

# Protocol for the Gas Cylinder Component of the NPL Gas Measurement and Gas Cylinder Proficiency Testing Scheme, Round 24 (Version 1)

## 1. INTRODUCTION

1.1 This document describes the procedure which should be adopted by participants in the **Gas Cylinder component** of the NPL Gas Measurement and Gas Cylinder PT Scheme. In order to ensure comparability of the results, it is important that, as far as possible, the same protocol is adopted by all participants. It is also important, and in the true spirit of a PT scheme, that the measurements of the test gases are carried out in a manner which is as close as possible to that used under operational conditions. This increases the benefit that participants will gain from the scheme and will make the scheme more appropriate for use with quality control systems.

## 2. OUTLINE OF THE SCHEME

2.1 The scheme consists of seven test gases which participants come to the NPL site to measure. The scheme will include the following test gases:

Gas		Standard deviation for proficiency testing (%)	Maximum concentrations
SO <sub>2</sub> balanced in N <sub>2</sub>		5	80 ppm
C <sub>3</sub> H <sub>8</sub> balanced in 10% O <sub>2</sub> + 90% N <sub>2</sub>		7.5	10 ppm
NO balanced in N <sub>2</sub>		5.0	150 ppm
CO balanced in N <sub>2</sub>		3.0	80 ppm
O <sub>2</sub> balanced in N <sub>2</sub>		2.0	21 %
CO <sub>2</sub> balanced in N <sub>2</sub>		2.0	20 %
NO/NO <sub>2</sub> balanced N <sub>2</sub>	NO <sub>x</sub>	5.0	150 ppm
	NO	5.0	140 ppm

*Note: For the NO/NO<sub>2</sub> cylinder both NO<sub>x</sub> and NO values are requested*

2.2 The cylinders will be analysed by NPL prior to each round, in order to certify the concentrations against NPL primary UK standards. These certified concentrations will be known only to NPL. The certified concentrations are the assigned value against which participant results will be compared, using z-scores derived from the standard deviation for proficiency testing given in the table above.

### 3. PROTOCOL

- 3.1 Participants should refer to the samples analysed by the **cylinder number** which will be clearly identified on the cylinders. NPL will coordinate when participants visit the NPL site. If the participant is unable to visit the NPL site at the allotted time they will be deemed to have failed to supply results for that round of the scheme.
- 3.2 Teams will be allocated a day in which to undertake testing. Both components of the Gas Measurement and Gas Cylinder PT Scheme will take place at the same time with measurements of the cylinders taking place in the gaps between test runs on the simulator. As up to three teams will be working at any one time it is requested that each team be limited to a maximum of 2 people. To enable teams to complete the testing in the allotted time you will be asked to observe the following timetable:

Time	Activity
08:00 – 10:00	Instrument calibration + CG
10:00 – 10:30	Test 1
10:30 – 11:45	Instrument calibration + CG
11:45 – 12:15	Test 2
12:15 – 13:30	Instrument calibration + CG
13:30 – 14:00	Test 3 (60 minutes if HCl is being measured)
14:00 – 15:00	Instrument calibration + CG
15:00 – 15:30	Pack up and depart

**This timetable will be strictly enforced. If a team is not ready for testing then they will have to forfeit that test to ensure that they are ready in time for the next test.**

On days where HCl is being measured the third test will be extended to last 60 minutes (13:30-14:30) rather than 30 minutes for all other tests. Subsequent items in the timetable above will be shifted back to allow for this with departure by 16:00.

Participants will be able to arrive on the day before testing after 16:00 to set up their equipment to ensure it is fully warmed up before testing. Teams will have to be off site by 18:00.

- 3.3 NPL will not provide tubing, Swagelok, extension reels and other connectors. NPL will provide one 110V socket, two 220V sockets and a bench top (120cm x 70cm) per team.
- 3.4 The cylinders held at NPL will have regulators attached and should not be removed. The regulators will have ¼ inch Swagelok fittings.
- 3.3 The participants should analyse each gas using **up to two** of their operational analysers. **The gases should be introduced direct to the analyser via the ‘sample in’.** **The sample should not be introduced through any available calibration port or via any sample probe/system.** The use of a bypass is requested to avoid over pressurising the sampling system and analyser. A rotameter must be used on the bypass. This should be monitored to ensure there is a positive bypass flow during the test. The bypass flow should be approximately 0.5-1 l/min.
- 3.4 The measurement should be performed in a similar manner to the participants' relevant field measurement calibration protocol in terms of the number and time period of samples and the sample flow. However, in order to preserve gas the sample flow rate should be

kept to below 2 l/min and a total sample period of less than half an hour per gas should be used. Participants may assume that the sample is free from particulate/aerosol and that the gas is dry. Any calibrations should be carried out according to the participants' usual practice, and at a level suitable for the nominal concentration of the test samples.

- 3.5 Participants are requested to make **one measurement per analyser** of each of the PT scheme gas cylinders. The sample gas should be flowed until a stable reading is achieved. Please record and average the analyser output for the last 4 minutes of stable readings.
- 3.6 Participants should treat results as they would a field sample. However, it will not be necessary to produce a full report of the measurements. The attached standardised report shall be used to return results.
- 3.7 Participants are requested to follow their normal field calibration procedure for calibrating the analysers. It is expected that this will include a post-test calibration check for analyser drift. If this check is carried out and if the analyser has been seen to drift by an amount that would lead to manipulation of the data to compensate for the observed drift, participants are asked to treat the PT scheme samples in the same way; i.e. to compensate for drift as appropriate.
- 3.8 Participants should record the pressure in the sample cylinder (read from the regulator gauge) before and after using each test gas.

## 4 COLLUSION

- 4.1 Participants are requested not to discuss results during the proficiency tests.
- 4.2 A member of the proficiency test team will make spot readings from participants' instruments during each proficiency test.

## 5 REPORTING RESULTS

- 5.1 **Results should be supplied direct to NPL on the day of the proficiency test.** Participants are requested to fill in the attached pro-forma and return it to the PT Scheme Co-ordinator or email to [results@stack-pt-schemes.net](mailto:results@stack-pt-schemes.net) and [gabrielle.stevenson@npl.co.uk](mailto:gabrielle.stevenson@npl.co.uk).
- 5.2 NPL will report the results anonymously. In addition each participant will be informed of their own performance.

## 6 Appeals or Complaints

- 6.1 Appeals or complaints should be directed to the PT Scheme Coordinator through the following e-mail address:

[info@stack-pt-schemes.net](mailto:info@stack-pt-schemes.net)

## Calibration Gas PT Scheme (Round 24) Reporting Sheet

<b>Organisation:</b>	
<b>Operator Name:</b>	
<b>Signature :</b>	
<b>Date :</b>	
<b>Instrument Identifier :</b>	

Species	Cylinder number	Measured results (ppm/%)	Uncertainty (ppm/%)	Pressure in cylinder (bar)	
				Before	After
SO <sub>2</sub>					
C <sub>3</sub> H <sub>8</sub>					
NO					
CO					
O <sub>2</sub>					
CO <sub>2</sub>					
NO / NO <sub>2</sub>	NO <sub>x</sub>				
	NO				

Participants are requested to make **one measurement per analyser** of each of the PT scheme gas cylinders

**Notes:**

**Results should be supplied direct to NPL on the day of the proficiency test.**

Please return this form to the PT Scheme Co-ordinator

or email to [results@stack-pt-schemes.net](mailto:results@stack-pt-schemes.net) and [gabrielle.stevenson@npl.co.uk](mailto:gabrielle.stevenson@npl.co.uk).