

Testing of the implementation of the analytical step of the EN 1911 during an ILC

Origin of the data

| | |
|-----------------------------|--|
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| Country | UK |
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| Date of transmission data | January 2023 |

How the data was obtained

| | |
|---|--|
| Modelling | - |
| Tests: in laboratory / on industrial site / on test bench (stack simulator) | - |
| Interlaboratory comprison: in laboratory / on industrial site / on test bench / with sample distribution... | Samples generated in the laboratory with know concentration and further samples from the stack simulator were split into equal samples and sent out to a selection of accredited laboratories as normal stack samples (blind ILC). |

Purpose of the ILC

Demonstrate laboratory performance and stated uncertainties for EN 1911 at low HCl levels.

If tests or ILC: description of test facilities

Identical samples sent to laboratories accredited to ISO 17025 for the required analysis in EN 1911.

Conditions for the implementation of ILC

| | |
|---------------------------------|---|
| Date of trials | June-August 2021 |
| Number of participants | 7 accredited laboratories |
| Number of tests | 14 test solutions and 5 blanks - Stack Sim samples; |
| Duration of each test | 30 minute sampling time for Stack Sim samples |
| Number of measurement lines | 1 |
| Number of samples / measurement | Each of the 14 test solutions was split and sent to all seven participant laboratories for analysis |
| Other results | Water Vapour Results |
| Characteristics of the matrix | HCl Test Concentrations: 1-8 mg.m-3 273.15 K/101325 Pa Dry, NO, CO, SO2: 130, 20, 40 mg.m-3 Dry, O2: 11%, Water Vapour: 8%-15%, CO2:4% (Only Test 7) |
| Data processing | Concentration (mg/m3) calculation from analysis results (mg/l) & ISO 5725-6 |
| Type of results | Chloride concentration (mg/l); Impinger volume; Mass of chloride (mg); Absorber efficiency; HCl concentration (mg.m ⁻³) |
| Other information | - |

Main conclusion

Challenging to meet uncertainty requirements with the current method if ELVs fall in future and there is limited scope for further improvement of the laboratory analytical uncertainty component.

Testing of the implementation of part of the sampling equipment used for EN 1911 in lab

Origin of the data

| | |
|-----------------------------|---|
| Institut/company/laboratory | VTT Technical Research Centre of Finland |
| Country | Finland |
| Contact | Tuula Pellikka, Tuula Kajolinna |
| Mail contact | tuula.pellikka@vtt.fi ; tuula.kajolinna@vtt.fi |
| Date of transmission data | January 2023 |

How the data was obtained

| | |
|---|----------------------|
| Modelling | - |
| Tests: in laboratory / on industrial site / on test bench (stack simulator) | in laborarory at VTT |
| Interlaboratory comprison: in laboratory / on industrial site / on test bench / with sample distribution... | - |

Purpose the tests

Aim was to make tests using four different glassware tips of impigners and assess if there is statistical differences between the results.

Conditions for the implementation of tests

| | |
|--|---|
| Date of trials | flowrate tests on May 2020, tip tests 1-9 on June 2020 and tests 10-15 on February 2021 |
| Number of glassware tips (glassware tests) | 4 |
| Number of tests (glassware tests) | 15 |
| Number of flowrates (flowrate tests) | 2 (1.5 and 8 l/min) |
| Number of tests (flowrate tests) | 10 |
| Duration of each test | 30 min |
| Number of measurement lines | |
| Number of samples / measurement | Flowrate tests: 10 tests * 2 sampling flowrates = 20 tests |
| Other results | absorption efficiency from one test per campaign |
| Characteristics of the matrix | Liquid vapourized to gas flow with Hovacal (incl. scale). Gas: N2 10 lpm, HCl liquid: 1 mmol/l, HCl concentration target 2 mg/m3 (dry, NTP), Water vapour 6.4 vol-%. NTP=273.15 K and |
| Data processing | In accordance with ISO 13528 / ISO 5725-2. Average, repeatability, between-laboratory variance, reproducibility |
| Type of results | HCl concentrations, mg/m3, NTP (0 °C, 101,3 kPa) , dry |
| Other information | Each participant sent their samples to the analytical laboratory they usually work with |

Main conclusions

Based on these tests, there is no statistical difference between different impinger tips or between tested flow rates.

Testing of the implementation of the measurement method EN 1911 during an ILC on a test bench

Origin of the data

| | |
|-----------------------------|--|
| Institut/company/laboratory | INERIS |
| Country | France |
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| Date of transmission data | January 2023 |

How the data was obtained

| | |
|--|--------------------------|
| Modelling | - |
| Tests: in laboratory / on industrial site / on test bench (stack simulator) | - |
| Interlaboratory comparison: in laboratory / on industrial site / on test bench / with sample distribution... | ILC on Ineris test bench |

Purpose of the ILC

to test the capability of the EN 1911 to measure new ELVs with acceptable uncertainty

If tests or ILC: description of test facilities

Matrix generated from gases emitted by a gas boiler: gas boiler for this ILC; matrix was spiked with compounds from gas cylinders to adapt the matrix to the ILC objective,
The bench has 12 sampling ports and therefore 12 participants can perform measurements simultaneously

Conditions for the implementation of ILC

| | |
|---------------------------------|---|
| Date of trials | 27 to 29 April 2021 |
| Number of participants | 9 |
| Number of tests | 15 |
| Duration of each test | 60 min |
| Number of measurement lines | 2 independent sampling systems implemented by each participant => 18 sampling lines |
| Number of samples / measurement | 15 x 2 = 30 measurements by each participant 9 x 2 measurements for each trial Duplicate analysis of each sample under repeatability conditions 15 x 2 x 2 x 9 = 540 results In fact 540 + 60 results = 600 results because 1 participant doubled his sampling lines to send his samples to 2 different analytical laboratories |
| Other results | Quality controls: field blank + absorption efficiency Samples of 3 participants analysed by the same analytical laboratory for 3 trials. |
| Characteristics of the matrix | HCl concentration: 2,4 to 12,9 mg/m ³ 273.15 K/101325 Pa/dry Auther doping: NH ₃ /SO ₂ /NH ₃ /SO ₂ |
| Data processing | - In accordance with ISO 13528 / ISO 5725-2 / ISO 5725-5 Assigned value=reference value for each trial: robust mean calculated from the participants' results and according to ISO 5725-5 Calculation of half confidence intervals and biases of participants - In accordance with Statistical treatment based on Eurachem Guide/Citac " - Measurement uncertainty arising from sampling » Evaluation of the sampling and the analysis contributions in repeatability |
| Type of results | <u>For each trial:</u> - Robust mean - Half confidence interval of repeatability - Half confidence interval of reproducibility - Biases for each trial and each sampling line of each participant - For each participant, the sampling and the analysis contributions in repeatability on average, for 15 trials <u>Other data:</u> - Field blanks, absorption efficiency - Metadata: > description of sampling lines > analysis method of solutions > analysis and measurement LoQ of each sampling and analytical laboratory > analysis and measurement uncertainties of each sampling and analytical laboratory analysis calculated by budget uncertainty approach |
| Other information | Each participant sent their samples to the analytical laboratory they usually work with |

Main conclusion

1/ Temperature is a parameter that has a strong influence on the results in case of the presence of compounds that can lead to the formation of salts like NH_3 . The temperature chosen should take into account the risk of salt formation that could lead to measurement bias, and it is important to avoid any cold spots in any part of the sampling system that is not flushed.

2/ Comparison of expanded uncertainties provided by participants from GUM approach and calculated from ILC (1/2 half confidence interval of reproducibility):

- GUM approach leads to expanded uncertainties that comply with the standard's criterion: $< 30\%$ of concentration

Whereas ILC approach leads to $\frac{1}{2} \text{IC}_{95} > 30\%$ of concentration

- Some uncertainty components not modelled in the GUM approach: e.g. human factor

Some implementation biases not modelled in the GUM approach: e.g. risk of salt formation in the presence of ammonia, loss in case of condensation

- GUM approach evaluates uncertainty for one measurement / implemented by **one measurement laboratory** / using **one equipment** / with analysis of the solutions by **an analytical laboratory** => as during periodic monitoring

vs ILC approach evaluates the variability for one measurement / implemented by **several measurement laboratories**, using **different equipments** with variable performance / with analysis of the solutions by **several analytical laboratories** => which does not quite correspond to the implementation of an on-site control

- Analyses uncertainties declared by participants were equal for the 15 tests for most participants: were they really?

Analyses uncertainties need to be refined to improve the estimation of measurement uncertainties

- In GUM approach, contribution of analysis in measurements uncertainties varies between participants. ILC approach seems to show that for HCl this contribution of analysis to the repeatability is limited and that the measurement bias is more related to sampling.

3/ Need to estimate the uncertainty for the analysis step, and not just repeatability as required in actual EN 1911

Moreover, uncertainty => in France analytical laboratories have to estimate the analytical uncertainty at least 3 concentration levels of the validated concentration range $[\text{LoQ}_{\text{analysis}} - C_{\text{max}}]$

4/ Setting an uncertainty criterion only in relative terms is not appropriate for the lowest concentrations as some sources of uncertainty are not proportional to concentration. In French Standard NF X 43-551, a concentration threshold below which the uncertainty criterion is expressed in mg/m^3 and is therefore constant; has been defined: if concentration is $< 5 \text{ mg} \cdot \text{m}^{-3}$, expanded uncertainty criterion is $< 1.5 \text{ mg} \cdot \text{m}^{-3}$.

Testing of the implementation of the measurement method EN 1911 on industrial site

Origin of the data

| | |
|-----------------------------|---|
| Institut/company/laboratory | VTT Technical Research Centre of Finland |
| Country | Finland |
| Contact | Tuula Pellikka, Tuula Kajolinna |
| Mail contact | tuula.pellikka@vtt.fi ; tuula.kajolinna@vtt.fi |
| Date of transmission data | January 2023 |

How the data was obtained

| | |
|--|--|
| Modelling | - |
| Tests: in laboratory / on industrial site / on test bench (stack simulator) | on industrial site, plant is located at Southern Finland |
| Interlaboratory comparison: in laboratory / on industrial site / on test bench / with sample distribution... | - |

Purpose of the tests

Aim was to measure on real field circumstances the HCl concentrations using two lines of EN1911 and two lines of P-AMS FTIR (Gaset D4000), and to compare the results

Conditions for the implementation of test

| | |
|---------------------------------|---|
| Date of trials | 20.-22.4.2021 |
| Number of sampling lines | 4 (2*EN1911 + 2*P-AMS FTIR) |
| Number of tests | 8 |
| Duration of each test | 30 min (EN1911 sampled gas volume ~ 120 l) |
| Number of samples / measurement | 8 tests * 4 lines/test = 32 samples (+efficiency tests of EN1911) |
| Other results | |
| Characteristics of the matrix | Fluegas. Fuel 70% biomass + 30% coal and peat. Temperature ~140°C, Ammonia was not present in the flue gas, SO ₂ levels ~35- 40 ppm, Moisture 19-20 %, O ₂ 4,5- 5,1 % (dry) |
| Data processing | |
| Type of results | HCl concentration as mg/m ³ at NTP dry (NTP 101.3 kPa, 0°C) |
| Other information | For EN1911 the sampling flowrate between 3 and 5 l/min was used. Sampling flowrate to each P-AMS was 4 l/min |

Main conclusions

Based on this data sets, both sampling lines (EN 1911 and P-AMS) seem to give uniform results. No further conclusions can be made based on this data set.

P-AMS testing on the NPL test bench

Origin of the data

Institut/company/laboratory
Country
Contact
Mail contact
Date of transmission data

NPL
UK
Chris Dimopoulos
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January 2023

How the data was obtained

Modelling

-

Tests: in laboratory / on industrial site / on test bench (stack simulator)

P-AMS intercomparison on NPL test bench

Interlaboratory comparison: in laboratory / on industrial site / on test bench / with sample distribution...

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Purpose of modelling / of the tests

Determine the performance of different P-AMS technologies in order to create a scientific base to facilitate future decisions on HCl SRM and when and if it should be replaced by an alternative portable optical technology under EN 16429:2021

If modelling: brief description of the model and the assumptions considered

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If tests or ILC: description of test facilities

Gas mixtures generated by blending test gases from source gas cylinders with purified air, nitrogen and water vapour. Facility has 4 x 127mm ports and a 1.5m cross stack path length. Reference concentrations determined through instrumental techniques using additional quality control/bracketing calibration.

Conditions for the implementation of the model

-

Conditions for the implementation of test

Date of tests
Number of P-AMS
Type of P-AMS
Sampling Systems

4th to 10th June 2021
4 Pairs of P-AMS (5th pair was not delivered in time For tests)
FTIR x2, TDL, NDIR-GFC, Reference instrument FTIR
M&C Heated Lines (2-5m length)

Number of tests
Duration of each test
Standard followed
Calibration method

M&C Heated Probes
PD-100E Portable Permeation Dryer (only for NDIR-GFC)
8 tests per Round of testing (two rounds of testing with 2 pair of P-AMS in each)
30 min
EN 16429:2021

Span Value
Characteristics of the matrix

Wet gas calibration generator using Aqueous HCl solution (<1% uncertainty), Bracketing calibration method for Reference instrument.
5-6 mg.m-3 Wet, 9-10% Water Vapour
HCl Test Concentrations: 1-8 mg.m-3 273.15 K/101325 Pa Dry, NO, CO, SO2: 130, 20, 40 mg.m-3 Dry, O2: 11%, Water Vapour: 8%-15%, CO2:4% (Only Test 7)

Data processing

- Uncertainty calculation based on EN 16429:2021.
- Drift calculation based on EN 16429:2021.
- ANOVA statistical tests on repeatability.
- ANOVA statistical tests on reproducibility.
- ANOVA statistical tests on interference effects.
- Comparison of uncertainty with requirements under EN 16429:2021 and current and future legislation requirements.

Type of results

For each P-AMS/test:
•30min average
•Uncertainty results
•Pre and post test calibration results/Drift
•Repeatability
•Response Time

Other information

1-3 Pair of P-AMS operated by one laboratory and pair 4 by a different.

Conditions for the implementation of ILC

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Main conclusion

Current state of the art P-AMS can meet the currentfor Waste Incineration Processes ELV of 10mg/m3 uncertainty requirement and also down to a future ELV of 6 mg/m3. Below that ELV things become more challenging for the P-AMS to meet the uncertainty criterion. In absolute terms a conservative 1.5mg/m3 uncertainty is a reasonable estimate.

CFD simulation of effect of particles in gas stream as different sizes and concentrations

Origin of the data

Institut/company/laboratory

Country

Contact

Mail contact

Date of transmission data

Czech Metrology Institute

Czech Republic

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January 2023

How the data was obtained

Modelling

Data obtained by simulations done in open source software OpenFOAM 5.x

Tests: in laboratory / on industrial site / on test bench (stack simulator)

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Interlaboratory comprison: in laboratory / on industrial site / on test bench / with sample distribution...

Purpose of modelling / of the tests

To asses the uncertainty caused by sampling points located under the standard EN 15259:2007.

If modelling: brief description of the model and the assumptions considered

CFD modelling used to analyse particle distributions in dependence on type of stack supply pipe and for different emission particles size and different inlet concentrations.

If tests or ILC: description of test facilities

Conditions for the implementation of the model

Particle size and density

Inlet emission concentration

Fluid properties

10, 20 and 50 m^{-6} , 2300 kg/m^3

0.1, 1 and 10 mg/m^3

kinematic viscosity 10cSt

Main conclusion

Particle size is decisive for the emission distribution in stack and for the uncertainty of the emission prediction given by sampling points according to the standard.