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## EXECUTIVE REPORT

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### 1 Introduction

Plastics Europe have engaged Eurofins Product Testing A/S with the intention to perform an extensive analysis program for the VOC emitted from expanded polystyrene (EPS) products representing roughly 80 per cent of the market according to the client.

The testing was divided into two parts with the first part being analysed approx. April 2015 and comprised of 9 samples of varying density with respect to VOC emissions and the content of free styrene in the samples. The second part was analysed in the approx. February 2016 on was performed on samples with similar density and was analysed for VOC emissions and free formaldehyde.

The emission testing was performed fully according to ISO 16000 -6 -9 -11 and in full accordance with CEN/TS 16516 and FprEN 16516. The testing was performed as a screening of both VOC and carcinogens. The performing laboratory is ISO 17025 accredited and the methods employed for emission testing were accredited test methods.

This report will summarize the results found in the testing (ref. 392-2015-00141500, 392-2015-00086301)



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## 2 Sample Information

The samples were received wrapped in aluminium foil and plastic to ensure a tight seal to avoid premature emissions and contamination during transport. All samples were received un-damaged and were approved for laboratory analysis. The samples were not unwrapped until the day of emission test start to preserve the samples.

## 3 Applied Test Methods

The chamber testing was performed in an ISO 16000-9 chamber and the samples were prepared with a loading ratio of  $0.66 \text{ m}^2/\text{m}^3$ . The loading ratio is in between the loading scenario for floor ( $0.4 \text{ m}^2/\text{m}^3$ ) and the loading scenario for wall ( $1 \text{ m}^2/\text{m}^3$ ). The results from the analysis can therefore be seen as the expected concentration in the reference room the given number of days after the installation of the products, if the products cover a surface area between floor and wall. The edges and back of the samples were covered before testing in order to only expose one surface during testing. The tested sample thickness was 5 cm.

The air change rate was 0.5/h equivalent to all the air in the chamber being changed every 2 hours. The temperature was 23 degrees Celsius and the relative humidity was 50%. This is the standard conditions adopted in ISO 16000 to simulate a standard real-world environment as close as possible.

Samples were taken after 3 and 28 days on tenax TA (ISO 16000-6) and DNPH tubes (ISO 16000-3). The result after the given number days can be seen as the expected concentration in the reference room from the tested product. The DNPH tubes were analysed using HPLC and the tenax tubes were analysed using ATD-GC/MS. Isomers of pentane were investigated using charcoal tubes with solvent desorption and analysis on HS-GC/MS. The analytical method is further discussed in the appendices.

The free content of styrene and isomers of pentane was also investigated. Note that this is the content in the samples and not the emissions measured from the samples. The measurement was performed by dissolving the EPS in dimethylformamide and subsequently performing the analysis using GC/MS. Content determination was performed as triple determinations to ensure representative sampling from the EPS.

For the testing of density in the first batch, there was performed density measurement before start of the emission test and after ended emission test. For content testing of free styrene and/or isomers of pentanes, there was also performed analysis before and after the chamber testing.

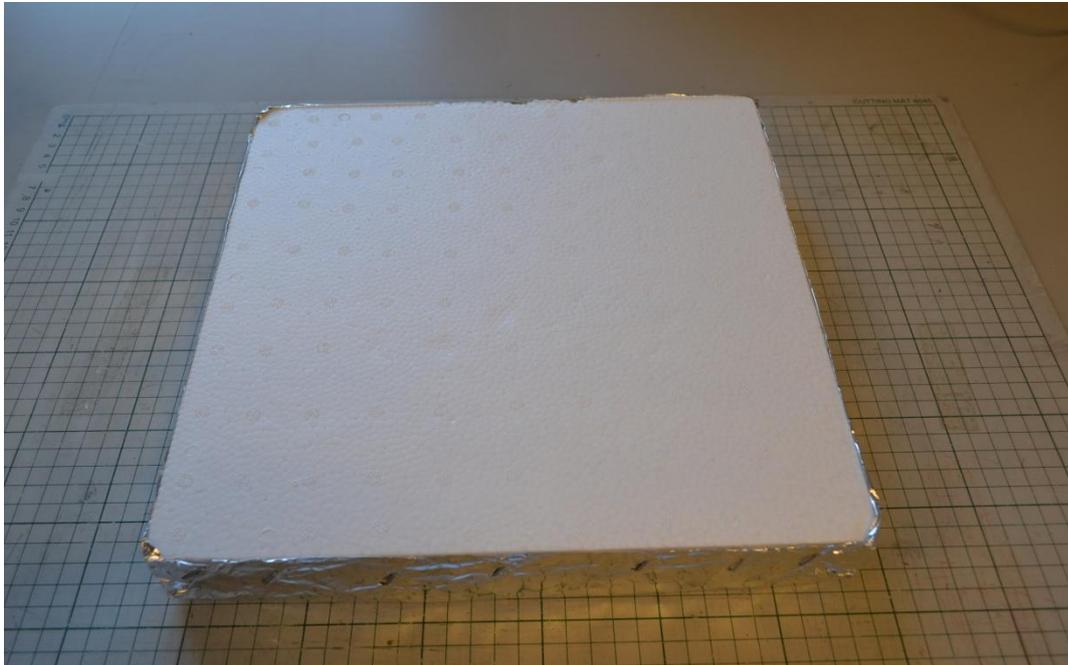
### 3.1 Specific Laboratory Sampling and Analyses

Procedure	External Method	Internal S.O.P.	Quantification limit	Analytical principle	Uncertainty <sup>2</sup> [RSD(%)]
VOC sample preparation	ISO 16000-11:2006, EN16402:2013, CDPH, AgBB/DIBt, EMICODE	71M549810	-	-	-
VOC emission chamber testing	ISO 16000-9:2006, CEN/TS 16516:2013	71M549811	-	Chamber and air control	-
Sampling of VOC	ISO 16000-6:2011, CEN/TS 16516:2013	71M549812	5 L	Tenax TA	-
Analysis of VOC	ISO 16000-6:2011, CEN/TS 16516:2013	71M542808B	5 µg/m <sup>3</sup>	ATD-GC/MS	10%
Sampling of isomers of pentanes in air	ISO 16000-6:2011	71M549812	-	Charcoal tube	-
Analysis of isomers of pentanes	Internal method	71M546209	20 µg/m <sup>3</sup>	HS-GC/MS	10%
Content analysis of free styrene content	Internal method	71M546209	0.2 mg/kg	HS-GC/MS	10%
Determination of density	Internal method	-	-	Gravimetric	5%

### 3.2 VOC Emission Chamber Test Parameters

Parameter	Value	Parameter	Value
Chamber volume, V[L]	119	Preconditioning period	-
Air Change rate, n[h <sup>-1</sup> ]	0.5	Area specific ventilation rate, q [m/h or m <sup>3</sup> /m <sup>2</sup> /h]	0.76
Relative humidity of supply air, RH [%]	50 ± 3	Loading factor [m <sup>2</sup> /m <sup>3</sup> ]	0.66
Temperature of supply air, T [°C]	23 ± 1		

### 3.3 Picture example of a prepared sample for VOC emission testing



## 4 Analytical Results

### 4.1 Density variation in test set

The tested samples in the first batch were with varying density from 14 to 30 kg/m<sup>3</sup>. It was observed that more dense samples had higher emissions, and it was also observed a tendency that emissions were becoming lower per mass the denser the product got.

### 4.2 VOC emissions from EPS samples

The emissions from the EPS samples were measured on tenax tubes in accordance with ISO 16000 and the main emissions of VOC was determined to be styrene and in smaller degree ethyl benzene.

	TVOC 3d µg/m <sup>3</sup>	Styrene 3d µg/m <sup>3</sup>	TVOC 28d µg/m <sup>3</sup>	Styrene 28d µg/m <sup>3</sup>
<b>Average</b>	72	54	25	20
<b>RSD</b>	50%	55%	67%	66%
<b>Highest measurement</b>	140	100	58	46
<b>Lowest measurement</b>	21	8.9	6.4	2.1

Table 1: The average results of TVOC and styrene emissions and min/max measurements from the testing of 21 EPS samples with varying density from 14 to 30 kg/m<sup>3</sup> with over half the test set being 22 ± 2 kg/m<sup>3</sup>.

From one of the samples, there was detected the emission of benzene in low amounts after 3 days but nothing could be measured after 28 days in the chamber.

The emissions were evaluated by the EU LCI values for the safety of the emissions. Styrene has an LCI value of 250 µg/m<sup>3</sup> and ethylbenzene have an LCI value of 850 µg/m<sup>3</sup>.

	28d R-Value
<b>Average</b>	0.084
<b>RSD</b>	60%
<b>Highest measurement</b>	0.19
<b>Lowest measurement</b>	0.029

Table 2: R-value calculated using the European LCI value for the compounds detected in the emissions.

### 4.3 VVOC emissions from EPS samples

There was observed significant emissions from pentane and isomers thereof in the initial testing with tenax sampling according to ISO 16000-6. Tenax is not a good adsorbent for VVOC, so therefore the second batch was also sampled on charcoal tubes.

	Pentane and isomers 3d $\mu\text{g}/\text{m}^3$	Pentane and isomers 28d $\mu\text{g}/\text{m}^3$
<b>Average</b>	19800	6900
<b>RSD</b>	16%	27%
<b>Highest measurement</b>	26000	10000
<b>Lowest measurement</b>	16000	3800

Table 3: The average results of pentane and isomers thereof and min/max measurements from the testing of 12 EPS samples with density being  $22 \pm 2 \text{ kg}/\text{m}^3$ .

#### 4.4 Aldehydes emissions from EPS samples

The analysis of the emission of aldehydes was measured using DNPH tubes according to ISO 16000-3 and there were not detected aldehydes in the testing.

	Single aldehydes (C1-C4) 3d $\mu\text{g}/\text{m}^3$	Single aldehydes (C1-C4) 28d $\mu\text{g}/\text{m}^3$
<b>Average</b>	< 5	< 5
<b>RSD</b>	0%	0%
<b>Highest measurement</b>	< 5	< 5
<b>Lowest measurement</b>	< 5	< 5

Table 4: The average results of single aldehydes and min/max measurements from the testing of 21 EPS samples with varying density from 14 to  $30 \text{ kg}/\text{m}^3$  with over half the test set being  $22 \pm 2 \text{ kg}/\text{m}^3$ .

There were not detected emissions from aldehydes. The tested single aldehydes were formaldehyde, acetaldehyde, propionaldehyde and butyraldehyde.

#### 4.5 Measurement of free styrene content before and after emission testing

The free styrene content was measured in the samples before starting the emission and also again after the emission test had ended.

	Free Styrene before emission testing [mg/kg]	Free Styrene after emission testing [mg/kg]
<b>Average</b>	483	453
<b>RSD</b>	40%	44%
<b>Highest measurement</b>	880	850
<b>Lowest measurement</b>	240	210

Table 5: The average of free styrene before and after the emission test in 12 EPS samples with density being  $22 \pm 2 \text{ kg}/\text{m}^3$ .

The measurement of free styrene content, and also the pentane incl isomers content, were performed by the client prior to sending the samples to Eurofins. These results can be seen in table 5 here below.

The results are only valid for the tested sample(s).

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	<b>Residual monomer Styrene (initial pearls) [mg/kg]</b>	<b>Residual monomer Styrene (before packing) [mg/kg]</b>	<b>Pentane, total sum of isomers (initial pearls) [mg/kg]</b>	<b>Pentane, total sum of isomers (before packing) [mg/kg]</b>
<b>Average</b>	563	437	56667	29108
<b>RSD</b>	40%	45%	10%	26%
<b>Highest measurement</b>	925	888	64300	40700
<b>Lowest measurement</b>	267	245	45200	15000

Table 6: Free styrene monomer and pentane incl isomers measurements performed by the client prior to sending the samples to Eurofins.

## 5 Discussion of the results

### 5.1 Density variation in test set

The higher emissions from more dense samples make sense, as there is essentially being tested more material in the chamber as the sample size is fixed.

There was observed a tendency of lower diffusion of VOC in the sample with increasing density, which also makes sense as the larger amount of material gives a longer diffusion path.

As the two above effects counteracts each other and overall it seems that the emissions isn't highly dependent on density for the entire test set, which is most likely due to different production optimizations of the test set.

### 5.2 VOC emissions from EPS samples

There is a significant reduction in the detected amount of VOC going from 3 days to 28 days, which is as expected. The reduction of styrene emissions going from 3 day to 28 day is calculated to be 37% on average.

The emission levels detected are far below the limit values of the European regulations with respect to total volatile organic compounds (TVOC). The French regulation have a specific limit value for styrene of 250 µg/m<sup>3</sup> after 28 days for an A+ rating, and the results are also well below this value.

The R-value is a parameter where there have been defined limits for a little over 100 VOC compounds. The R-value is calculated by taking the concentrations of a compound and dividing it by the LCI/NIK value and then summing up for all compounds with defined LCI/NIK value as in the equation presented below. The limit value of the R-value is 1 in the German and Belgian regulation.

$$R = \sum_i^n \left( \frac{c_i}{LCI_i} + \dots + \frac{c_n}{LCI_n} \right)$$

Styrene has a defined LCI/NIK value of 250 µg/m<sup>3</sup> which means that a detected concentration of styrene after 28 days above 250 µg/m<sup>3</sup> would give an R-value above 1. The R-values of the tested samples are listed here below.

The contribution to the R-value comes almost exclusively from styrene, and there is therefore a close correlation between the R-value and the styrene concentration. All R-values from the tested samples are below the limit value in the European regulations.

### 5.2.1 Point of Note of VOC Emission Testing

There can be said things about what have not been detected. Notably there were not detected formaldehyde emissions from the samples, which is a rising concern since the implementation of the French VOC regulation in 2011. The French regulation set an ambitious target for their A+ class at 10 µg/m<sup>3</sup> formaldehyde which was challenging for many product types. The focus on formaldehyde has increased since the classification was escalated from C3 to C1B carcinogenic in 2015 in Europe.

From the emissions, it is worth noting that several samples showed trace level emissions (<2 µg/m<sup>3</sup>) of the carcinogenic compound benzene after 3 days. The amount is below the limit value in any of the European regulations as of May 2016. The only protocol used in regulations which have a limit value on carcinogens after 3 days is the German AgBB protocol, and the AgBB limit value is 10 µg/m<sup>3</sup> of total carcinogens. The limit value of carcinogen after 28 days is 1 µg/m<sup>3</sup>.

The quantification of benzene on tenax can be a difficult task if the tenax adsorbent is not treated properly. It is important to track the amount of times tenax have been used, as old tenax will degenerate to more benzene than relatively fresh tenax. The analysis of tenax ATD tubes have been performed using a double desorption program, which enables separation of the background benzene from the adsorbed benzene from the sample. The general background level of benzene is below 0.5 µg/m<sup>3</sup> using the Eurofins S.O.P. Results can be confirmed using a charcoal sampling technique where the adsorbent does not degrade in to benzene, but this have not been performed for the testing discussed in this report as it was not necessary.

### 5.3 VVOC emissions from EPS samples

The results after 3 days tend to be a little overloaded on the MS detector and these results may therefore be underestimated. There is no apparent correlation between the emissions of pentane isomers and styrene content or styrene emissions.

As a point of comparison, the German AgBB test method has a TVOC limit value of 10000 µg/m<sup>3</sup> after 3 days and 1000 µg/m<sup>3</sup> after 28 days (note that VVOC does not have a limit value). This is of course depending on the loading scenario, where this emission test have been performed using a loading factor of 0.66 m<sup>2</sup>/m<sup>3</sup>, which sits between a wall and a floor covering scenario in the European reference room.

The North American standard test (CDPH, CA01350) defines VOC differently than the European tests. VOC, according to this protocol, is defined as any compound eluting between C5 and C17 and the isomers of pentanes will most likely be defined as VOC according to CDPH. CDPH does not have a limit value for TVOC and only regulates 35 specific compounds and isomers of pentane are not among the regulated compounds.

The difference in pentane and isomers free content in the samples tested in the emission chamber could not be detected in the measurements.

### 5.4 Measurement of free styrene content before and after emission testing

There is no apparent correlation between the loss of styrene and the styrene detected in the initial content determination, and the loss ranges from 2.9% to 18.2% taking the defined outliers into concern. If these are removed as outliers, then the picture gets somewhat clearer and the styrene

content loss from 28 days of emission testing can be approximated to 40 mg/kg with a standard deviation of 15 mg/kg.

### **5.5 Connection between emissions and content measurements**

The measurements of free styrene content and the emission of styrene were investigated and there was found a connection even though the correlation wasn't that good. The data could be split into two sets with a significantly better correlation. This points to a difference in production could have an impact on the emission of styrene.

### **5.6 Emission potential**

There was measured a low amount of styrene after 28 days, but the reduction in free styrene was less. It must be expected that the product will emit very low amounts of styrene over a long period. This is also true for pentane and isomers thereof.

## 6 Conclusion

All samples were tested according to ISO 16000-3 -6 -9 -11 and CEN/TS 16516 at an ISO 17025 accredited laboratory. The highest TVOC measured after 3 days was  $140 \mu\text{g}/\text{m}^3$  and after 28 days was  $58 \mu\text{g}/\text{m}^3$ . The main VOC compound detected was styrene with small amounts of ethyl benzene. There was not detected formaldehyde in the emissions from the products.

There was detected an amount of pentane and isomers thereof in the emissions which does not factor in to the TVOC, as pentane and the isomers were VVOC according to CEN/TS 16516.

All tested products live up to the current regulations in place around Europe and has emissions which are below AgBB limit values and would be rated A+ in the French VOC regulation.

## 7 Appendices

### 7.1 Qualitative Description of VOC Emission Test

#### 7.1.1 Test Chamber

The test chamber is made of stainless steel. A multi-step air clean-up is performed before loading the chamber, and a blank check of the empty chamber is performed.

The chamber operation parameters are as described in the test method section. (CEN/TS 16516, ISO 16000-9, internal method no.: 71M549811).

#### 7.1.2 Expression of the Emission Test Results

All test results are calculated as specific emissions rate, and as extrapolated air concentration in the European Reference Room (CEN/TS 16516, AgBB, EMICODE, M1 and Indoor Air Comfort).

#### 7.1.3 Testing of Carcinogenic VOC Emissions

The emission of carcinogens (EU Categories C1A and C1B, as per European law) is tested by drawing sample air from the test chamber outlet through Tenax TA tubes after the specified duration of storage in the ventilated test chamber. Analysis is performed by ATD-GC/MS (automated thermal desorption coupled with gas chromatography and mass spectroscopy using 30 m HP-5 (slightly polar) column with 0.25 mm ID and 0.25 µm film, Agilent) (CEN/TS 16516, ISO 16000-6, internal methods no.: 71M549812 / 71M542808B).

All identified carcinogenic VOCs are listed; if a carcinogenic VOC is not listed then it has not been detected. Quantification is performed using the TIC signal and authentic response factors, or the relative response factors relative to toluene for the individual compounds.

This test only covers substances that can be adsorbed on Tenax TA and can be thermally desorbed. If other emissions occur, then these substances cannot be detected (or with limited reliability only).

#### 7.1.4 Testing of VOC, SVOC and VVOC Emissions

The emissions of volatile organic compounds are tested by drawing sample air from the test chamber outlet through Tenax TA tubes after the specified duration of storage in the ventilated test chamber. Analysis is performed by ATD-GC/MS using HP-5 column (30 m, 0.25mm ID, 0.25µm film) (CEN/TS 16516, ISO 16000-6, internal methods no.: 71M549812 / 71M542808B).

All single substances that are listed with a LCI/NIK value in the latest publications (hereafter referred to as target compounds) are identified if present. All other appearing VOCs are identified as far as possible. Quantification of target compounds is done using the TIC signal and authentic response factors, or the relative response factors relative to toluene. For certain compound groups, which differ significantly in chemistry from toluene, quantification is performed relative to a representative member of the group for more accurate and precise results. This can include quantification of for example glycols and acids. In addition to that, all results are also expressed in toluene equivalents. All non-target compounds, as well as all non-identified substances, are quantified in toluene equivalents.

The results of the individual substances are calculated in three groups depending on their retention time when analyzing using a non-polar column (HP-1):

- Volatile Organic Compounds (VOC) are defined as: All substances eluting between and including

- n-hexane (n-C6) and n-hexadecane (n-C16)

- Semi-Volatile Organic Compounds (SVOC) are defined as: All substances eluting after n-hexadecane (n-C16) and before and including n-docosane (n-C22)

The results are only valid for the tested sample(s).

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- Very Volatile Organic Compounds (VVOC) are defined as: All substances eluting before n-hexane (n-C6).

Total Volatile Organic Compounds (TVOC) is calculated by summation of all individual VOCs with a concentration  $\geq 5 \mu\text{g}/\text{m}^3$ . The TVOC can be expressed either in toluene equivalents as defined in CEN/TS 16516 and similar to ISO 16000-6, or as the sum of concentrations using specific or relative response factors. In the case of summation of concentrations using authentic or relative response factors, the toluene equivalent is applied to all non-target and non-identified VOCs before summing up. Compounds regarded as VOC in line with the above definition but elute before n-C6 or after n-C16 on the HP-5 column are treated as VOC, and are thus added to the TVOC.

Total Semi-Volatile Organic Compounds (TSVOC) is calculated by the summation of all individual SVOCs expressed in toluene equivalents with a concentration  $\geq 5 \mu\text{g}/\text{m}^3$ , as defined in CEN/TS 16516. VOCs that are regarded as VOC in line with the above definition, but elute after n-C16 in this test, are not added to the TSVOC.

Total Very Volatile Organic Compounds (TVVOC) is calculated by the summation of all individual VVOCs with a concentration  $\geq 5 \mu\text{g}/\text{m}^3$  and expressed in toluene equivalents. VOCs that are regarded as VOC in line with the above definition, but elute before n-C6 in this test, are not added to the TVVOC.

This test only covers substances which can be adsorbed on Tenax TA and can be thermally desorbed. If emissions of substances outside these specifications occur then these substances cannot be detected (or with limited reliability only).

### 7.1.5 Testing of Aldehydes Emissions

The presence of aldehydes after the specified duration of storage in the ventilated test chamber is tested by drawing air samples from the test chamber outlet through DNPH-coated silicagel tubes after the specified duration of storage in the ventilated test chamber. Analysis is performed by solvent desorption and subsequently by HPLC and UV-/diode array detection (CEN/TS 16516, ISO 16000-3, VDI 3862 Blatt 3, internal methods no.: 71M549812 / 71M548400).

The absence of formaldehyde and other aldehydes is stated if UV detector response at the specific wavelength is lacking at the specific retention time in the chromatogram. Otherwise it is checked whether the reporting limit is exceeded. In this case the identity is finally checked by comparing full scan sample UV spectra with full scan standard UV spectra.

### 7.1.6 Testing of Isomers of Pentane Emissions

The emission of Isomers of Pentane is tested by drawing sample air from the test chamber outlet through charcoal tubes after the specified duration of storage in the ventilated test chamber. The analysis is carried with DMF desorption and HS-GC/MS (ISO/CD 16200-1:2001, EN 14662-2:2005, internal Method: 71M549812 / 71M546209).

## 7.2 Quality Assurance of VOC Emission testing

Before loading the test chamber, a blank check of the empty chamber is performed and compliance with background concentrations in accordance with CEN/TS 16516 / ISO 16000-9 is determined.

Air sampling at the chamber outlet and subsequent analysis is performed in duplicate. Relative humidity, temperature and air change rate in the chambers is logged every 5 minutes and checked daily. A double determination is performed on random samples at a regular interval and results are registered in a control chart to ensure the uncertainty and reproducibility of the method.

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The stability of the analytical system is checked by a general function test of device and column, and by use of control charts for monitoring the response of individual substances prior to each analytical sequence.

### **7.3 Measurement of Styrene Content**

The sample was dissolved in dimethyl furan (DMF) and subsequently analysed on HS-GC/MS with compound specific calibration (Internal methods no.: 71M526209).

### **7.4 Measurement of Density**

The density was measured by cutting the sample into blocks of approximately 5 x 5 x 10 cm and weighing them on a laboratory weight. The determination was performed in double.

### **7.5 Quality Assurance of content testing**

The stability of the analytical system is checked by a general function test of device and column, and by use of control charts for monitoring the response of individual substances prior to each analytical sequence. The analysis was done using methodology comparable to the requirements of ISO 17025.