National Institute for Public Health
and the Environment
Ministry of Health, Welfare and Sport

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RIVM Letter Report 680708017/2013

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This study has been conducted at the own initiative of the participants in this comparison with the aim of checking the quality of reference measurements of particulate matter.

Abstract

Inter-laboratory comparison of the determination of PM_{10} in ambient air using filter sampling and weighing

Reference measurements of particulate matter (PM) in ambient air are performed by sampling a known volume of air through a filter for 24 hours. By measuring the mass difference of the filter before and after sampling the concentration of particulate matter may be determined. Procedures for the performance of these measurements are described in European Standards; additional requirements for Dutch monitoring networks are laid down in a Netherlands Technical Agreement.

In order to investigate whether application of this procedure – when applied by different networks – leads to comparable results, an inter-laboratory comparison has been performed. Seven monitoring networks from Belgium and The Netherlands have measured PM_{10} simultaneously from April to July 2012 at a monitoring site of RIVM. A total of 9 reference samplers has been used.

After validation by each of the participants the measurement results have been sent to the pilot laboratory (RIVM) for evaluation. The evaluation has been performed using the international standards ISO 13528 and ISO 5725 part 2, resulting in performance indicators for each network but also for the reference method as such.

The mean PM_{10} concentration level found during the comparison is 16,1 $\mu g/m^3$, the range of concentrations is between 7,2 and 41,6 $\mu g/m^3$. These rather low levels may be explained from prevailing typical Dutch weather conditions.

The evaluations performed show that the results for all networks are generally in good agreement. On average, 96 per cent of all results have so-called z-scores, determined using ISO 13528 by applying a fit-for-purpose standard deviation of 8,3 per cent, that are good.
Application of ISO 5725-2 statistics indicate one set of results to be a straggler for means and one to be a straggler for standard deviation.
Both fin dings imply that all networks have an appropriate level of quality control for the performance of the measurements.

When evaluating the method as a whole, the resulting reproducibility relative standard deviation is 6,8 per cent resulting in an expanded measurement uncertainty of 2,5 $\mu g/m^3$. This is within the uncertainty calculated in prEN 12341:2012 by adding contributions derived from method performance requirements, showing that it is possible overall to meet these requirements.

Lastly, a number of permutations of the implementation of the reference method have been compared. It is noted, however, that the results of these comparisons are to be treated with care because of the low number of samples within single permutations.

Keywords:
particulate matter, PM, reference measurements, comparability

Rapport in het kort

Inter-laboratorium vergelijking van de bepaling van PM_{10} in buitenlucht door monsterneming op filters en filterweging

In het kader van de implementatie van haar referentiefunctie voor luchtkwaliteitsmetingen in Nederland heeft RIVM een vergelijkingsonderzoek georganiseerd voor het meten van fijnstof (PM_{10}) m.b.v. de zogenaamde referentiemethode. Bij deze metingen wordt gedurende 24 uur een bekend volume lucht door een filter gezogen, waarvan de massa voor en na monsterneming wordt bepaald.
Dergelijke metingen vormende basis voor alle metingen van PM_{10} met automatische meetapparatuur, waarvan de resultaten worden gebruikt voor informatie van burgers en de berekening van kengetallen voor rapportage, bijvoorbeeld aan het Ministerie van Infrastructuur en Milieu.

Zes Nederlandse luchtmeetnetten en het Belgische VMM hebben met goede resultaten deelgenomen aan dit onderzoek. Alle meetnetten meten hierbij conform de Europese norm EN 12341. Van alle resultaten blijkt 96 procent een zogenaamde z-score te hebben die als "goed" kan worden geclassificeerd. Slechts 0,6 procent krijgt als kwalificatie "onacceptabel".

De berekende uitgebreide meetonzekerheid (95 procent) bedraagt 2,5 $\mu g/m^3$ bij een 24-uursgemiddelde concentratie van 16 $\mu g/m^3$, d.w.z., 16 procent, waar bij het niveau van de grenswaarde voor 24-uursgemiddelde metingen van 50 $\mu g/m^3$ een eis geldt van maximaal 25 procent.
Dit impliceert dat de deelnemende meetnetten een niveau van kwaliteitscontrole hanteren dat voldoende is om aan de in EN 12341 gestelde eisen te voldoen. De verschillen in de implementatie van de referentie-methoden (type filter, vorm van filter-conditionering, gebruik van al dan niet gekoelde filteropslag) blijken hierop niet of nauwelijks van invloed.

Tenslotte mag uit de goede vergelijkbaarheid tussen de meetnetten worden afgeleid dat een basis bestaat voor de uitwisselbaarheid van meetgegevens tussen deze meetnetten.

Trefwoorden:
fijnstof, PM, referentie-metingen, vergelijkbaarheid

Contents

Summary

Inter-laboratory comparison of the determination of PM_{10} in ambient air using filter sampling and weighing

In order to investigate whether application of the reference method for the measurement of PM_{10} in ambient air described in prEN 12341:2012 – when applied by different networks – leads to comparable results, an inter-laboratory comparison has been organized. Seven monitoring networks from Belgium and The Netherlands have measured PM_{10} simultaneously from April to July 2012 at a monitoring site of RIVM. A total of 9 reference samplers has been used.

After validation by each of the participants the measurement results have been sent to the pilot laboratory (RIVM) for evaluation. The evaluation has been performed using the international standards ISO 13528 and ISO 5725 part 2, resulting in performance indicators for each network but also for the reference method as such.

The mean PM_{10} concentration level found during the comparison is 16,1 µg/m^3, the range of concentrations is between 7,2 and 41,6 µg/m^3. These rather low levels may be explained from prevailing typical Dutch weather conditions.

The evaluations performed show that the results for all networks are generally in good agreement. On average, 96 per cent of all results have so-called z-scores, determined using ISO 13528 by applying a fit-for-purpose standard deviation of 8,3 per cent, that are good.
Application of ISO 5725-2 statistics indicate one set of results to be a straggler for means and one to be a straggler for standard deviation.
Both findings imply that all networks have an appropriate level of quality control for the performance of the measurements.

When evaluating the method as a whole, the resulting reproducibility relative standard deviation is 6,8 per cent resulting in an expanded measurement uncertainty of 2,5 µg/m^3. This is within the uncertainty calculated in prEN 12341:2012 by adding contributions derived from method performance requirements, showing that it is possible overall to meet these requirements.

Lastly, a number of permutations of the implementation of the reference method have been compared. It is noted, however, that the results of these comparisons are to be treated with care because of the low number of samples within single permutations.

1 Introduction

Reference measurements of particulate matter (PM) in ambient air are performed by sampling a known volume of air through a filter for 24 hours. By measuring the mass difference of the filter before and after sampling the concentration of particulate matter may be determined. For this purpose filters are weighed under strict conditions of temperature and relative humidity, as prescribed in European Standards EN 12341:1998 [1] and EN 14907:2005 [2]. These two standards have been found to show considerable overlap in information, but also some potentially conflicting requirements. Current practice is therefore that reference measurements of PM_{10} are performed in accordance with EN 14907 rather than EN 12341:1998.

Apart from the measurements of $PM_{2.5}$ for the determination of the Average Exposure Indicator for the years 2008 to 2010 [3], these standards are rarely used for direct measurements of PM mass concentrations but rather for the evaluation and declaration of equivalence of automated continuous systems for the measurement of PM [4] and for subsequently checking the maintenance of equivalence over time.

During the application of EN 14907 it has been found that the flexibility introduced for practical reasons into this standard may lead to significant differences in results of measurements of $PM_{2.5}$ or PM_{10} [e.g., 5,6].
For this reason both standards have been reviewed and merged into one new EN 12341:2012 [7]. Novelties in the new standard include the selection of a single reference sampler with inlet characteristics based on particle separation efficiency, operating at a flow rate of 2,3 m^3/h.

In order to investigate the comparability and state of the art of the measurement of particulate matter using the reference method an inter-laboratory comparison has been conducted from April to August of 2012. In this comparison PM_{10} has been measured by laboratories that are members of the OLM, a cooperation between monitoring networks operating in Flanders and the Netherlands (in alphabetical order):

- the DCMR (Schiedam, Netherlands) (DCMR);
- the GGD Amsterdam (Amsterdam, Netherlands) (GGD-A);
- the Province of Gelderland (Arnhem, Netherlands) (PGld);
- the Province of Limburg (Maastricht, Netherlands) (PLmb);
- the Province of Noord-Brabant (Netherlands) (PNBr);
- the RIVM (Bilthoven, Netherlands) (RIVM);
- the VMM (Antwerp, Belgium) (VMM).

This report describes the methods used, results and findings of this comparison.

2 Methods

The proposed reference method for the measurement of mass concentrations of the PM_{10} fraction of suspended particulate matter in ambient air consists of [7]:

- drawing ambient air through a particle classifier capable of separating the fraction below PM_{10} (efficiency 50 per cent at 10 μm particle size) when applying a volume flow at ambient conditions of 2,3 m^3/h;
- collection for a period of 24 ± 1 h of the PM_{10} fraction onto a filter that has been conditioned and weighed under strict conditions of relative humidity and temperature;
- determination of the mass of PM_{10} collected by conditioning and weighing under the same conditions as used before sampling;
- calculation of the mass concentration of PM_{10} as the mass by volume –of-sampled-air ratio.

Currently, a wide variety of filters may be used for the collection of PM_{10}, although is has been demonstrated that this may lead to considerable differences in mass concentrations obtained. The Netherlands have therefore agreed that all networks measuring PM should use a single filter type; in addition, before the regular preconditioning the filters used are exposed for 3 weeks to a relative humidity of close to 100 per centrh at a temperature of 20 ± 1 °C in conformity with the Dutch National Agreement NTA 8019 [8].

All participants from the Netherlands have applied the methodology described above for this comparison. The VMM has applied the same procedures but without the exposure of the filters to high relative humidity prior to use.

Installations of equipment and periodic checks have been performed by each network individually, using its own procedures. In addition, each network has provided for the two-weekly transport and changes of its own filters.

Four of the seven networks are EN-ISO 17025 accredited for the full measurement procedure, one is accredited for the sampling procedure and one is accredited for the filter conditioning and weighing.

Table 1 summarizes the equipment and filters used by all participants. In addition, the laboratory responsible for the conditioning and weighing of the filters is indicated.

The comparison has been performed at the monitoring site of the Netherlands National Air Quality Network at Bilthoven (station code 627). This station is characterized as a rural background station.
All equipment has been installed and put into operation in the period of 13 to 24 April 2012. De-installation has been performed in the period of 20 to 31 July 2012.

During the period of 24 April to 20 July all participants have measured simultaneously.

In Annex 1 an overview is given of the meteorological conditions during this period. This overview shows that the comparison has been conducted under typical Dutch spring and summer conditions, with relatively low temperatures and a relatively high level of precipitation. The average temperature for the period in which all participants have performed measurements is 14,9 °C; the average precipitation level is 3 mm per day.

In the following period all networks have performed the necessary weighings and evaluations and have submitted their results to the coordinating network, the RIVM.

Table 1. Overview of equipment, filters used and weighing laboratories

Network	*Sampler*	*Filter*	*Weighing laboratory*
DCMR	Leckel 47/50 SEQ	Whatman QMA cf. NTA 8019	DCMR
GGD-A	Derenda PNS16	Whatman QMA cf. NTA 8019	GGD-A
PGld	Leckel 47/50 SEQ cooled	Whatman QMA cf. NTA 8019	GGD-A
PLmb (1)	Leckel 47/50 SEQ cooled	Whatman QMA cf. NTA 8019	PLmb
PLmb (2)	Leckel 47/50 SEQ cooled	Pall Tissuquartz cf. NTA 8019	PLmb
PNBr	Tecora + Charlie Sentinel	Whatman QMA cf. NTA 8019	Buro Blauw
RIVM	Leckel 47/50 SEQ	Whatman QMA cf. NTA 8019	RIVM
VMM (1)	Leckel 47/50 SEQ	Pall Tissuquartz	VMM
VMM (2)	Leckel 47/50 SEQ cooled	Pall Tissuquartz	VMM

3 Results and evaluations

3.1 Results

3.1.1 General

The results of the measurements are presented in Annex 2 in an anonimized way. Each individual sampler used has been given its own alphanumerical code. Missing values are for results that have not been submitted. No outlying values have been removed prior to data processing.

3.1.2 Data processing

After submission of the results outliers have been removed in consultation with the participants. The remaining data set has been used to calculate robust mean concentrations for each day. This procedure has been used:
- for lack of reference values;
- because all results are obtained using methods that fulfil the requirements of prEN 12341:2012;
- because all networks work in accordance with internationally accepted QA/QC procedures.

The procedure applied is that described in ISO 13528 [9]. After 2 iterations the robust mean values have been reached. These values have subsequently been used for evaluation purposes.

In Figure 1 the course of the mean concentration as a function of the date of measurement is shown. Concentrations are generally low and show no exceedances of the European Union daily limit value of 50 µg/m^3. The overall mean concentration during the comparison is 16,1 µg/m^3, the daily means vary between 7,2 and 41,6 µg/m^3. These low concentrations are due to the meteorological conditions during the comparison.

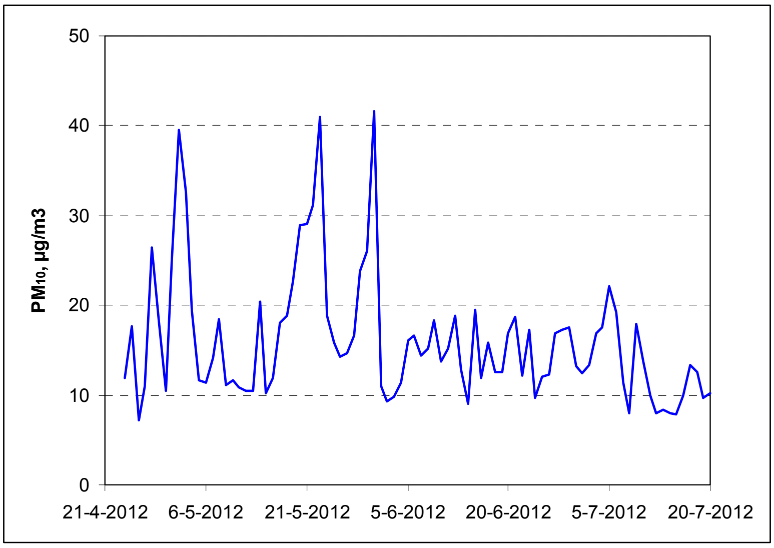

Figure 1. Mean daily concentrations of PM_{10} during the comparison

3.2 Evaluation

3.2.1 Laboratory performance from z-scores

For the evaluation of the results of the comparison firstly the *z*-score model has been applied. The parameter *z* is calculated as follows [9]:

$$z = \frac{x_p - x_{mean}}{\sigma} \qquad \text{(eq. 1)}$$

where

x_p = participant's result
x_{mean} = robust mean value
σ = assigned standard deviation of the results.

An z-score of $-2 \leq z \leq 2$ is an indication of good participant performance.
A z-score of $-3 \leq z < -2$ or $z > 2 \leq 3$ is an indication of questionable performance.
A z-score < -3 or > 3 indicates unacceptable performance.

In this way, a series of 86 – 88 z-scores per participant is obtained: one for each day for which participant's results and a robust mean are available.

Obviously, the magnitudes of the z-scores will be determined by the values of the assigned standard deviations of the results.

ISO 13528 recommends the use of standard deviations that represent "fitness-for-purpose". In a first attempt, the robust standard deviations obtained from the calculations of the mean daily values (see 3.1.2) have been used for this purpose. This results in high numbers of z-scores in excess of the unacceptability criteria. The cause for this is the trimming of extreme values for calculation of the robust mean when applying the ISO 13528 procedure.

In a second attempt the standard deviation has been based on the interval of the values that is supposed to encompass all results. In order to calculate this interval the uncertainty requirement from EU Directive 2008/50/EC [3] has been used: values are required to have a maximum expanded uncertainty of 25 per cent (at the 95 per cent confidence level).
Application of the ISO 13528 premises results in a "fit-for-purpose" relative standard deviation of 8,3 per cent.
When applying this standard deviation in the calculation of the z-scores, it is found that, as a whole, 4,1 per cent of the z-scores is < -2 or >2 and 0,6 per cent is < -3 or > 3. This is acceptable from the viewpoint that $|z| = 2$ represents a 95 per cent confidence interval and $|z| = 3$ a 99 per cent confidence interval.

The resulting numbers of z-scores per category are presented for each sampler in Table 2. In addition, Figure 2 gives a graphical representation of the z-scores for each sampler as a function of the sampling day.

Table 2. z-Scores per sampler per category

Sampler	*z < -3*	*-3 ≤ z < -2*	*-2 ≤ z ≤ 2*	*2 < z ≤ 3*	*z > 3*
A	0	1	87	0	0
B	0	6	80	0	0
C	0	0	88	0	0
D	0	3	76	6	1
E	0	1	85	1	1
F	0	0	81	5	1
G	0	3	83	1	0
H	0	0	87	0	0
I	1	0	85	0	1

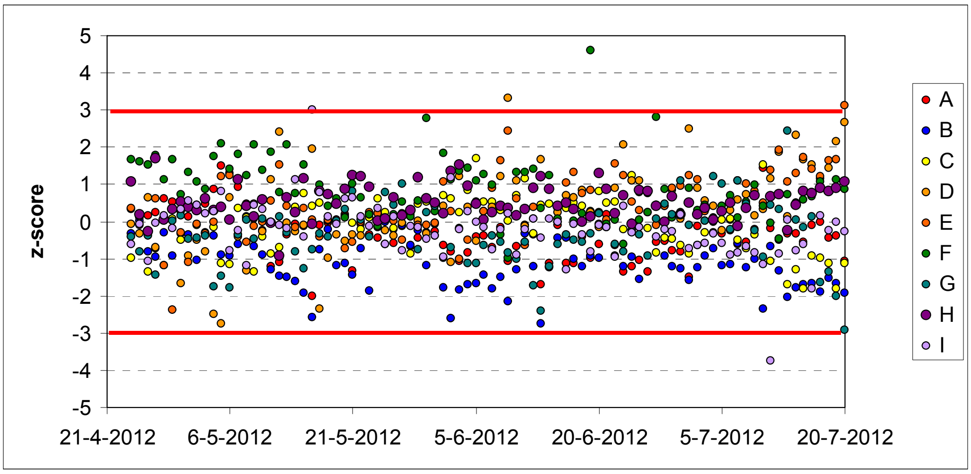

Figure 2. z-Scores per sampler for each measurement day

The tabulated z-scores show that samplers B, D, F have more than 5 per cent scores in categories with |z|> 2.
Of all z-scores, 96 per cent fall into the category "good", which indicates a good network performance on average. Only 0,6 per cent fall into the category "unacceptable".

3.2.2 *Laboratory performance from ISO 5725 evaluation*

Although no replicate measurements of samples are available in this comparison, the statistical approach of ISO 5725-2 [10] may be applied by normalizing all results to the daily mean values and treating the normalized results as replicates. Normalized results are calculated as follows:

$$x_{i,n} = \frac{x_{i,p}}{x_{i,mean}} \qquad \text{(eq. 2)}$$

where

$x_{i,p}$ = sampler's result for day *i*
$x_{i,mean}$ = robust mean for day *i.*

From the normalized results means and standard deviations are calculated that are submitted to Mandel's h- and k-tests for outliers of mean values and standard deviations, respectively, and to Grubb's and Cochran's outliers tests. The results of these calculations are given in Table 3.

Table 3. ISO 5725-2 statistics

Sampler	*Number of replicates*	*Means of $X_{i,n}$*	*Standard deviation*	*Mandel's h*	*Mandel's k*
A	88	0,972	0,054	-0,63	0,84
B	86	0,908	0,054	-1,91	0,83
C	88	0,991	0,061	-0,23	0,94
D	86	1,027	0,091	0,50	1,41
E	88	1,038	0,071	0,72	1,09
F	87	1,071	0,069	1,38	1,07
G	87	0,972	0,069	-0,62	1,07
H	87	1,043	0,037	0,81	0,58
I	87	0,982	0,062	-0,42	0,96

The cells marked in orange indicate the values to be stragglers from Mandel's tests. This indicates that the mean value for sampler B and the standard deviation for sampler D are significantly different from others at the 95 per cent significance level.
Grubb's and Cochran's tests do not indicate any stragglers or outliers.

3.2.3 Method performance

The ISO 5725-2 approach has been extended to calculate performance characteristics for the complete method of determination of PM_{10} in ambient air (repeatability, between-sampler variability and reproducibility).
The results are given in Table 4.

Table 4. Method performance characteristics

Performance characteristic	
Mean of means	1,003
Median of means	1,001
Repeatability standard deviation	0,0467
Between-laboratory standard deviation	0,0490
Reproducibility standard deviation	0,0677

These results show that:

- the ISO 13528 trimming procedure does not lead to a marked deviation of the mean result from 1.
- the method is quite robust: the ratio of reproducibility and repeatability standard deviations is < 2.
- the reproducibility is excellent at a level of 6,8 per cent.

When the reproducibility is converted into units of mass per volume by multiplication with the overall mean concentration observed in the comparison (16,1 $\mu g/m^3$), a reproducibility of 1,09 $\mu g/m^3$ results.

By using a coverage factor of 2,306 for 8 degrees of freedom (95 per cent confidence), the expanded measurement uncertainty is calculated to be 2,5 µg/m^3.
At the level of the daily limit value for PM_{10} (50 µg/m^3) this gives a relative measurement uncertainty of 5,0 per cent.[1] This is lower than the uncertainty given in [7], which, based on a GUM-approach, is calculated to be 7,7 per cent.

This finding implies that the level of quality control applied by all participants in this comparison is such that the requirements of [7] are met.

3.2.4 Additional information

Apart from information about the level of agreement of measurement results of participants and of method performance, the comparison has been used (on a small scale) to collect and compare other information, including:

- potential differences between the 2 filter types used: Whatman QMA and Pall Tissuquartz UP;
- potential differences between samplers with and without cooling of the filter storage compartment(s);
- levels of field blanks collected during the comparison;
- calibration results of samplers by parallel measurements with different flow calibrators.

The first two parts of information are inherent to the comparison, the latter two have been collected separately.

Comparability of filter types

In 7 samplers Whatman QMA filters have been used that are preconditioned according to the NTA 8019. In 3 samplers Pall Tissuquartz UP has been used. The VMM has used Pall Tissuquartz in a cooled and uncooled sampler, using the regular conditioning as prescribed in [7]. The Province of Limburg has used Whatman QMA and Pall Tissuquartz in cooled samplers, but has also preconditioned Pall Tissuquartz filters in accordance with NTA 8019. Comparison of the results obtained with the 2 samplers of the Province of Limburg leads to the plot in Figure 3. The regression equation in Figure 3 is obtained by ordinary least-squares regression.

The mean results obtained for both filters are:

- 16,5 µg/m^3 for Whatman QMA;
- 15,6 µg/m^3 for Pall Tissuquartz.

Resulting in a ratio QMA / Pall of 1,06.

[1] Because all contributions to the weighing uncertainty are absolute in nature, the uncertainty may be "translated" to other mass concentration levels of PM_{10}.

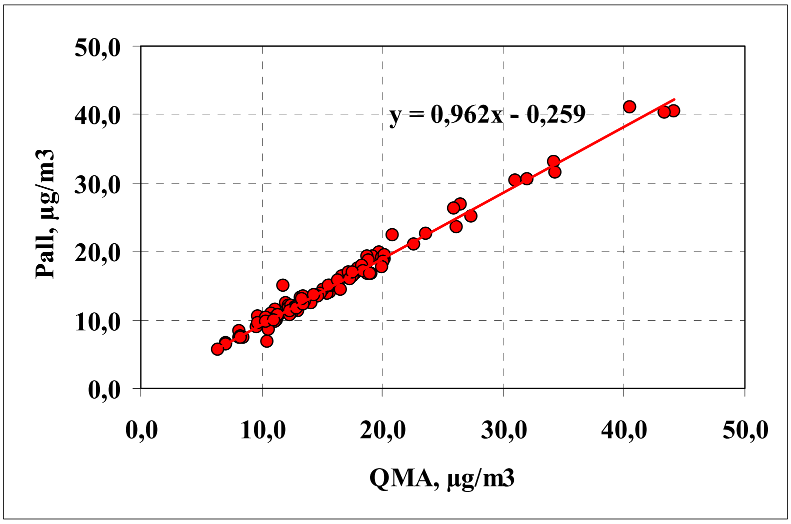

Figure 3. Results for Pall Tissuquartz vs. Whatman QMA

Comparison of results for preconditioned and regularly conditioned-only Pall Tissuquartz filters leads to the plot in Figure 4.

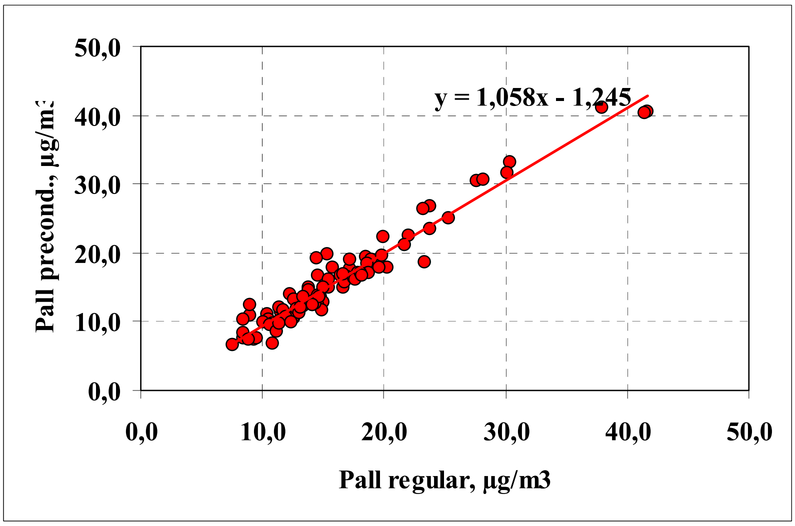

Figure 4. Results for Pall Tissuquartz regular vs. preconditioned

The mean results obtained for both filters are:
- 16,2 µg/m^3 for Pall Tissuquartz regular;
- 15,9 µg/m^3 for Pall Tissuquartz preconditioned.

Resulting in a ratio regular / preconditioned of 1,02.

When all results available for both Whatman QMA and Pall Tissuquartz are averaged, regardless of filter treatment, the plot in Figure 5 results.

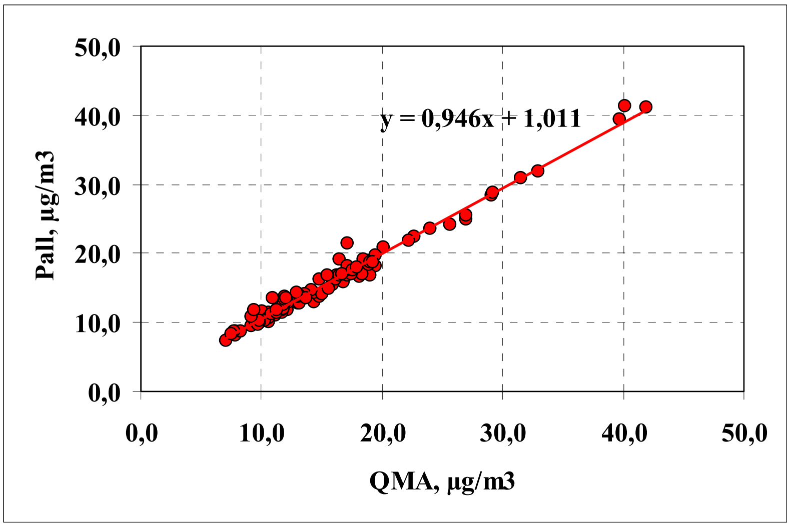

Figure 5. Mean results for Whatman QMA vs. mean results for Pall Tissuquartz

The mean results obtained for both filters are:

- 15,9 µg/m^3 for Whatman QMA;
- 16,1 µg/m^3 for Pall Tissuquartz.

Resulting in a ratio QMA / Pall of 0,99.

Although based on a limited number of experimental results, these comparisons tend to show that:

- when both filter types are preconditioned cf. NTA 8019, the mass increase for Whatman QMA on average is higher than that for Pall Tissuquartz; similar findings have been obtained when using regularly conditioned filters [11]
- preconditioning of Pall Tissuquartz filters leads to a marginal decrease in mass of about 2 per cent on average when compared to regularly conditioned Pall filters
- comparison of all results averaged for Whatman QMA and Pall Tissuquartz leads to similar results on average; however, the relationship between the results has a slope of 0,95 and an intercept of +1; this indicates that Whatman QMA preconditioned has a relatively higher mass increase but that an "offset" exists, most likely in the form of a higher blank level for Pall Tissuquartz (*vide infra*).

<u>Comparability of results for samplers with and without cooled filter storage</u>

Comparison of results for Pall Tissuquartz filters stored in a cooled and uncooled ("regular") sampler leads to the plot in Figure 6.

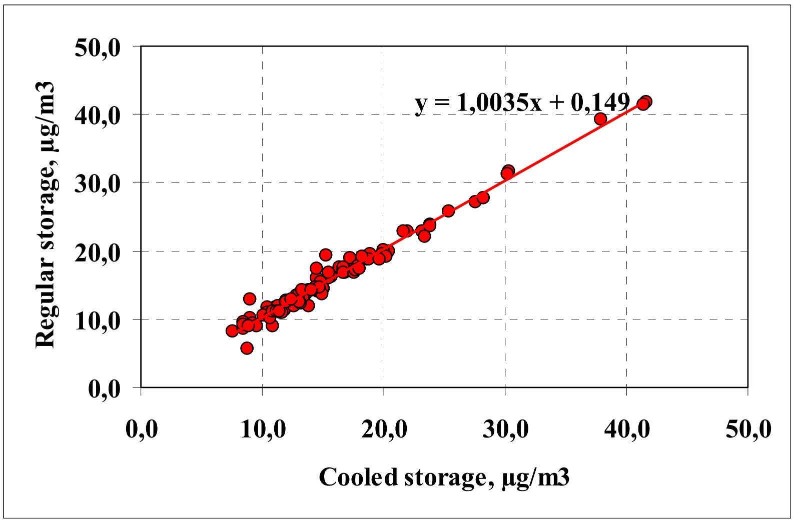

Figure 6. Results for Pall Tissuquartz with cooled and regular filter storage in the sampler

The mean results for both types of storage are 16,2 µg/m^3 for cooled storage, and 16,4 µg/m^3 for regular storage (ratio of means = 0,99). The application of cooled storage has no apparent effect on the loss of semi-volatile materials from the filters. This is readily explained from the moderate temperature conditions during the comparison, with a maximum daily mean concentration of 22 °C.

Field blank levels

For all samplers, results for field blanks collected during the comparison have been made available. The results are summarized below for each individual sampler in Table 5.

Table 5. Field blank values: mean and extremes.

a. Whatman QMA preconditioned		
Sampler	*Mean field blank (µg)*	*Range (µg)*
A	-31	-67 to +1
B	7	-7 to +26
C	-3	-30 to +26
F	24	+7 to +51
G	-6	-40 to +10
H	43	+4 to +117

b. Pall Tissuquartz regularly conditioned		
Sampler	*Mean field blank (µg)*	*Range (µg)*
D	38	-20 to +83
E	65	+34 to +104

c. Pall Tissuquartz preconditioned		
Sampler	*Mean field blank (µg)*	*Range (µg)*
I	39	-21 to +99

These figures show that preconditioning of Whatman QMA filters generally results leads in relatively low blank values, on average within the requirement of < 60 µg [7]. However, frequently negative values are found, implying that the field blank has lost mass – most likely water – upon storage in the sampler and subsequent storage in the weighing room. For samplers H and I the network reports a potential problem of contamination due to the construction of the filter storage compartment in the sampler. However, this has no apparent effect on the measurement results as judged by their performance scores.

Regularly conditioned Pall Tissuquartz filters show relatively higher blank values, with exceedances of the < 60 µg criterion.

As a whole, the blank values for most individual samplers show remarkably high ranges, with up to 120 µg difference between low and high levels.

Comparability of results for of flow measurements

During the comparison all networks have brought their flow field calibration equipment to the monitoring site for comparison with the flow field calibrator of the pilot laboratory. A series of bilateral comparisons has been performed by connecting the calibrators to the inlets of samplers operating at 2,3 m^3/h at ambient conditions. Where necessary network operators have converted the measured flows to ambient conditions using their own temperature and pressure measurement values. The results of the comparisons are shown as averages of values relative to the flow measured by the pilot laboratory in Table 6.

Table 6. Results of flow comparisons at 2,3 m^3/h nominally.

Network	*Flow ratio*
DCMR	1,008
GGD-A	0,996
PGld	0,982
PLmb	0,959
PNBr	0,979
VMM	1,009

These results show flow measurements generally to be in good agreement (within ±2 per cent), with the exception of those of the pilot laboratory (RIVM) and the Province of Limburg. However, when the comparison has been repeated using a sampler under laboratory conditions (20 °C) no significant differences have been observed between the results of both devices. The differences found in the field therefore remain unexplained.
It is noted that the uncertainties of the flow measurements include contributions from calibrations and from potential effects of field conditions.

4 Conclusions

Laboratory performance
Overall, the results of the individual network performance evaluations are satisfactory. When using a fit-for-purpose standard deviation of 8,3 per cent, derived from the maximum uncertainty criterion of 25 per cent [3], 96 per cent of all z-scores fall into the category "good", which indicates a good network performance on average.
Only 0,6 per cent scores fall into the category "unacceptable".

The evaluation based on ISO 5725 part 2 statistics shows similar results; only 2 Mandel's performance indicators qualify as "stragglers", no outliers are found.

Method performance
The method performance, expressed as the repeatability and reproducibility relative standard deviations of all normalized measurement results, calculated according to ISO 5725 part 2 are 4,7 per cent and 6,8 per cent, respectively. The ratio of both standard deviations indicates that the method is quite robust.

In addition, when comparing this uncertainty with that calculated in [7], which is based largely on performance requirements for the application of the reference method for the measurement of PM_{10}, it is found to compare favourably. This indicates that all networks apply an appropriate level of quality control to their measurements.

Filter type, conditioning and storage
Because of the use of different filter types (Whatman QMA and Pall Tissuquartz), different procedures for filter conditioning (with and without preconditioning at relatively high relative humidities) and filter storage (in cooled or regular sampler filter storage compartments), additional information has become available on the comparability of these parameters.
However, since each permutation only has a limited number of results, the results of the comparisons need to be considered as indicative.

<u>Filter type</u>

The results obtained show that on average the use Whatman QMA leads to higher results than that of Pall Tissuquartz. In this case both filter types have been preconditioned at high relative humidities.

<u>Filter conditioning</u>

Two networks have used Pall Tissuquartz filters, one with preconditioning and one without. In both cases, cooled filter storage has been used.
On average it is found that the preconditioning slightly reduces the concentration levels measured. When extrapolating the results it is noted that filters without preconditioning apparently have a higher mass level at zero PM_{10} concentrations.

It is found that preconditioning the Whatman QMA filters generally results in relatively low field blank levels. However, regularly negative field blank level are observed, which may be an indication of the loss of water during storage.

Filter storage

One network has used a cooled and non-cooled sampler of the same type with one common filter type and conditioning procedure. A comparison of the results obtained for both types of filter storage shows no significant differences.
Most likely this is due to the meteorological conditions during the comparison (relatively low temperatures).

Flow measurement comparison
A series of bilateral comparisons has shown that at the nominal flow of 2,3 m3/h flow measurements are comparable to within ± 2 per cent, with the exception of one. However, this appears to be due to hitherto unexplained differences between the behaviour of flow calibrators in the field and under laboratory conditions.
Note that the uncertainties of the flow measurements include contributions from calibrations and from potential effects of field conditions.

References

[1] EN 12341: 1998. Air quality – Determination of the PM_{10} fraction of suspended particulate matter – reference method and field test procedure to demonstrate reference equivalence of measurement methods.

[2] EN 14907: 2005. Ambient air quality- Reference gravimetric measurement method for the demonstration of the $PM_{2,5}$ mass fraction of suspended particulate matter.

[3] EU Directive 2008/50/EC on ambient air quality and cleaner air for Europe.

[4] Guidance to the Demonstration of Equivalence of Ambient Air Monitoring Methods, ed. January 2010. Available at http://ec.europa.eu/environment/air/quality/legislation/assessment.htm.

[5] EUR 24851 EN: 2011. A quality assurance and control program for PM2.5 and PM10 measurements in European air quality monitoring networks.

[6] LANUV Fachbericht 26: 2010. PM2,5-Vergleichsmessungen der deutschen Bundesländer im Rahmen der STIMES-Arbeitsgruppe.

[7] CEN/TC 264 WG 15. prEN 12341 version April 2012.

[8] NTA 8019: 2008. Luchtkwaliteit - Meeteisen voor fijnstofmetingen.

[9] ISO 13528: 2005. Statistical methods for use in proficiency testing by inter-laboratory comparisons.

[10] ISO 5725:1994 part 2. Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method.

[11] VMM: 2008. Comparative PM10 and PM2.5 Measurements in Flanders, Period 2006-2007.

Annex 1 – Meteorological conditions during the comparison[2]

Date	Pressure (kPa)	Temperature (°C)	RH (%)	Wind velocity (m/s)	Wind direction (°)	Precipitation (mm)	Precipitation duration (h)
1-4-2012	1018,1	4,9	77	2,8	297	0	0
2-4-2012	1010	8,1	79	1,8	264	0	0
3-4-2012	1003,1	9	71	1,9	193	0,7	0,7
4-4-2012	1009,1	7,5	89	3	53	-0,1	0
5-4-2012	1018	5,9	75	4,1	40	0	0
6-4-2012	1012,6	6	68	2,4	305	0,4	1,5
7-4-2012	1010,1	5,3	70	3,2	349	0,3	0,9
8-4-2012	1012,3	6,2	73	3,2	212	0,6	2,2
9-4-2012	998,7	9	93	5,2	216	9,2	14
10-4-2012	992,4	9,5	89	4,6	202	4,1	8,7
11-4-2012	998,3	7,9	83	3,4	218	0,2	0,6
12-4-2012	1002,4	7,1	84	1,9	250	1,7	0,9
13-4-2012	1005,1	7,3	85	1,8	300	3,5	1,4
14-4-2012	1006,6	7,6	75	2,8	346	0	0
15-4-2012	1015,6	6,6	71	4,5	4	0,2	0,6
16-4-2012	1021,7	5	69	3,1	335	0	0
17-4-2012	1005,9	4,9	79	5,3	189	2,3	2,6
18-4-2012	990,8	8,4	78	5,5	167	2,4	1,8
19-4-2012	988,9	8,9	75	4,7	177	0,4	1
20-4-2012	994,9	8,2	81	3,5	193	1,4	0,5
21-4-2012	997,8	7,8	83	3,8	211	3,1	3,3
22-4-2012	1003	8,8	79	4,4	215	0,8	0,8
23-4-2012	998,8	9,4	76	4,5	181	2,2	5,3
24-4-2012	995,6	9,7	80	3	222	0,4	1,3
25-4-2012	996,9	10	82	5,4	167	1,2	1,9
26-4-2012	1001,9	11,4	77	6,1	193	7,1	2,7
27-4-2012	1014,1	12,3	76	4,3	208	-0,1	0
28-4-2012	1012,8	10,5	91	5	34	5,3	2,7
29-4-2012	1007,8	12,5	84	3,2	67	-0,1	0
30-4-2012	1015,8	15,8	66	3,5	70	0	0
1-5-2012	1017,1	14,9	81	3,1	40	3,5	2,5
2-5-2012	1015	14,3	86	2,5	330	1,6	1,8
3-5-2012	1010,3	12,5	88	3	205	0	0
4-5-2012	1005	9,9	85	3,1	260	0,1	0,2
5-5-2012	1008,1	8,6	76	3,5	40	0,2	0,8
6-5-2012	1013	8,1	69	3,8	48	0	0
7-5-2012	1017,2	9,9	68	3	136	0	0
8-5-2012	1010,1	13,6	82	3,2	178	9,7	7,2
9-5-2012	1011,4	15,7	88	3,1	212	12,3	3,4
10-5-2012	1013,4	18,3	87	5,2	208	10,1	7,4
11-5-2012	1021,3	13,8	77	5,3	256	-0,1	0

[2] Source: KNMI, station De Bilt.

Date	Pressure (kPa)	Temperature (°C)	RH (%)	Wind velocity (m/s)	Wind direction (°)	Precipitation (mm)	Precipitation duration (h)
12-5-2012	1034,5	8,7	73	2,9	324	0,2	0,1
13-5-2012	1030,1	8,8	73	1,7	223	0	0
14-5-2012	1016,4	11,1	69	3,7	233	-0,1	0
15-5-2012	1010,7	8,9	83	3,2	288	4,9	3,6
16-5-2012	1022,7	8,2	70	4	303	0,9	0,9
17-5-2012	1018,9	10,4	64	3	142	0	0
18-5-2012	1007,9	13,4	74	3,5	143	1,5	1
19-5-2012	1010,6	15,4	74	2,8	235	-0,1	0
20-5-2012	1007	16,4	88	2,5	8	5	5,2
21-5-2012	1002,5	19	80	3,2	346	0	0
22-5-2012	1008,8	21,4	74	2,6	354	0	0
23-5-2012	1020,7	20,3	82	1,7	231	22,5	0,6
24-5-2012	1025,5	22,4	65	3	72	1	1,5
25-5-2012	1025,5	20,8	50	6,2	72	0	0
26-5-2012	1022	20,2	50	4,2	76	0	0
27-5-2012	1019,2	20,5	61	3	31	0	0
28-5-2012	1016	18,3	74	3	316	0	0
29-5-2012	1016,4	13,6	84	2	347	-0,1	0
30-5-2012	1018,1	16,2	73	1,7	294	0	0
31-5-2012	1016	14,4	93	3	268	10,9	5,8
1-6-2012	1018,5	12,7	78	2,6	323	0,1	0,4
2-6-2012	1016,7	11,8	67	2,4	10	0	0
3-6-2012	1009,9	9,1	89	2,6	61	8,6	9
4-6-2012	1012,3	9,1	87	2,6	13	9	8,7
5-6-2012	1016,4	11	70	2,1	184	0	0
6-6-2012	1005,2	13,9	85	3,5	190	8,1	9
7-6-2012	1002,2	16,2	82	3,3	186	1,7	2,8
8-6-2012	1004,2	15,2	68	6,6	219	0,5	0,6
9-6-2012	1010,1	13,3	67	6,4	224	-0,1	0
10-6-2012	1009,3	14,9	68	2,8	229	-0,1	0
11-6-2012	1002,7	14,8	81	2,5	84	4,6	3,1
12-6-2012	1006,2	13,6	81	3,5	7	0	0
13-6-2012	1015,7	11,7	70	2,5	314	0	0
14-6-2012	1019	12,5	65	1,8	36	0	0
15-6-2012	1012,5	14,9	84	4,1	175	8,7	7,4
16-6-2012	1012,4	16,7	69	5	217	-0,1	0
17-6-2012	1018,5	15,6	71	4,7	225	-0,1	0
18-6-2012	1016,3	14,4	81	2,9	312	10,2	2,2
19-6-2012	1019,8	15,3	74	1,8	155	0	0
20-6-2012	1015,8	17,7	74	3,3	31	0	0
21-6-2012	1009,9	17,8	82	4,2	128	16,9	3,1
22-6-2012	1014,6	15,4	71	6,3	215	5,4	1,2
23-6-2012	1020,5	15,7	68	5,1	227	0	0
24-6-2012	1011,9	13,7	84	4,5	232	11,4	9,8
25-6-2012	1016,3	14,3	79	4	274	0	0
26-6-2012	1020,9	16,2	72	1,9	318	0	0
27-6-2012	1017	18	88	2,6	245	0,7	1,1
28-6-2012	1008,1	22,3	77	3,3	148	-0,1	0

Date	Pressure (kPa)	Temperature (°C)	RH (%)	Wind velocity (m/s)	Wind direction (°)	Precipitation (mm)	Precipitation duration (h)
29-6-2012	1008,2	19,1	69	3,2	220	0,3	0,3
30-6-2012	1011,1	19,1	71	3,8	218	4,9	2,2
1-7-2012	1015,9	15,4	70	4,6	230	0,2	0,5
2-7-2012	1018,8	16,5	64	2,8	174	0	0
3-7-2012	1016,5	19	72	2,3	182	0	0
4-7-2012	1012,4	21,2	71	2,5	136	0	0
5-7-2012	1009,2	21,9	75	2,1	52	0,4	1
6-7-2012	1008,8	19	82	3,1	192	-0,1	0
7-7-2012	1010,6	18,4	74	2,5	182	2,6	1,2
8-7-2012	1005,6	16,8	89	3,1	216	19,9	6,6
9-7-2012	1010,8	16,6	83	4,3	237	0,7	0,4
10-7-2012	1011,5	16,4	79	3,7	216	0,4	1,1
11-7-2012	1009,9	15,6	75	5	228	2,7	0,8
12-7-2012	1011,7	14,5	76	3,7	249	7,8	5,9
13-7-2012	1003,3	15,5	83	3,5	214	5	10,6
14-7-2012	1003,9	14,9	85	3,3	241	3,7	0,8
15-7-2012	1014,3	15,3	75	2,5	272	0,2	0,4
16-7-2012	1019,3	14,9	89	4,8	227	20,7	13,3
17-7-2012	1020	17,5	83	3,8	260	0,4	0,2
18-7-2012	1012,5	17,1	80	5,9	234	1,5	1,8
19-7-2012	1008,5	15,3	82	4,3	264	9	4,2
20-7-2012	1016	14,6	73	2	321	-0,1	0
21-7-2012	1023,4	13,3	76	2,1	344	0	0
22-7-2012	1027,8	15	74	1,3	173	0	0
23-7-2012	1024,6	18,8	68	2,3	173	0	0
24-7-2012	1018,5	20,9	66	1,5	127	0	0
25-7-2012	1015,8	22,3	67	2,2	343	0	0
26-7-2012	1017,8	21,2	71	2,9	39	0	0
27-7-2012	1011,2	21,4	76	2	343	8,3	2,5
28-7-2012	1008,6	18,5	84	2	347	0,8	0,4
29-7-2012	1012	16,4	73	3	247	4,9	1,1
30-7-2012	1015,2	15,4	72	3,8	231	1,2	0,8
31-7-2012	1016,8	15,3	84	3	195	0,9	1,7

Annex 2 – All measurement results

All results are in μg/m3 at ambient conditions

Date	A	B	C	D	E	F	G	H	I	Mean
17-4-2012	21,1	20,9		20,4	20,0					
18-4-2012	11,3	11,3		11,1	11,8					
19-4-2012	8,1	8,1	6,1	8,7	5,7	9,7				
20-4-2012	17,6	17,6	17,7	15,8	16,3	19,6		18,4	17,7	
21-4-2012	13,6	13,4	13,3	12,3	12,6	16,1		15,6	14,0	
22-4-2012	16,6	15,2	15,9	14,6	16,1	18,5		17,8	16,7	
23-4-2012	13,7	11,1	11,9	12,6	12,7	15,2		13,3	13,3	
24-4-2012	12,3	11,6	11,0	11,9	12,3	13,6	11,5	13,0	11,3	12,0
25-4-2012	17,8	17,7	17,9	17,2	17,5	20,1	16,5	18,0	17,6	17,7
26-4-2012	7,3	6,7	6,4	7,6	8,1	8,1	7,0	7,0	6,6	7,2
27-4-2012	11,3	10,2	10,4	10,4	11,6	12,7	9,7	12,6	11,0	11,1
28-4-2012	27,8	25,8	26,6	23,8	23,9	28,9	26,6	26,5	26,8	26,4
29-4-2012	18,9	16,7	18,2	6,9	14,5	20,6	18,1	18,6	17,9	18,1
30-4-2012	10,7	10,2	10,1	9,0	10,2	11,1	10,6	11,0	10,8	10,5
1-5-2012	25,4	24,4	26,1	23,2	22,9	28,0	24,2	26,0	26,3	25,2
2-5-2012	41,0	36,1	39,2	37,9	39,4	43,0	38,1	40,6	41,1	39,6
3-5-2012	33,9	31,8	31,7	30,4	31,7	34,9	31,5	34,3	33,2	32,6
4-5-2012	20,7	18,7	19,1	15,3	19,3	22,2	16,5	19,8	19,8	19,3
5-5-2012	13,1	10,8	10,6	9,0	12,9	13,7	10,3	12,1	12,5	11,7
6-5-2012	12,6	10,5	10,3	12,0	12,6	12,8	9,7	11,5	10,7	11,4
7-5-2012	15,2	13,4	14,6	13,7	13,7	16,3	13,7	15,5	13,9	14,2
8-5-2012	19,2	18,6	18,6	16,4	17,6	20,3	18,5	19,1	16,6	18,4
9-5-2012	11,1	10,4	9,8	11,5	11,0	13,0	10,6	11,2	11,5	11,1
10-5-2012	11,4	10,8	11,3	11,4	11,7	13,0	11,7	12,3	12,0	11,7
11-5-2012	9,8	9,7	10,1	11,9	11,3	12,5	10,8	10,8	10,8	10,8
12-5-2012	9,6	9,2	9,9	12,6	11,9	11,2	10,6	9,7	10,4	10,5
13-5-2012	10,2	9,2	10,5	10,5	10,9	12,3	10,3	11,1	10,4	10,5
14-5-2012	20,3	17,7	21,1	20,0	20,2	21,7	19,1	20,8	22,3	20,4
15-5-2012	10,1	8,6	10,9	10,1	10,5	11,5	9,2	11,2	9,9	10,2
16-5-2012	9,9	9,4	12,3	13,9	12,0		11,2	11,9	14,9	11,9
17-5-2012	17,9	16,9	18,8	14,5	17,4	19,5	17,6	19,3	19,3	18,1
18-5-2012	18,8	18,5	18,8	17,3	18,9	19,5	18,1	20,0	19,0	18,8
19-5-2012	22,7	20,5	22,7	22,0	22,8	23,9	22,7	23,6	22,6	22,7
20-5-2012	29,3	26,2	29,2	27,6	27,1	29,2	29,7	31,0	30,4	28,9
21-5-2012	25,8	25,6	29,0	28,2	27,7	31,5	31,1	32,0	30,6	29,1
22-5-2012	31,0	29,3	30,6	30,2	31,2	32,8	31,3	34,3	31,5	31,2
23-5-2012	39,7	34,6	41,3	41,6	41,8	41,6	39,5	44,1	40,5	41,0
24-5-2012	18,1	18,5	19,8	18,5	19,1	18,6		18,8	19,3	18,8
25-5-2012	15,0	15,8	16,1	15,5	15,9	15,9	16,8	15,9	15,0	15,8
26-5-2012	13,6	14,1	14,1	13,8	14,3	14,5	14,8	15,1	14,5	14,3
27-5-2012	14,5	14,6	14,9	14,6	14,1	15,3	15,9	14,9	13,7	14,7
28-5-2012	15,7	15,7	15,4	16,8	16,9	16,8	17,0	17,0	16,4	16,6
29-5-2012	22,9	23,3	24,7	23,8	23,6	24,9	22,3	26,1	23,5	23,8
30-5-2012	25,4	23,5	27,2	25,4	25,9	32,1	26,6	27,4	25,0	26,1
31-5-2012	40,6	40,3	41,5	41,4	41,5	42,5	43,1	43,4	40,2	41,6
1-6-2012	10,4	9,3	11,6	11,4	11,9	12,6	10,1	11,2	10,1	11,0
2-6-2012	8,7	7,3	9,8	8,5	9,6	10,2	8,8	10,4	10,3	9,3

Date	A	B	C	D	E	F	G	H	I	Mean
3-6-2012	9,0	8,3	10,1	10,9	9,0	11,0	9,7	11,1	9,7	9,9
4-6-2012	10,5	9,7	12,1	11,7	10,9	12,7	10,3	12,2	11,6	11,3
5-6-2012	15,6	13,9	18,4	15,5	16,9	17,7	14,7	16,8	16,1	16,2
6-6-2012	16,1	14,6	17,4	17,0	17,0	18,4	15,8	16,7	16,4	16,6
7-6-2012	13,5	12,2	15,0	14,8	15,5	15,6	13,9	14,7	13,5	14,4
8-6-2012	15,0	13,3	15,6	16,7	17,3	14,7	14,5	15,7	14,9	15,2
9-6-2012	16,7	15,1	18,4	23,4	22,1	16,8	17,0	18,9	18,7	18,3
10-6-2012	13,0	12,3	14,6	15,1	14,5	15,3	12,6	14,0	12,8	13,8
11-6-2012	15,1	14,8	16,6	15,0	14,4	16,8	14,5	15,6	14,9	15,2
12-6-2012	18,3	16,9	18,3	19,0	19,4	21,0	16,1	20,2	19,0	18,8
13-6-2012	11,0	9,9	13,3	14,6	14,1	13,2	10,3	14,1	12,4	12,9
14-6-2012	8,1	8,0	9,3		9,6	9,9	8,1	9,6	9,0	9,0
15-6-2012	19,4	18,8	20,1	19,9	19,5	19,6	18,5	20,2	19,5	19,5
16-6-2012	10,8	11,5	12,2	12,6	12,9	12,4	11,6	12,4	10,6	11,9
17-6-2012	14,4	14,5	16,5	16,7	17,6	15,9	15,2	16,3	15,8	15,9
18-6-2012	12,1	11,2	12,9	13,4	13,3	12,7	11,9	12,5	12,1	12,5
19-6-2012	11,6	11,7	12,9	12,9	13,5	17,5	12,3	12,8	11,8	12,6
20-6-2012	16,1	15,6	18,1	16,7	16,8	17,3	17,2	18,7	16,8	16,9
21-6-2012	19,0	18,5	19,5	18,7	18,8	18,7	18,5	20,1	18,5	18,8
22-6-2012	11,5	11,3	12,7	13,1	13,8	12,2	11,5	12,4	11,2	12,2
23-6-2012	15,3	15,6	17,0	20,2	19,1	16,4	16,1	18,3	17,9	17,3
24-6-2012	8,8	9,0	10,7	10,6	10,2	9,7	9,5	9,7	9,5	9,7
25-6-2012	11,0	10,5	12,9	13,2	12,4	12,1	11,7	12,9	11,9	12,1
26-6-2012	10,9		12,1	13,1	12,6	12,6	11,4	13,1		12,3
27-6-2012	16,2		15,8	17,6	16,8	20,9	17,1	17,5	16,4	16,9
28-6-2012	16,5	16,0	16,7	17,9	17,9	18,6	18,0	17,3	17,0	17,3
29-6-2012	16,4	15,8	16,8	18,7	18,8	17,7	17,8	18,5	17,1	17,5
30-6-2012	12,4	11,9	12,6	13,3	14,3	13,5	14,5	13,5	13,5	13,3
1-7-2012	10,8	10,8	11,5	14,9	13,7	13,3	12,3	12,9	11,6	12,4
2-7-2012	13,3	12,0	12,7	14,4	14,6	13,7	13,6	13,6	12,7	13,4
3-7-2012	16,9	15,5	16,4	17,6	17,2	17,5	16,9	17,4	16,0	16,9
4-7-2012	17,6	16,4	17,0	18,1	17,5	17,6	19,3	17,5	17,0	17,5
5-7-2012	21,5	20,0	22,3	21,7	22,8	22,9	24,0	22,7	21,1	22,2
6-7-2012	19,3	17,4	19,3	19,7	18,8	20,3	18,9	20,0	17,8	19,2
7-7-2012	10,8	10,9	10,9	12,0	12,5	11,5	12,5	11,3	10,6	11,4
8-7-2012	7,6	7,1	7,5	8,4	8,6	8,3	8,2	8,2	7,5	7,9
9-7-2012	17,7	16,5	17,5	18,2	19,2	19,5	17,6	19,0	16,7	18,0
10-7-2012	15,4	11,1	15,5	14,1	14,3	14,2	12,6		12,4	13,7
11-7-2012	9,7	9,3	9,0	10,8	11,0	9,8	10,3	10,5	6,8	9,9
12-7-2012	7,7	7,1	7,7	9,3	9,3	7,7	8,2	8,5	7,4	8,0
13-7-2012	8,2	7,0	7,2	8,5	9,1	8,9	10,1	8,2	8,4	8,4
14-7-2012	7,8	6,8	7,2	9,6	8,9	8,4	8,1	8,3	7,5	8,0
15-7-2012	7,4	6,7	6,6	8,9	8,9	8,2	7,7	8,3	7,4	7,8
16-7-2012	9,9	8,6	9,2	11,2	11,1	10,5	10,1	10,6	8,5	10,0
17-7-2012	13,4	11,3	12,3	14,7	14,7	14,6	11,6	14,4	13,6	13,4
18-7-2012	12,1	11,0	11,4	14,1	14,3	13,4	11,2	13,5	12,3	12,6
19-7-2012	9,4	8,4	8,2	11,4	11,0	10,6	8,1	10,4	9,7	9,7
20-7-2012	9,3	8,5	9,2	12,4	12,8	10,9	7,7	11,1	9,9	10,2
21-7-2012	5,8	5,9	5,5			7,2	4,6	7,1	6,4	

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