European Metrology Research Programme

Delivering Impact

Better digestion for mercury analysis

Mercury is highly toxic and once released into the environment bioaccumulates into fish and seafood. Released from burning fossil fuels and broken fluorescent light fittings its emissions are regulated by international treaties and EU Directives. For industrial polluters to demonstrate regulatory compliance mercury emissions are carefully monitored. But the continuing use of an empirical equation for calibration and non-optimised chemical analysis methods hinder a robust measurement hierarchy.

Europe's National Measurement Institutes working together

The European Metrology Research Programme (EMRP) brings together National Measurement Institutes in 23 countries to address key measurement challenges at a European level. It supports collaborative research to ensure that measurement science meets the future needs of industry and wider society.

Challenge

Highly toxic mercury affects the immune system and damages the nervous system. Produced naturally by volcanoes, and forest fires, it is also released during burning fossil fuels and from broken energy saving fluorescent lamps. Once in the atmosphere it bioaccumulates into fish and seafood. For these reasons EU Directives classify mercury as a Priority Hazardous Substance and require member states to progressively reduce emissions to zero over the next 20 years.

Currently all mercury in air measurements are traceable to an empirical equation derived in a 1980s that was subsequently adopted internationally by instrument manufacturers. Reference measurement data from using this equation is reported in atmospheric monitoring, but confirming the accuracy of this equation is difficult. A more rigorous approach based on a measurement hierarchy with robust links to SI units would help improve the quality of atmospheric mercury monitoring.

Plants discharging mercury into the environment use filters and impingers, devices that trap particles in a liquid, for this purpose. Collected particles are digested using a chemical procedure that produces a syrupy solution for transfer through thin tubes into sensitive analytical instruments – a process prone to blockages. This method, underpinned by European Normative documentary standards, is based on operators using highly reactive HF acid which can cause deep painless burns that have the potential to destroy tissue unless treated immediately.

Greater accuracy for determining mercury that is trapped by industrial plants and that released into the atmosphere is needed to enable improved compliance with EU Directives.

Solution

The EMRP project *Traceability for Mercury Measurements* developed a new digestion method based on aqua regia, a strong acid, for preparing mercury-containing samples for analysis. This method produces a solution that is easy to transfer to the analytical instruments used - removing previous problems with clogged analytical instrumentation. Now incorporated into an annex to the relevant mercury measurement EN standard it has made the first steps towards use in regulatory emission monitoring.

To improve the accuracy of measurements of mercury in air the project developed a consistent method based on weighing mercury losses for use in instrument calibration. This method has robust links to SI units and good reproducibility. Increased use in instrument calibrations will decrease reliance on the interpretation of an empirical equation and assist in greater result comparability.

Impact

The mercury measurement community requires accurate reference data and robust instrument calibrations to make meaningful measurements. Currently there is an increasing debate about the validity of reference data arising from different sources. There are now several competing datasets that do not agree, some with historical authority and others with more robust links to SI units. To overcome this dilemma an international committee is required to propose the way forward and methods to confirm data reliability.

The NMI community is proposing that the CCQM could be the arbiter for data quality. The BIPM maintains a materials database of reference data and this could be extended to include NMI derived data on mercury in air with robust links to the SI. An international decision on this is required to make progress in this area. The introduction of a robust measurement hierarchy underpinned by accurate reference data will help industrial plant comply with EU Directives on mercury emissions.

Monitoring mercury in the environment

The EMRP project *Traceability for Mercury Measurements* developed a calibration system for airborne mercury and used it to calibrate environmental monitoring sensors based on accurately determining mercury vaporisation weight loss, to provide a potential replacement for currently used empirical equations. This calibration system was used to characterise an SI traceable transfer instrument that circulated between the project partners enabling the generation of international equivalence for the first time. It also confirmed the performance of project-developed innovative, low-cost, mercury-in-air monitoring sensors demonstrating their suitability for use in monitoring for atmospheric traces of mercury. These have subsequently been trialled by an international monitoring network. Working with multiple biota samples, the project optimised and validated preparation procedures for determining mercury content in fish and used these to establish inter-species differences resulting from variation in fat content or habitat. This is important for analysing samples drawn from rivers and oceans to determine mercury uptake in the food chain.

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