Recovery spiked sample

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What is spiking ?

Matrix spiking is a technique used in analytical chemistry to evaluate the performance of an analytical method for a specific sample type (matrix). It helps ensure that the method produces accurate and valid results for that particular sample method.

Matrix spike

A matrix spike is when a known amount of an analyte (analytical standard) is added to a sample. This will increase the concentration by a known amount. By testing the spiked sample you will be able to determine if the added analyte is recovered. Prepare six matrix spiked samples for good recovery and reliable results.

Spike solution (analytical standard)

The spike solution is the solution that is chosen for preparing a matrix spike. The concentration of this solution is determined by the linearity and detection limit. Depending on the method the spike solution should always be within the linearity concentration range. This concentration is chosen to ensure that the spike solution does not change the sample volume. This makes calculating easier and avoids unknown effects from happening.

In this example the spike solution is slightly more concentrated than the lowest linearity standard, the linearity standards were prepared between 0.0003 and 0.005 µg/mL. The spike solution was chosen as 0.0005 µg/mL.

Sample Preparation

The following sample preparation was used in PCB with MS app note;

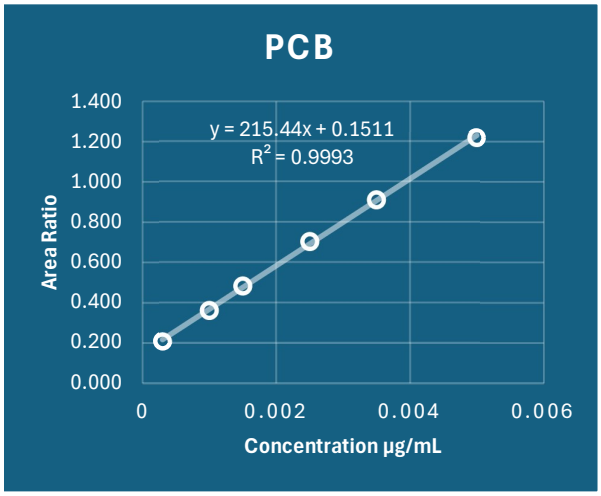
Tap water samples were prepared in triplicate by an extraction of 50 mL tap water with 10 mL iso-octane containing 0.003 µg/mL IS. The solution was stirred for 15 minutes with a magnetic stirrer, two layers appeared. The top (organic) layer was transferred into a sample container then concentrated under N_2-gas to approximately 1 mL. The extract was transferred into an injection vial ready for analysis.

To determine the recovery, six water samples were prepared as the water sample above and spiked with 0.0005 µg/mL PCB standard prior to extraction. These spiked samples were used to determine the LOQ in accordance with the method.

Blank injections of iso-octane were performed in between samples, to ensure that the system was not contaminated after sample injections.

Calculations

Conc. [PCB] µg/mL	0.0003	0.001	0.0015	0.0025	0.0035	0.005
Area Ratio	0.205	0.362	0.483	0.702	0.909	1.218



215.44 =***A*** , 0.1511 =***B***

Sample unspiked	Sample spiked	Ratio concentration	Spiked concentration	Recovery %
0.00	0.257	0.00049	0.0005	98.44

Sample unspiked: area ratio found in unspiked sample

Sample spiked: area ratio found in spiked sample

Ratio concentration: $\frac{\text{Sample spiked} - B}{AX}$

Spiked concentration: known spiked concentration

Recovery %: $\frac{\text{Ratio concentration}}{spiked\ concentration} * 100$

Conclusion

Compare the recovery results with the applicable validated method requirements. When the recovery results are good and within specification, it means that you have good method efficiency and reliability.