# Laboratory comparison of measurement procedures for workplace monitoring of mercury vapor

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**ABSTRACT** Measurement procedures used for workplace measurements of chemical agents need to fulfill certain quality criteria. According to the European standard EN 482 the expanded uncertainty is the crucial factor in deciding whether a measurement procedure can be used for workplace measurements. For the purpose of this study, one personal sampler and four direct-reading instruments (DRIs) commonly used for workplace measurements of mercury were tested. In a first step, a measurement procedure consisting of a personal air sampler with adsoQUICK as sorbent material and a DMA-80 mercury-measuring device using atomic absorption spectrometry (AAS) was validated according to EN 482. The expanded uncertainty of this analytical method was found to be below 30%, deeming it suitable for workplace measurements of mercury vapor according to EN 482. In a second step a round robin test including four DRIs (three different manufacturers and one replicate) was performed to determine the comparability of these devices to the validated analytical method as well as among themselves. Three of the four DRIs yielded average values between 96 and 106% when compared to the personal air sampler results, whereas the forth yielded 122%. The round robin test demonstrated a good comparability of the DRIs with each other and with the personal air sampler.

## Laborvergleich zwischen Messverfahren zur Bestimmung von Quecksilberdämpfen am Arbeitsplatz

**ZUSAMMENFASSUNG** Messverfahren zur Bestimmung von Gefahrstoffen am Arbeitsplatz müssen bestimmte Qualitätsanforderungen erfüllen, um sie für Arbeitsplatzmessungen einsetzen zu können. Die erweiterte Messunsicherheit ist entsprechend der Norm DIN EN 482 das Hauptkriterium zur Beurteilung der Leistungsfähigkeit von Messverfahren. In dieser Studie wurden ein personengetragenes Sammelverfahren sowie mehrere direkt anzeigende Messgeräte miteinander verglichen. In einem ersten Schritt wurde ein Messverfahren, basierend auf einem Sammelröhrchen mit adsoQuick als Adsorber und anschließender analytischer Bestimmung mittels Atomabsorptionsspektrometrie, entsprechend der Norm DIN EN 482 validiert. Die erweiterte Messunsicherheit für das Sammelverfahren lag unter 30 % und erfüllte dementsprechend die Anforderungen für Arbeitsplatzmessungen von Quecksilberdämpfen nach DIN EN 482. In einem zweiten Schritt wurde ein Ringversuch mit vier direkt anzeigenden Quecksilbermessgeräten (drei unterschiedliche Hersteller und ein Replikat) durchgeführt, um die Vergleichbarkeit der direkt anzeigenden Geräte mit dem validierten Sammelverfahren zu untersuchen. Drei der vier direkt anzeigenden Messgeräte ergaben Werte zwischen 96 und 106 % des für das Sammelverfahren gefundenen Wertes; das vierte Messgerät ergab einen Wert von 122 %. Der Ringversuch zeigte eine gute Vergleichbarkeit der direkt anzeigenden Messgeräte sowohl untereinander als auch mit dem validierten Sammelverfahren.

## 1 Introduction

Mercury (Hg) is a chemical element that can be found in nature either in its pure, metallic form, as mineral ore (i.e. mercuric sulphide, HgS), or contained in bituminous coal. At standard conditions, elemental mercury is a liquid with a relatively high vapor pressure.

In industry, mercury is used in the amalgam process chloralkali electrolysis for production of chlorine, in gold extraction and in the manufacturing of fluorescent lamps and energy saving lamps, computer monitors and light switches. Furthermore, it finds widespread use as amalgam filling material in dentist offices. In the past, mercury was incorporated into thermometers and barometers. Many devices containing mercury are disposed at local waste collecting centers, causing concerns that environmental pollution as well as occupational exposure for the workers employed in this sector may occur.

Mercury as well as most of its compounds has a high toxicity for living organisms [1]. Combined with a long bio-persistence, it poses a hazard for the environment as well as for human health. In order to avoid occupational diseases for exposed workers, the European Commission has set an indicative Occupational Exposure Limit (OEL) value of 0.02 mg/m$^3$ [2]. A number of European countries (Austria, Finland, Germany, Poland, Romania, Sweden) adopted this value as a national OEL [3]. Also in Germany, the Technical Rule for Hazardous Substances (TRGS) 900 [4] states the occupational exposure limit of 0.02 mg/m$^3$ valid for mercury and its inorganic compounds.

Because mercury has a relative high vapor pressure at room temperature, its vapor is readily released into workplace atmosphere. Its saturated vapor concentration at 25 °C of 22 mg/m$^3$ [5] may lead to vapor concentrations in workplace air exceeding the occupational exposure limit (OEL) of 20 µg/m$^3$. Owing to its high toxicity, it can lead to adverse health effects.

Because the saturated vapor concentration can reach levels of three orders of magnitude above the OEL, it is important to identify potential sources and monitor workers´ exposure.

Workplace measurements are an important instrument to assess risks for workers. Personal air samplers positioned in the breathing zone of workers are preferred for measuring concentrations of chemical agents. Personal air samplers were used by *Decharat* et al. [6] and *Darvishi* et al. [7] to sample Hg in the breathing zone of workers. The samplers contained hopcalite sorbent tubes that were connected to a personal sampling pump. After sampling, the hopcalite sorbent was digested in a mixture of nitric acid and hydrochloric acid and later analyzed by cold vapor atomic absorption spectroscopy according to the NIOSH method 6009 [8]. *Hebisch* et al. [9] also used hopcalite as sorbent material, however the analysis consisted of thermal decomposition instead of acid digestion of the sorbent, providing for a more time-effective analysis.

Direct-reading instruments (DRIs) on the other hand can be used to detect possible local emission sources as well as to evaluate any exposure peaks. They can also be deployed in the vicinity of workers to measure their occupational exposure. *Al-Zubaidi* and *Rabee* [10] performed workplace measurements using a Mercury Tracker 3000 IP DRI, while the studies of *Lindberg* et al. [11] and *Southworth* et al. [12] were performed using an Lumex RA 915+ DRI. Both these DRIs measure Hg vapor by atomic absorption spectroscopy, with quantification of the absorption of Hg at a wavelength of 253.7 nm. *Aucott* et al. [13] and *Singhvi* et al. [14] used a Jerome Gold Film Mercury Vapor Analyser for their studies. This type of DRI uses a thin gold foil to form an amalgam with the Hg vapor, which in turn results in a change in the electrical potential across the foil that is proportional to Hg vapor concentration.

A series of European standards were adopted to provide guidance in the field of workplace measurements. EN 482 [15] defines performance requirements that need to be fulfilled by measuring procedures used for monitoring occupational exposure to chemical agents. These performance criteria are especially related to the accuracy and variability of the method in relation to three defined concentrations linked to 10%, 100% and 200% of the OEL. Suitability criteria for workplace measurements procedures are the results of the expanded uncertainty. The expanded uncertainty should be below 50% in the concentration range from 0.1 times to 0.5 times OEL and below 30% in the concentration range from 0.5 times to 2 times OEL, respectively.

This study was aimed to (1) test the suitability of an analytical method involving a personal air sampler to measure mercury in workplace air according to EN 482 and (2) to investigate the comparability of DRIs for mercury measurements. The sampler validation was performed by (1a) spiking technique and by (1b) sampling of Hg vapors. The investigated sampler and DRIs used were designed to monitor the occupational exposure against Hg vapor.

## 2 Methods

### 2.1 Measurement using Personal Sampling Tubes

Quartz sampling tubes (MLS GmbH, Germany, length 30 mm, ID 8 mm, OD 10 mm) were filled with approx. 50 mg of novel adsoQUICK sorbent material (MLS GmbH, Germany). The sorbent consists of spherical shaped graphite material. The porous surface of the adsoQUICK material allows mercury vapor to adsorb on its surface. Porous ceramic fiber septa (Fa. MLS, Germany) were placed on the inlet and outlet of the sampling tubes in order to keep the sorbent material in place. The sorbent tubes were conditioned at 650 °C for a period of 6 minutes, in order to eliminate any contaminants. Two sorbent tubes were used in series for the purpose of this study, the second tube served as a backup to quantify a possible breakthrough from the first tube.

The samplers were operated at 125 ml/min using GilAir plus personal sampling pumps (Fa. Gillian, USA). The airflow was set and verified with a DryCal Defender 530L (Fa. Mesa Labs, USA). After the sampling of mercury vapor, the samplers were stored in 5 ml capped glass vials at room temperature.

To extract adsorbed mercury thermally from the sorbent, the sampling tubes were heated for 6 minutes and analyzed by an atomic absorption spectrometer (DMA-80 analyzer, Fa. MLS). In short, mercury is thermally desorbed from the collection media without further sample preparation, concentrated on a gold amalgamator and then released into the spectrophotometer optical path of the analyzer. The preparation of the standard solutions used for the instrument calibration and the temperature program for thermal desorption were done analogue to the method described by *Hebisch* et al. [16].

### 2.2 Spiking of Samplers (1a)

The samplers were spiked with mercury standard solutions using microliter syringes. The standard solutions contained 30 ng, 300 ng and 600 ng of mercury as target values. These values are equivalent to sampling mercury with a flow rate of 125 ml/min for two hours at the concentrations of 2 µg/m³, 20 µg/m³ and 40 µg/m³ required in EN 482. After spiking, clean air was passed through the sampler with a GilAir plus pump for a period of two hours at 125 ml/min.

### 2.3 Vapor Generation System (1b)

A dynamic mercury vapor generation system was set up in a fume hood inside an air-conditioned laboratory. The temperature and relative humidity within the laboratory were kept constant at 21 °C and 50%, respectively. Particle-free, purified air originating from a compressor was used as dilution air in a major flow and as carrier gas for mercury vapor in a minor flow. No further enrichment of the air with water vapor has been performed for this study. The flow rates of both dilution and mercury enriched airstreams were controlled by needle valves, the total flow rate was varied between 5 l/min and 6 l/min. The minor flow was directed through a small vessel containing elemental mercury (Fa. Merck, Germany) in order to be enriched with its vapor. The variation of the flow rate of the air that bubbles through the vessel adjusts the mercury vapor concentration. The mercury-enriched airstream was mixed with the main flow in a T-shaped connector in order to achieve the required mercury vapor concentration. Finally, the airstream containing mercury vapor was directed through a sampling route offering simultaneous sampling from seven serially mounted ports. Sorption tubes and/or DRIs can be connected to these ports. Mercury concentrations of 2, 20 and 40 µg/m³ were generated and monitored using a direct-reading Fa. Mercury Tracker 3000IP (Fa. Mercury Instruments, USA).

The expanded uncertainty according to EN 482 [15] was calculated for the personal air sampler using a web tool developed by the Institute for Occupational Safety and Health of the Ger-

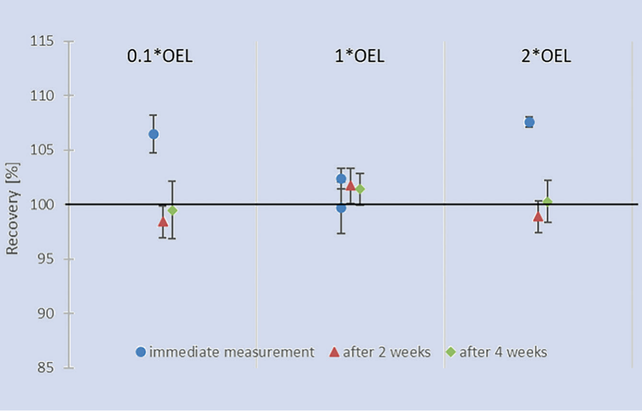

**Figure 1** Mercury recovery rate for personal samplers spiked with standard solutions of Hg in HNO3 (1a validation). The 100% recovery line is based on the spiked Hg amount (30 ng, 300 ng and 600 ng for 0.1, 1 and 2*OEL based on 15 liters sample volume or 2 hours of sampling). The error bars represent one standard deviation. *Graphic: Authors*

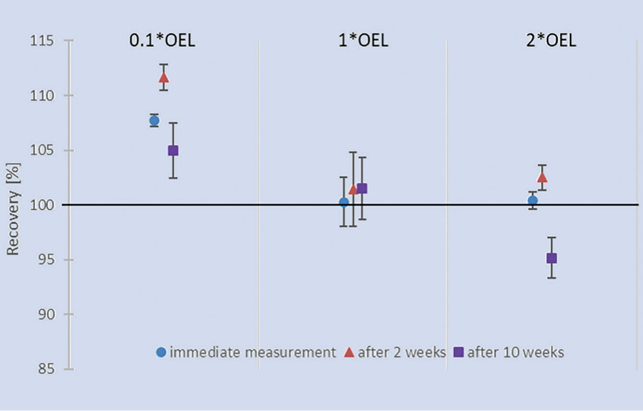

**Figure 2** Mercury recovery rate for personal samplers at the Hg vapor generating system (1b validation). The 100% recovery line is based on the results of the Mercury Tracker 3000IP no. 1. The samples were collected over 2 hours, with 15 liters sampled volume. The error bars represent one standard deviation. *Graphic: Authors*

man Social Accident Insurance [17]. The expanded uncertainty is a measure of the uncertainty of random and non-random components for concentrations of 0.1, 1 and 2 times OEL, in brief a combination of accuracy, precision and confidence. More details can be found in the publications of *Ashley* and *Bartley* [18], *Breuer* et al. [19], *Breuer* [20], *Hebisch* et al. [21], *Ashley* [22] and on the calculation tool webpage.

### 2.4 Direct-Reading Instruments / Round Robin Tests (2)

Comparison measurements took place in a round robin test for mercury vapor on four DRIs. The instruments were: two Mercury Tracker 3000IP (Fa. Mercury Instruments, USA) operated at 1.7 l/min, a Mercury Analyzer RA-915M (Fa. Lumex Instruments, Canada) operated with an external Gillian pump at 0.8 l/min and a X-Stream XEGK (Fa. Emerson, USA) operated at 0.8 l/min. The inlets of the devices were connected to the vapor generating system using flexible Tygon tubing (Fa. Carl Roth, Germany). All devices use atomic absorption spectrometry for measuring mercury. The Tracker 3000IP and the RA-915M can also be operated on batteries. The instruments were stored in the lab to acclimatize for 16 hours and turned on at least 2 hours before the beginning of the round robin tests.

Three test gas atmospheres containing mercury vapor with target concentrations of 2 µg/m³, 20 µg/m³ and 40 µg/m³ were generated for the round robin tests. Four sets of one-hour measurements have been carried out for each concentration with two DRIs in parallel. Simultaneously, three sampling tubes were used during each run to account for the reference concentration. The flow rate of the sampling tubes was set at 125 ml/min using GilAir plus pumps. The reported concentration for each DRI was averaged over the sampling duration of one hour and used to compare the results of each DRI with the personal air sampling tubes.

## 3 Results

### 3.1 Personal Air Sampler Validation with Spiking Tests (1a)

Both recovery and storage test were performed with the spiking method (1a) as well as with sampling at the vapor generating system (1b). For storage tests, the recovery from samplers stored at room temperature after certain time periods was measured. **Figure 1** shows the results of the recovery and storage tests conducted for the spiked personal air samplers. Each data point represents the average value of six measurements. The error bars represent one standard deviation. Two sets of measurements were taken on different days for the immediate measurement of the OEL concentration to test for possible day-to-day variability of the system. The recovery rates of mercury from the spiked samples ranged from 98% to 108%. The average recovery (± one SD) across all 1a measurements was 101.6 (±3.1) %. The average recovery (0.1, 1 and 2 times OEL) for samples measured immediately after spiking (t = 0) was 104 (±3.7) %. Average recoveries of 99.7 (±1.8) % and 100.4 (±1) % were determined for samples after a storage period of two and four weeks, respectively. The average recovery rates for the spiking solutions representing 0.1, 1 and 2 times OEL were 101.5%, 101.8% and 102.3%, respectively. Using the calibration curve method according to the German standard DIN 32645 [23; 24] the limit of quantification (LOQ) was calculated with spiked samplers and found to be 0.44 ng mercury or 29.3 ng/m³ for a sample volume of 15 l.

### 3.2 Personal Air Sampler Validation using the Vapor Generation System (1b)

Recovery and storage tests were performed after sampling for two hours from the mercury vapor generation system. The recovery rate was calculated in relation to the measurement result obtained with the Mercury Tracker 3000IP no. 1. **Figure 2** shows recovery rates from 95% to 112% for mercury vapor sampling. The average recovery (± one SD) across all 1b measurements was 102.8 (±4.8) %.The average recovery (0.1, 1 and 2 times OEL) for the samples measured immediately after sampling (t = 0) was 102.8 (±4.8) %. An average recovery of 105.2 (±5.6) % was obtained for samples stored for two weeks and of 100.5 (±5) % for samples stored for ten weeks. The average recoveries for the vapor generation system representing 0.1, 1 and 2 times OEL were 108.1 (±3.3) %, 101.1 (±0.7) % and 99.3 (±3.8) %, respectively. Because nearly 100% recovery was found for 2 hours sampling at 40 µg/m³, the capacity of the sampler for the tested flow rate and temperature is at least 600 ng Hg. The values for the expanded uncertainty calculated according to EN 482 were 17.3%, 15.9% and 15.5% for the

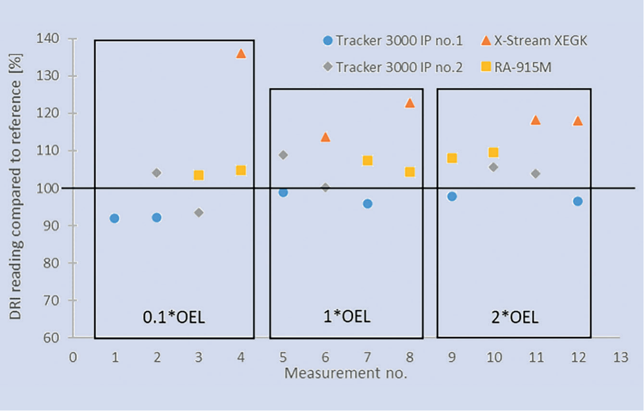

**Figure 3** Round robin results of the participating direct-reading instruments compared to the reference personal sampler measurement as 100 % line. Results are based on one hour sampling time for target concentrations of 2, 20 and 40 µg/m$^3$. *Graphic: Authors*

corresponding concentrations of 0.1, 1 and 2 times OEL, respectively.

The breakthrough time, which indicates the maximum duration of sampling at the given environmental conditions without losing more than 5% of the analyte due to breakthrough was also determined. Sampling occurred at a target concentration of 10 µg/m$^3$ (0.5*OEL) with 0.125 l/min. This concentration was chosen in order to distinguish between breakthrough caused by air passing through the sampler and the capacity of the sorbent material being reached. At this concentration, the sorbent capacity of 600 ng Hg would have been reached after 8 hours. Thus, a decrease in recovery before 8 hours can be attributed to breakthrough time/volume and not to sorbent capacity. The recovery rates of the samplers were compared to the readings of a DRI (3,000 IP no.1) after sampling for three, four and eight hours. The three-hour samples revealed a recovery rate of the first sampling tube of 98.8%, with 2.3% found in the breakthrough tube. This shows that the breakthrough time exceeds three hours, equivalent to a breakthrough volume of at least 22.5 liters. Increasing the sampling time to four and eight hours led to a decrease of the analytical recovery to 82.3% (additional 21.3% in the breakthrough tube) and 57.7% (additional 35.2% in the breakthrough tube), respectively. Therefore, sampling duration should not be longer than three hours in order to avoid sampling losses due to breakthrough.

### 3.3 Round Robin Tests using the Vapor Generation System (2)

**Figure 3** depicts the results of the DRIs during the round robin tests, using the results from the personal air sampler as the 100% reference. The first measurement with the X-Stream XEGK was considered to be an outlier (Nalimov and Grubbs outlier tests, with 99% certainty) and consequently removed from further evaluations. The tests performed yielded mercury results ranging from 92% to 99% for the first Mercury Tracker 3000IP, 93 to 109% for the second Tracker 3000IP, 103% to 109.5% for the RA-915M and 118 to 136% for the X-Stream XEGK, when compared to the personal air sampler as reference. The results obtained with the RA-915M and the X-Stream XEGK were constantly above the 100% line when compared to the reference sampler, with average results of 106 (±2.4) and 122 (±8.6) % respectively. The first Mercury Tracker 3000IP had an average reading of 96 (±2.9) %, though slightly lower at a mercury concentration of 2 µg/m$^3$ (92%). The second Mercury Tracker 3000IP showed an average recovery of 103 (±5.4) %.

## 4 Discussion

### 4.1 Personal Air Sampler Validation with Spiking Tests (1a)

The spiking tests using solutions of known mercury quantities were a first step towards the validation of the sorbent tube method. These tests showed that the total amount of spiked mercury can be extracted using thermal desorption followed by a precise quantification by AAS analysis. From the ten tests performed with six samples each (Figure 1), only two measurements had an overestimation of more than 5%. Probably, this may be attributed to a slight error during spiking of the samples. Nonetheless, the obtained results were not affected neither by the spiked mercury concentration, nor by the storage duration.

### 4.2 Personal Air Sampler Validation using the Vapor Generation System (1b)

A further possibility to validate methods according to EN 482 is by actively drawing mercury-enriched air through the sampler with a pump. Usually, this method is preferred to the previously mentioned spiking tests because this procedure better simulates sampling at real workplaces. Similar to the spiking tests where an average recovery of 101.6% (±3.1%) was measured, an average recovery of 102.8% (±4.8%) was found for sampling and analysis of mercury vapor. This shows that the recovery rates were not substantially affected by the dosing method used, both spiking and vapor sampling yielded similar results. The difference between the two methods was not statistically significant (two sided t-test). However, some differences (average of 108% vs. 101.5%) were measured at the lowest mercury concentration, with vapor sampling yielding a higher nominal recovery rate when compared to spiking. This difference is explainable by the different methods used to measure the reference concentration. For the spiking method, mercury standard solutions served for the calculation of the reference value, whereas for vapor sampling the Mercury Tracker 3000IP no. 1 was used as reference. The different recovery rates found only at 2 µg/m$^3$ were caused by a lower sensitivity of the DRI for this low concentration, which was confirmed by the round robin tests, too.

Because the storage stability of the 1a samples was nearly 100% even after four weeks, an extended storage time of ten weeks for the samples with mercury vapor was investigated. The samples were stable even after ten weeks of storage, with recovery rates of at least 95%.

The sampler breakthrough test has shown that no sample loss occurs if the sampling period does not exceed three hours at the recommended flow rate of 125 ml/min. The samplers proved to be suitable for measuring mercury vapors, considering the good analytical recovery rates, long storage stability and sampling time up to three hours. Hence, the samplers served as a reference for the round robin tests.

The results of the expanded uncertainty were below 30% at all times. This demonstrates the capability of the personal air sampling method to meet the performance requirements of EN 482.

### 4.3 Round Robin Tests using the Vapor Generation System (2)

The round robin tests showed that three of the four DRIs tested deviated less than 10% from the reference method. The investigated mercury concentrations did not have a major influence on the results of the DRIs, the coefficient of determination $R^2$ being larger than 0.998 in all cases, as shown by the linear regression analysis (**Figure 4**). The first Tracker 3000IP was the least biased compared to the reference values, with a slope of 0.97. However, at the lowest concentration (0.1*OEL) the difference amounted up to 8%. This is a confirmation of the results obtained during the sampler validation, where the results from this DRI were lower than the sampler measurements at 2 µg/m³. The results of the second Tracker 3000IP were in good agreement with the reference values, with a slope of 1.05. The X-Stream XEGK always overestimated the concentration, yielding a slope of 1.18. A mismatched calibration may be the cause, as this overestimation was not affected by the mercury concentration, as shown by the linear regression analysis (Figure 4). The RA-915M overestimated the reference values in all cases, with a slope 1.08, which may also been caused by the devices' calibration. A spread of 20% in average sensitivities were measured in comparison to the reference method, the results of both Tracker 3000IP being closest to the reference measurements.

## 5 Conclusions

The results presented in this study show that workplace measurements of mercury may be carried out using both personal air sampling methods as well as with DRIs over a concentration range from 2 to 40 µg/m³. The performance of the measurement procedure using sampling and subsequent analytical determination agrees with the requirements described in the European standard EN 482. In addition, the DRIs showed comparable results to the sampler method.

The presented measurement procedures were tested in a laboratory setup, under controlled temperature and humidity conditions. Field studies under varying environmental conditions may lead to different results. DRIs can provide a real-time screening of potentially contaminated areas and identify areas of high concern. The personal air samplers are in turn better suited to monitor the personal exposure of individual workers. ■

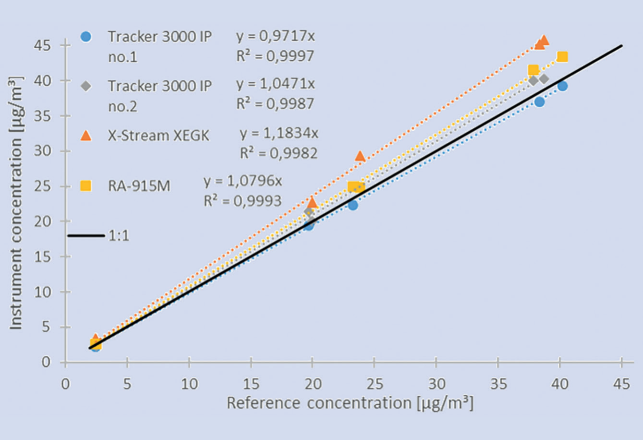

**Figure 4** Results of the four participating direct-reading instruments and their linear regression compared to the reference personal sampler measurement. Results are based on one hour sampling time for target concentrations of 2, 20 and 40 µg/m³. *Graphic: Authors*

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