Supelco®
Analytical Products

Application Note

Identification of matrix effects by means of standard addition

This method is especially suited for samples where the analyte content lies in the lower half of the measuring range for the corresponding test kit.

Experimental Background

As a rule, a sample contains not only the substance to be analyzed (the analyte), but also further components (foreign substances, accompanying substances). Their interference can, under given circumstances, be so great that the recovery of the analyte deviates from 100% to a significant degree. For this reason, a variety of foreign substances have been investigated to define the concentration up to which they may be present in samples without their interfering with the determination in question. These limits are stated in the instruction for use of the Spectroquant®, Reflectoquant®, or MQuant® test kits, generally in the form of a table.

In the case of samples with a complex, in many cases inexactly known composition (matrix) - such as, for example, foods - it is particularly difficult to estimate the potential influence of the foreign substances on the analysis (matrix effect). The following instructions describe a method by means of which the user can test whether a matrix effect is present or not.

Standard addition method (spiking)

In this method, volumes of the sample solution are gradually decreased by the same amount a pure standard solution of the analyte (addition solution) is added. Subsequently, each sample to which the addition solution has been added (spiked sample) is analyzed with the respective test kit to determine and calculate the recovery rate of the added, defined quantity of the analyte:

Step 1: Measurement of the sample (result A)

Step 2: Measurement of the spiked sample (sample + addition solution) (result B)

Step 3: Calculation of the recovery rate:

$$\text{Recovery rate} = \frac{\text{Result } \mathbf{B} - \text{Result } \mathbf{A}}{\text{Specified value } \mathbf{B} - \text{Result } \mathbf{A}} \cdot 100\%$$

The specified value B (= c_3) is determined as follows:

$$V_1 \cdot C_1 + V_2 \cdot C_2 = V_3 \cdot C_3 \longrightarrow C_3 = \frac{V_1 \cdot C_1 + V_2 \cdot C_2}{V_3}$$

where: v_1 = volume of the sample solution [mL]

c_1 = concentration of the sample solution [mg/L] (result **A**)

v_2 = volume of the addition solution [mL]

c_2 = concentration of the addition solution [mg/L]

v_3 = volume of the spiked solution, $v_3 = v_1 + v_2$ [mL]

c_3 = concentration of the spiked solution [mg/L] (specified value **B**)

In order to securely determine matrix effects by means of standard addition, the following aspects must be considered:

1. At least four spiked solutions should be prepared.
2. The measurement values expected for the spiked solutions must lie within – preferably in the middle of – the measuring range of the test kit in question.
3. The addition solution should have a relatively high concentration to ensure that the resulting measurement values A and B differ from each other to as great an extent as possible. Furthermore, in this case only small quantities of addition solution need be added, so that the matrix of the sample is hardly affected by the spiking process (dilution).

Example

Measuring range of the test kit: 10-150 mg/L.

Concentration of the addition solution: 1000 mg/L (i.e., 1 mg/mL)

To obtain 100 ml of spiked solution in each case, the volumes of sample solution and addition solution given in the following table are mixed.

Sample solution [mL]	Addition solution [mL]	Result B resp. A [mg/L]	Specified value B [mg/L]	Specified value B – Result A [mg/L]	Result B – Result A [mg/L]	Recovery rate
100.0	0	50.0 (=A)	-	-	-	-
99.8	0.2	52.5 (=B)	52.0	2.0	2.5	125%
99.6	0.4	54.3 (=B)	54.0	4.0	4.3	108%
99.4	0.6	56.1 (=B)	56.0	6.0	6.1	102%
99.2	0.8	58.0 (=B)	58.0	8.0	8.0	100%
99.0	1.0	59.6 (=B)	60.0	10.0	9.6	96%

Assessment of the results

A matrix effect is present when the recovery rate lies considerably below 80% or considerably above 120%. When only very small quantities of addition solution are added (see line 2 of the table), the range of variation of the measurement values may result in these limits being undermet or exceeded! The decisive factor here is always the assessment of the measurement series as a whole. In the example above, therefore, a matrix effect can be excluded.

The presence of a matrix effect means that the sample in question cannot be properly investigated without appropriate pretreatment. Please check our available application notes whether the respective sample/analyte combination is already described or contact us.

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SigmaAldrich.com/photometry

Merck KGaA
Frankfurter Strasse 250
64293 Darmstadt, Germany

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