**Review of Method Performance and Uncertainty for 2021**

**Actual and Apparent Alcohol Strength (OP221 & OP222)**

1. **Determination of Apparent Alcohol Strength (OP221)**

A total of 4 QC samples were used in 2021 and performance data is summarised in Table 1. The data from each QC is compared against the uncertainty document from 2007[[1]](#footnote-2)&[[2]](#footnote-3).

**Table 1:** Summary statistics for apparent strength quality control samples in 2021

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **QC Name** | **AppS048** | **AppS049** | **AppS050** | **AppS051** |
| **Dates this QC was in use** | 03/02/2020 to 05/05/2021 | 19/03/2021 to 20/07/2021 | 06/07/2021 to 13/01/2022 | 12/11/2021 to 19/01/2022 |
| **No. of Points** | 86 | 42 | 48 | 10 |
| **Mean** | 42.50 | 42.33 | 40.47 | 40.55 |
| **Standard Deviation** | 0.009 | 0.024 | 0.008 | 0.009 |
| **%CV** | 0.02 | 0.02 | 0.02 | 0.02 |
| **Minimum on QC Chart** | 42.47 | 42.26 | 40.45 | 40.53 |
| **Maximum of QC Chart** | 42.52 | 42.36 | 40.48 | 40.56 |
| **Uncertainty (2007)** | 0.05% abv | 0.05%abv | 0.05%abv | 0.05%abv |
| **Lower Limit** | 42.47 | 42.27 | 40.44 | 40.52 |
| **Upper Limit** | 42.53 | 42.39 | 40.50 | 40.61 |

Precision data from the 2007 uncertainty document used data from 6 quality control samples, all approximately 40% alcohol v/v, and repeated analyses of these gave standard deviations between 0.009 and 0.021% abv. Standard deviations for two of the QC samples used in 2021, AppS048 and AppS051, are within this range. AppS049 is slightly above this range with a standard deviation of 0.024% abv and AppS050 is slightly below this range at 0.008% abv. This indicates the method has been performed with satisfactory precision during 2021. Note that the expiry date for AppS048 was extended for a further 6 months after review when the laboratory reopened during the COVID-19 pandemic.

**Certified Reference Material**

Certified reference materials with strengths between 5 and 70.08% abv were analysed 12 times in 2021 and the results listed in Table 2.

Across all strengths the results give a root mean square bias of 0.020% abv. Taking into account the uncertainties of the certified reference material gives a combined uncertainty arising from the accuracy of the method of 0.029% abv. This is greater than the values in the uncertainty document (0.020% abv at 40%). Combined with the maximum standard deviation from Table 1, this would give a standard uncertainty for the method of 0.038% abv and expanded uncertainty of 0.075% abv.

**Table 2**: Summary of the analysis of certified reference materials in 2021

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| **Date** | **SWRI ref no.** | **Certified Strength** | **Uncertainty** | **SWRI Result** | **Bias** |
| 15/03/2021 | C-7235 | 5.00 | 0.03 | 4.99 | -0.01 |
| 02/07/2021 | C-7236/7 | 5.00 | 0.03 | 4.99 | -0.01 |
| 15/03/2021 | C-7215 | 15.00 | 0.04 | 14.97 | -0.03 |
| 02/07/2021 | C-7216/7 | 15.00 | 0.04 | 14.98 | -0.02 |
| 14/01/2021 | C-7224 | 40.04 | 0.04 | 40.039 | -0.001 |
| 15/03/2021 | C-7225 | 40.00 | 0.06 | 40.05 | 0.05 |
| 04/06/2021 | C-7630 | 40.04 | 0.04 | 40.05 | 0.01 |
| 02/07/2021 | C-7629 | 40.04 | 0.04 | 40.03 | -0.01 |
| 12/07/2021 | C-7628 | 40.04 | 0.04 | 40.05 | 0.01 |
| 07/10/2021 | C-7632 | 40.04 | 0.04 | 40.03 | -0.01 |
| 15/03/2021 | C-7229 | 70.00 | 0.07 | 70.01 | 0.01 |
| 02/07/2021 | C-7228/30 | 70.08 | 0.03 | 70.06 | -0.02 |

***Proficiency Schemes***

DAPS analysis performed very well over 2021 with Z scores between 2 and -2 being achieved. There was only one case where Z scores outside of 2 and -2 were achieved (Round 104 B4, Z = 2.33). Summary of reported results, their bias compared with the assigned values, assigned values and their associated uncertainty are given in Table 3 for the different matrices analysed. B1 = Scotch whisky; B2 = dark spirit; B3 = clear spirit and B4 = simulant.

**Table 3**: Summary of DAPS apparent strength results in 2021

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| **Round** | **Sample** | **SWRI Result** | **Assigned (DAPS)** | **Uncertainty** | **Bias** |
| 104 | B1 | 39.83 | 39.90 | 0.010 | -0.07 |
| 104 | B3 | 37.41 | 37.42 | 0.00 | -0.01 |
| 104 | B4 | 38.03 | 38.10 | 0.00 | -0.07 |
| 105 | B1 | 39.89 | 39.89 | 0.006 | 0.00 |
| 105 | B2 | 39.29 | 39.30 | 0.006 | -0.01 |
| 105 | B4 | 47.43 | 47.43 | 0.005 | 0.00 |
| 106 | B1 | 39.79 | 39.80 | 0.005 | -0.01 |
| 106 | B3 | 37.61 | 37.61 | 0.010 | 0.00 |
| 106 | B4 | 39.16 | 39.19 | 0.029 | -0.03 |

Across all sample types the results give a root mean square bias of 0.035% abv. Combined with the uncertainties of the assigned value this gives a combined uncertainty arising from the accuracy of the method of 0.036% abv. This is close to but above the standard uncertainty calculated in the 2007 document (0.026% abv). DAPS covers a wider range of matrices than the 2007 document and this document is already pending revision to reflect this.

***Technical Anomalies /Departures***

* TA357: 10 run on the same side of the mean Status: CLOSED
* TA387: Difference between QC app and act is 0.0 Status: CLOSED
* TA391: Bracket check failedStatus: CLOSED
* TA392: QC issues Status: CLOSED
* TA395: Failed bracket check and QC points on warning limit Status: CLOSED

A total of 5 Technical Anomalies were raised for this method in 2021, all of which have been closed. This was less than the number recorded in 2020. The proportion of Technical Anomalies to runs performed in 2020 was 8 whilst in 2021 it was 17. Technical Anomalies generally related to bracket check/repeatability failures (resolved after following instrument maintenance procedures) and environmental effects on the density meter and QC samples (humidity and temperature increase). 4 Technical Anomalies were opened against control chart bias. One of which was attributed to humidity and temperature changes and the other two were resolved by following instrument maintenance procedures. All 4 have since been closed.

No departures were recorded against this method in 2021.

***Conclusions***

The precision of this method during 2021 was within the limits of the 2007 uncertainty document. Based on the analysis of the certified reference materials and proficiency scheme samples, the uncertainty of the method accuracy is greater than in the 2007 document and this should be revised.

The Technical Anomalies raised were all actioned appropriately and thorough investigations carried out. Close attention will continue to be paid to the control chart to monitor the bias, but since all points are within/on two standard deviations of the mean the performance of the method is not significantly impacted.

**Determination of Actual Alcohol Strength (OP 222)**

A total of 4 QC samples were used in 2021 and performance data is summarised in Table 4. The data from each QC is compared against the uncertainty document from 2007.

|  |  |
| --- | --- |
| **Table 4** | Summary statistics for actual strength quality control samples in 2021 |

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **QC Name** | **ActS040** | **ActS041** | **ActS042** | **ActS043** |
| **Dates this QC was in use** | 07/09/20 to 13/05/21 | 02/04/21 to 03/08/21 | 12/07/21 to 04/10/21 | 08/09/21 to 19/01/21 |
| **No. of Points** | 26 | 17 | 22 | 15 |
| **Mean** | 41.23 | 42.44 | 40.55 | 44.46 |
| **Standard Deviation** | 0.12 | 0.073 | 0.081 | 0.042 |
| **%CV** | 0.15 | 0.14 | 0.15 | 0.13 |
| **Minimum on QC Chart** | 40.71 | 42.28 | 40.30 | 44.38 |
| **Maximum on QC Chart** | 41.38 | 42.58 | 40.69 | 44.56 |
| **Uncertainty (2007)** | 0.11% abv | 0.11% abv | 0.11% abv | 0.11% abv |
| **Lower Limit** | 40.87 | 42.22 | 40.31 | 44.33 |
| **Upper Limit** | 41.59 | 42.66 | 40.79 | 44.59 |

Precision data from the 2007 uncertainty document used data from 8 quality control samples, all approximately 40%, and repeated analyses of these gave standard deviations between 0.025 and 0.072% abv. Standard deviations for one of the QC samples used in 2021, ActS043, is within this range. ActS041 and ActS042 are both slightly above this range with a standard deviation of 0.073% abv and 0.081% abv respectively. The standard deviation for ActS040, 0.12% abv, is much higher than the range given in the 2007 uncertainty document, 0.025 to 0.072% abv. However, this QC sample includes a data point significantly lower than usual. This data point is from a run during which the gas was turned off to the laboratory whilst the distillations were running. Removing that data point from ActS040 results in a standard deviation of 0.059% abv which falls within the range given in the 2007 uncertainty document. This indicates the method has been performed with satisfactory precision during 2020.

***Proficiency Schemes***

DAPS analysis performed well over 2021 with Z scores between 2 and -2 being achieved. There was only one case where Z scores outside of 2 and -2 were achieved (Round 104 B4, Z = 25.12). Summary of reported results, their bias compared with the assigned values, assigned values and their associated uncertainty are given in Table 5 for the different matrices analysed. B1 = Scotch whisky; B2 = dark spirit; B3 = clear spirit; B4 = simulant; D1 = white wine; D2 = red wine; E1 = “ready to drink” beverage and E2 = liqueur.

Across all sample types the results give a root mean square bias of 0.54% abv. Combined with the uncertainties of the assigned value and the uncertainty from the determination of apparent strength, this gives a combined uncertainty arising from the accuracy of the method of 0.54% abv. Omitting round 104 B4 from the calculations gives a root mean square bias of 0.055% abv and a combined uncertainty arising from the accuracy of the method of 0.058% abv. This is close to but above the standard uncertainty calculated in the 2007 document (0.056% abv). DAPS covers a wider range of matrices than the 2007 document and this document is already pending revision to reflect this.

**Table 5:** Summary of DAPS actual strength results in 2021

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| **Round** | **Sample** | **SWRI Result** | **Assigned (DAPS)** | **Uncertainty** | **Bias** |
| 104 | B1 | 39.96 | 39.97 | 0.010 | -0.01 |
| 104 | B3 | 37.37 | 37.40 | 0.010 | -0.03 |
| 104 | B4 | 36.14 | 38.15 | 0.020 | -2.01 |
| 104 | D2 | 13.30 | 13.33 | 0.020 | -0.03 |
| 104 | E1 | 4.86 | 4.92 | 0.020 | -0.06 |
| 105 | B1 | 39.91 | 40.00 | 0.009 | -0.09 |
| 105 | B2 | 40.10 | 40.05 | 0.024 | 0.05 |
| 105 | B4 | 47.39 | 47.47 | 0.022 | -0.08 |
| 105 | E2 | 35.06 | 35.07 | 0.032 | -0.01 |
| 106 | B1 | 39.91 | 39.93 | 0.017 | -0.02 |
| 106 | B3 | 37.58 | 37.60 | 0.026 | -0.02 |
| 106 | B4 | 39.10 | 39.22 | 0.029 | -0.12 |
| 106 | D1 | 11.59 | 11.63 | 0.015 | -0.04 |
| 106 | E1 | 3.91 | 3.92 | 0.012 | -0.01 |

***Technical Anomalies/Departures.***

* TA360: Discrepancy in results of repeated sample Status: CLOSED
* TA374: Actual strength QC below 2sd Status: CLOSED
* **TA382: 200ml dilution (SA) Status: Open**
* TA387: Difference between QC app and act is 0.0 Status: CLOSED
* TA395: Failed bracket check and QC points on warning limit Status: CLOSED
* TA405: Failed QC check Status: CLOSED
* TA408: Filling error for first measurement of bracket check Status: CLOSED

A total of 7 Technical Anomalies were raised for this method over 2021, though a number of the Technical anomalies listed for OP221, also affected results from this method and are listed in both sections. This is higher than the number of TAs in 2020 but consideration must also be given to the fact that the lab was closed for 3 months during the COVID-19 lockdown in 2020. The proportion of Technical Anomalies to runs performed in 2020 was 14 whilst in 2021 it was 12. 3 of the Technical Anomalies were related to bracket check/repeatability failures and were resolved after following instrument maintenance procedures. 3 of the Technical Anomalies were related to analyst error (overfilling the flask during the distillation/not maintaining the glycol temperature below 15 °C). The remaining Technical Anomaly related to a filling error by which was resolved by tightening the peristaltic pump.

No departures were recorded against this method in 2021.

***Conclusion***

This method performed satisfactorily during 2021 with uncertainties due to precision and accuracy comparable to the 2007 uncertainty document. Based on the analysis of the proficiency scheme samples, the uncertainty of the method accuracy is greater than in the 2007 document and this should be revised.

The Technical Anomalies raised were all actioned appropriately and thorough investigations carried out.

1. UNCERTAINTY For Operating Procedure 221 Document U221.V03 (2007):

   <https://365swri.sharepoint.com/:b:/r/quality/Quality%20System%20Document%20Library/Uncertainty/Uncertainty%20for%20OP221.pdf?csf=1> [↑](#footnote-ref-2)
2. UNCERTAINTY For Operating Procedure 222 (2007):

   <https://365swri.sharepoint.com/:b:/r/quality/Quality%20System%20Document%20Library/Uncertainty/Uncertainty%20for%20OP222.pdf?csf=1> [↑](#footnote-ref-3)