



EffecTech

Global Leaders in Gas Measurement



4719

Stack Emissions Proficiency Testing (PT) Scheme

Instructions for Participants

EffecTech is accredited by the United Kingdom Accreditation Service (UKAS) to provide this Proficiency Testing Scheme in accordance with the requirements of ISO/IEC 17043:2010

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1. Introduction

EffecTech provides and organises the Stack Emission Proficiency Testing Scheme (SEPTS).

Proficiency testing (PT) schemes provide an objective way of assessing the performance of laboratories by a series of regular inter-laboratory comparisons. This PT scheme is aimed at laboratories and testing organisations working in the field of stack emissions.

The results, reports and certificates issued during this scheme can be used as objective evidence of competence during audits and inspections from interested third parties or accreditation bodies.

In this PT scheme one round is scheduled per year. PT scheme items are binary or tertiary mixtures provided in cylinders each containing one of the target measurands of sulphur dioxide, nitric oxide, nitrogen dioxide, carbon monoxide, carbon dioxide, oxygen or propane.

The mixtures are checked for stability and reference values are assigned prior to shipment. After each round, the mixtures will remain at the participant's laboratories for use in either calibration or quality control.

The gas mixture preparation and assignment of reference values are carried out by EffecTech.

2. References

The following list of references contains information which, through reference in the main body of this document, are necessary to understand fully the provisions of these instruction. At the time of writing, and unless otherwise stated in the main body of this document, these instructions are consistent with the principles contained within all those listed below.

All documents in this list, particularly at the present time, are subject to revision and the provider of this scheme makes every effort to ensure that provisions in more recent revisions than those listed below are adopted.

- [1] ISO/IEC 17043 *Conformity assessment - General requirements for proficiency testing*
- [2] ISO Guide 35 *Reference materials – Guidance for characterization and assessment of homogeneity and stability*
- [3] ISO Guide 98-3 *Uncertainty of measurement - Part 3: Guide to the expression of uncertainty in measurement (GUM : 1995)*
- [4] ISO 6142-1 *Gas analysis - Preparation of calibration gas mixtures - Part 1: Gravimetric method for Class I mixtures*
- [5] ISO 6145 *Gas analysis - Preparation of calibration gas mixtures using dynamic volumetric methods - Part 7 Thermal Mass Flow Controllers*
- [6] ISO/IEC 17025 *General requirements for the competence of testing and calibration laboratories*
- [7] ISO 12963 *Gas analysis - Comparison methods for the determination of the composition of gas mixtures based on one- and two-point calibrations*
- [8] ISO 16664 *Gas analysis - Handling of calibration gases and gas mixtures - Guidelines*
- [9] ISO 13528 *Statistical methods for use in proficiency testing by interlaboratory comparisons*

3. Points of contact

The PT scheme is provided by EffecTech.

EffecTech has appointed key personnel and management representatives responsible for providing the scheme. Contact details of the scheme coordinator, the logistics manager and technical authority are given below

Scheme coordinator

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4. Accreditation status

Effectech provides this proficiency testing scheme in accordance with the requirements of ISO/IEC 17043 [1] (formerly ISO Guide 43) for which it is accredited by the United Kingdom Accreditation Service (UKAS). Please see the UKAS website for our official scope of accreditation ([UKAS proficiency testing provider 4719](#)).

Effectech holds an accreditation awarded by UKAS to ISO/IEC 17025 [6] as a calibration laboratory. Please see the UKAS website for our official scope of accreditation ([UKAS calibration laboratory 0590](#)). Effectech uses these methods for the calibration of gas mixtures issued in this PT scheme.

5. Mixtures

5.1 Introduction

Each measurand in this PT scheme is issued as a binary or tertiary mixture (see Table 1).

Table 1: Gas mixture types

description	cylinder volume (dm ³)	cylinder outlet	nominal pressure (bar)
carbon monoxide in nitrogen	5	BS341-No.4	50
carbon dioxide in nitrogen	5	BS341-No.3	50
oxygen in nitrogen	5	BS341-No.3	50
nitric oxide in nitrogen	5	BS341-No.15	50
nitric oxide and nitrogen dioxide in nitrogen	5	BS341-No.15	50
sulphur dioxide in nitrogen	5	BS341-No.15	50
propane in 10% oxygen / nitrogen	5	BS341-No.4	50

5.2 Composition

Table 2: Composition range

component	range (mol/mol)
carbon monoxide in nitrogen	50 ppm to 1000 ppm
carbon dioxide in nitrogen	1% to 10%
oxygen in nitrogen	2% to 14%
nitric oxide in nitrogen	5 ppm to 500 ppm
nitric oxide (NO) in NO/NO ₂ mix	40 ppm to 400 ppm
nitrogen oxides (NO _x) in NO/NO ₂ mix	50 ppm to 500 ppm
sulphur dioxide in nitrogen	50 ppm to 1000 ppm
propane in 10% oxygen / nitrogen	1 ppm to 50 ppm

6. Mixture preparation and reference value assignment

6.1 Preparation of mixture batches

One or more parent mixtures are pre-prepared by a gravimetric method in accordance with ISO 6142-1 [4]. A batch of mixtures of each type is then produced by decanting the parent mixture into a batch of lower volume pre-prepared and evacuated daughter cylinders.

The parent mixtures and daughter mixture are then calibrated.

6.2 Mixture calibration

All parent mixtures are calibrated using a two-point calibration design with bracketing (TPC), with the exception of propane which is calibrated using a single-point through origin calibration (SPO). Both of these methods are in accordance with ISO 12963 [7] for which EffecTech is accredited to ISO 17025 [6] by UKAS.

All decant mixtures were calibrated by a single point exact matching technique (SPEM) also in accordance with ISO 12963 [7] by the comparison of the decant mixture with its nominally identical parent mixtures.

The uncertainty on amount fraction of each measurand in the mixtures resulting from this calibration is termed the characterisation uncertainty, u_{char} (ISO Guide 35 [2]).

All calibrations are performed in accordance with EffecTech's ISO 17025 [6] accredited calibration methods.

6.3 Reference mixture traceability

An analytical comparison method is used for the calibration of all mixtures in this scheme. Parent mixtures are calibrated by comparison with reference gases generated dynamically in accordance with ISO 6145-7 [5]. The reference mixtures used for blending are traceable either directly, or by internal verification through an in-house primary standard, to a National Metrology Institute (NMI). This process ensures that the values assigned to the mixtures in this scheme are metrologically traceable to international standards, through an unbroken chain of comparisons, and ultimately to the amount of substance (mole) defined in the SI (International System of Units).

6.4 Homogeneity assessment

Statistical analysis of the spread of reference values obtained for each batch of mixtures (derived through calibration in 6.2 above) is used to assess the homogeneity between the set of decant mixtures to be distributed to each participant. The dispersion of the amount fraction of each component due to batch inhomogeneity is known as the between-bottle standard deviation. The uncertainty arising from this is u_{hom} (ISO Guide 35 [2]). This uncertainty should be less than or equal to the characterisation uncertainty in order to accept the batch.

6.5 Stability of the mixtures

Over several years EffecTech has built up a history of intercomparisons of mixture types featured in this PT scheme. Data from these intercomparisons show clearly that all mixtures remain stable within their stated uncertainty for a minimum of 12 months.

The stability of each mixture is guaranteed for a period of 12 months. Within this time period there is no additional uncertainty ascribed to the reference values resulting from the stability of the mixtures, u_{ITS} . This is valid providing the mixtures are not used beyond this stability period.

Most of the mixtures will be stable (within their stated uncertainty) for considerably longer but this period has not been determined.

In conjunction with your existing gas mixtures used for calibration or quality control, these mixtures can be used likewise after the submission of results for each round in this scheme. Cylinders that are no longer required may be returned directly to EffecTech if the costs are not prohibitively expensive.

6.6 Reference value assignment

For each mixture the measurand is assigned a reference value, x_{ref} , calculated from the average of those determined in the calibration stage (see section 6.2 above).

A combined uncertainty, u_c , is first calculated from the equation below. This uncertainty is dominated in all cases by the calibration uncertainty, u_{char}

$$u_c = u_{\text{char}} + u_{\text{hom}}$$

Following this calculation, the expanded uncertainty, $k \cdot u_c$, ($k=2$), is compared to the Calibration and Measurement Capability (CMC) for which EffecTech is accredited to ISO 17025 (See [UKAS calibration laboratory 0590](#)). If $U_{\text{CMC}} (k=2)$ is greater than $k \cdot u_c (k=2)$ then the uncertainty on the reference value is assigned to that stated in our published CMC in accordance with accepted practice such that

$$U_{\text{ref}} = \max (U_{\text{CMC}} , 2u_c)$$

The use of a coverage factor of $k=2$ in the assignment of U_{ref} provides a level of confidence of approximately 95%.

6.7 Approximate reference values

Approximate reference values will be given on the shipped mixtures to enable participants to calibrate their instrument appropriately prior to measurement of the mixture.

7. Schedule

7.1 Shipping

The scheme shall operate once a year and mixtures shipped to participants during May.

EffecTech sub-contracts all shipping and freight forwarding to specialist transport providers whose performance has been evaluated by regular and on-going monitoring of their ability to deliver to time and quality.

Participants will be responsible for taking receipt of each consignment and collecting the reference mixtures once they have been notified by EffecTech (or the freight forwarder / courier) that they are due for delivery on site. If the consignment is not received at the destination within **one week** of the due date of this notification then they should contact EffecTech.

On receipt of the Proficiency Testing item(s) participants will be responsible for completing and returning the "Sample Receipt Form". This is securely attached to the neck of one of the sample cylinders supplied. The contact details, timescale and methods for return of the completed form are contained on the form itself.

Airfreight delivery to airport

The cargo handling agent at the destination airport will notify the consignee (participant) of the need to clear the goods for customs. Proofs of Delivery (PODs) are requested from the destination cargo handling agent via EffecTech's freight forwarder. Once beyond the Incoterms™ defined delivery point, EffecTech will periodically request shipment status updates from the participant and their freight forwarder, if known to us, regarding the current status of the shipment at the time of request. Participants should respond to these requests with the latest available information and should, in any event, inform EffecTech of any issues or delays in transportation or customs clearance as soon as they are known.

Airfreight delivery to door

The cargo handling agent at the destination airport will notify the destination freight agent/forwarder (known as the *Notify Party*) who will in turn notify the consignee (participant). PODs are requested from the *Notify Party* by EffecTech's freight forwarder. However, this may be done by EffecTech directly if the *Notify Party* is known to us.

UK and European road transport

The UK freight forwarder will pass information through the carrier network from carrier to carrier until delivery is made. A Proof of Delivery (POD) will then pass back through the network of carriers to the originating freight forwarder who will then pass back the POD to EffecTech. In the case of UK road transport, EffecTech takes the place of the freight forwarder.

Ex-Works (EXW) & Free Carrier (FCA EffecTech) shipments

For shipments where the participant has elected to arrange their own transportation, EffecTech will notify the participant and their freight forwarder, if supplied, by means of a notification stating the date the samples will be available for collection. This notification will contain an electronic version of all shipping documents generated by EffecTech as well as the collection address, hours of opening and contact details for the collection.

Given the timescales involved with this scheme, the availability date given in this notification will start a maximum 5 week (35 day) period for the collection, transit and delivery of the sample(s) to the participant to be completed.

During transportation EffectTech will periodically request information from the participant and their freight forwarder, if known to us, regarding the current status of the shipment at the time of request. Participants should respond to these requests with the latest available information and should, in any event, inform EffectTech of any issues or delays in transportation or customs clearance as soon as they are known.

The participant must notify EffectTech on receipt of the sample(s). This will prompt the reporting form and reporting deadline to be issued. However, should 35 days from the stated sample collection availability date pass without the participant confirming receipt of the sample(s), the reporting form and reporting deadline will be issued automatically to the participant on the 36th day following the sample(s) being made available for collection.

All logistics and delivery queries should be directed to

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EffectTech undertakes every reasonable effort to prevent delays in the dispatch of the gas mixtures.

7.2 Reporting

Laboratories are requested to report their results well before the deadline. Participants will be informed of the deadline for reporting which shall usually be **2 weeks** after receipt of the cylinders. Results received after the deadline will not be processed or included in the report. In case of foreseeable delays, participants are requested to report such delays to the [scheme coordinator](#) with an expected date of submission of their results. The new date requested cannot be regarded as a new deadline unless it is confirmed by the scheme coordinator.

Detailed instructions for reporting your results can be found below in section 8.6 & 8.7.

8. Instructions for measurement

8.1 Sample treatment

Laboratories shall introduce the reference gas mixtures provided directly through the “sample in” port of each analyser. The aim of this PT scheme is to assess the accuracy and traceability of your calibrated analysers in isolation; it is not the intention to assess any additional elements such as heated filters, heated sample probes, heated transport lines or chillers. Hence, the sample should not be introduced through any sample treatment / conditioning systems routinely used when sampling real stack gas samples at site.

In introducing the gas mixture from the cylinder to the analyser directly, appropriate consideration should be given to the advice below (in sections 8.2, 8.3 & 8.4) concerning storage and use of the gas mixture.

8.2 Cylinder storage

Before and after measurement, the ideal way to store the reference gas mixtures for longer periods is by laying the cylinders in a horizontal position well protected against rolling and falling. For safety reasons it is necessary to separate cylinders containing flammable gases from cylinders containing oxidising components.

For the mixture types provided in this PT scheme there is no minimum or maximum recommended temperature of storage. Ambient temperatures in the range 0°C to 30°C will have no deleterious effect on the stability of the gas mixture.

8.3 Gas handling during use

Normally the withdrawal of the reference gas from a cylinder is regulated by a pressure regulator and / or a flow controller (needle valve, mass flow controller or capillary device). Due to reversible adiabatic expansion of the gas on withdrawal from the cylinder, cooling (Joule-Thomson cooling) of the sampled gas mixture will occur. Joule-Thomson cooling occurs particularly with mixtures containing condensable components and can result in retrograde condensation of the sampled gas. It is important that the pressure drop through pressure regulators and across flow controllers is minimised for mixtures where this is an issue and that withdrawal is done at an appropriate ambient temperature.

However, for mixture types provided in this PT scheme there is no minimum recommended usage temperature as the measurands at the amount fractions provided do not suffer from retrograde condensation in the ambient temperature range 0°C to 30°C.

8.4 Sample integrity and handling

It is important to use the most appropriate pressure and flow reduction equipment to deliver the required flow rate of gas at the required pressure to each analyser. The equipment needs to regulate to the required pressure and flow and be constructed of the most appropriate material for the gas being sampled.

In many cases the integrity of high quality and expensive calibration gases can be compromised through the use of poor quality or inappropriate pressure and flow reduction equipment. This should be avoided.

Adequate leak tightness of the sampling system should be established when sampling your gas mixtures. The mixture should be purged through the pressure reduction and sample conditioning system for an appropriate time such that the sample being measured onto your instrument is truly representative of the contents of the cylinder.

If in doubt, participants should follow the advice given in the International Standard ISO 16664 [8].

8.5 Measurement methods

Laboratories shall, wherever possible, use their default calibration and measurement methods for analysing the PT scheme mixtures. The test method and number of replicates used for measurement shall be the same as for routinely analysed samples.

8.6 Submission of results

Each laboratory is required to submit their results by completing the spreadsheet provided with these instructions. Data reported in an alternative format will not be processed. Please do not make any modifications to the format of the spreadsheet provided as this will prohibit automatic processing. If any participant wishes to comment on this spreadsheet or wishes a revision or update thereof then please send an email request to the [scheme coordinator](#).

All results should be submitted in the approved / supplied spreadsheet before the deadline date and by email to

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Each laboratory is requested to submit

- the result (with up to ten repeat measurements)
- the expanded uncertainty associated with the result using a coverage factor $k=2$ (see section 8.7)
- the method and type of calibration gas used for your measurement (included in the *Comments* section of the form)

Following receipt of each participant's results by EffecTech they will be acknowledged by the scheme coordinator. The submitted results will then be inspected by a designated authority. Any typographical errors, miscalculations or other gross errors in reporting shall not be referred back to

the participant for review. If such errors in reported results are observed then EffectTech will make an explicit note of this in the report of the round.

All reported data, comments and other correspondence will be kept strictly confidential by the scheme provider. In the scheme reports (see section 10.1) results and selected comments will be reproduced using the laboratory's designated code only.

8.7 Reporting of measurement uncertainty

Wherever possible each laboratory should report an estimate of the measurement uncertainty associated with each reported measurement result.

Document JCGM 100:2008 GUM 1995 with minor corrections "Evaluation of measurement data- Guide to the expression of uncertainty in measurement" describes the preferred method of uncertainty estimation. [JCGM 100:2008](#) can be downloaded from the website of the Bureau International des Poids et Mesures www.bipm.org

A more chemistry related document on the estimation of uncertainty is published by Eurachem and CITAC. This guide CG4 "Quantifying Uncertainty in Analytical Measurements" can be downloaded from www.eurachem.org.

These documents can also be obtained from the [scheme coordinator](#) on request.

8.8 Collusion with other participants

In the interests of fairness and accuracy, participants are instructed not to collude or share data with other participants on the results which they have obtained from measurements of any gas mixture.

If you are aware of others that participate in the scheme and wish to share measurement results information with them then, subject to permission from local management, do not do so until the final report has been published by EffectTech.

9. Evaluation of performance

9.1 Rating per component and per mixture type

z-score

The evaluation of performance was carried out by means of a z-score, which gives the relative deviation of the participant's result from the reference value.

The z-score is calculated with the following general formula

$$z = \frac{x_{meas} - x_{ref}}{\sigma} \quad (1)$$

where x_{meas} is the measured result reported by the laboratory

x_{ref} is the assigned reference value and

σ is the absolute standard deviation used for performance assessment which can be calculated from the relative standard deviation for performance assessment S_{PT} by

$$\sigma = \frac{S_{PT}}{100} \cdot x_{ref} \quad (2)$$

The relative standard deviation for performance assessment used for calculating the z-scores has been fixed for all components by EffectTech and based upon a reasonable expectation of the performance capabilities that should be demonstrated by each laboratory. These values have been chosen with reference to relevant CEN standards, the Waste Incineration Directive (WID) and the Large Combustion Plant Directive (LCPD).

In addition, as the measurement of a gas mixture from a cylinder does not assess the additional uncertainty that would be introduced by sampling from a real stack / chimney / exhaust then the expectations have been revised downwards so that the process of calibration and sampling from a cylinder only can be assessed in isolation.

These are given in the tables below

Table 3: Standard deviation for performance assessment

component	S_{PT} (mol/mol)
carbon monoxide in nitrogen	1.5 % relative
carbon dioxide in nitrogen	1.0 % relative + 0.01 %mol/mol
oxygen in nitrogen	1.0 % relative + 0.01 %mol/mol
nitric oxide in nitrogen	2.5 % relative
nitric oxide (NO) in NO/NO ₂ mix	2.5 % relative
nitrogen oxides (NO _x) in NO/NO ₂ mix	2.5 % relative
sulphur dioxide in nitrogen	2.5 % relative
propane in 10% oxygen / nitrogen	5.0 % relative

The qualification of the z-scores is given in Table 4 below

Table 4: Relationship between z-score and quality of result

z-score	quality of result
$ z \leq 2$	satisfactory result
$2 < z < 3$	questionable result
$ z \geq 3$	unsatisfactory result

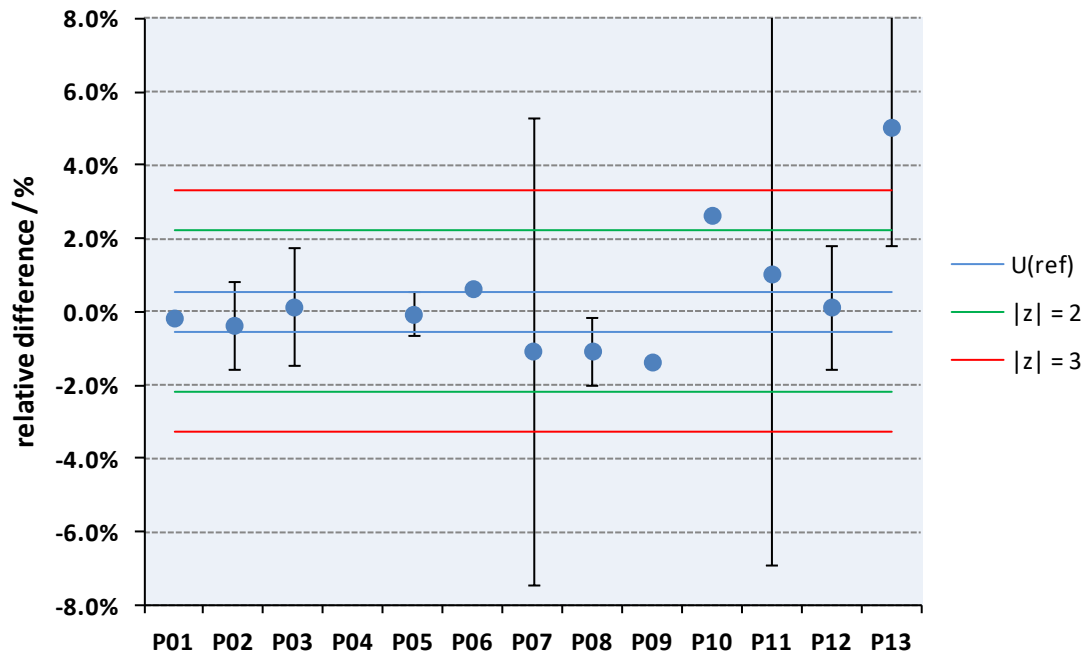


Figure 1: z-scores

From the example shown in Figure 1, it is clear that laboratories P01, P02, P03, P05, P06, P07, P08, P09, P11 and P12 report a satisfactory result, laboratory P10 a questionable result and laboratories P13 and unsatisfactory result.

E_n number

In addition, an E_n number is calculated which assesses the difference in the reference and measured (reported) values relative to their respective uncertainties. The calculation of E_n numbers is dependent upon the laboratory reporting estimates of uncertainties associated with their measurement results. Laboratories are encouraged to report such estimates in accordance with accepted practices (see section 8.7).

The E_n number is calculated with the following general formula

$$E_n = \frac{x_{meas} - x_{ref}}{\sqrt{U_{meas}^2 + U_{ref}^2}} \quad (3)$$

where x_{meas} is the measured result reported by the laboratory

x_{ref} the assigned reference value and

U_{meas} and U_{ref} their respective uncertainties (using a coverage factor $k=2$)

The qualification of the E_n number is given in Table 5 below

Table 5: Relationship between E_n -number and quality of result

z-score	quality of result
$ E_n \leq 1$	satisfactory result
$ E_n > 1$	unsatisfactory result

Reference	x_{ref}	$U(x_{ref}) k=2$		S_{PT}	
	1.000	0.005	%mol/mol	0.011	%mol/mol

Reported data

participant id	value (%mol/mol)	U (k=2) (%mol/mol)	relative difference	z-score	E_n -number
P01	0.998		-0.16%	-0.18	
P02	0.996	0.012	-0.36%	-0.36	-0.31
P03	1.001	0.016	0.14%	0.09	0.06
P04					
P05	0.999	0.006	-0.06%	-0.09	-0.13
P06	1.006		0.64%	0.55	
P07	0.989	0.063	-1.06%	-1.00	-0.17
P08	0.989	0.009	-1.06%	-1.00	-1.07
P09	0.986		-1.36%	-1.27	
P10	1.026		2.64%	2.36	
P11	1.010	0.080	1.04%	0.91	0.12
P12	1.001	0.017	0.14%	0.09	0.06
P13	1.050	0.034	5.04%	4.55	1.45

Figure 2: E_n numbers

From the same dataset as used for Figure 1, the E_n numbers are shown in the far right hand column of Figure 2. The E_n numbers of laboratories P08 and P13 are unsatisfactory. However, the

performance of all other laboratories is satisfactory by this measure (for those reporting uncertainties).

10. Reports

10.1 Confidentiality

In all reports bound for general distribution, the participants are identified by means of a laboratory code (P01, P02,...,Pxx) which is assigned by EffecTech. Hence, the details of all participating laboratories, their results and their performance assessment are anonymous.

The laboratory code assigned to each participant will be kept strictly confidential and is known only to the [scheme coordinator](#) and [technical authority](#) within EffecTech.

In addition, on the day of publication of the report (see section 10.2 below), the registered contact at each laboratory shall also be informed of their laboratory code.

Only the Certificate of Participation (see section 10.3 below) includes details of the laboratory name, address, results and performance in the current round of the scheme. This is **ONLY** submitted to the registered contact at the participant laboratory.

10.2 Report

Following each round of the scheme a report will be issued containing at least the following information:

- calibration and reference value assignment by EffecTech
- individual results and expanded uncertainties reported by each participant
- ratings of each laboratory's performance by means of **z**-scores and E_n numbers
- graphical displays showing participants' results against reference values and the range of a satisfactory result
- general observations shall be made based upon the participants' results including advice on the interpretation and comments or recommendations based on the outcome of the round.

10.3 Use of Reports

EffecTech do not impose any restrictions on the use of reports by individuals or organisations but reports, when disseminated further, must only be reproduced in full.

10.4 Certificate of Participation

Following each round an individual Certificates of Participation shall be issued to the registered contact at the participant laboratory. This can be used, for example, as a proof of competence during inspections and audits by interested third parties or accreditation bodies.

The Certificate of Participation shall include the name of the scheme and round number, the participant's name and address and a table of each gas mixture type each containing

- reference values and their uncertainties assigned by EffecTech
- results reported by the individual laboratory for which the certificate is created

- **z**-scores and E_n -numbers for the laboratory
- statement of authenticity issued, dated and signed by the scheme coordinator and technical authority

11. Appeals

Following issue of a report or certificate, participants may appeal their results or scores should they consider the assessment presented is unfair, incorrect or misleading.

All appeals should be reported directly to the [scheme coordinator](#) in the first instance who will acknowledge and then consider the appeal. He may, if necessary, refer the appeal to the technical authority for a final decision to be agreed.

Following a successful appeal the laboratory shall be informed of the outcome in the first instance. A new revision of the quarterly report shall be issued to all participants. In addition, dependent upon the consequences of the appeal, it may be necessary to issue one or more Certificates of Participation.