrothermal graphite tube atomiser has to provide easy handling of samples and operate as a L'vov platform inside the tube.

The aspects stated are studied with respect to different sample supports. The requirements to the aforementioned systems can be met provided that the graphite tube is sufficiently large, mainly to allow a safe handling of the sample, which is one of the most critical steps of the measurement procedure.

1. Introduction

Ever since the electrothermal graphite furnace AAS has been developed, many approaches have been attempted to analyse solid samples directly. There were, however, several drawbacks which hampered routine application of this technique for a long time. These problems were diminished by a number of new developments during the last years. Direct solid sample analysis by AAS has thus reached a state which allows a simple application in many cases.

Langmyhr [1] reviewed developments and state-of-the-art of this analytical technique.

Technical requirements for the analysis of solid samples may be comprised as follows:

- In order to suppress chemical interferences, samples have to be atomised into a hotter gas atmosphere. The L'vov platform [4] approximates this requirement.
- Solid sample analyses require permanent work close to the atomic absorption spectrometer. An ergonomically projected working place thus becomes necessary, covering spectrometer, balance, data station, and sample insertion system.
- The sample insertion system must be adapted to peculiarities of solid sample handling concerning preparation, dosage, and insertion of samples into the atomising unit.
- Easy handling and good stability of the microbalance are mandatory to profit from the rapidity of solid sample analysis.
- Microprocessor data treatment must meet the requirements of solid sample analysis with respect to hardware and software.
- Samples have to be homogenised thoroughly to obtain representative results. The type of homogenising technique depends on the type of sample as well as the analytical problem to be solved.

This paper will point out those topics concerning chemical interferences and the insertion of solid samples, and examine how far the insertion systems presently offered met the requirements stated.

2. Insertion Systems for Solid Samples

Handling of solid and liquid samples differ significantly from each other. These differences are discussed elsewhere [5] in detail. For the purpose of easy handling, a device to insert solid materials into an electrothermal atomizer has to meet the following demands:

a) The sample aliquot to be measured must be easily deposited on a sample support prior to the weighing step.

b) The insertion system and the atomising unit should fit a large range of weight of <0.1 to >10 mg.

c) Sample support tare must not exceed the electronic weighing range of a modern microbalance.

d) The sample support has to be easily and safely transported from the balance to the atomizer; in particular, seizing and holding and losses by partial dropping of weighed samples are to be considered.

e) The sample has to be inserted free of jerks, reproducibly, avoiding secondary contaminations, and
Table 1. Comparison of properties of different sample supports

<table>
<thead>
<tr>
<th>Criterion</th>
<th>Microboat</th>
<th>Platform boat</th>
<th>Miniature cup</th>
<th>Central probe</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample deposition on sample support</td>
<td>Simple, deposition area is open and bordered. Size 6 mm x 4 mm</td>
<td>Simple, deposition area is open and bordered. Size 7 mm x 4 mm</td>
<td>Open cylindrical vessel, inner diameter 2.5 mm. Sample insertion on the balance tray is difficult, as particles may fall aside</td>
<td>Closed capsule, insertion aperture 3.5 mm x 4.2 mm. Sample deposition cannot be observed, therefore rather difficult</td>
</tr>
<tr>
<td>approx. Maximum weight of organic samples</td>
<td>10 mg</td>
<td>15 mg</td>
<td>1 mg</td>
<td>2 mg</td>
</tr>
<tr>
<td>approx. Weight of sample support</td>
<td>120 mg</td>
<td>130 mg</td>
<td>35 mg</td>
<td>180 mg</td>
</tr>
<tr>
<td>Transport from balance to atomising unit</td>
<td>Safe hold by means of a special claw; simple seizing on the balance from aside</td>
<td>Safe hold by means of a special claw; simple seizing on the balance from aside</td>
<td>No holding appliance; special claw seizes on the inner surface of the vessel, making sample contact possible; claw seizes from top, direct removal from the balance tray is difficult</td>
<td>No holding appliance; Special claw seizes into gas outlet from top, therefore no direct removal from the balance tray; hitherto existing seizing appliance causes jerks on insertion</td>
</tr>
<tr>
<td>Insertion of sample support into graphite tube</td>
<td>Support is pushed through a narrow slit in the tube wall; bumps and sample losses are possible; contamination risk is low because of insertion into the “clean part” of the tube</td>
<td>Axial insertion by means of a mechanical guide free of jerks and reproducible; low contamination risk, safe distance from tube walls on sample insertion</td>
<td>Radial insertion by the enlarged gas outlet; risk of jerks. Contamination risk is low, because sample is inserted into the “clean part” of the tube</td>
<td>Radial insertion; gas is lead through the capsule, minimum allowance between tube and capsule, insertion accompanied by jerks. High contamination risk, as the capsule is lead along the cold graphite pressure parts (condensated analyte)</td>
</tr>
<tr>
<td>Removal of sample residue</td>
<td>Simple on account of open deposition area</td>
<td>Simple on account of open deposition area</td>
<td>Rather difficult on account of the small aperture; additional seizing tool required</td>
<td>Rather difficult, inaccessible deposition area</td>
</tr>
</tbody>
</table>