



## **Report No. 802**

### ***Waters Proficiency Testing***

### **Round 151**

### ***- Nitrate, Ammonia, Orthophosphate -***

**March 2013**

#### **Acknowledgments**

PTA wishes to gratefully acknowledge the technical assistance provided for this program by Ms K Timms, Department of Primary Industry and Fisheries (Northern Territory, Australia). Also our thanks go to the Environmental Resource Associates (ERA) for the supply of samples and to Global Proficiency Pty Ltd (Melbourne, Australia) for distributing the samples.

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## 1. Foreword

This report summarises the results of a proficiency testing program on the determination of Nitrate, Ammonia and Orthophosphate in waters. This is round 151 in a planned series of programs involving the analysis of chemical and physical parameters of waters.

The exercise was conducted in January 2013 by Proficiency Testing Australia (PTA). The main aim of the program was to assess laboratories' abilities to competently perform the prescribed analyses.

The Program Coordinator was Ms D Mihaila and the Technical Advisor was Ms K Timms of the Department of Primary Industry and Fisheries. This report was authorised by Ms W Fajloun, PTA Quality Coordinator.

## 2. Program Features and Design

2.1 Each laboratory was randomly allocated a unique code number for the program to ensure confidentiality of results. Reference to each laboratory in this report is by code number only. Please note that a number of laboratories reported more than one set of results and, therefore, their code numbers (with letter) could appear several times in the same data set.

2.2 Laboratories were provided with the "Instructions to Participants" and "Results Sheet" (see Appendix C). Laboratories were requested to perform the tests according to their routine methods.

2.3 Participants were provided with one sealed vial (labelled R151) containing solutions of Nitrate, Ammonia and Orthophosphate.

2.4 A total of 56 laboratories received samples, comprising:

- 46 Australian participants; and

- 10 overseas participants, including:

- Brunei Darussalam (1), Indonesia (2), Malaysia (1), Papua New Guinea (1), Qatar (1), Singapore (2), Thailand (1), Vietnam (1).

Of these 56 laboratories, 3 were unable to submit results by the due date.

2.5 Results (as reported by participants) with corresponding summary statistics (i.e. number of results, median, uncertainty of the median, normalised interquartile range, robust coefficient of variation, minimum, maximum and range) are presented in Appendix A (for each of the analyses performed).

2.6 A robust statistical approach, using z-scores, was utilised to assess laboratories' testing performance (see Section 3). Robust z-scores and ordered z-score charts relevant to each test are presented in Appendix A.

The document entitled *Guide to Proficiency Testing Australia, 2012* (reference [1]) defines the statistical terms and details the statistical procedures referred to in this report.

- 2.7 A tabulated listing of laboratories (by code number) identified as having outlier results can be found on page 15.
- 2.8 Prior to sample distribution, a number of randomly selected samples were analysed for homogeneity and stability. Based on the results of this testing (see Appendix B) it was considered that the samples utilised for this program were homogeneous and stable. As such, any results later identified as outliers could not be attributed to any notable sample variability.

### 3. Statistical Format

For each test, the following information is given:

- a table of results and calculated z-scores;
- a list of summary statistics; and
- ordered z-score charts.

#### 3.1 Outlier Results and Z-scores

In order to assess laboratories' testing performance, a robust statistical approach, using z-scores, was utilised. Z-scores give a measure of how far a result is from the consensus value (i.e. the median), and gives a "score" to each result relative to the other results in the group.

A z-score close to zero indicates that the result agrees well with those from other laboratories, whereas, a z-score with an absolute value greater than or equal to 3.0 is considered to be an outlier and is marked by the symbol "§".

Each determination was examined for outliers with all methods pooled. The table on page 15 summarises the outlier results detected.

#### 3.2 Results Tables and Summary Statistics

The tables in Appendix A contain the results returned by each laboratory, including the code number for the method used and the robust z-score calculated for each result.

Results have been entered exactly as reported by participants. That is, laboratories which did not report results to the precision (i.e. number of significant figures) requested on the Results Sheet have **not** been rounded to the requested precision before being included in the statistical analysis.

A list of summary statistics appears at the bottom of each of the results tables and consists of:

- *No of Results*: the total number of results for that test/sample;
- *Median*: the middle value of the results;
- *Uncertainty of the Median*: a robust estimate of the standard deviation of the *Median*;
- *Normalised IQR*: the normalised interquartile range of the results;
- *Robust CV*: the robust coefficient of variation expressed as a percentage, i.e.  $100 \times \text{Normalised IQR} / \text{Median}$ ;
- *Minimum*: the lowest laboratory result;
- *Maximum*: the highest laboratory result; and
- *Range*: the difference between the *Maximum* and *Minimum*.

The median is a measure of the centre of the data.

The normalised IQR is a measure of the spread of the results. It is calculated by multiplying the interquartile range (IQR) by a correction factor, which converts the IQR to an estimate of the standard deviation. The IQR is the difference between the upper and lower quartiles (i.e. the values above and below which a quarter of the results lie, respectively).

For normally distributed data, the uncertainty of the median is approximated by:

$$\frac{\sqrt{\pi}}{\sqrt{2}} \times \frac{\text{normIQR}}{\sqrt{n}}$$

$n$  = number of results

Please see reference [1] for further details on these robust summary statistics.

### 3.3 Ordered Z-score Charts

The charts in Appendix A indicate each laboratory's robust z-score, in order of magnitude, marked with its laboratory code number. From these charts, each laboratory can readily compare its performance relative to the other laboratories.

These charts contain solid lines at +3.0 and -3.0, so that outliers are clearly identifiable as those laboratories whose "bar" extends beyond these "cut-off" lines. The y-axis of these charts has been limited, so very large z-scores appear to extend beyond the chart boundary.

#### 4. PTA and Technical Advisor's Comments

##### 4.1 Metrological Traceability and Measurement Uncertainty of Assigned Values

Consensus values (median) derived from participants' results are used in this program. These values are not metrologically traceable to an external reference.

Sample preparation was undertaken according to Environmental Resource Associates' Standard Operating Procedures to ensure samples were fit-for-purpose, homogeneous and stable.

Solutions were stable and homogeneous, and medians obtained from this proficiency round were in good agreement with the expected levels (manufacturer's assigned values), as shown in Table 1.

As the assigned value for this program is the median of the results submitted by the participants, the uncertainty of the median has been calculated and is presented in Table 1.

| Analyte             | Expected Levels (mg/L) | Median (mg/L) | Uncertainty of the median (mg/L) |
|---------------------|------------------------|---------------|----------------------------------|
| Nitrate as N        | 21.6                   | 21.80         | 0.23                             |
| Ammonia as N        | 18.5                   | 18.75         | 0.16                             |
| Orthophosphate as P | 3.90                   | 3.935         | 0.030                            |

Table 1. Comparison of expected levels and proficiency medians. The values of the calculated uncertainty of the median are also presented.

Overall, the performance of participants in this round was good, with robust CVs less than 6% for all analytes.

## 4.2 Analysis of Round 151 Results

### 4.2.1 Nitrate as N

Table 2 compares the Nitrate median and robust CV from this round to those obtained in previous PTA rounds.

| Round      | Sample | Median (mg/L) | Robust CV (%) | Participants |
|------------|--------|---------------|---------------|--------------|
| This study | R151   | 21.80         | 5.8           | 46           |
| Report 746 | R138   | 7.690         | 7.1           | 49           |
| Report 675 | R123   | 22.00         | 4.9           | 56           |

Table 2. Comparison of current round variability and proficiency median of Nitrate testing with the results of the previous two rounds.

### Bias / Accuracy

The Nitrate testing was successfully performed, with satisfactory results ( $|z\text{-scores}| \leq 2.0$ ) ranging between 19.3 – 24.3 mg/L. Out of 46 participants, one questionable result ( $2.0 < |z\text{-scores}| < 3.0$ ) was reported (laboratory 269b). Seven outlier results ( $|z\text{-scores}| \geq 3.0$ ) were obtained, requiring follow-up action by laboratories 188, 217, 402, 453, 512, 525 and 608.

Figure 1 exhibits the spread of results and the methods used for Nitrate testing in this round.

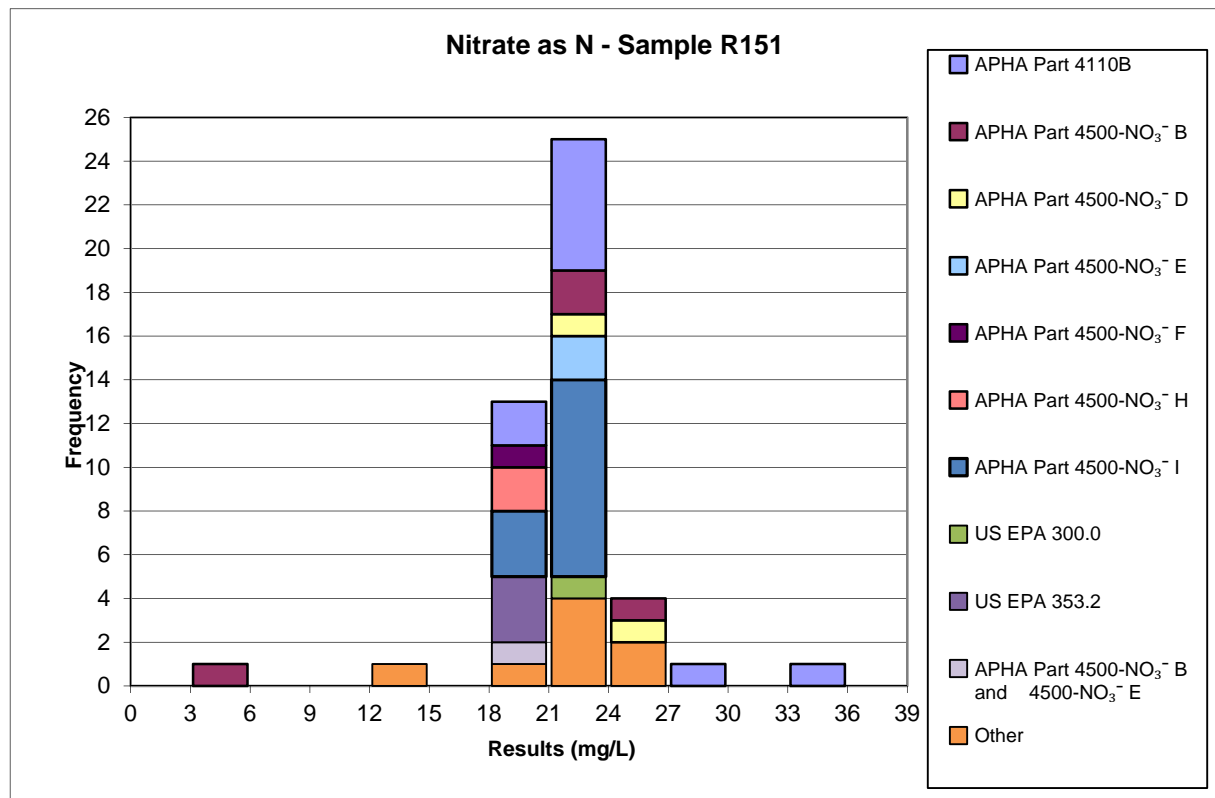


Figure 1. Spread of results for Nitrate testing of Sample R151, with a median of 21.80 mg/L.

The data set formed an approximately normal distribution with no significant bias attributable to any one method. There was a large variety of methods used (Figure 1), with approximately 26% of participants using the method APHA Part 4500-NO<sub>3</sub><sup>-</sup> I (Cadmium Reduction Flow Injection Analysis), followed by approximately 22% of participants who used the method APHA Part 4110B (Ion Chromatography with Chemical Suppression of Eluent Conductivity). The remaining participants used other APHA protocols (26%), US EPA methods (9%) or other methods (17%).

### Measurement Uncertainty (MU)

The MU reported by participants can be seen in Figure 2. Out of 46 participants, 34 (74%) submitted MU information. Many of the stated MUs did not accurately reflect the difference between the median and the participant's result for this proficiency sample.

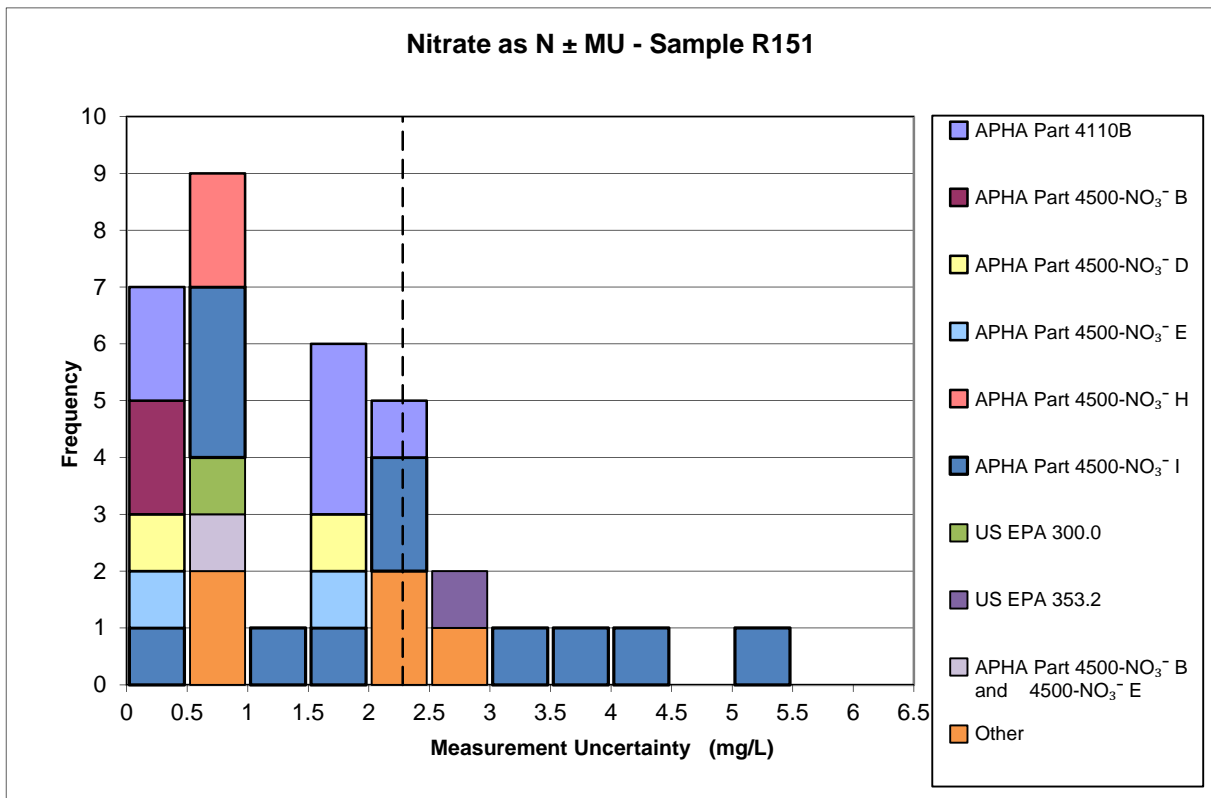


Figure 2. MU for Nitrate testing of Sample R151, as reported by participants, compared with 95% confidence interval for overall reproducibility (----) ( $\pm 2.28$  mg/L) in this round.

A t-test of all the results (outliers removed), indicated the overall reproducibility for Nitrate testing was  $21.45 \pm 2.28$  mg/L for Sample R151 (95% CI).

Laboratories 109, 118, 142, 181, 217, 268, 269b, 273, 365, 376, 427, 461 and 608 may wish to re-examine their MU calculations, as their result was further from the median than their stated MU, as shown in Figure 3 below.



### Nitrate as N - Sample R151

*Results of Sample R151, including MU, compared to the median*

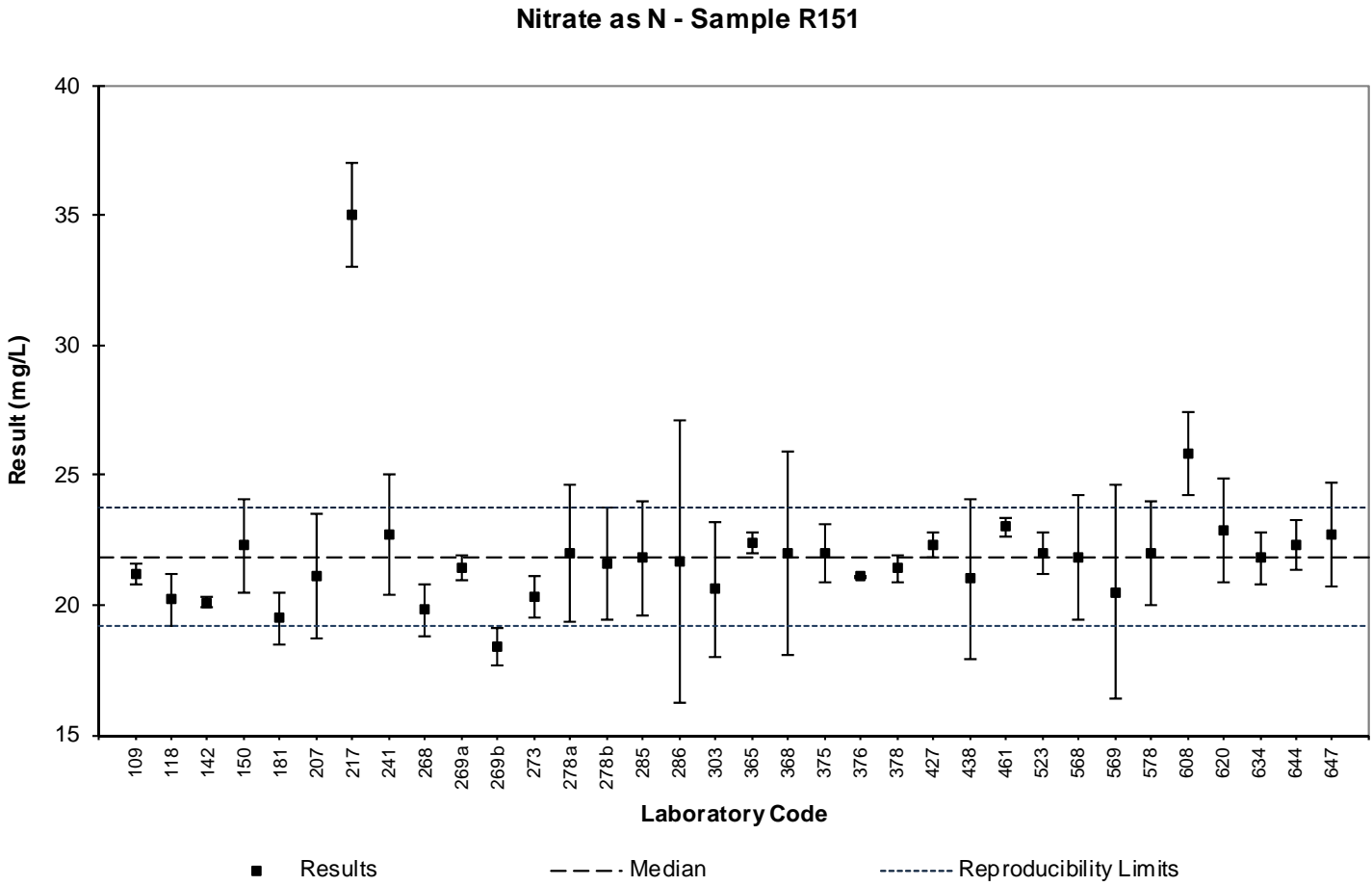


Figure 3. Nitrate as N - Results of Sample R151, including MU, compared to the median

#### 4.2.2 Ammonia as N

Table 3 compares the Ammonia median and robust CV from this round to those obtained in previous PTA rounds.

| Round      | Sample | Median (mg/L) | Robust CV (%) | Participants |
|------------|--------|---------------|---------------|--------------|
| This study | R151   | 18.75         | 4.8           | 52           |
| Report 746 | R138   | 9.000         | 4.2           | 56           |
| Report 675 | R123   | 10.73         | 5.5           | 58           |

Table 3. Comparison of current round variability and proficiency median of Ammonia testing with the results of the previous two rounds.

#### Bias / Accuracy

The Ammonia testing was successfully performed, with satisfactory results ( $|z\text{-scores}| \leq 2.0$ ) ranging between 16.9 – 20.6 mg/L. Out of 52 participants, two questionable results ( $2.0 < |z\text{-scores}| < 3.0$ ) were reported (laboratories 525 and 647). Two outlier results ( $|z\text{-scores}| \geq 3.0$ ) were obtained, requiring follow-up action by laboratories 181 and 608.

Figure 4 exhibits the spread of results and the methods used for Ammonia testing in this round.

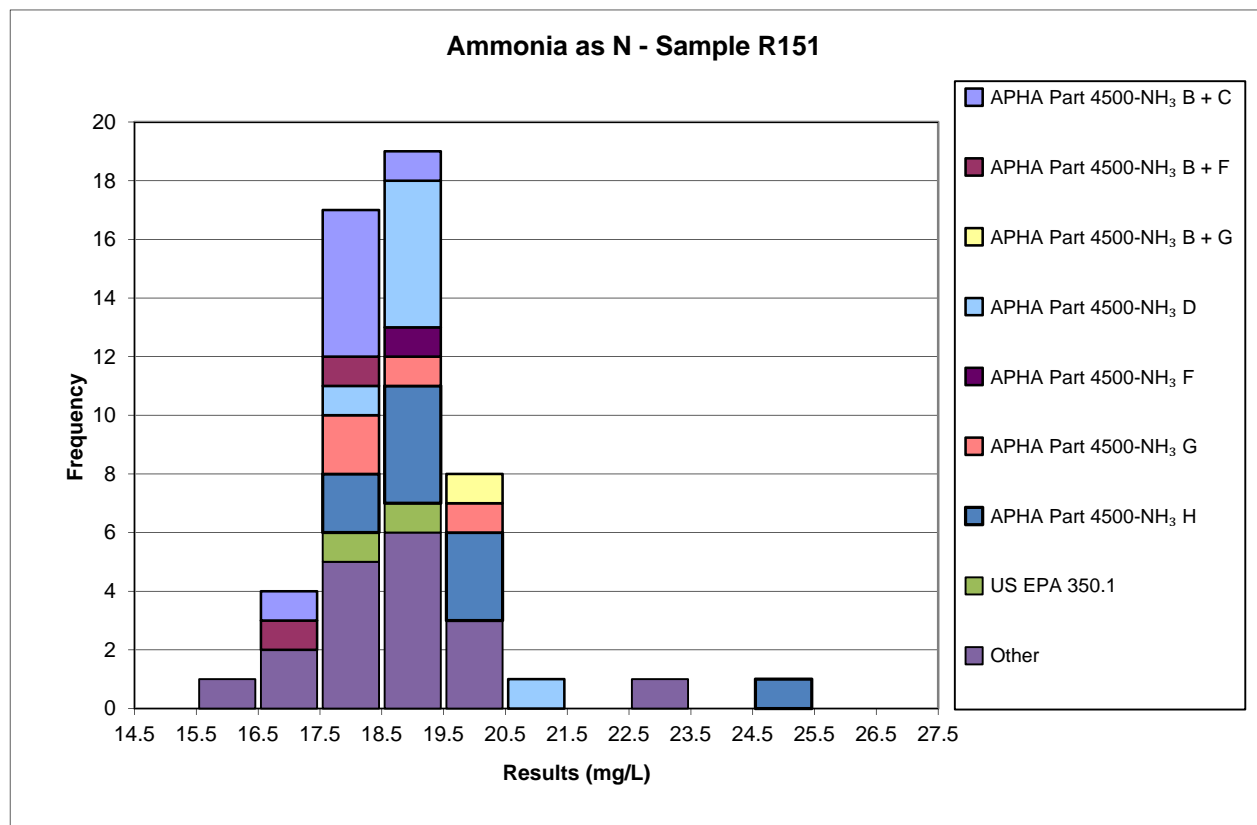


Figure 4. Spread of results for Ammonia testing of Sample R151, with a median of 18.75 mg/L.

The data set formed an approximately normal distribution with no significant bias attributable to any one method. There was a large variety of methods used (Figure 4), with approximately 19% of participants using the method APHA Part 4500-NH<sub>3</sub> H (Flow Injection Analysis), followed by approximately 13% of participants who used the method APHA Part 4500-NH<sub>3</sub> B + C (Preliminary Distillation Step and Titrimetric Method) and 13% of participants using APHA Part 4500-NH<sub>3</sub> D (Ammonia-Selective Electrode Method). The remaining participants used other APHA protocols (16%), US EPA methods (4%) or other methods (35%).

### Measurement Uncertainty (MU)

The MU reported by participants can be seen in Figure 5. Out of 52 participants, 39 (75%) submitted MU information. Many of the stated MUs did not accurately reflect the difference between the median and the participant's result for this proficiency sample.

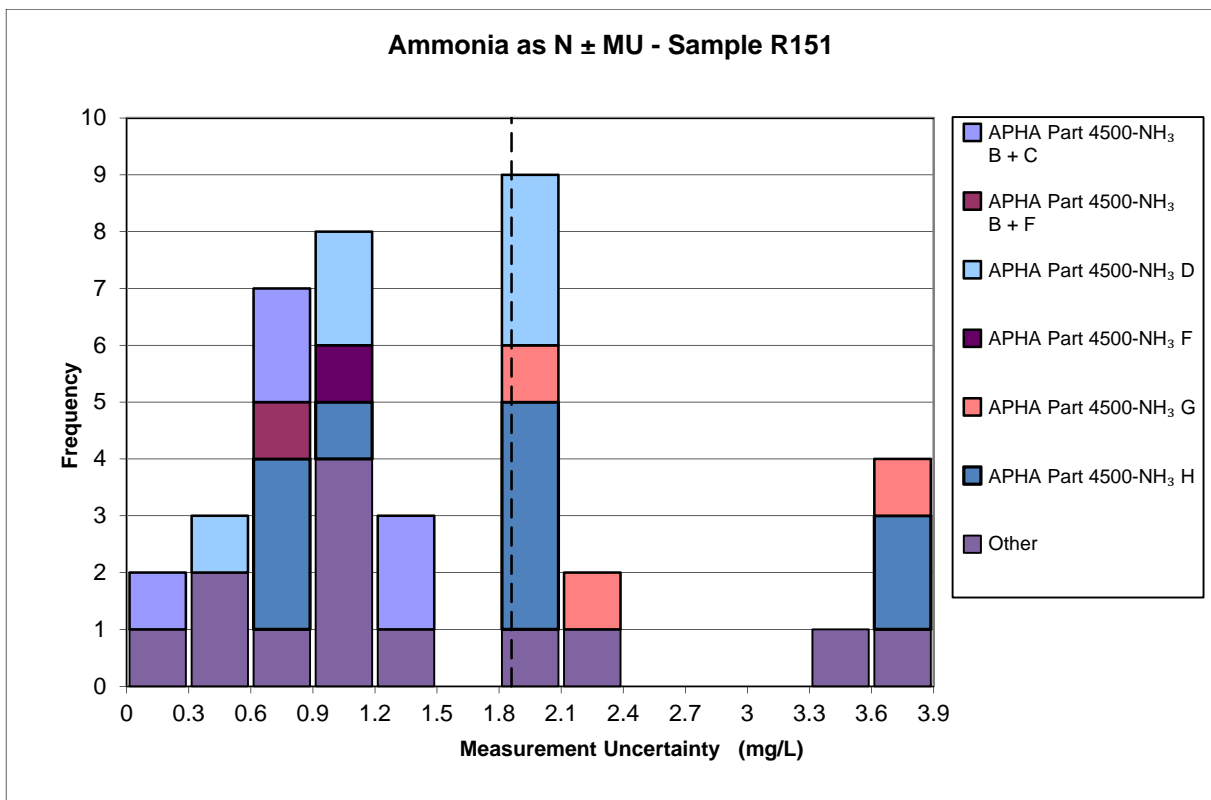


Figure 5. MU for Ammonia testing of Sample R151, as reported by participants, compared with 95% confidence interval for overall reproducibility (----) ( $\pm 1.86$  mg/L) in this round.

A t-test of all the results (outliers removed), indicated the overall reproducibility for Ammonia testing was  $18.71 \pm 1.86$  mg/L for Sample R151 (95% CI).

Laboratories 142, 181, 269a, 269b, 343, 375, 390a, 390b, 427, 461, 608 and 647 may wish to re-examine their MU calculations, as their result was further from the median than their stated MU, as shown in Figure 6 below.

### Ammonia as N - Sample R151

*Results of Sample R151, including MU, compared to the median*

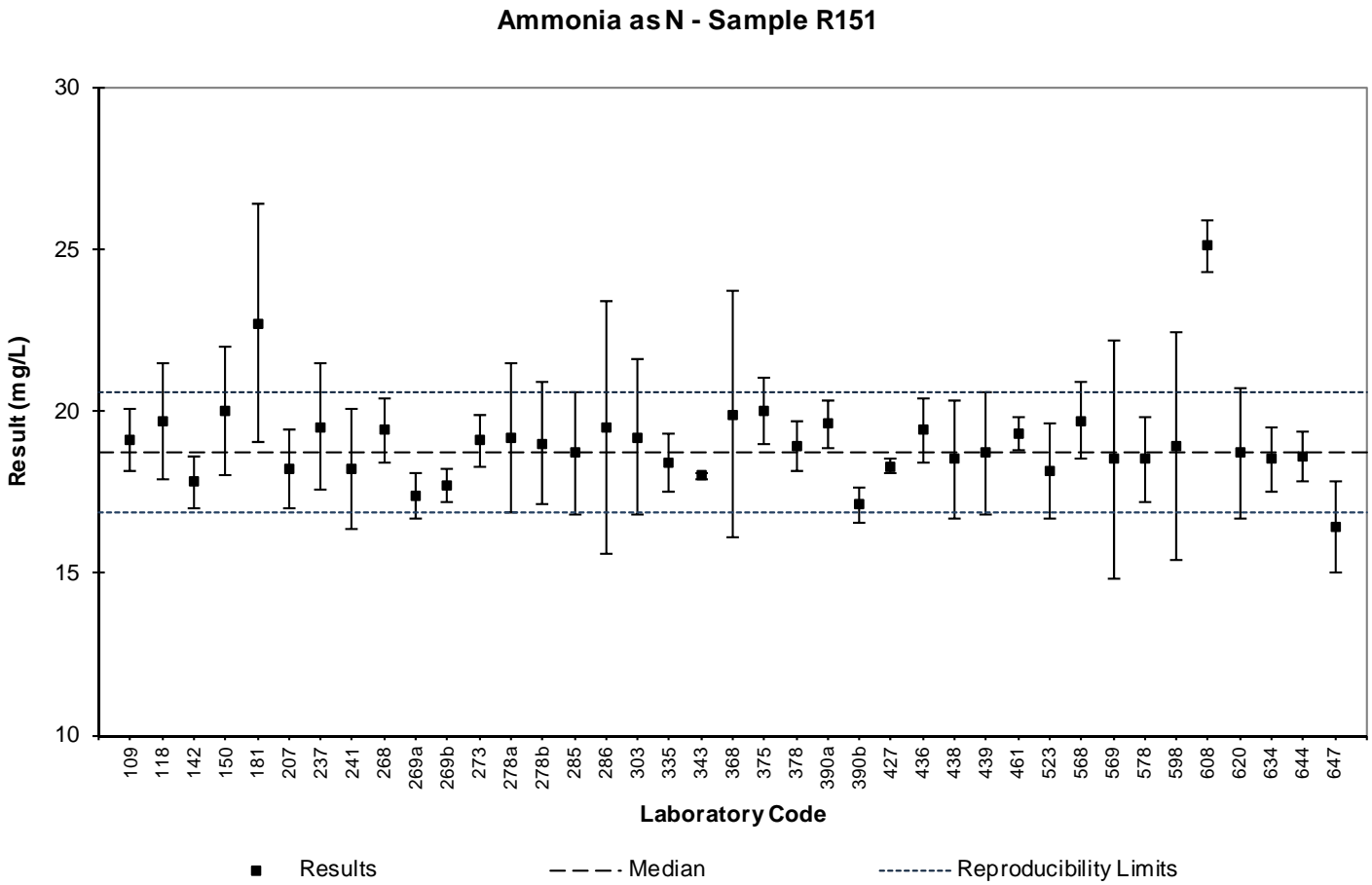


Figure 6. Ammonia as N - Results of Sample R151, including MU, compared to the median

### 4.2.3 Orthophosphate as P

Table 4 compares the Orthophosphate median and robust CV from this round to those obtained in previous PTA rounds.

| Round      | Sample | Median (mg/L) | Robust CV (%) | Participants |
|------------|--------|---------------|---------------|--------------|
| This study | R151   | 3.935         | 4.1           | 46           |
| Report 746 | R138   | 2.610         | 4.7           | 47           |
| Report 675 | R123   | 3.000         | 5.1           | 55           |

Table 4. Comparison of current round variability and proficiency median of Orthophosphate testing with the results of the previous two rounds.

#### Bias / Accuracy

The Orthophosphate testing was successfully performed, with satisfactory results ( $|z\text{-scores}| \leq 2.0$ ) ranging between 3.61 – 4.26 mg/L. Out of 46 participants, four questionable results ( $2.0 < |z\text{-scores}| < 3.0$ ) were reported (laboratories 269a, 568, 620 and 639). Five outlier results ( $|z\text{-scores}| \geq 3.0$ ) were obtained, requiring follow-up action by laboratories 196, 217, 343, 353 and 647.

Figure 7 exhibits the spread of results and the methods used for Orthophosphate testing in this round.

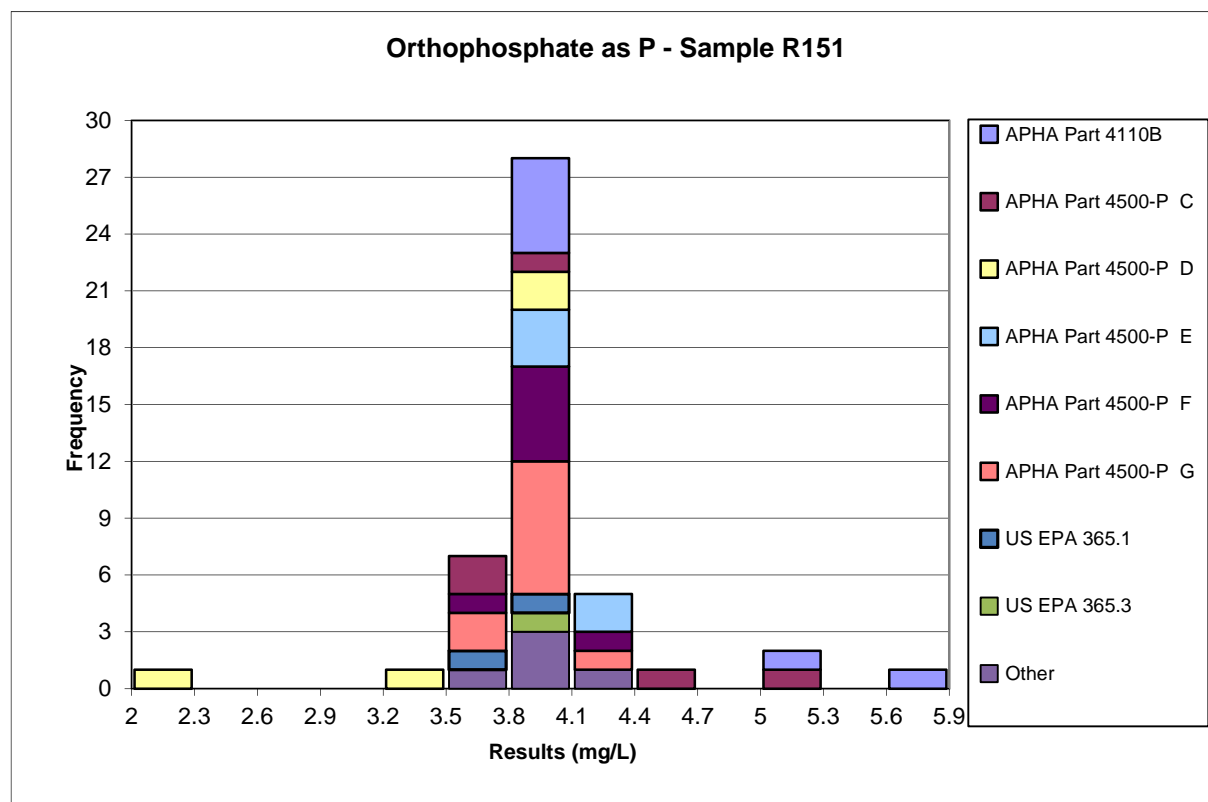


Figure 7. Spread of results for Orthophosphate testing of Sample R151, with a median of 3.935 mg/L.

The data set formed an approximately normal distribution with no significant bias attributable to any one method. There was a large variety of methods used (Figure 7), with approximately 22% of participants using the APHA Part 4500-P G (Flow Injection Analysis for Orthophosphate), followed by approximately 15% of participants who used the method APHA Part 4110B (Ion Chromatography with Chemical Suppression of Eluent Conductivity) and 15% of participants using APHA Part 4500-P F (Automated Ascorbic Acid Reduction Method). The remaining participants used other APHA protocols (30%), US EPA methods (7%) or other methods (11%).

### Measurement Uncertainty (MU)

The MU reported by participants can be seen in Figure 8. Out of 46 participants, 35 (76%) submitted MU information. Many of the stated MUs did not accurately reflect the difference between the median and the participant's result for this proficiency sample.

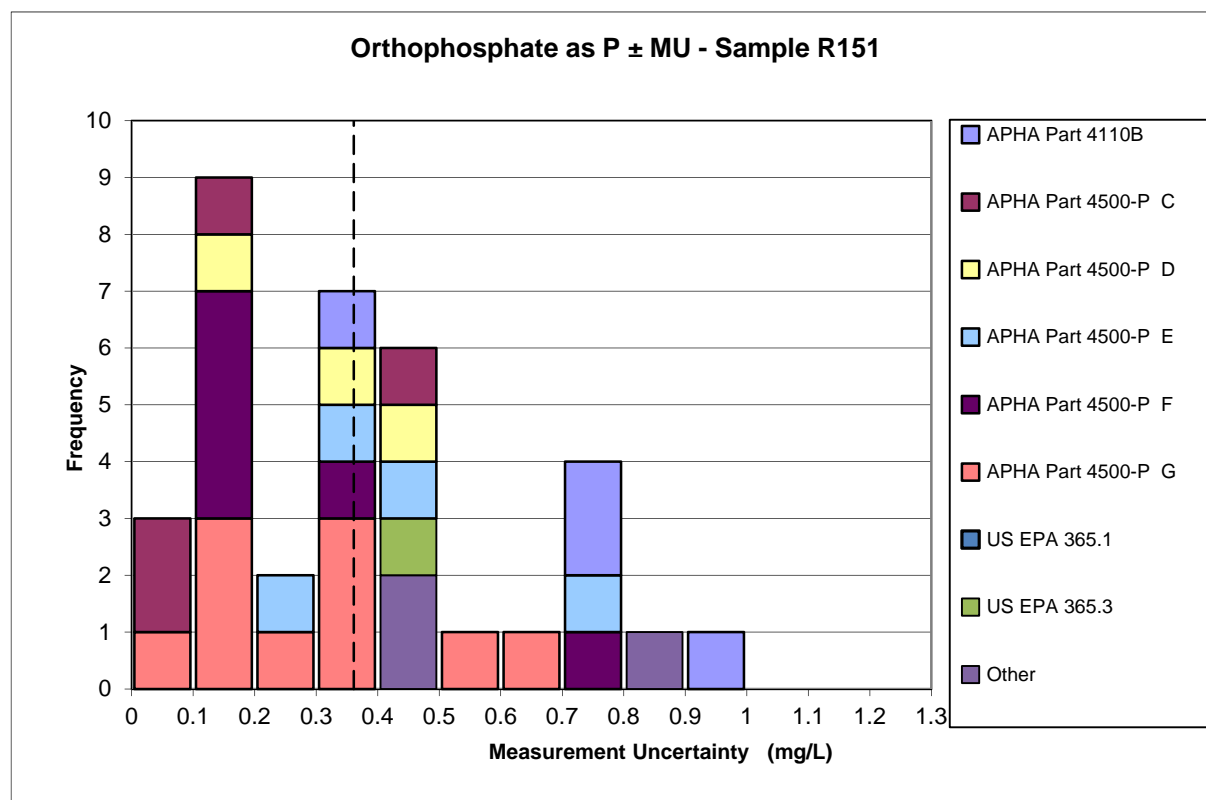


Figure 8. MU for Orthophosphate testing of Sample R151, as reported by participants, compared with 95% confidence interval for overall reproducibility (----) ( $\pm 0.361$  mg/L) in this round.

A t-test of all the results (outliers removed), indicated the overall reproducibility for Orthophosphate testing was  $3.921 \pm 0.361$  mg/L for Sample R151 (95% CI).

Laboratories 217, 335, 343, 375, 378, 568, 620 and 647 may wish to re-examine their MU calculations, as their result was further from the median than their stated MU, as shown in Figure 9 below.

### Orthophosphate as P - Sample R151

Results of Sample R151, including MU, compared to the median

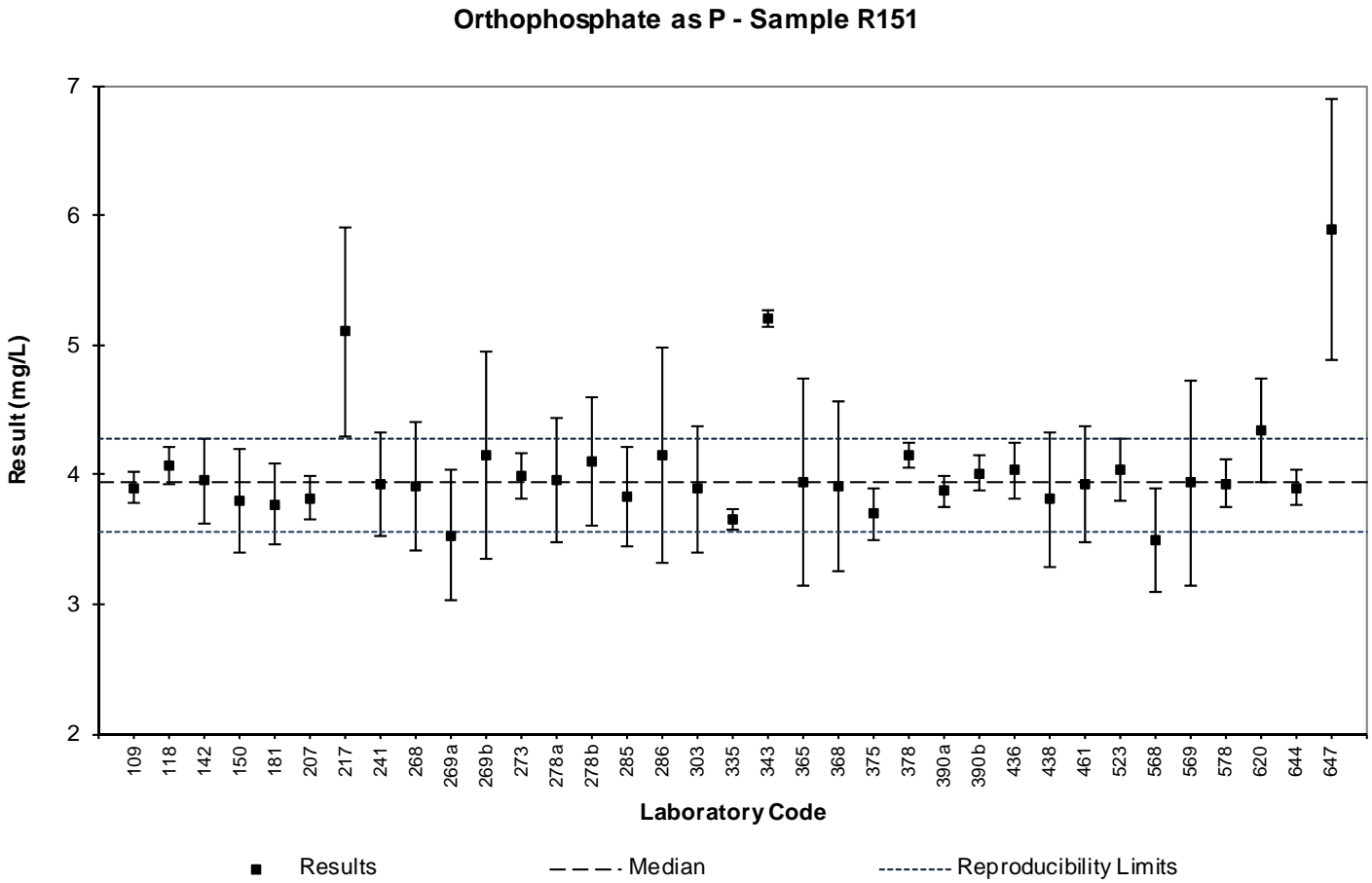


Figure 9. Orthophosphate as P - Results of Sample R151, including MU, compared to the median

### 4.3 Analysis of Results by Method Groups

Further analysis of results by method groups is undertaken to provide specific information on individual method performance.

The participants in this round used a large variety of methods for analysis of Nitrate, Ammonia and Orthophosphate.

The method APHA Part 4500-NO<sub>3</sub><sup>-</sup> I - Cadmium Reduction Flow Injection Analysis (method code 23) was most frequently employed for Nitrate analysis, with 12 laboratories indicating the use of this method. Table 5 below presents the median, uncertainty of the median and robust CV for Nitrate results obtained by method 23.

| <b>Sample</b> | <b>Method code</b> | <b>Participants</b> | <b>Median ±<br/>Uncertainty of the Median<br/>(mg/L)</b> | <b>Robust<br/>CV (%)</b> |
|---------------|--------------------|---------------------|--|--------------------------|
| R151          | 23                 | 12                  | 21.75 ± 0.27   | 3.4                      |

Table 5. Variability and proficiency medians of Nitrate results obtained by method 23.

In order for methods to be grouped for analysis, PTA requires more than ten sets of results from the same method group. For methods other than those presented above, there were ten or less results submitted, therefore reliable conclusions cannot be drawn from analysing them separately and these results have not been analysed by grouped methods.



## 5. Outlier Results

Laboratories reporting results that have been identified as outliers are listed in Table 6 below.

| Lab Code | Analysis     |              |                     |
|----------|--------------|--------------|---------------------|
|          | Nitrate as N | Ammonia as N | Orthophosphate as P |
| 181      |              | §            |                     |
| 188      | §            |              |                     |
| 196      |              |              | §                   |
| 217      | §            |              | §                   |
| 343      |              |              | §                   |
| 353      |              |              | §                   |
| 402      | §            |              |                     |
| 453      | §            |              |                     |
| 512      | §            |              |                     |
| 525      | §            |              |                     |
| 608      | §            | §            |                     |
| 647      |              |              | §                   |

Table 6. Laboratory results identified as outliers for each analysis performed.

Note:

1. A "§" indicates the occurrence of a z-score outlier result (i.e. those results for which  $|z\text{-score}| \geq 3.0$ ).

## 6. Reference

- [1] *Guide to Proficiency Testing Australia*, 2012 (This document can be found on the PTA website, [www.pta.asn.au](http://www.pta.asn.au)).

# APPENDIX A

## Results and Data Analysis

|                           |    |
|---------------------------|----|
| Nitrate as N .....        | A1 |
| Ammonia as N.....         | A4 |
| Orthophosphate as P ..... | A7 |

# **Nitrate as N Results**

Sample R151

**Nitrate as N****Results by Laboratory Code**

| Sample R151 |                    |                 |                                |                             |    |
|-------------|--------------------|-----------------|--------------------------------|-----------------------------|----|
| Lab Code    | Result ±<br>(mg/L) | MU <sup>1</sup> | Robust<br>z-score <sup>2</sup> | Method<br>Code <sup>3</sup> |    |
| 109         | 21.2 ±             | 0.42            | -0.48                          | 17                          |    |
| 118         | 20.2 ±             | 1.0             | -1.27                          | 22                          |    |
| 142         | 20.1 ±             | 0.2             | -1.35                          | 16                          |    |
| 150         | 22.3 ±             | 1.8             | 0.40                           | 23                          |    |
| 181         | 19.5 ±             | 0.995           | -1.83                          | 22                          |    |
| 183         | 20.6               | #               | -0.95                          | 25                          |    |
| 188         | 26.1               | #               | 3.41                           | §                           | 28 |
| 196         | 22.0               | #               | 0.16                           | 16                          |    |
| 207         | 21.1 ±             | 2.4             | -0.56                          | 28                          |    |
| 217         | 35.0 ±             | 2.0             | 10.47                          | §                           | 16 |
| 241         | 22.7 ±             | 2.30            | 0.71                           | 23                          |    |
| 268         | 19.8 ±             | 1               | -1.59                          | 17,20                       |    |
| 269a        | 21.440 ±           | 0.50            | -0.29                          | 17                          |    |
| 269b        | 18.400 ±           | 0.70            | -2.70                          | 28                          |    |
| 273         | 20.3 ±             | 0.8             | -1.19                          | 23                          |    |
| 278a        | 22.0 ±             | 2.64            | 0.16                           | 28                          |    |
| 278b        | 21.6 ±             | 2.16            | -0.16                          | 28                          |    |
| 285         | 21.8 ±             | 2.18            | 0.00                           | 23                          |    |
| 286         | 21.7 ±             | 5.43            | -0.08                          | 23                          |    |
| 303         | 20.6 ±             | 2.58            | -0.95                          | 25                          |    |
| 343         | 20.6               | #               | -0.95                          | 16                          |    |
| 353         | 24.3               | #               | 1.98                           | 17                          |    |
| 365         | 22.4 ±             | 0.4             | 0.48                           | 16                          |    |
| 368         | 22.0 ±             | 3.92            | 0.16                           | 23                          |    |
| 375         | 22.0 ±             | 1.1             | 0.16                           | 23                          |    |
| 376         | 21.10 ±            | 0.04            | -0.56                          | 23                          |    |
| 378         | 21.4 ±             | 0.51            | -0.32                          | 23                          |    |

1 Where reported, results are shown with their corresponding measurement uncertainty (MU).

2 "§" denotes outliers (i.e. those results for which  $|z\text{-score}| \geq 3.0$ ). Robust z-scores are calculated as:  
 $z = (A - \text{median}) \div \text{normalised IQR}$ , where A is the participant laboratory's result.

3 Please refer to Appendix C (page C3-C4) for method code descriptions.

**Nitrate as N - cont.****Results by Laboratory Code**

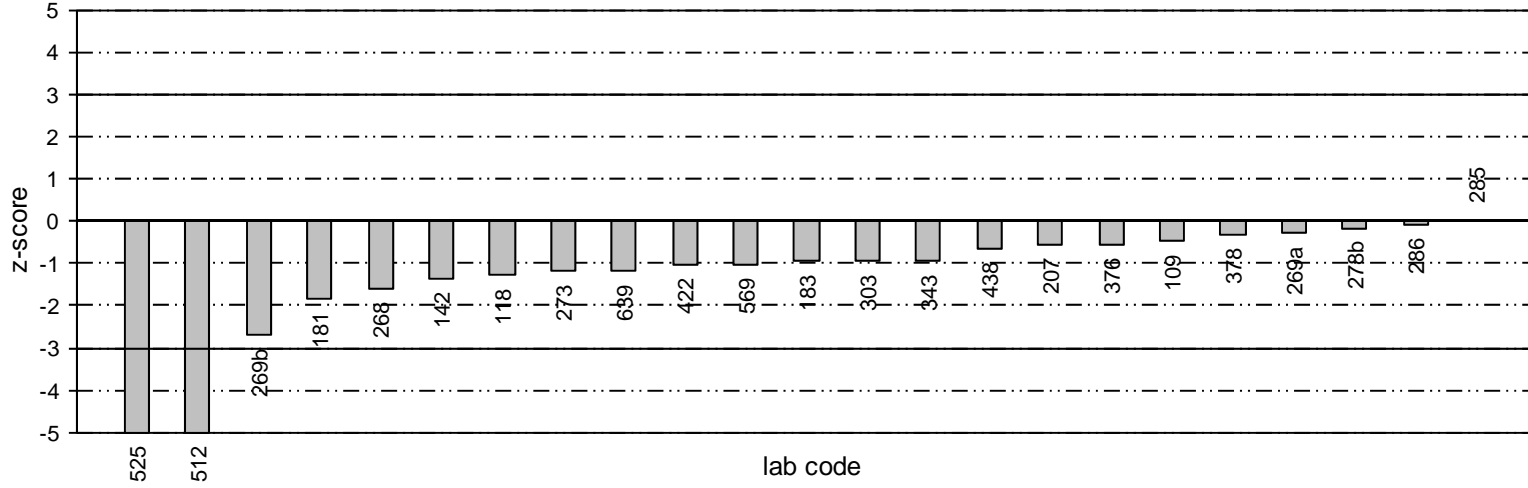
| Sample R151 |                 |                 |                             |   |                          |
|-------------|-----------------|-----------------|-----------------------------|---|--------------------------|
| Lab Code    | Result ± (mg/L) | MU <sup>1</sup> | Robust z-score <sup>2</sup> |   | Method Code <sup>3</sup> |
| 402         | 27.5            | #               | 4.52                        | § | 16                       |
| 422         | 20.5            | #               | -1.03                       |   | 25                       |
| 427         | 22.3 ±          | 0.45            | 0.40                        |   | 20                       |
| 438         | 21.0 ±          | 3.052           | -0.63                       |   | 23                       |
| 453         | 27              | #               | 4.13                        | § | 28                       |
| 461         | 23.0 ±          | 0.34            | 0.95                        |   | 19                       |
| 512         | 14.9            | #               | -5.48                       | § | 28                       |
| 523         | 22.0 ±          | 0.79            | 0.16                        |   | 28                       |
| 525         | 4.174           | #               | -13.99                      | § | 17                       |
| 568         | 21.8 ±          | 2.4             | 0.00                        |   | 16                       |
| 569         | 20.5 ±          | 4.1             | -1.03                       |   | 23                       |
| 578         | 22.0 ±          | 2.0             | 0.16                        |   | 20                       |
| 601         | 22.2            | #               | 0.32                        |   | 16                       |
| 608         | 25.8 ±          | 1.59            | 3.17                        | § | 19                       |
| 620         | 22.9 ±          | 2.0             | 0.87                        |   | 16                       |
| 634         | 21.8 ±          | 1.0             | 0.00                        |   | 24                       |
| 639         | 20.3            | #               | -1.19                       |   | 21                       |
| 644         | 22.3 ±          | 0.941           | 0.40                        |   | 23                       |
| 647         | 22.7 ±          | 2.0             | 0.71                        |   | 16                       |

|                                   |        |
|-----------------------------------|--------|
| <i>No of Results:</i>             | 46     |
| <i>Median:</i>                    | 21.80  |
| <i>Uncertainty of the Median:</i> | 0.23   |
| <i>Normalised IQR:</i>            | 1.26   |
| <i>Robust CV:</i>                 | 5.8%   |
| <i>Minimum:</i>                   | 4.174  |
| <i>Maximum:</i>                   | 35.0   |
| <i>Range:</i>                     | 30.826 |

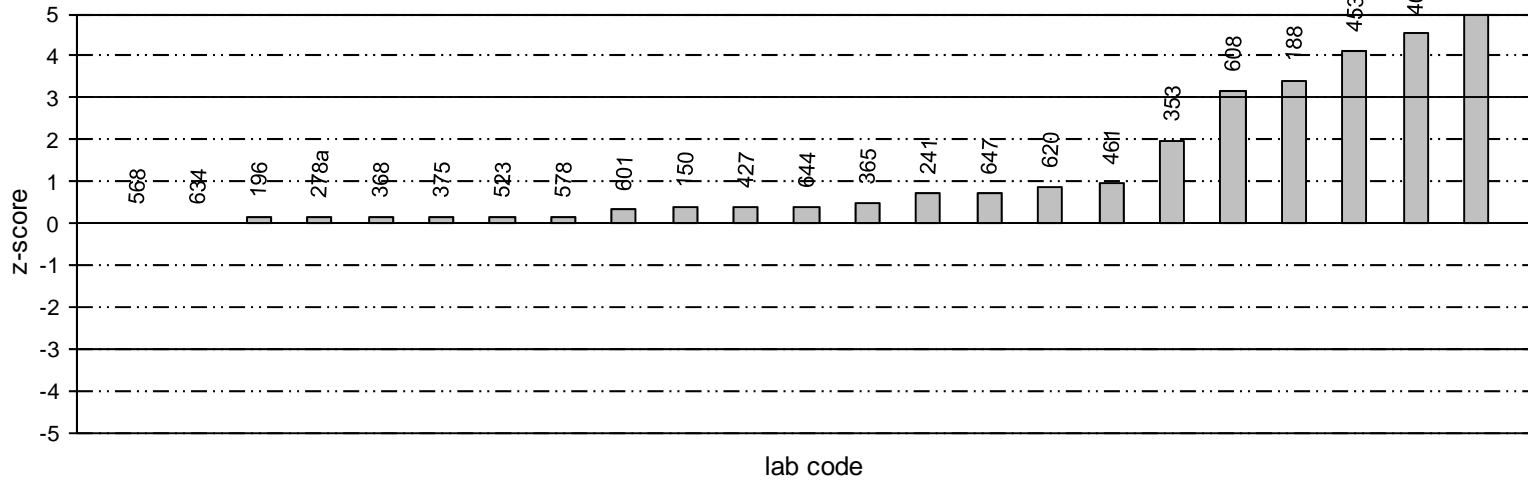
- 1 Where reported, results are shown with their corresponding measurement uncertainty (MU).
- 2 "§" denotes outliers (i.e. those results for which  $|z\text{-score}| \geq 3.0$ ). Robust z-scores are calculated as:  

$$z = (A - \text{median}) \div \text{normalised IQR}$$
 where A is the participant laboratory's result.
- 3 Please refer to Appendix C (page C3-C4) for method code descriptions.

**Nitrate as N - Sample R151 - Robust Z-Scores**



**Robust Z-Scores**



**Ordered Robust Z-Score Charts**

**Nitrate as N - Sample R151**

# **Ammonia as N Results**

Sample R151

**Ammonia as N****Results by Laboratory Code**

| Sample R151 |        |   |                 |                             |                          |
|-------------|--------|---|-----------------|-----------------------------|--------------------------|
| Lab Code    | Result | ± | MU <sup>1</sup> | Robust z-score <sup>2</sup> | Method Code <sup>3</sup> |
|             | (mg/L) |   |                 |                             |                          |
| 109         | 19.1   | ± | 0.96            | 0.39                        | 7                        |
| 118         | 19.7   | ± | 1.8             | 1.05                        | 10                       |
| 142         | 17.8   | ± | 0.8             | -1.05                       | 1                        |
| 150         | 20.0   | ± | 2.0             | 1.38                        | 11                       |
| 179         | 17.0   |   | #               | -1.93                       | 1                        |
| 181         | 22.7   | ± | 3.677           | 4.35                        | 15                       |
| 183         | 18.9   |   | #               | 0.17                        | 12                       |
| 196         | 18.2   |   | #               | -0.61                       | 15                       |
| 207         | 18.2   | ± | 1.2             | -0.61                       | 15                       |
| 237         | 19.5   | ± | 1.95            | 0.83                        | 7                        |
| 240         | 19.2   |   | #               | 0.50                        | 15                       |
| 241         | 18.2   | ± | 1.85            | -0.61                       | 11                       |
| 268         | 19.4   | ± | 1               | 0.72                        | 15                       |
| 269a        | 17.40  | ± | 0.70            | -1.49                       | 4                        |
| 269b        | 17.70  | ± | 0.50            | -1.16                       | 15                       |
| 273         | 19.1   | ± | 0.8             | 0.39                        | 11                       |
| 278a        | 19.2   | ± | 2.30            | 0.50                        | 15                       |
| 278b        | 19.0   | ± | 1.90            | 0.28                        | 15                       |
| 285         | 18.7   | ± | 1.87            | -0.06                       | 11                       |
| 286         | 19.5   | ± | 3.90            | 0.83                        | 11                       |
| 303         | 19.2   | ± | 2.40            | 0.50                        | 10                       |
| 335         | 18.4   | ± | 0.90            | -0.39                       | 15                       |
| 343         | 18.0   | ± | 0.1             | -0.83                       | 1                        |
| 353         | 19.6   |   | #               | 0.94                        | 5                        |
| 365         | 18.8   |   | #               | 0.06                        | 15                       |
| 368         | 19.9   | ± | 3.8             | 1.27                        | 11                       |
| 375         | 20.0   | ± | 1.0             | 1.38                        | 11                       |
| 376         | 17.62  |   | #               | -1.24                       | 1                        |
| 378         | 18.9   | ± | 0.78            | 0.17                        | 1                        |
| 390a        | 19.6   | ± | 0.75            | 0.94                        | 15                       |
| 390b        | 17.1   | ± | 0.56            | -1.82                       | 15                       |
| 402         | 17.0   |   | #               | -1.93                       | 15                       |
| 422         | 17.9   |   | #               | -0.94                       | 12                       |

- 1 Where reported, results are shown with their corresponding measurement uncertainty (MU).
- 2 "**S**" denotes outliers (i.e. those results for which  $|z\text{-score}| \geq 3.0$ ). Robust z-scores are calculated as:  

$$z = (A - \text{median}) \div \text{normalised IQR}$$
 where A is the participant laboratory's result.
- 3 Please refer to Appendix C (page C3-C4) for method code descriptions.



**Ammonia as N - cont.****Results by Laboratory Code**

| Sample R151                       |        |   |                 |                             |                          |
|-----------------------------------|--------|---|-----------------|-----------------------------|--------------------------|
| Lab Code                          | Result | ± | MU <sup>1</sup> | Robust z-score <sup>2</sup> | Method Code <sup>3</sup> |
|                                   | (mg/L) |   |                 |                             |                          |
| 427                               | 18.3   | ± | 0.20            | -0.50                       | 15                       |
| 436                               | 19.4   | ± | 1.0             | 0.72                        | 9                        |
| 438                               | 18.5   | ± | 1.825           | -0.28                       | 11                       |
| 439                               | 18.7   | ± | 1.9             | -0.06                       | 7                        |
| 461                               | 19.3   | ± | 0.51            | 0.61                        | 7                        |
| 512                               | 19.6   |   | #               | 0.94                        | 15                       |
| 523                               | 18.16  | ± | 1.48            | -0.65                       | 1                        |
| 525                               | 21.4   |   | #               | 2.92                        | 7                        |
| 568                               | 19.7   | ± | 1.2             | 1.05                        | 15                       |
| 569                               | 18.5   | ± | 3.7             | -0.28                       | 10                       |
| 578                               | 18.5   | ± | 1.3             | -0.28                       | 1                        |
| 598                               | 18.9   | ± | 3.5             | 0.17                        | 15                       |
| 601                               | 18.4   |   | #               | -0.39                       | 4                        |
| 608                               | 25.1   | ± | 0.79            | 6.99 §                      | 11                       |
| 620                               | 18.7   | ± | 2.0             | -0.06                       | 7                        |
| 634                               | 18.5   | ± | 1.0             | -0.28                       | 7                        |
| 639                               | 18.0   |   | #               | -0.83                       | 10                       |
| 644                               | 18.6   | ± | 0.750           | -0.17                       | 11                       |
| 647                               | 16.4   | ± | 1.4             | -2.59                       | 15                       |
| <i>No of Results:</i>             | 52     |   |                 |                             |                          |
| <i>Median:</i>                    | 18.75  |   |                 |                             |                          |
| <i>Uncertainty of the Median:</i> | 0.16   |   |                 |                             |                          |
| <i>Normalised IQR:</i>            | 0.91   |   |                 |                             |                          |
| <i>Robust CV:</i>                 | 4.8%   |   |                 |                             |                          |
| <i>Minimum:</i>                   | 16.4   |   |                 |                             |                          |
| <i>Maximum:</i>                   | 25.1   |   |                 |                             |                          |
| <i>Range:</i>                     | 8.7    |   |                 |                             |                          |

1 Where reported, results are shown with their corresponding measurement uncertainty (MU).

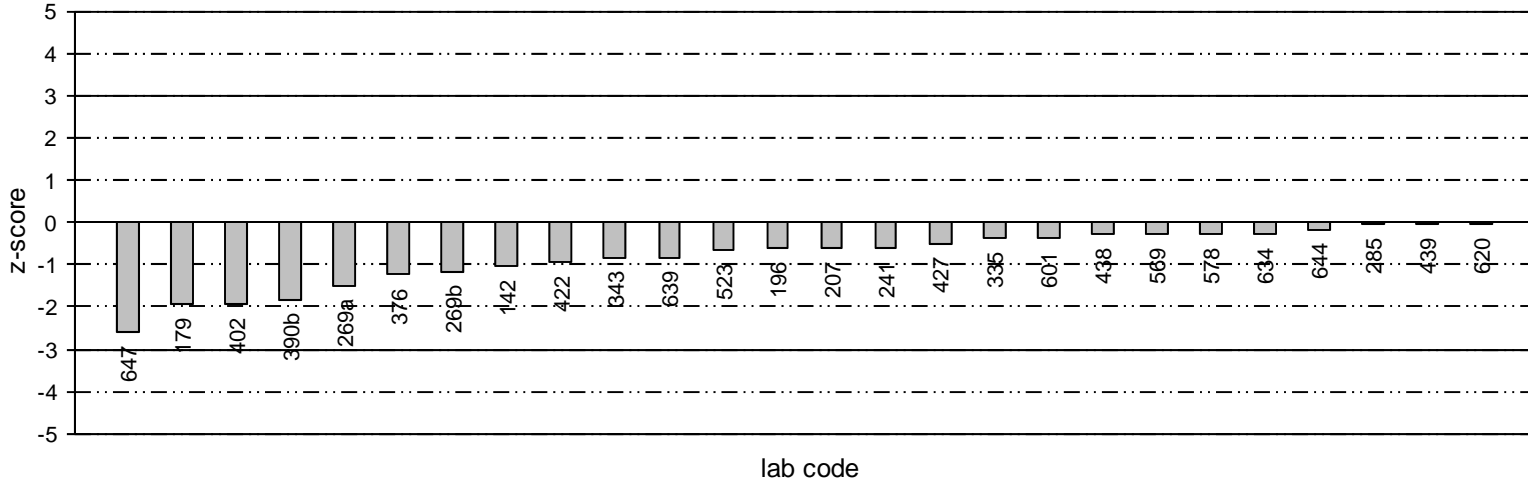
2 "§" denotes outliers (i.e. those results for which  $|z\text{-score}| \geq 3.0$ ). Robust z-scores are calculated as:  
 $z = (A - \text{median}) \div \text{normalised IQR}$ , where A is the participant laboratory's result.

3 Please refer to Appendix C (page C3-C4) for method code descriptions.

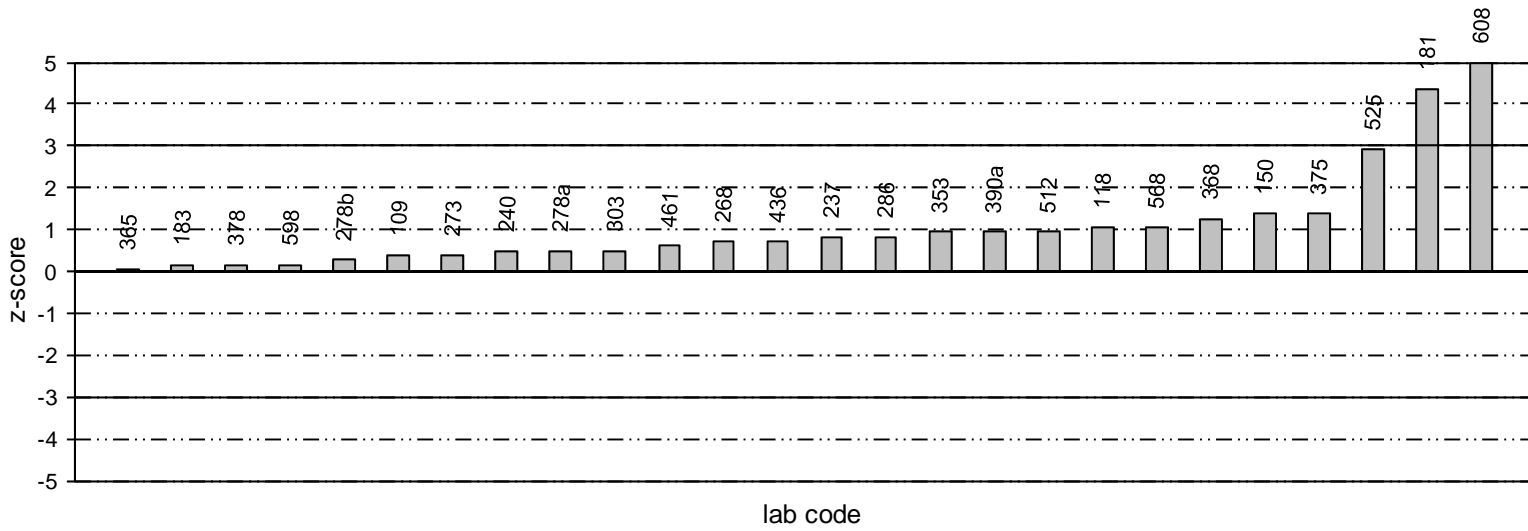
### Ammonia as N - Sample R151

#### Ordered Robust Z-Score Charts

#### Ammonia as N - Sample R151 - Robust Z-Scores



#### Robust Z-Scores



# Orthophosphate as P Results

Sample R151

**Orthophosphate as P**  
**Results by Laboratory Code**

| Sample R151 |        |   |                 |                             |                          |
|-------------|--------|---|-----------------|-----------------------------|--------------------------|
| Lab Code    | Result | ± | MU <sup>1</sup> | Robust z-score <sup>2</sup> | Method Code <sup>3</sup> |
|             | (mg/L) |   |                 |                             |                          |
| 109         | 3.90   | ± | 0.12            | -0.22                       | 30                       |
| 118         | 4.07   | ± | 0.15            | 0.84                        | 33                       |
| 142         | 3.95   | ± | 0.33            | 0.09                        | 29                       |
| 150         | 3.80   | ± | 0.4             | -0.84                       | 34                       |
| 181         | 3.77   | ± | 0.313           | -1.02                       | 33                       |
| 183         | 3.67   |   | #               | -1.64                       | 35                       |
| 188         | 3.70   |   | #               | -1.46                       | 38                       |
| 196         | 2.16   |   | #               | -11.01                      | § 31                     |
| 207         | 3.82   | ± | 0.17            | -0.71                       | 33                       |
| 217         | 5.10   | ± | 0.8             | 7.23                        | § 29                     |
| 240         | 3.84   |   | #               | -0.59                       | 29                       |
| 241         | 3.92   | ± | 0.40            | -0.09                       | 34                       |
| 268         | 3.91   | ± | 0.5             | -0.16                       | 32                       |
| 269a        | 3.53   | ± | 0.50            | -2.51                       | 30                       |
| 269b        | 4.15   | ± | 0.80            | 1.33                        | 32                       |
| 273         | 3.99   | ± | 0.18            | 0.34                        | 34                       |
| 278a        | 3.96   | ± | 0.475           | 0.16                        | 38                       |
| 278b        | 4.10   | ± | 0.492           | 1.02                        | 38                       |
| 285         | 3.83   | ± | 0.38            | -0.65                       | 34                       |
| 286         | 4.15   | ± | 0.83            | 1.33                        | 38                       |
| 303         | 3.89   | ± | 0.486           | -0.28                       | 37                       |
| 335         | 3.66   | ± | 0.08            | -1.71                       | 30                       |
| 343         | 5.2    | ± | 0.06            | 7.85                        | § 30                     |
| 353         | 4.49   |   | #               | 3.44                        | § 30                     |
| 365         | 3.94   | ± | 0.8             | 0.03                        | 29                       |
| 368         | 3.91   | ± | 0.66            | -0.16                       | 34                       |
| 375         | 3.7    | ± | 0.2             | -1.46                       | 34                       |

1 Where reported, results are shown with their corresponding measurement uncertainty (MU).

2 "§" denotes outliers (i.e. those results for which  $|z\text{-score}| \geq 3.0$ ). Robust z-scores are calculated as:  

$$z = (A - \text{median}) \div \text{normalised IQR}$$
 where A is the participant laboratory's result.

3 Please refer to Appendix C (page C3-C4) for method code descriptions.

**Orthophosphate as P - cont.****Results by Laboratory Code**

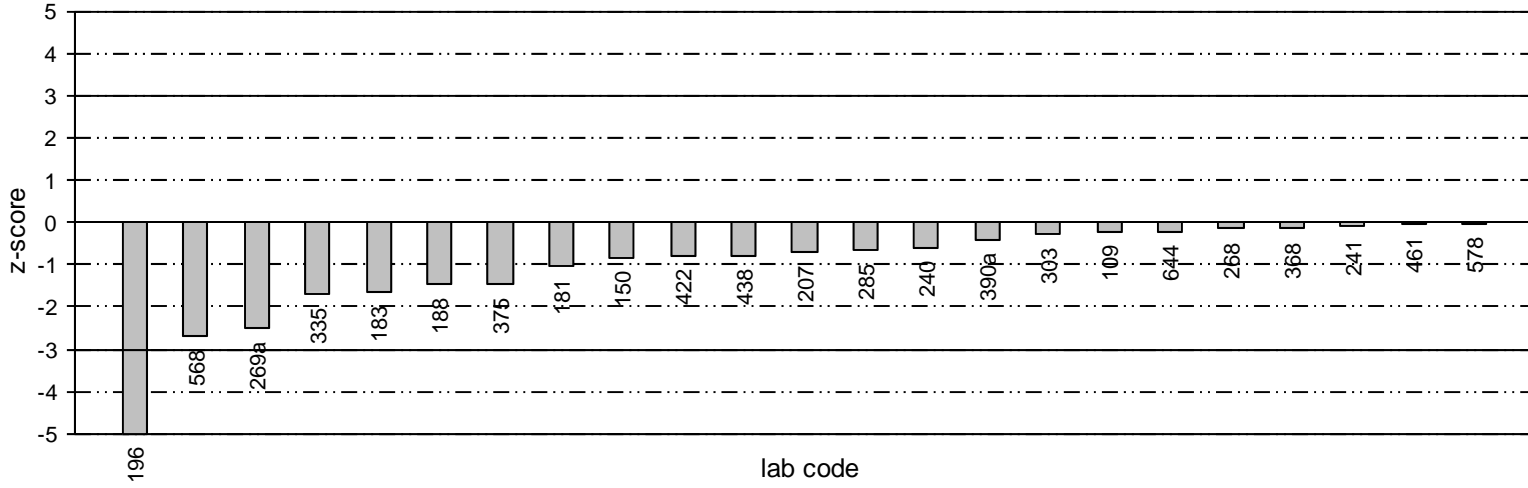
| Sample R151 |        |   |                 |                             |                          |
|-------------|--------|---|-----------------|-----------------------------|--------------------------|
| Lab Code    | Result | ± | MU <sup>1</sup> | Robust z-score <sup>2</sup> | Method Code <sup>3</sup> |
|             | (mg/L) |   |                 |                             |                          |
| 378         | 4.15   | ± | 0.10            | 1.33                        | 34                       |
| 390a        | 3.87   | ± | 0.12            | -0.40                       | 33                       |
| 390b        | 4.01   | ± | 0.14            | 0.47                        | 33                       |
| 422         | 3.81   |   | #               | -0.78                       | 35                       |
| 436         | 4.03   | ± | 0.22            | 0.59                        | 32                       |
| 438         | 3.81   | ± | 0.522           | -0.78                       | 34                       |
| 461         | 3.93   | ± | 0.45            | -0.03                       | 31                       |
| 512         | 4.04   |   | #               | 0.65                        | 38                       |
| 523         | 4.04   | ± | 0.24            | 0.65                        | 34                       |
| 525         | 4      |   | #               | 0.40                        | 29                       |
| 568         | 3.50   | ± | 0.40            | -2.70                       | 31                       |
| 569         | 3.94   | ± | 0.79            | 0.03                        | 33                       |
| 578         | 3.93   | ± | 0.18            | -0.03                       | 31                       |
| 601         | 3.96   |   | #               | 0.16                        | 29                       |
| 620         | 4.34   | ± | 0.4             | 2.51                        | 32                       |
| 621         | 4.04   |   | #               | 0.65                        | 32                       |
| 639         | 4.32   |   | #               | 2.39                        | 33                       |
| 644         | 3.90   | ± | 0.133           | -0.22                       | 34                       |
| 647         | 5.89   | ± | 1.00            | 12.13 §                     | 29                       |

|                                   |       |
|-----------------------------------|-------|
| <i>No of Results:</i>             | 46    |
| <i>Median:</i>                    | 3.935 |
| <i>Uncertainty of the Median:</i> | 0.030 |
| <i>Normalised IQR:</i>            | 0.161 |
| <i>Robust CV:</i>                 | 4.1%  |
| <i>Minimum:</i>                   | 2.16  |
| <i>Maximum:</i>                   | 5.89  |
| <i>Range:</i>                     | 3.73  |

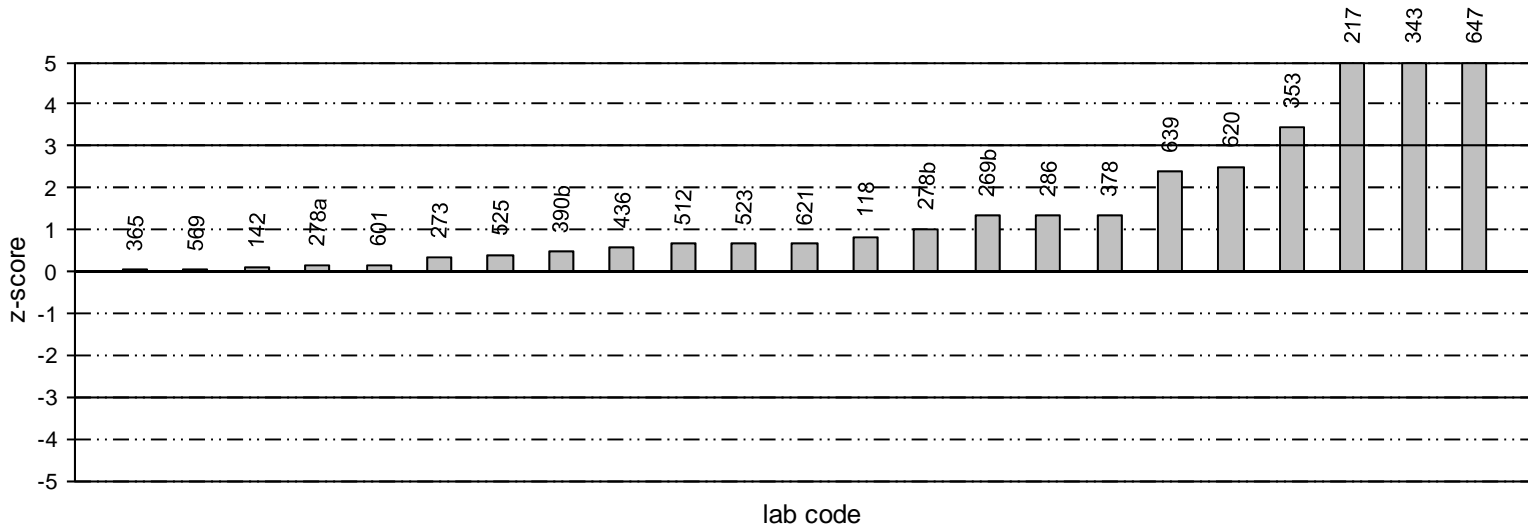
- 1 Where reported, results are shown with their corresponding measurement uncertainty (MU).
- 2 "§" denotes outliers (i.e. those results for which  $|z\text{-score}| \geq 3.0$ ). Robust z-scores are calculated as:  

$$z = (A - \text{median}) \div \text{normalised IQR}$$
 where A is the participant laboratory's result.
- 3 Please refer to Appendix C (page C3-C4) for method code descriptions.

**Orthophosphate as P - Sample R151 - Robust Z-Scores**



**Robust Z-Scores**



**Orthophosphate as P - Sample R151  
Ordered Robust Z-Score Charts**

# **APPENDIX B**

## **Sample Homogeneity and Stability**

Homogeneity and Stability Testing..... B1

**Homogeneity and Stability Testing**

Certified reference samples for this program were obtained from the Environmental Resource Associates (ERA). As such, all samples are subjected to rigorous stability and homogeneity testing. On the basis of this testing, the vials utilised for this program were considered to be homogeneous and stable.

Table 7 below presents the certified values for the samples used in Round 151.

| <b>Analyte</b>      | <b>Certified Value<sup>1</sup><br/>(mg/L)</b> | <b>Uncertainty<sup>2</sup></b> |
|---------------------|---|--------------------------------|
| Nitrate as N        | 21.6  | 6.9%                           |
| Ammonia as N        | 18.5  | 0.6%                           |
| Orthophosphate as P | 3.90  | 4.8%                           |

Table 7. Certified values of the certified reference samples used in Round 151.

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<sup>1</sup> The Certified Values are the actual “made-to” concentrations confirmed by ERA analytical verification.

<sup>2</sup> The stated Uncertainty is the total propagated uncertainty at the 95% confidence interval. The uncertainty is based on the preparation and analytical verification of the product by ERA, multiplied by a coverage factor which is equal with the Student t factor at 95% confidence interval at n-1 degrees of freedom. The uncertainty applies to the product as supplied and does not take into account any required or optional dilution and/or preparations the laboratory may perform while using this product.



# APPENDIX C

## Documentation

|                                    |    |
|------------------------------------|----|
| Instructions to Participants ..... | C1 |
| Method Codes .....                 | C3 |
| Results Sheet .....                | C5 |

## PROFICIENCY TESTING AUSTRALIA



## WATERS PROFICIENCY TESTING PROGRAM

**CHEMICAL ANALYSIS ROUND 151**

January 2013

**Nitrate, Ammonia, Orthophosphate****INSTRUCTIONS TO PARTICIPANTS*****\*\*Please record (on the Results Sheet) the approximate temperature of the samples upon receipt\*\****

Please note the following before commencing the analysis of the samples.

**1. Samples**

- i) One sealed vial labelled PTA R151 supplied by the ERA.
- ii) Please note the temperature of vial on receipt.
- iii) The vial contains approximately 14mL of artificial waste water concentrate.
- iv) The sample has been acidified with 1% hydrochloric.
- v) The sample must be thoroughly mixed prior to analysis.
- vi) The vial will require 200-fold dilution in deionised water.
- vii) The vial may be stored at room temperature.

**Please Note:** Where possible, proficiency testing samples should be treated as a routine laboratory sample.**2. Sample Preparation****Caution:** Analysis must begin immediately after vial is opened.

- i) Add 100 – 200mL deionised water to a clean 1000mL volumetric flask.
- ii) Invert the sample vial prior to opening, to ensure the sample is mixed.
- iii) Using a clean pipette, volumetrically pipette 5.0mL of the sample into the 1000mL volumetric flask.
- iv) Dilute the flask to the final volume with deionised water.
- v) Stopper and mix by inversion.
- vi) Immediately analyse the diluted sample using your normal procedures.
- vii) Report your results as mg/L for the diluted sample.

**3. Tests Requested**

For the sample prepared from the vial.

- i) Nitrate
- ii) Ammonia
- iii) Orthophosphate

**Be sure to report the Nitrate result as N (not as  $\text{NO}_3^-$ ), the Ammonia result as N (not as  $\text{NH}_3$ ) and the Orthophosphate result as P (not as  $\text{PO}_4^{3-}$ ).**

#### 4. Safety

- i) Samples are for laboratory use only.
- ii) Participants should have sufficient experience and training to take the necessary precautions when handling the samples and reagent chemicals and during disposal.
- iii) Use of safety glasses, gloves, and fume hoods, where appropriate during the determinations, is recommended.

#### 5. Reporting

- i) Report results using three significant figures.
- ii) Report results in milligrams per litre (mg/L).
- iii) Do not correct results for recovery.
- iv) In addition to reporting the results, record the method of analysis using the attached codes.
- v) Laboratories are also requested to calculate and report an estimate of measurement uncertainty (MU) for each reported measurement result. All estimates of MU must be given as a 95% confidence interval (coverage factor  $k \approx 2$ ) and reported in milligrams per litre (mg/L).

6. Testing should commence as soon as possible after receiving the sample and results reported **NO LATER THAN 15 FEBRUARY 2013 to:**

Delfina Mihaila  
 Proficiency Testing Australia  
 PO Box 7507  
 SILVERWATER NSW 2128  
 AUSTRALIA  
**Phone:** +612 9736 8397  
**Fax:** +612 9743 6664  
**Email:** [dmihaila@pta.asn.au](mailto:dmihaila@pta.asn.au)

7. For this program your laboratory has been allocated the code number shown on the attached Results Sheet. All reference to your laboratory in reports associated with the program will be through this code number, thus ensuring the confidentiality of your results.

8. As a guide, ranges for the samples can be expected to be (in mg/L):

| Analyte             | Range (mg/L) |
|---------------------|--------------|
| Nitrate as N        | 0.1 - 50     |
| Ammonia as N        | 0.5 - 30     |
| Orthophosphate as P | 0.1 - 10     |

**Method Codes to be used for the Results Sheet**

| ANALYSIS                              | METHOD REFERENCE       | METHOD DESCRIPTION  | CODE |
|---------------------------------------|------------------------|---|------|
| Ammonia Nitrogen (NH <sub>3</sub> -N) | APHA SM                | Part 4500 – NH <sub>3</sub> B. + C. Preliminary Distillation Step and Titrimetric Method                                      | 1    |
|                                       |                        | Part 4500 – NH <sub>3</sub> B. + D. Preliminary Distillation Step and Ammonia-Selective Electrode Method                      | 2    |
|                                       |                        | Part 4500 – NH <sub>3</sub> B. + E. Preliminary Distillation Step and Ammonia-Selective Electrode Method Using Known Addition | 3    |
|                                       |                        | Part 4500 – NH <sub>3</sub> B. + F. Preliminary Distillation Step and Phenate Method  | 4    |
|                                       |                        | Part 4500 – NH <sub>3</sub> B. + G. Preliminary Distillation Step and Automated Phenate Method                                | 5    |
|                                       |                        | Part 4500 – NH <sub>3</sub> B. + H. Preliminary Distillation Step and Flow Injection Analysis                                 | 6    |
|                                       |                        | Part 4500 – NH <sub>3</sub> D. Ammonia-Selective Electrode Method   | 7    |
|                                       |                        | Part 4500 – NH <sub>3</sub> E. Ammonia-Selective Electrode Method Using Known Addition  | 8    |
|                                       |                        | Part 4500 – NH <sub>3</sub> F. Phenate Method   | 9    |
|                                       |                        | Part 4500 – NH <sub>3</sub> G. Automated Phenate Method   | 10   |
|                                       |                        | Part 4500 – NH <sub>3</sub> H. Flow Injection Analysis  | 11   |
|                                       | US EPA                 | Method Number: 350.1: Ammonia (as nitrogen) - Colorimetric / Ammonia (as N) Semi-Automated Colorimetry                        | 12   |
|                                       |                        | Method Number: 350.2: Ammonia (as N) -Colorimetric, Titrimetric   | 13   |
|                                       |                        | Method Number: 350.3: Ammonia (as N) - Potentiometric   | 14   |
|                                       | Other (please specify) | 15  |      |
| Nitrate Nitrogen (NO <sub>3</sub> -N) | APHA SM                | Part 4110 B. Ion Chromatography with Chemical Suppression of Eluent Conductivity  | 16   |
|                                       |                        | Part 4500 – NO <sub>3</sub> <sup>-</sup> B. Ultraviolet Spectrophotometric Screening Method                                   | 17   |
|                                       |                        | Part 4500 - NO <sub>3</sub> <sup>-</sup> C. Second-Derivative Ultraviolet Spectrophotometric Method (Proposed)                | 18   |
|                                       |                        | Part 4500 - NO <sub>3</sub> <sup>-</sup> D. Nitrate Electrode Method  | 19   |
|                                       |                        | Part 4500 – NO <sub>3</sub> <sup>-</sup> E. Cadmium Reduction Method  | 20   |

| ANALYSIS  | METHOD REFERENCE                                     | METHOD DESCRIPTION  | CODE  |
|---|--|---|---|
| Nitrate Nitrogen<br>(NO <sub>3</sub> <sup>-</sup> -N)<br>(cont.)        | APHA SM<br>(cont.)                                   | Part 4500 – NO <sub>3</sub> <sup>-</sup> F.<br>Automated Cadmium Reduction Method                                 | 21  |
|   |  | Part 4500 – NO <sub>3</sub> <sup>-</sup> H.<br>Automated Hydrazine Reduction Method                               | 22  |
|   |  | Part 4500 – NO <sub>3</sub> <sup>-</sup> I.<br>Cadmium Reduction Flow Injection Analysis                          | 23  |
|   | US EPA   | Method Number: 300.0<br>Nitrate-N by IC   | 24  |
|   |  | Method Number: 353.2<br>Nitrate-Nitrate by Automated Colorimetry<br>Nitrate-Nitrite (as N) - Colorimetric/Cadmium | 25  |
|   |  | Method Number: 353.3<br>Nitrate-Nitrite (as N) -Manual Cadmium  | 26  |
|   |  | Nitrate by the enzyme Nitrate Reductase method  | 27  |
|   |  | Other (please specify)  | 28  |
|   | Orthophosphate<br>(PO <sub>4</sub> <sup>3-</sup> -P) | APHA SM   | Part 4110 B. Ion Chromatography with Chemical<br>Suppression of Eluent Conductivity                                     |
| Part 4500 – P C.<br>Vanadomolybdophosphoric Acid Colorimetric<br>Method |  |   | 30  |
| Part 4500 – P D.<br>Stannous Chloride Method                            |  |   | 31  |
| Part 4500 – P E.<br>Ascorbic Acid Method                                |  |   | 32  |
| Part 4500 – P F.<br>Automated Ascorbic Acid Reduction Method            |  |   | 33  |
| Part 4500 – P G.<br>Flow Injection Analysis for Orthophosphate          |  |   | 34  |
| US EPA  |  |   | Method Number: 365.1<br>Orthophosphate (as P) - Automated Colorimetry<br>Phosphorus, All Forms - Colorimetric/Automated |
|   |  | Method Number: 365.2<br>Phosphorus - manual colorimetric/1 reagent  | 36  |
|   |  | Method Number: 365.3<br>Phosphorus - colorimetric/ 2 reagents   | 37  |
|   |  | Other (please specify)  | 38  |

#### Method Reference Key

- i) APHA SM      APHA “Standard Methods for the Examination of Water and Wastewater” (18, 19, 20, 21 and 22 Edition).
- ii) USEPA      U.S Environmental Protection Agency,  
<http://www.epa.gov/osa/fem/methcollectns.htm>.



**PROFICIENCY TESTING AUSTRALIA**  
**WATERS PROFICIENCY TESTING PROGRAM**  
**CHEMICAL ANALYSIS ROUND 151**  
**NITRATE, AMMONIA, ORTHOPHOSPHATE**

**JANUARY, 2013**

**RESULTS SHEET**  
**(mg/L)**

Laboratory Code «Code»

\*Approximate temperature of samples upon receipt:

| ANALYSIS            | R 151  |      | METHOD CODE |
|---------------------|--------|------|-------------|
|                     | Result | ±MU* |             |
| Nitrate as N        |        |      |             |
| Ammonia as N        |        |      |             |
| Orthophosphate as P |        |      |             |

**Please Note:**

- Where possible, proficiency testing samples should be treated as a routine laboratory sample.
  - Be sure to report the Nitrate result as N (not as  $\text{NO}_3^-$ ), the Ammonia result as N (not as  $\text{NH}_3$ ) and the Orthophosphate result as P (not as  $\text{PO}_4^{3-}$ ).
- i) For the prepared sample only a single result is requested.
  - ii) Report the results for the diluted sample.
  - iii) Report results using three significant figures (eg.: 0.123, 1.23, 12.3).
  - iv) Report results in milligrams per litre (mg/L).
  - v) Do not correct results for recovery.
  - vi) MU\* Laboratories Measurement Uncertainty (MU) if known for the result. Please report in milligrams per litre (mg/L).

**DATE** \_\_\_\_\_

**SIGNATURE** \_\_\_\_\_

|  |  |
|--|--|
| Return results <b>NO LATER THAN 15 FEBRUARY 2013</b> to: |  |
| Delfina Mihaila  |  |
| Proficiency Testing Australia                            |  |
| PO Box 7507  |  |
| SILVERWATER NSW 2128                                     | <b>Phone:</b> +61 2 9736 8397  |
| AUSTRALIA  | <b>Fax:</b> +61 2 9743 6664  |
|  | <b>Email:</b> <a href="mailto:dmihaila@pta.asn.au">dmihaila@pta.asn.au</a> |

INSTRUCT WATERS PROF TEST PROG 151

*- End of Report -*