User Guide for NPL VOC Gas Standards

This information is provided as a general guide to help achieve optimum results when using an NPL VOC gas standard.

1. Cylinders

NPL VOC gas standards are supplied in 10-litre aluminium cylinders treated internally with the proprietary *Quantum* passivation process (Air Products). The gas standard is normally supplied at 100 bar. The cost of a standard includes the outright purchase of the cylinder, which is financially beneficial as this means there are no cylinder rental costs. Each cylinder has a unique identifier, which is engraved on the cylinder body, and is referenced on the certificate of calibration as the cylinder number.

2. Handling, Storage and Use

Cylinders can be stored between – 10 ºC and 70 ºC, in accordance with the safe operating range of the valve. Cylinders can also be used within this temperature range, however special care must be taken if a cylinder is used at the extremes of this recommended temperature range, as condensation and other transfer problems within the manifold may lead to a non-representative sample at the analyser. For this reason it is most straightforward for cylinders to be used at room temperature. Tests carried out at NPL confirm there is no measureable change in the standards stored at – 10 ºC for 46 hours and also at 50 ºC for 7 days. These tests confirm the expected behaviour of the standards and we would expect no further change if standards are exposed to similar temperatures over longer periods. Direct heat should not be applied to the cylinder as this may damage the cylinder walls and alter the composition of the gas mixture. Cylinders are supplied with metal caps, which should be kept on when not in use to avoid damage to the valve. If cylinders are stored outside then the valves must be kept clean.

3. Analysis

a) Sample path

The cylinders are fitted with DIN477 No. 1 valve outlets. A DIN 1 connector can be used to connect to the sample line. NPL recommends the use of a specialised VOC one-stage regulator for Scott Gases (Part No. 05119VOCG(DIN1)) if pressure regulation is required. This regulator has been specially designed for handling VOCs, with a low internal volume and treated metal surfaces. If pressure regulation is not required then a low flow restriction should be adopted, such as a needle valve with treated metal surfaces. The regulator or flow restrictor should be connected to the instrument by 1/16” silcosteel (Restek Ltd) tubing. Narrow sample paths reduce the surface area in contact with the gas stream and treated metals are proven to improve performance in measuring high molecular weight VOCs.

For in-house measurements NPL use a minimised dead volume (MDV) connector, in place of a DIN 1 connector, and a low volume restriction device to control sample flow. The MDV connector screws directly into the head of the valve, reducing the surface area in contact with the sample and significantly lowering the dead volume. This device reduces the necessary purge time, particularly in the measurement of high molecular weight VOCs.

b) Gas Chromatography

A Gas Chromatograph is the recommended instrument for the analysis of VOCs with the injection and detection mechanisms dependent on the class of VOCs and the concentration range. The multi-component VOC standards are used for the calibration of Gas Chromatographs used in the measurement of a wide range of VOCs.

-30 Component Ozone Precursor VOC standard

A PLOT Al_2O_3/KCl column is recommended for the separation of light compounds and resolution of highly unsaturated compounds, such as acetylene and isoprene. If a multi-dimensional configuration is available then the light compounds may be separated on the PLOT column and the heavier compounds (> C6) may be separated on a faster polysiloxane column (e.g. CP-Sil 5 CB, DB-1 etc).

-Terpene standards

A CP-Sil 13 CB column is recommended for the separation of terpenes. If the terpenes are combined with other VOCs, then multiple analyses on different columns, such as a PLOT Al_2O_3/KCl column, will be necessary to separate all the components.

c) Injector

The injector should be either a gas sampling valve (GSV) for high concentration (> 100 ppb) or a preconcentration device (eg Sample Preconcentration Trap, SPT (Bruker)) for low concentration (100 ppb). The recommended adsorbent for preconcentration is glass beads, where liquid nitrogen is available. The temperature set point of the SPT during sampling should be determined by the boiling point of the VOCs. If liquid nitrogen is available a multi-bed adsorbent may be used for trapping. Otherwise other absorbents (Tenax, Carbograph) can be used to preconcentrate the sample, with other cooling systems, such as Peltier elements. The strong sorbent must be positioned at the back end (or outlet) of the bed otherwise the heavier compounds will not be released upon thermal desorption.

d) Detector

The recommended choice of detector for the analysis of the majority of VOCs is the Flame Ionisation Detector (FID). The FID is linear over a large dynamic range and can detect virtually all organic compounds.

4. Uncertainties

The uncertainty of NPL VOC gas standards is stated on the certificate as an expanded uncertainty with a coverage factor of approximately 95% in the units of the gravimetric amount fraction. This uncertainty is a combination of the gravimetric uncertainty in the preparation of the mixtures and an allowance for drift over the two-year period the certificate is valid.

5. Stability

NPL carries out continuous stability checks on a suite of trace VOC standards. This data is published periodically and covers a range of VOC classes. Please see our website for further details.

For further information please contact (gases@npl.co.uk)

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