

LAQM Helpdesk – March 2017

Summary of Laboratory Performance in AIR NO₂ Proficiency Testing Scheme (April 2015 – February 2017).

Reports are prepared by LGC for BV/NPL on behalf of Defra and the Devolved Administrations.

Background

AIR is an independent analytical proficiency-testing (PT) scheme, operated by LGC Standards and supported by the Health and Safety Laboratory (HSL). AIR PT is a new scheme, started in April 2014, which combined two long running PT schemes: LGC Standards STACKS PT scheme and HSL WASP PT scheme.

AIR offers a number of test samples designed to test the proficiency of laboratories undertaking analysis of chemical pollutants in ambient indoor, stack and workplace air. One such sample is the AIR NO₂ test sample type that is distributed to participants in a quarterly basis.

AIR NO₂ PT forms an integral part of the UK NO₂ Network's QA/QC, and is a useful tool in assessing the analytical performance of those laboratories supplying diffusion tubes to Local Authorities for use in the context of Local Air Quality Management (LAQM). With consent from the participating laboratories, LGC Standards provides summary proficiency testing data to the LAQM Helpdesk for hosting on the web-pages at <http://laqm.defra.gov.uk/diffusion-tubes/qa-qc-framework.html>. This information will be updated on a quarterly basis following completion of each AIR PT round.

Defra and the Devolved Administrations advise that diffusion tubes used for Local Air Quality Management should be obtained from laboratories that have demonstrated satisfactory performance in the AIR PT scheme. Laboratory performance in AIR PT is also assessed, by the National Physical Laboratory (NPL), alongside laboratory data from the monthly NPL Field Intercomparison Exercise carried out at Marylebone Road, central London.

The information is used to help the laboratories to identify if they have problems and may assist devising measures to improve their performance and forms part of work for Defra and the Devolved Administrations under the Local Air Quality Management Services Contract.

AIR NO₂ PT Scheme overview

Purpose of scheme

The AIR PT scheme uses laboratory spiked Palmes type diffusion tubes to test each participating laboratory's analytical performance on a quarterly basis and continues the format used in the preceding WASP PT scheme. Such tubes are not designed to test other parts of the measurement system e.g. sampling. Every quarter, roughly January, April, July and October each year, each laboratory receives four diffusion tubes doped with an amount of nitrite, known to LGC Standards, but not the participants. At least two of the tubes are usually duplicates, which enables precision, as well as accuracy, to be assessed. The masses of nitrite on the spiked tubes are different each quarter, and reflect the typical analytical range encountered in actual NO₂ ambient monitoring in the UK.

LAQM Helpdesk – March 2017

Preparation of test samples

Diffusion tubes are spiked using a working nitrite solution prepared from a stock solution. The concentration of this stock solution is initially assayed using a titrimetric procedure. All steps in the subsequent test sample production process, involving gravimetric and volumetric considerations, are undertaken using calibrated instruments employing traceable standards. As an additional cross check, 12 spiked Palmes tubes are picked at random from each spike loading level and submitted to a third party laboratory which is accredited to ISO 17025 to undertake this analysis using an ion chromatographic procedure.

In summary, the tube spiking precision is calculated to be better than 0.5 %, expressed as a standard deviation, and this is derived from repeat gravimetric checking of the pipette device used to spike the test samples. The calculated spike values, derived from titrimetric, gravimetric and volumetric considerations, are found to be typically within ± 3 % of results obtained by the third party laboratory using an ion chromatographic analytical procedure.

Scheme operation

The participants analyse the test samples and report the results to LGC Standards via their on-line PORTAL data management system. LGC Standards assign a performance score to each laboratory's result, based on how far their results deviate from the assigned values for each test samples. The assigned values are best estimates of the levels of nitrite doped onto the test sample tubes and are calculated from the median of participant results, after the removal of test results that are inappropriate for statistical evaluation, e.g. miscalculations, transpositions and other gross errors. At the completion of the round, laboratories receive a report detailing how they have performed and how their results relate to those of their peers.

Performance scoring

The z-score system is used by LGC to assess the performance of laboratories participating in the AIR PT NO₂ scheme.

The Z_{score} may be defined as:

$$Z_{\text{score}} = \frac{(x_{\text{lab}} - \bar{x}_{\text{assigned}})}{\sigma_{\text{SDPA}}}$$

Where:

x_{lab} = participant result from a laboratory

$\bar{x}_{\text{assigned}}$ = assigned value

σ_{SDPA} = standard deviation for performance assessment (currently set at 7.5 % of $\bar{x}_{\text{assigned}}$)

LAQM Helpdesk – March 2017

Performance score interpretation

A Z_{score} is interpreted as described below:

$|Z_{\text{score}}| \leq 2$ indicates satisfactory laboratory performance

$2.0 < |Z_{\text{score}}| < 3$ indicates questionable (warning) laboratory performance

$|Z_{\text{score}}| \geq 3$ indicates unsatisfactory (action) laboratory performance

As a general rule of thumb, provided that a laboratory does not have systematic sources of bias in their laboratory measurement system, then on average, 19 out of every 20 z-scores should be $\leq \pm 2$. In this scheme each laboratory receives 4 test samples per round and therefore submits 4 z-scores per round. Hence over 5 rounds laboratories would receive 20 test samples and report 20 z-scores.

Assessing the performance of a laboratory

End users that avail of analytical services from laboratories should satisfy themselves that such laboratories meet their requirements. A number of factors ideally need to be considered including

- Expertise and skills of staff within the laboratory?
- Does the laboratory follow accepted measurement standards, guidance?
- Does the laboratory operate a robust internal quality control system?
- Is the laboratory third party accredited to relevant standards such as ISO 17025?
- Does the laboratory successfully participate in relevant external proficiency testing schemes?
- How good is their customer care (communication, turnaround times, pricing etc)?

Participation therefore, in an external proficiency-testing scheme such as AIR PT, represents but one factor in such considerations.

Participation in a single round of an external proficiency-testing scheme represents a “snap-shot” in time of a laboratory’s analytical quality. It is more informative therefore to consider performance over a number of rounds.

Following on from above, therefore over a rolling five round AIR PT window, one would expect that 95 % of laboratory results should be $\leq \pm 2$. If this percentage is substantially lower than 95 % for a particular laboratory, within this five round window, then one can conclude that the laboratory in question may have significant systematic sources of bias in their assay.

A summary of the performance, for each laboratory participating in the AIR PT scheme, is provided in Table 1. This table shows the percentage of results where the absolute z-score, for each laboratory, was less than or equal to 2, i.e. those results which have been assessed as satisfactory.

LAQM Helpdesk – March 2017

Contacts

Further **specific** information on the LGC AIR NO₂ PT scheme is available from LGC proficiency testing on 0161 7622500 or by email at customerservices@lgcgroup.com.

For **general** questions about the scheme within the context of wider LAQM activities please contact Nick Martin at NPL on 0208 943 7088 or nick.martin@npl.co.uk.

Table 1: Laboratory summary performance for AIR NO₂ PT rounds AR007, 9, 10, 12, 13, 15, 16 and 18

The following table lists those UK laboratories undertaking LAQM activities that have participated in recent AIR NO₂ PT rounds and the percentage (%) of results submitted which were subsequently determined to be **satisfactory** based upon a z-score of $\leq \pm 2$ as defined above.

| AIR PT Round | AIR PT AR007 | AIR PT AR009 | AIR PT AR010 | AIR PT AR012 | AIR PT AR013 | AIR PT AR015 | AIR PT AR016 | AIR PT AR018 |
|--|------------------|--------------------|-------------------------|-------------------------|------------------|--------------------|--------------------------|-------------------------|
| Round conducted in the period | April – May 2015 | July – August 2015 | October – November 2015 | January – February 2016 | April – May 2016 | July – August 2016 | September – October 2016 | January – February 2017 |
| Aberdeen Scientific Services | 100 % | 75 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % |
| Cardiff Scientific Services | NR [3] | NR [3] | NR [3] | NR [3] | NR [3] | NR [3] | NR [3] | NR [3] |
| Edinburgh Scientific Services | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % |
| Environmental Services Group, Didcot [1] | 100 % | 100 % | 100 % | 100 % | 75 % | 75 % | 100 % | 100 % |
| Exova (formerly Clyde Analytical) | NR [3] | NR [3] | NR [3] | NR [3] | NR [3] | NR [3] | NR [3] | NR [3] |
| Glasgow Scientific Services | 100 % | 100 % | 100 % | 75 % | 100 % | 0 % | 100 % | 100 % |
| Gradko International [1] | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % |
| Kent Scientific Services | NR [3] | NR [3] | NR [3] | NR [3] | NR [3] | NR [3] | NR [3] | NR [3] |
| Kirklees MBC | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | NR [2] | NR [2] |
| Lambeth Scientific Services | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 75 % | 100 % |
| Milton Keynes Council | 100 % | 100 % | 100 % | 50 % | 100 % | 100 % | 75 % | 100 % |
| Northampton Borough Council | 100 % | 100 % | 100 % | 50 % | 100 % | NR [2] | 75 % | 0 % |
| Somerset Scientific Services | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % | 100 % |
| South Yorkshire Air Quality Samplers | 100 % | 100 % | 75 % | 100 % | 100 % | 75 % | 100 % | 100 % |
| Staffordshire County Council | 100 % | 75 % | 75 % | 75 % | 75 % | 100 % | NR [2] | 100 % |
| Tayside Scientific Services (formerly Dundee CC) | NR [2] | NR [2] | NR [2] | 100 % | NR [2] | 100 % | NR [2] | 100 % |
| West Yorkshire Analytical Services | 75 % | 75 % | 75 % | 75 % | 100 % | NR [2] | 50 % | 100 % |

[1] Participant subscribed to two sets of test samples (2 x 4 test samples) in each AIR PT round.

[2] NR No results reported

[3] Kent Scientific Services, Cardiff Scientific Services and Exova (formerly Clyde Analytical) no longer carry out NO₂ diffusion tube monitoring and therefore did not submit results.