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Nanotechnologies — Method to quantify air concentrations of carbon black and amorphous silica in the nanoparticle size range in a mixed dust manufacturing environment

Nanotechnologies — Quantification du noir de carbonne et de la silice amorphe nanométriques en suspension dans l'air en ambiance de production

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 229, *Nanotechnologies*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Nanomaterials are widely used in industrial settings in the manufacture of consumer products. Carbon black and/or amorphous silica are commonly used in consumer products, such as rubber products, insulating materials, and others. Although these materials typically exist as agglomerates in dimensions larger than the nanoscale, there is also potential for worker exposure to these materials in the nanoscale size range. In spite of the widespread use of nanomaterials such as these, quantification of air concentrations of specific nanomaterials in mixed dust settings, such as a manufacturing environment, have been challenging to date and has been identified as a hindrance to the development of nano-specific occupational exposure limits (Gordon, et al. 2014; Hansen, et al. 2012; van Broekhuizen, et al. 2013). This method outlines a technique whereby particles carbon black and amorphous silica can be identified, distinguished, and quantified (in terms of air concentrations) by size in such manufacturing settings. It is anticipated that although this method is specific to carbon black and amorphous silica, the general principles of the method can be applied to many materials in a variety of manufacturing environments. This method advances beyond existing techniques for analysis in that it provides quantitative information regarding exposure to specific materials by size; many other methods provide quantitative information on nanoparticle exposures that are incapable of differentiating by material type. This method includes both a defined methodology for collecting air samples in the manufacturing settings as well as a methodology for analyzing the sample to obtain appropriate information for quantifying air concentration of the materials of interest. Application of this methodology has recently been published in the peer-reviewed literature (Kreider, et al. 2015).

This document will provide guidelines to quantify and identify particles carbon black and/or amorphous silica in air samples collected in a mixed dust industrial manufacturing environment. The guidelines describe air sample collection and the characterization of the particles in the air samples by both particle size and elemental composition. The particles in the air sample are collected in the various stages of a cascade impactor with cut-offs for median particle size between 6 nm and 10 µm. This impactor determines the number particle size distribution in real-time based on the particle aerodynamic diameter. Particles collected on each stage are collected for off-line analysis using Transmission Electron Microscopy (TEM) and Energy Dispersive Spectrometry (EDS) to identify amorphous silica and carbon black particles. The TEM-EDS measurement provides the elemental composition and source of the particles in each stage. Scanning Electron Microscopy (SEM) is also an option to TEM in the electron microscopy/dispersive spectrometry combination. The concentration of particles of a specific nanomaterial in a given size range (#/cm^3) is given by the product of the total particle count for size range (#/cm^3) obtained from the cascade impactor and the fraction of particles identified as the specific material of interest (e.g. carbon black or amorphous silica) from the TEM-EDS results. Though this technique is described for carbon black and amorphous silica, the technique can be applied to the measurement of other particle types, provided they are in the size range of 6 nm to 2,5 µm and can be observed by TEM/SEM and chemically characterized by EDS.

At this time, this methodology represents the one of the methods available to quantify chemical-specific exposures to nanoparticles by size with this degree of sensitivity. Many of the other existing methods that can speciate and quantify chemical exposure in this size range are mass-based, and thus are limited by mass-based detection limits that are high when compared to the mass of particles in this size range. Furthermore, although other sampling methods may be amenable to the techniques described herein, none have been evaluated or validated for this purpose. Therefore, this methodology offers increased sensitivity for quantification of exposure to specific particle types in the nanoscale when such an interest arises. This methodology could be implemented as a higher tier step in an occupational exposure assessment sampling strategy for nanomaterials, particularly in the event hot spots for exposure are identified using other methods and there is an interest in understanding the nature of the exposure. Results from this analysis can be used to compare to health benchmarks, as they become available, to understand potential health risk of workers. In addition, it could be useful in selecting appropriate personal protective equipment (PPE) at a very early stage of the manufacturing process, when required.

Nanotechnologies — Method to quantify air concentrations of carbon black and amorphous silica in the nanoparticle size range in a mixed dust manufacturing environment

1 Scope

This document provides guidelines to quantify and identify air concentration (number of particles/cm^3) of particles of carbon black and/or amorphous silica by size in air samples collected in a mixed dust industrial manufacturing environment.

The method is defined for air samples collected with an electrical low pressure cascade impactor (ELPCI). on a 25 mm polycarbonate substrate. The method is suitable for sampling in manufacturing environments where there are a variety of particle types contributing to the overall atmosphere. This method is applicable only to environments with chemically and physically distinct particles contributing to aerosols or when confounders can be controlled (e.g. diesel sources). Other sampling methods can also be suitable, though this document is limited to describing methods associated with the electrical low pressure cascade impactor.

Samples collected with the electrical low pressure cascade impactor are analyzed via TEM and EDS to for particle morphology and elemental composition, respectively, to permit identification of particles by type. This information is then used, in conjunction with particle concentration by size range, as determined by the electrical low pressure cascade impactor, to determine concentration of the materials of interest by size.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 10312, *Ambient Air — Determination of asbestos fibres — Direct-transfer transmission electron microscopy method*

ISO 4225, *Air quality — General aspects — Vocabulary*

ISO/TS 80004-1, *Nanotechnologies — Vocabulary — Part 1: Core terms*

ISO 22309, *Microbeam analysis — Quantitative analysis using energy-dispersive spectrometry (EDS) for elements with an atomic number of 11 (Na) or above*

ISO/TS 10798, *Nanotechnologies — Charaterization of single-wall carbon nanotubes using scanning electron microscopy and energy dispersive X-ray spectrometry analysis*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 4225 and ISO/TS 80004-1 apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
particle aerodynamic diameter
diameter of a sphere of density 1,000 kg m^{-3} that has the same settling velocity as the irregular particle

3.2
cascade impactor
device for simultaneously collecting particles separately in a number of size ranges by impaction, depending on the momentum

3.3
cut-off
size of particles at which the retention efficiency of an instrument device drops below a specified value under defined conditions

3.4
nanoscale
size range from approximately 1 nm to 100 nm

3.5
nanomaterial
material with any external dimension in the nanoscale or having internal structure or surface structure in the nanoscale

3.6
particle
minute piece of matter with defined physical boundaries

3.7
sampling time
interval of time over which a single sample is taken

4 Principle

Air sampling is conducted using an electrical low pressure cascade impactor (ELPCI). The ELPCI is a cascade impactor with multiple stages representing different size bins, distinguished based on aerodynamic diameter. The cascade impactor is connected to a vacuum pump, which pulls air (and subsequently airborne particles) into the impactor. As particles enter the cascade impactor, they are charged and then subsequently separated by aerodynamic size onto different stages. As the particles come into contact with the surface of their appropriate stage, they transmit an electrical current to the ELPCI. This electrical current is then converted into a particle count for that stage, thus permitting the ELPCI to conduct real-time particle counting. This particle count is reported as number of particles per cm^3 of air. In addition to the particle counting function, the ELPCI also allows for collection of samples on each stage, using a 25 mm polycarbonate substrate.

Following completion of air sampling, these substrates are analyzed via TEM to identify the particles on each stage by morphology coupled with EDS to identify the particles on each stage by elemental composition. A minimum of 100 distinct particles per stage are analyzed using TEM- EDS to identify the particle type, based on the chemical signature of the particles. Carbon black, made up primarily of elemental carbon, elicits a strong carbon signal (and absence of other signals) on the EDS, whereas silica (SiO_2) elicits a strong silicon and oxygen signal. These profiles are used to designate particles as carbon black or amorphous silica. Where there are potential confounders present in the sample, such as soot (for carbon black) or crystalline silica (for amorphous silica), particle morphology and/or diffraction pattern must be relied upon to identify the particles. Morphology is the main attribute that distinguishes carbon black from soot, whereas morphology and diffraction can be used to distinguish crystalline silica from amorphous. Furthermore, comparison to the morphology of the source materials (e.g. carbon black or amorphous silica) can be evaluated under TEM to ensure appropriate assignation of the particle type. The relative proportion of particles (e.g. number of particles by type out of 100 total particles identified per size fraction) identified by type of particle (carbon black, amorphous silica, or other) is the key outcome of this analysis.

To determine air concentration of the materials of interest (e.g. carbon black or amorphous silica), the proportion of particles identified by type is multiplied by the total number of particles in each size bin determined during the real-time air sampling. The outcome of this analysis is the air concentration of each material type by size in number of particles/cm^3.

Though the method described herein will specifically focus on the analysis of carbon black and amorphous silica, this methodology is transferrable to other materials, provided they have a unique signal under EDS and/or are distinguishable via morphology under TEM.

5 Abbreviations

EDS	energy dispersive spectroscopy
ELPCI	electrical low pressure cascade impactor
HEPA	high efficiency particulate arrestance
LOD	limit of detection
PM10	airborne particles with an aerodynamic diameter less than 10 µm
SEM	scanning electron microscopy
TEM	transmission electron microscopy

6 Reagents

During the analysis, use only reagents of recognized analytical grade. All agents listed are required to execute the transfer of the polycarbonate substrate content onto a TEM grid in accordance with ISO 10312. No additional reagents are required for execution of this specification.

WARNING — Use the reagents in accordance with the appropriate health and safety regulations.

6.1 Water, fibre-free.

A supply of freshly distilled, fibre-free water, or another source of fibre-free, pyrogen-free water shall be used.

6.2 Chloroform, analytical grade.

Distilled in glass, preserved with 1 % (WV) ethanol; used to dissolve polycarbonate substrate.

6.3 l-Methyl-2-pyrrolidone.

Used to dissolve polycarbonate substrate.

6.4 Dimethylformamide.

Used to dissolve polycarbonate substrate.

6.5 Glacial acetic acid.

Used to dissolve polycarbonate substrate.

6.6 Acetone.

Used to dissolve polycarbonate substrate.

7 Apparatus

7.1 Air sampling — Equipment and consumable supplies

7.1.1 Electrical low cascade pressure impactor (ELPCI)

An electrical low pressure cascade impactor, consisting of a minimum of 14 size bins, should be used for collection of air samples. Of the size bins, at least 3 should be within the nanoscale, defined as less than 100 nm. The ELPCI should be operated in accordance with the manufacturer's instructions.

An example of measurement capability for a commercially available electrical low pressure impactor operating at a 10 l/min flow rate is presented in the Table 1.

Table 1 — Measurement range for commercially available electrical low pressure cascade impactor (Dekati, 2011)

D50 % nm	Di nm	Number min $1/cm^3$	Number max $1/cm^3$	Mass conc. min $\mu g/m^3$	Mass conc. max $\mu g/m^3$
10 000	Unavailable	Unavailable	Unavailable	Unavailable	Unavailable
6 800	8 200	0,1	2,40E+04	30	10 000
4 400	5 500	0,1	2,40E+04	10	3 000
2 500	3 300	0,15	5,40E+04	3	1 000
1 600	2 000	0,3	1,10E+05	1,4	450
1 000	1 300	0,5	1,90E+05	0,7	210
640	800	1	3,50E+05	0,3	100
400	510	2	6,40E+05	0,1	50
260	320	3	1,20E+06	0,07	20
170	210	5	2,10E+06	0,03	10
108	140	10	3,70E+06	0,02	5
60	80	20	7,30E+06	0,005	2
30	42	50	1,70E+07	0,002	0,5
17	22	100	3,40E+07	0,001	0,25
6	1	250	8,30+07	0,000 4	0,13
D50 % = Aerodynamic diameter cut-offs for 50 % efficiency; Di = Geometric mean aerodynamic diameter of stage					

7.1.2 Limit of Detection

Based on the table above and the expected volumetric flow rate of the sampling equipment (10 l/min), the practical limits of detection (LOD) for both carbon black and amorphous silica (based on a one-second sample) by particle size are found in Table 2.

Table 2 — Limits of detection by particle size (Dekati, 2011)

D50 % nm	Di nm	LOD in # of particles
10 000	Not applicable	
6 800	8 200	16,6
4 400	5 500	16,6
2 500	3 300	24,9
D50 % = Aerodynamic diameter cut-offs for 50 % efficiency; D_i = Geometric mean aerodynamic diameter of stage; LOD = Limit of detection.		

Table 2 *(continued)*

D50 % nm	**Di** nm	**LOD** **in # of particles**
1 600	2 000	49,8
1 000	1 300	83
640	800	166
400	510	332
260	320	498
170	210	830
108	140	1 660
60	80	3 320
30	42	8 300
17	22	16 600
6	1	41 500

D50 % = Aerodynamic diameter cut-offs for 50 % efficiency;

D_i = Geometric mean aerodynamic diameter of stage;

LOD = Limit of detection.

Detection limits presented in this table are approximate based on a typical impactor; each impactor has exact specifications that may result in deviations from the reported detection limits. The detection limit for a specific duration can be calculated by converting the minimum concentration of particles detectable to number of particles based on sample duration and volumetric flow rate of the impactor. Increasing the duration of sampling will decrease the detection limit based on increased volume of collection.

7.1.3 Real-time aerosol monitor

A real-time aerosol monitor, capable of measuring mass of particulate consistent with the size range of the ELPCI (e.g. PM10 if largest size bin of ELPCI is 10 µm), should be operating coincident with the ELPCI sampling. Output of the real-time aerosol monitor will be used to appropriately designate sample time of the ELPCI to prevent under/overloading of the substrate (see 8.2 and Table 3). The real-time aerosol monitor should be operated in accordance with the manufacturer's instructions.

7.1.4 Vacuum pump

A vacuum pump, calibrated to a flow rate consistent with what is required for appropriate size fractionation in the ELPCI (i.e. 10 l/min), will be required to introduce air and particles into the cascade impactor. The vacuum pump should be operated in accordance with the manufacturer's instructions.

7.1.5 Polycarbonate substrate

A 25-mm polycarbonate substrate. The substrate should be compatible with the stages of the ELPCI. Polycarbonate substrates should be handled using tweezers or similar to prevent transfer of oils from the skin. This polycarbonate substrate is mounted in the ELPCI for sample collection.

Other collection media, such as polycarbonate filters, mixed cellulose ester filters, or others, could be considered only if and when proper steps are taken to ensure size fractionation is unaffected by the selected media.

7.1.6 Hydrocarbon grease

A silicone- and halogen-free vacuum grease is recommended for use on the surface of the selected stages to prevent particle bounce within the cascade impactor. Select stages will be treated with this grease at the time of sampling.

7.2 Analytical/microscopy laboratories

The laboratories used for sample analysis, including sample preparation immediately prior to microscopy analysis, shall be sufficiently free of contamination such that blank substrate analyses demonstrate an absence of carbon black or amorphous silica as established by the method detection limit.

7.3 Sample analysis — Equipment and consumables

7.3.1 Transmission electron microscope (TEM)

A transmission electron microscope (TEM) is the preferred microscope for analysis of these samples, though scanning electron microscopes (SEM) or scanning-transmission electron microscopes (STEM) are also acceptable. The selected microscope must be capable of being coupled with an EDS. The TEM is used for morphological identification of the collected particles.

7.3.2 Energy dispersive spectrometry (EDS)

The electron microscope must be equipped with an EDS to allow for chemical identification of individual particles. The EDS unit should be capable of providing spectra for particles of interest, background spectra, and providing estimations of peak size to allow for identification of particle type. In order to ensure proper carbon detection, TEM settings, such as energy beam, or sample coatings are issues to consider.

7.3.3 Computer

Collection and storage of many particle images and spectra are necessary, and these may be collected and analyzed conveniently by various computer programs. Therefore, a computer with adequate memory is required to accommodate the programs and storage of images and spectra for subsequent analysis.

7.3.4 TEM Grid

Air samples collected in the polycarbonate substrates shall be transferred to TEM grid to facilitate analysis. Copper TEM grids are recommended for this analysis. To facilitate the relocation of individual grid openings for quality assurance purposes, the use of grids with numerical or alphabetical indexing of individual grid openings is recommended.

8 Air sample collection

8.1 Preparation for sampling

In preparation of air sampling, the ELPCI should be assembled, connected to the vacuum pump, and connections evaluated for leaks according to manufacturer's instructions. For characterization of nanoscale particles of carbon black and/or amorphous silica, each stage of interest (e.g. all of the nanoscale stages, plus any additional stages of interest) shall be mounted with a polycarbonate substrate. The largest stages (at least the stages representing median aerodynamic diameters ≥2,5 µm) should be mounted with a polycarbonate substrate treated with a hydrocarbon grease, which prevents particle bounce of the largest particles. These stages will not be analyzed further, as the grease prevents analysis via TEM-EDS. If information from the larger stages is desired, the polycarbonate substrates should not be sprayed with the hydrocarbon grease to permit analysis of the particles landing on those stages; however under these conditions, there is an increased risk of particle bounce. Additional preparations specified by the manufacturer, such as electrometer zeroing, should also be completed with substrates in place. Desired conditions for operation of the ELPCI should be established prior to sampling, including vacuum pump flow rate, establishment of designated pressure within the cascade impactor, electrometer zeroing, and any other instrument set-up requirements recommended by the manufacturer.

8.2 Determination of sampling time

Prior to initiation of sampling, it is necessary to determine sampling time based on total relevant particle air concentration in the vicinity of which the sampling is to occur. Control of sampling time will ensure appropriate loading onto the substrates. Where possible, sampling time should be consistent with the most appropriate duration for characterizing potential exposure during the activity of interest, as much as possible. The recommended sampling durations are:

Table 3 — Recommended Air Sampling Durations based on Particle Concentrations during Sampling

Reported air concentrations in relevant size range (e.g. PM_{10}) mg/m^3	**Recommended sample duration** min
0 to 0,5	15
0,5 top 1,5	10
>1,5	5

These values are based on the air concentration in mg/m^3 as measured by the real-time particle monitor. The sampling times are recommended to prevent overloading that would impair microscopy of individual structures (primary particles, aggregates, agglomerates) on any individual stage. Depending on the volumetric flow rate used during sampling and mass per stage that can be deposited as per equipment specifications, longer sampling times may be possible, if desired. In the event longer sampling durations are required to more accurately characterize the full scope of activity, longer sampling durations can be used. It is recommended to conduct a test-run to evaluate loading across the size bins at longer sample durations using optical microscopy and tailor sample time accordingly; if longer sampling times still permit distinction of individual structures deposited on the substrates, they can be employed, as needed

8.3 Sample collection procedure

The air samples collected for this effort are area samples, and as such should be collected in the vicinity in which characterization of air concentration the material of interest (e.g. carbon black or amorphous silica) is desired. Where possible, it is recommended to reduce or eliminate sources of particulate with similar chemical and morphological composition to particles of interest. For example, for sampling for carbon black, removing or reducing sources of combustion products (e.g. diesel equipment) is recommended to reduce confounding of results and ease analysis. If an appropriate background location can be identified, background samples could also be collected and considered. All samples should be taken in accordance with the sampling duration recommended in Table 2. Upon completion of sampling, the vacuum pump should be turned off, and data recorded from the ELPCI, as described in manufacturer instructions. Following completion of air sampling, polycarbonate substrates should be carefully removed from all stages of the cascade impactor and saved in appropriate filter media containers for subsequent analysis.

For each round of air sampling, a blank sample should also be collected. Blank samples should be handled in the same manner, undergoing all steps of the original sampling. To collect a blank sample, the ELPCI should be fitted with a HEPA filter at the inlet to prevent particles from entering the impactor. All other steps remain the same as in-facility sampling.

9 Procedure for analysis

9.1 General

Analysis of samples involves preparation of substrates for mounting onto a TEM grid, microscopic analysis of the sample, and collection of data in the form of particle images and EDS spectra. To analyze the sample, each sample is scanned at low magnification first to identify areas of particle impaction and subsequently analyzed via EDS at higher magnification. EDS spectra are used to identify particle types and determine fractional percentage of particles of interest.

9.2 Preparation of substrates

Polycarbonate substrates should be prepared for TEM-EDS analysis and mounted to a TEM grid according to ISO 10312. At least three grid preparations per substrate should be prepared, focusing preparations to central areas of the substrate to maximize likelihood of capturing particle impaction locations on the grid. Where possible, visual information regarding location of particles should be used in grid preparation.

9.3 Sample analysis

9.3.1 Instrument conditions

To begin analyzing samples, the microscope should be fixed to a low magnification (e.g. 100X) to scan the grid for transfer quality and to identify location of impaction spots within the grid. Magnification during analysis of impaction spots will vary, depending on particle size; magnification should be set to identify ten to 15 particles per field. TEM settings, such as energy beam or sample coatings are issues to consider to properly detect carbon.

9.3.2 Data collection

To analyze the samples, first, the grid is scanned at low magnification to identify a suitable area with high quality transfer for evaluation (e.g. the substrate is no longer visible and the grid intact), after which an area just outside of the impaction spots, where particles or structures (aggregates or agglomerates) are in a clear monolayer with minimal contact. If a grid is not cleared appropriately (e.g. residual substrate is present on the grid), the grid should be rejected and an alternate grid evaluated. Following verification of grid acceptability and identification of impaction areas, the microscope should be adjusted to an appropriate magnification to identify ten to 15 particles or structures within the field. EDS spectra and images for each structure (primary particle, aggregates or agglomerates) that is completely within the field should then be manually collected, and peaks identified in accordance with ISO 22309. EDS spectra should be collected from a central point in the structure. Quantification of peak concentrations is not necessary. This process should be repeated until at least 100 structures are analyzed from a minimum of 7 fields. In addition, for each field, a background spectrum should also be collected in a location absent of particles to characterize the potential influence from the grid and coating.

Following collection of spectra and images, each spectrum should be analyzed for primary elemental composition to assist with particle identification. In addition to EDS spectra, morphology based on images can also be used for identification. Bulk samples of carbon black and amorphous silica can be used to compare against images collected to verify the identification of the selected particles in the images. As an example, particles with strong carbon peaks in the absence of other chemical signals (when considering the background spectrum) and morphologies consistent with carbon black are identified as carbon black (see Figure 1 for an example of an EDS spectra and structure identified as carbon black). This method will permit for both identification of carbon black and amorphous silica using chemical and morphological information, but also serve to identify any other particles by chemistry found in the aerosol. Once a minimum of 100 structures from a stage have been identified by type, fractions for each particle type (by chemistry/morphology combination) are calculated for each stage based on the 100 structures. One hundred structures is recommended based on the requirement in NIOSH Method 7400 that indicates evaluating 100 structures is considered sufficient for characterizing the distribution of fibre types on an air sample (NIOSH, 1994).

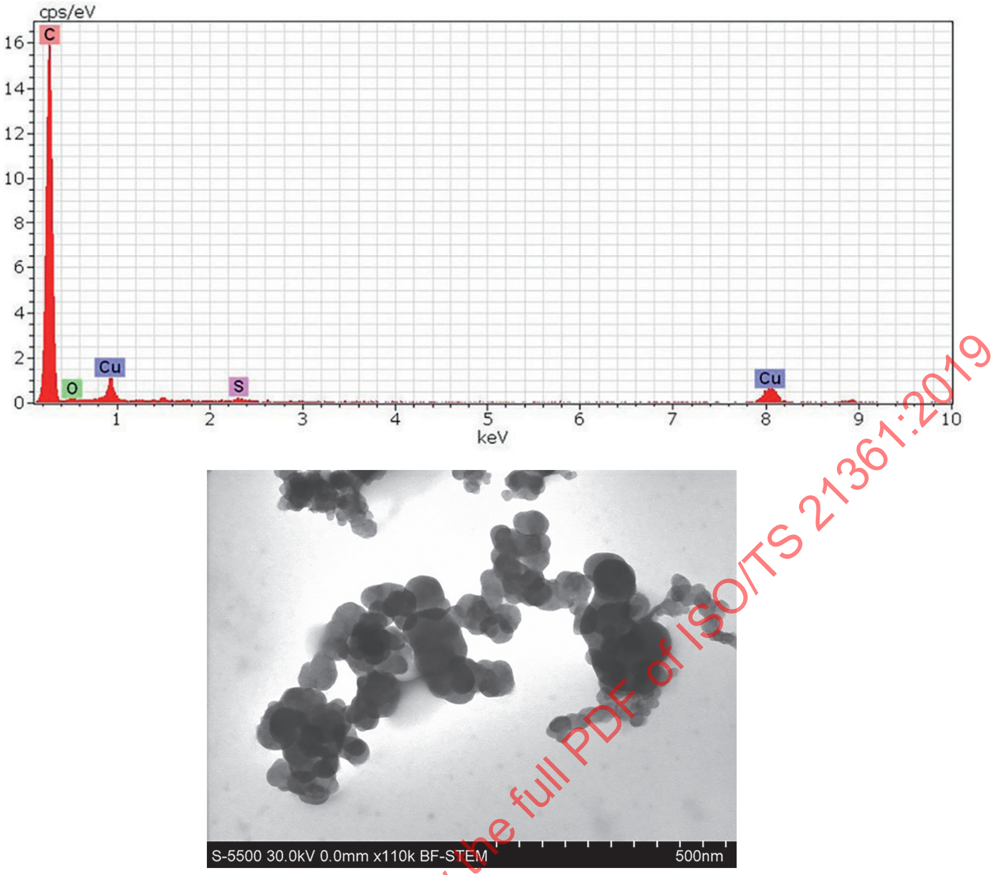

Figure 1 — EDS spectra (top) and microscopic image (bottom) of particle identified as carbon black (from Kreider, et al. 2015)

9.4 Calculation of air concentration

Following microscopic analysis of all samples and determination of fraction of particles identified by particle type, air concentration of carbon black and/or amorphous silica can be calculated using the following formula:

$$C_{\text{mat,x}} = F_{\text{mat,x}} \times C_{\text{total,x}}$$

such that:

$C_{\text{mat,x}}$ = concentration of material of interest in air on *x* stage impactor;

$F_{\text{mat,x}}$ = fraction of partgicles identified as material of interest on *x* stage of impactor;

$C_{\text{total,x}}$ = total air concentration of all particles on *x* stage of impactor.

The number-based air concentration of total particles for each size bin should be obtained from readings from the ELPCI. Particle air concentration as reported by the ELPCI is dependent on particle density of the material of interest. If information on particle density for the specific materials of interest are available, these shall be used. Otherwise, for mixed dust environments, the default particle density assumed by the ELPCI shall be used. Fraction of particles for each type are determined from microscopy results.

Air concentration by particle type can be calculated for any size ranges of interest, with the exception of inclusion of the largest size bin. To specifically calculate a total concentration of particles in the nanoscale by particle type, the following formula shall be used:

$$C_{\text{mat,nano}} = \sum C_{\text{mat,x}} \text{ for all nanoscale stages}$$

10 Uncertainties and performance criteria

The measurement uncertainties of particle concentration of carbon black or amorphous silica by size can be attributed to uncertainties related to particle counting with the ELPCI and/or particle identification with TEM/EDS. These uncertainties are not quantifiable, but should be considered as outlined in 10.1 and 10.2, respectively.

10.1 Particle counting with the ELPCI

ELPCIs are engineered with strict specifications to maintain proper size fractionation and measurement of particle concentrations. Within an ElectricalLow Pressure Cascade Impactor, size fractionation is dictated by a strict control of internal pressure, which allows for size fractionation via impaction on the stages of the column. Real-time monitoring of pressure during sampling and maintenance of pressure within manufacturer specifications will help to ensure proper size fractionation in accordance with the prediction of the manufacturer. For well-maintained and properly functioning ELPCIs, uncertainty associated with deviations in pressure are not anticipated. In the event pressure readings deviate from manufacturer specifications during sampling, caution should be taken in interpreting particle counting by size.

In addition to size fractionation, particle charge is a key to particle counting, as currently is the primary measurement used to determine the number of particles for each stage. As particles enter the ELPCI, they are charged. Depending on the stage on which the particle lands, the ELPCI software assigns a given current detected in the stage to represent a particle present in the stage. A rigorous establishment of a baseline charge by stage is necessary for accurate determination of current changes based on particle impaction. Maintaining the electrometer within recommended specifications prior to sampling (e.g. electrometer zeroing) will reduce uncertainty associated with influence of background current. Without proper electrometer zeroing, particle counts may be overpredicted by the ELPCI.

In addition, because the ELPCI fractionates by aerodynamic diameter, and all assumptions regarding charge and current are based on spherical particles, materials that deviate from aerodynamic behavior of spheres may carry more or less charge than a spherical particle, and therefore over or under predict the number of particles per stage. Comparing results of an ELPCI to other particle counting equipment may provide information regarding the accuracy of particle counting by size for specific materials. Such a comparison for carbon black and amorphous silica has indicated that for particle with aerodynamic diameters of 30 nm or greater, particle counts for the ELPCI are similar to those predicted with other equipment. Measurement uncertainty from the calibration of the ELPCI should be considered.

Lastly, though the ELPCI currently represents one of the currently available equipment capable of counting particles by size and allowing for collection of samples for off-line analysis to meet the goals of this analysis, the rate of air flow present at high pressures within the impactor may cause particles to bounce to lower stages for particles to deagglomerate, resulting in high concentrations of particles at lower stages. While steps are taken to reduce the amount of particle bounce through the use of hydrocarbon grease at the higher stages, this phenomenon may still occur. However, should particle bounce or deagglomeration occur, the results would overestimate exposure in the occupational setting, rather than underestimate. This is an important consideration with respect to uncertainty in the results, as any conclusions drawn based on these results would still be health-protective.