FINAL PUBLISHABLE REPORT

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Project short name	Heroes
Project full title	Determining new uncertainty requirements for increasingly stringent legislative HCl industrial emission limits

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Internal Funded Partners: 1. NPL, United Kingdom 2. CMI, Czech Republic 3. VTT, Finland	External Funded Partners: 4. EA, United Kingdom 5. INERIS, France	Unfunded Partners:
RMG: -		

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1 Overview

This project validated aspects of the HCl Standard Reference Method (SRM, EN 1911), which is legislatively required for measuring industrial emissions, demonstrating that it is unable to meet uncertainty requirements brought in via Best Available Techniques (BAT) Conclusions documents (which set permit conditions for regulated industrial processes). The regulatory framework and the ability of national regulators to carry out enforcement interventions is compromised by this. HEROES has devised new uncertainty requirements for revising the SRM, based on results of the project. Beyond this HEROES has demonstrated the current performance of portable optical technologies as a potential replacement SRM in future. The regulator guidance document will help ensure that future operational measurements are made with compliant uncertainties. The results of this project have provided the evidence required to move forward with revision of the EN 1911 standard.

2 Need

Limiting emissions of HCl from industrial processes is critical, as it is acutely toxic and impacts on far more ecosystems than previously thought. The incorrect implementation of the Industrial Emissions Directive and the BAT Conclusions documents it adopts led to premature deaths / years of life lost in Europe of 13 000 and 125 000, respectively. Economically, if the legislation was not enforced, the European taxpayer may have incurred high costs (€7 – €28 billion p.a.).
The HCl emission limit for industrial processes regulated under the Industrial Emissions Directive used to be 10 $mg.m^{-3}$. However, BAT Conclusions documents have brought in increasingly stringent emissions limits impacting a range of industries (e.g. 2-6 $mg.m^{-3}$ for waste incineration, <1-3 $mg.m^{-3}$ for iron and steel production, 3-12 $mg.m^{-3}$ for power stations).
The legislatively required SRM for monitoring HCl emissions was described in EN 1911 and is based on extracting stack gas through deionised water in glass impingers before off-line analysis, generally by ion chromatography. It was a requirement that the emission measurement meets an uncertainty of 30 % (k = 2) of the emission limit, but this is not possible at limits below 10 $mg.m^{-3}$. The method uncertainty is subtracted from the reported emission prior to comparison to the emission limit, thus ensuring any breach is 'beyond reasonable doubt' (i.e. 95 % confidence) and that there is justification for national regulator enforcement intervention. Hence, if the method uncertainty was unclear then enforcement becomes untenable.
To address these issues three key needs were identified, from which the project's objectives were derived:

- CEN/TC 264 'Air Quality' have listed in their future priority work document the need for "assessment of current SRM to meet stricter limit values";
- CEN/TC 264 'Task Force Emissions – who provide recommendations to the European Commission – have stated that a regulatory guidance document was needed on, "…stationary source emissions providing information on the field of application of the methods (measurement range, validation range, uncertainty, etc.)";
- CEN/TC 264 have identified the need for work on "automated methods for measuring emissions".

3 Objectives

The overall objective of the project was to facilitate the monitoring and enforcement of emission limits at industrial processes regulated via BAT Conclusions documents. The specific objectives were:

1. Extending Stack Simulator capability in Europe to generate emissions in-line with industries regulated via BAT Conclusions (<10 $mg.m^{-3}$) adopted under the Industrial Emissions Directive (IED 2010/75/EU).

2. Characterised the performance of the HCl SRM (EN 1911) using the capability developed in objective 1 at BAT Conclusion processes. Characterised new uncertainty sources that became significant at low concentrations.

3. Produced a metrologically valid evidence base of uncertainty contributions to EN 1911 and from this developed a series of recommended uncertainty requirements for regulatory monitoring.

4. Created a scientific evidence base of the performance of a range of portable optical technologies, using the extended Stack Simulator capability developed in objective 1, with a view to the future

replacement of the existing SRM. This underpins current work at CEN on CEN/TS16429 (optical techniques) and CEN/TC264/WI00264151 (Fourier transform infrared spectroscopy).

5. Produced a National Regulator Guidance Document of recommended measurement uncertainty requirements. Disseminated the document to EU DG Environment (Directorate-General for Environment), Task Force Emissions, CEN/TC 264 'Air Quality', and more broadly, to the emission community.

6. Contributed to a revision of EN 1911 by providing to CEN/TC 264 the data, methods, guidelines and recommendations, necessary for new uncertainty requirements for HCl industrial emission limits.

4 Results

4.1 To extend Stack Simulator capability in Europe to generate emissions in-line with industries regulated via BAT Conclusions (<10 mg.m^{-3}) adopted under the Industrial Emissions Directive (IED 2010/75/EU).

In order to assess the performance of new and existing methods for HCl measurement it is necessary to have facilities where it is possible to control the measurand, while also providing realistic measurement conditions. A typical industrial stack will be hot, wet and contain a matrix of cross interfering gases, all of which will contribute to a very different measurement challenge to lab based testing and direct measurement from cylinders. To address this challenge there are a number of stack simulator facilities across Europe that can provide this more complex test bed for demonstrating the real-world performance of existing and new methods. NPL and INERIS have these facilities and validated them for low HCl concentrations.

The NPL Stack Simulator facility is able to reproduce a wide range of simulated stack gases under controlled operation conditions. It has a rectangular shape with cross-stack path length of 1.5m equipped with two 127 mm BSP (British Standard Piping) ports at one end of the path-length and two ports at the other end. These are positioned to allow two cross-stack instruments to be installed. Alternatively, a number of gas analysers or manual extractive systems may be attached. Different sized port adaptors allow the installation of all standardised gas sampling apparatus.

The facility is a recirculating system which allows flows of air, nitrogen and a selection of ‘pollutant’ gases into the stack simulator chamber at controlled rates to create predictable mixture concentrations. Complex gas mixtures are generated by blending the test gases from source gas cylinders with nitrogen and air from reservoirs. The bulk gas delivery is controlled by two 150 l.min^{-1} mass flow controllers, allowing a blend of nitrogen and zero air, this enables reduced oxygen concentrations to be generated, simulating combustion gas conditions. Further mass flow controllers (between 1 l.min^{-1} and 20 l.min^{-1}) allow the introduction of test gases. Typically, these consist of gas mixtures representative of combustion or waste incineration processes, i.e. SO_2, CO, Nox and HCl but more complex matrices can be generated that can include any interferent gases of interest. The stack simulator also incorporates a water injector to provide a controlled water vapour concentration. Figure 1 represents a facility schematic and Figure 2 a schematic of external and internal components. Specifications include: four 127mm BSP sample ports (v), centrifugal fan (w), internal heaters (z) and water vapour generator. The flow restriction (x) ensures a constant velocity across a duct of uniform cross-section (y).

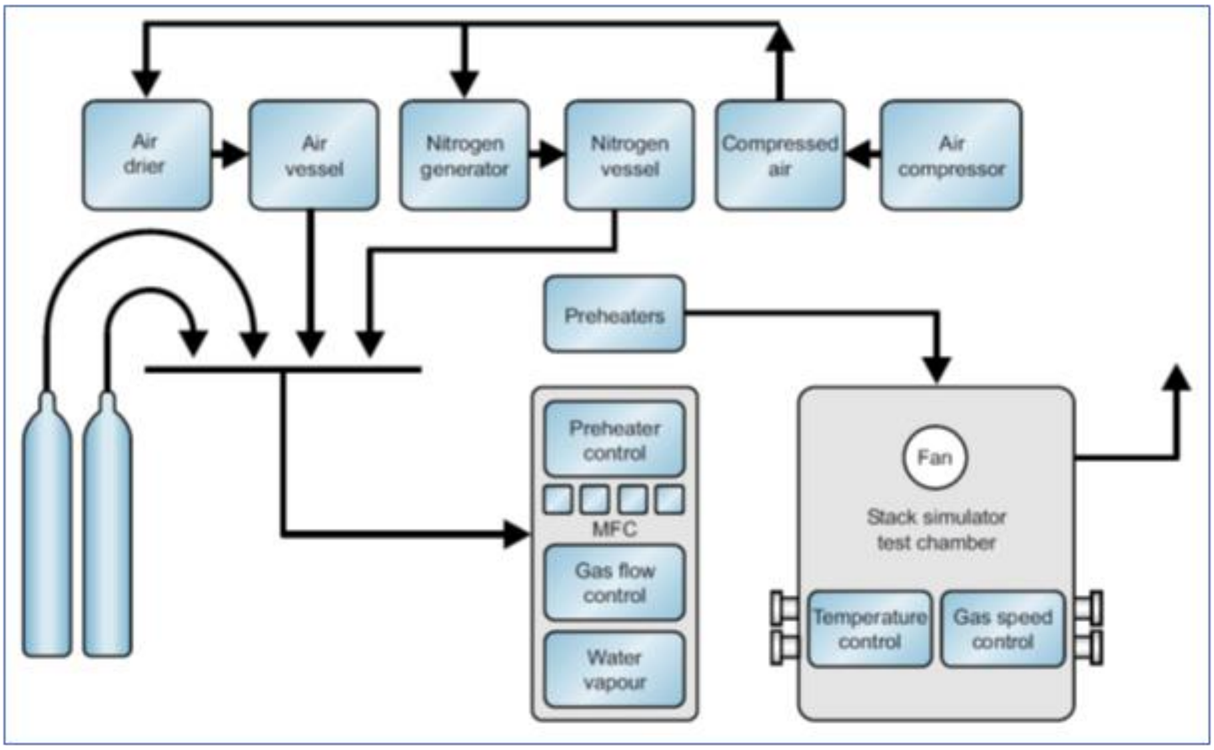


Figure 1 Facility Schematic.

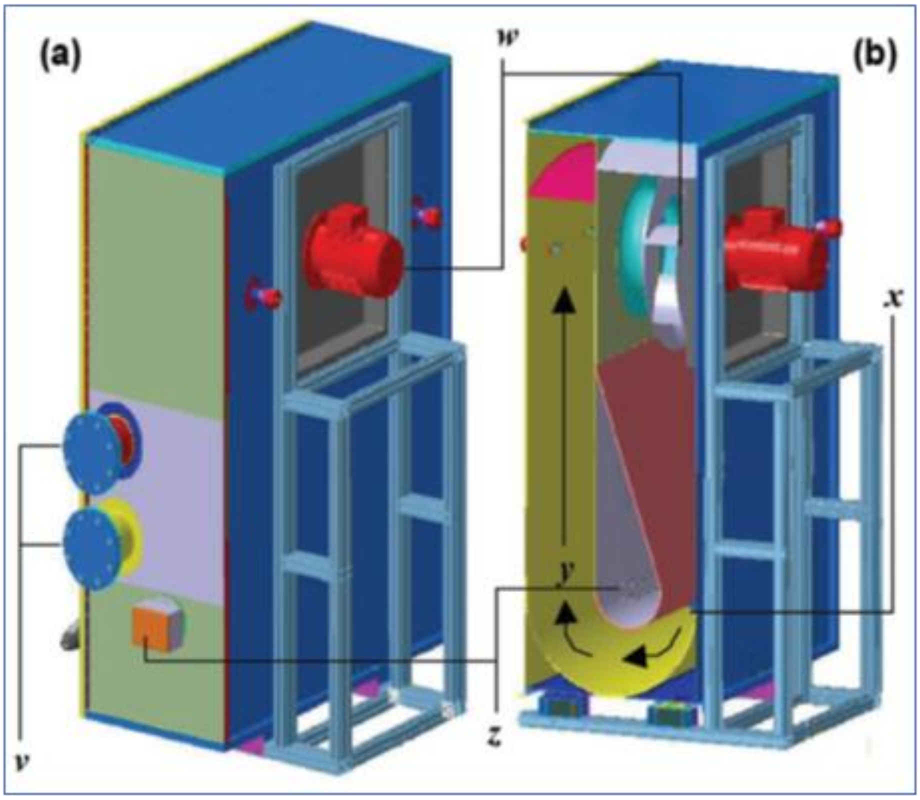


Figure 2 Schematic of external (a) and internal (b) components.

The gases are recirculated to create a high vertical stack velocity with low gas consumption and low release to the atmosphere. The velocity profile is approximately constant along the length of the test paths. The internal electric heaters allow the 'stack temperature' to be set above the dew point of the recirculating gas mixture. Table 1 outlines the performance characteristics of the facility.

Table 1 Performance Summary of NPL Stack Simulator.

Temperature	Maximum 180℃ controlled to ±2℃ and measured to ±0.5℃
Gas vertical velocity	Maximum 12 m.s-1
Water Vapour	Maximum 25% volume concentration
Oxygen	Maximum 20% volume concentration
Input gas flow rate	Maximum 300 l.min-1

The Ineris test bench at Verneuil-en-Hallate (Oise, France) simulates gaseous effluents from combustion or waste incineration facilities. It has twelve ports that allow large scale parallel testing, in measurement conditions that are similar to those that can be encountered on real facilities. The sampling ports are identical and separated by 2.75m. The total length of the test bench is near 36 meters.

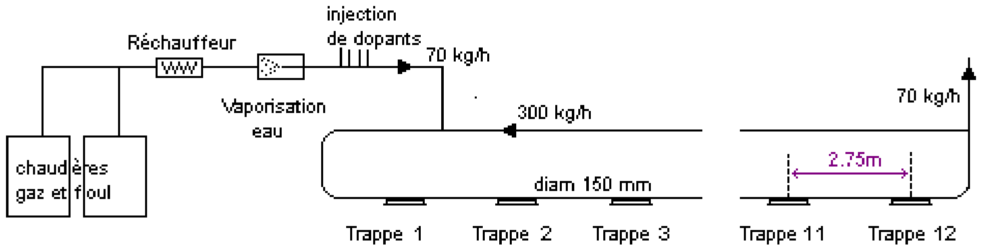


Figure 3: *Ineris test bench schematic*

The gas generated goes into a titanium loop, where it circulates at up to 400 kg/h flow rate (Figure 3). This loop is maintained in temperature by electric tracing (the setpoint temperature is fixed between 130 and 170°C). The internal diameter of the duct is 150 mm. The test bench is maintained at positive pressure to avoid the sampling from one port disturbing the homogeneity of the characteristics of the effluent in the measurement section downstream.

Flue gases are generated by domestic combustion facilities fuelled with gas, light fuel oil or biomass. (≈40-50 kW). Boiler settings can be modified to operate at different excess air, which has an influence on the levels of O_2, CO_2, but also in CO, VOC and NOx (Figure 4).

Prior to their introduction in the loop, gases produced by the combustion can be heated, moistened to simulate effluents coming out of a wash tower, and the concentrations of some compounds can be increased by the addition of gases from gas-cylinders (CO, NO, SO_2, HCl, HF, CH_4, C_3H_8, etc.) or by stripping liquids (generation of gas vapor from aqueous solution + evaporator: e.g. HCl, specific VOC, etc.).

Some of the pollutant measured at the ports comes from the combustion gas of the boiler, with the remainder coming from compounds added. The content of pollutant added to the gas effluent can be determined however the portion coming from the boiler can't be accurately quantified since the settings for the boiler are changed voluntarily to work in different conditions and produce different gas composition.

The generated levels of concentration are followed by means of continuous analysis equipment when it is possible: this follow-up allows adjusting the levels of gas injection settings and the regulation of boilers to reach the aimed levels of concentration.

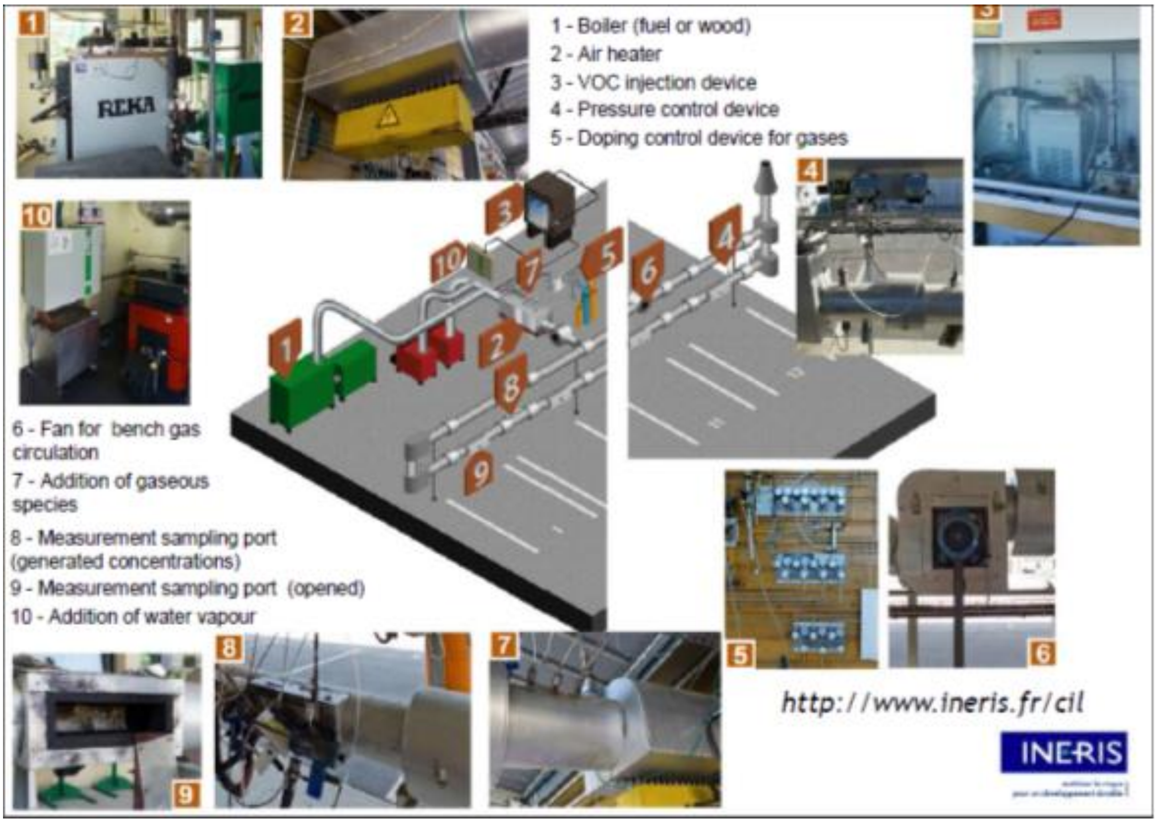


Figure 4: Features of the Ineris test bench

Ineris carried out rounds of testing in 2018 and 2020, with both sets of results confirming the findings. Response times were around 4 minutes or better and there was no evidence of sample loss along the test bench. The between port standard deviation was between 0.6-3.8% relative, which is less than the 4.5% level required for proficiency assessment, so it is not necessary to consider the between sample standard deviation. This confirms that performance at all twelve measuring ports was homogeneous with a between sampling ports standard deviation fixed at 4% of the concentration.

NPL carried out initial tests in 2020 focused on single port homogeneity, before further investigations in 2021 that confirmed the earlier results, then looked at operating conditions homogeneity and sampling homogeneity. In total eleven tests were carried out, with ten passing the stability check criteria. The remaining test narrowly failed due to an operational error that led to a sampling port being left open between tests, so the concentration levels in the simulator had not yet stabilised before the test was started.

The Ineris validation exercise included cross contaminant species (NH_3 and SO_2) which can form salts with HCl, but the facility demonstrated homogeneous HCl concentration in the gas stream even when these were present (4% inhomogeneity uncertainty). The NPL facility was validated to a lower concentration using a dry gas stream before additional tests investigated different levels of water vapour, demonstrating homogeneous HCl concentration at H_2O volumes up to 11% (3.6% inhomogeneity uncertainty).

Validating the facilities in parallel like this allowed Ineris and NPL to share experiences and push their facilities to the lowest possible values, benefiting the subsequent work carried out at these facilities. The different set ups allowed the consortium to investigate alternative cross contaminant options and lower detection limits. This allowed these facilities to host interlaboratory comparisons (ILCs) at low HCl concentrations to assess the performance limitations of existing methodologies later in the project.

Through the validation tests both facilities have successfully extended their operating range for the generation of HCl down to 1mg.m^{-3} and 2.3 mg.m^{-3} for NPL and Ineris facilities respectively, with the target under Heroes being <10mg.m^{-3}. This will allow the development and validation of new techniques for measuring low concentration HCl emissions. These results meet this objective in full.

4.2 To characterise the performance of the HCl SRM (EN 1911) using the capability developed in objective 1 at BAT Conclusion processes. To characterise new uncertainty sources that become significant at low concentrations.

VTT organised a measurement campaign at a biomass power plant in Finland to test assumptions in the EN 1911 method. The plant had the following characteristics:

- biomass power plant CHP
- Fuel 70% biomass + 30% coal and peat
- If Flue gas heat recovery (FGR) is operating, then the flue gas temperature is about ~50°C and the moisture ~11 vol-%
- If FGR is not in use, the temperature is ~140°C and the moisture ~15 vol-%
- Emission limit value for HCl 47 mg.m^{-3}(n), given as daily average
- During the measurements, FGR was not used since then HCl levels are typically very low. The aim in this field campaign was to have ¨measurable¨ levels of HCl

About the measurements:

- Non-isokinetic sampling
- ”Main stream” approach
- Out-stack filter, quartz filter, 47 mm
- Absorber type: ”normal”, volume of the absorption solution used ~80 ml
- Ammonia was not present in the flue gas
- SO_2 levels ~35- 40 ppm
- Moisture 19-20 %
- O_2 4,5- 5,1 % (dry)

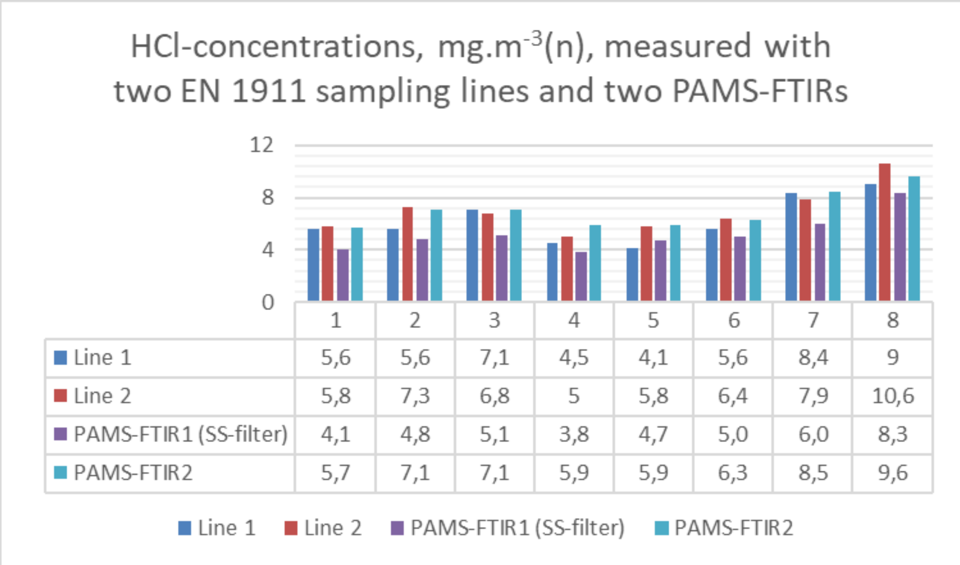
The same impingers and sampling lines were used one day, then changed to new ones. Field blanks were taken before and after the measurements in order to test if any HCl was left in the impingers. Absorption efficiency was checked once during the measurement campaign. Leak tests were performed before the measurements and also, during the sampling, by measuring O_2 at the end of the sampling line

Observations from the measurements:

- Field blanks were below LOQ both before and after the sampling. This demonstrated that the rinsing efficiency was good so no chlorides were left in the impingers. According to chapter 5.3.3.3 in EN 1911, the glassware can be re-used after rinsing, which this test proved is acceptable.
- Checks for absorption efficiency showed that both lines results were ok (in the last absorber concentrations were < LOQ)

Eight samples were taken using EN 1911, with HCl concentrations varying between 4-11 mg.m^{-3}, using two sampling lines. The measurement uncertainty for the EN 1911 sampling lines was calculated using the GUM approach for a 6 mg.m^{-3} concentration. This was 1.3 mg.m^{-3} or 23% of the measured concentration. In this calculation, the uncertainty coming from the analysis was used, not the standard deviation of the analytical repeatability of chloride analysis (which is mentioned in EN 1911).

Based on these studies, the results obtained with the two EN 1911 sampling lines (see Figure 5, line 1 and line 2) were consistent.



	1	2	3	4	5	6	7	8
Line 1	5,6	5,6	7,1	4,5	4,1	5,6	8,4	9
Line 2	5,8	7,3	6,8	5	5,8	6,4	7,9	10,6
PAMS-FTIR1 (SS-filter)	4,1	4,8	5,1	3,8	4,7	5,0	6,0	8,3
PAMS-FTIR2	5,7	7,1	7,1	5,9	5,9	6,3	8,5	9,6

Fig 5. Results from the field test campaign at biomass power plant, Finland

ILC organised by Ineris on the implementation of the EN 1911 standard

Objectives of the ILC organised on the Ineris test bench

- Evaluate the ability of the standard reference method to measure low concentrations, in relation to the lowering of the ELVs following the revision of the BREFs (Best Available Technique Reference Document)
- Evaluate the contributions of sampling and analysis to the measurement repeatability
- To look for improvements of the reference method to reduce the measurement uncertainty

Organisation of the ILC

- 9 French participants, accredited for EN 1911 measurements for sampling by COFRAC (French committee of accreditation)
  Each participant sent their samples to their usual analytical laboratory (accredited analytical laboratory).
  The participants were chosen so that the analytical laboratories were different.
  The variability of the measurements considers:
  - The variability between the sampling laboratories
  - The variability between the analytical laboratories

  Each laboratory was asked to provide:
    - the analytical LoQ and the measurement LoQ
    - the expanded uncertainty associated with the analysis of each sample and the expanded uncertainty associated with each measurement including the analytical contribution and the sampling contribution, calculated by “GUM approach” (ISO/IEC Guide 98-3 "Guide to the Expression of Uncertainty“) (GUM), consisting of establishing an uncertainty budget).
- 15 trials of 60 minutes each; quality controls: leak tests before each trial, field blanks, absorption efficiencies

Implementation of EN 1911 conditions:

- Each team implemented 2 independent sampling systems to evaluate the repeatability of the method.
- The sampling systems were with out-stack filtration, and could be either with or without side stream.
- Non isokinetic sampling required: isokinetics was not required in the absence of droplets
- **T**emperature filtration: in EN 1911: at least 20 degrees above the dew point (§ 5.2.1.1.2)

  - Gas temperature during trials: 137-142°C; water vapor dew point < 60 °C
  - Filtration temperature during ILC: the usual temperature applied, which is 180°C for some participants, and always 160°C for other, except for 2 trials: 180°C for all participants
- Duplicate analysis of each sample under repeatability conditions in order to evaluate contribution of sampling and of analysis in intralaboratory variability
- Analyses by the same analytical laboratory for 3 trials and 3 participants who had kept the absorption solutions: the analyses were repeated by the same analytical laboratory (Ineris) to evaluate the differences in concentrations calculated for these trials, depending on whether the analyses were carried out by the same laboratory or different laboratories
- Matrix:
  - Possibility of varying the HCl concentration by spiking of the matrix
  - Test of influence of NH_3 and SO_2: the boiler flue gas was also spiked with other compounds and in particular NH_3 and SO_2 to assess the potential impact of these 2 compounds on HCl measurement

    In case of NH_3 it can lead to the formation of ammonium salts in the duct, but also in the sampling line (e.g. on the filter) if the temperature is lower or equal to the temperature of salt formation; if salts are formed and deposited at parts of the sampling line that are not flushed, the measurement may be underestimated.

Figure 6: Ineris test bench

Data processing:

- In accordance with ISO 13528 / 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

Main conclusions:

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 (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 ½ ICR (half confidence interval of reproducibility) > 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 that evaluates the variability for one measurement / implemented by several measurement laboratories, using different equipment 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
- Analysis uncertainties declared by participants were equal for the 15 tests for most participants: were they really?
  Analysis uncertainties need to be refined to improve the estimation of measurement uncertainties
- In the GUM approach, the contribution of analysis in measurement 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.

In France analytical laboratories have to estimate the analytical uncertainty and at least 3 concentration levels of the validated concentration range [LoQ analysis - Cmax], because the analytical uncertainty is probably not the same at the LoQ level as at Cmax.

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 $mg.m^{-3}$, expanded uncertainty criterion is < 1.5 $mg.m^{-3}$.

The combination of testing from VTT and Ineris has demonstrated that assumptions about the EN 1911 method are correct and viable. The results also identify and characterise the general performance of the method. This meets in full the requirements for this objective.

4.3 To produce a metrologically valid evidence base of uncertainty contributions to EN 1911 and from this to develop a series of recommended uncertainty requirements for regulatory monitoring.

The EN 1911 standard sets out the standard reference method for measuring HCl in emission streams. Uncertainty in the method comes from two main areas, the sampling and the laboratory analysis. Both areas need to be understood and quantified in order to limit overall uncertainty in these measurements, something that is increasingly important as emission limit values are lowered, resulting in a related tightening of uncertainty requirements, which are typically a percentage of the emission limit values (ELVs), e.g. EN 1911 stipulates a maximum permissible uncertainty of 30% of the ELV. Additionally, national regulators can apply additional requirements for EN 14181 calibrations of automated monitoring systems, e.g. Environment Agency (EA) in England specifies that reference methods should meet a 20% of the ELV uncertainty requirement.

This project has investigated the uncertainty sources from both key areas to identify areas with potential improvements for lowering uncertainty. Collaboration between NPL, Ineris, VTT and CMI has unlocked a greater degree of understanding than would have been possible doing the research individually.

Chronologically the first source of uncertainty in EN 1911 comes from the sampling. There are several types of impinger setup that are used, but little previous evidence that all types are truly equivalent. VTT set up tests in their laboratory to compare the performance of four different glassware set ups by parallel measurement.

VTT tested four different impinger tips, over a set of 15 tests. A Hovacal calibrator was used to produce 2 $mg.m^{-3}$ HCl and an FTIR analyser was used to measure the produced concentrations (Figure 7). Figure 8 illustrates the level of variation between the different impinger tips and the FTIR and Hovacal results for each test. The statistical treatment to calculate the repeatability and reproducibility was carried out with the principles of ISO 5725-2 (Accuracy (trueness and precision) of measurement methods and results - part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method)

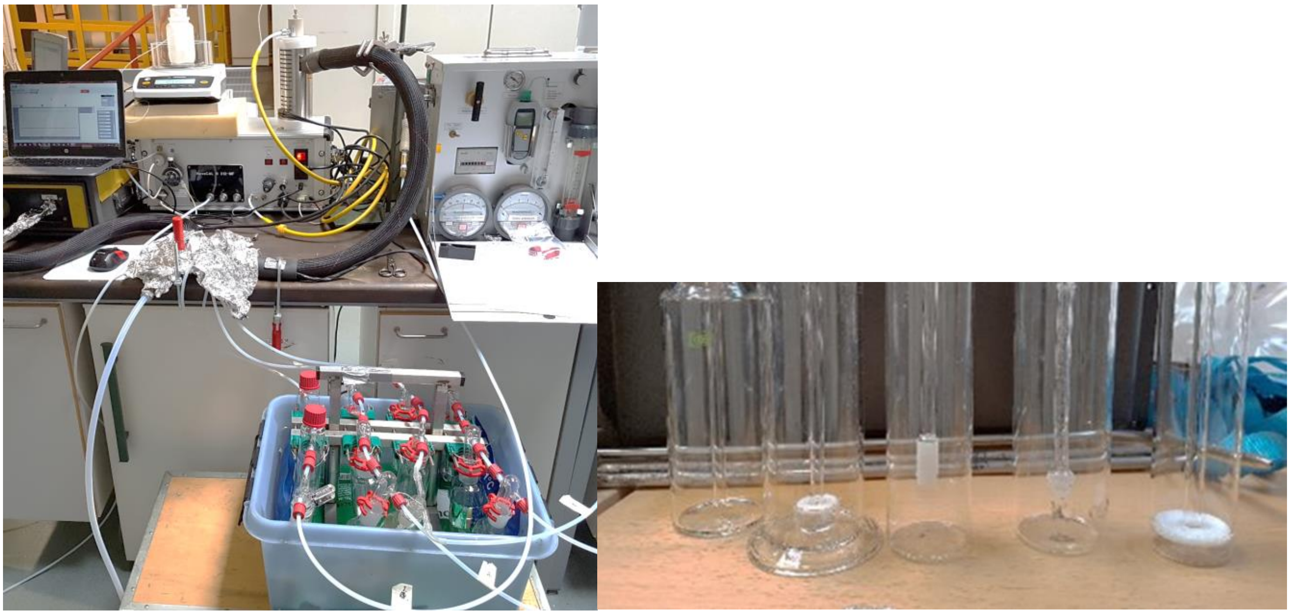
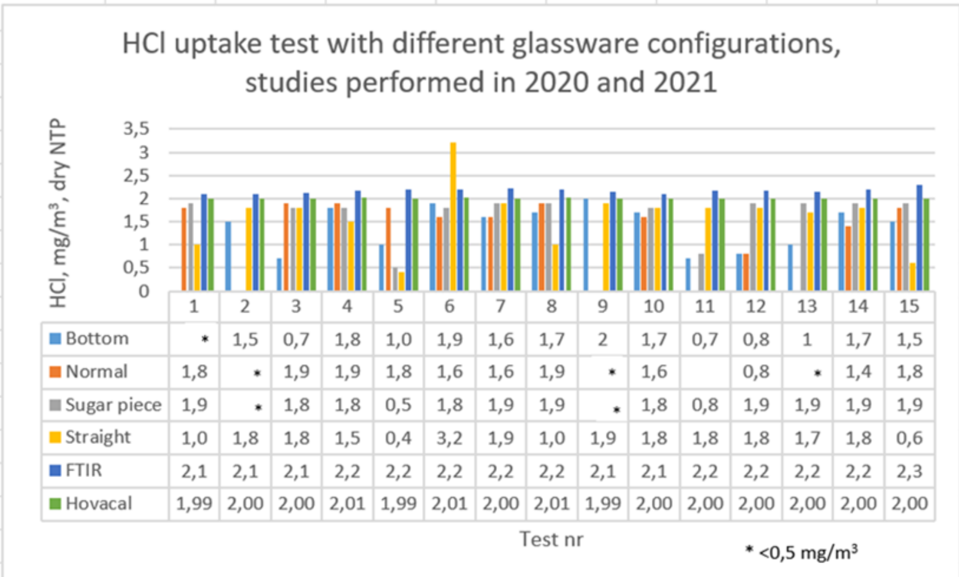


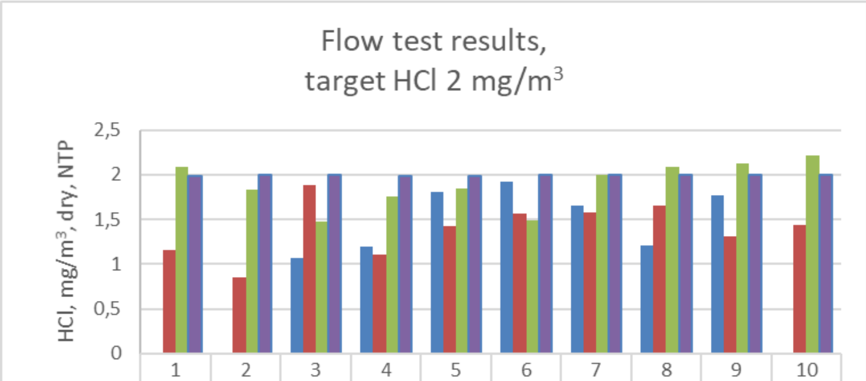
Fig. 7 Experimental set-up for glassware tip tests and tested impinger tips



	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
Bottom	*	1,5	0,7	1,8	1,0	1,9	1,6	1,7	2	1,7	0,7	0,8	1	1,7	1,5
Normal	1,8	*	1,9	1,9	1,8	1,6	1,6	1,9	*	1,6		0,8	*	1,4	1,8
Sugar piece	1,9	*	1,8	1,8	0,5	1,8	1,9	1,9	*	1,8	0,8	1,9	1,9	1,9	1,9
Straight	1,0	1,8	1,8	1,5	0,4	3,2	1,9	1,0	1,9	1,8	1,8	1,8	1,7	1,8	0,6
FTIR	2,1	2,1	2,1	2,2	2,2	2,2	2,2	2,2	2,1	2,1	2,2	2,2	2,2	2,2	2,3
Hovacal	1,99	2,00	2,00	2,01	1,99	2,01	2,00	2,01	1,99	2,00	2,00	2,00	2,00	2,00	2,00

Fig 8 HCl uptake tests

Another variable in the EN 1911 sampling method is the flow rate through the impingers. To test this two different collection flow rates were tested, 1.5 lpm and 8 lpm (Figure 9).



	1	2	3	4	5	6	7	8	9	10
1.5 lpm			1,1	1,2	1,8	1,9	1,7	1,2	1,8	
8 lpm	1,2	0,9	1,9	1,1	1,4	1,6	1,6	1,7	1,3	1,4
FTIR	2,1	1,8	1,5	1,7	1,8	1,5	2,0	2,1	2,1	2,2
Hovacal	1,99	1,99	2,00	1,98	1,98	1,99	2,00	1,99	1,99	2,00

Figure 9: Flow rate test results

Based on these calculations, the *statistical difference for both Repeatability and Reproducibility is negligible*, both for:

- Different flow rates (1.5 l/min and 8 l/min) and
- Different impinger tips

Demonstrating the equivalence of these variables is an important finding that provides added confidence in the performance of the method.

In addition to the effects of the measurement equipment, sampling location can have a significant effect on measurement results. CMI have used computer fluid dynamic modelling in the past to look at the effects of obstructions and direction changes on the flow of emissions in a stack. These can introduce swirl and inhomogeneity to the flow and those tests highlighted the importance of following the sampling site selection guidelines in EN 16911. HCl is known to “stick” to the walls when sampling, so CMI used their expertise in CFD modelling, supported by NPL, to investigate the impact this would have. CMI also investigated the potential for cross interference when flow and concentration probes interact (e.g. S-type pitot and isokinetic sampling probe).

Tests were performed in a wind tunnel at CMI. Since emissions can be reported as mass emissions the flow rate is an important variable, so operators need to be able to measure it in a way that will limit impact on the concentration measurement. Tests were made with an S-type Pitot and six configurations of sampling probe and filter holder. Different layouts led to variation of 1.6% on the calibration of the Pitot. This is an uncertainty source that is not generally considered, but the CMI results demonstrate that it will have an impact on the overall measurement uncertainty.

Once the samples have been collected, they are sent to a laboratory accredited for EN 1911 to carry out the analysis. To investigate the real-world laboratory uncertainty NPL carried out a blind laboratory inter-comparison, sending out samples with known levels of Cl^-, disguised as general stack emission monitoring samples. This was carried out in two rounds, one to accredited UK laboratories and another to accredited laboratories in UK and Europe. This work ascertained the analytical uncertainty phase of EN 1911.

The UK only tests had two different types of samples, synthetic samples (‘Synthetic’) and real samples taken from the NPL Stack Simulator facility (‘s-Real’). The synthetic samples were prepared by dissolving NaCl in de-ionised water to produce samples with metrologically traceable known chloride concentrations, equivalent to concentrations of 1.9, 3.9, 5.9, 7.7 & 9.8 mg.m^{-3}. Each solution was split and sent to six laboratories accredited to ISO/IEC 17025 for chloride analysis in accordance with EN 1911. The s-Real samples were collected from the stack simulator following the EN 1911 procedure by staff qualified to MCERTS level 2 under the EA personal competency standard. The simulator had realistic emission matrices containing representative concentrations of expected potential cross interfering species (SO_2, CO, NO, H_2O, CO_2, O_2, CH_4, VOCs), providing a more realistic analysis challenge. Exact chloride levels of the s-Real samples were not known, but as with the synthetic samples each sample was split and sent to three of the accredited laboratories for analysis. None of the laboratories was aware of the testing and assumed these were standard stack emission samples, so the results should be representative of the actual laboratory uncertainty for this method.

Results from the s-Real tests showed far greater variation than the synthetic samples (Figure 10), with 42.3% of sample results deviating from the mean by more than the stated laboratory uncertainty, compared with just 5% for the synthetic samples. This indicates the potential scale of issues caused by cross interference in real sample matrices. Generally synthetic samples are used for proficiency testing, but this test has indicated that this may provide an optimistic view of the achievable repeatability of EN 1911 analysis.

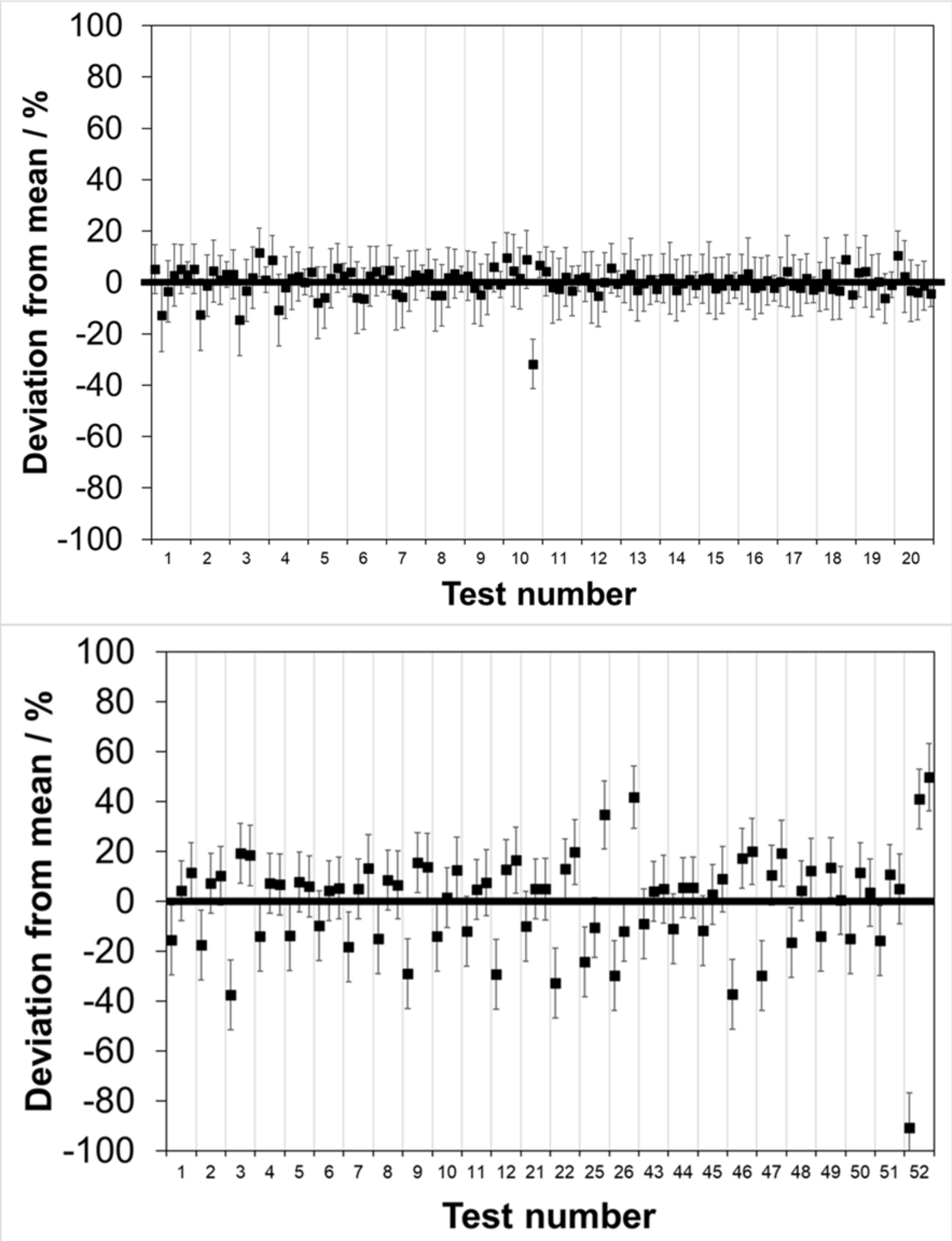


Figure 10: Plots of the reported values and stated laboratory uncertainties in terms of deviation from the mean for all samples in the 0-10 mg.m^{-3} range (synthetic on top and s-Real on the lower plot)

The deviation in the s-Real results were compared to the limits stated previously (30% of ELV for EN 1911 and 20% of ELV for EA calibration requirement) to indicate the ability of EN 1911 to meet the uncertainty requirements at low ELVs. After consulting with sampling teams an estimate of 7.2% of the value was given as a potential sampling uncertainty. This was then subtracted from the overall uncertainty limit to indicate the uncertainty available for the analysis. The results were plotted for ELVs of 10mg.m^{-3} (0-15 mg.m^{-3} range) and 3mg.m^{-3} (0-4.5 mg.m^{-3} range) to indicate current performance of the method at these levels (Figure 11). Some of the more recent best available technique reference document conclusions (BATC) have AELs as low or lower than 3mg.m^{-3} so these uncertainty levels will soon be required.

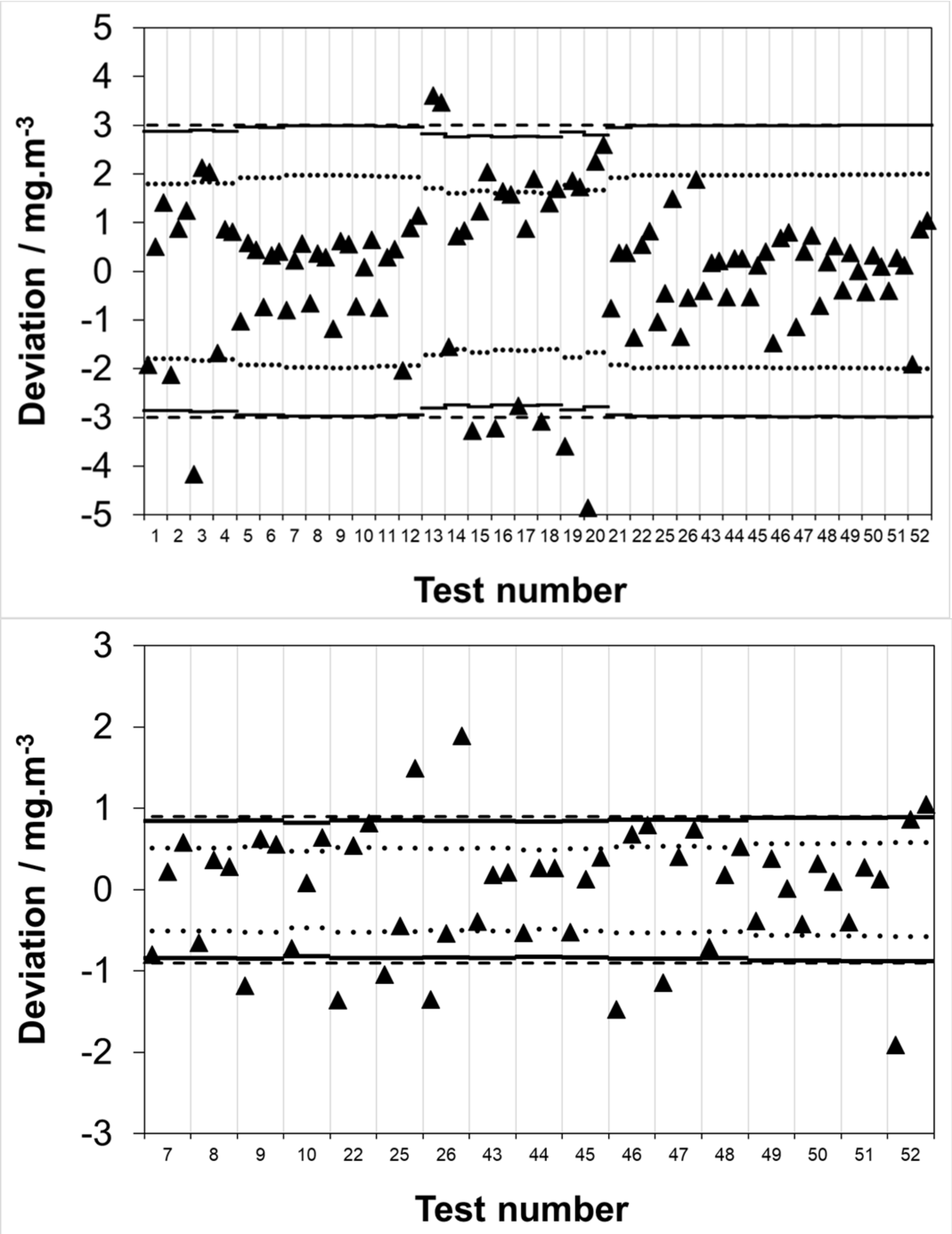


Figure 11: Plots of deviation from the mean value for s-Real samples and lines indicating the allowable uncertainties at 30% of ELV (---), 30% of ELV less 7.2% of value for sampling uncertainty (▬) and 20% of ELV less 7.2% of value for sampling uncertainty (…), at 10mg.m^{-3} ELV (top) and 3mg.m^{-3} ELV (lower)

Combining the findings from VTT, CMI and NPL on uncertainties for different sections of the measurement process, along with the Ineris and NPL test bench experiments, provides a more complete picture of the actual achievable uncertainty for using EN 1911 for HCl emission measurement. By breaking down the uncertainty in this way, we can target the areas where most improvement can be made. This work meets the objective in full.

4.4 To create a scientific evidence base of the performance of a range of portable optical technologies, using the extended Stack Simulator capability developed in objective 1, with a view to the future replacement of the existing SRM. This will underpin current work at CEN on CEN/TS16429 (optical techniques) and CEN/TC264/WI00264151 (Fourier transform infrared spectroscopy).

Given the limitations of EN 1911 it is necessary to assess the capability of alternative methods to replace EN 1911 as the potential new SRM. In order to make this change the uncertainties of the alternative method would have to widely demonstrated and supported by a scientific evidence base. This objective was to put together the evidence to support (or dismiss) the potential for optical techniques, including FTIR, to fulfil this role in future.

NPL and VTT have carried out performance testing for portable optical technologies, NPL using their test bench facilities that have been described above, while VTT did parallel testing using a real stack. The VTT approach is a true representation of the measurement challenges (cross interferents, dust, hot/wet sampling, etc.), but it is not possible to control the emission concentrations, so limits the experiments that can be completed. The NPL test bench has control of the test emission matrix so can investigate specific scenarios, including very low HCl concentrations, but are a less realistic test of the overall measurement method. CMI also completed some CFD modelling work to look at the impact of particles within the gas stream.

During the VTT field campaign measuring a real stack at a biomass combustion plant, the experiments with EN1911 equipment were done in parallel with testing alternative FTIR sampling set ups (Figure 12). The FTIR needs particulate free sample gas, since particulates will adhere to optical surfaces, altering the measurement behaviour of the instrument. To remove particulates from the gas stream filters are deployed within the sample line. The performance of different filter mediums will impact on the measurement uncertainty and measured quantities. VTT tested PTFE, stainless steel and ceramic filters to investigate the significance of this factor on overall uncertainty.

Two Gasmet portable FTIR analysers (DX 4000) were used, with two individual sampling systems (heated filters, heated lines). For FTIRs the field quality checks were completed with an HCl feed from a Gasmet portable syringe calibrator (tested also in lab before the measurements), with wet gas.

About FTIR measurements:

- Two independent sampling and analysis equipment
- 2 * Heated sampling probes 1.3 m, 180°C
- 2 * Heated filters (2*M&C PSP4000) 180°C
- 1st line had all the time stainless steel filter F-3SS
- 2nd line had alternately filters: Stainless steel F-3SS, Teflon M&C F-2T and ceramic M&C SP-2K
- Heated lines 10 m and 6 m, after pump 1 m lines to analysers, all 180°C
- 2 * Portable sampling units incl. pump and integrated oxygen measurement
- 2 * FTIR's (Fourier Transform Infrared) Gasmet Dx4000 were used in the measurements

FTIRs were operated according to the instructions given in CEN/TS 17337. Below are given some observations from the checks that were made during measurement campaign.

Injecting and vaporizing HCl solution was performed according to wet gas injection approach

- Hovacal vaporization unit with scale, used in laboratory
- Gasmet portable syringe vaporizing unit, on field

Zero check performed in the field (according to CEN/TS 17337):

- Each day at the start and the end

- All of the measured compounds shall not exceed 2,0 % of respective certification range
- "Certification range" of 10 mg.m^{-3} was used in calculations
- All checks were accepted (see Table 2)

Table 2. Zero Checks for FTIRs during field campaign

		FTIR 1			FTIR 2			Criteria	Notes
		Day 1	Day 2	Day 3	Day 1	Day 2	Day 3		
zero start	HCl mg/Nm^3	0,08	0,11	0,13	0,08	0,02	0,06		
zero end	HCl mg/Nm^3	0,00	0,00	0,07	0,13	0,02	0,00		
zero start	% of range (10)	0,8	1,1	1,3	0,8	0,2	0,6	< 2	accepted
zero end	% of range (10)	0,0	0,0	0,7	1,3	0,2	0,0	< 2	accepted

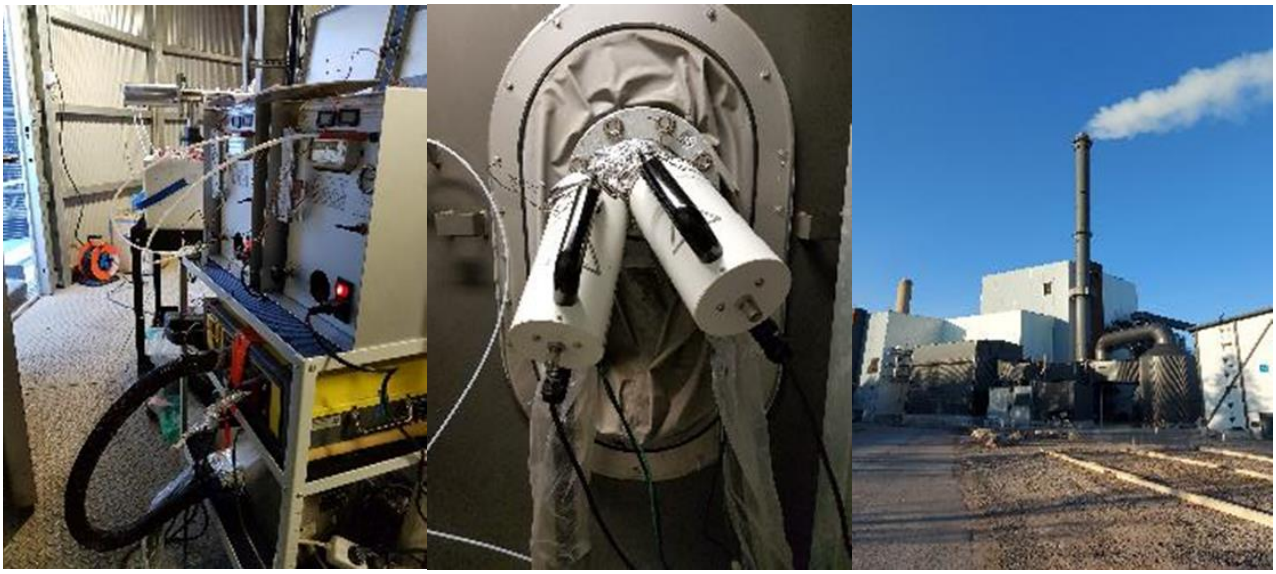


Figure 12: Field measurement set-up, with the photo on power plant

Response time tests in laboratory and in field:

- Laboratory: Both stainless steel filters had response time about 1 min, Teflon and ceramic filter had a response time between 2 and 3 minutes
- Field: Both stainless steel filters and ceramic filter had response time between 2 and 3 min (Teflon filter was not tested due to technical challenges of syringe system caused by low ambient temperature)
- All response times < 200 sec (requirement is < 400 sec), see Figure 13

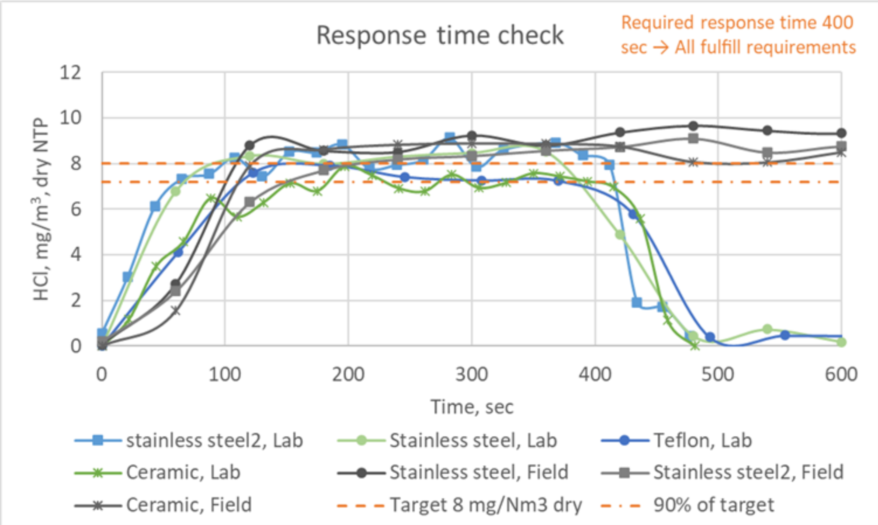
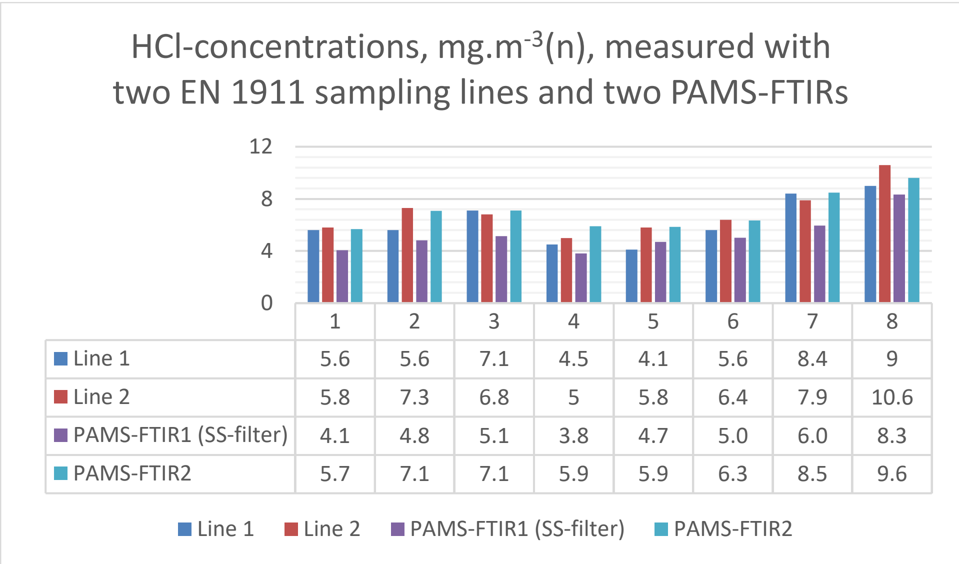


Figure 13: Response time checks for FTIRs



	1	2	3	4	5	6	7	8
Line 1	5.6	5.6	7.1	4.5	4.1	5.6	8.4	9
Line 2	5.8	7.3	6.8	5	5.8	6.4	7.9	10.6
PAMS-FTIR1 (SS-filter)	4.1	4.8	5.1	3.8	4.7	5.0	6.0	8.3
PAMS-FTIR2	5.7	7.1	7.1	5.9	5.9	6.3	8.5	9.6

Figure 14: HCl measured concentrations from parallel tests using two EN 1911 sampling lines and two FTIR instruments. One of the FTIR instruments (PAMS-FTIR2) used different filter materials for some tests to quantify that effect on the overall measurement quality

PAMS-FTIR 1 had stainless steel (SS) filter in all tests and **PAMS- FTIR 2** had:

- stainless steel (SS) filter during tests 1-3
- ceramic filter (C) for tests 4-6
- PTFE filter in tests 7-8

Measured concentrations varied between ~4–10 mg.m^{-3} (Figure 14). Based on these results, statistical testing demonstrated that there was no significant difference between the performance of the FTIR with any of the filter materials. The HCl measurement uncertainty using the FTIR, calculated from the principles of CEN/TS 17337, was shown to be <2 mg.m^{-3}, for a 6 mg.m^{-3} test concentration.

The NPL Stack Simulator facility, which was validated down to HCl concentrations of 1mg.m^{-3} during objective one, was used to compare different measurement technologies. Eight 30 minute rounds of testing were carried out with parallel sampling using EN 1911 and a variety of P-AMS. The intension when the test plan was devised was to include a broader selection of instrument types and for VTT and Ineris to be involved in the testing, but due to limitations in place during COVID, some instruments including a cavity ring down spectrometer (CRDS), were not included and only NPL took part. The final P-AMS instruments were three FTIR instruments, a tunable diode laser (TDL) system and a Non-Dispersive Infrared Gas Filter Correlation (NDIR-GFC) system, the last of which operates dry, so was run with a permeation drier. Heated probes and heated sample lines were used for all P-AMS (Figure 15). There were issues with the amount of time it took to condition and calibrate the dry system which resulted in some missed tests for the NDIR-GFC.

Figure 15: Sampling systems used in the NPL Stack Simulator facility during parallel P-AMS testing.

Table 3: Zero and span drift levels over the period of each test

% Drift (either zero or span)	
<2%	25% of checks
2-5%	31% of checks
>5%	44% of checks

The tests were bracketed with zero and span checks to detect instrumental drift. This used a wet gas calibration generator with aqueous HCl solution (<1% uncertainty) with a span value of 5-6 mg.m^{-3} (wet) with 9-10% water vapour. Some analyser drift was found (Table 3), which may suggest an issue with understanding the wet calibration uncertainties, with a maximum drift of 26% in one test. In general drift less than 2% is accepted, 2-5% would require correction and >5% drift would lead to rejection of the test, so this could be a significant issue.

When looking at the measurement uncertainties compared to the 20% of ELV requirement included in EN 16429:2021, these tests have highlighted the current limitations (Table 4). All uncertainties met this requirement for an ELV of 10 mg.m^{-3}, while 85% were acceptable for an ELV of 6 mg.m^{-3}. At 5 mg.m^{-3} only 48% met the requirement and the average uncertainty was 21% of the ELV, indicating that further improvement is required. EN 16429:2021 requires uncertainty of 1 mg.$^{-3}$ for ELVs below 5 mg.m^{-3} and 36% of uncertainty values met this with an average uncertainty of 1.1 mg.m^{-3}. Given this, a conservative estimate of 1.5 mg.m^{-3} would be an achievable uncertainty limit for ELVs below 5 mg.$^{-3}$ given current methodologies.

Table 4: Uncertainty of test results for tests at different ELVs

Example ELV	Average Uncertainty	Uncertainty spread
10	11% of ELV	5-17%
6	18% of ELV	9-29%
5	21% of ELV	11-35%
<5	1.0mg.m^{-3}	0.5-1.4mg.m^{-3}
	1.1mg.m^{-3} <5mg.m^{-3} Tests	0.5-1.7mg.m^{-3} <5mg.m^{-3} Tests

Particulates will impact performance of optical technologies, so CMI carried out computer fluid dynamic flow modelling work to investigate the distribution of particulates within the gas stream after various obstructions (e.g. joints in the duct or changes of flow direction). They compared the results to the requirements for measurement locations to estimate the potential impact this could have on point sampling techniques.

Motion of clouds of particles (droplets) with three different diameters of 10 mm, 20 mm and 50 mm has been investigated in three stack geometries generating different flow patterns by means of CFD (computational fluid dynamics) modelling in OpenFoam software. It was shown that flow patterns which are typical for stacks can lead to significant particle redistributions and inhomogeneities in particle concentrations in stacks. The larger the particles are the more significant the effect is. CMI found that concentrations at specific sampling points which are prescribed for isokinetic sampling can then be non-representative for the overall average concentration leading to measurement errors up to 50 %.

The combination of results from NPL, VTT, Ineris and CMI forms an evidence base for the performance of portable optical technologies, demonstrating their potential to provide future reference measurements for monitoring low HCl emitting processes regulated via the BAT conclusions. This meets the objective requirements in full.

4.5 To produce a National Regulator Guidance Document of recommended measurement uncertainty requirements. To disseminate this document to EU DG Environment (Directorate-General for Environment), Task Force Emissions, CEN/TC 264 'Air Quality', and more broadly, to the emission community.

National Regulators, like the Environment Agency (EA) in England, set monitoring requirements based on national and European requirements. This requires knowledge of the measurement landscape for all monitored pollutants. Therefore the EA produced a national regulator paper on the current state of HCl emission monitoring from stacks as part of the project to demonstrate the issues involved with these measurements.

In England EN 1911 is the most commonly used method, but the EA also recognise the benefits of using automated methods, allowing the use of EN 16429 (P-AMS using infrared measurement method) and CEN/TS 17337 (FTIR). Using P-AMS for calibration provides real time results, is less labour intensive and can have lower uncertainties and data quality compared to the wet-chemistry based method. Since the EA have accepted their use for regulatory monitoring, this enables development of new techniques if they can demonstrate equivalence.

EN 1911 is specified in the large combustion plant (LCP) and non-ferrous metal industries (NFM) BATC for periodic monitoring, since these specify gaseous chlorides expressed as HCl. Measuring gaseous chlorides could result in systematically higher concentration compared to an automated method directly measuring HCl. However for continuous monitoring all AMS are measuring HCl, so there is an issue calibrating with a method that is measuring a different measurand. Since EN 16429 and CEN/TS 17337 measure HCl the EA don’t need them to prove equivalence with the manual method.

As EN 16429 was only released in 2021, no English test laboratory has chosen to use it and it is not yet referenced in any of their regulatory guidance. The national regulator guidance document published by EA as part of this project asks if there are any suitable P-AMS other than FTIR currently available but expects to allow its use.

CEN TS 17337 has been more widely used and in the UK this is an approved alternative method for HCl. FTIR is beneficial for the test laboratory since the HCl measurements can be combined with the measurement of other pollutants also measured by it. It is therefore used for QAL2 and ASTs, mainly at waste incinerators. The P-AMS should be tested to EN 15267-4, but no P-AMS yet meet this, so the EA allows P-AMS meeting EN 15267-3 for now.

EA reviewed 31 EN 14181 calibration reports for continuous HCl AMS. Several test laboratories have been using both manual and automated methods, with a slight preference for the later (55%). Both methods were meeting the uncertainty requirements specified by legislation based on ELVs at the time of testing. Comparing results for QAL2 and AST reports suggests that EN 1911 could result in systemically higher concentrations than automated methods. Nine of the twelve campaigns using EN 1911 produced results below the AMS result, suggesting a negative bias in practice. In contrast TS 17337 did not show a bias compared to the AMS.

In chapter IV of the IED for waste incineration the daily ELV for HCl is 10 mg.m-3, whereas the BAT AEL for new plants is 2-6 mg.m^{-3} (2-8 mg.m^{-3} for existing plants). Due to the challenging uncertainty requirement the UK will be adopting at the high end (6 mg.m^{-3} for new plants) due to the issues there might be with legally enforcing the lowest limits.

EN 1911 requires overall uncertainty of <30% of ELV. EN 16429 requires an expanded uncertainty of <20% of ELV or 1 mg.m^{-3} for ELVs below 5 mg.m^{-3}. The EA national regulator guidance requires CEN TS 17337 to have an expanded uncertainty of 25% of ELV, although this is not specified in the standard. So only the more recent EN 16429 considers the issues with uncertainties as ELVs fall further, by introducing a fixed uncertainty at the lowest ELVs.

To ensure the findings of the project have a significant impact on the broader emission monitoring community there are a number of dissemination channels that will be used. Beyond the overarching uncertainty results required by IED and the BAT Conclusions, national regulators can require additional uncertainty information. To set out these additional requirements and provide general information on all implementation issues national regulators produce guidance documents. Based on the findings of this project the English national regulator, the EA, has produced a new guidance document as a project deliverable.

This suggests a number of points to be considered for the revision of EN 1911, based on the findings of the Heroes project:

- It should include an estimation of uncertainty of the analysis, rather than only a determination of the repeatability of the analysis. The repeatability of the analysis is only one component of the uncertainty associated with the analysis. Currently a laboratory would be acceptable if it under or over-reads as long as it is consistent, ignoring the bias that this would introduce. Limit values for precision and bias are specified in the MCERTS performance standard for laboratories testing samples from stack emissions monitoring. For analytical laboratories they could also apply ISO 11352 “Water quality – Estimation of measurement uncertainty based on validation and quality control data”, which would satisfy this requirement, which the French regulator already requires. France also requires that uncertainty be determined at least at the limit of quantification (LoQ) and two other points on the analytical range.
- It is necessary to clarify how analytical results below the limit of detection or LoQ are to be treated, and that uncertainties associated with concentrations <LOQ cannot be determined and are not considered.
- It is necessary to define a concentration level above which the measurement uncertainty is set in relative value, as a percentage of the concentration, and below which the uncertainty criterion is set in mg/m^3 and therefore becomes constant.

Project partners have contributed to the following working groups of CEN TC 264:

- Task Force Emissions (TFE) – NPL, Ineris
- WG 3: Emissions – HCl and HF – Ineris, NPL
- WG45: Emissions – Proficiency testing schemes – NPL, VTT

EA, Ineris, NPL and VTT have also contributed to their respective mirror committees for CEN TC 264 in their respective countries. On top of this NPL have contributed to ISO TC 146 SC4/WG9, working on a technical report on validation of measurement methods.

Project partners have ensured that the findings of the Heroes project have been communicated to the wider emission monitoring community, through standardisation, trade journal and peer review publications, along with workshops and training sessions. This has ensured that the work has significant impact on future implementation of HCl measurements. This meets in full the requirements for this objective.

4.6 To contribute to a revision of EN 1911 by providing the data, methods, guidelines and recommendations, necessary for new uncertainty requirements for HCl industrial emission limits, to CEN/TC 264.

Various deliverables from the Heroes project have produced new data to verify aspects of the existing methods (e.g. validation of different EN 1911 equipment carried out by VTT), investigate the impacts of particulates within the gas stream (e.g. CMI flow modelling work) and contribute to adoption of instrumental measurement techniques (e.g. testing of FTIR and other optical P-AMS by NPL). The EA, with the help of other project partners, produced a report on the current state of HCl monitoring and the challenges facing the industry as emission limits evolve. By expanding upon the existing validation data available for EN 1911, the Heroes project has demonstrated what is working and highlighted the main areas for improvement in the future revision of EN 1911.

EN 1911 sits within a suite of emission monitoring standards which make up the quality assurance framework for monitoring in this field (Figure 16). Changes to the standards around EN 1911 will have an impact on how it will be revised in future. Partners have contributed to the whole web of related standards to ensure that revision of EN 1911 will complement and build upon the recent developments.

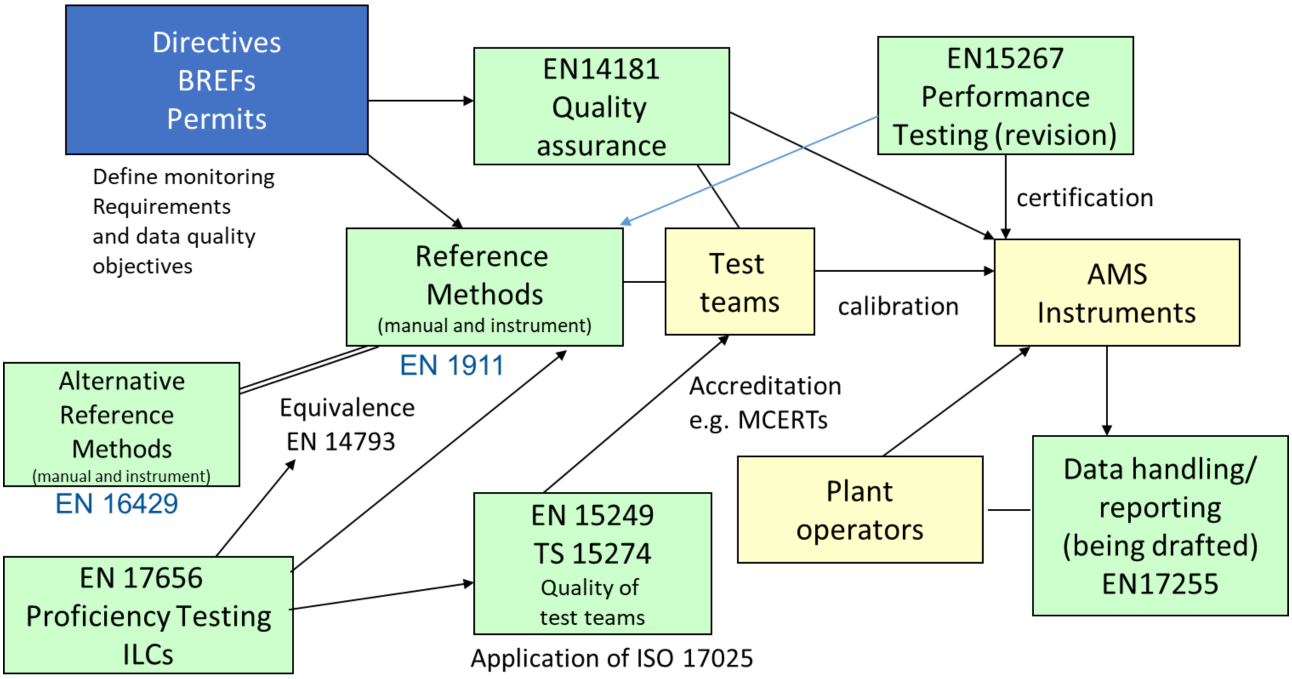


Figure 16: Visualisation of the stationary source emission monitoring standardisation landscape, indicating the wide variety of standards required to regulate this field

NPL and Ineris have contributed to a working group developing a technical report to be used as a guideline for developing future measurement method standards, which will obviously influence the future revision of EN 1911. This will include the improved handling of analysis uncertainty that has been developed for EN 1911 in the Heroes project. This technical report is expected to be published in 2023.

NPL, Ineris and VTT have also contributed to standardisation of other HCl methods, including EN 16429, which was published in 2021 and outlines a reference method for determining the concentration of gaseous hydrogen chloride. This instrumental method directly measures HCl, rather than total chlorides measured by EN 1911. Validation studies for the new standard were carried out on the NPL Stack Simulator facility down to concentrations of 3 $mg.m^{-3}$ along with field validation at waste incinerator sites.

Additionally, NPL and VTT contributed to the development and publication of EN 17656, which sets out stationary source emission monitoring specific proficiency testing requirements. Regular proficiency testing checks the ability of test laboratories to meet their monitoring requirements and is required by National Regulators like the EA in order to demonstrate ongoing competence. Heroes has demonstrated that the test benches at NPL and Ineris have the capability to implement HCl PT schemes.

Project partners have produced large amounts of data on quality control for the EN1911 reference method for HCl, that will be fed into a future revision of the standard. In addition to this work has been done on the standardisation of related stationary source emission monitoring areas. This meets in full the requirements for this objective.

5 Impact

The consortium has been active in many standardisation activities and has given presentations to different groups, as described below. The dissemination highlights are as follows:

- Two invited presentations given at Air Quality and Emissions conference (AQE2021):
  - ‘Measurement Uncertainty – Performance of methods at low concentrations’
  - ‘Do existing standard reference methods remain fit for purpose give increasingly stringent emission limits?’
- Presentation ‘Introducing the HEROES project’ given at SR 215 Air quality, Oct 2019, Helsinki, Finland.
- Presentation ‘Introducing the HEROES project’ given at French standardisation committee X43B Air quality - Stationary source emissions.
- Presentation ‘EURAMET Pre-Normative Metrology Research Activities’ given at CEN/TC 264 Annual Plenary, May 2019, Copenhagen, Denmark.
- Representations at: UK, French and Finnish national mirror groups; Source Testing Association.
- Two training courses have taken place as part of a 3-day course ‘Industrial Air Pollution Monitoring’, hosted by University of Leeds Faculty of Engineering and Physical Sciences entitled:
  - ‘Calculations of uncertainties in stack monitoring‘;
  - ‘Understanding instrument performance standards’
- A poster presentation was made at the International Metrology Congress in September 2021. This covered the objectives of the Heroes project and the ILC on the EN 1911 method.
- Presentation ‘Heroes project and it’s results’ given at SR 215 Air quality, Oct 2022, Finland
- Presentation at the FLOMEKO 2022 conference, "Modelling of uncertainties of an emission concentration measurement in stacks”, November 2022.
- Four presentations at the Emission Monitoring conference (CEM 2023):
  - Evaluation of P-AMS for measuring HCl at the NPL stack simulator in order to meet the requirements of EN1649:2021 (Instrumental reference method for HCl) under increasingly stringent emission limit values
  - Appropriate measurement places and their importance in emission measurements
  - Does the performance of the HCl standardized reference method remain suitable in a context of lowering emission limit values?
  - Standardising proficiency testing for emission monitoring: EN 17656:2022
- Publication of the standard EN 17656:2022 Stationary source emissions – Requirements on proficiency testing schemes for emission measurements, October 2022.
- Baltic workshop presenting the outcomes of the Heroes project to an audience of regulators, stack testers, analyser manufacturer, plant operators and researchers, October 2022, Finland.
- A training course on “Performance of measurement methods: LoQ, measurement uncertainties” as part of a 3-day “Monitoring of air pollutant emissions” in-company training course, France
- Trade journal article: Pienten HCl-päästöpitoisuuksien mittaamisen haasteet- Heroes-projektin havaintoja, November 2022, Finland.
- End of project workshop for Heroes Stakeholders, January 2023, Paris, France.

Impact on industrial and other user communities

The project partners have presented findings from the project at workshops and/or training courses for industry. NPL made two presentations as part of a three-day training course in the UK on “Industrial Air Pollution Monitoring”. VTT organised a workshop in October 2022 for representatives in the Baltic nations, including regulatory and industrial figures, entitled “Observations from the HEROES project”. This covered a wide range of findings from the project including presentations given by other project partners. A trade journal article was also published to communicate the findings to relevant communities in Finland. In November 2022 INERIS included their HEROES ILC results as part of a 3 day “Monitoring of air pollutant emissions” in-company training course, which is usually run twice a year. EA periodically meet with Scottish and Welsh national regulators and have communicated findings from the project to ensure consistent implementation of the findings across the UK. The consortium also hosted an online end of project workshop for the whole user community, at which all findings of the project were presented by representatives of all the project partners.

Multiple presentations have been made at conferences focussed on emission monitoring: the Air Quality and Emissions conference (AQE) and the Conference on Emission Monitoring (CEM). These large-scale conferences have audiences of over two hundred delegates from across the industry, regulators and instrument manufacturers, communicating the findings of the HEROES project to the key stakeholders.

By communicating the findings of the project in this way, the wider community will be more aware of the issues around uncertainty for monitoring very low concentrations and, more specifically for HCl monitoring, areas of EN 1911 that are likely to be improved upon when it is revised. This will allow the industry to adopt best practice techniques validated by the HEROES project as soon as possible, making emission measurements better.

Impact on the metrology and scientific communities

By validating some aspects of the existing measurement methods (e.g. variations in impinger tips, different sampling head configurations, etc.), which were previously assumed to be equivalent, the project has quantified uncertainties and highlighted the areas that require the most and least focus as part of future revisions. In particular the parallel testing of optical P-AMS has demonstrated on-going issues with measurement uncertainty for monitoring HCl at the lowest concentrations under the EN 16429 methodology. Along with the work on characterising a more complete uncertainty calculation for EN 1911, covering both sampling and analysis in a more complete way, will guide future work to improve equipment and implementation of these methods.

The HEROES project was represented at the International Metrology Congress (CIM2021) with a presentation and a poster, highlighting the metrological need for the project and how it would benefit industrial emission measurement and reporting. Results were presented by CMI at the FLOMEKO 2022 conference which focuses on metrological issues around measurement of flow, since flow measurement is an important part of converting the concentration measurements into reportable mass emissions.

A peer-reviewed article by NPL in the journal Metrologia investigated the impact of different flow measurement reference methods on overall reported mass emission uncertainty. A further paper by CMI and NPL on modelling uncertainties of emission measurement will be submitted to the journal Measurement. EA and INERIS are drafting another paper on historical trends of AMS calibration using EN 1911 or P-AMS to be submitted to the Journal of the Air and Waste Management Association. NPL and INERIS are drafting a fourth paper on trends in the proficiency of accredited service providers performing measurements in accordance with EN 1911, which will also be submitted to the Journal of the Air and Waste Management Association. A final peer review publication on the uncertainty associated with measuring new low level HCl emissions associated with BAT conclusions emission limits will be submitted to the Journal of the Air and Waste Management Association.

Impact on relevant standards

One of the main aims of the HEROES project was supporting the revision of the existing standard reference method, EN 1911. Through the evidence base compiled by the project partners there is now a compelling case for this revision to take place, with a focus on improving the uncertainty quantification and how it is handled at very low measurement concentrations. One of the project deliverables is a draft uncertainty annex for the revised standard, setting out the required changes for meeting uncertainty requirements at lower emission limits.

When the EN 16429 standard was published in 2021 it provided a standard reference method for HCl measurement by optical P-AMS. However, no instruments had been validated against the requirements of the new standard. The HEROES project has produced some data on implementation and highlighted the areas where instruments need to be able to improve in order to meet the required uncertainty levels for the lowest upcoming emission limit values.

NPL and VTT contributed to the development of EN 17656 – Stationary source emissions – Requirements on proficiency testing schemes for emission measurements. This sets out methodologies for checking the performance of measurement laboratories. The INERIS and NPL test benches both host annual proficiency testing schemes, so having been validated for low HCl concentrations this can be added to these schemes in future to provide a better indication of true performance. The HEROES ILC activities highlighted that real sampling introduced far higher variability in results, than would be expected from the uncertainty analysis.

VTT, NPL and INERIS have also contributed to Task Force Emissions, working on a technical report (an informative (guidance)) to provide guidelines for the elaboration of standardised measurement methods. This will benefit the development of future reference method standards like the revision of EN 1911, by formally setting out recommendations for the structure and content. This will have significant benefits for all future standard development in this field.

Longer-term economic, social and environmental impacts

The work carried out in the HEROES project has demonstrated the level of achievable uncertainty with current measurement methods and highlighted areas where this can be improved. In order to enforce the lower emission limits introduced in BAT conclusions the measurement technologies and methods need to keep up. This project has contributed to this process, helping to support legislation that will protect the environment and human health from the dangerous effects of HCl emissions.

The work testing P-AMS systems will help the future development of new technologies in this field. No current technology (tunable diode laser (TDL), cavity ring down spectrometer (CRDS) or Fourier transform infrared (FTIR), etc.) has demonstrated the ability to perform at the required level to implement EN 16429 for the lowest concentrations. The continuing race to be recognised as the new SRM will drive further investment, then once the breakthrough has been made all ISO/IEC 17025 accredited stack testing organisations providing compliance measurements for HCl will have to replace their existing equipment. To give an idea of value, process plant operators currently spend $1 billion p.a. on their own analysers to monitor stacks, with Europe making up a significant portion of this. This gives an indication of market size if all accredited providers of HCl compliance measurements needed to acquire portable optical instruments.

With respect to stack testing organisations, whilst portable analysers are more expensive than glassware, they are less labour intensive to operate. During the lifetime of an analyser (~10 years) the cost of the analyser compared to the labour costs is relatively insignificant. Furthermore, coal fired powered stations are being phased out by several smaller biomass stations which is a convenient consequence of the ongoing shift in energy production. As a result, the workload for stack testing organisations is steadily increasing.

6 List of publications

Smith, T.O.M.; Robinson, R. A.; Coleman, M. D.; (2021), Monte-Carlo modelling to demonstrate the influence of alternative flow reference techniques on annual mass emission uncertainty. Metrologia. 10.1088/1681-7575/ac3564

This list is also available here: https://www.euramet.org/repository/research-publications-repository-link/

7 Contact details