

Automation of QC Testing Using the Agilent Sample Prep WorkBench

QUALITY CONTROL



The accuracy, reliability and ease of use of the Agilent 7696A Sample Prep WorkBench provide accurate and error-free preparation of QC samples for the production of standards.

Reliable and accurate calibration of reference standards are a must for any laboratory running GC/MS. ULTRA Scientific provides such certified analytical standards, using stringent QC procedures to ensure their accuracy, reliability, and now adherence to ISO Guide 34 requirements as a certified reference material producer.

The ULTRA Scientific procedures require selection of random samples from the unitizing process to monitor homogeneity and ensure proper concentrations for each analyte in a standards mix. The concentrations of the components used to prepare the standard set are determined by constructing a calibration curve for each component, using a procedure that requires precise dilutions as well as the addition of an internal standard. The same internal standard is added in prescribed amounts to each vial sampled from the unitizing process. The actual concentration of each standard compound in the vial is determined by GC analysis, measuring the ratio of the area of the compound peak relative to that of the internal standard. This relative response is compared to the same ratio determined in a calibration run at the beginning of the QC sequence and comprised of known amounts of each analyte present in the standard being packaged. The result is a highly accurate determination of the actual concentrations of the compounds in the vial.

Traditionally, ULTRA Scientific has performed this QC procedure by manually pipetting the internal standards. However, the company recently tested the Agilent Sample Prep WorkBench to automate pipetting, using both a volatile and a semi-volatile standards mix. The WorkBench provided accuracy measurements in most cases equal to or better than those determined for the manual method. This was true for the volatile standard set, even though WorkBench punctures the vial seal to add the internal standard.

The Sample Prep WorkBench is a valuable tool for automating applications requiring reliable and accurate pipetting, including QC in a manufacturing environment.

Key Benefits

- Eliminates the opportunity for error inherent in manual methods
 - Accuracy equal to or better than that of manual methods
 - QC methods can be stored in the WorkBench for rapid access
 - User-friendly, templated software that is easy to master
 - Bar code reading for easy sample tracking
-

This work was performed as a collaboration between Scott A. Lorimer of ULTRA Scientific and Jared Bushey of Agilent Technologies.

The Measure of Confidence



Agilent Technologies

Method Used to Generate the QC Samples on the Agilent WorkBench

Method steps

1. Add 315 µL of methylene chloride (volatile standards set) or methanol (semivolatile standards set) to Empty Vials 1 at Back Tower.
2. Add 35 µL of sample to Empty Vials 1 at Front Tower.
3. Add 35 µL of biphenyl (volatile standards set) or fluorobenzene (semivolatile standards set) internal standard to Empty Vials 1 at Front Tower.
4. Mix Empty Vials 1 by vortex at 2,000 RPM, 5 sec spin, bidirectional, 2 cycles.
5. Flag Empty Vials 1 as 'Results'.

WorkBench configuration

| | |
|-----------------------------|------------------|
| Front injector syringe size | 100 µL |
| Rear injector syringe size | 500 µL |
| Barcode heater | enabled at 50 °C |

The amount of each analyte in the QC sample was calculated according to the following formula:

$$\left(\frac{\left(\frac{\text{Analyte area}}{\text{istd area}} \right)_{\text{Sample}}}{\left(\frac{\text{Analyte area}}{\text{istd area}} \right)_{\text{Calibration}}} \right) \times (\text{istd concentration})_{\text{Sample}}$$

Accuracy results for the QC of vials removed from a production run*

Volatile Standards Set

| Standard component | Manual % difference | WorkBench % difference |
|-----------------------------|---------------------|------------------------|
| 2-picoline | -1.1960 | 0.6202 |
| Acetophenone | -3.2765 | 0.3384 |
| N-nitrosopiperidine | -3.3925 | -2.6848 |
| a,a-dimethylphenethylamine | 0.9115 | 1.0354 |
| N-nitrosodi-n-butylamine | -0.2920 | 0.2560 |
| 1,2,4,5-tetrachlorobenzene | -0.8785 | 0.8129 |
| 1-chloronaphthalene | 9.4015 | 0.1805 |
| Pentachlorobenzene | -2.2000 | 0.7535 |
| Diphenylamine | -0.0380 | -0.4711 |
| Phenacetin | -2.1165 | 2.0917 |
| 4-Aminobiphenyl | -9.6435 | 5.1187 |
| Pentachloronitrobenzene | -2.5130 | 1.0443 |
| Pronamide | -3.7865 | 2.5000 |
| p-(dimethylamino)azobenzene | 2.1465 | 3.1419 |
| 7,12-dimethylbenz[A]anthra | -1.5785 | 0.0849 |
| 3-methylcholanthrene | 1.6965 | -0.4717 |
| Dibenz[A,J]acridine | 2.2478 | 1.7602 |

Semi-volatile Standards Set

| Standard component | Manual % difference | WorkBench % difference |
|---------------------------|---------------------|------------------------|
| 1,1-dichloroethene | -7.7578 | -1.0566 |
| trans-1,2-dichloroethene | -1.4115 | -3.8288 |
| cis-1,2-dichloroethene | 3.0163 | -1.6658 |
| Benzene | 3.1771 | -0.5289 |
| Trichloroethene | 3.2273 | -5.9651 |
| cis-1,3-dichloropropene | 5.7795 | -4.1192 |
| Toluene | 4.5507 | -1.5242 |
| trans-1,3-dichloropropene | 6.4737 | -2.8953 |
| Tetrachloroethene | 4.1876 | -4.7692 |
| Chlorobenzene | 7.0025 | -1.8823 |
| Ethylbenzene | 6.4856 | -0.9086 |
| Meta+para-xylene | 6.5132 | -1.9300 |

Learn more:

www.agilent.com/chem

Email:

info_agilent@agilent.com

Find a customer center in your country:

www.agilent.com/chem/contactus

This information is subject to change without notice.

© Agilent Technologies, Inc. 2012
Published in USA, January 6, 2012
5990-9671EN



Agilent Technologies