

ON-SITE TESTING OF FILTERS WITH SPECIAL REFERENCE TO SOLID AEROSOLS

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Abstract—A lithium flame photometer test is proposed for on-site testing of filter installations. The lithium test is intercalibrated with the standard Sodium Flame Test. The spraying time needed to introduce the lithium salt into a ventilation system is considered inconvenient for systems handling more than 10^6 m³/hr. For such systems photoelectric particle counters can be used with the dust naturally present in the local atmosphere. Where the dust level is too low, Aitken nuclei can easily be artificially generated in sufficient amounts. The relation between filter evaluation by such particle counting and by lithium measurements is established.

INTRODUCTION

The on-site testing of installed filters presents problems which are generally absent from the techniques involved in the laboratory testing of filter elements.

In the laboratory a small filter element or system can be treated as a single unit and the test set-up arranged so that the efficiency of the unit appears in the results as the ratio between truly representative samples of particle concentrations taken before and after the test unit.

In on-site testing, the physical layout might make it impossible to carry out determinations equivalent to those on the laboratory scale. Usually large filter banks are dealt with, containing one or more dust filters and the same number of absolute filters. These filter banks are installed to accommodate one or more large ventilation channels servicing an entire building section. The air flow in these channels is of the order of thousands to tens of thousands of cubic metres per hour.

It is the intention here to discuss some of the laboratory methods available for measuring the effectiveness of filters and to show how one of these methods can be adapted to accommodate the on-site evaluation of filter installations.

DISCUSSION OF SOME STANDARD LABORATORY FILTER TESTS IN RELATION TO ON-SITE TESTING

An estimation of the performance of a highly efficient filter is made on the basis of an experi-

ment in which a cloud of particles in the micron range, moving at a certain velocity, is passed through the filter. The ratio between the concentrations of particles in the air before and after the filter is then determined.

The experimental techniques discussed here involve the use of an inhomogeneous test cloud consisting of solid particles with an approximate mean size of about 0.5 micron. Examples of the employment of an inhomogeneous test cloud are the two British Standard tests, the Methylene Blue Test⁽¹⁾ and the later developed Sodium Flame Test.⁽²⁾ Both tests involve an atomizer which delivers a spray from a solution of methylene blue or sodium chloride. The spray is dried in an evaporation tube, thus forming a particulate cloud with particles of about 0.5 micron.

A common feature of the Methylene Blue and the Sodium Flame Test is that the attainment of reliable results depends on a most careful control of the atomizer air-flow system.

In the Methylene Blue Test a subjective comparison is made of the stain intensities on two filter papers sampling the air on either side of the test filter. The air sample from before the filter is obtained within a few seconds by means of a small hand pump, while the collection of an air sample from behind the filter takes about half an hour for highly efficient test filters. For a test filter with a penetrability of 0.2% the unfiltered volume of methylene-blue cloud may have to be about 12 ml (drawn evenly with a

hand pump) as compared with 60 l. of the filtered cloud, so that the two stain intensities lie close together for reliable comparison. In a comparable situation the lithium-flame test requires one or two litres as a sufficient sample. The lithium test⁽³⁾ is furthermore free of subjective approximations, and the lithium amounts sampled before and after the test filter are measured quite independently of each other.

The Sodium Flame Test requires an even more carefully controlled atomizer air-flow system as no air sample is taken before the filter, the efficiency of the latter being evaluated only by measurement of the concentration of sodium in the air after the filter. The measurement is made by means of a specially designed flame photometer that can analyse a continuous air stream. The instrument has to be calibrated every time it is to be used, by allowing a diluted sample of the unfiltered test gas to enter the flame. In an on-site test, the required calibration equipment, which may involve the use of two flow meters and a mixing bottle for each decade by which the unfiltered test gas is diluted, is very inconvenient. In some, not uncommon, cases the section before a filter bank is in fact isolated from the effluent side by the wall in which the filter bank has been installed. In such cases it may prove impossible to connect the calibration equipment to the flame apparatus.

To avoid such difficulties, an essential specification for on-site equipment is that the sampling units before and after the test filter must be independent of each other. The equipment for the lithium flame test fulfils this demand and avoids the tedious task of repeated calibration of an air-flow system.

The hardware required for the construction of the sodium flame photometer is not among standard production items and must therefore be produced by each individual laboratory from the Porton specifications.⁽⁴⁾ Any type of high quality flame photometer may be used for the lithium test.

The application of the lithium flame test on-site will be discussed later in connection with a practical example.

The basic scheme of the lithium flame photometer test is briefly as follows:

A 2% aqueous solution of Li_2SO_4 is atomized

by a Collison atomizer (see Fig. 1). The spray is diluted with prefiltered dry air in an evaporation tube, which thus yields a diluted particulate

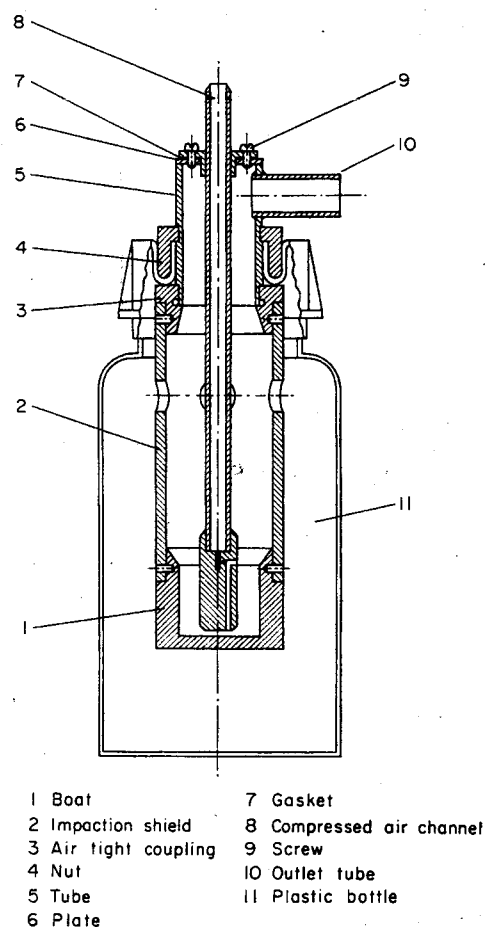


FIG. 1. Modified Collison atomizer. This design allows the atomizer to be used for small quantities of radioactive solutions. The cap construction ensures complete tightness of the system.

stream of Li_2SO_4 crystals. The stream enters a mixing chamber where a few litres per minute is tapped and passed through a membrane filter. A portion, or all, of the remaining flow is passed through the filter to be tested. The effluent air enters a second mixing chamber identical with the first, and an appropriate amount of the flow is tapped and passed through a second membrane filter. The membrane filters are then processed

chemically, and two solutions are produced in which concentrations of lithium as low as 0.0002 γ /ml can be detected with a sensitive flame photometer.

The ratio between the lithium contents of the two solutions is then, after compensation for differences of air volumes sampled, a measure of the effectiveness of the test filter.

Lithium has been chosen as a tracer substance although a flame photometer is about ten times as sensitive to sodium as to lithium, because of the relative rarity of lithium in laboratory surroundings and the ease with which it is measured on a flame photometer because of the lack of interference from other alkali metals present.⁽⁶⁾ In coastal areas, concentrations of NaCl in the air⁽⁶⁾ of up to 20,000 μg NaCl per m^3 have been measured.

A set of membrane filters rubbed over various surfaces at this laboratory were processed for lithium determinations on a photometer capable of detecting 0.0002 γ Li/ml in the 5 ml solution used. In no case was any lithium found.

The inapplicability of sodium as a tracer component is further emphasized by the variations in the amounts of sodium found on fresh membrane filters. An amount of sodium large enough to allow us to neglect the variation in the sodium content of the filters should be sampled from the air after the filter to be tested, which means in practice that sodium would be much less sensitive than lithium as a tracer component.

The mean size by mass of the Li_2SO_4 particles was not measured, but the atomizers employed in all the experiments deliver NaCl particles of a mean size by mass of 0.6 micron from a 2% NaCl solution.

Electron microscopy reveals that Li_2SO_4 crystals appear as elongated rhombic forms unlike the cubic NaCl crystals from a Sodium Flame Test cloud.

As the original atomizer made from the Porton specifications was of steel, it was reconstructed in a hard plastic to avoid corrosion. At the same time the container was changed, the glass jar originally used being replaced by a plastic bottle to avoid the difficulties of tightening the screw-lid. For radioactive tracer experiments, in which the amount of radioactivity applied in the atomizer should not exceed that

necessary for the experiment, a special beaker was attached to the bottom of the impaction shield. In this case the holes in the impaction shield should be closed to avoid waste of liquid.

CALIBRATION OF THE LITHIUM TEST WITH THE SODIUM FLAME TEST AND POLLAK COUNTERS

In order to determine what differences a filter might display between a sodium chloride cloud and a lithium sulfate cloud, a series of investigations was made. The importance of such inter-calibration is apparent as the Sodium Flame Test is a standard test widely used by filter manufacturers.

The inter-calibration was carried out by spraying 2% solutions of Li_2SO_4 and NaCl into the open end of a cylindrical duct of diameter 8 cm leading to the test filter. From the latter the duct continued to a larger one through which a ventilation fan drew the air. The air flow through the test filter was measured by a flow meter of the rotameter type and controlled by adjusting a valve opening into the large duct.

The cylindrical duct from inlet to test filter was about 8 metres long and contained two baffle plates. It had a sharp bend two metres from the test filter.

A before-filter tap-hole was situated after the bend and an after-filter tap-hole about 8 m from the test filter, after a baffle plate.

The atomizer was of the type shown in Fig. 4. An aluminium pot contains three atomizers of the collision type, made from the Porton specifications. The atomizers are interconnected by a distributor plate allowing a general feed of compressed air to them of about 7 atm. In order to obtain an efficient impingement of the atomized liquid, the atomizers are surrounded by a special baffle-plate arrangement allowing an equally efficient impingement of the droplets from all sides. Fast evaporation of the droplets and an improved yield of lithium crystals are obtained by the addition of an air flow through a range of small holes placed along the circumference of the aluminium pot. The extra air is distributed through the holes by means of a circular tube welded to the outside of the pot.

The total output from the atomizer pot was measured at about 8 ml liquid/min. With a compressed-air flow at 7 atm and an auxiliary

evaporation air flow of about 100 l./min the consumption of compressed air is 252 l. air (760 mm Hg)/min.

The NaCl solution was spiked with ^{24}Na so that the test-filter penetration could be measured directly by counting the activities sampled from the air before and after the filter.

The lithium measurements were made by the method mentioned previously. The details of this method are described in the following section.

The test filters comprised the types 33, 44, 55 manufactured by the British filter manufacturing company Vokes Ltd. The manufacturer's penetration ratings for the filter in the above order were 25%, 2% and 0.05%. The filters were tested at a flow rate of 850 litres per minute.

In addition to these tests the natural particles found in the building were used to test the filters. The before and after particle concentrations were determined by means of two Pollak counters⁽⁷⁾ operating from the two tap-holes. The penetration of these natural aerosols is shown together with the results of the intercalibration. The reason for using the Pollak counters is to demonstrate the relation between the mass penetration of the lithium and sodium salts and the particle-number penetration of the natural aerosol.

In cases where the natural particles found in the air are present in concentrations too low to be measured accurately after passage through a highly efficient filter, particles can be generated artificially by inflating a large rubber balloon with air containing particles from a heated filament over which a boat of TUFNOL is suspended. Within a short time a large reservoir can be loaded with a very high particle concentration.

The particles generated in this way have a diameter of about 10^{-5} cm provided that during the heating process, the air around the Tufnol boat is only circulated by the free convection from the heat developed. If forced convection is applied, the diameter of the particles may fall in the range of 10^{-6} to 10^{-7} cm.

A test with a 66 type filter (manufacturer's rating 0.1% penetration) displays the so-called pinhole effect, see Fig. 2. The effect, when present, can easily be demonstrated by means of a

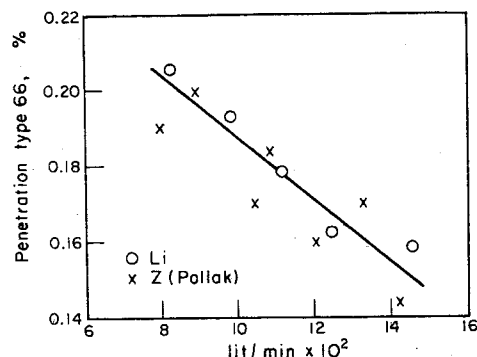


Fig. 2. Plot of results from tests with filter 66.

Pollak counter as well as by a lithium test as the penetration appears to decrease with increasing air speed through the filter. The pinhole effect may dominate the performance of a filter.

Whether there were actual pinholes in the filter material or pinhole leaks due to damage or faulty installation, was not determined. It can only be said that the unit did not function according to the manufacturer's specifications.

PREPARATION OF THE FLAME-PHOTOMETER SOLUTIONS FOR THE DETERMINATION OF LITHIUM ON MEMBRANE FILTERS

A Göttingen membrane filter type MF of 50 mm diam. can be dissolved under gentle heating in 1 ml conc. HNO_3 . If this solution is diluted with H_2O to 5 ml, a Zeiss flame photometer (type PF 5) is able to detect the presence of 0.003 γ/ml Li. It has been found that the response of the photometer to these solutions is not linear in the range 0.003 γ/ml Li to 0.03 γ/ml Li. Further, the responses are quite unstable.

In order to increase the sensitivity of the photometer readings, the addition of 1 part of acetone to 4 parts of the final solution was tried.

A membrane filter was dissolved in 1 ml concentrated HNO_3 in a 5 ml measuring flask by heating gently over a bunsen flame and shaking. After cooling to room temperature 1 ml of distilled water was added, and the solution was again allowed to cool to room temperature. The following quantities of a 3 γ Li/ml solution were added to the measuring flasks: 5, 10, 20, 25, and 50 μl , giving standard solutions of

0.003, 0.006, 0.012, 0.015, and 0.03 γ Li/ml. 1 ml of acetone was added producing a white precipitate. Distilled H_2O was added up to the 5 ml mark, and the flask was shaken thoroughly. After filtration through a Whatman glass-fibre filter the clean effluent was set aside in another 5 ml measuring flask. On account of the constant formation of gas from this solution it is advisable to leave the flasks only lightly covered.

In the determination of the amount of lithium adsorbed on a membrane filter, a total solution of 5 ml (minimum) is used to allow a reasonable measuring time of up to 45 sec. One must be particularly careful with these small amounts to measure the 1 ml of acetone accurately.

It was noted that even after 7 days the solutions, standing in open measuring flasks, did not show any change in their concentration of lithium. In order to ascertain whether any of the lithium was lost in the precipitate, another calibration series was made in which the aliquots of 3 γ Li/ml sol. were added in the final step after filtration.

The measurements on the flame photometer are shown in Fig. 3. In addition to these tests, one was performed in which the photometer

was set at its maximum sensitivity and the null solution adjusted to zero on the scale. In this case the 0.003 γ Li/ml sol. read 25 units on the scale. If the instrument is assumed to respond approximately linearly to these solutions at this setting, then a solution containing about 0.0002 γ Li/ml would give a scale deflection of more than one unit.

All the above readings were quite stable and easily reproducible.

DISCUSSION OF AND CONCLUSIONS FROM INTER-CALIBRATION TEST

Some initial experiments were performed in which the test filter was omitted to ensure that losses to the walls and baffle plates did not affect the Li and Na contents in the air samples from the different tap-holes. It was found that the Li and Na contents of the air samples were within the measuring accuracy of the gasometers.

The Li and Na calibration experiments for the two filter types 55 and 44 (see Figs. 5 and 6), while very consistent individually revealed a qualitative shift in the Li-Na ratio of penetrability of the two filters. The shift was somewhat surprising and could not be explained by means

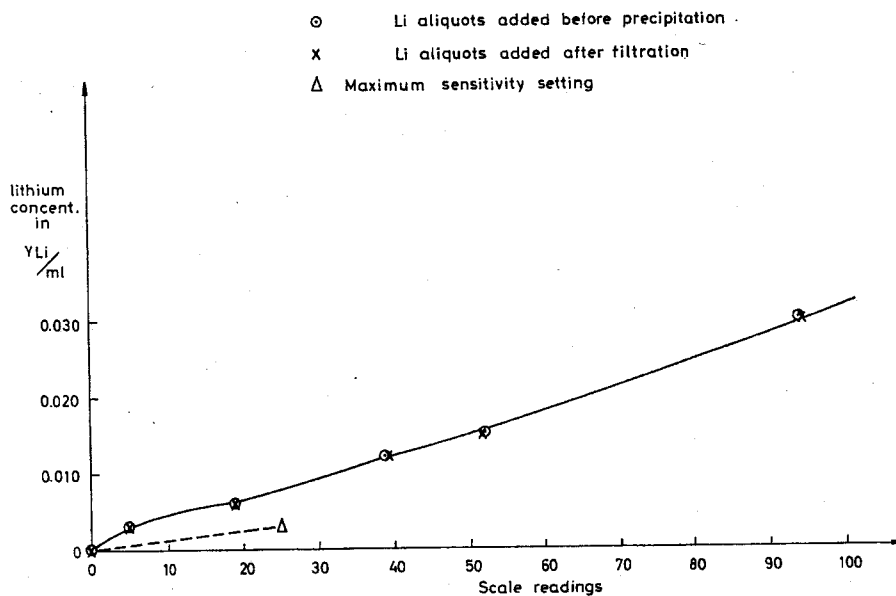


FIG. 3. Calibration curves for Zeiss PF5 flame photometer: air pressure = later; acetylene pressure = 90 mm W.

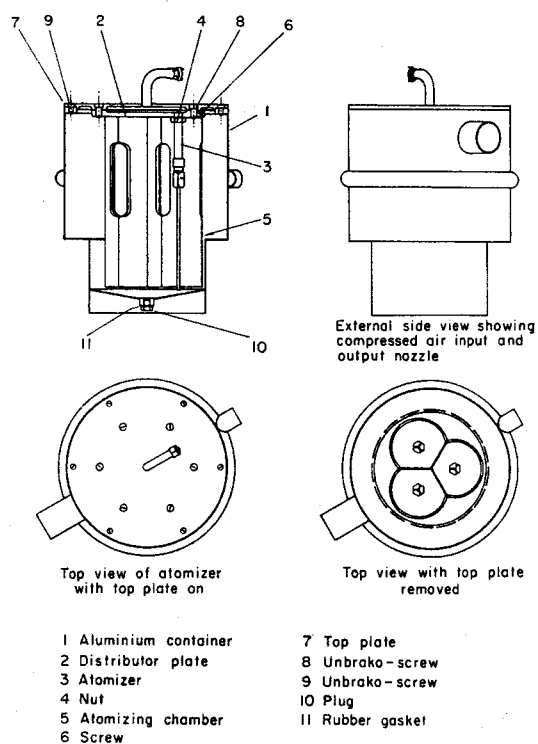


FIG. 4. Portable atomizer for on-site testing. The three atomizer elements included are equivalent in structure to the elements used in the Collision Atomizer shown in Fig. 1.

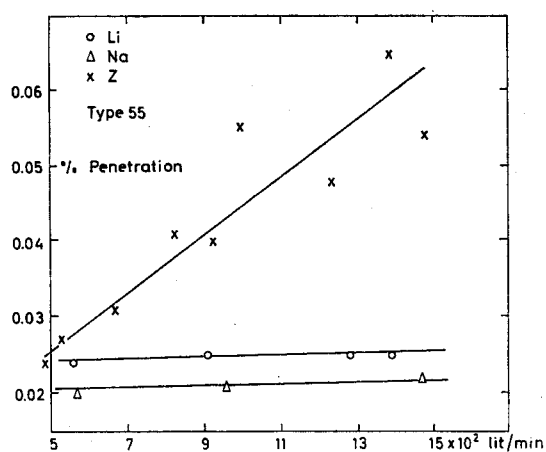


FIG. 5. Plot of results from tests with filter 55.

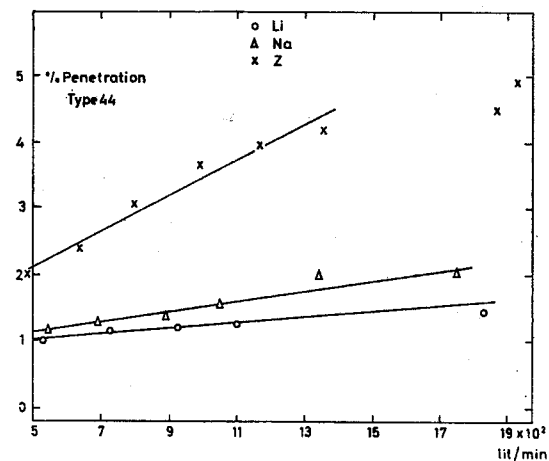


FIG. 6. Plot of results from tests with filter 44.

of the filtration theory. The filters were consequently reinstalled, and the calibration experiments were repeated. Figures 8 and 9 display the results. Comparing the results from Fig. 5 with those from Fig. 8 and the results from Fig. 6 with those from Fig. 9, we notice the same qualitative shift in the Li-Na ratio of penetrability, but in this particular case it is not related

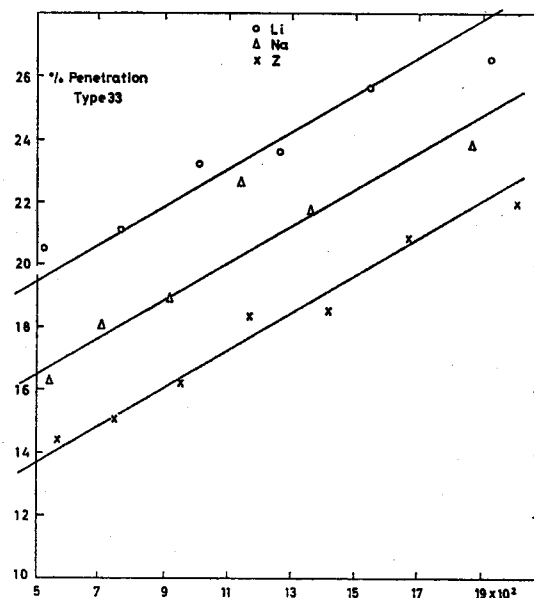


FIG. 7. Plot of results from tests with filter 33.

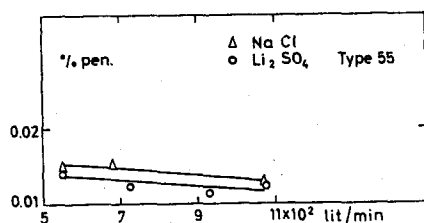


FIG. 8. Repetition of tests with filter 55 after removal and reinstallation.

to a shift in filter type, but connected with the same individual filter.

The observed difference may have been caused by using two different G.M. counters, depending upon the activity to be measured. Therefore, a new series of similar experiments with the same 44-filter was performed in which all the counting was done on a single scintillation counter. The shifts between the effectivity curves from test to test for both sodium and lithium were found to be the same as those observed previously. Therefore it was concluded that the counting technique was not a cause of this apparent shifting.

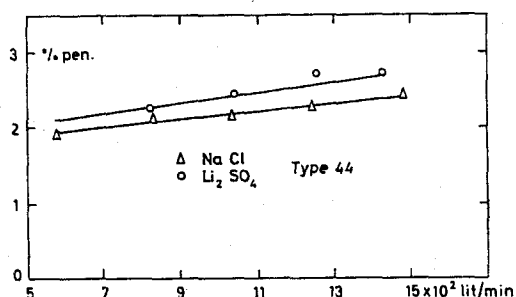


FIG. 9. Repetition of tests with filter 44 after removal and reinstallation.

There were two other possibilities that might each lead to such variations.

(1) If a variation in the atomizer mechanism were responsible for the shifts then one could hope to observe such variations by taking effectivity measurements before and after stopping and starting the atomizer again.

(2) If there is an intrinsic variation in the filter itself from test to test then one could try to produce such variations by varying the air-flow through the filter from extreme to extreme and seeing if a given effectivity determination taken before and after these impressed flow changes holds the same value.

It was shown that no significant shifts were present due to either cause but that there was a shift when the air flow was completely stopped then started again. For the 44 type filter, there was a change from a penetrability of 4.85% to a penetrability of 4.50%, at a flow of 1000 l/min.

Two experiments were performed to confirm this phenomena.

A test cloud of ^{24}Na spiked NaCl was used with the 44-filter. Points on each curve were determined as the air flow was increased from zero to maximum and back to zero again. The shift between the two curves shown in Fig. 10 indicates the existence of the start phenomenon.

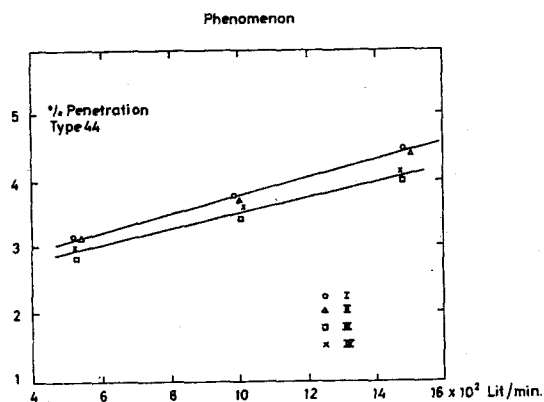


FIG. 10. Repetition of tests with filter 44 demonstrating the start phenomenon.

Therefore, according to our measurements a given filter of the type 33, 44 or 55 displays a start phenomenon that dominates any discrimination of these filters between Li_2SO_4 and NaCl particles. The Li_2SO_4 test is therefore practically equivalent to the NaCl test.

The Pollak counter curves, on the other hand, are consistent relative to the lithium and sodium curves as shown.

DISCUSSION OF AN EXAMPLE OF AN ON-SITE TEST

An example of a large installed filter system is that cleansing the air of the reactor building DR 3 at Risö.

The installation consists of a single ventilation channel, cross-section about 1 m^2 , in which the flow is $6000 \text{ m}^3/\text{hr}$. In this duct a primary dust filter and an absolute filter are installed. The under-pressure in the channel is about 80 mm H_2O before the primary filter and about 150 mm H_2O after the absolute filter.

At a point about six metres from the face of the primary filter a cloud of droplets atomized from a 2% Li_2SO_4 aqueous solution is sprayed into a tap-hole. At a point between the primary and the absolute filter another tap-hole is connected to a piston pump which draws about 20 l./min from the channel air through a membrane filter. Further downstream, about ten channel diameters from the end face of the absolute filter and round a gradual right-angle bend in the channel, a final tap-hole draws 20 l./min of air through a membrane filter.

The flow of sampled air is monitored by two flow meters of the float type, and the air volumes are measured by two gasometers. The atomizer used was of the type shown in Fig. 4. Four runs were performed, and the results were as follows: trial 1: 0.69%, trial 2: 0.70%, trial 3: 0.10%, and trial 4: 0.60% penetration.

According to recent determinations,⁽⁸⁾ thorough mixing in such a duct is assumed for points lying more than 30 channel diameters downstream of a source of contamination or the effluent face of a filter, and then only if there is some baffle arrangement within this length. If the duct has one or more bends within the region from the point of injection of the aerosol to the final hole, these bends might, if sufficiently acute, be effective as baffle surfaces. If sampling points exist which satisfy these criteria and are accessible, the above procedure can yield a measurement of the efficiency of the filter bank as a whole.

A second on-site test that fulfilled the above requirements was therefore carried out on the same filter at the DR 3 reactor. In this case, the aerosol, a ^{24}Na spiked 2% NaCl solution from a modified Collison atomizer delivery 15 ml/hr was introduced into one of the inlets of a ventila-

tion duct in the reactor hall itself. The tap-hole for the before filter was situated just before the primary filter face and the after tap-hole about 15 m downstream of the absolute filter; the length of duct in between had four sharp bends. The measured penetration was 0.06%.

A total of 10 mCi ^{24}Na was sprayed into the ventilating system in order to ensure good after-filter measurements. The spraying time was 6 hr. The same DR 3 filter bank could have been evaluated by means of a 2% Li_2SO_4 solution atomized by the portable atomizing unit capable of delivering 8 ml solution per min. The total number of litres to be sampled by the after filter in order to ensure a good flame photometer reading was calculated at about 80.

The above test using ^{24}Na was performed on an auxiliary filter unit situated in parallel to the first, the penetration was 0.10%.

A test was then performed with the Pollak counters, using the natural aerosol. The first filter displayed a particle-number penetration of 0.12%, twice that of the NaCl cloud. The auxiliary filter displayed a particle-number penetration of 0.25%, $2\frac{1}{2}$ times that of the NaCl cloud.

CONCLUDING REMARKS

The question arises as to the significance of the efficiency measured by these methods. It is not the inherent penetrability of the adsorbing material that is of principal interest since it is always specified by the manufacturer. What concerns us here is the performance of the installed units, since such a determination can reveal a malfunction in the filter bank, e.g. a badly installed or damaged filter.

When the results of an on-site lithium test indicate that the manufacturer's specifications are not fulfilled by the installation as a whole, one knows exactly the extent of the failure because the lithium test is directly comparable with the Sodium Flame Test used by the manufacturer. Thus one is in a position to quantitatively evaluate the performance of a filter installation by direct comparison with the figures offered by the manufacturer as to the expected performance of the unit. In other words, the lithium on-site test represents an easy way of comparing the actual efficiency of a filter installation in operation with the efficiency attainable according to the manufacturer's test.

The spraying time needed to introduce sufficient amounts of lithium salts into a ventilation system sets a practical upper limit to the utilization of a lithium salt as a tracer in very high capacity systems. For example, in a ventilating system through which the flow is 100,000 m³/hr and whose filter bank shows a penetration of 0.01%, it would be necessary to sample about 8000 l. on the after filter. This figure is based upon the output of the portable atomizer unit mentioned previously. The time of sampling based upon a sampling rate of 20–50 l./min through a 5 cm diameter membrane filter would be from 7 to 3 hr.

For these very high capacity systems the use of Pollak counters measuring the natural particles found before and after the filter bank is a reliable indication of the integrity of the filter bank. Allowance must be made for the difference between the effectiveness of a filter bank as determined by Pollak counting and the effectiveness as an expression of mass concentrations before and after the filter bank. This difference is demonstrated and established by the inter-calibration curves (Figs. 5–9). These curves are valid only for filters in which the pinhole effect is not significant.

In cases where it is possible to vary the air flow through a filter bank and thus demonstrate the existence of a pinhole effect by Pollak counting, the effectiveness thus obtained converges with the effectiveness as measured by the lithium and sodium methods (Fig. 2).

It might be desirable to determine the exact location of a pinhole defect (if it cannot be tolerated from a safety point of view). This can be done by scanning the back surface of each filter with a flexible probe introduced through a hole in the conduit wall. The air injected into this probe, e.g. by means of an ejector, is passed directly into a flame, and the intensity of the lithium line is registered on a photometer. In this way a continuous profile of the lithium in the effluent air is obtained. In this procedure the lithium concentrations due to a leak are very high, and thus it suffices to use a cheap and simple flame-photometer device. Such a system has been constructed (Fig. 11) by modifying a cheap, commercially available flame photometer (Evans Electroselenium Ltd.). At the most sensi-

tive setting a scale deflection of 10 units (~ 1.5 cm) is obtained from a channel concentration of $1.2 \times 10^{-2} \gamma$ Li/l. air.

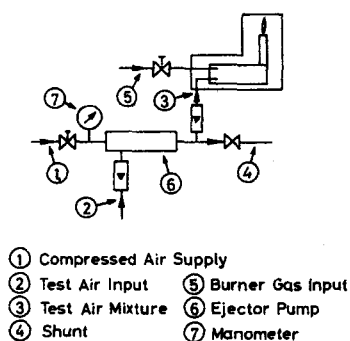


FIG. 11. Flow diagram of the modified EEL flame photometer.

ACKNOWLEDGEMENTS

We wish to thank P. O. Heidemann for his help in planning and drawing the atomizer assemblies shown in Figs. 1 and 4.

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