

METHOD STATEMENT



Determinand:

Determination of Suspended and Ashed Suspended solids including Settled Suspended Solids (1 hour), Part Settled Suspended Solids (30 minutes) and Neutralised (~pH 7) Suspended Solids

Matrix:

Sample Type: Final effluents, trade discharges and crude sewage samples

Principle of Method:

Suspended matter is removed from a measured volume of sample by filtration under reduced pressure through a pre-treated, pre-weighed glass fibre filter paper and determined gravimetrically after washing and drying at $105 \pm 5^{\circ}\text{C}$. If ashed suspended solids are required, this is followed by ignition at 450°C and weighing the residue.

The sample may be pre-treated by settling the solids for 1 hour after adjusting the pH to 6.5 - 7.5 pH units (see method WWC4).

Sampling and Sample Preparation:

Samples are normally received in 1L pet bottles.

Samples are stored at $3 \pm 2^{\circ}\text{C}$ until ready for analysis. Samples should be shaken before analysis to ensure that any subsample is as homogeneous as possible.

Details of sample pre-treatment for Yorkshire Water settled and neutralised suspended solids are found in method WWC4.

Samples are stable for 7 days (Coventry In House Data).

Interferences

Interferences can occur when substances such as oil or dissolved solids are erroneously counted as suspended solids.

Performance of Method:

Range of Detection:

2.4 mg/l upwards

Normal reporting level is 2.4mg/l.

Limit of Quantification:

2.384 mg/l.

The statistically obtained limit of detection was generated using 200mls of sample. If a smaller volume is used the reporting limit should be adjusted accordingly.

Recoveries of Compounds, Bias and Uncertainty of measurement:

Suspended solids

| | Low Standard | High Standard | Final Eff | Final Low Spike | Trade | Trade Low Spike | Crude | Crude Low Spike |
|--------------------------------|--------------|---------------|-----------|-----------------|-------|-----------------|-------|-----------------|
| Concentration mg/l | 33.1 | 463.5 | 2.0 | 33.4 | 10.0 | 32.8 | 360.4 | 930.0 |
| Total Standard Deviation, mg/l | 1.8 | 25.5 | 0.8 | 2.4 | 1.8 | 1.9 | 21.3 | 48.0 |
| Recovery % | 94.6 | 92.7 | - | 94.9 | - | 87.7 | - | 91.9 |

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Ashed Solids

| | AQC | Final | Trade | Crude |
|----------|----------|-------|-------|-------|
| Ash mg/l | 18555.46 | -0.14 | 0.89 | 79.46 |
| SD mg/l | 1235.00 | 0.94 | 0.88 | 6.58 |

Note: As the majority of the suspended solids within the samples were volatile at 450°C, the ashed solids results approximate to 0.0mg/l, giving artificially high %RSD values during validation testing.

The absolute standard deviation for the samples were all below 5mg/l and as ashed solids is the residue from the volatile suspended matter, the performance of both methods are fundamentally linked

Uncertainty of measurement:

± 20.7%

References:

Suspended, Settleable, and Total Dissolved Solids in Waters and Effluents 1980. ISBN 011 751957X.
Wakefield Solids Instrument Interfacing Manual (Version 1.0)