

What's up with the European PM reference methods?

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Quite recently it has become clear that the European reference methods for measuring **PM₁₀ (EN12341)** and **PM_{2.5} (EN14907)** can lead to much larger ranges of results than what was first assumed. It turns out that certain artefacts were underestimated or even unknown when the standards were created. In the last couple of years the Flemish Environment Agency has carried out a range of experiments and tests in order to investigate some of the issues, and to put them on the agenda of CEN TC264 WG15 (which created both standards).

Some 5 years ago various types and brands of 47-mm filters were used in parallel in several field campaigns. To our surprise the differences between the different variations of the reference method were sometimes as high as 25% (as campaign averages). Of the two filters that were used the most, the **Whatman QM-A** (which contains 5% borosilicate and is pre-fired in the factory) gave results that were on average 14% higher than **Macherey-Nagel QF10** (a 100% pure quartz fiber filter). Tests with the standard EMERY 3004 aerosol (a poly alpha olefin) showed that the observed differences were not due to variations in physical separation efficiency, as all filters scored better than 99.9%.

In a more recent project ("Chemchar PM₁₀" a chemical characterisation project) the Whatman QM-A filters (which were extra pre-fired to obtain lower OC blanks) captured on average 7% more mass than teflon filters (Pall Teflo) that were sampled in parallel. Part of this difference was due to the higher blanks for the QM-As (112 µg absolute resulting in ± 2 µg/m³) than for the teflon filters (56 µg absolute resulting in ± 1 µg/m³).

A 3-step experiment with 5 filter types (Table 1) was done to investigate both the effects of pre-firing and the use of the 'Amsterdam' approach (= 'pre-conditioning' the blank filters by exposing them to 100% humidity for some weeks, before weighing and sampling). Each set of a filter type consisted of 3 new, identical filters.

In the first step of the experiments types 3 and 5 were fired (by Ghent University) for 24 h at 550 °C. This led to a mass loss of 10 mg for the QF10s and 2 mg for the QM-As.

Table 1: Filter types used in 3-step experiment.

1. Pall Teflo	teflon
2. Macherey-Nagel QF10	100% quartz
3. Macherey-Nagel QF10*	100% quartz
4. Whatman QM-A	95% quartz + 5% glass
5. Whatman QM-A*	95% quartz + 5% glass

*to be fired in step 1

In the second step all 5 sets of filters were exposed to 100% relative humidity (RH) for 9 weeks (Fig 1). All quartz filters showed a rapid increase in the first couple of days and did not stabilise within the 9 weeks. Similar profiles, but in a lower mass range, had already been obtained at 50% RH and for field blanks.

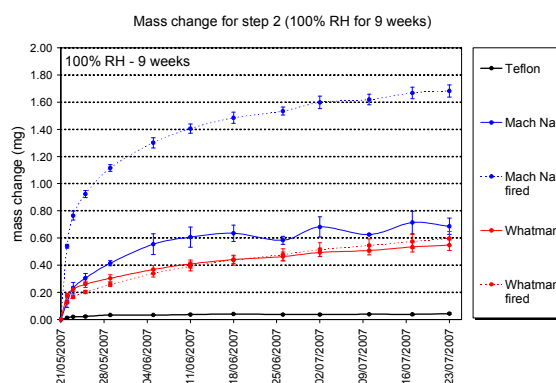


Figure 1: Mass change for step 2, at 100% RH.

In the third step all the filters were exposed to 50% humidity for 30 weeks. The results of the first 4 weeks are presented in Fig 2 and show that 3 of the 4 types of quartz filters reach an equilibrium weight within 48 h. For these 3 types this weight turned out to be the same as the weight they had after exactly 3 weeks in step 2 (at 100% RH) indicating that at least 3 weeks of 'pre-conditioning' is desirable. The 4th quartz type, the pre-fired QM-A showed only a small loss in mass and even started gaining weight again after 3 days. After the full 30 weeks this type had gained about 100 µg compared to the start of step 3. For the other types the mass was constant or less than 50 mg from the equilibrium weight.

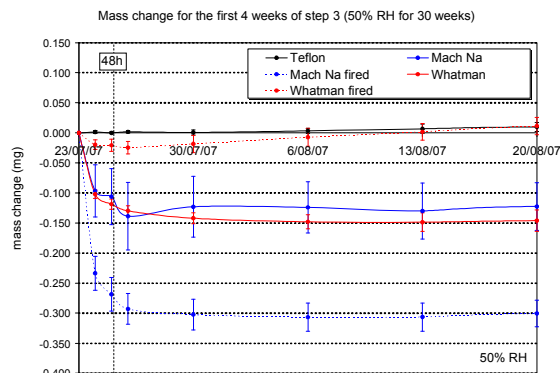


Figure 2: Mass change for step 3, at 50% RH.