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Ambient air — Measurement of the mass of particulate matter on a filter medium — Beta-ray absorption method

Air ambiant — Mesurage de la masse des matières particulaires sur un milieu filtrant — Méthode par absorption de rayons bêta

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 10473 was prepared by Technical Committee ISO/TC 146, *Air quality*, Subcommittee SC 3, *Ambient atmospheres*.

Ambient air — Measurement of the mass of particulate matter on a filter medium — Beta-ray absorption method

1 Scope

This International Standard describes a method for the measurement of the mass of particulate matter in ambient air and is based on the absorption of beta rays by the particulate matter.

This method applies to the determination of concentrations ranging from a few micrograms per cubic metre to a few milligrams per cubic metre contained in the atmospheres of urban, rural or industrial areas.

The lower mass detection limit of the method is usually 15 µg to 30 µg of deposited mass per square centimetre of surface area, S, of the filter. This means, for a sampling time t of 3 h and a flowrate q of 1 m^3/h, that the concentration detection limit ranges between 5 µg/m^3 and 10 µg/m^3, computed as follows:

$$\text{Concentration}\left(\mu\text{g}/\text{cm}^2\right)\cdot\frac{S\,(\text{cm}^2)}{q\,(\text{m}^3/\text{h})}\cdot\frac{1}{t\,(\text{h})}$$

Sampling techniques are not included in the scope of this International Standard.

NOTE The concentration of particulate matter is calculated by dividing the mass deposited on a filter tape or individual filter, by the known volume of air sampled. However, concentration is dependent on the sampling technique used, for example, the design of the sampling inlet. Normally, for ambient-air particle sampling, large particles are filtered out by means of a size-selective inlet (for example cascade impactor or cyclone filtration). The particle size limit is defined by the characteristics of the sampling head.

2 Term and definition

For the purposes of this International Standard, the following term and definition applies.

2.1
beta ray
radiation emitted by electrons during the nuclear decay of radioactive elements

NOTE In this International Standard, elements such as ^{147}Pm, ^{14}C or ^{85}Kr may be used.

3 Principle

3.1 Description

A known volume of ambient air is drawn through a filter on which the particulate matter is collected. The total mass of the particulate matter is determined by the measurement of absorption of beta rays. This measurement follows the following empirical absorption law:

$$N = N_0 \cdot e^{-km} \qquad (1)$$

where

N_o is the number of incident electrons per unit of time (counts per second);

N is the number of electrons transmitted per unit of time (counts per second) measured after the filter;

k is the coefficient of absorption per unit of mass (cm^2/mg);

m is the areic mass (mg/cm^2) of matter encountered by the beta radiation.

Practically, it is not necessary to determine N_o, and the areic mass of the collected particulate matter is determined as follows.

a) Step one: a measurement is made on a blank filter:

$$N_1 = N_o \cdot e^{-km_o} \quad (2)$$

where

N_1 is the number of electrons transmitted per unit of time (counts per second) measured after the blank filter;

m_o is the areic mass (mg/cm^2) of the blank filter.

b) Step two: a measurement is made on the same filter loaded with particulate matter:

$$N_2 = N_o \cdot e^{-k(m_o + \Delta m)} \quad (3)$$

where

N_2 is the number of electrons transmitted per unit of time (counts per second) measured after the filter loaded with particulate matter;

Δm is the areic mass (mg/cm^2) of particulate matter collected on the filter.

Combining equations (2) and (3):

$$N_1 = N_2 \cdot e^{+k \Delta m} \quad (4)$$

or

$$\Delta m = \frac{1}{k} \ln \frac{[N_1]}{[N_2]} \quad (5)$$

This method of measurement has the following characteristics:

— the empirical exponential law [equation (1)] is valid in the practical working range. There is, however, an upper limit which is directly proportional to the maximum energy of the emission spectrum of the beta source used.

3.2 Limitations

The absorption law (1) can slightly depend on particle density and size for large particles (diameter more than 20 µm). To minimize this effect, large particles are usually filtered out by means of a cascade impactor in the sampling head.

Errors in mass determination can be caused by irregularities in the spatial distribution of the stream of beta electrons and heterogeneous deposits of particulate matter due to a deterioration of the sampling system.

Changes in atmospheric pressure and temperature cause the density of air between the source and the detector to change. This can affect the determination of the mass of particulate matter deposited on the filter. The error can be minimized by keeping the time between the measurement of N_1 and N_2 as short as possible and can be corrected by measuring the atmospheric pressure and temperature, or by use of a dual detection system; in this case, the result of the second measurement (N_2) has to be recorded continuously as mass accumulates over time.

The elemental and chemical composition of atmospheric particulate matter has a small effect on the value of the coefficient of absorption per unit of mass, k.

The effect of radioactivity in particulate matter is negligible for long-lived radioisotopes. However, in locations where low levels of radon and its daughters are present, the response of beta gauges may be affected. The error depends on the type of equipment used.

4 Apparatus

The apparatus should be installed in a room which is controlled for temperature and humidity.

The apparatus may be either automatic with sequential or simultaneous sampling and analysis, consisting of a single assembly, or automatic with separate sampling and analysis, consisting of two sub-assemblies, one for sampling the particulate matter and the other for measuring it.

Schematics of four typical apparatus that allow sequential or simultaneous sampling and analysis, using one or two beta gauges, are shown in Figure 1:

— automatic simultaneous sampler and analyser with 1 beta gauge [see Figure 1 a)];

— automatic sequential sampler and analyser with 2 beta gauges [see Figure 1 b)];

— automatic sequential sampler and analyser with 1 beta gauge [see Figure 1 c)];

— automatic sequential sampler and analyser with 1 beta gauge on separate filters [see Figure 1 d)].

Whichever mode is adopted, the apparatus includes the following main components.

4.1 Apparatus for sequential or simultaneous sampling and analysis

4.1.1 Sample inlet, also called sampling head, generally made of stainless steel, for sampling the particulate matter contained in ambient air. Its characteristics, in conjunction with sampling flowrate, determine the sampling efficiency.

The sampling head should be made from a material resistant to atmospheric corrosion.

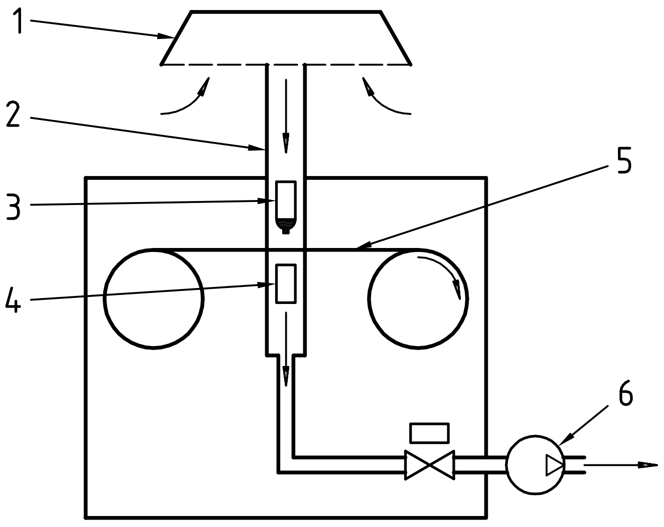
4.1.2 Sample intake tube, preferably straight, perpendicular to the filter, and made of stainless steel, intended to carry the sampled particulate matter to the filter.

It is essential that this tube be designed to prevent losses of particulate matter before reaching the filter. In addition, the tube shall be slightly heated (40 °C to 50 °C) in order to prevent any condensation on the filter. The internal cross-section of the tube and its outlet shall be equal to the exposed area of the filter.

4.1.3 Sealing device to prevent any leak between the lower end of the sample intake tube and the filter, thus preventing any loss of particulate matter and intake of air.

It may consist of a retractable nozzle which stays on the filter during sampling.

A permanent magnet shall not be used as a sealing device.

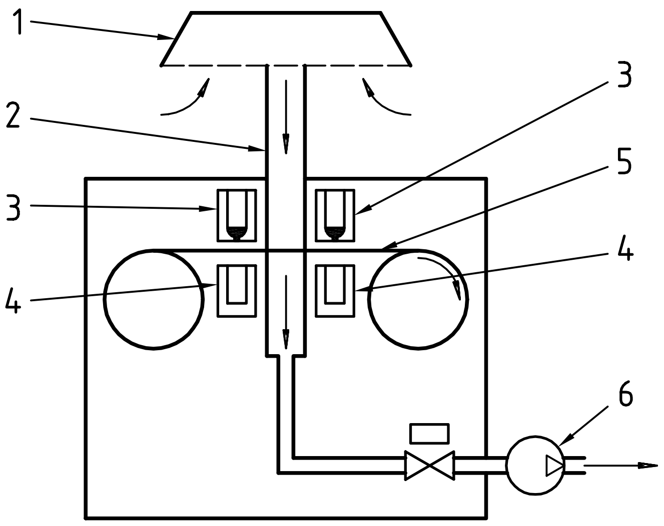


Key

1 Sampling head
2 Sample intake tube
3 Beta gauge emitter unit
4 Beta gauge receiver unit
5 Filter
6 Pump

NOTE The filter is measured before sampling with the pump off to determine the blank. During sampling the beta absorption is recorded. At the end of the sampling period, a new filter portion is then placed in position. In order to minimize the influences described in section 4 a dual detection system may be used.

a) Simultaneous sampler and analyser with one beta gauge

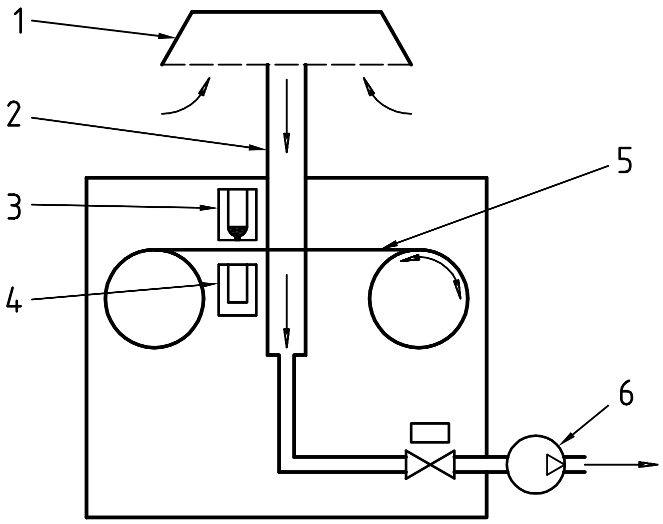


Key

1 Sampling head
2 Sample intake tube
3 Beta gauge emitter unit
4 Beta gauge receiver unit
5 Filter
6 Pump

NOTE The blank filter is measured by the first beta gauge unit. After the blank is determined, the filter passes through the sample collection area. Once sampling is completed, the filter is moved outside of the sampling area, and measured by the second beta gauge unit. The reel moves in only one direction.

b) Sequential sampler and analyser with two beta gauges

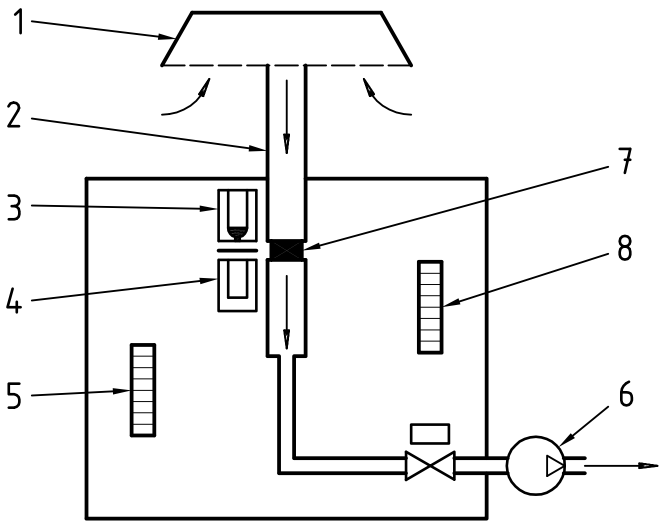


Key

1 Sampling head
2 Sample intake tube
3 Beta gauge emitter unit
4 Beta gauge receiver unit
5 Filter
6 Pump

NOTE The blank value for the filter is measured before sampling. The filter is then placed in the sampling section and after sampling is completed, measured again by the beta gauge. The filter reel moves in both directions.

c) Sequential sampler and analyser with one beta gauge



Key

1 Sampling head
2 Sample intake tube
3 Beta gauge emitter unit
4 Beta gauge receiver unit
5 Blank filters
6 Pump
7 Sampling step
8 Sampled filters

NOTE These instruments do not use reels. Rather, the filters are supported on suitable holders for transportation through the instrument and storage. The filters move back and forth to the beta gauge and to the sampling section in order to provide several measurements on the same filter. The filters can be removed for the analysis of specific compounds. Blanks and used filters are stacked in carriers.

d) Sequential sampler and analyser with one beta gauge on separate filters

Figure 1 — Typical automatic instruments for sequential or simultaneous sampling and analysis

4.1.4 Filter, on which the sampled particulate matter is collected.

The following parameters are important in the choice of the filter:

- minimum collection efficiency of 99 % for particle diameters above 1 µm;
- low resistance to air flow;
- low sensitivity to water vapour in the air;
- uniformity of areic mass and of mechanical and chemical properties;
- thermal stability;
- retention efficiency.

4.1.5 Filter holder

For apparatus using individual filters [see Figure 1 d)], each filter shall be supported by a filter holder having a diameter suited to that of the filter used.

The filter shall be protected during sampling against any deformation due to the pressure drop by close contact with a support grid. The grid shall be made from a corrosion free material. The grid shall have a smooth surface and a sufficiently small mesh to prevent any deterioration in the filter and to ensure a high degree of uniformity of the deposit of particulate matter.

The holder assembly can be heated slightly (40 °C to 50 °C) in order to prevent any condensation on the filter in the case of humid atmospheres.

The holder assembly should be designed to facilitate the operations of positioning and unloading the filter, and to ensure correct support of the filter.

4.1.6 Beta gauge, intended to measure the absorption of the beta rays by particulate matter, consisting of:

- **an emitter unit** made up of a sealed beta ray source emitting only soft rays, free from gamma radiation;
- **a receiver unit** made up of a beta ray detector, for example a high-sensitivity Geiger-Müller counter or a solid-state surface.

These two units shall be placed on each side of the filter.

The following precautions shall be taken to ensure the optimum geometry of the beta gauge:

- verification of the symmetrical distribution of the incident beta radiation;
- optimization of the distance between the emitter unit and the receiver unit, with respect to the count-rate.

The beta gauge shall be designed so that the measurement is always made on the same area of the filter before and after sampling.

The measurement of the absorption of the blank filter, prior to sampling, is extremely important. The area on which the deposit is made during sampling needs to be reproduced exactly. If the working surface is not returned to exactly the same position after exposure, substantial errors in the measurement of mass may occur.

4.1.7 Flowrate control device, to maintain the sampling rate within ± 5 % of the initial value by mean of electronic flow control.

4.1.8 Pump, placed at the end of the sampling train, after the beta gauge device, to draw the air sample through the whole sampling train.

Typically, the sampling flowrate is between 1 m^3/h and 3 m^3/h.

It is essential to check for the correct operation of the pump as well as the tightness of the whole sampling train. It is recommended that a pressure-drop indicator be set up between the pump and the filter.

4.1.9 **Flowrate meter** or **volume meter**

Either of these devices shall be used. The flowrate meter shall allow the reading of the air flowrate through the filter. The volume meter shall allow the measurement of the total volume of ambient air sampled in the case of a sequential mode.

Since the data from beta gauge instruments are the mass concentrations, it is essential to calibrate these devices periodically under normal operating conditions (temperature, pressure drop, etc.).

4.1.10 Electronic components, to allow the various automatic processes required for sampling and analysis to be carried out in accordance with the procedure described in clause 7.

They shall allow the total mass of particulate matter collected on the filter to be determined directly from the areic mass.

4.2 Apparatus for separate sampling and analysis

4.2.1 First sub-assembly for sampling and collection of particulate matter, containing all the components described in 4.1.

4.2.2 Second sub-assembly for measurement using absorption of beta rays, consisting of a beta gauge as described in 4.1.6, and the electronic components for controlling the mechanical operations and for processing measurements as described in 4.1.10.

5 Calibration of the beta gauge

5.1 Zero calibration and variability

5.1.1 General

This procedure allows the user to set the analysers to a true instrumental zero, i.e. on a blank filter without any particles, for each of the different instrument designs.

To provide a uniform procedure, disconnect the pump from the sampling unit in order to avoid air and particles entering the sampling section.

The following procedures are advised.

5.1.2 **Automatic simultaneous sampler and analyser with one beta gauge** [see Figure 1 a)]

5.1.2.1 Zero calibration

Run the sampler and record the signal output for 1 h. Calculate and report the mean and the standard deviation of the observed signal. Calibrate the zero setting.

5.1.2.2 Repeatability

On a regular basis, run the zero check on several sequential filter sections. The variability of the zero is expressed as the standard deviation of at least five filter sections running for at least 24 h each.

5.1.3 Automatic sequential sampler and analyser with two beta gauges [see Figure 1 b)]

5.1.3.1 Zero calibration

Programme the instrument for a sampling time as short as possible and record the signal output for at least ten measuring cycles. Calculate and report the mean and the standard deviation of the observations. Calibrate the zero setting.

5.1.3.2 Repeatability

In order to measure the zero variability, repeat the procedure for a longer sampling time.

5.1.4 Automatic sequential sampler and analyser with one beta gauge [see Figure 1 c)]

The same procedure as 6.2 is used.

5.1.5 Automatic sequential sampler and analyser with one beta gauge on separate filters [see Figure 1 d)]

5.1.5.1 Zero calibration

For instruments using individual filters, the zero procedure requires several measurements (at least ten) on the same filter for a sampling period as short as possible. Report the mean and the standard deviation of the observations.

5.1.5.2 Repeatability

The variability is measured by repeating the procedures over a longer sampling time.

Instruments for separate sampling and analysis can be zeroed by repeating the measurement on the same filter for the typical counting time. Repeat at least ten measurements and report the mean and the standard deviation.

NOTE It should be recalled that the pump is disconnected (no air or particles are sampled by the instrument). Repeated measurements on the same filter portion or the same filter will be distributed according to Poisson's distribution; in this case, the standard deviation is equal to the square root of the mean value of the number of the effected particles (counts). In order to verify that the Poisson's distribution is achieved, a direct access to the measurement of total counts is necessary.

5.2 Calibration

NOTE The procedures described do not describe the calibration of the sampling section, since they are common to any sampler of atmospheric particles.

5.2.1 Pre-weighed membrane method

The calibration of the beta gauge is generally performed by using a membrane (or sheet) of inert material [for example polycarbonate, aluminium, gold, etc. (see Figure 2)] of uniform areic mass, which is determined gravimetrically using a balance having an accuracy to within 0,01 mg. The sheet is placed upon the blank filter and a calibration factor is obtained, after dual measurements of the absorption of beta rays by the blank filter, alone and then with the sheet.

Instruments are directly calibrated by recording the signal obtained when the sheet is placed on the filter. The variability of the calibration can be checked by repeating several measurements. Express the variability in terms of the standard deviation of the observations.

Using several membranes of different areic mass allows a check of linearity.

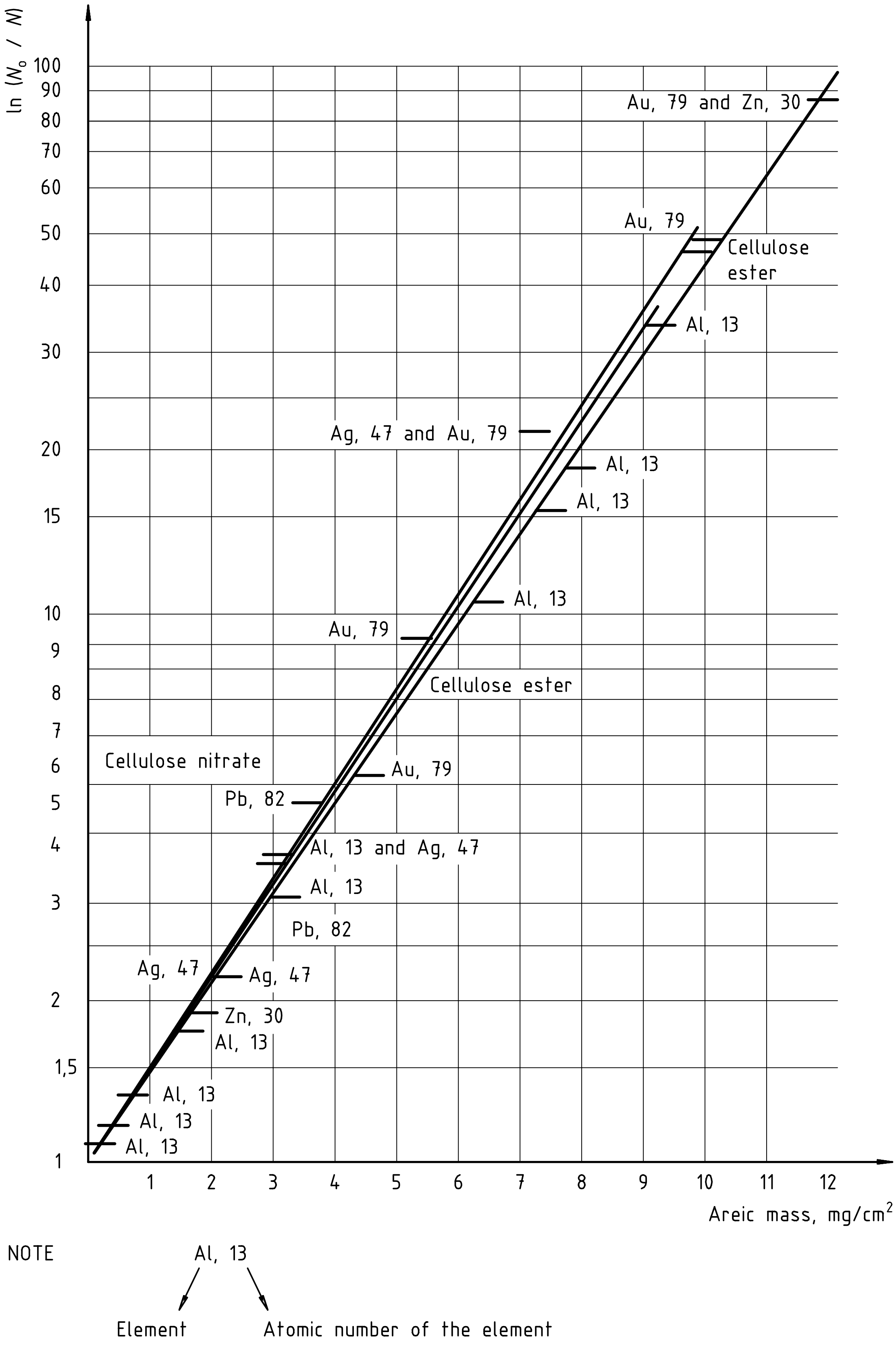


Figure 2 — Effect of calibration material on the exponential law for a ^{14}C source as a function of areic mass

5.2.2 Gravimetric method

Instruments and devices using a beta gauge can be also calibrated by weighing the deposit of the sampled particulate matter. At least five blank filters are weighed and measured as blanks in the instrument. Particles are then sampled on these filters in order to accumulate five different amounts of particulate matter. After sampling, the mass of the particulate matter sampled on the filters is measured gravimetrically and a calibration factor is obtained by a linear regression analysis between the gauge readings and gravimetric data.

For instruments using different types of filter, a series of measurements shall be performed in order to determine any variation in the calibration factor due to different filters. Since experimental observations demonstrate that the response of any beta gauge is dependent upon the type of membrane used, particular care should be paid to the expression of results.

6 Procedure

6.1 Sampling time

First select the sampling time. Choose this time so that the mass measured shall be as large as possible but shall not exceed a total mass which would reduce N_1 by more than 75 %.

6.2 Automatic apparatus for sequential or simultaneous sampling and analysis

Operate the apparatus described in 4.1 in a sequential or simultaneous manner as follows:

— measure the absorption of beta rays on the blank filter;

— select the sampling time;

— sample a known volume of air through the filter tape to collect the particulate matter;

— measure the absorption of beta rays by the filter tape loaded with the particulate matter either at the end of the sampling period (sequential) or during sampling (simultaneous);

— calculate the total mass using the calibration factor; calculate the concentration of particulate matter in the volume of air sampled.

6.3 Automatic apparatus for separate sampling and analysis

First, in the laboratory, assign an identification code to each blank filter. Using the second sub-assembly (see 4.2.2) located in the laboratory (or in the apparatus), measure the absorption of beta rays by each blank filter. Then load these filters in to the storage part of the sampling device.

Then draw a known volume of air through the filter with the sampling device, in order to sample and collect the particulate matter in a sequential manner.

Measure the absorption of beta rays on the loaded filters (returned to the laboratory, or in the apparatus), the original blank values are subtracted from the loaded filter values and the corresponding total mass of particulate matter is determined.

The problems caused by varying humidity are the same for the beta gauge measurement method as for direct microgravimetric methods. It is therefore necessary to take the usual precautions with regard to handling, conditioning, stabilization and drying of filters (such as air-conditioned room or box with controlled environmental conditions).

7 Expression of results

Record the mean values of the temperature T and pressure p during the sampling period.

The results of the measurements shall be expressed in micrograms per cubic metre or in milligrams per cubic metre by dividing the mass m of particles sampled (computed from the measured beta-ray absorption values) by the sample volume, corrected to standard temperature T_0 and pressure p_0.

8 Test report

The test report shall include the following information:

a) reference to this International Standard;

b) all details required for the complete identification of the samples;

c) the sampling method used;

d) the results;

e) details of any deviation from the procedure specified in this International Standard or any circumstances that may have influenced the result.

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