Quantitative analysis of minimal aqueous sample by PIXE

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Abstract

For quantitative analysis of liquid sample by PIXE, internal standard method is commonly used by adding a standard solution to the sample to an optimal concentration usually at 5-10 ppm. In order to add the standard accurately with a micropipette, the amount of the sample is favorably more than 1 mL. But some kind of liquid, such as $[^{18}O]H_2O$, the material of F-18 labeled PET pharmaceutical, is too scarce or precious to get enough amount of sample for analysis by the ordinal internal standard method. With the aim of developing techniques for quantitative analysis applicable to minimal sample of such liquid, we have tested the following four methods by analyzing two multi-element standard solutions and examined their accuracy and reproducibility.

1. External standard method for a spot target: the whole target is irradiated with a uniformly adjusted beam and a specific element in the target is firstly quantified by comparing the spectrum with that of equally irradiated standard target. The remaining elements are quantified by taking the element's value as the internal standard.
2. Two-step method: a portion of the minimal sample is firstly analyzed by adding the internal standard of the ordinal amount (usually 10 µL to a 1 mL sample, so it is too much for the minimal portion) to identify the highest element in the sample and obtain its value. Taking the value as the internal standard, the rest sample is analyzed.
3. Standard dilution: the standard solution is diluted by 10 or 100 times to be added accurately to the minimal sample.
4. Standard covering: after drying 5-10 µL of the sample on the backing film, the appropriate amount of the standard solution is dropped to cover the whole residue.

The results were mostly satisfactory with 10-15 per cent accuracy except two-step method where the self absorption of indium, the excessively added internal standard, affects the measurement. For external standard method and covering method, correct adjustment and positioning of the beam, and precise location of the standard dropping are found to be crucial respectively. As both the methods require no sample conditioning, they are expected to be applicable even to a sample of less than 10µL. Standard dilution method with a 100 times diluted solution is shown to be most stable in accuracy and reproducibility. The ordinal internal standard procedure using optimally diluted solution is supposed to be most advantageous though it takes more amount of sample than the former two methods.