3 Identification, monitoring and control of dust exposures

Frank J. Hearl

INTRODUCTION

The recognition, evaluation and control of dust exposures in occupational environments can be complex. An occupational hygienist must assess the nature of the hazard, the likely target of the dust, and the properties of that dust responsible for the biological response of interest. Dust measurement and dust hazard evaluation is complex because of the need to characterize properties beyond the intensity of exposure, i.e. the dust concentration. It may be necessary to describe the exposure in terms of the particle size distribution, and the often inhomogeneous chemical or morphological properties of the dust. For fibrous minerals such as asbestos, particle shape may have profound impact on the toxicity of the material. Several descriptors of the particles may be used to characterize the concentration including: the mass of the particles, the mass of one chemical species in the particles, the active surface area, the number of particles, or the crystalline properties of the particles.

Some studies show that freshly generated dust containing crystalline silica will exhibit increased toxicity in lung cells compared with aged dust, due to the recent creation and presence of surface free radicals (Castranova *et al.*, 1996). Therefore, specific knowledge about the process that generated the dust and the interactions of the aerosol with the environment provide important information. Particles of varying sizes deposit in different regions of the lungs based on their aerodynamic size. The largest of the particles have settling velocities greater than the air velocity at the nostril and drop harmlessly away. Other smaller particles may be captured by the hair and mucus-covered surfaces of the nose and be quickly expelled or ingested. Still smaller particles can travel to the furthest depths of the lungs and deposit in the alveolar regions where gas exchange takes place with the blood.

The large particles captured in the nose, mouth or upper airways are cleared to the gastrointestinal tract within minutes or hours. They may contain soluble toxic materials that can be absorbed producing systemic toxicity, or they may produce local effects such as nasal cancer. Particles depositing in the upper airways may be responsible for chronic obstructive pulmonary disease, asthmatic reactions or lung cancer. The smallest particles – those that deposit in the alveolar regions of the lungs – are often associated with hypersensitivity pneumonitis (extrinsic allergic alveolitis), pneumoconiosis, including coal workers pneumoconiosis, asbestosis and silicosis.

In the preceding discussion, we have touched on the complexities of describing dust concentrations and the importance of characterizing particle size distributions, chemical properties, morphologic features, and the crystallinity of the inhomogeneous mixture we call 'dust'. Developing a strategy for appropriate identification, evaluation and control of dust must necessarily involve some or all of these factors. Monitoring

Occupational Lung Disease. Edited by Daniel E. Banks and John E. Parker. Published in 1998 by Chapman & Hall. ISBN 0 412 73630 6

exposure concentrations usually has one of three purposes:

- finding a dose–response relationship or mechanism of action as part of an epidemiologic study;
- evaluating the effectiveness of an engineering exposure control system; or
- checking compliance with an accepted exposure limit.

Typically, monitoring strategies developed for epidemiologic purposes are more complex than those needed for the other purposes. Reasonable feed-back and control strategies for recognizing and characterizing significant dust exposures often make assumptions about the size and composition of the dust being monitored to allow for rapid assessment. Likewise, enforcement monitoring strategies are usually based on some standardized sampling systems; however, they may require specific information about the chemical composition, morphology or crystallinity to demonstrate compliance or non-compliance with specific standards. Generally, complete characterization of the aerosol is not feasible or necessary for these routine dust exposure monitoring scenarios. However, strategies for characterizing exposure for assessing health risk in epidemiologic studies, animal exposure experiments, cell-culture studies and health risk assessments are often intricate. To improve the chances of detecting a true dose–response relationship in these studies, the most appropriate exposure metric must be used. Since it is often difficult or impossible to predict which metric will be the best correlate to the biological response, full characterizations of the exposure are usually required. The following sections will discuss these exposure metrics and the circumstances under which under each should be or has been used.

EXPOSURE METRICS

To quantify aerosol exposures, a variety of exposure metrics has been used. Some common descriptors of the exposure include:

- the total *mass* of particles in a given volume of air;
- the mass of particles with a particular size or aerodynamic property in a given. volume of air, usually *respirable* dust;
- the total *number* of particles in a given volume of air;
- the total number of particles with a particular size and/or shape (e.g. length to width *aspect* ratio) in a given volume of air;
- the mass of specific *chemical species* contained in a collected mass of particles in a given volume of air;
- the *relative composition* of a specific species compared with another species or the total mass or count (percentage composition);
- the *crystalline structure* of the dust particles; and
- the *active* surface area or number of surface functional groups in a given volume of air.

Over the years, each of these measures has been used to describe workplace exposures to particulate. Most of these metrics are quantified in terms of mass or number per stated volume of air, and have customarily been reported as milligrams per cubic meter (mg/m^3), or million particles per cubic foot (mppcf), or fibers per cubic centimeter (f/cc).

Generally, the sampling technique involves collecting and concentrating the particles from a measured volume of air. Some early dust collection systems relied upon gravitational settling of the dust onto an open-faced collection surface. Later techniques included accelerating the dust-laden air stream through a glass tube and impinging the particles in the bottom of a liquid-filled glass bottle. Other devices used thermal precipitation or electrostatic precipitation to attract the airborne particles toward surfaces that could later be analyzed. Similar to impinging the particles into a liquid-filled device, particles may be collected by impaction onto a flat-plate substrate that is often coated with grease or other sticky and non-volatile substance. Impactors are *size-selective samplers*, with the size of particles collected determined by the velocity with which the air stream is accelerated toward the collection surface. Perhaps the most common method for collecting dust samples is to use an air-mover (pump) to draw a known volume of dust-laden air through a filter for further analysis. If the air passes through a size-selective device, such as an impaction surface or a cyclone, before collection by a filter the largest particles are removed and only the small particles are collected on the filter.

Particle count

Once the dust is removed from the air and is concentrated in either a liquid impinger solution, non-volatile impactor grease, glue, or on a filter, it must be quantified. Generally, dusts collected using the impinger method were quantified by particle counting, while dusts collected using impactors or filters were analyzed for particle mass. A notable exception is the method used for counting fibers. In this case, a special filter is used that has properties that allow it to be 'cleared', that is it becomes transparent to visible light so that the particles can be seen, sized and counted by optical microscopy (NIOSH, 1994a).

Dust particles collected in the impinger solution are counted using a counting cell and a method adapted from those used to count plankton in water (Ayer, 1969). Rules for counting have not been standardized. The main steps in particle counting begin with the removal of a known portion of the solution containing the collected dust particles. This aliquot is transferred to a counting cell, which is a glass microscope slide constructed with a 1 mm-deep well to contain the solution. Particles that settle to the floor of the cell are counted using light field microscopy with a 10-power objective lens (Hall *et al.*, 1965). Opaque particles as small as 0.5 μm can be detected by trained observers under these conditions. The total particle count can then be computed, noting the ratio between the withdrawn portion and the total volume of the sample. Concentrations reported from impinger samples were usually stated in million particles per cubic foot, mppcf, computed by:

$$C = \frac{\text{COUNT}}{Q \times t}$$

where C is the concentration, COUNT is the number of particles in the impinger solution, Q is the pump flow rate, and t is the sampling time.

One investigator contacted 45 industrial hygiene laboratories and documented significant differences in their standard procedures involving: collection medium (alcohol or water or mixtures thereof), type of illumination (dark field versus light field microscopy), length of time between collection and counting, method of counting (i.e. counting only those particles settled on the bottom of the slide or focussing through the entire 1 mm depth of field), and settling time of the sample before counting was begun (Halley, 1946). As understanding developed among industrial hygienists that it was the respirable fraction that was most closely associated with pneumoconiosis, many industrial hygienists rejected counts of particles with observed diameters larger than 10 μm since these were 'not respirable' (Lippmann, 1983). Impinger count data were perceived to be highly inaccurate and subject to training and experience bias among counters (Ayer, 1969). With few exceptions, most occupational exposure limits and standards for dusts enforced since 1970 have been based on mass sampling methods. The preceding section was presented primarily to put historical dust data in proper context.

Particle mass

Particle mass is measured by *gravimetric* analysis. The particle collection substrate, which is usually either a filter or a greased impactor substrate, is pre-weighed on a precision microbalance under controlled conditions. This *tare* weight is carefully recorded along with the filter number. After sampling, the substrate is reweighed, under the same controlled environmental conditions. The difference between the final weight and the tare weight is the mass of particles collected.

For gravimetric mass measurements, the airborne concentration is computed by dividing the collected mass by the known volume of air from which they were collected. The air volume is usually computed by using a flow control device in conjunction with the pump to regulate the air flow rate, and to sample for a measured period of time, computing the concentration, usually reported as mg/m^3, by:

$$C = \frac{W_f - W_i}{Q \times t}$$

where C is the concentration, W_f is the final weight of the substrate, W_i is the initial weight of the substrate, Q is the pump flow rate, and t is the sampling time.

PARTICLE SIZE CRITERIA

Total dust

The Greenburg–Smith impinger, developed in 1925 by the US Bureau of Mines, the American Society of Heating and Ventilating Engineers, and the US Public Health Service, along with the more convenient size midget impinger (Figure 3.1), was used before 1970 as the dust sampling method of choice in the United States. The impinger sampler collects dust particles with diameters larger than 0.7 μm aerodynamic diameter with efficiencies ranging from 68 to 95%, depending on the particle density and the specific solution used in the impinger (Jacobson *et al.*, 1970). Impingers collect *total dust* samples. A common total dust sampler currently in use is the 37-mm or 25-mm

Figure 3.1 Total dust impinger sampler.

Figure 3.2 Total dust filter cassettes. Left, closed-faced; right, open-faced.

Figure 3.3 Inhalable dust sampler.

diameter filter housed in a plastic cassette (Figure 3.2), connected to an air sampling pump. The sample is collected by removing the protective end caps and sampling through the small 4-mm cassette opening for a known time, at a known air flow rate, usually from 1–2 l/min. Alternatively, the filter may be mounted in a three-piece cassette such that the entire end section of the cassette is removed for sampling giving a 37-mm or 25-mm open-faced filter for collection of the sample. Both sampling configurations have their respective problems. When sampling through the cassette's small-diameter opening, some large particles are not efficiently sampled. In the open-faced configuration, the sample is subject to contamination since some particles can be propelled onto the filter. Samples collected using mannequins in a wind tunnel showed that the amount of dust that enters the nose of the mannequin can be substantially greater than the amount of dust sampled by total dust samplers (Mark *et al.*, 1994). Clearly, the two configurations described are working as size-selective samplers and not collecting a true total dust samples. However, with the mannequin studies previously reported, it was shown that for large particles, up to 100 μm in diameter, the nose is only 50% efficient for collection of those particles (Vincent and Armbruster, 1981; Armbruster and Breuer, 1982; Vincent and Mark, 1982). Therefore, a more appropriate assessment of whole-body dust burden might be *inhalable dust*, where the sampler is designed to collect particles as efficiently as the nose. Such inhalable dust samplers have been designed and described elsewhere (Figure 3.3) (Mark and Vincent, 1986; Vincent, 1995).

Another problem with characterizing total dust concentrations occurs because the size distribution is constantly changing. Dust may be generated continuously or intermittently by the dust-generating process with some particular size-distribution. However, once airborne, the particles begin to settle due to gravitational forces according to the relationship:

$$c_s = \frac{\rho_p \, d_p^2 \, g}{18 \, \mu}$$

where c_s is the particle settling velocity, ρ_p is the particle density, d_p is the particle diameter, g is the gravitational constant, and μ is the viscosity of the air (Friedlander, 1977). Since the settling velocity of particles is dependent on particle size, over time with no new dust generation, the airborne size distribution will appear depleted of large particles and enriched in small-sized particles, perhaps setting up a vertical spatial concentration gradient in the environment.

In spite of these inherent problems, measures of total dust have been successfully used for exposure control monitoring, even where the health risk is primarily associated with the respirable dust fraction. For example, in the Peoples Republic of China, a total dust device has been used for over 30 years to track and control dust exposures (Figure 3.4). In the US, for many years the prescribed sampling system for silica dust monitoring was the impinger combined with particle counting. By controlling the total dust concentration so that the levels were below recommended

Figure 3.4 Total dust sampler – Chinese sampler.

limits, the incidence of silicosis was significantly reduced (Costello and Graham, 1988). This sampling strategy was successful because the concentration of total dust and the concentration of respirable dust and its chemical components are often sufficiently correlated for a particular dust-generating operation (Wu *et al.*, 1992). It is this principle that simplifies dust characterization tasks for occupational hygienists involved in controlling dust exposures and those involved in monitoring compliance and non-compliance with specific standards. Additionally, where systemic toxicity of soluble particles or local effects in the head or upper airways may be a concern, exposure control should be based on the total or inhalable dust concentration.

Respirable dust

In 1970, the American Conference of Governmental Industrial Hygienists, (ACGIH) proposed a specific definition for respirable dust based on human pulmonary deposition data. This definition specified the fraction of particles that could pass through a size-selective pre-separator and be collected on a filter for gravimetric analysis by specifying several points on the respirable size-selection curve (Lippmann, 1970):

Aerodynamic diameter (μm)	*% Passing selector*
≤ 2	90
2.5	75
3.5	50
5.0	25
10	0

Figure 3.5 Respirable dust sampler – 10-mm nylon cyclone.

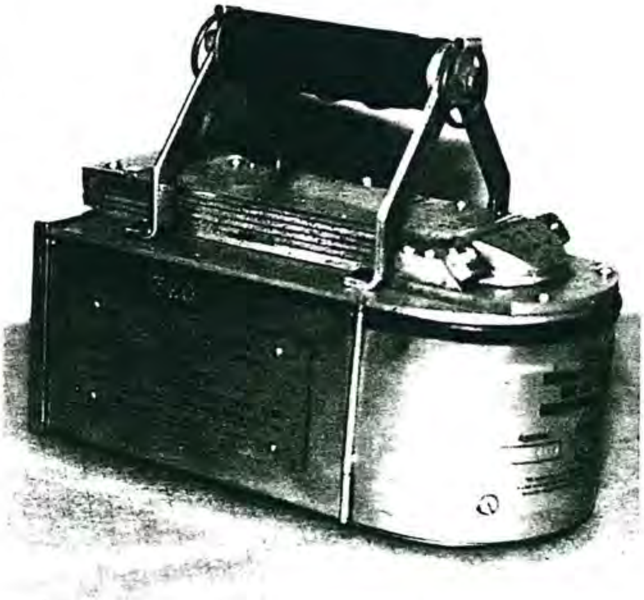

Figure 3.6 Respirable dust sampler – horizontal elutriator (MRE).

Another early approach to the definition of respirable dust was to define it as that dust collected by a particular instrument. In the United States, the 10-mm nylon cyclone (Figure 3.5) has been used as the size-selective pre-separator which defines respirable dust. The British Medical Research Council (BMRC) adopted the horizontal elutriator, also known as the Medical Research Establishment instrument or MRE (Figure 3.6), to provide the basis for the respirable dust definition in Britain. The MRE instrument is portable, battery operated, and has collection properties such that particles less than 1 μm aerodynamic diameter are collected with nearly 100% efficiency, particles with 5 μm aerodynamic diameters are collected with 50% efficiency, and particles larger than 7 μm aerodynamic diameter are excluded from collection on the filter.

Defining respirable dust by specifying a particular sampling system is not without its problems. For example, the 10-mm nylon cyclone's collection efficiency curve shifts with changes in operating flow rate. From the outset, there has been controversy about whether the flow rate should be 1.4 l/min, 1.7 l/min or 2.0 l/min (Bartley and Brauer, 1982; Caplan *et al.*, 1977a; Knuth, 1969; Ettinger *et al.*, 1970; Tomb and Raymond 1970; Tomb and Treaftis, 1976). After examining the flow rate bias curves, which measure the difference between the ACGIH respirable dust definition and the sampler's observed performance, the US National Institute for Occupational Safety and Health (NIOSH) specified 1.7 L/min as the flow rate for respirable dust sampling using the 10-mm nylon cyclone preseparator (NIOSH, 1994b). However, in selecting a sampler for underground coal mines, where the US coal mine dust standard was based on data obtained from Britain (using MRE-collected dust), it was observed that if a cyclone sampler was operated at 2.0 L/min, and a correction factor of 1.38 was applied, an acceptable correlation to MRE-collected samples was obtained for a wide range of coal mining settings (Tomb *et al.*, 1973; Caplan *et al.*, 1977b). This procedure, using the 10-mm nylon cyclone operated at 2.0 L/min and multiplying the concentration by a constant factor of 1.38, has been the legal definition of respirable coal mine dust in US coal mines since the early 1970s. The use of the '1.38 correction factor' has been disputed, with some investigators noting that these same data could be used to obtain different correction factors (NRC, 1980). The use of correction factors to adjust measurements made with samplers having varying particle collection characteristics can create some unusual biases in the data. For example, the 38% positive correction applied to cyclone-collected respirable dust provides adjustment for dust in the size range from 3.5 to 7 μm, where the MRE instrument has a higher collection efficiency than the cyclone. However, if the mine environment contains a surplus of particles in the smallest size ranges, below 2 μm, where both instruments collect essentially 100% of the particles, then the 1.38 factor introduces a positive 38% bias in the measurement (Bartley and Breuer, 1982). This scenario becomes particularly relevant with the introduction of diesel-powered equipment into coal mining environments. Diesel equipment produces a particle fume that is usually smaller than 1 μm and can represent a sizable fraction of the collected dust (McCawley and Cocalis, 1986; Marple *et al.*, 1991).

In summary, whenever two samplers, such as the MRE or the 10-mm nylon cyclone, are used to sample the same environment, the conversion between measured concentrations will be dependent on the particle size distribution.

International size-selective dust definition

Recognizing the limitations of the existing respirable dust definitions, the international community has attempted to standardize the absolute definition of respirable dust (ISO, 1981). These efforts also extend the definitions to other physiologically significant fractions, and provide a criterion for assessing the adequacy of instruments used to evaluate concentrations. The ACGIH Air Sampling Procedures Committee developed criteria for size-selective sampling (Phalen, 1984). They defined the rationale for size-selective sampling and the recommended definitions and sampler performance characteristics for sampling inhalable (formerly called *inspirable*), thoracic and respirable mass fractions (Hinds, 1984; John, 1984; Lioy *et al.*, 1984; Soderholm, 1984). An accuracy criterion that could be applied to specific instruments which meet the various size-selective criteria was provided (McCawley, 1984). The committee also established a logical framework for selecting the appropriate size fraction based on the dust-hazard being controlled (Stuart *et al.*, 1984). Others tested these criteria against a range of observed work place particle-size distributions and found that the acceptability criteria were too broad and found two samplers acceptable even though they may report dust concentrations as different as 0.71 mg/m^3 and 4.3 mg/m^3 (Bartley and Doemeny, 1986).

The International Standards Organization (ISO), Comité Européen de Normalisation (CEN) and ACGIH cooperated in the most recent efforts to establish specific definitions for size-selective dust measurements (Soderholm, 1989). In an attempt to harmonize the various definitions in common use, e.g. the ACGIH and the BMRC definitions of respirable dust, several compatible dust size definitions were developed and adopted by the various national and international organizations (Figure 3.7) (ISO, 1991; CEN, 1993; ACGIH, 1995a).

Inhalable (or inspirable) fraction

The *inhalable* or *inspirable* fraction describes those particles that enter through the nose or mouth during inhalation. The definition was developed based on data collected using mannequins of human heads and torsos in a wind tunnel (Vincent and Armbruster, 1981; Armbruster and Breuer, 1982; Vincent and Mark,

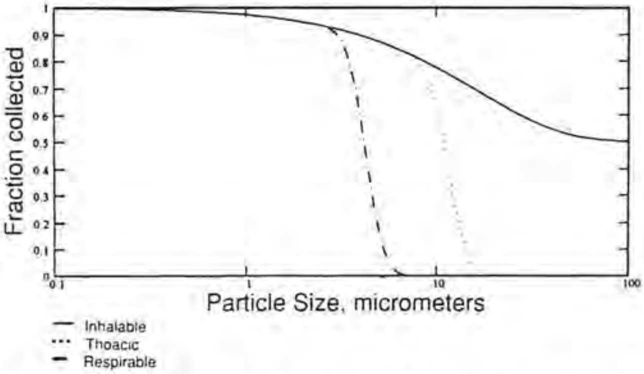

Figure 3.7 International standard definitions for inhalable, thoracic and respirable dust.

1982). The inhalable fraction is described by the equation:

$$SI(d) = 0.5\ (1 + e^{-0.06d})$$

where $SI(d)$ is the inhalable particle sampling efficiency for particles with aerodynamic size of d in μm. Using the inhalable size fraction for monitoring control of dust exposures is appropriate for substances that are hazardous or toxic when deposited anywhere in the respiratory system including nose and head, or when they are ingested. This creates some complications with regards to prior occupational exposure limits that may have been based on total dust sampling. The ratio of side-by-side concentrations measured by total dust sampling and inhalable dust sampling have been reported to range between factors of 0.7 to 3.4 (Vinzents *et al.*, 1995). As with the case of different respirable dust samplers, the relationship between total dust sampling and inhalable dust sampling is dependent on other factors such as the overall particle size distribution and wind speed and direction with respect to the sampling port (Lidén and Kenny, 1994).

Thoracic size fraction

The thoracic fraction was previously defined by a cumulative lognormal function with a mass median diameter of 10 μm and a geometric standard deviation, σ_g of 1.5 (Raabe, 1984). This definition includes those particles small enough to penetrate past the nasal–pharyngeal passages of the head, and includes particles that deposit in the trachea–bronchial tree and the alveolar region of the lung. The ISO, CEN and ACGIH definition of the thoracic fraction begins by considering only those particles first determined to be inhalable, and then applying the cumulative normal frequency distribution as:

$$ST(d) = SI(d\ [\ 1 - F(x)\ ]$$

where $ST(d)$ is the thoracic particle sampling efficiency for particles with an aerodynamic diameter of d, $\Gamma = 11.64$ μm (the geometric mean of the log-normal distribution). $\Sigma = 1.5$ (the geometric standard deviation of the log-normal distribution), $SI(d)$ is the inhalable dust sampling efficiency, and the function, $F(x)$ which is defined by:

$$F(x) = \int_0^x \frac{e^{-(x^2/2)}}{\sqrt{2\pi}}\, dx$$

where,

$$x = \frac{\ln(d\ /\ \Gamma)}{\ln(\Sigma)}$$

Respirable size fraction

The respirable dust definition that was adopted by the ISO, CEN and ACGIH is based on the cumulative probability density function, $F(x)$, of a standardized normal random variable, e.g. based on this function, the respirable fraction is defined by:

$$SR(d) = SI(d)\ [\ 1 - F(x)\ ]$$

where $\Gamma = 4.25$ μm (the geometric mean of the log-normal distribution), and $\Sigma = 1.5$ (the geometric standard deviation of the log-normal distribution), and $SI(d)$ is the inhalable dust sampling efficiency. The function $F(x)$, and x are as defined in the preceding section.

Dust deposition

Each of these definitions describes 'penetration' curves for particles, based on their aerodynamic diameters. Particles with aerodynamic diameters much smaller than the median cut-point are collected with almost 100% efficiency. Samplers designed to measure penetration do not measure 'deposition' in any particular region of the lung. Alveolar deposition is less than 100% for particles between 0.1 and 1.0 μm aerodynamic diameter because many particles in this size range are subsequently exhaled (Chan and Lippmann, 1980; Stahlhoffen, *et al.*, 1980). Respirable dust samplers will overestimate the fraction deposited in the alveolar region of the lung for sub-micrometer size particles. The fraction of dust which deposits in the lungs shows a marked reduction from 80–95% deposition of 5 μm particles, dropping to 20–40% deposition for 0.5 μm particles, then rising to between 50–70% deposition for particles smaller than 0.1 μm (Chan and Lippmann, 1980). The deposition curve shifts downward as settling and impaction become less important for depositing particles in the smaller size range, while the process of diffusive deposition becomes more important for the particles smaller than 0.1 μm. For mechanically generated dusts which exist primarily in the 2 to 15 μm size-range, this issue may be insignificant. However, for fumes generated through diesel combustion or by welding processes, there could be a significant difference between measured exposure and deposited dose (Soderholm and McCawley, 1990). Likewise, tracheobronchial deposition is not measured by the thoracic fraction samplers because those samplers include and collect particles that would be deposited in the alveolar region, i.e. the respirable fraction. Extrathoracic deposition (the fraction deposited in the head and pharynx, down to and including the larynx, i.e. the 'nasal–pharyngeal' fraction) is not measured with an inhalable fraction sampler because that fraction includes particles that would penetrate further and be deposited in the tracheobronchial and alveolar regions of the lung (Raabe, 1984).

Other size-selective fractions

(a) Ambient sampling using the PM_{10} sampling system

The US Environmental Protection Agency (EPA) specified in its National primary and secondary air quality standards for particulate matter, that particulate matter shall be measured in the ambient air as PM_{10} particles with an aerodynamic diameter less than or equal to a nominal 10 μm (EPA, 1995). The requirements for a PM_{10} sampler (Figure 3.8) were based on the lung deposition curve proposed by Chan and Lippmann (1980), Kashdan *et al.* (1986) and Liu and Pui (1986).

(b) Vertical elutriator for cotton dust sampling

Another instrument-specified size fraction is collected by the Lumsden–Lynch vertical elutriator (Figure 3.9). This device was designed to collect airborne cotton dust with a mass-median diameter of 15 μm. The vertical elutriator is specified in US regulation as the standard method for measuring airborne cotton dust in textile mills and selected secondary cotton processing industries (OSHA, 1995). The size-selection criteria is similar to the thoracic fraction adopted by the ACGIH, CEN and the ISO.

Figure 3.8 PM_{10} sampler – environmental aerosols.

Criteria for selection of an appropriate size

The selection of an appropriate sampling system should be based on the criteria that the measured quantities are related to the risk being controlled (Roach, 1953; Ayer, 1969). The selection of one or more of the size-selective criteria could be guided by the following classification scheme:

- Respirable dust sampling – suggested when the aerosol is insoluble and the disease is associated with the gas exchange regions of the lungs, as in fibrotic diseases like silicosis or coal workers' pneumoconiosis, or emphysema.
- Thoracic dust sampling – suggested when the aerosol is believed to produce asthma, to aggravate chronic bronchitis, or to be responsible for inducing bronchogenic tumors. Thoracic sampling is appropriate if the material which deposits in the head airways does not contribute to its toxicity.
- Inhalable or inspirable dust – suggested when the aerosol is likely to be absorbed by the body wherever it deposits, or if the dust is associated with nasal cancer as in wood workers (ISO, 1981; Stuart *et al.*, 1984).

As an example, for environmental particulate exposures, concern should be based on health effects including asthma, bronchitis, emphysema, fibrosis and pneumoconiosis. Based on these broad needs, the

Figure 3.9 Lumsden–Lynch vertical elutriator.

selection of PM_{10} sampling for the National Ambient Air Quality Standard for particulate is appropriate. Alternatively, the most appropriate size-selective criterion for measuring silica concentrations, exclusive of other particulate exposures, would be the respirable dust criterion. If the sampled aerosol has a relatively stable size distribution, then thoracic sampling (PM_{10}) would provide an acceptable surrogate for respirable mass sampling when measuring ambient exposures.

THE COMPOSITION OF DUST

Crystalline silica in dust particles

Because the earth's crust is predominantly composed of crystalline silica, whenever earth is disturbed through mining, milling, or earth moving operations, there exists a potential for exposure to crystalline silica. Historically, the development of dust sampling programs and systems revolve around the need to sample quartz-containing dusts.

The ACGIH recognized that the health risk of exposure to dust was directly related to the total dust concentration and to the concentration of crystalline silica, also known as quartz, in the dust. In 1962, after reviewing available data on dust-exposed populations, the ACGIH adopted a Threshold Limit Value (TLV) for dust containing quartz (ACGIH, 1986):

$$\text{TLV} = \frac{250}{\%\text{quartz}+5}\ \text{mppcf}$$

To apply this exposure limit, the concentration of crystalline silica (%quartz) was quantified through chemical analysis of collected airborne samples. The TLV for that exposure setting was then calculated using the formula above. The total dust level was then measured using the impinger sampler, and that result was compared with the calculated TLV. For example, if the %quartz was computed as 5%, the calculated TLV would be 25 mppcf. Any dust sample over this amount would exceed the exposure limit. In 1970, to make the TLV consistent with a nuisance dust standard of 30 mppcf, the formula was revised to (ACGIH, 1986):

$$\text{TLV} = \frac{300}{\%\text{quartz}+10}\ \text{mppcf}$$

For both of these formulas, the measurement method was the impinger, coupled with particle count analysis (ACGIH, 1986). Based on growing body of literature in the US, summarized by Morrow (1964), and the findings of the Johannesburg Conference (Orenstein, 1960), in 1968 the ACGIH proposed a respirable dust TLV based on size-selective gravimetric sampling. The revised formula, based on the particle count formula, would be designed to provide approximately equal protection from dusts containing quartz, and to limit the exposure to total airborne dust. Based on findings of the US Public Health Service, a value of 0.1 mg/m^3 of quartz was found equivalent to the 10 mppcf exposure concentrations observed from the Vermont Granite Shed study where the quartz was 25% (Ayer *et al.*, 1968; Ayer, 1995). Besides the quartz limit, it was decided to limit size-selected respirable dust to 5 mg/m^3, so the total dust concentration would not exceed 15 mg/m^3, based on an assumption that the respirable dust was approximately one-third of the total dust concentration. Combining these limits into an analogous formula led the ACGIH to adopt the following formula for control of respirable dust (ACGIH, 1986):

$$\text{TLV} = \frac{10}{\%\text{quartz}+2}\ \text{mg/m}^3$$

The %quartz value in the formula was to be measured from airborne samples except where it was decided that other methods were acceptable (Ayer, 1969). In 1986, the ACGIH adopted a revised silica standard that requires the direct measurement of respirable crystalline silica, comparing the result against a 0.1 mg/m^3 TLV (ACGIH, 1986). The revised TLV was proposed without reference to the mixture formula, that was implicitly included in the former TLV formula (Hearl, 1996).

Particle size dependence in analysis of crystalline silica

There are three principal analytical methods used to analyze crystalline silica. The wet-chemistry method is based on the differential solubility of crystalline silica, various silicates, and amorphous silica when treated with hot phosphoric acid. Following the dissolution of the amorphous silica and silicates, the remaining crystalline 'free silica' was quantified either by weighing the residue (Talvitie, 1951) or, more precisely by colorimetric determination (Talvitie and Hyslop, 1958; Talvitie, 1964). In an attempt to find suitable alternatives to the Talvitie method, investigators examined other spectral methods such as infrared spectroscopy (IR) and X-ray diffraction (XRD). One study examined particle counting using a light microscope with dispersion staining (Edwards, 1965). In a comparative study of IR and XRD methods, it was found that both IR and XRD could quantify crystalline silica with approximately equal precision and ruggedness (NIOSH, 1983). However, both methods were subject to interference by other common minerals, and both exhibited a response dependent on the particle size of the sample. When a pure silica standard (Min-U-Sil®) was used to establish a calibration curve for the XRD method, the instrumental response for the 10 μm Min-U-Sil® was 1.6 times the response obtained for the 5 μm Min-U-Sil® (NIOSH, 1983). Similarly, it was found that the quartz absorbance peaks observed using the IR method varied by as much as 30% for particle diameters varying from 0.65 μm to 5.8 μm (Lorberau, 1989). Due to these response characteristics of the methods, the choice of a suitable reference standard was critical to making accurate measurements of silica concentrations (Verma *et al.* 1992). To improve the standards for XRD analysis of silica, the National Bureau of Standards produced a Standard Reference Material (NBS-SRM#1878) by cycloning Min-U-Sil® 5, which was an α-quartz sold by U.S. Silica, West Virginia. The particle size distribution of the SRM approximated that found on respirable dust samples, thus reducing systematic bias from particle-size effects on instrument response. However, where the particle size distribution has a low dispersion (i.e. a small geometric standard deviation of the distribution, approaching a monodisperse aerosol) the size-dependence of the analysis could become important. For example with ground silica (silica flour) samples, the particles tend to be very small and the X-ray diffraction response is weak. Comparison of such finely ground samples to the larger respirable dust standard reference material will result in an underreporting of the true concentration in the sample. Selection of an appropriately-sized standard reference is essential where crystalline silica analysis is required (Bhaskar *et al.*, 1994).

Coal dust sampling

The selection of an appropriate sampler for coal mine dusts is based on the basic criteria that the measured quantity should be related to the risk of developing occupational lung disease from that exposure (Roach, 1953; Ayer, 1969). A variety of devices has been used over the years to quantify airborne dust levels including: filters, impactors, impingers, elutriators, electrostatic precipitators, thermal precipitators and cyclones (Lippmann, 1983; Lioy *et al.*, 1984; Glenn and Kraft, 1986).

In British coal mines, thermal precipitators were used beginning in 1936, to monitor airborne dust levels (Fay and Rae, 1959; Roach, 1959; Fay and Ashford, 1964). The samples were analyzed microscopically. Dust concentrations were assessed by particle counting, reporting the number of particles in the size range 1–5 μm diameter per cubic foot of mine air. Following the recommendations from the Johannesburg Pneumoconiosis Conference that mine dust sampling be based on respirable size-selective gravimetric measurements (Orenstein, 1960; Chamberlain *et al.*, 1971), the British National Coal Board (NCB) chose to use the Medical Research Establishment MRE dust sampler, a horizontal elutriator described earlier. Since 1965, the MRE system has been used to measure dust exposures in British mines.

Based on the toxicity of coal mine dust reported in British studies (Jacobsen *et al.*, 1971) the US Federal Coal Mine Health and Safety Act of 1969 as amended in 1977 (US Code, 1977) specified that the US standard for coal mine dust would be 3.0 mg/m^3 on the effective date of the Act (1969), and the standard would be reduced to 2.0 mg/m^3 three years after the date of enactment of the Act. The Act did not explicitly define respirable dust, stating only that respirable dust means dust measured with a device approved by the Government. As noted earlier, the chosen instrument was a 10-mm nylon cyclone and a 37-mm filter in series with a pump operated at 2.0 l/min air flowrate.

The sampling procedure used to assess a coal miner's exposures to crystalline silica in US mines is indirect. A respirable dust sample is analyzed for crystalline silica. If the coal mine dust sample contains greater than 5% crystalline silica, a reduced respirable dust standard is calculated and compared with the respirable dust concentration measured by gravimetric methods. The MSHA Respirable Dust Standard (RDS) is reduced according to the formula (MSHA, 1995):

$$\mathrm{RDS} = \frac{10\,\mathrm{mg/m^3}}{\text{Percent Quartz}}\ .$$

A RDS established for a particular work place may be enforced for extended periods of time (up to one year) without additional analysis of the percentile composition of silica. This approach assumes that the concentration of silica is relatively constant in the material being processed. Some have argued that this is a poor assumption for many mining situations (Corn *et al.*, 1985).

MIXED DUST EXPOSURES

Other toxic substances may be present in the environment, and may exist as airborne particulate. Particulate, liquid aerosol or vapors capable of adsorbing on the surface of airborne particles may work as carriers for the condensed vapors, transporting the substances and depositing them in the deepest regions of the lungs. Depending on environmental conditions, particle-adsorbed materials may, after collection on a filter, be desorbed back into the environment (Soderholm and Ferron, 1992). This would introduce a *negative* bias as an artifact of the sampling system. These types of problems have been observed even when sampling high-molecular weight materials such as some polynuclear aromatic hydrocarbons that are often associated with diesel emissions. As a result, sampling methods for such substances may require the use of a sorbent back-up tube placed in series behind the particulate filter in order to capture vapors desorbing from particle surfaces (NIOSH, 1994c; Soderholm, 1995).

Early control limits on dust, in the generic sense, were not based on toxicological data. Maximum allowable concentration limits were set for generic dust, termed 'nuisance dust' or 'inert or nuisance particulate' at 50 mppcf or 15 mg/m^3 in order to gain some control over excessively dusty operations. Part of the definition of 'nuisance dust' was that they did not produce significant disease or toxic effect (e.g. air spaces remain intact, no fibrosis or scarring, and tissue reactions are reversible) when inhaled in controlled amounts. Recent inhalation studies have shown that high exposures to dust, generically, can significantly retard the lungs' protective clearance mechanisms, leading to various additional dysfunctional and pathologic changes (Morrow *et al.*, 1991). Based on these findings there is justification for sampling and controlling respirable dust exposure *per se*, even when there are no specific observable toxic constituents in the dust.

Some specific effects caused by dust exposures with specific chemical compositions are cataloged in Tables 3.1 and 3.2 (Langer, 1986). From the description of the expected health response, the appropriate size-selective sampling criteria can be selected using the decision logic outlined on p. 37.

INTERNATIONAL COMPARABILITY OF DUST MEASUREMENTS

As described in the preceding sections, there are a number of choices available for exposure metrics, for instruments to use in quantifying those exposures, and for subsequent analysis to further describe the composition, crystallinity and morphology of the dust particles. However, there is an additional factor that confounds attempts to achieve international comparability for dust measurements and exposure standards. The sampling strategy and protocol itself can have a dramatic influence on the measured exposure. These elements of the sampling strategies that can influence measurements include:

- the duration for sample collection;
- the location for sample collection; and
- the frequency of sample collection and statistical treatment of the results.

Sample duration is sometimes dictated by the sampling system being used as well as the prevailing exposure concentration. For measurement of very low concentrations by gravimetric methods, large air sample volumes are required to concentrate a sufficient mass on the sampling substrate for quantification. In cases of high exposure concentrations, relatively short sampling periods are required to avoid overloading the sampling media, resulting in an unusable or unreliable quantitative result.

By regulation, US coal mine dust samples are required to be collected on a 'portal-to-portal' basis, i.e. sampling the entire work-shift from the time the worker enters the workplace to the time they leave (MSHA, 1995). Generally, industrial hygienists in the US will attempt to collect full-shift dust samples for comparison with 8-hour time-weighted average exposure limits. However, as is done in Chinese dusty trades, the sampling strategy may be limited to those periods of time when the dust-generating tasks are in progress (Wu *et al.*, 1992). Obviously for such task-based sampling schemes, higher levels will generally

Table 3.1 Natural materials associated with human disease: minerals

Rock type	*Components/uses*	*Comments*
Amosite Anthophyllite Crocidolite Chrysotile	Used as asbestos	Asbestosis, lung cancer; cancer of the gastrointestinal tract; pleural and peritoneal mesothelioma; possible increase in other malignancies.
Bauxite	Hydrated aluminum oxides	Some reports of lung scarring
Biotite Muscovite Phlogopite	Insulation and filler	Pulmonary fibrosis; silicosis; strong association with free silica, likely a major factor in producing fibrosis.
Coal	Anthracite Bituminous ± quartz	Coal workers' pneumoconiosis; 'miner's consumption'; focal emphysema; anthracosis, silicoanthracosis; may be related to rank (anthracite >bituminous); 'melanosis' or 'black lung'; greater incidence of tuberculosis; may progress to 'massive fibrosis'.
Diatomaceous earth	Opaline diatom fragments	Some lung scarring developed; much greater in processing when calcined (conversion of opal to cristobalite); silicosis with progressive massive fibrosis.
Feldspar	Ceramics	Silicosis; often attributed to the included crystalline silica.
Fluorite	Industrial flux	Fluorosis; silicosis.
Fullers earth	Butonites and related morillonites ± quartz	Pneumoconiosis without massive fibrosis and nodules (diffuse); mottled X-ray appearance; some related silicosis.
Granite Quartzite Sandstone Slate	All with large amounts of quartz	Silicosis; silico-tuberculosis; nodular silicosis; fibrosis; enlarged and hardened lymph glands. Silicotic nodules in spleen
Graphite	Crucibles; high temperature facings; electrodes; paints	Graphite pneumoconiosis; tuberculosis; resembles silicosis.
Gypsum Anhydrite	Gypsum ± evaporites Anhydrite ± sulfur	Some bronchitis; when calcined for industrial purposes, toxic effect to skin increases some irritation to eyes, nose and pharynx.
Kaolin	Ceramics; filler	Some lung scarring observed, but only in areas associated with crystalline silica.
Kyanite	Ceramics	Some lung scarring observed.
Limestone Marble Dolomite	Calcite ± quartz Dolomite ± quartz	Some bronchitis; some emphysema, some scarring reported; when calcined for industrial purposes, toxicity increases; caustic burns; dermatitis; ulceration of skin; injury to conjunctiva and cornea.
Nepheline	Industrial uses	Nephelosis; some lung scarring.
Olivine	Industrial uses	Silicosis; some lung scarring.
Pumice	Volcanic glass, some devitrification to quartz	Resembles silicosis; some linear scarring.
Silica Quartz Cristobalite Tridymite Chalcedony Flint	Abrasive; industrial uses	Silicosis; 'potters' asthma'; 'potters' consumption'; silicotic nodules in spleen; silico-tuberculosis; progressive pulmonary fibrosis; progressive pulmonary fibrosis. Cristobalite and tridymite are more fibrogenic than quartz.
Talc	Industrial uses; filler	Talcosis; talc pneumoconiosis.

From Langer (1986).

Table 3.2 Natural materials associated with human disease: elements

Metal	*Mineral*	*Comments*
Aluminum	Bauxite ore Corundum	Aluminosis; some lung scarring; pneumoconiosis; emphysema; crystalline silica associated with corundum deposits.
Arsenic	Cobaltite Enargite Realgar Orpiment Arsenopyrite Smaltite	Local skin and mucous irritant; carcinogen; anemia (hemolytic agent); hemoglobinuria; mixed dust pneumoconioses.
Beryllium	Beryl Chrysoberyl Bertrandite	Berylliosis; pulmonary lesions; acute poisoning; granuloma; pneumonitis; ore minerals are associated with crystalline silica.
Cadmium	Greenockite	Renal and pleural involvement.
Chromium	Chromite	Some reports of lung cancer; ore may be associated with chrysotile asbestos.
Cobalt	Smaltite Linnaeite Cobaltite Erythrite	Some reports of excess deaths due to lung cancer and cancer of the main bronchus; hard metal disease.
Iron	Hematite Magnetite Taconite	Siderosis; some reports of increased lung cancer among taconite ore miners.
Lead	Galena Cerussite Anglesite	Pneumoconiosis; diseases of the central nervous system; nephritis; 'plumbism'; anemia.
Manganese	Pyrolusite Braunite Manganite Hausmanite Rhodochrosite	Pneumoconiosis; affects the central nervous system (Parkinsonism syndrome); usually high rate of pneumonia among ore processors.
Mercury	Cinnabar	A range of systemic diseases; nephrosis (renal lesions); salivation; vertigo; paralysis; 'hatters shakes'; erethism; stomatitis; 'mercury poisoning'; perforation of nasal septum.
Nickel	Pentlandite Niccolite Millerite Garnierite	Pneumoconiosis; some observations indicate a higher than normal lung cancer rate; higher incidence of lung and nasopharynx cancer.
Phosphorus	Apatite	Minor scarring of lungs; some neurological disorders; phosphine and pesticide forms toxic-to-lethal.
Platinum	Platinum Sperrylite	Some lung scarring observed which resembles asbestosis.
Selenium	Tiemannite Guanajuatite Clausthalite Naumannite Eucairite Chalcomenite	Pneumoconiosis; severe irritation of nose and eyes; gastrointestinal disorders; dental caries.
Silver	Silver Acanthite	Argyria-discoloration of the skin to a pale gray or blue-gray.

continued

Table 3.2 continued

Metal	*Mineral*	*Comments*
Tellurium	Montanite Emmonsite Durdenite Tetradymite	Pneumoconioses; some lung scarring; gastrointestinal disorders; renal disorders.
Tin	Cassiterite	Severe X-ray changes in miners; tin pneumoconiosis.
Titanium	Rutile Sphene Ilmenite	Severe lung scarring, titaniosis; lung scarring;
Tungsten	Wolframite Tungstite	Lung scarring; tungsten pneumoconiosis.
Uranium	Uraninite Carnotite Pitchblende Thorium ores Vanadium ores	Excess lung cancers.
Vanadium	Vanadinite Carnotite	Lung scarring; respiratory irritant; irritant to eyes; increased susceptibility to pneumonia.
Zinc	Sphalerite	Shortness of breath; some minor lung changes; pneumoconiosis.

be observed compared with systems that sample and average exposures over a full work shift. Where discretion is allowed, some occupational hygienists use their professional judgement to remove the samplers or otherwise stop the sampling and discount certain non-operational portions of a work shift, such as during the lunch hour.

Sampling location, which has a significant impact on the measured dust level, varies in different countries. In German mines, respirable dust is measured at the location of a working area in which the maximum dust concentration is expected. In British coal mines, dust samples are collected at a fixed point, located in the return airway approximately 70 m behind the face. In the former Soviet Union, dust with particle-sizes up to 74 μm are collected at a fixed point, determined by the type of mining, generally located near the cutting machine. In US coal mines, respirable dust is collected using personal samplers, attached to the 'high risk' miners on the working section. Since these measurement locations vary as noted, exposure limits and reported dust levels cannot be directly compared between these countries (Prinz and Stolz, 1990).

The sampling frequency and the statistical treatment of measured concentrations can also affect the reported dust values. For example, when partial-period sequential samples are collected, there are two accepted practices for dealing with the unsampled portions of the shifts. Compliance officers in the US may be required to assume the unsampled periods have 'zero' exposure when computing the 8-hour time-weighted average. However, if the data are reported from sources not related to regulatory compliance sampling, the occupational hygienist might well have decided to assume that the average concentration measured during the partial-shift period, extended during the balance of the shift. This is a reasonable assumption when the work operation continued uninterrupted during these unsampled time intervals. In some instances, data handling differences may not be noted with the reported data, making direct comparisons difficult or impossible.

DUST CONTROL

Control of dust exposure follows some common themes regardless of industry. Exposure control can be achieved by:

- substitution of less toxic materials in the process;
- suppression of dust generation;
- capture of the particles at their source;
- separating the dust generation source from the worker;
- diluting the concentration with supplied fresh air; and
- using personal protective equipment.

These various techniques for exposure control have been well described elsewhere (Talty, 1988; Burgess, 1994).

Water has often been used to suppress dust generation. Wetting down roadways, using water sprays on cutting machines, industrial drills and other devices where material is mechanically broken or ground are common dust control measures. Where material is drilled or cut, using sharpened bits can often reduce the amount of dust generated in the process. Good maintenance of cutting tools provides an easy means of dust control, preventing dust from being generated.

Perhaps the most common techniques for dust control involve ventilation systems (ACGIH, 1995b). By providing exhaust air movement away from the workers at sufficient velocity, small particles entrained in the air can be exhausted safely where no one is exposed. Barriers containing dusty operations, such as sandblasting booths, glove boxes or brattice curtains and permanent stoppings in mines, serve to separate the workers from the dust generation sources. Generally less effective are systems that supply copious amounts of fresh air to dilute the concentration of dust. Due to the high costs of air conditioning or heating the large quantities of fresh air that is needed for adequate dilution ventilation, this method is not frequently used.

When the use of engineering controls is not possible, or during times when the control systems are being installed, maintained or repaired, respiratory protective equipment may be used. This type of personal protective equipment may include: supplied air respirators, self-contained breathing apparatus, powered-air filter respirators, cartridge filter particulate respirators and disposable (paper mask) type respirators. These each have operational limits that should be researched before recommending an appropriate unit for a particular application.

REFERENCES

ACGIH (1986) *Documentation of the Threshold Limit Values and Biological Exposure Indices*, Fifth edition, American Conference of Governmental Industrial Hygienists (ACGIH), Cincinnati, Ohio, pp. 523–5.

ACGIH (1995a) *1995–1996 Threshold Limit Values (TLVs®) for Chemical Substances and Physical Agents and Biological Exposure Indices (BEIs®)*, American Conference of Governmental Industrial Hygienists (ACGIH), Cincinnati, Ohio.

ACGIH (1995b) *Industrial Ventilation: A Manual of Recommended Practice*, 22nd edition. American Conference of Governmental Industrial Hygienists (ACGIH), Cincinnati, Ohio.

Armbruster, L. and Breuer, H. (1982) Investigations into defining inhalable dust. *Ann. Occup. Hyg.* **26**(1–4), 21–32.

Ayer, H.E. (1969) The proposed ACGIH mass limits for quartz: review and evaluation. *Am. Ind. Hyg. Assoc. J.*, **30**, 117–25.

Ayer, H.E. (1995) Origin of the U.S. respirable mass silica standard. *Appl. Occup. Environ. Hyg.*, **10**(12), 1027–30.

Ayer, H.E., Sutton, G.W. and Davis, I.H. (1968) Size-selective gravimetric sampling in dusty industries. *Am. Ind. Hyg. Assoc. J.*, **29**, 336–41.

Bartley, D.L. and Breuer, G.M. (1982) Analysis and optimization of the performance of the 10 mm cyclone. *Am. Ind. Hyg. Assoc. J.*, **43**, 520–8.

Bartley, D.L. and Doemeny, L.J. (1986) Critique of 1985 ACGIH report on particle size-selective sampling in the workplace. *Am. Ind. Hyg. Assoc. J.*, **47**(8), 443–7.

Bhaskar, R., Li, J. and Xu, L. (1994) A comparative study of particle size dependency of IR and XRD methods for quartz analysis. *Am. Ind. Hyg. Assoc. J.*, **55**(7), 605–9.

Burgess, W.A. (1994) Philosphy and management of engineering control, in *Patty's Industrial Hygiene and Toxicology*, 3rd edn, Volume 3, Part A, (eds R.L. Harris, L.J. Cralley and L.V. Cralley), John Wiley & Sons, Inc., New York, pp. 129–71.

Caplan, K.J., Doemeny, L.J. and Sorenson, S.D. (1977a) Performance characteristics of the 10 mm cyclone respirable mass sampler: Part I – monodisperse studies. *Am. Ind. Hyg. Assoc. J.*, **38**, 83–95.

Caplan, K.J., Doemeny, L.J. and Sorenson, S.D. (1977b) Performance characteristics of the 10 mm cyclone respirable mass sampler: Part II – coal dust studies. *Am. Ind. Hyg. Assoc. J.*, **38**, 162–73.

Castranova, V., Dalal, N.S. and Vallyathan, V. (1996) Role of surface free radicals in the pathogenicity of silicosis, in *Silica and Silica-Induced Lung Diseases* (eds V. Castranova, V. Vallyathan and W.E. Wallace). CRC Press, Inc., New York, pp. 91–105.

CEN (1993) Comité Européen de Normalisation. Standard on workplace atmospheres. Size fraction definitions for the measurement of airborne particles in the workplace, EN 481. Brussels, Belgium.

Chamberlain, E.A.C., Makower, A.D. and Walton, W.H. (1971) New gravimetric dust standards and sampling procedures for British coal mines, in *Inhaled Particles III*, (ed. W.H. Walton), Unwin Brothers Ltd., Vol. 2, Old Woking, UK, pp. 1015–30.

Chan, T.L. and Lippmann, M. (1980) Experimental measurements and empirical modelling of the regional deposition of inhaled particles in humans. *Am. Ind. Hyg. Assoc. J.*, **41**, 399–409.

Corn, M., Breysse, P., Hall, T., Chen, G., Risby, T. and Swift, D.L. (1985) A critique of MSHA procedures for determination of permissible respirable coal mine dust containing free silica. *Am. Ind. Hyg. Assoc. J.*, **46**, 4–8.

Costello, J. and Graham, W.G.B. (1988) Vermont Granite workers' mortality study. *Am. J. Ind. Med.*, **13**, 483–97.

Edwards, G.H. (1965) Comparison of X-ray diffraction, chemical (phosphoric acid) and dispersion staining methods of determination of quartz in dust. *Am. Ind. Hyg. Assoc. J.*, **26**, 532–6.

EPA (1995) Environmental Protection Agency. Code of Federal Regulations: Protection of Environment, Title 40: Parts 50 and 53, U.S. Government Printing Office, Washington, DC.

Ettinger, H.J., Partridge, J.E. and Royer, G.W. (1970) Calibration of two-stage air samplers. *Am. Ind. Hyg. Assoc. J.*, **31**, 537–45.

Fay, J.W.J. and Ashford, J.R. (1964) Environmental exposures of British coal miners. *Arch. Environ. Health*, **9**, 144–59.

Fay, J.W.J. and Rae, S. (1959) The pneumoconiosis field research of the National Coal Board. *Assoc. Occ. Hyg.*, **1**, 149–61.

Friedlander, S.K. (1977) *Smoke, Dust and Haze*, John Wiley and Sons, New York.

Glenn, R.E. and Kraft, B. (1986) Air sampling of particulates. In *Occupational Respiratory Diseases* (eds J.A. Merchant, B.A. Boehlecke, G. Taylor and M. Pickett-Harner. DHHS (NIOSH) Publication No. 86–102, Superintendent of Documents, Washington, DC, pp. 69–82.

Hall, F.E., Kupel, R.E. and Harris, R.L., Jr (1965) Particle settling times in ethyl alcohol–water mixtures as affected by variables in impinger sampling. *Am. Ind. Hyg. Assoc. J.*, **26**, 537–43.

Halley, P.D. (1946) Are dust counts made by different laboratories comparable? *Ind. Hyg. Quarterly*, **7** (2), 15–22.

Hearl, F.J. (1996) Guidelines and limits for occupational exposure to crystalline silica, in *Silica and Silica-Induced Lung Diseases* (eds V. Castranova, V. Vallyathan, and W.E. Wallace), CRC Press, Inc., New York, pp. 15–22.

Hinds, W.C. (1984) Sampler efficiencies: inspirable mass fraction. *Ann. Am. Conf. Ind. Hyg.*, **11**, 67–74.

ISO (1981) International Organization for Standardization: Size Definitions for Particle Sampling: Recommendations of Ad Hoc Working Group Appointed by Committee TC 146 of the International Standards Organization. *Am. Ind. Hyg. Assoc. J.*, **42**, A64–8.

ISO (1991) International Organization for Standardization: Air Quality – Particle Size Fraction Definitions for Health-Related Sampling, CD 7708, Geneva.

Jacobsen, M., Rae, S., Walton W.H. and Rogan, J.M. (1971) The relation between pneumoconiosis and dust-exposure in British coal mines, in *Inhaled Particles III*, Vol. 2, (ed. W.H. Walton), Unwin Bros., Old Woking, UK.

Jacobson, M., Terry, S.L. and Ambrosia, A. (1970) Evaluation of some parameters affecting the collection and analysis of midget impinger samples. *Am. Ind. Hyg. Assoc. J.*, **31**, 442–5.

John, W. (1984) Sampler efficiencies: thoracic mass fraction. *Ann. Am. Conf. Ind. Hyg.*, **11**, 75–9.

Kashdan, E.R., Madhav B.A.R., Purdue, L.J. and Rehme, K.A. (1986) Interlaboratory evaluation of two inlets of sampling particles less than 10 μm. *Environ. Sci. Technol.*, **20**(9), 911–16.

Knuth, R.H. (1969) Recalibration of size-selective samplers. *Am. Ind. Hyg. Assoc. J.*, **30**, 379–85.

Langer, A. (1986) Characterization and measurement of the industrial environment: Mineralogy, in *Occupational Respiratory Disease*, (eds J.A. Merchand, B.A. Boehlecek, G. Taylor and M. Pickett-Harner), DHHS (NIOSH) Publication No. 86–102, U.S. Government Printing Office, Washington, DC.

Lidén, G. and Kenny, L.C. (1994) Errors in inhalable dust sampling for particles exceeding 100 μm. *Ann. Occup. Hyg.*, **38**(4), 373–84.

Lioy, P.J., Lippmann, M. and Phalen, R.E. (1984) Rationale for particle size-selective air sampling. *Ann. Am. Confl. Ind. Hyg.*, **11**, 27–34.

Lippmann, M. (1970) 'Respirable' dust sampling. *Am. Ind. Hyg. Assoc. J.*, **31**, 138–159.

Lippmann, M. (1983) Size-selective health hazard sampling, in *Air sampling Instruments*, (eds P.J. Lioy, and M.J.Y. Lioy), 6th edn, ACGIH, Cincinnati, Ohio, pp. H1–H22.

Liu, B.Y.H. and Pui, D.Y.H. (1986) Aerosol sampling and sampling inlets, in *Aerosols – Research, Risk Assessment and Control Strategies*, (eds S.D. Lee, T. Schneider, L.D. Grant and P.J. Verkerk), Lewis Publishers, Inc., Chelsea, Michigan, pp. 175–83.

Lorberau, C. (1989) *Identification of the Determination of Respirable Quartz on Filter Media Using Fourier Transform Infrared Spectrophotometry*, National Technical Information Service, Springfiedl, Virgine, PB90–130105.

Mark, D. and Vincent, J.H. (1986) A new personal sampler for airborne total dust in workplaces. *Ann. Occup. Hyg.*, **30**(1), 89–102.

Mark, D., Lyons, C.P., Upton, S.L. and Kenny, L.C.(1994) Wind tunnel testing of the sampling efficiency of personal inhalable aerosol samplers. *J. Aerosol Sci.*, **25**(Suppl.1), S339–40.

Marple, V.A., Robow, K.L. and Tao, Y. (1991) A personal diesel exhaust sampler, in *Third Symposium on Respirable Dust in the Mineral Industries*, (eds R.L. Frantz and R.V. Romani) Society for Mining Metallurgy and Exploration, Inc., Littleton, Colorado, pp. 73–91.

McCawley, M.A. (1984) Performance considerations for size-selective samplers. *Ann. Am. Conf. Ind. Hyg.*, **11**, 97–100.

McCawley, M.A. and Cocalis, J. (1986) Diesel particulate measurement techniques for use with ventilation control strategies in underground coal mines. *Ann. Am. Conf. Ind. Hyg.* **14**, 271–81.

Morrow, P.E. (1964) Evaluation of inhalation hazards based upon the respirable dust concept and the philosophy and application of selective sampling. *Am. Ind. Hyg. Assoc. J.*, **25**, 213–36.

Morrow, P.E., Muhle, H. and Mermelstein, R. (1991) Chronic inhalation study findings as a basis for proposing a new occupational dust exposure limit. *J. Am. Coll. Tox.*, **10**(2), 279–89.

MSHA (1995) Mine Safety and Health Administrations. Code of Federal Regulations: 'Mineral Resources', Title 30: Parts 56, 57, 70, 71 and 75, US Government Printing Office, Washington, DC.

NIOSH (1983) Collaborative Tests of Two Methods for Determining Free Silica in Airborne Dust. DHHS (NIOSH) Publication No. 83–124 (NIOSH Publications Dissemination, 4676 Columbia Parkway, Cincinnati, OH 45226).

NIOSH (1994a) *Manual of Analytical Methods, 4th edition. Method 7400 – Asbestos and Other Fibers by PCM*, Revised 08/15/94. DHHS (NIOSH) Publication No. 94–113 (NIOSH Publications Dissemination, 4676 Columbia Parkway, Cincinnati, OH 45226).

NIOSH (1994b) *Manual of Analytical Methods, 4th edition. Method 0600 – Particulates Not Otherwise Regulated, Respirable*, Revised 08/15/94. DHHS (NIOSH) Publication No. 94–113 (NIOSH Publications Dissemination, 4676 Columbia Parkway, Cincinnati, OH 45226).

NIOSH (1994c) *Manual of Analytical Methods, 3rd edition. Method 5506 – Polynuclear Aromatic Hydrocarbons by HPLC*, Revised 08/15/94. DHHS (NIOSH) Publication No. 94–113 (NIOSH Publications Dissemination, 4676 Columbia Parkway, Cincinnati, OH 45226).

NRC (1980) Measurement and Control of Respirable Dust in Mines. Report of the Committee on Measurement and Control of Respirable Dust, National Materials Advisory Board, Commission on Socioetechnical Systems, National Research Council. NMAB-363, National Academy of Sciences, Washington, DC.

Orenstein, A.J. (1960) *Proceedings of the Pneumoconiosis Conference, Johannesburg, 1959*, Little, Brown & Co., Boston.

OSHA (1995) *Occupational Safety and Health Administration. Code of Federal Regulations: 'Labor'*, Title 29: Parts 1000.1043, US Government Printing Office, Washington, DC.

Phalen, R.F. (1984) Particle size-selective sampling in the workplace – Report of the ACGIH Technical Committee on air sampling procedures – Introduction and Recommendations. *Ann. Am. Conf. Ind. Hyg.*, **11**, 23–6.

Prinz, B. and Stolz, R. (1990) Effect of the measuring strategy on the determination of the respirable dust concentration in the breathable air at underground workplaces, in *Proceedings, VIIth International Pneumoconiosis Conference*, Pittsburgh, Pennsylvania. DHHS(NIOSH)-Publication No. 90–108, Part II, pp. 1148–1151.

Raabe, O.G. (1984) Size-selective sampling criteria for thoracic and respirable mass fractions. *Ann. Am. Conf. Ind. Hyg.*, **11**, 53–65.

Roach, S.A. (1953) Relating the incidence of pneumoconiosis to airborne dust exposure. *Br. J. Ind. Med.*, **10**(4), 220–6.

Roach, S.A. (1959) Measuring dust exposure with thermal precipitator in collieries and foundries. *Br. J. Ind. Med.*, **16**(2), 104–22.

Soderholm, S.C. (1984) Size-selective sampling criteria for inspirable mass fraction. *Ann. Am. Conf. Ind. Hyg.*, **11**, 47–52.

Soderholm, S.C. (1989) Proposed international conventions for particle size-selective sampling. *Ann. Occup. Hyg.*, **33**(3), 301–20.

Soderholm, S.C. (1995) Particle and gas phase interactions in air sampling, in *Air Sampling Instruments*, American Conference of Governmental Industrial Hygienists, Inc., Cincinnati, Ohio, Chapter 4, pp 67–80.

Soderholm, S.C. and Ferron, G.A. (1992) Estimating effects of evaporation and condensation on volatile aerosols during inhalation exposures. *J. Aerosol Sci.*, **23**(3), 257–77.

Soderholm, S.C. and McCawley, M.A. (1990) Should dust samplers mimic human lung deposition? *Appl. Occup. Environ. Hyg.*, **5**(12), 829–35.

Stahlhoffen, W., Gebhart, J. and Heyder, J. (1980) Experimental determination of the regional deposition of aerosol particles in the human respiratory tract. *Am. Ind. Hyg. Assoc. J.*, **41**, 385–98.

Stuart, B.O., Lioy, P.J. and Phalen, R.F. (1984) Use of size-selection in establishing TLVs. *Ann. Am. Conf. Ind. Hyg.*, **11**, 85–96.

Talty, J.T. (1988) *Industrial Hygiene Engineering: Recognition, Measurement, Evaluation and Control*, 2nd edn. Noyes Data Corporation, Park Ridge, New Jersey.

Talvitie, N.A. (1951) Determination of quartz in presence of silicates using phosphoric acid. *Anal. Chem.*, **23**(4), 623–6.

Talvitie, N.A. (1964) Determination of free silica: gravimetric and spectrophotometric procedures applicable to air-borne and settled dust. *Am. Ind. Hyg. Assoc. J.*, **25**, 169–78.

Talvitie, N.A. and Hyslop, F. (1958) Colorimetric determination of siliceous atmospheric contaminants. *Ind. Hyg. J.*, **19**, 54–8.

Tomb, T.F. and Raymond, L.D. (1970) Evaluation of the penetration characteristics of a horizontal plate elutriator and of a 10-mm nylon cyclone elutriator. US Bureau of Mines Report of Investigations No. USBM-RI-7367.

Tomb, T.F. and Treaftis, H.N. (1976) Review of published experimental calibrations performed on the 10 millimeter nylon cyclone. Mining Enforcement and Safety Administration, Informational Report 1040.

Tomb, T.F., Treaftis, H.N., Mundell, R.L. and Parobeck, P.S. (1973) Comparison of respirable dust concentration measured with MRE and modified personal gravimetric sampling equipment. US Bureau of Mines Report of Investigation, USBM-RI-7772.

US Code (1977) Federal Mine Safety and Health Act of 1977: 30 U.S.C. §§ 801–961 (1971 and Supp. 1978); Public Law 91–173 as amended by Public Law 95–164. (Federal Coal Mine Health Act of 1969 [PL 91–173] amended by the Federal Mine Safety and Health Amendments Act of 1977 [PL 95–164], Superintendent of Documents, Government Printing Office, Stock 024–019–00023–3, Washington, DC.

Verma, D.K., Sebestyen, A., Julian, J.A. and Muir, D.C.F. (1992) A comparison of two methods of sampling and analysis for crystalline silica α-quartz. *Appl. Occup. Environ. Hyg.*, **7**(11), 758–63.

Vincent, J.H. (1995) Progress towards implementation of new aerosol industrial hygiene standards, with special reference to the aluminium industry. *Sci. Total Environ.*, **163**, 3–9.

Vincent, J.H. and Armbruster, L. (1981) Short communication on the quatitative definition of the inhalablity of airborne dust. *Ann. Occup. Hyg.*, **24**(2), 245–8.

Vincent, J.H. and Mark, D. (1982) Applications of blunt sampler theory to the definition and measurement of inhalable dust. *Ann. Occup. Hyg.*, **26**(1–4), 3–19

Vinzents, P.S., Thomassen, Y. and Hetland, S. (1995) A method for establishing tentative occupational exposure limits for inhalable dust. *Ann. Occup. Hyg.*, **39**(6), 795–800.

Wu, Z., Hearl, F.J., Peng, K.L. *et al.* (1992) Current occupational exposures in Chinese iron and copper mines. *Appl. Occup. Environ. Hyg.*, **7**(11), 735–43.