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.

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-butlamine	-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
Pentachloronitorbenzene	-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

*Accuracy is defined as the % difference between the amount of a given analyte determined to be in the sample versus the amount that should have been present, based on the calibration run data.

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