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# Gravimetric Analysis of Particulate Matter using Air Samplers Housing Internal Filtration Capsules

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## Summary

An evaluation was carried out to investigate the suitability of polyvinyl chloride (PVC) internal capsules, housed within air sampling devices, for gravimetric analysis of airborne particles collected in workplaces. Experiments were carried out using blank PVC capsules and PVC capsules spiked with 0,1 – 4 mg of National Institute of Standards and Technology Standard Reference Material® (NIST SRM) 1648 (Urban Particulate Matter) and Arizona Road Dust (Air Cleaner Test Dust). The capsules were housed within plastic closed-face cassette samplers (CFCs). A method detection limit (MDL) of 0,075 mg per sample was estimated. Precision $S_r$ at 0,5 - 4 mg per sample was 0,031 and the estimated bias was 0,058. Weight stability over 28 days was verified for both blanks and spiked capsules. Independent laboratory testing on blanks and field samples verified long-term weight stability as well as sampling and analysis precision and bias estimates. An overall precision estimate $\hat{S}_{rt}$ of 0,059 was obtained. An accuracy measure of ±15,5% was found for the gravimetric method using PVC internal capsules.

## Introduction

Gravimetric methods of sampling and analysis are commonly used to measure quantities of airborne particulate matter collected from workplace atmospheres. Such measurements are often carried out to estimate occupational exposures to airborne particles and/or to evaluate the efficacy of air pollution control technologies. Gravimetric analysis of airborne particles is often complemented by other analytical methods which are employed to measure concentrations of specific chemical agents in occupational atmospheres.

Examples of government-promulgated methods for gravimetric measurement of airborne particles in workplaces include those developed by the US National Institute for Occupational Safety and Health (NIOSH) [1] and the UK Health and Safety Executive (HSE) [2]. These methodologies, which are conceptually uncomplicated, entail pump-based collection of airborne particles onto pre-weighed, weight-stable filters that are housed within

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plastic air samplers. Subsequent weighing of the total collected particulate matter plus filter yields the weight of the sampled aerosol by difference. Accurate gravimetric analysis of air particles by such means requires minimization of electrostatic effects, control of moisture sorption and/or other volatiles, temperature stability and exercising extreme care in sample handling [3].

Airborne particles that are collected from workplace atmospheres using filtration-based samplers can deposit elsewhere within the sampler than on the filter [4]. Studies have shown that material deposited on the inside surfaces of air sampling devices often constitutes a significant fraction of the aerosol that enters the sampler [5, 6, 7]. NIOSH considers that all particles entering air samplers should be included as part of the sample whether they deposit on the filter or on the inside surfaces of the sampler [8]. As a result of these concerns, we have undertaken steps to update aerosol sampling methods in order to include particulate material not collected onto filters, which would otherwise be lost to subsequent analysis.

Earlier issues of NIOSH gravimetric methods for “nuisance” dust (“total” collected airborne particles) and carbon black (NIOSH Methods 0500 and 5000, respectively) specify the use of polyvinyl chloride (PVC) filters housed in closed-face filter cassettes (CFCs) for collection of airborne particles for subsequent gravimetric analysis [9]. As described in the previous versions of these NIOSH methods, the PVC filter is weighed before and after sample collection, with the reported weight of sampled particulate matter being the difference between pre- and post-weighing. However, airborne particles collected using CFC samplers can deposit elsewhere than on the PVC filter, and thus would not be included in gravimetric analysis of the filter alone [7, 8].

An alternative technique for ensuring that internal non-filter deposits are included in the analysis is to collect airborne particles within an internal capsule, housed within the sampler, which is weighed in its entirety before and after sample collection. PVC capsules for gravimetric analysis of airborne particulate matter have been commercially available for several years. The filter capsules are designed to be inserted into 37-mm plastic CFC samplers on top of cellulose support pads, as illustrated in Figure 1. The evaluation described in this report was undertaken in order to provide necessary performance data for validation of newly updated NIOSH gravimetric methods, which will be based on the use of PVC internal capsules.

## Experimental

Polyvinyl chloride (PVC) Accu-caps® with 5-μm pore size PVC filters (Lot #11136-7DBPASK-104, SKC, Inc., Eighty-Four, PA, USA) were allowed to equilibrate to room temperature and humidity in the laboratory under a dust cover box for at least two hours (most were equilibrated for 24 hours). A $^{210}$Po static control device (NRD, Grand Island, NY, USA) was used to eliminate electrostatic effects during weighing.

After equilibration, the Accu-caps were weighed on a Mettler Toledo high-precision analytical balance (Model XP205DR, Greifensee, Switzerland) before spiking with certified reference material (CRM) dust; balance calibration was checked before each use with ASTM Class 1 weights. The pre-spiked weight of each PVC Accu-cap was recorded to the nearest 0,01 mg. The Accu-caps were then placed into two-piece closed-face plastic air sampling cassettes (Lot #2039-7D6PASK-010, SKC, Inc.) and were sealed within the cassettes. For spiking with CRM, each cassette was connected to a personal sampling pump (Aircheck Sampler, Model #224-PCXR7, SKC, Inc.) set to a calibrated flow rate of 2,5 (±0,1) $l \cdot min^{-1}$.

The certified reference materials (CRMs) used were US National Institute of Standards and Technology (NIST) Standard Reference Material® (SRM) 1648, Urban Particulate Matter (NIST, Gaithersburg, MD, USA) and Arizona Road Dust – Air Cleaner Test Dust (General Motors AC Spark Plug Division, Flint, MI, USA). These materials were chosen in consideration of their median particle sizes ≈10 μm, similar to that of inhalable particles.

On glassine weighing paper, different levels of CRM (0,1 – 4 mg), with at least three samples at each spiking level, were weighed to the nearest 0,01 mg. The test materials were loaded into separate Accu-caps via 23-mm plastic micro funnels (Poly Micro Funnel, The Science Company, Denver, CO, USA), which were inserted snugly into the inlets of the cassettes housing the Accu-caps. For each sampler, once the weighed test material was poured into the funnel, the sampling pump was activated so that the particulate material was drawn completely out of the funnel and into the PVC Accu-cap housed within the cassette. The pump was used to draw air through the system for approximately one minute, which enabled maximum transfer of CRM from the funnel into the Accu-cap.

After the cassettes were disengaged from the pump, the PVC Accu-caps were removed from the cassettes using a cassette opener and were then weighed individually. The weights of the spiked Accu-caps for all replicate levels were obtained using the same balance and were recorded as weight for Day 0. Collection efficiency was calculated by dividing the collected weight of the test material by the total material weight before transfer to the Accu-cap. For each of the sets of CRM samples (i.e., NIST SRM 1648 and Arizona Road Dust – Air Cleaner Test Dust), six media blanks were weighed and sealed, recording their Day 0 weights as well.

Once the initial (Day 0) weights were recorded for all replicate samples and media blanks, the PVC Accu-caps (sealed in their labeled testing cassettes) were stored in cardboard boxes (organized by sample set) for a 4-week storage study. On seven-day intervals, the Accu-caps were weighed and the results recorded for comparison to their Day 0 weights. Percent change in total weight was calculated by subtracting the Day 0 weight from the Day 7 (and then Day 14, 21, and 28) weights, dividing by the Day 0 weight and multiplying by 100%.

As an external check of the internal capsule weighing procedure, gravimetric analyses of PVC Accu-caps, both blank media and spiked capsules, were performed by an independent laboratory (Bureau Veritas North America (BVNA), Novi, MI, USA). These data were also used to estimate the overall accuracy of the method through examination of differences between results from NIOSH and BVNA.

Pressure drop experiments [10] were carried out in order to further characterize the PVC Accu-caps. Five different Accu-caps were placed in 37-mm plastic cassettes and the absence of leakage was verified before measurement [11]. The pressure drop from 5-µm pore size PVC filters (SKC, Inc.) was measured for comparison.

For field comparison studies, Accu-caps within plastic CFCs, as well as Institute of Occupational Medicine (IOM, Edinburgh, United Kingdom) plastic samplers with PVC filters, were obtained from SKC, Ltd. (Dorset, United Kingdom).

## Results

### Storage stability

Storage stability data for PVC Accu-caps are presented in Tables 1 and 2 for NIST SRM 1648 and Arizona Road Dust sample sets, respectively. In each table, sample weight (i.e., weight of added dust corrected for the tare weight of internal capsule plus the change in blank capsule weight) is indicated by descending order (in sets of rows) for each spiking level (≈4 mg; ≈2 mg; ≈1 mg; etc.). Media blank data for each sample set are also included and are indicated in Tables 1 and 2. The magnitudes of percent changes in CRM weights of ≈1 mg and above were generally less than a few percent for both NIST SRM 1648 (Table 1) and Arizona Road Dust (Table 2) for time periods of up to four weeks. It can be seen from these tables that the most significant weight changes occur during the first week of storage.

For NIST SRM 1648, Urban Particulate Matter (Table 1), weight changes as a percent of the loaded dust weight are less than -5% for weights of ≈1 mg and above, but become appreciable (in excess of -25% and less) at weights of ≈0,5 mg and below. On the other hand Arizona Road Dust, Air Cleaner Test Dust (Table 2) is weight stable for yet lower spiking levels, as evidenced by percent weight changes of <±9% for weights of ≈0,5 mg and greater.

### Limits of detection and quantitation

The estimated method detection limit (MDL) and limit of quantitation (LOQ) of the gravimetric procedure was obtained from PVC Accu-cap blank data based on Day 0 to 7 weight changes (n=12). The overall mean weight change was -0,000019 g and the standard deviation was 0,000025. Thus the estimated MDL is obtained by three times this value or 0,000075 g = 0,075 mg (75 µg). The estimated LOQ is ten times the standard deviation of blank results, i.e., 0,25 mg (250 µg).

### Spiking recovery

Measured recoveries of spiked Accu-caps are shown in Table 3 for capsules spiked with ≈1 mg, ≈2 mg and ≈4 mg of NIST SRM 1648 and Arizona Road Dust. Recoveries were computed as the ratio of the amount of material weighed after spiking to the amount of material weighed prior to spiking. (These gravimetric measurements attempted to account for trace CRM losses onto weighing paper during spiking; recovery calculations of course subtracted the weights of Accu-caps obtained before spiking was carried out.) Measured recoveries for these spiked PVC Accu-caps ranged from ≈92% to ≈102%.

## Pressure drop tests

At sampling pump flow rates of 2 and 4 $l \cdot min^{-1}$, the average pressure drop values for PVC Accu-caps were 0,924 (±0,055) kPa and 1,86 (±0,18) kPa, respectively. For PVC filters, these values were 1,06 kPa and 2,08 kPa for flow rates of 2 and 4 $l \cdot min^{-1}$, respectively. (Similar pressure drop results for PVC filters have been reported elsewhere [10].) Thus the measured pressure drops for PVC Accu-caps and PVC filters were comparable. No collapse or other physical failures of the Accu-caps were observed when using sampling pump flow rates of up to 5 $l \cdot min^{-1}$.

## Independent laboratory tests

It was of interest to conduct an independent test of the gravimetric procedure by a different laboratory, but performed on the same samples analyzed by NIOSH, in consideration of established validation protocols. Accordingly, a comparison study was carried out whereby PVC Accu-cap NIOSH-analyzed samples (i.e., CRM dust spikes, as well as capsule media blanks) were conveyed to BVNA for gravimetric analysis. The results for Arizona Road Dust from BVNA are listed vs. the final weights obtained at the NIOSH laboratory in Table 4. (Data from NIST SRM 1648 are not shown owing to weight instabilities observed for this material.) For dust loadings of ≈5 mg and above, results from the two laboratories are within ±10% of one another. For blank sampling media, independently reported results are within 0,1 % of the weights obtained by the NIOSH laboratory.

In testing the internal capsule weighing procedure in an independent laboratory (BVNA), PVC Accu-cap gravimetric measurements were carried out; analysts at BVNA performed MDL and LOQ measurements as well as separate long-term stability studies. From seven Accu-cap blanks, the reported standard deviation was 0,0000189 g, which yielded estimated values for the MDL and LOQ of 0,057 mg (≈60 μg) and 0,189 mg (≈190 μg), respectively. For seven Accu-cap blanks that were stored for six months, the average deviation of the individual blank measurement from the average blank was 0,02 mg, with a maximum deviation of 0,05 mg. The average percent deviation over this time period was less than -0,01 %. These figures of merit are comparable to those obtained separately in the NIOSH laboratory.

## Field data

To test the gravimetric method using PVC internal capsules in workplace environments, gravimetric results from PVC Accu-caps housed in closed-face cassettes (CFCs) and Institute of Occupational Medicine (IOM) samplers were compared in air samples obtained at metal foundries in France. The purpose of the field experiments was to evaluate the use of internal capsules housed within CFCs against an established ‘reference’ sampler for inhalable aerosols, the IOM sampler [12]. Side-by-side static (area) samples for gravimetric analysis were obtained using the Institut de recherché Robert-Sauvé en santé et en sécurité du travail (IRSST) Method 48-1 [13], which specifies PVC capsule inserts, and HSE Method 14/3 [2], which relies on IOM samplers housing PVC filters. The IRSST method calls for a flow rate of 1,5 $l \cdot min^{-1}$ and a minimum sample volume of 180 L, while the HSE method specifies a flow rate of 3,5 $l \cdot min^{-1}$ and a minimum sampling time of 4 h. Results from these experiments are presented in Table 5, and demonstrate the comparability of the PVC capsule-based gravimetric method and the IOM sampling method.

# Discussion

## Sample stability and analytical range

The data presented in Tables 1 and 2 illustrate the long-term weight stability of blank and spiked PVC Accu-caps for up to at least 28 days. For loadings of ≈1 mg and greater, measured weights are all within 100% ± 5% for NIST SRM 1648 and Arizona Road Dust (Air Cleaner Test Dust). Arizona Road Dust is observed to be more weight stable than NIST SRM 1648 (Tables 1-2), and this is attributed to trace moisture loss / off-gassing from the latter material, especially during the first week of storage. Despite this, the excellent weight stability of the PVC internal capsule material is clearly seen. The weight stability demonstrated by the PVC capsules is greatly superior to that of plastic cassettes, which have been shown to adsorb or desorb significant amounts of moisture (as much as 1-2 mg over a several-day period), depending on humidity conditions [14].

At loading (spiking) levels of ≈1 mg and greater, recoveries of NIST SRM 1648 and Arizona Road Dust (Air Cleaner Test Dust) from PVC Accu-caps (spiked as described above) were >90% (Table 3). However, for spiking levels of ≈0,5 mg and ≈0,1 mg, SRM 1648 measured recoveries were inconsistent, varying from <40% to >150% (data not shown); and for Arizona Road Dust spiked at ≈0,1 mg, measured recoveries were ≈60% to ≈180% (data not shown). To investigate whether there was any appreciable background dust being collected during spiking, air was drawn through a separate set of blank PVC Accu-caps; the capsules were weighed before and after air was drawn through them. These experiments did not show any statistically significant differences in weights between the un-spiked capsules, indicating that there was no measureable dust collected by sampling background laboratory air.

It should be noted that the MDL for all sample loadings was less than the expected weight for the gravimetric method using the Accu-cap samplers. However, spiking levels of ≈0,5 mg are close to the estimated LOQ of the method, and spiking levels of ≈0,1 mg are below the LOQ estimate of 0,25 mg, thereby illustrating the limits of gravimetric analysis using this technique. Causes of greater imprecision in CRM weights at dust spiking levels of ≈0,5 mg and below are ascribed in part to inherent difficulties in accurately weighing CRM weights less than 1 mg.

## Independent laboratory tests

It is pointed out that the differences in Day 0 to Day 7 gravimetric results provide conservative estimates of MDLs and LOQs. In practice, actual MDLs and LOQs estimated from same-day weighing of PVC internal capsules may be better, as was found by BVNA in their independent tests of the method.

Independent weight measurements obtained by BVNA were very close to the results obtained by NIOSH (Table 4), thereby demonstrating the ruggedness of the gravimetric method when using PVC capsules.

### Field data

Owing largely to sampling variability and a limited number of samples, the results shown in Table 5 yielded no statistically significant differences between the mean gravimetric results obtained from PVC AccuCaps vs. IOM samplers. These gravimetric data are consistent with reported metals analysis results from CFC vs. IOM comparisons when wall deposits are taken into account in the former sampler [5-7]. This work further supports the notion that there is no practical difference in the performance of the two samplers (i.e., CFCs with capsule inserts or wall deposits included vs. IOMs) when used in the occupational settings investigated.

### Special precautions

Because of the delicate nature of the PVC Accu-caps, there should be as little pressure on the plastic tops of the capsules as possible, being that indentations in the Accu-cap domes could occur. Deformation of the Accu-caps might lead to misalignments of the collection holes of the Accu-caps with the cassette inlet ports, which in turn could result in incomplete aerosol collection and poor collection rates. Accu-caps having misaligned inlet holes were not used in this evaluation. During sample preparation, when removing the PVC Accu-caps from the cassettes in which they were housed, the capsules often adhered to the top sections of the cassettes. In practice, it is suggested that the filters be removed by using a scalpel blade, but for the PVC Accu-caps this technique was not always effective. At times the Accu-caps were difficult to remove from the cassettes and required additional applied pressure to remove them from the samplers. It was often necessary to use a forceps and, with gloved fingers, physically grab the outer plastic edge of the Accu-cap and pull as hard as practicable. Because of the effort that was required to remove the Accu-caps from the cassettes, misplacement or slippage of the forceps can occur, often resulting in tearing of the Accu-cap filters. Accu-caps that were damaged during removal from cassettes were discarded.

It was observed that some Accu-caps were prone to sticking inside the cassettes, and this was ascribed to their non-uniformity in manufacture. From batch to batch, or even capsule to capsule, the appearance and construction of the different Accu-cap inserts varied significantly. For example, the fringe plastic on one Accu-cap can be noticeably wider than that of a different Accu-cap. The top shell of one Accu-cap can also be considerably different from that of another capsule (in terms of thickness of the capsule dome and/or position of the inlet hole). About one in twenty capsules was found to have fabrication deficiencies. It is apparent that these problems are due to the manufacturing technique used to make the capsules, thus the manner in which these imperfections are avoided during fabrication should be addressed and corrective action taken.

Additionally, it is strongly recommended to use long cassette inlet plugs that will ensure complete sealing of the PVC internal capsules after sample collection. This will help to prevent collected particulate matter from being lost through the inlet hole of each capsule during transport and handling. The use of short inlet plugs that have historically been used with closed-face cassette sampling should be avoided.

### Method accuracy

Two sets of CRMs were used for this investigation. It was observed that NIST SRM 1648 was not ideally weight stable, and this was attributed to moisture uptake or off-gassing by this material depending on humidity conditions. Hence results from Arizona Road Dust, which was found to be much more weight stable than NIST SRM 1648, were used to estimate the bias, precision and accuracy of the gravimetric method. The bias of the method was estimated by examining the difference between the NIOSH blank-corrected dust weights and the BVNA blank-corrected weights obtained independently.

Data for Arizona Road Dust at weight levels of 0,1 mg and below were below the estimated LOQ of the method and thus were excluded from the subsequent accuracy computations. Homogeneity of bias was tested by means of analysis of variance (ANOVA) [15] of the NIOSH vs. BVNA results and indicated that bias was not homogeneous. As a conservative approach, the maximum mean bias value was used for evaluation of method accuracy. Homogeneity of precision was investigated using Bartlett's test [15], which suggested that the precision was poolable across the tested levels, thus pooled RSD was used for the accuracy evaluation.

To summarize, we examine the performance of the gravimetric method using PVC internal capsules, in consideration of the applicable criterion for method accuracy established by NIOSH [15]. Because of superior weight stability (as mentioned previously), reported results for Arizona Road Dust (Table 2) at spiking levels above the estimated LOQ of the method were used to estimate overall method accuracy of gravimetric analysis using PVC capsules. For Arizona Road Dust samples of loadings of 0,5 - 4 mg, the precision estimate was 0,031. From the differences in reported gravimetric results by NIOSH and BVNA for Arizona Road Dust (for weight loadings ≈0,5 mg and above), a maximum mean bias of 0,058 was obtained. A pooled estimate of method precision (as total relative standard deviation $\hat{S}_{rt}$) of 0,059 was computed from the NIOSH and BVNA results for dust loadings of ≈0,5 mg and greater. Using the these figures of merit relating accuracy to precision and bias [15, 16], an accuracy estimate of ±15,5% was obtained (we note that this accuracy figure is equivalent to expanded uncertainty for coverage factor $k \approx 2.8$ [17]). An upper limit of accuracy at the 95% confidence limit is 21,7%. Given that the NIOSH criterion for method accuracy is ±25%, the above analytical figures of merit demonstrate that the performance criteria for accuracy for acceptance as a NIOSH method are satisfied for gravimetric measurement using PVC capsule inserts. Consequently new NIOSH gravimetric methods are being drafted to specify the use of PVC internal capsules in lieu of PVC filters.

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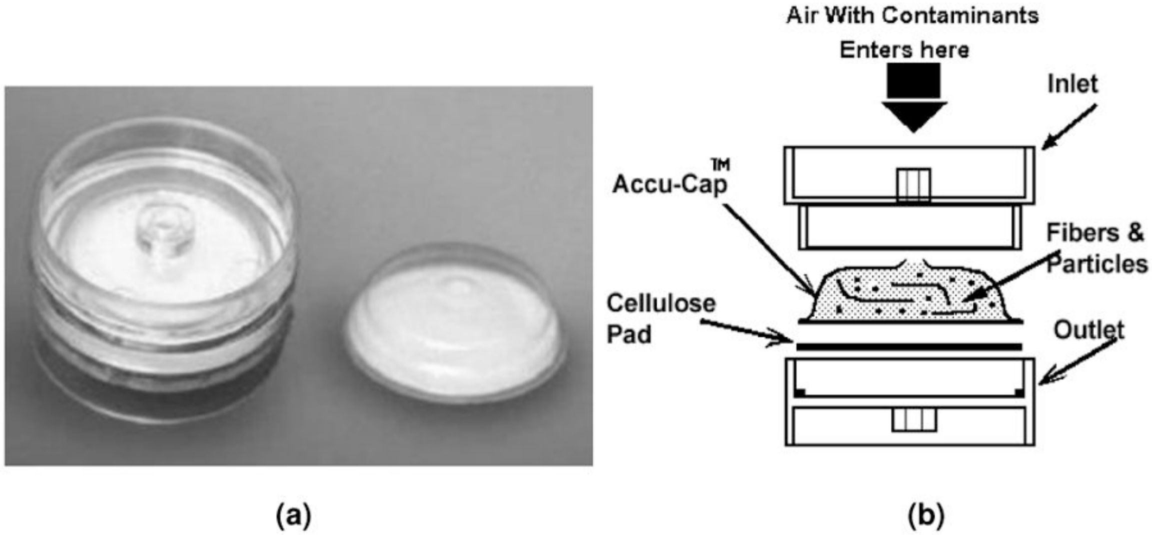


**Figure 1.**

(a) Photograph of a 37-mm diameter plastic closed-face cassette (CFC) sampler (left) and an internal filter capsule (right); (b) Schematic of the CFC sampler showing placement of the internal filter capsule (shaded portion) and cellulose back-up pad within the cassette. (Courtesy of SKC, Inc.)

**Table 1**

Storage stability data for PVC Accu-caps spiked with NIST SRM 1648, Urban Particulate Matter; percent change (%Δ) values are with respect to Day 0 weights (corrected for capsule weight and error in mean blank tare weight).

| **SRM 1648 ≈4 mg** | **Day 0 wt. (g)** | **Day 7 wt. (g)** | **%Δ** | **Day 14 wt. (g)** | **%Δ** | **Day 21 wt. (g)** | **%Δ** | **Day 28 wt. (g)** | **%Δ** |
|---|---|---|---|---|---|---|---|---|---|
| Sample 1 | 0,00386 | 0,00378 | -2,07 | 0,00375 | -2,85 | 0,00376 | -2,59 | 0,00376 | -2,59 |
| Sample 2 | 0,00383 | 0,00376 | -1,83 | 0,00375 | -2,09 | 0,00371 | -3,13 | 0,00374 | -2,34 |
| Sample 3 | 0,00385 | 0,00379 | -1,56 | 0,00378 | -1,82 | 0,00379 | -1,56 | 0,00377 | -2,08 |
| **%Δ mean ± std. dev.** | | | -1,82 ± 0,26 | | -2,25 ± 0,54 | | -2,43 ± 0,80 | | -2,34 ± 0,26 |
| **≈2 mg** | | | | | | | | | |
| Sample 1 | 0,00169 | 0,00166 | -1,78 | 0,00165 | -2,37 | 0,00166 | -1,78 | 0,00165 | -2,37 |
| Sample 2 | 0,00130 | 0,00126 | -3,08 | 0,00125 | -3,84 | 0,00124 | -4,62 | 0,00124 | -4,62 |
| Sample 3 | 0,00170 | 0,00165 | -2,94 | 0,00165 | -2,94 | 0,00167 | -1,76 | 0,00165 | -2,94 |
| **%Δ mean ± std. dev.** | | | -2,60 ± 0,71 | | -3,05 ± 0,74 | | -2,72 ± 1,65 | | -3,31 ± 1,17 |
| **≈1 mg** | | | | | | | | | |
| Sample 1 | 0,00090 | 0,00088 | -2,2 | 0,00088 | -2,2 | 0,00088 | -2,2 | 0,00088 | -2,2 |
| Sample 2 | 0,00110 | 0,00111 | +0,91 | 0,00110 | -0- | 0,00111 | +0,91 | 0,00111 | +0,91 |
| Sample 3 | 0,00117 | 0,00114 | -2,56 | 0,00113 | -3,42 | 0,00115 | -1,71 | 0,00113 | -3,42 |
| **%Δ mean ± std. dev.** | | | -1,28 ± 1,91 | | -1,87 ± 1,75 | | -1,00 ± 1,67 | | -1,57 ± 2,23 |
| **≈0,5 mg** | | | | | | | | | |
| Sample 1 | 0,00035 | 0,00020 | -43 | 0,00017 | -51 | 0,00020 | -43 | 0,00020 | -43 |
| Sample 2 | 0,00035 | 0,00031 | -11 | 0,00023 | -34 | 0,00023 | -34 | 0,00024 | -31 |
| Sample 3 | 0,00049 | 0,00040 | -18 | 0,00039 | -20 | 0,00039 | -20 | 0,00040 | -18 |
| Sample 4 | 0,00045 | 0,00029 | -36 | 0,00026 | -42 | 0,00025 | -44 | 0,00026 | -42 |
| **%Δ mean ± std. dev.** | | | -27 ± 15 | | -37 ± 13 | | -35 ± 11 | | -34 ± 12 |
| **≈0,1 mg** | | | | | | | | | |
| Sample 1 | 0,00005 | -0,00006 | -220 | -0,00008 | -250 | -0,00006 | -220 | -0,00006 | -220 |
| Sample 2 | 0,00016 | 0,00018 | +13 | 0,00017 | +6,3 | 0,00017 | +6,3 | 0,00017 | +6,5 |
| Sample 3 | 0,00024 | 0,00024 | -0- | 0,00022 | -8,3 | 0,00024 | -0- | 0,00024 | -0- |
| Sample 4 | 0,00018 | 0,00019 | +5,6 | 0,00019 | +5,6 | 0,00019 | +5,6 | 0,00020 | +11 |
| **%Δ mean ± std. dev.** | | | -50 ± 110 | | -64 ± 130 | | -52 ± 110 | | -51 ± 110 |

| **SRM 1648 ≈4 mg** | **Day 0 wt. (g)** | **Day 7 wt. (g)** | **%Δ** | **Day 14 wt. (g)** | **%Δ** | **Day 21 wt. (g)** | **%Δ** | **Day 28 wt. (g)** | **%Δ** |
|---|---|---|---|---|---|---|---|---|---|
| **Media blank** | | | | | | | | | |
| Sample 1 | 0,28025 | 0,28021 | -0,0143 | 0,28023 | -0,0071 | 0,28021 | -0,0143 | 0,28019 | -0,0214 |
| Sample 2 | 0,29180 | 0,29179 | -0,0034 | 0,29177 | -0,0103 | 0,29177 | -0,0103 | 0,29177 | -0,0103 |
| Sample 3 | 0,28421 | 0,28421 | -0- | 0,28419 | -0,0070 | 0,28419 | -0,0070 | 0,28419 | -0,0070 |
| Sample 4 | 0,30742 | 0,30744 | +0,0065 | 0,30744 | +0,0065 | 0,30744 | +0,0065 | 0,30745 | +0,0098 |
| Sample 5 | 0,31681 | 0,31676 | -0,0158 | 0,31677 | -0,0126 | 0,31676 | -0,0158 | 0,31677 | -0,0126 |
| Sample 6 | 0,32797 | 0,32791 | -0,0183 | 0,32791 | -0,0183 | 0,32792 | -0,0152 | 0,32792 | -0,0152 |
| **%Δ mean ± std. dev.** | | | -0,0092 ± 0,0075 | | -0,0081 ± 0,0131 | | -0,0094 ± 0,0085 | | -0,0095 ± 0,0166 |

## Table 2

Storage stability data for PVC Accu-caps spiked with Arizona Road Dust – Air Cleaner Test Dust; percent change (%Δ) values are with respect to Day 0 weights (corrected for capsule weight and error in mean blank tare weight).

| **AZ Rd. dust ≈4 mg** | **Day 0 wt. (g)** | **Day 7 wt. (g)** | **%Δ** | **Day 14 wt. (g)** | **%Δ** | **Day 21 wt. (g)** | **%Δ** | **Day 28 wt. (g)** | **%Δ** |
|---|---|---|---|---|---|---|---|---|---|
| Sample 1 | 0,00418 | 0,00421 | +0,72 | 0,00420 | +0,48 | 0,00420 | +0,48 | 0,00420 | +0,48 |
| Sample 2 | 0,00394 | 0,00396 | +0,51 | 0,00397 | +0,76 | 0,00397 | +0,76 | 0,00396 | +0,51 |
| Sample 3 | 0,00374 | 0,00373 | -0,27 | 0,00373 | -0,27 | 0,00373 | -0,27 | 0,00375 | +0,27 |
| **%Δ mean ± std. dev.** | | | +0,32 ± 0,52 | | +0,32 ± 0,53 | | +0,32 ± 0,53 | | +0,42 ± 0,13 |
| **≈2 mg** | | | | | | | | | |
| Sample 1 | 0,00221 | 0,00219 | -0,90 | 0,00220 | -0,45 | 0,00219 | -0,90 | 0,00219 | -0,90 |
| Sample 2 | 0,00185 | 0,00186 | +0,54 | 0,00186 | +0,54 | 0,00186 | +0,54 | 0,00186 | +0,54 |
| Sample 3 | 0,00185 | 0,00189 | +2,16 | 0,00189 | +2,16 | 0,00190 | +2,70 | 0,00190 | +2,70 |
| Sample 4 | 0,00187 | 0,00190 | +1,60 | 0,00186 | -0,53 | 0,00188 | +0,53 | 0,00188 | +0,53 |
| **%Δ mean ± std. dev.** | | | +0,85 ± 1,34 | | +0,43 ± 1,25 | | +0,72 ± 1,49 | | +0,72 ± 1,49 |
| **≈1 mg** | | | | | | | | | |
| Sample 1 | 0,00100 | 0,00103 | +3,0 | 0,00103 | +3,0 | 0,00103 | +3,0 | 0,00103 | +3,0 |
| Sample 2 | 0,00114 | 0,00116 | +1,8 | 0,00118 | +3,5 | 0,00117 | +2,6 | 0,00117 | +2,6 |
| Sample 3 | 0,00098 | 0,00099 | +1,0 | 0,00098 | -0- | 0,00100 | +2,0 | 0,00099 | +1,0 |
| Sample 4 | 0,00097 | 0,00096 | -1,0 | 0,00095 | -2,1 | 0,00097 | -0- | 0,00097 | -0- |
| **%Δ mean ± std. dev.** | | | +1,2 ± 1,7 | | +1,1 ± 2,6 | | +1,9 ± 1,3 | | +1,7 ± 1,4 |
| **≈0,5 mg** | | | | | | | | | |
| Sample 1 | 0,00036 | 0,00037 | +2,8 | 0,00037 | +2,8 | 0,00037 | +2,8 | 0,00039 | +8,3 |
| Sample 2 | 0,00041 | 0,00044 | +7,3 | 0,00044 | +7,3 | 0,00044 | +7,3 | 0,00044 | +7,3 |
| Sample 3 | 0,00044 | 0,00047 | +6,8 | 0,00046 | +4,5 | 0,00048 | +9,1 | 0,00048 | +9,1 |
| Sample 4 | 0,00051 | 0,00054 | +5,9 | 0,00053 | +3,9 | 0,00055 | +7,8 | 0,00055 | +7,8 |
| **%Δ mean ± std. dev.** | | | +5,7 ± 2,0 | | +4,6 ± 1,9 | | +6,8 ± 2,8 | | +8,1 ± 0,8 |
| **≈0,1 mg** | | | | | | | | | |
| Sample 1 | 0,00032 | 0,00033 | +3,1 | 0,00034 | +6,3 | 0,00019 | -41 | 0,00019 | -41 |
| Sample 2 | 0,00008 | 0,00013 | +63 | 0,00014 | +75 | 0,00014 | +75 | 0,00014 | +75 |
| Sample 3 | 0,00010 | 0,00011 | +10 | 0,00012 | +20 | 0,00012 | +20 | 0,00014 | +40 |

| AZ Rd. dust ≈4 mg | Day 0 wt. (g) | Day 7 wt. (g) | %Δ | Day 14 wt. (g) | %Δ | Day 21 wt. (g) | %Δ | Day 28 wt. (g) | %Δ |
|---|---|---|---|---|---|---|---|---|---|
| Sample 4 | 0,00008 | 0,00012 | +50 | 0,00010 | +25 | 0,00011 | +38 | 0,00011 | +38 |
| **%Δ mean ± std. dev.** | | | +32 ± 29 | | +32 ± 30 | | +23 ± 48 | | +28 ± 49 |
| **media blank** | | | | | | | | | |
| Sample 1 | 0,32999 | 0,33000 | +0,0030 | 0,33002 | +0,0091 | 0,33000 | +0,0030 | 0,33002 | +0,0091 |
| Sample 2 | 0,23152 | 0,23150 | -0,0086 | 0,23153 | +0,0043 | 0,23150 | -0,0086 | 0,23151 | -0,0043 |
| Sample 3 | 0,26775 | 0,26772 | -0,0112 | 0,26775 | -0- | 0,26774 | -0,0037 | 0,26775 | -0- |
| Sample 4 | 0,30638 | 0,30637 | -0,0033 | 0,30639 | +0,0033 | 0,30638 | -0- | 0,30639 | +0,0033 |
| Sample 5 | 0,32830 | 0,32830 | -0- | 0,32834 | +0,0122 | 0,32831 | +0,0030 | 0,32832 | +0,0061 |
| Sample 6 | 0,32176 | 0,32172 | -0,0124 | 0,32177 | +0,0031 | 0,32176 | -0- | 0,32177 | +0,0031 |
| **%Δ mean ± std. dev.** | | | -0,0054 ± 0,0063 | | +0,0053 ± 0,0045 | | -0,0011 ± 0,0045 | | +0,0029 ± 0,0048 |

**Table 3**

Computed recoveries from spiked PVC Accu-caps for certified reference material (CRM) spiking weights of ≈0,5 to ≈4 mg per sample.

| Spiking level | CRM | Mean recovery ± std. dev., % |
|---|---|---|
| ≈4 mg | NIST SRM 1648 | 96,1 ± 1,6 (n=7) |
| | Arizona Road Dust | 96,8 ± 4,9 (n=3) |
| ≈2 mg | NIST SRM 1648 | 93,1 ± 6,2 (n=6*) |
| | Arizona Road Dust | 97,4 ± 1,3 (n=4) |
| ≈1 mg | NIST SRM 1648 | 91,6 ± 9,0 (n=7) |
| | Arizona Road Dust | 102 ± 3,2 (n=4) |
| ≈0,5 mg | Arizona Road Dust | 87,5 ± 9,4 (n=4) |

*One statistical outlier omitted (Q-test, p=0,05)

## Table 4

Sample weights for Arizona Road Dust and PVC capsules obtained by NIOSH vs. independent gravimetric laboratory results reported by Bureau Veritas North America (BVNA).

| Sample | NIOSH wt. (g) | BVNA wt. (g) | Δ (g) | % Δ |
|---|---|---|---|---|
| *Arizona Road Dust* | | | | |
| ≈4 mg #1 | 0,00418 | 0,00408 | -0,00010 | -2,4 |
| ≈4 mg #2 | 0,00394 | 0,00379 | -0,00015 | -3,8 |
| ≈4 mg #3 | 0,00374 | 0,00361 | -0,00013 | -3,5 |
| ≈2 mg #1 | 0,00221 | 0,00210 | -0,00011 | -5,0 |
| ≈2 mg #2 | 0,00185 | 0,00188 | +0,00003 | +1,6 |
| ≈2 mg #3 | 0,00185 | 0,00186 | +0,00001 | +0,54 |
| ≈2 mg #4 | 0,00187 | 0,00178 | -0,00093 | -4,8 |
| ≈1 mg #1 | 0,00100 | 0,00105 | +0,00005 | +5,0 |
| ≈1 mg #2 | 0,00114 | 0,00116 | +0,00002 | +1,7 |
| ≈1 mg #3 | 0,00098 | 0,00094 | -0,00004 | -4,1 |
| ≈1 mg #4 | 0,00097 | 0,00098 | +0,00001 | +10 |
| ≈0,5 mg #1 | 0,00036 | 0,00038 | +0,00002 | +5,6 |
| ≈0,5 mg #2 | 0,00041 | 0,00042 | +0,00001 | +2,4 |
| ≈0,5 mg #3 | 0,00044 | 0,00047 | +0,00003 | +6,8 |
| ≈0,5 mg #4 | 0,00051 | 0,00056 | +0,00005 | +9,8 |
| ≈0,1 mg #1 | 0,00032 | 0,00031 | -0,00001 | -0,97 |
| ≈0,1 mg #2 | 0,00008 | 0,00009 | +0,00001 | +13 |
| ≈0,1 mg #3 | 0,00010 | 0,00003 | -0,00007 | -70 |
| ≈0,1 mg #4 | 0,00008 | 0,00008 | -0- | -0- |
| *PVC capsule media blanks* | | | | |
| #1 | 0,28019 | 0,28036 | +0,00017 | +0,061 |
| #2 | 0,29177 | 0,29195 | +0,00018 | +0,062 |
| #3 | 0,28419 | 0,28436 | +0,00017 | +0,060 |
| #4 | 0,30745 | 0,30762 | +0,00017 | +0,055 |
| #5 | 0,31677 | 0,31698 | +0,00021 | +0,066 |
| #6 | 0,32792 | 0,32812 | +0,00020 | +0,061 |
| #7 | 0,33002 | 0,33018 | +0,00016 | +0,048 |
| #8 | 0,23151 | 0,23165 | +0,00014 | +0,060 |
| #9 | 0,26775 | 0,26792 | +0,00017 | +0,063 |
| #10 | 0,30639 | 0,30659 | +0,00020 | +0,065 |
| #11 | 0,32832 | 0,32853 | +0,00021 | +0,064 |
| #12 | 0,32177 | 0,32197 | +0,00020 | +0,062 |

## Table 5

Summary of field gravimetric results obtained from sets of PVC Accu-caps housed in closed-face cassette (CFC) samplers (n=15) and Institute of Occupational Medicine (IOM) samplers (n=12) collected in metal foundries.

| Data set | Accu-cap ($mg \cdot m^{-3}$) | IOM ($mg \cdot m^{-3}$) | Avg. ratio ([Accu-cap$\cdot$IOM$^{-1}$]) |
|---|---|---|---|
| 1 | 9,0 | 9,7 | |
| | 9,1 | 10,8 | |
| | avg. = 9,1 | avg. = 10,3 | 0,88 |
| 2 | 3,9 | 5,2 | |
| | 4,0 | 5,5 | |
| | avg. = 4,0 | avg. = 5,4 | 0,74 |
| 3 | 9,3 | 12,4 | |
| | 8,7 | 5,8 | |
| | avg. = 9,0 | avg. = 9,1 | 0,99 |
| 4 | 10,3 | 11,0 | |
| | 9,8 | 11,1 | |
| | 6,6 | 8,1 | |
| | 9,0 | | |
| | avg. = 9,0 | avg. = 10,1 | 0,89 |
| 5 | 4,7 | 5,4 | |
| | 4,8 | 6,0 | |
| | 4,6 | 3,8 | |
| | 4,9 | | |
| | 5,1 | | |
| | avg. = 4,8 | avg. = 5,1 | 0,94 |
| Mean overall ratio ([Accu-cap$\cdot$IOM$^{-1}$]) = 0,89 | | | |