1987

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Current Calibration Practices for Flame Atomic Absorption Spectrometry
Part 1. Initial Results From a Survey

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Information about 309 different routine analyses by flame atomic absorption spectrometry has been obtained from a questionnaire survey. The information is analysed according to the methods of overcoming interferences, the methods of curve fitting and the reasons for choosing a particular calibration method. The results indicate that most methods suffer from interference effects and that the addition of matrix modifiers or the matching of standards is the most popular approach to overcoming these effects. Manual curve fitting procedures are still widely used and 5-point calibrations are the most used. No clear cut reasons for choice of calibration strategy emerge from this preliminary analysis of the returns to date.

Experimental

Questionnaire
The first version of the questionnaire was sent to members of the committee of the ASG as a pilot survey. Comments and suggestions received from this pilot survey were incorporated into the final version of the questionnaire, which is shown in Fig. 1.

As can be seen from Fig. 1, the questionnaire is divided into a number of sections. These request information about: (1), analyte element, sample type and dissolution procedure (if the sample is not a liquid); (2), treatment of samples following dissolution such as might be used to overcome or compensate for interference effects; (3), the pre-treatment of standards; (4), instrument operating parameters (including sample presentation) and optimisation; (5), method of data acquisition; (6), the fitting of a curve to the calibration data; and (7), any other relevant information. This version was distributed to the 794 UK members of the ASG.

Results and Discussion
So far, 98 questionnaires have been returned, representing information on 309 different analyses. At this stage only information concerning the approach to overcoming interferences, the number of calibration points and the curve fitting method, and the reason for choosing a particular calibration
method is presented and discussed. A full analysis of all of the information will be given in a later publication.

**Approaches to Overcoming Interferences**

The results of section 2 of the questionnaire are given in Fig. 2. From this it can be seen that almost one third of the analyses reported as routine were considered not to have any interference effects. The reagent most often added in this addition in the sample pre-treatment as a means of suppressing substantial number being classified as ionisation suppressants. The reagent most often added in this group was classified as a releasing agent, though with a notable influence on the sample matrix components. Some analyses used a combination of reagent addition and matrix matching. Just over one tenth of the analyses used the standard additions method and about 5% involved a separation step in which the analyte was isolated from other matrix components. Just under one fifth of the analyses made use of the nitrous oxide-acetylene flame, although only half of these involved the use of the addition of an ionisation suppressant. A suspected “outlier” has been omitted from Fig. 3(a). One respondent indicated the use of 25 calibration points together with a manual curve fit method, the over-all strategy being selected for speed.

**Choice of Calibration Method**

Table 1 shows the breakdown of the analyses reported by number of calibration points used with respect to the criteria speed, precision and accuracy. Table 2 shows a similar breakdown of the method adopted to deal with interference effects. No clear pattern emerges from these tables, although there is a slight tendency to the diagonal relationship of the high numbers that might be expected.

<table>
<thead>
<tr>
<th>Number of calibration points</th>
<th>Speed</th>
<th>Precision</th>
<th>Accuracy</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>21</td>
<td>1</td>
<td>12</td>
</tr>
<tr>
<td>2</td>
<td>21</td>
<td>4</td>
<td>18</td>
</tr>
<tr>
<td>3</td>
<td>27</td>
<td>25</td>
<td>36</td>
</tr>
<tr>
<td>4</td>
<td>28</td>
<td>8</td>
<td>28</td>
</tr>
<tr>
<td>5</td>
<td>33</td>
<td>29</td>
<td>43</td>
</tr>
<tr>
<td>6</td>
<td>2</td>
<td>1</td>
<td></td>
</tr>
<tr>
<td>7</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>8</td>
<td></td>
<td>2</td>
<td>2</td>
</tr>
</tbody>
</table>

Many respondents indicated that their choice of calibration was made for reasons of: both speed and accuracy; accuracy and precision; speed, accuracy and precision; and occasionally speed and precision. This is the reason for the rather flat distribution along the rows. Respondents obviously felt that, as far as a choice of calibration strategy was concerned, “speed” and “accuracy” were not necessarily mutually exclusive. It is difficult to avoid the impression that respondents interpreted this section as asking reasons for choosing flame atomic absorption spectrometry. Some respondents commented that the reasons for choosing a particular calibration method were a
judicious balance of all three criteria listed. One other criterion was proposed by a few respondents, namely "simplicity." This might be interpreted as a compromise between speed and accuracy. One or two commented that the choice of calibration strategy depended on the particular problem to be solved. While this is true, the questionnaire sought information about analyses that were considered routine, and thus the decision about the calibration strategy to be adopted should have been taken some time ago, presumably at the time the method was being developed and evaluated.

**Table 2. Numbers of analyses classified by method of approach to dealing with interference effects and reason for choosing the calibration method**

<table>
<thead>
<tr>
<th>Approach to dealing with interferences</th>
<th>Speed</th>
<th>Precision</th>
<th>Accuracy</th>
</tr>
</thead>
<tbody>
<tr>
<td>No pre-treatment</td>
<td>53</td>
<td>23</td>
<td>45</td>
</tr>
<tr>
<td>Matrix modification</td>
<td>59</td>
<td>27</td>
<td>72</td>
</tr>
<tr>
<td>Matrix matching</td>
<td>40</td>
<td>29</td>
<td>65</td>
</tr>
<tr>
<td>Standard additions</td>
<td>7</td>
<td>6</td>
<td>16</td>
</tr>
<tr>
<td>Matrix isolation</td>
<td>9</td>
<td>11</td>
<td>14</td>
</tr>
</tbody>
</table>

It is possible that a clearer picture will emerge concerning the choice of calibration strategies when the types of sample and elements sought are classified as well.

**Conclusions**

The extent to which conclusions can be drawn and generalisations formulated from the results presented here is limited. Firstly, the target sample (members of the ASG) might not be representative of the entire population of AAS users, and secondly, the sub-sample of questionnaires returned could also be unrepresentative. In addition to these limitations, some shortcomings in the questionnaire became apparent when respondents interpreted the questions in different ways. For example, it is clear that not everyone agrees on what constitutes an "addition" in the standard additions method. Some respondents who indicated $n$ additions in section 2, indicated $n$ calibration points in section 6; some indicated $n + 1$ calibration points. It would probably have been sensible to ask about the strategy used for drift correction and re-calibration.

Also, to include, in section 6, a question concerning the use of hard-wired curve-linearisation facilities.

It appears that, as far as routine analyses are concerned, the most frequently determined elements are calcium, copper, lead and zinc. Over two thirds of such analyses involve attempts to overcome interference effects, but of these attempts the standard additions method does not feature to any extent. The same comment can be made about techniques such as solvent extraction, co-precipitation, etc. The use of the nitrous oxide - acetylene flame accounts for about one fifth of routine analyses but the use of an ionisation suppressant with this flame type is by no means automatic. Five calibration points is the most popular number to use, and the use of ruler and flexicurve to draw the calibration curve is still widespread.

The use of instruments' integral computers does not dominate the picture yet.

The authors thank the Trustees of the Analytical Chemistry Trust Fund of the Royal Society of Chemistry for the award of an SAC studentship and members of the Atomic Spectroscopy Group for their co-operation.

The survey continues; anyone wishing to participate should contact the authors for a copy of the questionnaire.

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**Fig. 3. Numbers of analyses classified by the number of calibration points. (a), Normal calibration curves; (b), standard additions method**