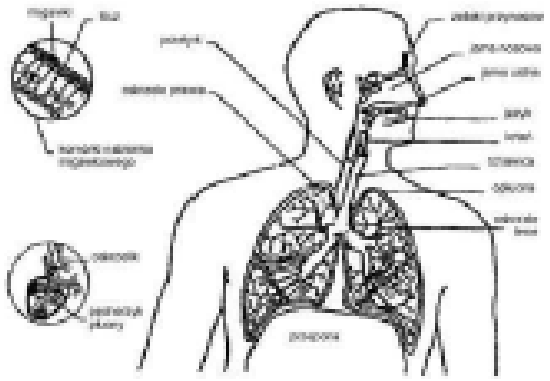
DETERMINATION OF TOTAL AND RESPIRABLE DUST USING FILTRATION – WEIGHING METHOD ON WORKSTATIONS



The purpose of this publication is to bring issues related to the determination of total and respirable dust in the workplace. The filtration-weighing method is presented with consideration of purely practical notions which are usually found in the use of this method. The scope of publications does not cover all issues relating to the problem – it more precisely shows the weighing process and potential sources of errors which may occur in this process.

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1. Introduction

Dust is collection of particles, which if thrown into the air remain there for some time. It is assumed, that these are particles with dimensions below 300 μm. Their shape depends on the source of dust. Particles of organic origin are shaped differently than the non-organic ones. A notion of particle as a single object is thus conventional. Size of a particle can be specified in certain intervals:

– very coarse particles /$d_a > 10\mu m$/
– coarse particles /$2{,}5\mu m < d_a \leq 10\mu m$/
– fine particles /$0{,}1\mu m < d_a \leq 2{,}5\mu m$/
– very fine particles /$d_a \leq 0{,}1\mu m$/

For the purpose of defining their size, below terms are employed:

- Substitutory diameter of a dust grain
  It is the diameter of a sphere with density equal to the density of tested dust, and which falling velocity in still air is equal to falling velocity of tested dust grain.

- Aerodynamic diameter of a dust grain
  It is density of a sphere with diameter 1g/cm3, and the same falling velocity in still air, as the falling velocity of tested dust grain;

- Projective diameter
  It is a diameter of a circle, which size is equal to the projection surface of a dust grain on observation surface.

From the hygienic point of view, dust is not a loose substance, but a biphasic system called aerosol. One of phases is the air (dispersing phase) and the other is the solid (dispersed phase). In reviewing the harmfulness of dusts, the **respirable** fraction is the most harmful one, as it reaches the vesicles. The measurement of respirable fractions is made by cyclone selectors, which separate respirable fractions from the other ones.

According to EN-481 regulation, there are another kinds of dust fractions defined:
- inspiratory fraction (passes through the mouth and nose)
- pulmonary fraction (dust reaches the bronchus)

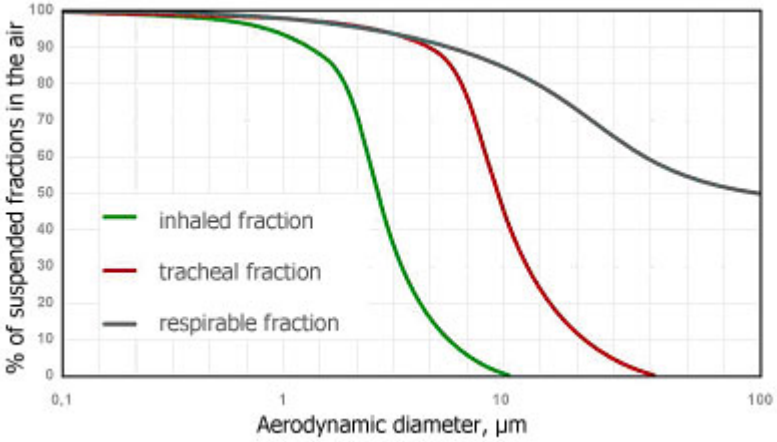

Fig.. 1. Dust division into fractions

2. Sources of dust and their effect on human organism

The surrounding air includes various particles, which are created in natural and artificial (technological) processes. It can be stated, that dust is created in:

– technological processes, which utilize dust as one of the components
– transport of loose materials
– production and packing of substances
– disintegrating and crushing of substances
– combustion of fuels
– agricultural works
– welding, cutting, and other material processing activities, etc.

Above statement is general in its character, and it does not name all sources generating dust in industrial environment.

The way dust influences human organism depends on dust concentration, its chemical composition and dimensions of particles. Generally speaking, inspiratory fractions are the ones with dimension below 100μm. The upper respiratory tract (nose, oral cavity, throat and larynx) blocks particles which are over 30μm, and they are excreted with mucus.

Pulmonary fraction, which dimension is up to 20μm, reaches the middle of respiratory tract (trachea, bronchus, bronchiole). The area of gas exchange (pulmonary alveolus) is infected by particles with dimension below 7μm. They create respirable dust which cumulates for a long period of time and causes pathological changes. The character of these changes depends on the dust structure that has been absorbed by the organism.

Among the dust, one can differentiate between: irritating, allergenic, cancerigenic, and fibrous. Another division specifies kind of dust that is source of pathology, i.e. asbestosis, silicosis, berylliosis, anthracosis, etc. When assessing risk degree of dust, exposure time is the main factor, and other important factors are:

- Concentration degree
- Size reduction
- Content of free silica in dust

The problem of air pollution id presently comprehended as a global one, which is reflected in the European directives
– no. 96/62/EC of 27 September 1996 on ambient air quality assessment and management.
– no. 1999/30/EC of 22 April 1999 relating to limit values for sulphur dioxide, nitrogen dioxide and oxides of nitrogen, particulate matter and lead in ambient air
– no. 2000/69/EC of 16 November 2000, relating to limit values for benzene and carbon monoxide in ambient air
– no. 2002/3/EC of 12 February2002 relating to ozone in ambient air
– no. 2004/107/EC relating to arsenic, cadmium, mercury, nickel and polycyclic aromatic hydrocarbons in ambient air.

3. Normative acts – testing methods

The problem of dust is so important that it requires constant monitoring, which is reflected in recommendations, standards and official documents. These should include:

- **PN-Z-04008-7:2002 + Az 1:2004**
  Air cleanness protection. Sampling. Rules governing the sample collection in the working environment & interpretation of results

  This standard gives rules for air sampling and interpretation of results to access occupational exposure to chemical substances and industrial dusts. They relate to individual dosimetry and stationary measurements.

- **PN-91/Z-04030/05**
  Air purity protection. Test for dust content. Matching total dust on workstations with filtration-weighing method

  This standard describes rules for methodology for matching the mass of dust sampled into the filter from determined air volume. It specifies the means of measurement with stationary and individual dust meters.

- **PN-91/Z-04030/06**
  Air purity protection. Test for dust content. Matching respirable dust on workstations with filtration-weighing method

  This standard describes rules for methodology for matching the mass of dust sampled into the filter from determined air volume. Respirable fraction is obtained through application of pre-selector during sampling. It specifies the means of measurement with stationary and individual dust meters.

In addition, there are other standards connected to this phenomenon, such as:

- PN-ISO 4225:1999 +Ak1 "Air quality. General terms. Terminology"
- PN-EN 1540:2004 "Air on workstations. Terminology"
- PN-ISO 7708 "Air quality. Dust fractions definitions used for sampling and health hazard assessment"
- PN-EN -13205:2004 "Air on workstations. Functionality assessment of measuring instruments for dust concentration in air"
- PN-EN -482:2009 "Air on workstations. General requirements for test procedures of chemical agents"
- PN-EN -689:2002 "Air on workstations. Guidelines for exposure assessment of inhalation of chemical agents by comparing the permissible values and measuring strategies"
- PN-91/Z -04222/02 "Air purity protection. Study of content of thermal black. Determination of thermal black on workstations using filtration-weighing method".

In the USA and in European Union the assessment of risk for dust is performed with filtration-weighing method. It has been assumed, that dust concentration should refer to the dimensions of the particles. This is the only way to determine the harmfulness effect of particles on human organism, due to the particles deposition on each part of respiratory tract.

3.1. Measuring instruments in use

Sampling in so called individual dosimetry requires application of below specified devices:

- **personal aspirator**
  it is to guarantee the constant flow of air with maximal deviation not bigger than 7% and the flow between 1 liter/min to 2 liters/min.

- **measuring indicator**
  for the mounting of a holder which contains a filter in the worker respiration area

- **cyclone selector**
  it provides proper flow intensity equaling 1,9 liter/min – separation of respirable dust from total dust

- **measuring filters**

they are manufactured of polipropylene microfibers or polivinyl chloride or other non-hygroscopic filters with filtering efficiency above 95%.

- **stop-watch**
- **analytical balance**
  with reading unit at least 0,01mg

- **exsiccator**

Special attention should be paid to personal aspirator, and test the air flow deviation before and after sampling.

$$\frac{V_{przed} - V_{po}}{V_{przed}} \cdot 100\% \leq 7\%$$

Vprzed – Vbefore
Vpo – Vafter

The other important component is the cyclone selector, which flow intensity has to ensure respirable dust uptake. If this condition is not performed, than the filter absorbs some intermediate phase. In such case, the measurement will focus on totally other fraction, and measuring results and conclusions will be incorrect.

In case of an analytical balance, it is recommended to do calibration in at least three points ranging from 10mg to 100mg. one of the points should equal 50mg. Recommended balance readability is:

- at least 0,05mg for total dust
- 0,01mg for respirable dust /0,05 mg is acceptable in case where dust mass on filter is bigger than 1mg/

3.2. Testing procedures

The principles for air sampling, storage and transport of filters are specified in corresponding documentation. It is a detailed description which allows for repeatable and reproducible performance of all activities. Standard no. PN-91-0430/05 for total dust specifies the possibility of sampling through stationary or personal dust meter. Independently on sampling method, the minimal dust mass on filter should equal at least 0,5mg and maximal concentration of dust on filter should be 0,5mg/cm2.

The measurement of respirable dust in accordance with PN-91 Z-04030/06 norm can be realized through stationary or individual dust meter. Sampling time has to guarantee the uptake of minimal sample mass onto the filter:

- 0,3mg, if an analytical balance with 0,01 mg reading unit is in use
- 1mg, if an analytical balance with 0,05 mg reading unit is in use.

Maximal sample mass on filter should not exceed 5mg.

Concentration of respirable dust in tested air is calculated according to a formula:

$$X = \frac{m_2 - m_1}{V} \cdot 1000$$

where:

X – respirable dust concentration [mg/m3]
m2 – filter mass after sampling [mg]
m1 – filter mass before sampling [mg]
V – air sample volume calculated as a product of volumetric intensity of uptaken air flow and sampling time [l]

All quoted standards and regulations, deal with a balances as with measuring instruments, but they do not specify the procedures for their checking. It can be, however, assumed, that all testing units are accredited, and they are operating in accordance to PN-EN ISO/IEC 17025 norm , and thus they have their own system of supervision over measuring instruments.

Unfortunately, none of the norms specifies the metrological characteristics of a balance with regard to its possible errors and sources for such errors. Total "*blank page*" is the description of sources which generate errors in case of electronic balances – special attention should be paid to this aspect. It is quite obvious here, as such recommendations can only be formulated by manufacturers of balances, and their experience.

Bearing in mind above mentioned issues, Sanitary-Epidemiologic Unit in Rzeszow province, laboratory section in Tarnobrzeg, and MRC company have organized the 1st Interlaboratory Research on filtration-weighing method, which aimed to:

- Determining characteristic features of filtration-weighing method in repeatable and reproducible conditions, where mass is the measurand.
- Checking the laboratory expertise on weighing analysis and filtration-weighing method.

The program was coordinated by M.Sc. Grazyna Czaderska (Sanitary-Epidemiologic Unit in Tarnobrzeg). For the purpose of the tests, MRC has created a procedure for filter weighing which guaranteed repeatability for each weighing process. The procedure also covered checking of the balance before and after the weighing process.

The procedure in such shape both presents and secures the operator against balance errors resulting from various drifts. This procedure does not take into account individual system solutions, but is provides a means of operation which may be accepted for repeatability assurance in filtration-weighing method. The descriptions in the procedure are general in their character, and thus they may be applied to most of electronic balances.

3.3. Procedure No.1: Weighing filters for laboratory tests

A. A balance used for testing filters should be plugged to mains at least 8 hours before the beginning of test procedure. It is assumed, that balances with "stand-by" function of the display, are ready to operate immediately on display activation. If a balance manufacturer recommends other warm-up periods, than they should taken as obligatory.

B. Balance should be checked before test procedure for its correct weighing parameters by placing a standard weight with mass relevant to the mass that is going to be measured. For measurements of contamination, it is assumed, that standard mass equaling 0,5 g or 1 g is sufficient. If balance indication is different from expected, perform adjustment process according to procedure described in balance documentation. On adjustment process completion, repeat checking of balance indications.

C. If balance adjustment does not cause any change of mass indication for standard weight, than such indications should be assumed as correct. The observed deviation between standard weight and balance indication is known as balance linearity error (on condition that mass of standard weight is correct – reliable).
Notes:
In case of differential weighing, it is very important to maintain repeatability of measurements and correctness of balance indications during complete testing procedure.

D. Before beginning of filter weighing procedure, weighing chamber of a balance should be closed. As the indication on balance display stabilizes, press ZERO/TARE button. It is necessary to set accurate zero indication of a balance.

E. Smoothly and gently open the balance weighing chamber, and place the filter in the centre of weighing pan. Do not exert any additional pressure on the weighing pan. As the filter is placed, do not correct its position on the weighing pan.
Notes:
The filter must not protrude beyond the edge of the weighing pan. Positioning the filter on the very centre of the weighing pan is not the most important factor in weighing process. It is more like a result of Good Laboratory Practice.

F. Close the weighing chamber of a balance and as the weighing result stabilizes, read the mass indication.
G. Open the weighing chamber of a balance and take the filter off the weighing pan.
H. As the balance returns to zero indication, set accurate zero point by pressing ZERO/TARE button.
I. Perform the second weighing of the same filter, and record its mass.
J. Calculate mass of a filter by taking average from performed measurements.

K. If long series of weighing are performed, than on completion of such series place a standard weight 0,5 g or 1 g on the weighing pan and compare the indication with the one from beginning of weighing process. Evaluate the drift of balance indication in time.
Notes:
Drift of balance indication is an objectionable factor, as by the operator it is interpreted increase or decrease of mass indication (depending on its direction). Thus, it is important to control this balance parameter. In practice, when evaluating drift,, one should also consider repeatability of a balance for specified load.

3.4. Personnel

Weighing seems to be an easy activity, thus is seems hard to define strict requirements for the personnel operating a balance. However, such simple activities as placing a filter on pan, read the indication, take the filter off, etc. are easy, but as practice shows, each user can perform them differently.
It starts from the approach to the issue:

- how to hold the filter?
- how to open the weighing chamber of a balance?
- when is the weighing result stable? How long to wait if weighing result is stable?
- how to react to balance indications if they are different from expected?
- adjust the balance or better not adjust it?
- check the balance before starting weighing process?
- work slowly for more precise indications? Or maybe work quickly?

There are multiple questions, and the answers are as plenty and variable. Generally speaking, an operator should be dependable; operator is to work precisely and rather quick with continuous observation of performed work. A good operator is a responsible person who can immediately evaluate measuring results, features of a tested object (filter) and a balance. Thus, it is expected, that an operator is experienced in:

- methods of collecting samples for tests,
- filter characteristics (its climatic features and stabilization in time),
- filter transporting (possibility of weight loss),
- weighing process (errors resulting from balance drifts, dispersion of indications)
- influence of ambient conditions on weighing process

Definitely, reaching the perfection is not possible, but it is recommended to extend one's knowledge and experience. However, it should be remembered, that one of the quality management system requirements is to self-improvement, which should refer not only to personal work but also to procedures.

Human factor in weighing process may have substantial importance, thus personnel should be carefully chosen for their activities.

4. Balances utiliezed in filtration-weighing method

The accuracy of balances is specified in norms. For total dust norm no PN-91 Z -04030/05 point 5 specifies balance accuracy to be at least 0,05 mg.
Respirable dust is described in norm PN-91 Z-04030/06 point 5 on apparatus, instruments and materials, which mentions semi-microbalance with weighing accuracy ±0,01mg. It also allows a balance with readability ±0,05 mg, if sample mass on a filter is at least 1 mg. According to practice, balances with readability 0,01 mg are commonly used for filtration-weighing method. The reason for application of such balances depends more on market supply than on user choice.

Specific norms related to monitoring of air pollution require **other solutions** on balances design. On one hand, norms focus on balance readability, but they do not specify other parameters, like repeatability or stabilization in time. These parameters are required for **differential weighing, which is applied for weighing dust**. On the other hand, most of balances used in filtration-weighing method have relatively high capacity ranging from 100 g to 200 g, and dust samples are much lighter. So, weighing process takes place at the very beginning of balance weighing range – it means that a balance is incorrectly suited for its application. Application of such balance results from market offer, which prefers so called universal weighing instruments for multiple applications..

Users of measuring instruments accepts this situation, as they want to have a kind of "universal device" for measuring multiple substances. Unfortunately, this is not s correct procedure. Each balance contains a mechanical set, and in case this set is loaded with heavy load (e.g. 150 g) and then with a light load (e.g. 80 g) than significant error is very likely to occur. Knowledge of above requirements is recommended for proper technical supervision over weighing instruments.

The other problem refers to pan size and design of weighing chamber (and influence of ambient conditions on weighing factors). Most important factor is the air movement in weighing chamber and its influence on weighing result.

4.1. Balances with readability d=10μg

In case of differential weighing, balance series **XA 52X** can be utilized. It is an analytical balance for operation in laboratory conditions. Series XA 52X is equipped with electromagnetic measuring system with DSP processor for impulse signal processing. Such design assures proper operation and maintaining very good repeatability and linearity parameters in full weighing range. Balance precise indications are continuously checked by system of automatic internal calibration.

Balance is equipped with detachable indicator. Such

solution significantly decreases any vibration transferred to weighing chamber and decreases total dimensions of the weighing instrument.
Weighing process documentation is realized through GLP procedures or through non-standard printouts.
Balance series XA 52X has type approval document and 3 years warranty period.

Design:

- Electromagnetic measuring system;
- Accuracy adjustment: automatic start-up calibration, defined according to time, temperature or manually;
- Detachable balance graphic indicator installed on a cable with possibility of placing next to the balance; digit height 20mm;
- 12 key membrane keyboard;
- PS/2 computer keyboard input;
- Balance weighing chamber dimensions 167mm x 161mm x 228mm (width x depth x height) with sliding side glass doors and openable top glass door;
- Under hook weighing
- Stainless steel weighing pan

Technical data

XA 52X	
Maximal capacity	52g
Reading unit	10µg
Repeatability	10µg
Working temperature	+ 18ºC / + 30 ºC
Stabilization time	5 sec
Pan size	85 mm

4.2. Balances with readability d=1µg

MYA 5/F is one of MRC microbalances designed for differential weighing of filters. Main freature of this microbalance is very good leakproofness. Such design provides very good repeatability even if the microbalance is used in unsatisfactory conditions with short weighing time. Weighing pan of MYA 5'F series is Φ100 mm, for weighing filters with considerable diameters.

Openwork surface of the weighing pan eliminates influence of air movement on

filters with small diameter Φ 20 – 50mm,
and provides stable support for filters with
big diameter.

Design:

- Electromagnetic measuring system
- Accuracy adjustment: automatic start-up calibration, defined according to time, temperature or manually.
- Detachable balance touch screen indicator (touch panel) 88 x 115mm with possibility of placing next to a balance; adjustable digit height;
- 8 key membrane keyboard
- System of user defined keys
- User defined infrared sensors
- Electronic level with alarm function
- PS/2 external computer keyboard input
- Aluminum weighing chamber with dimensions : Φ 115mm x 15m (depth.)
- Weighing pan construction – aluminum

Parametry techniczne

MYA 5/F	
Maximal capacity	5g
Reading unit	1µg
Repeatability	2µg
Working temperature	+ 18ºC / + 30 ºC
Stabilization time	10sec
Pan size	100mm

4.3. Errors which occur during differential weighing

Determination of dust content takes place by two times weighing of a filter. The first weighing is determination of mass of a clean filter
M_1 (without dust on a filter), the second one, is weighing a filter with dust content M_2 . As the dust mass is determined by ratio M_2 - M_1 , thus the weighing formula name is differential weighing. Differential weighing requires a balance to be stable in time, due to considerably long weighing procedure – mainly because of long period of filters stabilization time.

Errors that may occur during balance operation are:

- Weighing accuracy errors resulting from:
- drifts caused by changeable ambient temperature
- air blasts on workstation
- incorrect balance adjustment /performed in bad conditions/
- incorrect weighing procedure

- Zero point error resulting from:
- zero drifts caused by changeable ambient temperature
- incorrect weighing procedure

in practice, evaluation of above specified factors allows for determination and correction of these errors (or take them into account).

5. Report z I Interlaboratory Research on weighing filtration method

On 15-18 Spetember 2009 in Jantar, in Wiking resting/training centre, the I Interlaboratory Research on weighing-filtration method took place. The meeting was organized by Sanitary-Epidemiologic Unit in Rzeszow, Laboratory Unit in Tarnobrzeg, Auxiliary Farm of - Epidemiologic Unit in Rzeszow, Tarnobrzeg unit, and MRC Research and Development laboratory. The meeting was coordinated by M.Sc. Grazyna Czaderska (Sanitary-Epidemiologic Unit in Tarnobrzeg), MRC laboratory was represented by M.Sc. Sławomir Janas and Measuring laboratory by M.Sc. Andrzej Hantz.

The aim of the research was to:

- Learn binding regulations and norms on dust sampling procedures on workstations
- Determination of main error sources which occur during dust sampling and weighing
- Improvement of weighing process
- Learn the supervision processes for balances, standard masses and weights
- Presentation of principles for uncertainty evaluation and qualification process of filtration-weighing method
- Discussion on possibility of quality controlling during sample collection, its preparation and weighing analysis

The aim of proficiency testing program was:

- Determination of characteristic features of a weighing method in repeatability and reproducibility conditions, in measurement of mass.
- Checking laboratory proficiency in weighing analysis and in filtration-weighing menthod

Processes connected to measuring techniques were discussed during a lecture presented by MRC workers, and covered below issues:

1. Introduction to the problem of weighing
2. Metrological infrastucture in Poland and in Europe
3. Legal metrology in relation to weighing instruments
4. Supervision over weighing instruments and standard masses
5. Ambient conditions on workstations equipped with weighing instruments – practical aspects.

Problems referring to dust sampling techniques, and its aspects were presented by M.Sc. Andrzej Uzarczyk. The listeners of the lecture were presented with data on sources and characteristic features of dust, means of collecting air samples in individual dosimetry, evaluation of danger of total and respirable dust presence and finally calibration and checking flowmeters.

A separate part of the lecture was dedicated to phenomenon of uncertainty of testing methods (uncertainty budget during sample collection, uncertainty components, standard and extended uncertainty) and qualification process (repeatability, reproducibility, assessment of variance uniformity, working range, qualification process of air collection sampling for total and respirable dust assessment).

While testing, the participants performed real tests on air samples for total and respirable dust in accordance to
PN-91/Z-04030/05 and PN-91/Z-04030/06 norms. During tests, devices manufactured by Two-Med., SKC and Radiotechnika-Cassela were presented.

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