KF Application Note K–48

# Sample preparation with the oven technique – relative blank

Large sample sizes can lead to subtraction of too high blank values. This Application Note describes the calculation of a relative blank and thus helps to improve the accuracy of the method.

Metrohm

# Relative blank values

Generally, samples with water contents in the low ppm range require large sample sizes. If the sample should be analyzed using the oven technique, large sample sizes can lead to wrong or even negative results. One possible reason for the wrong results can be the subtraction of too high blank values.

Why are the blank values too high?

Blank values are determined using empty sample vials. If for the sample determination, half of the volume (or even more) of the vial is filled with sample, the blank value determined with the empty vial is too high and to obtain correct results, only half of the blank should be subtracted.

For water determinations in large sample sizes, we therefore recommend calculating with relative blank values.

$$\text{blank}_{\text{rel.}} = \text{blank}_{\text{empty vial}} \times \frac{V_{\text{vial}} - V_{\text{sample}}}{V_{\text{vial}}}$$

| | |
|---|---|
| $\text{blank}_{\text{rel.}}$ | relative blank value |
| $\text{blank}_{\text{empty vial}}$ | blank value of empty sample vial |
| $V_{\text{sample}}$ | volume of sample |
| $V_{\text{vial}}$ | total volume of sample vial |

## Examples

| Volume of vial filled with sample [%] | Total blank value subtracted from EP [%] |
|---|---|
| 50 | 50 |
| 60 | 40 |
| 70 | 30 |
| 80 | 20 |

## Results

The following results of water determinations in oil illustrate the influence and the importance of the relative blank. Measurements were performed using direct injection and oven technique.

### Direct injection

| Mean (n = 10) [µg/g] | RSD [%] |
|---|---|
| 19.2 | 0.45 |

### Oven technique (885 Compact Oven Sample Changer/899 Coulometer)

| Mean (n = 10) [µg/g] | RSD [%] |
|---|---|
| 14.0 | 2.34 |

Result with subtraction of $\text{blank}_{\text{empty vial}}$

| Mean (n = 10) [µg/g] | RSD [%] |
|---|---|
| 20.7 | 1.45 |

Result with subtraction of $\text{blank}_{\text{rel.}}$

### Oven technique (874 Oven Sample Processor/851 Titrando)

| Mean (n = 10) [µg/g] | RSD [%] |
|---|---|
| 14.7 | 1.91 |

Result with subtraction of $\text{blank}_{\text{empty vial}}$

| Mean (n = 10) [µg/g] | RSD [%] |
|---|---|
| 20.7 | 1.45 |

Result with subtraction of $\text{blank}_{\text{rel.}}$

## Conclusion

Water determinations obtained by the oven technique show a too low water content when the blank value of the empty vial is subtracted. In contrast, the subtraction of the relative blank value leads to results that agree with those using direct injection.