# Before you begin – What is needed to determine LOD and measurement uncertainty?

Mårten Dario Linköpings Universitet What is the LOQ (or LOD)?

What is the measurement uncertainty?

Why is the LOQ (LOD) so high?

Why is the measurement uncertainty so high?

#### What is the LOQ and LOD?

LOD = 3 \* stdev of blank results

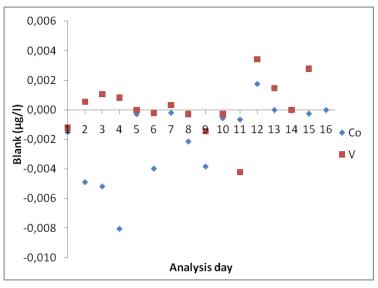
LOQ = 3 \* (average + 3\*stdev) of blank results

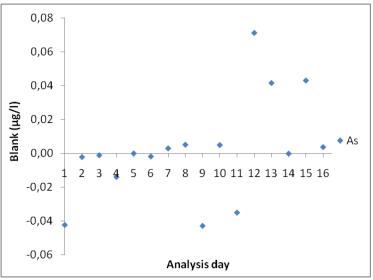
#### Example

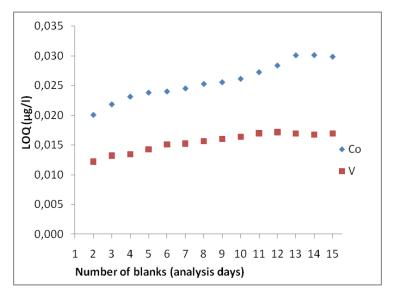
Analysis of metals in surface waters.

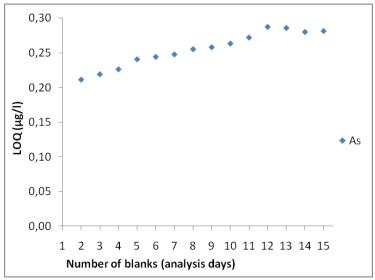
Blank samples (1%  $HNO_3$  in MQ water) analysed 16 different days.

#### Calculation of LOQ from different number of blanks

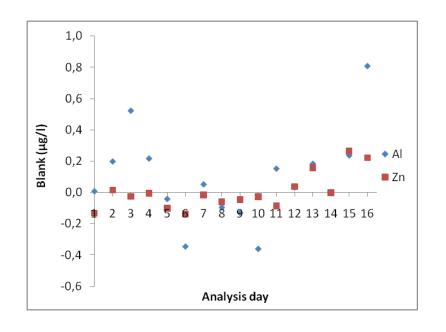


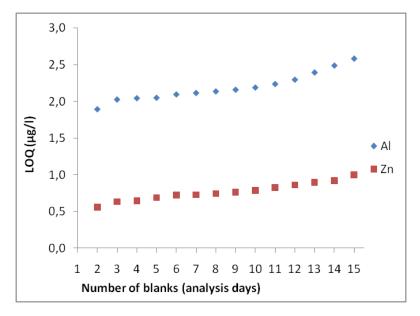






#### Calculation of LOQ from different number of blanks





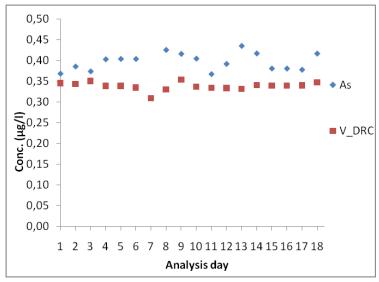
# What is the measurement uncertainty?

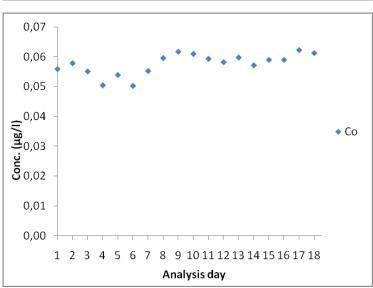
#### Example

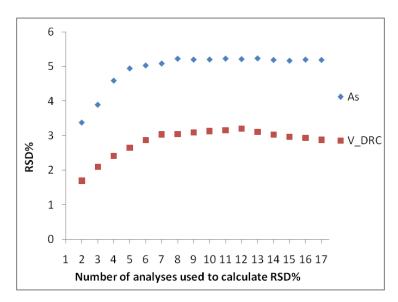
Analysis of metals (ICP-MS) in SLRS-5, a surface water.

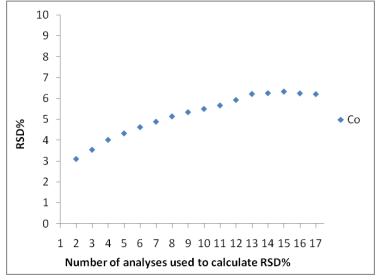
Analysed 18 times during 1.4 years.

#### What is the measurement uncertainty?

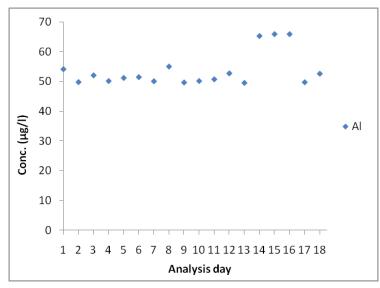


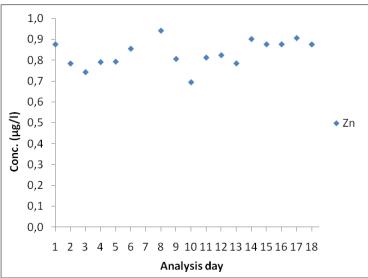


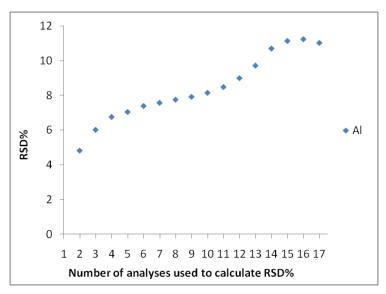


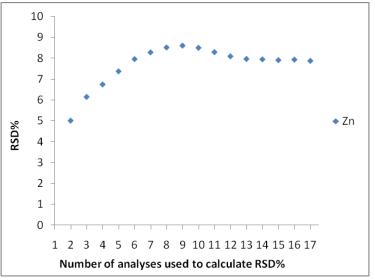


# What is the measurement uncertainty?









# When there is enough data to estimate LOQ and measurement uncertainty

Why is the LOQ (LOD) so high?

Why is the measurement uncertainty so high?

# Why is the LOQ so high?

#### Example

Biogas sludge

Digestion method: 0,25 g sample

20 ml 7 M HNO<sub>3</sub>

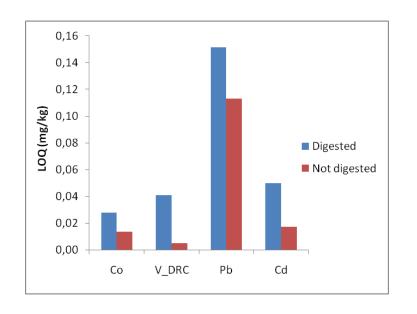
120 °C

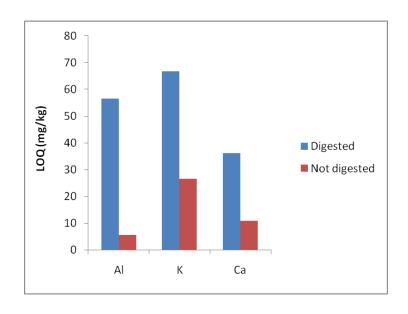
30 min

Diluted to 500 ml after digestion, before ICP-MS analysis.

Comparison of digested and undigested (1% HNO<sub>3</sub> solution) blanks.

# Why is the LOQ so high?





LOQ calculated from digested and not digested blank samples.

# Why is the measurement uncertainty so high?

#### Example

Biogas sludge

Digestion method: 0,25 g sample

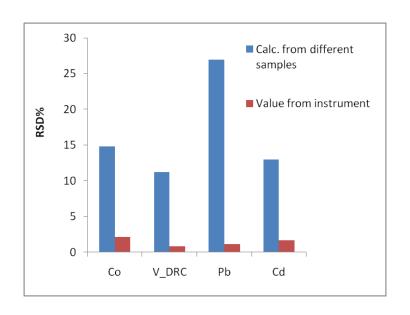
20 ml 7 M HNO<sub>3</sub>

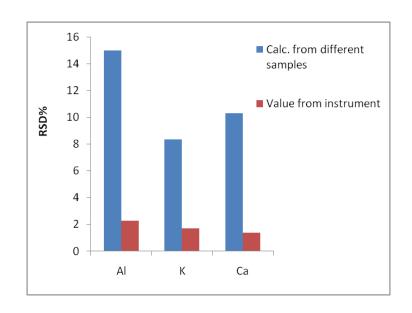
120 °C 30 min

Diluted to 500 ml after digestion, before ICP-MS analysis.

Analysis of a certified sludge, CRM 029-050.

# Why is the measurement uncertainty so high?





Calc. from different samples: Sludge (CRM 029-050) digested and analysed at different days.

Value from instrument: the same sample (CRM 029-050) analysed three times by the instrument.