Development of conversion factors for results of early gravimetric dust measurements

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Abstract We performed 35 parallel measurements using historical (Double Cone DC and Gravikon WL 10/40) and modern (Gravikon VC 25) dust samplers in a wind tunnel as well as on-site in porcelain plants. It was the aim to establish a correlation between results of historic gravimetric dust measurements in the German porcelain industry before 1972 and data obtained with modern gravimeters. Grain size analysis of the historic dust samples was accomplished by the original sedimentation process. The sampling device factor, obtained from the different sampling characteristics of the devices, combined with the analytical scaling factor yielded a total conversion factor of 1.5. This factor has already been used as a multiplier to convert historic gravimetric data from 1959 to 1971 into respirable dust results in the course of an epidemiological study in the German porcelain industry. This approach should also be applied for epidemiological investigations in other industries.

Umrechnungsfaktoren für Messergebnisse von älteren gravimetrischen Staubmessungen

Zusammenfassung Es wurden 35 Parallelmessungen mit historischen („Doppelkegel" DC und Gravikon WL 10/40) und modernen (Gravikon VC 25) Staubmessgeräten in einem Staubkanal und vor Ort in Porzellanbetrieben durchgeführt. Ziel war es, eine Beziehung zwischen den Ergebnissen gravimetrischer Staubmessungen vor 1972 und den mit modernen Geräten erhaltenen Daten in der deutschen Porzellanindustrie herzustellen. Die Korngrößenanalyse der mit historischen Geräten gewonnenen Proben wurde mit der damals verwendeten Sedimentationsmethode durchgeführt. Der sich aufgrund unterschiedlicher Sammelcharakteristiken ergebende Gerätefaktor und der analytische Faktor ergeben zusammen einen Umrechnungsfaktor von 1,5. Dieser Faktor kam bereits im Rahmen einer epidemiologischen Studie in der deutschen Porzellanindustrie zur Umrechnung von gravimetrischen Daten der Jahre 1959 bis 1971 in A-Staub-Werte zur Anwendung. Dieser Ansatz sollte auch für epidemiologische Untersuchungen in anderen Industriezweigen genutzt werden.

1 Introduction

In the early years of monitoring airborne particles at the workplace many different methods were developed and tested to quantify dust exposure. Most of the techniques were based on particle counting (e.g. by a konimeter after sampling in an impinger), thermal diffusion, light scattering or on gravimetric determination of mass [1]. A generally accepted standard method, however, did not exist at that time.

The first devices for systematic gravimetric measurements of dust concentration were designed and used beginning in 1950 [2; 3]. In Germany, regular and routinely performed gravimetric sampling started approximately in 1959 [4 to 7]. It was not until 1971 that a standard method for gravimetric dust measurements was implemented; this method is still in use today [8 to 10]. The driving forces behind this process were the prevention departments of the Berufsgenossenschaften (BG, German statutory accident insurance institutions), and the Staubforschungs-Institut (Dust research institute, now: Institute for Occupational Safety and Health of the German Social Accident Insurance (IFA)) which is a comprehensive service unit for the BG. In 1972 the exposure database MEGA was created containing at present more than 100,000 measurement data for respirable dust in various sectors of industry.

Unfortunately, the results for the "fine dust" fraction (the term "A-fraction" has been widely used in Germany) obtained by the various gravimetric methods and devices before the introduction of the European Standard EN 481 [11] cannot be compared directly because of the changing sampling and analytical techniques. Nevertheless, a comparison and comprehensive synthesis of measurement results over a long period of time is necessary for a variety of analytical purposes. This synthesis is necessary, for example, to evaluate the effectiveness of dust control measures in certain plants and industries, or to obtain a reliable job-exposure matrix in the course of epidemiological studies. Presently, the European Standard EN 481 and the nearly identical International Standard ISO 7708 [12] define the reference sampling conventions used for the respirable dust fraction for silica. Historical exposure results have to be transformed based on these standards [13].

The present paper describes the parallel dust measurement results obtained by historic and modern sampling devices commonly used to monitor industrial dust in the German porcelain industry. On the basis of these experiments, we established a conversion algorithm which allows the combination of diverse gravimetric measurement data for a period of nearly 50 years. With these factors it is possible to convert exposure data for respirable dust as well as for respirable quartz dust, over different periods of time, and thus put them into relation with each other. The results of this study have been used to establish a quantitative silica exposure assessment for a historical cohort epidemiological study in the German porcelain industry, described in a separate paper by *Birk* et al. [14].

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Figure 1. Double Cone (DC) sampling unit in use from1959 until approximately 1971.

Figure 2. Gravikon WL 10/40 sampling unit in use from 1969 until approximately 1975.

2 Methods

2.1 Dust sampling devices for gravimetric dust determination

The use of gravimetric devices for sampling and quantitative determination of dust at workplaces in the German porcelain industry was first documented in 1959 as reviewed by *Blasum* and *Claus* [7] and by *Winkel* [15]. None of these measurements were conducted by individual companies. Rather, all were obtained and analyzed by industrial hygiene experts from the institution for statutory accident insurance and prevention in the ceramics and glass industry (Berufsgenossenschaft der keramischen und Glas-Industrie in Würzburg, BGGK, since 2009: Verwaltungs-Berufsgenossenschaft, VBG). The measurement files reveal the use of two different sampling devices to monitor dust in the porcelain and ceramics industry between 1959 and 1975. Fortunately, some of the original gravimeters remained on the premises of the BGGK, enabling us to perform parallel measurements with historical and modern devices.

The first gravimetric measurements were conducted using a double-cone shaped sampling unit (DC) which was in use in the ceramics industry until the late 1960's (**Figure 1**). During a period from 1969 until about 1971 the DC was gradually replaced by the Gravikon WL 10/40 with a sampling head consisting of an annular gap (**Figure 2**), already very similar to the VC 25 G device used today for the sampling of inhalable dust. Both historical devices were developed and manufactured by Heinz Wertebach Mechanik und Feinkonstruktionen, Bonn, Germany. The airborne dust was collected with both devices on a Microsorban filter suspended on a metal screen within the sampling head. The filter material was provided by Delbag Luftfiltergesellschaft, Berlin, Germany. The air flow of the pump was controlled manually by a gas meter [16].

Since 1973 the device VC 25 F was established as a measurement instrument to collect respirable dust according to the Johannesburg convention and EN 481 [9; 10]. The separation of the respirable dust fraction takes place during sampling by filtration in combination with impaction: fine particles, following the radially redirected air flow within the head of the sampler are deposited on a concentric area of the filter by filtration; coarser particles are impacted on the central part of the filter and either stay there impacted or are reflected to the outer rim of the filter. Tests have verified that about 50% of the respirable fraction is collected on a defined concentric region of the filter which is then cut out for the following analysis. The deposited mass of respirable dust can be calculated after weighing (the blank mass of the cut out filter ring is calculated from the relation of its surface compared with the total filter surface). Usually the mass of dust is determined by

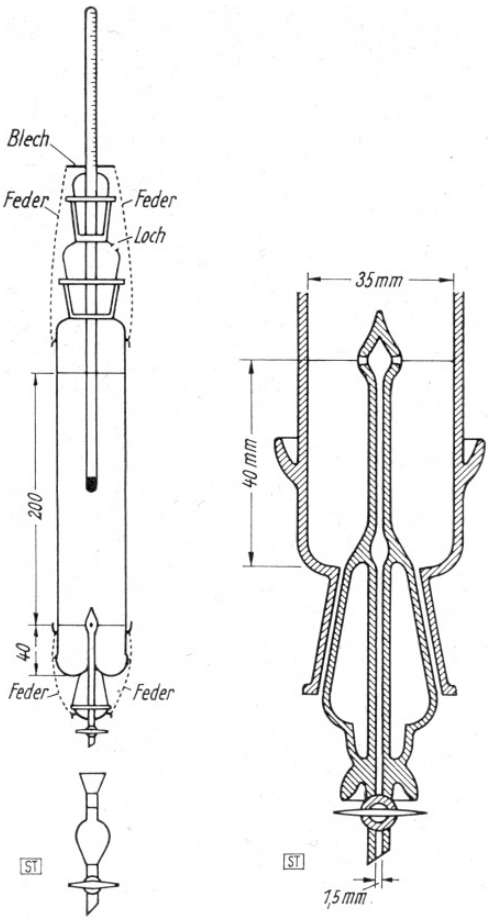


Figure 3. Section drawing of the sedimentation apparatus used in the Andreasen pipette method to separate the respirable fraction from collected dust, as described by *Schmidt*.

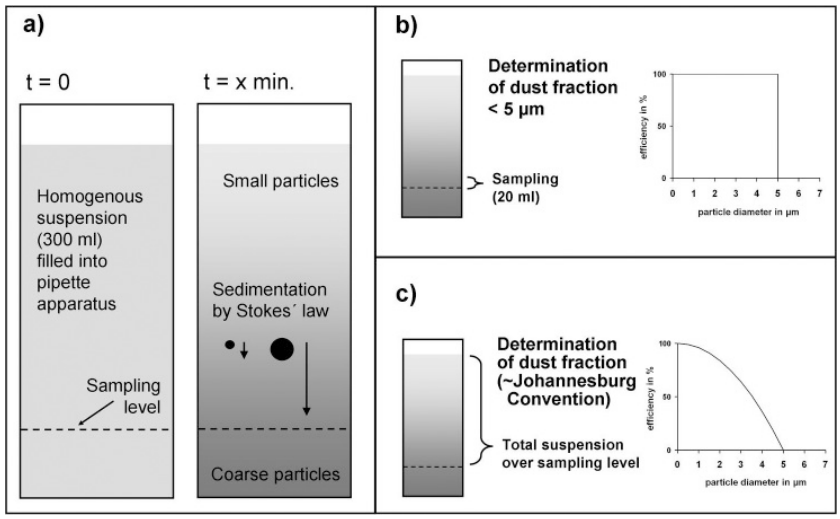


Figure 4. Simplified scheme of sedimentation analysis (pipette method by *Andreasen,* see Figure 3). a): A homogeneous suspension of dust is filled into the pipette apparatus. After a defined time of gravimetric sedimentation, coarser particles sink down faster than smaller particles. Particles larger than a specific diameter (e.g. 5 µm, calculated for quartz, specific gravity 2.65 g/cm³) sink down below the sampling level. b) and c): Different sampling methods of the suspension in the pipette apparatus after sedimentation yield different dust fractions.

beta-absorption. Tests with dust loaded filters from different industries confirmed that the absorption of beta-radiation shows a linear correlation with the dust mass; this correlation is independent from the kind of dust within acceptable statistical variation.

2.2 Analytical determination of the respirable fraction

When sampling dust at workplaces by use of the DC or Gravicon WL 10/40 the respirable fraction was still part of the collected dust and was not directly available after separation. The collected dust was historically called "total dust" and is different from the inhalable dust fraction collected using modern devices (see Section 3). After dust collection using the DC and WL 10/40, the polystyrene Microsorban filter was dissolved in an organic solvent (TRI, trichloroethene) and the resulting suspension then filtered by suction. The particles collected by this process were subsequently suspended in an aqueous sodium pyrophosphate solution ($Na_4P_2O_7$) of well-specified concentration and density. The respirable dust fraction was determined by dispersion of a defined amount of collected dust and subsequent isolation by sedimentation analysis, the Andreasen pipette method described by *Schmidt* [17]. The fluid is added to the pipette apparatus (**Figure 3**). After a given time, particles with the density of quartz (2.65 g/cm³) and coarser than 5 µm in diameter sink down a defined distance by gravitational sedimentation (see **Figure 4**a). Historically, two different analytical processes have been applied, depending on the intended result. The mass of dust obtained by separating a limited volume of suspension (20 ml exactly at the sampling level) and subsequent filtration represents the fraction < 5 µm (see Figure 4b). This fraction was used as the respirable dust fraction before the Johannesburg convention was established in Germany in 1971 [18].

As a second possibility, the complete suspension above the sampling level can be used for separation and gravimetrical analysis. The particle distribution of the dust in this liquid represents the respirable fraction with regard to the Johannesburg convention specifically (Figure 4c). For practical analytical reasons this procedure was also used to obtain the dust fraction for the analytical determination of quartz. Historically, the amount of quartz was determined by the phosphoric acid method [19; 20] and, beginning at about 1962, by X-ray diffraction and later also by IR spectroscopy. The concentration of quartz was reported in relation to the dust fraction < 5 µm.

Coenen [8] compared the different dust fractions used before and after 1971 and calculated factors to compare the results based on different conventions. The concentrations of respirable dust and quartz (determined before 1971) can be compared to exposure data with regard to the Johannesburg convention by an analytical scaling factor of 0.5 (**Figure 5**). But it must be stressed that this scaling factor does not take into consideration the different sampling efficiencies of the various sampling instruments used (see Section 2.3).

Besides the VC 25 F, other sampling instruments like the MPG II (Wazau, Berlin) were developed which directly fulfill the Johannesburg convention. Personal air samplers like FSP-BIA and FSP-10 (cyclones with flow rate of 2 l/min and 10 l/min) also fulfill the requirements for sampling of respirable dust according to EN 481.

For our investigations we weighed the dust in compliance with ISO 15767 [21]. A detailed description of the procedure and the quality aspects of the determination of dust as proceeded in Germany are given by *Hebisch* et al. [22].

Skilled personnel who knew the methods from their own practical experience performed the complete sedimentation procedures as prescribed by *Andreasen*. Figure 4c lays out the methods used in these experiments for the determination of fine dust.

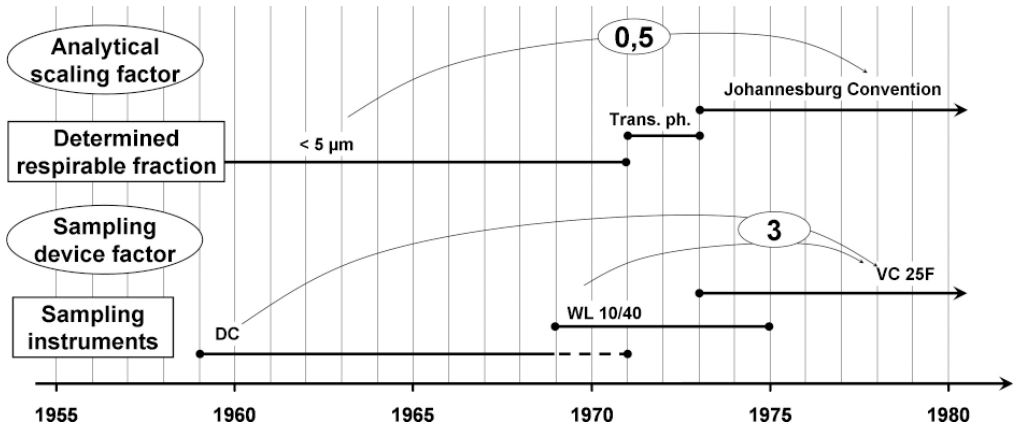


Figure 5. Timeline showing the use of different sampling instruments and different separation methods for respirable dust in Germany. The documented factors allow conversion of results obtained during the early years (for details see text; Trans. ph.: transition phase with varying methods).

2.3 Evaluation of sampling device factors

A total of 35 parallel measurements were carried out using the two "historic" sampling devices side by side with their successor model, VC 25 F. We performed eight parallel runs using the whole set of the three devices on-site in a porcelain plant and in a plant processing raw materials for the porcelain industry (**Figure 6**), and 27 parallel runs (17 runs: DC, WL 10/40 and VC 25 F; 10 runs: only WL 10/40 and VC 25 F simultaneously) in the wind tunnel of the Institute for Research on Hazardous Substances (Institut für Gefahrstoff-Forschung, IGF) at Dortmund, Germany (**Figure 7**).

The parameters of the sampling procedure described in 1962 by *Blasum* and *Claus* for the first gravimetric samples are very similar to the present specifications: flow rate of the

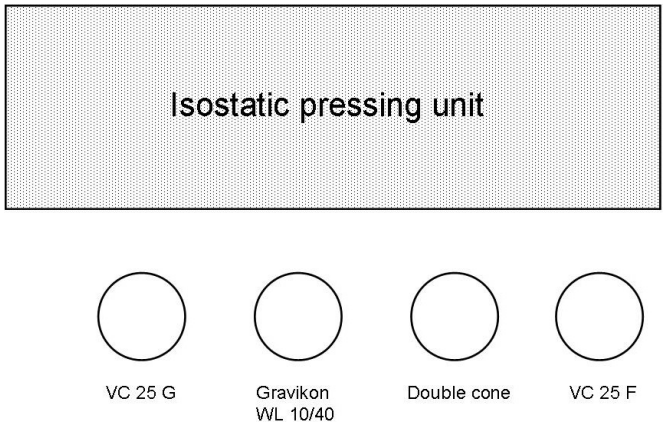
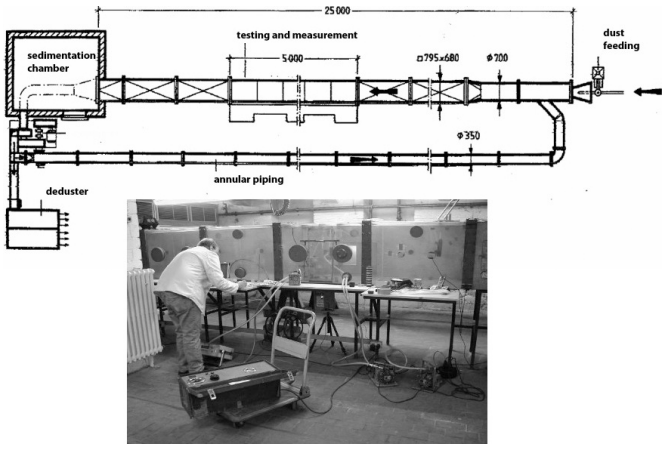


Figure 6. Left: On-site sampling with historic and modern sampling devices in a porcelain plant next to an isostatic pressing unit for plates. Right: Arrangement of sampling devices next to an isostatic pressing unit during on-site sampling.



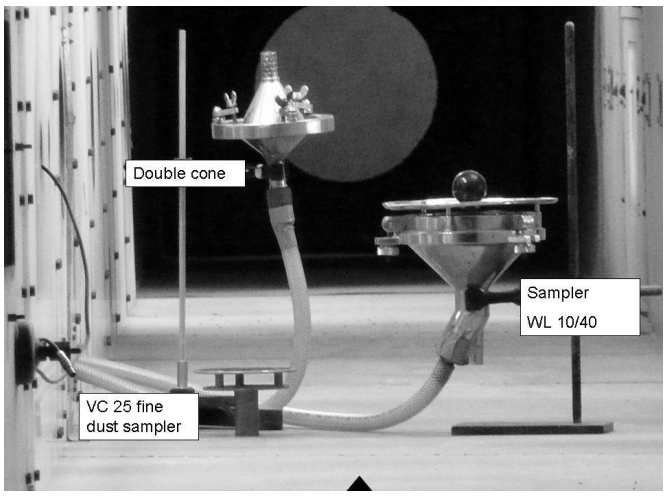


Figure 7. Left: Schematic design in the wind tunnel of the Institute for Research on Hazardous Substances (IGF). Right: Arrangement of sampling devices inside the wind tunnel of the IGF (line of sight along the direction of the diffusion of dust).

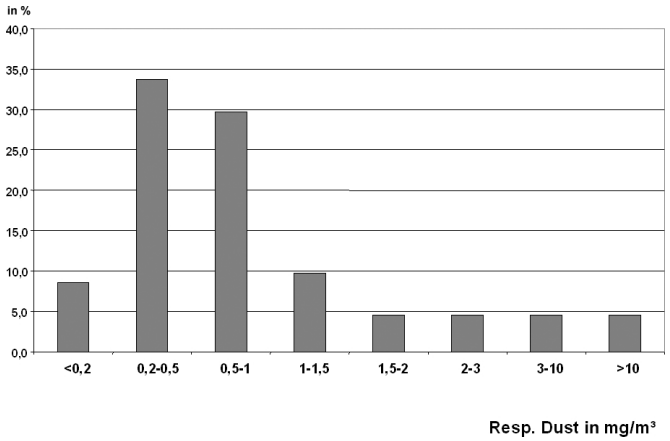


Figure 8. Distribution of results of respirable dust measurements in the years from 1959 to 1968 (original data).

pump was 20 m³/h compared to 22.5 m³/h today and sampling time 2 to 3 h compared to 2 h minimum today [7]. A comparison of the two different sets of parameters in preliminary experiments showed no significant difference between collected dust masses. Therefore, we applied the modern parameters as a standard for all parallel runs on-site and in the wind tunnel.

The on-site samples were taken in a modern porcelain factory in the forming department (isostatic pressing: two samples (Figure 6); dry fettling of shaped articles: one sample) and in the raw material preparation area (weighing of powdered materials and filling mixers: two samples). Another set of measurements was performed in a plant producing raw materials for the porcelain industry in the dry milling, in the screening and in the bagging department (one sample each).

For the measurements in the wind tunnel of the IGF (Figure 7) we collected dust samples from the dust extraction units of the materials preparation and the forming department of the porcelain factory exactly from the same workplaces where the on-site sampling was conducted.

The generation of airborne particles in the wind tunnel was controlled in such a way that the concentration of airborne respirable dust covered roughly a range from 0.1 to 4 mg/m³, thus matching roughly the set of original unconverted data generated in the early years from 1959 to 1968 (**Figure 8**). In contrast, the levels of airborne respirable dust during on-site sampling in the modern factories were rather low, ranging from 0.5 down to 0.035 mg/m³ due to modern industrial hygiene standards.

3 Results

Tables 1 and **2** show the results of the parallel measurements of the Double Cone (DC) and the WL 10/40, side by side with the modern VC 25 F device. The sampling device factor (last column in Tables 1 and 2) was calculated by dividing the measurement result of the VC 25 F by the corresponding result of the Double Cone (Table 1) or WL 10/40 (Table 2), as obtained by the sedimentation procedure described above (see Figure 4 c).

The results in Table 1 and 2 are displayed graphically in **Figure 9** and **10** respectively. For the Gravikon WL 10/40, the

Table 1. Results of parallel measurements for Double Cone (DC) and VC 25 F; VC 25 F operating according to the Johannesburg Convention and EN 481.

Measuring point	VC 25 F (respirable dust c_1 in mg/m³)	Double cone (fine dust by sedimentation c_2 in mg/m³)	Sampling device factor c_1/c_2
Wind tunnel	5.84	2.88	2.03
	6.63	3.25	2.04
	6.63	3.38	1.96
	6.63	2.99	2.22
	5.84	1.84	3.17
	5.84	1.87	3.12
	1.29	0.63	2.03
	2.01	0.51	3.97
	3.71	0.43	8.65
	1.09	0.15	7.14
	1.36	0.18	7.51
	1.27	0.13	9.48
	0.66	0.12	5.74
	0.68	0.06	10.63
	0.25	0.02	10.97
	0.25	0.02	14.18
	0.32	0.06	5.43
On site	0.44	0.20	2.20
	0.86	0.16	5.49
	0.17	0.10	1.77
	0.27	0.05	5.40
	0.93	0.24	3.96
	0.18	0.11	1.64
	0.11	0.14	0.79
	0.13	0.04	3.76

Table 2. Results of parallel measurements for Gravikon WL 10/40 and VC 25 F; VC 25 F operating according to the Johannesburg Convention and EN 481.

Measuring point	VC 25 F (respirable dust c_1 in mg/m³)	WL 10/40 (fine dust by sedimentation c_2 in mg/m³)	Sampling device factor c_1/c_2
Wind tunnel	1.07	0.30	3.57
	1.62	0.23	7.04
	1.39	0.29	4.79
	6.39	2.80	2.28
	7.61	2.78	2.74
	6.63	2.99	2.22
	6.63	3.28	2.02
	6.63	4.18	1.59
	5.90	3.32	1.78
	5.84	4.22	1.38
	5.84	1.63	3.58
	6.88	1.95	3.53
	5.68	1.43	3.97
	5.84	2.74	2.13
	6.38	1.69	3.78
	5.48	1.21	4.53
	1.29	0.83	1.56
	2.01	1.31	1.54
	3.71	1.50	2.48
	1.09	0.54	2.02
	1.36	0.67	2.03
	1.27	0.64	1.98
	0.66	0.29	2.24
	0.66	0.09	7.57
	0.68	0.22	3.03
	0.25	0.10	2.64
	0.25	0.07	3.39
On site	0.44	0.20	2.20
	0.86	0.17	5.16
	0.17	0.13	1.31
	0.27	0.10	2.70
	0.93	0.51	1.82
	0.18	0.09	2.00
	0.11	0.10	1.10
	0.13	0.08	1.63

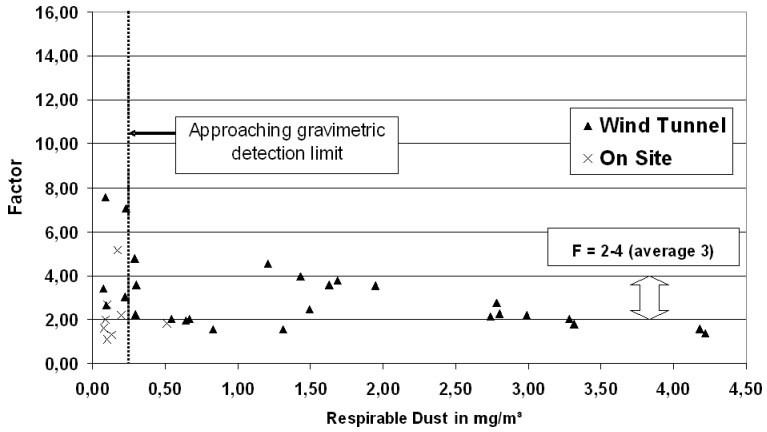


Figure 9. Sampling device factor (= c_{resp}[VC 25F]/c_{resp}[WL 10/40]) for different concentrations of respirable dust for the Gravikon WL 10/40.

variation of data is rather low. For airborne concentrations of respirable dust below approximately 0.5 mg/m³, the scattering increases to a level that precludes precise estimation of a sampling device factor. This can be explained by the fact that the sampling devices are approaching the gravimetric detection limit under the experimental conditions.

The sampling device factor of the Gravikon WL 10/40 exhibits a range of approximately 2 to 4 (Figure 9), with values averaging about 3 for concentrations ranging from 0.5 to 2 mg/m³, approaching 2 at respirable dust concentrations exceeding 2.5 mg/m³, and possibly falling slightly below 2 above 4 mg/m³.

For the DC sampling unit versus VC 25 F, a distinctly higher variation in the sampling device factor is observed. Because of the particular geometry of the

aspiration port of the DC, the air flow into the sampling head is poorly defined. Therefore, it is much more difficult to maintain identical parameters for all selected sampling conditions. Additionally for the range below 0.5 mg/m³, the same restrictions as for the WL 10/40 apply. Nevertheless, the sampling device factor for the DC also covers roughly a range from 2 to 4 (Figure 10), remaining at about 2 for concentrations over 2 mg/m³.

Because most of the measurement results (approximately 81%) in the respective time period were in a range of 0.2 to 3.0 mg/m³ respirable dust, we concluded that for both the WL 10/40 and DC, an average sampling device factor of 3 may be reasonable and – especially considering the lower concentration ranges – a conservative estimate. However, this factor applies to the sampling procedure only and therefore is referred to as the sampling device factor.

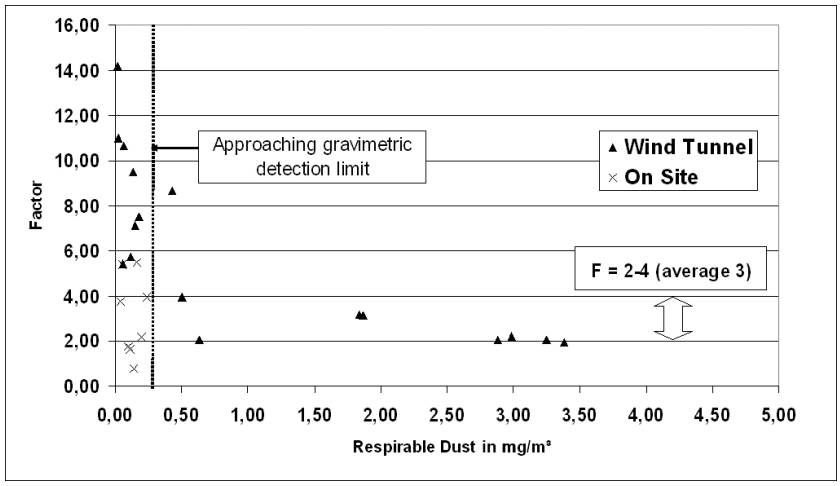


Figure 10. Sampling device factor (= c_{resp}[VC 25F]/c_{resp}[Double Cone]) for different concentrations of respirable dust for the double cone device.

These results are supported by some cursory measurements of total dust collected by the DC and the Gravikon WL 10/40 operated side by side with the VC 25 G, the standard sampling instrument for inhalable dust today.

4 Discussion

The historical equipment was operated as originally intended, and we do not assume significant errors in our experiments due to deviations from the historical operation procedures. Therefore, the results can be regarded as representative for those conditions.

Although the historic devices were not designed to sample specific health-related dust fractions, the results of the sedimentation analysis, always employed in conjunction with the samplers and using the actually collected dust, show good correlation to directly-determined respirable dust obtained by instrumentation working according to EN 481.

The wind tunnel experiments were well-suited to compare the historical samplers to modern equipment at dust concentrations above 0.5 mg/m³. Below that level, the limit of detection of the gravimetrical determination generated a poor correlation.

Therefore, an important conclusion of our experiments is that it is actually not necessary to exactly describe the separation efficiencies of the historic equipment in the case we studied. The historical samplers were simply not designed to perform any of the separation functions required by EN 481. However, the dust collected does indeed contain the complete respirable fraction, as shown in our experiments.

As long as this can be guaranteed and subsequent analytical investigation using a suitable sedimentation method is conducted, it is not necessary to exactly describe the separation function of those samplers. Of course the results of the sedimentation analysis do not translate into respirable dust directly. Instead, a sampling device factor can be determined from our experiments.

In order to convert historical exposure data generated before 1971 to modern respirable dust or quartz values, a recalculation process must be applied which takes into account both the influence of the sampling procedure and the historical analytical process. An analytical scaling factor of 0.5 can be obtained from the literature [8]. Taking into account the sampling procedures – which are associated with a sampling device factor of roughly 3 – we suggest as a convention a total conversion factor of 1.5 as a multiplier for the historical exposure data.

Thus, it is possible to convert the existing gravimetric data from 1959 to 1971 into respirable dust results. This method was performed for the epidemiological study published by *Birk* et al. [14]. Exposure data from 1974 to the present day do not need conversion as they have been obtained by methods directly compatible with EN 481 and other modern procedures. For the short transitory period between 1971 and 1973, where both methodological approaches were employed, we suggest a linear interpolation procedure already applied for the study mentioned above. In epidemiological studies this conversion process is necessary to obtain an appropriate and comprehensive description of the exposure situation, as can be shown in the case of the porcelain industry. A direct use of the uncorrected data generated before 1971 would lead to a severe underestimation of historic exposure. Without this correction, the steady improvements in working conditions and average exposure in the porcelain industry since the 1960s would not be apparent.

5 Conclusions

The recalculation procedure presented here is applicable for exposure data obtained by the DC and the WL 10/40 as dust samplers in conjunction with sedimentation analysis by the *Andreasen* pipette method as described above. It should be noted that this procedure cannot be used for other combinations of sampling and analytical procedures without modification. Nevertheless, researchers can apply the conversion process described in this paper to the roughly 5,000 exposure data generated in Germany between 1959 and 1969 by the DC and the WL 10/40, mainly in the ceramics, construction, mineral industries and foundries areas.

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