THE ACCURACY OF WEIGHING PROCEDURES DURING GRAVIMETRIC DUST SAMPLING

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The accuracy of gravimetric dust sampling is greatly dependent upon the accuracy of the filter paper weighing procedures. The purpose of this paper is to describe the procedures which have been used in one laboratory for just over a year, and to show how the weighing results have been used to assess the level of accuracy of filter weighing.

An earlier paper by the authors[1] documented full details of the sampling and weighing procedures. Since these procedures form the basis for the present paper certain relevant information is of necessity repeated here.

Weighing Procedures

In principle the measurement of the mass of dust collected on a filter paper is simple. The filter paper is weighed both before and after use, the difference in mass being the mass of dust collected on the paper. Problems arise because the mass of dust collected on the filter paper is in just about every instance a very small fraction of the mass of the filter paper itself. The filter papers used are cellulose nitrate, have a diameter of 37 mm and a mass of approximately 40 mg. The mass of dust deposited on the paper is typically less than 1 mg, although this quantity can be very much higher in very dusty situations.

Inherent in the procedure are two significant problem areas which must be recognised:

- Firstly, the occurrence of random errors during weighing can be of considerable importance. This is generally overcome by repeated weighing. In the present instance each filter paper is weighed three times both before and after use. The three values are only accepted for averaging if they differ by not more than 50 µg. In practice there are very few measurements where the difference exceeds 20µg.
- Secondly, problems frequently arise due to moisture absorption or desorption by the filter paper. This can alter the mass of the filter paper by a few hundred micrograms. This problem is overcome by either working in a temperature and humidity controlled weighing room, or by the use of one or more reference filter papers. In the present instance a temperature and humidity controlled weighing room was not available, and the procedure of utilising two reference filter papers was adopted.

The complete weighing procedure practised is therefore as follows:

1. Two reference filter papers are selected from each packet of 100 papers, and are used whenever any of the remaining filter papers from the pack are weighed, both before and after use.
2. Prior to weighing, both the two reference filter papers and the filter papers, whether new or used, are allowed to reach equilibrium with the atmosphere by keeping them in a cabinet for at least eighteen hours.
3. Weighing is then carried out. Each reference filter paper is weighed three times, and then up to five filter papers are weighed, also three times each. Should more than five filter papers have to be weighed at one session the weighing of the two reference filter papers is repeated.

Weighing Results

The above weighing procedure has been practised for more than a year and during that time some 900 filter papers have been weighed before and after use.

The principal reason for using two reference filter papers instead of just one is that it is felt that this practice provides a means for both checking the results in order to identify errors and establishing the accuracy of the weighing procedures.

Figure 1 *Reference Paper IR 1*

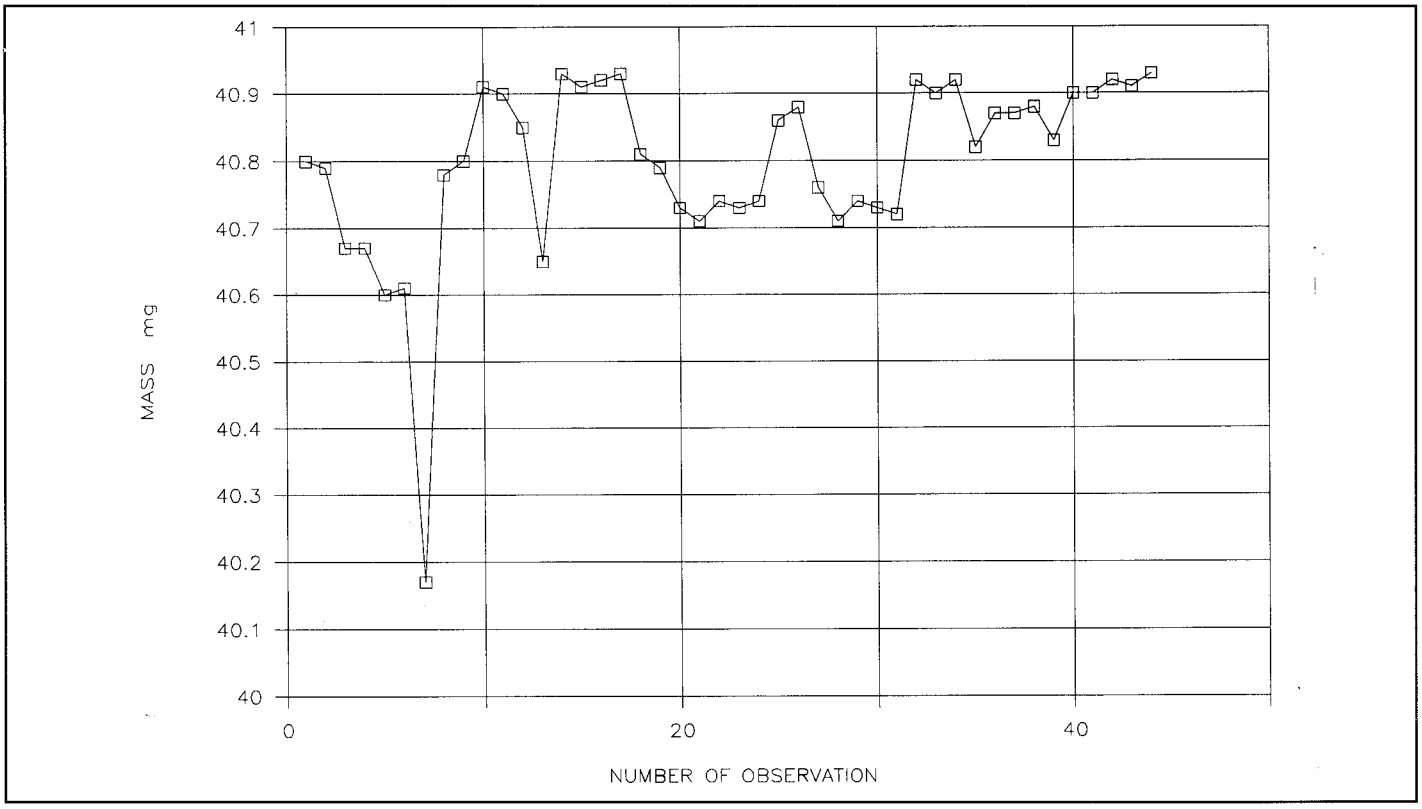


Figure 2 *Reference Paper IR 2*

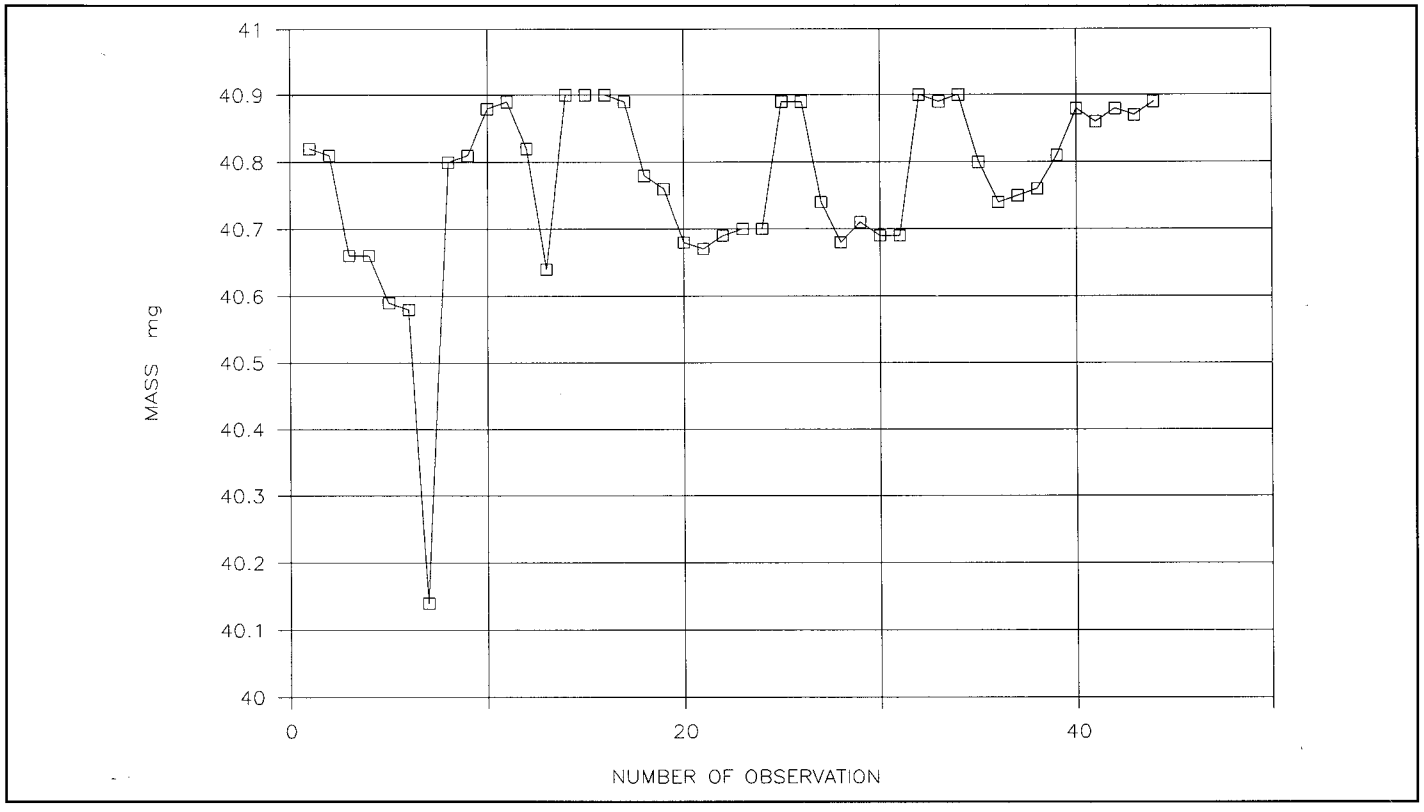


Figure 3 *Reference Papers IR 1 and IR 2*

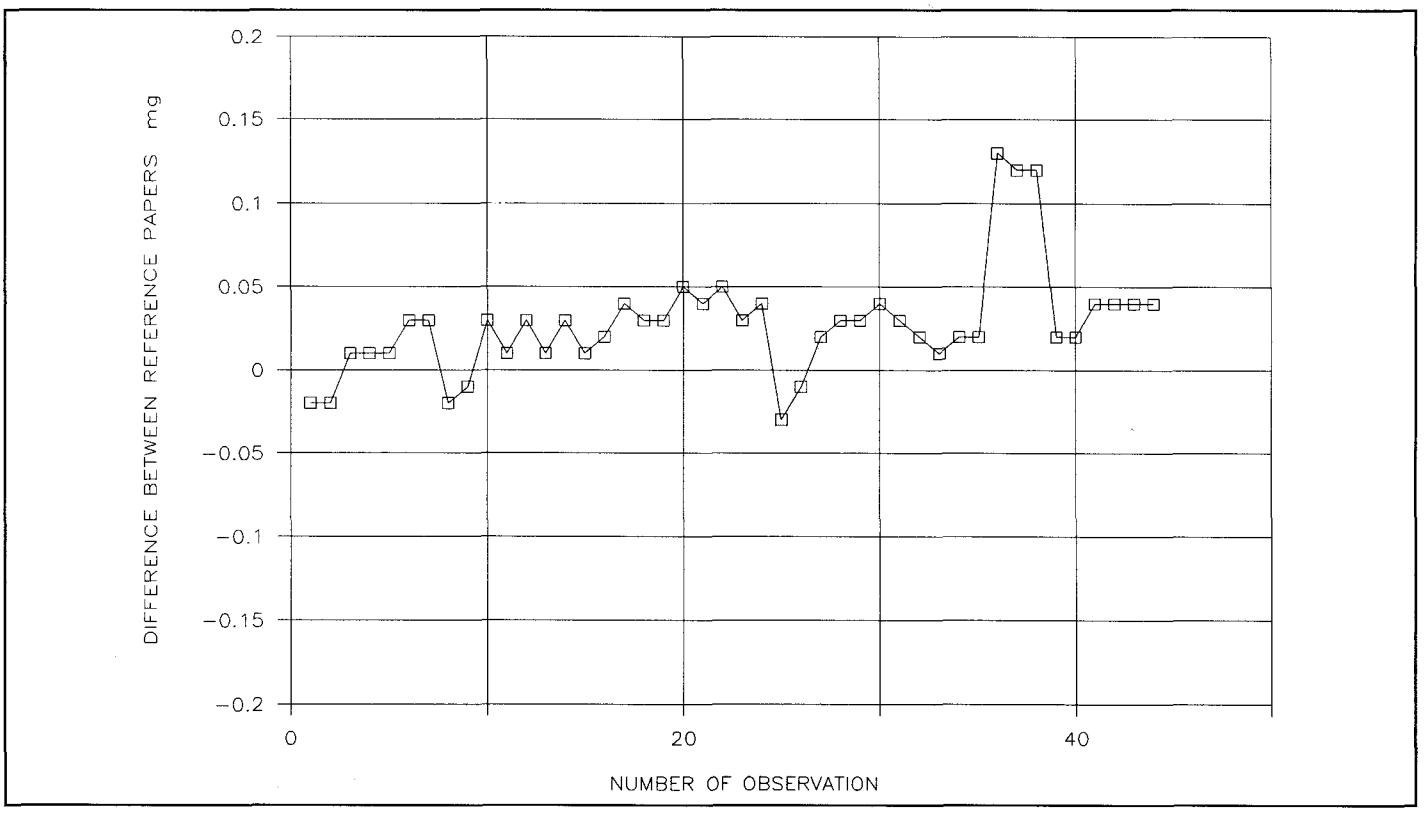
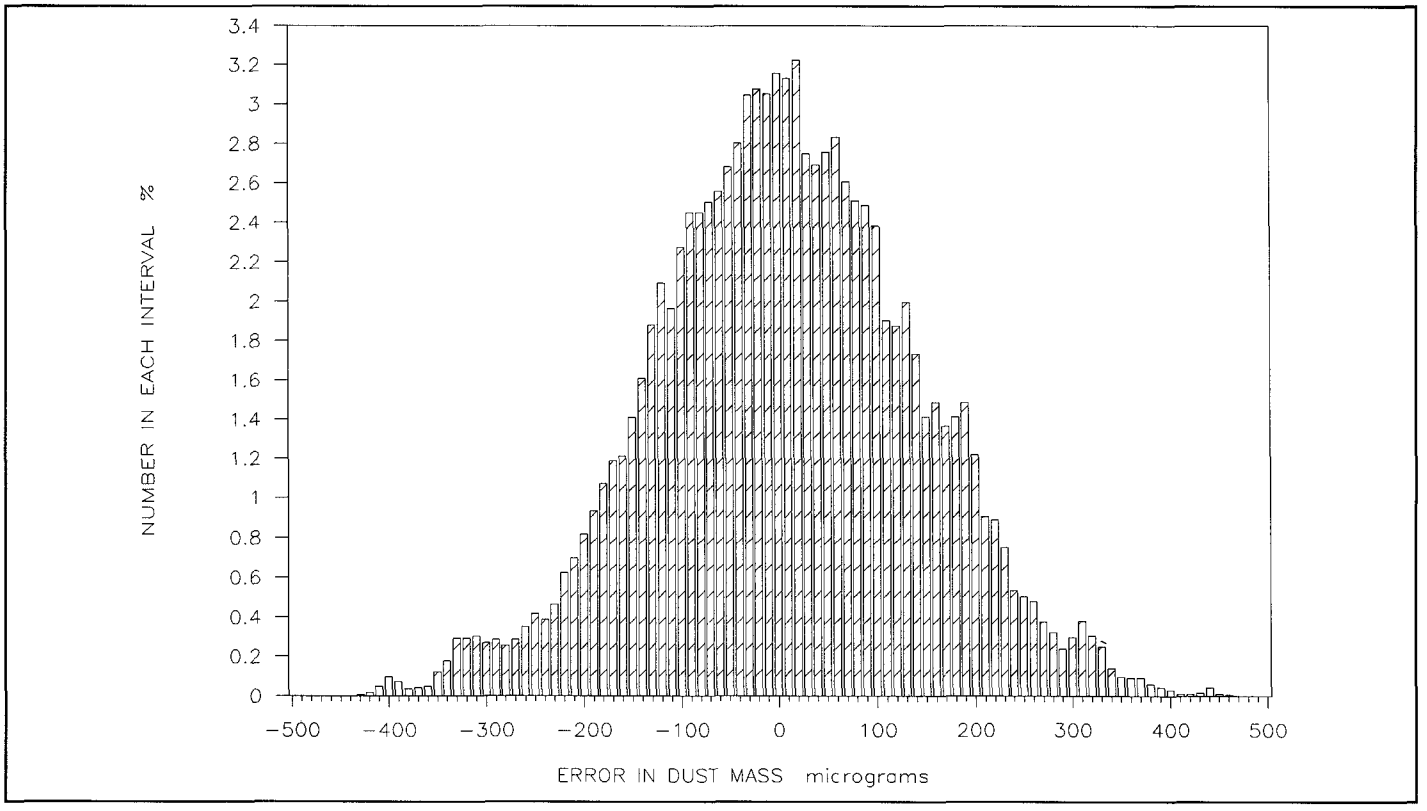


Figure 4 *No Reference Paper, Different Days*



The first aspect, **"the checking of results",** is illustrated in three graphs.These show the variation in mass of a pair of reference filter papers (**Figures 1** and **2**), and the variation in the difference between the masses of the two reference filter papers (**Figure 3**).

Measurement 7 reveals a difference between the two reference filter masses which fits in well with the other readings. However the individual masses differ to such an extent that the observations are suspect.

Measurements 36, 37 and 38, all observed during one day, have individual masses which fit in well with measurements made both before and after this day. The difference graph, however, indicates that these three measurements are also suspect.

During the period under review this situation arose on four or five occasions and in each case it was decided to repeat the sampling affected.

The second aspect, **"establishing the accuracy of weighing procedures",** will now be described.

During the period under review a total of 12 pairs of reference filter papers had been used, each pair having been weighed anything from 11 to 52 times. These weighing data, consisting of a total of 428 weighings of pairs of reference filter papers, were analysed as follows.

In the first instance it was considered worthwhile to establish likely weighing errors as if a policy of using no reference filter papers had been adopted. This was achieved by assuming that a reference filter paper had been used for dust sampling, and had been weighed both before and after use. As the paper had not in fact been exposed to any dust the difference between the two masses indicates the weighing error, both as a result of actual balance errors and as a result of the effects of changes in humidity. The term "weighing error" thus really implies the error in determining the mass of dust collected on a filter paper.

The analysis took each weighing of a particular paper, assumed this was the before use mass, and then assumed that each later weighing, providing this was on a different day, was an after use mass. Calculations were confined to readings taken on different days because it was considered that readings taken on the same day would tend to display a very low variance, and in practice a used filter paper can only be weighed at least one day (and very likely two or more days) after its initial weighing.

This procedure is perhaps better illustrated by considering a specific example.

Assume that a reference filter paper was weighed on five separate occasions, each on a different day. The five measured masses are Ml, M2, M3, M4 and M5. These five masses can be used to calculate ten separate values of the dust mass. Seeing that no dust was in fact deposited on the filter paper it is more correct to call the dust mass a weighing error. The ten weighing error values arise from the following mass differences

M2 - Ml

M3 - Ml

M4 - Ml

M5 - Ml

M3 - M2

M4 - M2

M5 - M2

M4 - M3

M5 - M3

M5 - M4

On this basis the 428 weighings of pairs of reference filter papers resulted in a total of 16 482 values of weighing error.

These values are shown on the histogram (**Figure 4**) and illustrate

- that in no instance was the error greater than 460 μg.
- that in 95% of the cases the weighing error was less than 270 μg.

A more relevant analysis results when it is assumed that one of the pair of reference filter papers is the reference paper, while the other is the paper used for dust sampling. As before, because this second paper had not been exposed to any dust the resulting dust mass is once again an indication of weighing errors. As before these errors include actual balance errors as well as those effects of humidity not compensated for by the reference filter paper method.

The same procedure as before was adopted, with each set of weighings of a particular pair of reference filter papers being considered as the before use measurements, and each subsequent set, again on a different day, being considered as the after use measurements. This analysis resulted in 8 241 values of weighing error.

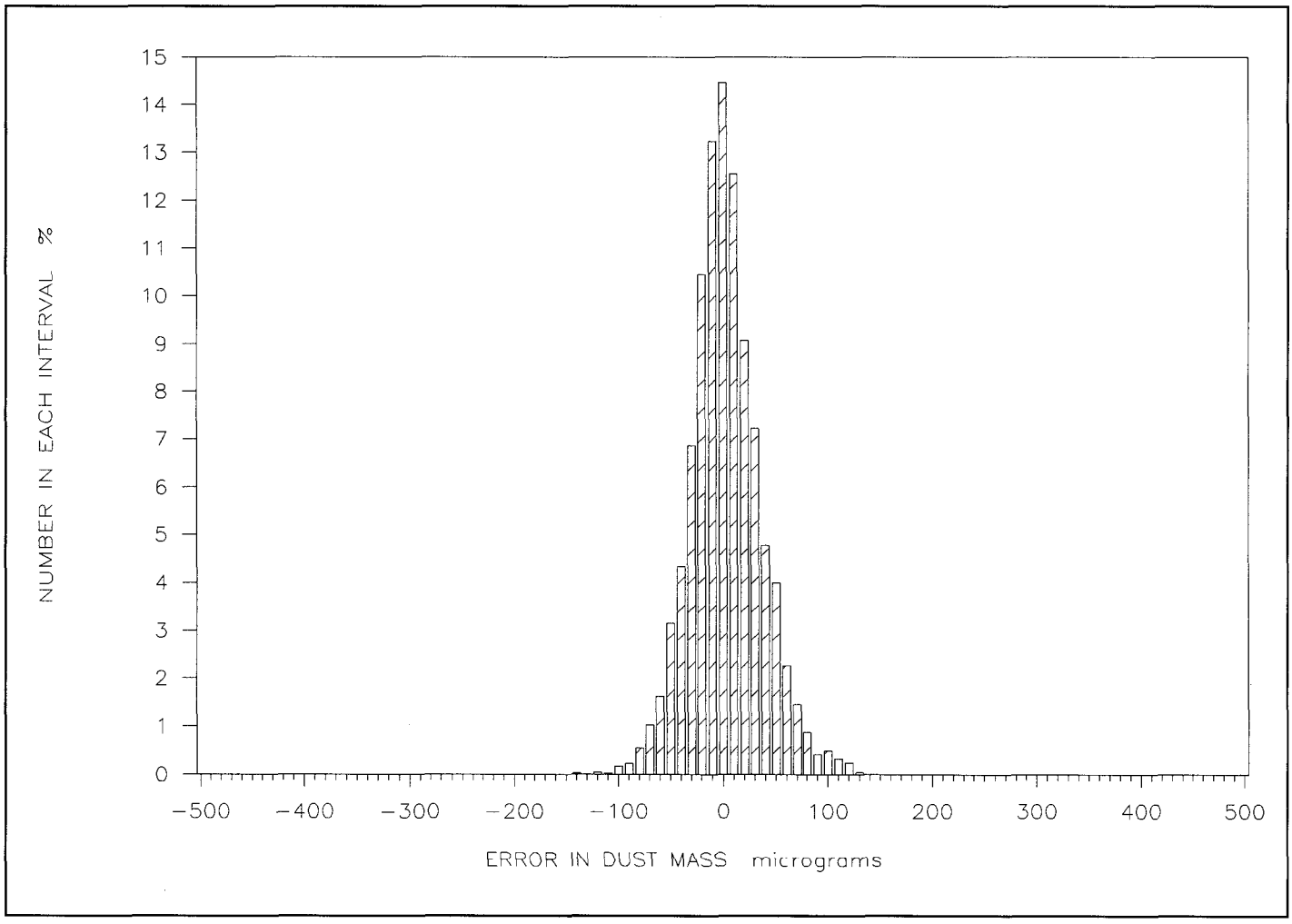
These values are depicted in the second histogram (**Figure 5**) and show

- that in no instance was the error greater than 140 μg.
- that in 95% of the cases the weighing error was less than 70 μg.

Conclusions

The conclusions to be drawn from these observations clearly indicate the following:

Figure 5 *Reference Paper, Different Days*



- The procedure of using more than one reference filter paper has definite advantages, and is therefore recommended. The major benefits to be derived are that an examination of the reference filter paper weighings provides a check on the weighing system and further that, as illustrated in this paper, an analysis of the reference filter paper weighings makes it possible to establish the accuracy of the weighing procedure.
- There is no indication at all of any accumulative loss (or gain) in mass of reference filter papers due to handling. This is illustrated in **Figs. 1** and **2**.
- The reference filter paper method provides a substantial decrease in weighing errors over the method where no reference filter paper is used. Weighing errors are typically twenty-five percent of those experienced with no reference filter paper.
- While the reference filter paper method does reduce weighing errors significantly it does not reduce these to a point where they can be attributed solely to balance errors. Thus it can be concluded that the method does not compensate totally for the effects of varying humidity.

Acknowledgement

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Reference

1. **DEGLON P** AND **HEMP R.** A gravimetric sampling procedure for opencast coal mines. Mine Ventilation Society Symposium : Dust and Radon : Considerations for the Future, Kimberley, February 1990.