



Introduction

The AXIO proficiency testing (PT) schemes are well established, with a number of schemes and sample types having been active for over 35 years. During the development of these schemes, protocols were established so that each of the participants receives an equivalent material (sufficient homogeneity) and that there are no significant changes to the material over the duration of the PT round (sufficient stability).

This is a requirement of the international standard for PT ISO/IEC 17043 where it is described fully in section 7.3.2.

As a result of a combination of effects from both the UK's exit from the EU ('Brexit') and the on-going impacts of the COVID pandemic, there has been an increase in the average time taken to distribute PT samples to the PT scheme participants.

Due to the observed increase in the time required for participants to receive PT samples, AXIO has carried out a study to determine whether this impacts laboratory performance scores.

Methodology

PT results submitted by participants for several LGC AXIO PT schemes in the fields of microbiology and chemical analysis have been investigated in order to determine whether a number of factors, particularly the recent increases in transport time, has had any significant impact on the performance assessment of the participants. The study aimed to be broadly relevant to all customers and therefore samples, schemes, and rounds were selected to be representative of the most sensitive samples across various parameters (spoilage, analyte degradation etc). The chosen samples were scheduled for dispatch at different points in the year, from April to October 2021.

The reported results from the participants, and assessments of their performance have been analysed according to:

- The time between dispatch date and reported date of analysis
- The effect of the geographical location of the participant
- The time taken for the participants to receive the PT samples

Information was reviewed from the following schemes:

QWAS

The Quality in Water Analysis Scheme (QWAS) covers the microbiological analysis of waters and effluent sludge. QWAS has a large participant base, meaning that each round can provide a high number of results and includes the broadest distribution of recipient countries. All samples from the chosen round were included in the investigation.

AQUACHECK

The Aquacheck scheme includes water, agricultural soils, and sludges for the analysis of a wide range of analytes including major inorganic and organic compounds, elements, herbicides, and organochlorine pesticides. Sample 2 from the Aquacheck scheme was selected as this includes the analytes nitrate and nitrite. Nitrite can be converted to nitrate, the most stable oxidation state, under typical ambient conditions. As such it is recommended during environmental monitoring that such analysis is undertaken as soon as possible after receipt of the sample. Samples are shipped under ambient conditions.

QDCS

The Dairy Chemistry (QDCS) scheme provides test materials to laboratories undertaking analysis in the dairy sector and includes matrices such as butter, cheese, and milk for parameters to measure quality and contaminants. Dairy samples for this type of analysis can be challenging given that the materials need to be refrigerated during routine storage and the potential stabilisation measures are limited, typically restricted to chemical stabilisation to prevent microbial growth. The data for cheese and cream samples was reviewed as any excessive transport time can mean that samples go mouldy in the case of cheese, or curdle (split) in the case of the cream samples.

Data from PT rounds

Time between dispatch and analysis: Microbiology

PT samples for round 300 of the QWAS scheme were dispatched on 12 April 2021, a time which was particularly challenging for shipping of goods in and out of the UK due to the impact of both Brexit, and Covid restrictions on the transport network. The performance in quantitative testing in this round was evaluated according to the time interval (days) between the dispatch date and the participant's reported date of analysis.

In addition, the rate (%) of satisfactory performance scores according to the location (country) of the participant was also compared. In total more than 1,300 results were returned in this round, which included a comprehensive range of inoculum levels and test analytes, including TVC, coliforms, Clostridium, E. coli, Pseudomonas and Enterococci.

Every sample dispatched was included in the data as the samples all have a common format whereby the stability of the organisms relates to the vial rather than the matrix used. The PT samples were sent to 56 countries worldwide at ambient temperature. The time interval from dispatch to analysis ranged from 1 to 46 days.

As detailed in **Figure 1**, overall, the percentage of satisfactory results received was >96%, with only 3 of the 56 countries, (where more than 10 results were reported) having a percentage of satisfactory results below 90%. A satisfactory result in this type of proficiency test is an absolute z score \leq 2.00.

Figure 2 shows all performance scores for the QWAS round by time interval i.e. days between dispatch and analysis, with no observable decrease in participant performance over the time interval.

Of particular interest given the potential sensitivity of test materials, all participants who tested samples more than 30 days after the date of dispatch obtained a satisfactory performance score.

Figure 3 shows a summary of the performance scores according to country in which the participants are located.

All countries achieved average performance scores between -2 and +2 except for Uruguay. This shows that there is no variation in performance due to location of participant. Participants in Uruguay only returned a total of four results, of which two were low and had absolute z scores of >3 and would be considered unsatisfactory and two were satisfactory. The average performance score for Uruguay, therefore, was -2.04, although larger variations in 'average performance' may be expected where the number of results returned is small, as is the case.

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Figure 1: Performance evaluation data, by country for all quantitative samples in QWAS round 300.

Participant country of origin	Number of participants	Average absolute Z score	Number of satisfactory results	% satisfactory results	Average number of days from dispatch to analysis
ARGENTINA	10	0.53	10	100.0	21.9
AUSTRIA	9	0.27	9	100.0	23.9
BELGIUM	13	0.44	13	100.0	12.2
BOSNIA AND HERZEGOVINA	4	0.50	4	100.0	8.0
CANADA	7	1.62	4	57.1	10.0
COLOMBIA	3	0.32	3	100.0	10.0
COSTA RICA	5	0.56	5	100.0	21.0
CROATIA	19	0.24	19	100.0	18.2
DENMARK	8	0.65	8	100.0	10.5
ESTONIA	13	0.13	13	100.0	12.8
FINLAND	4	0.15	4	100.0	21.0
FRANCE	39	0.62	37	94.9	11.8
GERMANY	26	0.41	26	100.0	20.7
GREECE	17	0.62	15	88.2	21.1
GREENLAND	3	0.29	3	100.0	25.3
HUNGARY	7	0.61	7	100.0	22.0
INDIA	5	1.00	5	100.0	5.0
IRELAND	69	0.55	65	94.2	12.8
ITALY	212	0.43	205	96.7	15.2
JERSEY	6	0.63	6	100.0	2.0
KENYA	10	0.40	10	100.0	28.4
KUWAIT	3	0.78	3	100.0	19.0
LATVIA	5	0.63	5	100.0	14.2
LEBANON	6	0.77	6	100.0	15.0
LUXEMBOURG	12	0.22	12	100.0	5.1
MALTA	4	0.18	4	100.0	4.0
MARTINIQUE	8	0.46	8	100.0	19.5
MAURITIUS	11	0.62	11	100.0	18.7
NETHERLANDS	15	0.64	15	100.0	19.0
NORWAY	8	0.11	8	100.0	7.0
OMAN	15	0.32	15	100.0	16.3
POLAND	119	0.22	119	100.0	11.5
PORTUGAL	46	0.30	45	97.8	13.1
QATAR	4	0.86	3	75.0	9.0
ROMANIA	50	0.24	50	100.0	18.1
RUSSIAN FEDERATION	6	0.10	6	100.0	14.8
SERBIA	16	0.48	16	100.0	19.9
SINGAPORE	3	0.45	3	100.0	22.3
SLOVENIA	11	0.31	11	100.0	17.2
SPAIN	102	0.66	93	91.2	10.9
SWITZERLAND	26	0.40	26	100.0	16.1
TAIWAN	3	0.21	3	100.0	7.0
THAILAND	19	0.34	19	100.0	14.0
TURKEY	25	1.00	22	88.0	15.9
UGANDA	6	0.20	6	100.0	29.0
UNITED ARAB EMIRATES	24	0.38	24	100.0	20.5
UNITED KINGDOM	226	0.46	219	96.9	16.8
UNITED STATES	18	0.68	16	88.9	21.7
URUGUAY	4	2.05	2	50.0	10.0
VIETNAM	8	0.38	8	100.0	14.0
Averages		0.51		96.38	15.5

Figure 2: Performance score in QWAS 300 vs the date of analysis (days post-dispatch).

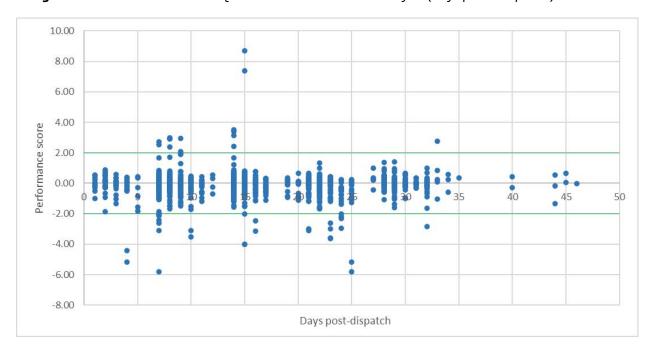
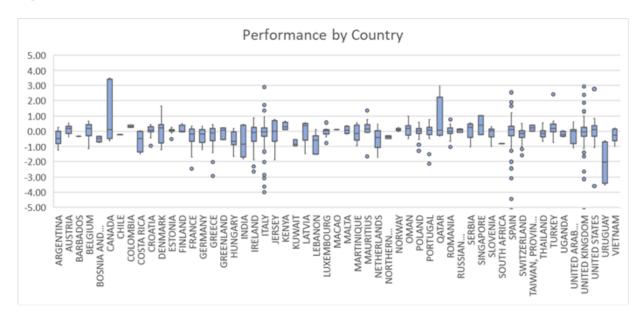


Figure 3: Performance scores in QWAS 300 according to the location of the participants.



The horizontal line is the median (50th Quartile) which is used as the assigned value.

The box represents the interquartile range between the 25th and 75 quartiles

The vertical lines (whiskers) are the range of data included in the analysis, defined as the 25th or 75th quartile +/- 1.5*IQR.

The circles are the resulting values outside of the whiskers (outliers).

Data from PT rounds

Time between dispatch and analysis: Chemistry

PT samples for round 613 of the Aquacheck scheme were dispatched on 13 September 2021. In total 91 participants in 25 countries received the PT samples, shipped at ambient temperature.

Figure 4 shows the measured nitrate concentration reported by the participants plotted against the number of days after dispatch on which the analysis was performed. The green lines represent the satisfactory performance range, and the dotted line represents the assigned value. No significant difference can be observed over the period that the PT round was in operation, with average values consistently around the 25 mg/L nitrate concentration on all days.

Figure 5 shows the measured nitrate concentration results from participants in each country and the average number of days after the dispatch date on which the analysis was performed. The performance of the participants from each country was very consistent, only four individual countries having an average performance outside of the satisfactory range for the PT of 22.7 to 30.71 mg/L, of which two countries only had a single participant. (Note that some countries have been grouped to ensure participant anonymity.)

Figure 4: Nitrate concentration result vs. analysis date

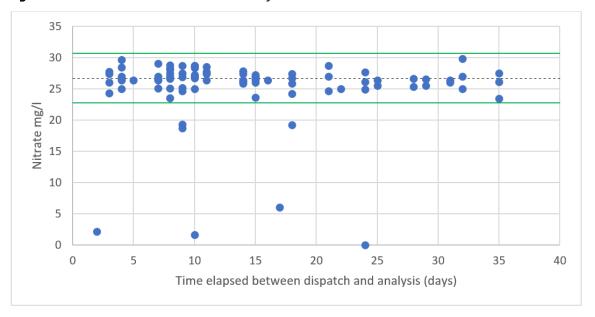


Figure 5: Measured nitrate concentration and days taken to complete analysis, for laboratories in specified countries or areas

Country	Number of participants	Average Nitrate result (mg/l)	Average number of days between dispatch and analysis
BELGIUM	7	26.6	7.3
LITHUANIA	4	19.4	7.5
UNITED ARAB EMIRATES	3	25.0	8.3
POLAND	8	25.8	10.0
UNITED KINGDOM	20	25.1	11.7
CROATIA	3	26.2	13.0
PORTUGAL	7	27.0	13.1
IRELAND	4	28.6	15.8
ROMANIA	4	26.5	16.0
SLOVENIA, LATVIA, SERBIA, SWEDEN	4	26.5	18.0
SAN MARINO, GUERNSEY	2	25.9	18.0
ITALY	8	26.6	18.5
MEXICO, BRAZIL, ARGENTINA, MALI	4	20.3	18.8
SPAIN	8	27.4	19.9
GREECE, CYPRUS, TURKEY	5	25.2	27.0

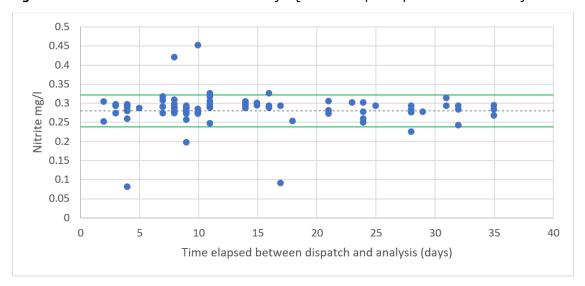
Figure 6 shows the concentration of Nitrite measured by Aquacheck participants vs date of analysis. The green lines represent the satisfactory performance range, the dotted line represents the assigned value.

The same pattern of good performance is observed for the determination of nitrite as for the determination of nitrate. There is no significant difference in the performance of participants who analysed the samples between zero and five days after dispatch, and those who analysed the samples between twenty-five and thirty-five days after dispatch.

In addition, the median average result returned by the participants was 0.287 mg/L, which compares well to the formulation value for this sample of 0.280 mg/L.

The consistent performance of the participants indicates that there has been no significant conversion of nitrite to nitrate, as a result of the sample conditions and time elapsed during transport and storage.

Figure 6: Concentration of Nitrite measured by AQUACHECK participants vs date of analysis.



Data from PT rounds

Time in transit

PT samples for round 306 of the QDCS scheme were dispatched on 18 October 2021. In this, more recent round, data was available for the transit time for a proportion of the participants, meaning that a comparison could be made between this data and the date of analysis reported by these participants. Two samples which might, or would be expected to be sensitive to transport and storage were selected, cheese (sample 37) and cream (sample 39).

Figure 7 shows the measured moisture content of the cheese sample compared to the number of days after dispatch on which the analysis was performed. Although there may be an increase in the variability of the date after 30 days post-dispatch, the average value is consistent with the results obtained by participants who analysed the sample shortly after dispatch.

Over the whole of the PT round, 86.6% of participants returned results which were within the satisfactory range of 33.2% to 35% moisture.

Figure 8 shows the measured moisture content of the cheese sample compared to the number of days the materials were in transit.

Although an unsatisfactory result was reported for the only sample to take 9 days to reach the participant, in general the results returned were close to the assigned value of 34.1%, irrespective of the number of days it took to reach the participants.

Figure 7: Measured moisture content of cheese vs. analysis date

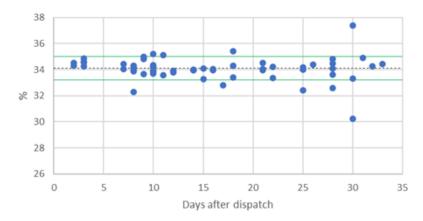


Figure 8: Measured moisture content of cheese vs. days in transit

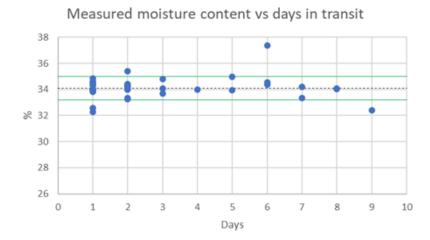


Figure 9 shows the measured fat content of the cream sample compared to the number of days after dispatch on which the analysis was performed. Although a decreasing trend was observed in the measured content, the size of the decrease was not significant compared to the Standard Deviation for Proficiency Assessment (SDPA). The mean result for the 11 labs which analysed the samples >27 days after dispatch was 32.8%, compared to the overall assigned value of 33.0%

Figure 10 shows the measured fat content of the cream sample compared to the number of days the materials were in transit. Although the data is limited there is no observed change in the results, based on the time it took for the samples to reach the participant.

Figure 9: Measured fat content content of cream vs. analysis date

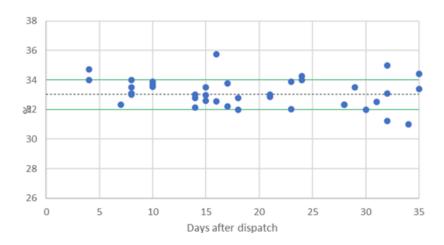
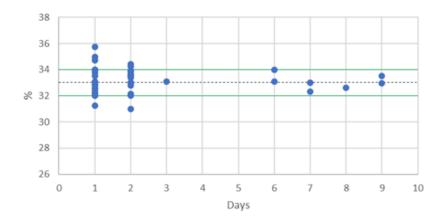


Figure 10: Measured fat content of cream vs. days in transit



Conclusions

The data analysed, from participant results for PT samples where stability could be a potential issue, do not show any trend towards poor participant performance as a result of either the date on which the analysis was carried out or on the time taken for the participants to receive their samples (which might be indicative of transit issues).

In addition, an analysis of the countries in which the participants were located did not show any significant differences in performance, that could be linked to geographical location

Despite the increase in the transportation time which has been observed throughout 2021, there is no indications from the data included in this study that there have been any negative effects on the robustness and validity of the samples and hence on the performance assessments of the participants.

While at the time of writing this report, transportation times appear to be stabilising, LGC continually monitors the performance of transportation, and if any further increase in the delivery times is observed, further data analysis will be carried out.

LGC AXIO leads a continuous development of new schemes and products to meet customer needs, and will consider increased transportation times within the stability assessment of all new samples.

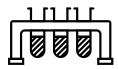
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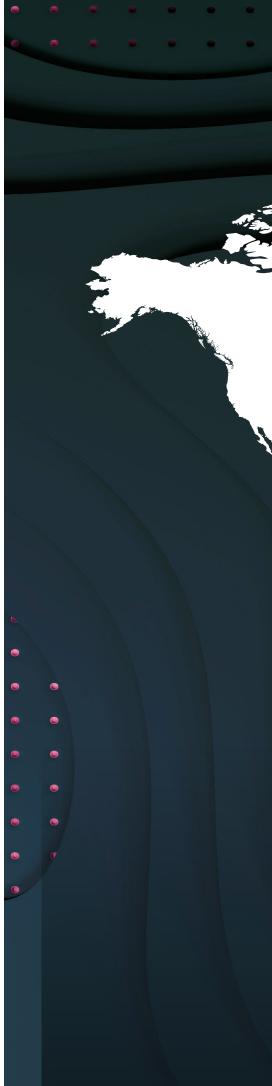
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