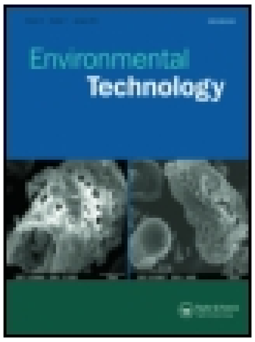

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Pre-selection test of jury for improvement of olfactometric certification efficiency

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Abstract

To certificate an olfatometric jury, laboratories usually follow up the panelist screening methodology described in the European Standard EN 13725/2003. The procedure takes a lot of time, labor and money. In laboratory routine of LCQAr – Laboratory of Air Quality Control, of Federal University of Santa Catarina, Brazil, it was found that the efficiency of jury approvals used to be as low as around 30%. In order to improve the efficiency, a quick preselection test was proposed and tried for late certification recommended by EN 13725. The methodology to create the preselection test was based on the conceptions of the standards EN 13725 (CEN, 2003), ASTM 679 (2011) and ASTM 544 (2010). In the trial test, 31 volunteers participated and then screened according to the EN13725 standard. It was verified that the efficiency increased to 46% from about 30% after the introduction of pre-selection test. The experiments were conducted at LCQAr, with the contribution of Water Research Centre of University of New South Wales, Australia

Keywords: olfactometry, certification, pre-selection, panelist screening.

1 Introduction

Odour measurement is a crucial element of odour management and regulation. Significant researches have been conducted to provide a basis for quantifying odour strength in techniques and methodologies. Dynamic olfactometry is the most common approach to quantify odour strength in Europe and America [1, 2]. This method is based on the dilution-to-threshold principle, on which a sample of odorous air can be described in terms of the volume of clean air for its odour intensity to be reduced to the sensory threshold level. In other words, the more dilutions required to make an odour sample undetectable, the higher the odour concentration must be [3]. The dilution factor of an odorous mixture represents the odour level of that sample. Odour levels can be used as input for dispersion models to calculate setback distances as well as for efficiency of odour abatement techniques, providing important information to the authorities and air quality managers [4].

Standards were developed to ensure optimal consistency within samples and laboratories in order to achieve comparable olfactometry measurement results [3]. The most widely accepted standard is European Community EN 13725 [5]. This standard stipulates the application of qualified assessors, a calibrated dilution apparatus and presentation of odorants in ascending strength or in random order. Furthermore, laboratories shall comply with quality criteria for trueness and precision (repeatability) [4].

Several issues which could affect the repeatability and accuracy of olfactometry results have been studied by [2]. According to the authors, the most significant issue by a wide margin for repeatability and reproducibility among laboratories and samples is panel selection. Obviously, the olfactometric analyses requires trained panel as EN 13725 defined criteria for panel member selection. As stated in EN 13725, assessors with specific qualities shall be selected from the general population to serve as panel members. However, the panel selection is a time consuming task, each test with 6 people takes around 20 to 40 minutes depending on the olfactometer type (Yes/No or forced choice) From historical register of Laboratório de Controle da Qualidade do Ar (LCQAr) in Brazil, the panel member selection using an olfactometer has around 30% of successfulness i.e. in a group of 6 person, only 1 or 2 are qualified. The total cost could be decreased if the panel selection time can be shortened. In order to do so, a novel, simple and fast methodology was developed and tested to preselect of the panel members. The objective of preselection test is absolutely not the substitution of the traditional selection test, but exclude candidates who may not meet standard requirements.

2 Methodology

The preselection test was employed before the traditional selection test of EN 13725. The individual threshold estimate – ITE, of 31 persons were examined by both tests. Statistical analysis of the results was conducted to determine the effectiveness of the preselection test on traditional selection test approval efficiency. All the members tested were students from Federal University of Santa Catarina, with age ranging from 18 to 55 (comprising 56% female and 44% male).

2.1 Traditional selection test methodology

The traditional selection test (also known as panelist screening test) was conducted using a dynamic olfactometer (Odile 3500 model; Odotech Inc., Canada). This device is designed to generate a stream of air that is composed of the odour sample mixed with odorless air, at a known and fixed ratio. The olfactometer ODILE is equipped with odour sniffing station operating simultaneously and configured for six persons. Three sniffing ports are used for the olfactory perception thresholds analysis. The odorous gas is brought to one of them while the other two ports are fed with pure air.

During a routine dynamic olfactometry analysis, the panel is exposed to a series of sample dilutions. The odour sample is initially highly diluted such that none of the panelists can distinguish the odour from a stream of odorless air. Then, the operator gradually decreases the dilution factor until all panelists can distinguish between the odorous and the odorless air streams. The responses of the odour panel to the dilution series are collected and the individual perception threshold is calculated by means of EN 13725 [7].

According to the EN 13725, to be selected as panel member, the data collected for that member shall comply with the following criteria:

- the antilog of the standard deviations calculated from the logarithms of the individual threshold estimates, expressed in mass concentration units of the reference gas (n-butanol), has to be less than 2.3;
- the geometric mean of the individual threshold estimates, expressed in mass concentration units of the reference gas, has to fall between 0.5 times and 2 times the accepted reference value for that reference material (for n-butanol 62 to 246 µg.m^{-3} or 20 a 80 ppbv).

As no pre-mixed gas phase n-butanol standard is available in Brazil during the test period, all n-butanol gas samples were prepared by injection of 2.3µL of n-butanol liquid standard into a Nalophan® bag filled with 30 L of pure air using a microsyringe (Hamilton, 0-5 µL).

2.2 Preselection test methodology

The preselection test methodology was introduced based on the standards ASTM E544/2010 and ASTM E679 [8, 9]. The methodological concepts used from ASTM E544/2010 to create the pretest were:

- n-butanol used as reference odour (as used in standard EN13725);
- Scale of n-butanol in water to determine odour intensity;
- Geometric progression scale of concentrations is used, with a factor of 2;
- Odour threshold of n-butanol in water of 2.5 ppm at 21°C.

Based on those prerogatives, a scale of ten n-butanol solutions was tested. The center of the scale (fifth flask) was the odour threshold of n-butanol, in analogy of ASTM E544/2010. From the first to the tenth flask, the concentrations increased in the factor of 2.

From ASTM E679, the preselection test considers the layout of test presentation and the ascending scales of odour concentrations. The layout of the test consisted in ten rows of three Erlenmeyers; two Erlenmeyers filled with 200 mL of distilled water, and one Erlenmeyer filled with 200 mL of n-butanol solution as shown in Figure 1. Only the operator of the preselection test knows which Erlenmeyers contain n-butanol solutions. Before the preselection test, volunteers were familiarised with the reference compound by sniffing a flask filled with diluted n-butanol in water above its odour recognition limit.

< Approximate location of Figure 1>

In order to find out concentrations of n-butanol in air, Henry constant of 8.81 x 10^{-6} atm.m³.mol^{-1} (25°C) [10, 11, 12] was used for calculation. Its molecular mass 74.12 g.mol^{-1} and density of 0.81 g.mL^{-1} at 20°C and atmospheric pressure of 1 atm were adopted directly from the standard supplier.

Table 1 presents the volume of n-butanol standard solution added in each flask. The calculated concentrations of n-butanol in liquid and gas phases were also listed based on calculation accordingly.

< Approximate location of Table 1>

3 Results

The results of the traditional certification test were used to establish the limits of approval/disapproval of candidates. Figure 2 shows the n-butanol Individual Threshold Estimate (ITE) of the preselection test. The percentage of individuals were segregated by flask number and by certified or not certified according to the standard test of EN 13725.

< Approximate location of Figure 2>

It can be seen a similar trend between the preselection and standard test certification test. High frequency of certified members with ITE was between the 4rd and 7th flask. 42% of the certified candidates perceived a n-butanol smell at the 5th flask that equivalent to the the odour perception threshold of n-butanol (2.5 ppmv - ASTM E544/2010). On the other hand, the 7th flask accumulated a large percentage of non certified (33%).
In order to eliminate possible non-certifiable candidates in traditional panelist selection tests, three hypothetical conditions were evaluated to set the optimal cutoff limits for the preselection test. The following hypotheses were tested:

1) Selection without considering the result of ITE in preselection test;
2) Selection with candidates preselected exclusively by the flask 5;
3) Selection with candidates preselected between flasks 4 and 6.

The main objective of these hypotheses was to find out the best way to exclude those candidates who may not be able to selected as panelists in olfactometry screening tests.

Figure 03 presents the results of Hypothesis 1, which has not considered ITE of all 31 candidates in the preselection test.

< Approximate location of Figure 3>

This graph shows certification results of all 31 candidates with the natural approval efficiency of 61%. To verify the Hypothesis 2, only candidates with their ITE equal to 5th flask were selected in the test. Figure 04 exhibits the certification ITE results of those preselected candidates.

<Approximate location of Figure 4>

For Hypothesis 2, the number of candidates has drastically reduced compared to Hypothesis 1 due to a very restrictive condition on preselection. This restriction had an impact on certification efficiency that increased to 89% from 61% of Hypothesis 1, and achieved higher elimination rate (58%).

Figure 05 shows the ITE certification results of candidates with preselection ITE equals to flasks 4, 5 or 6 – Hypothesis 3.

< Approximate location of Figure 5>

For hypothesis 3, the efficiency of certification was 74% that is between that of Hypothesis 1 (64%) and Hypothesis 2 (82%) (Table 2). Table 2 also shows the selected number of certifiable candidates.Table 2 shows the resumed outcome of each hypothetical condition of the preselection test on the selection of certifiable candidates and the standard certification efficiency.

< Approximate location of Table 2>

4 Conclusions

Following conclusions can be drawn after analysis of preselection test results:

- The most observed value of ITE of preselection test was flask number 5, equivalent to ASTM odour perception threshold of n-butanol [8].Candidates with their ITE equal or near to the flask five on preselection test will most likely to be qualified panelists;

- With help of preselection test and pre-determined criteria such as the Hypothesis 2 and 3, the number of certifiable candidates can be restricted and efficiency of certification can be improved;
- Elimination of candidates for olfactometry screening using preselection test can improve the screening efficiency, which will save time and cost to certify a group of odour panelists.

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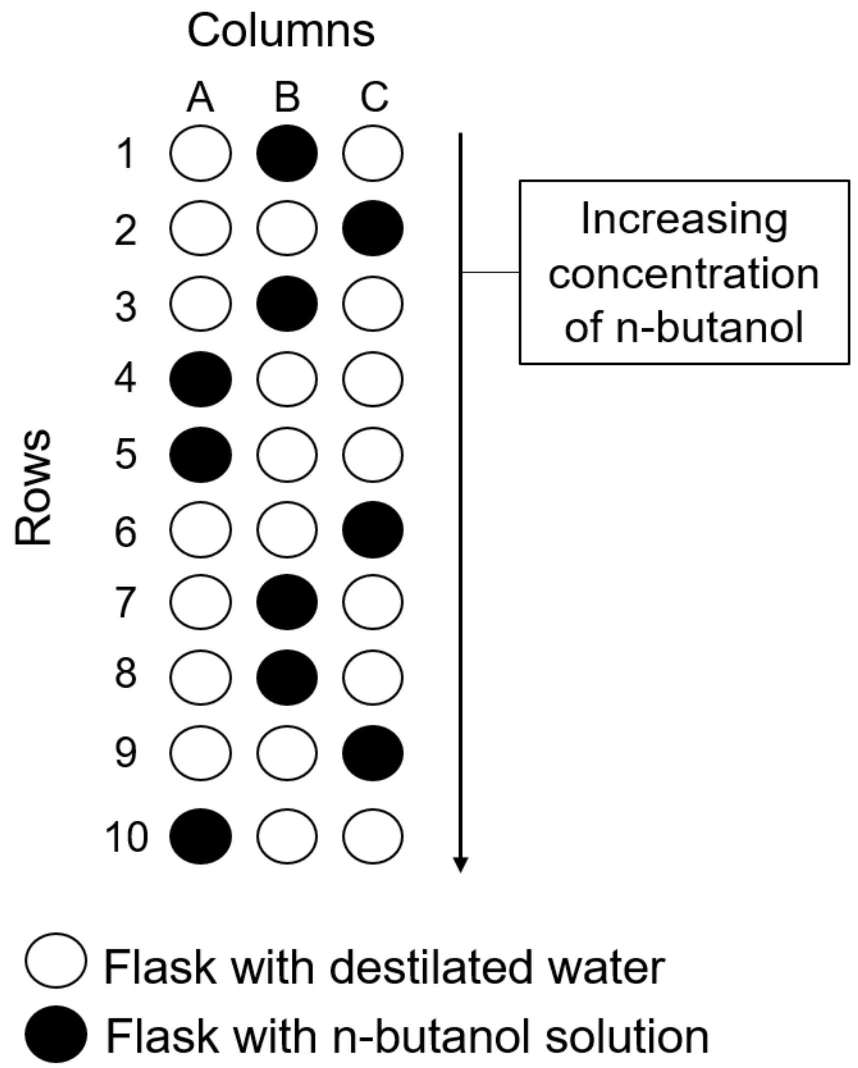
TABLE 1

Flasks	**Concentration of n-butanol (ppmv liquid phase)**	**Volume of n-butanol (µl)**	**Concentration of n-butanol (ppbv gas phase)**
1	0.16	0.03	15
2	0.31	0.06	30
3	0.62	0.12	60
4	1.25	0.25	120
5	**2.50**	**0.50**	**240**
6	5.00	1.00	480
7	10.00	2.00	960
8	20.00	4.00	1920
9	40.00	8.00	3840
10	80.00	16.00	7680

TABLE 2

Advantage	**Hypothesis 1**		**Hypothesis 2**		**Hypothesis 3**	
	Proportion		Proportion		Proportion	
Selection of certifiable candidates (a)	19/19	**00**	8/19	**2**	14/19	**4**
Efficiency of certification (ef)	19/31	**1**	8/9	**9**	14/17	**2**


Columns
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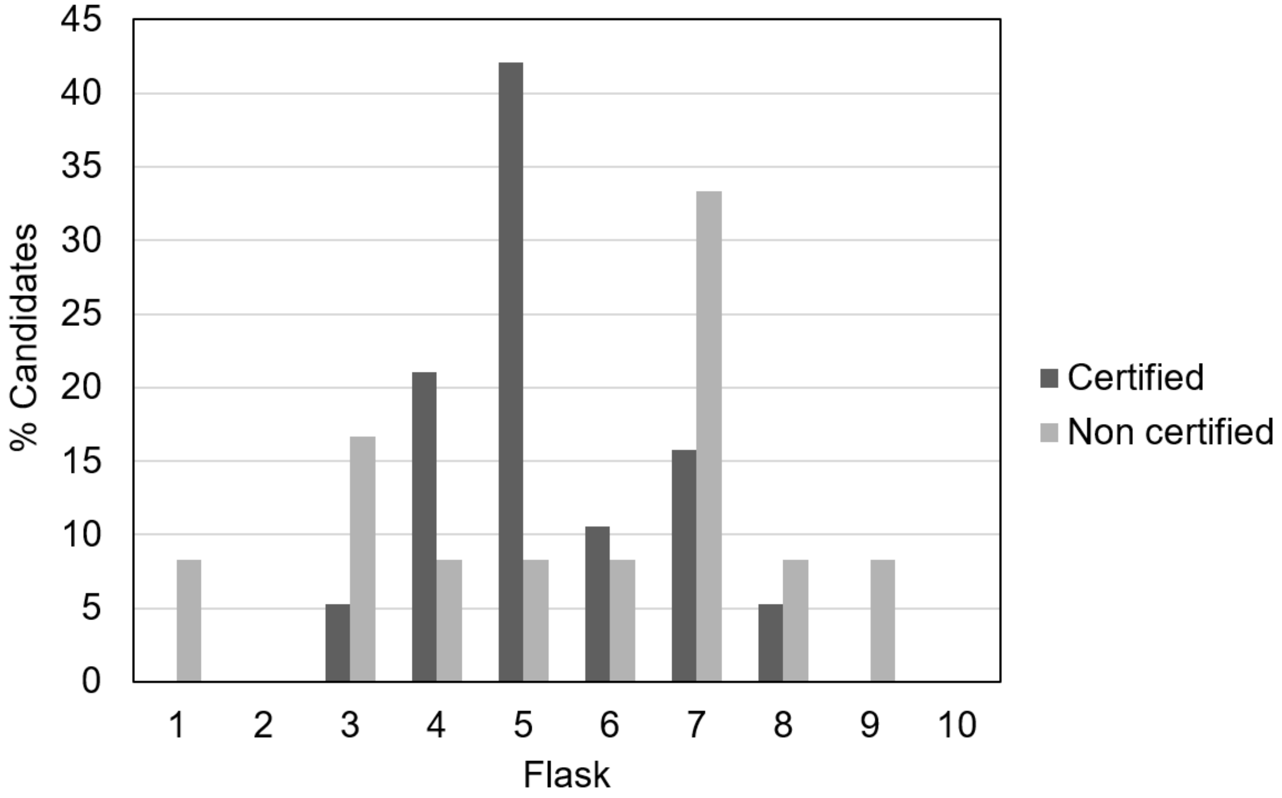
Increasing concentration of n-butanol
Flask with destilated water
Flask with n-butanol solution


45
40
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30
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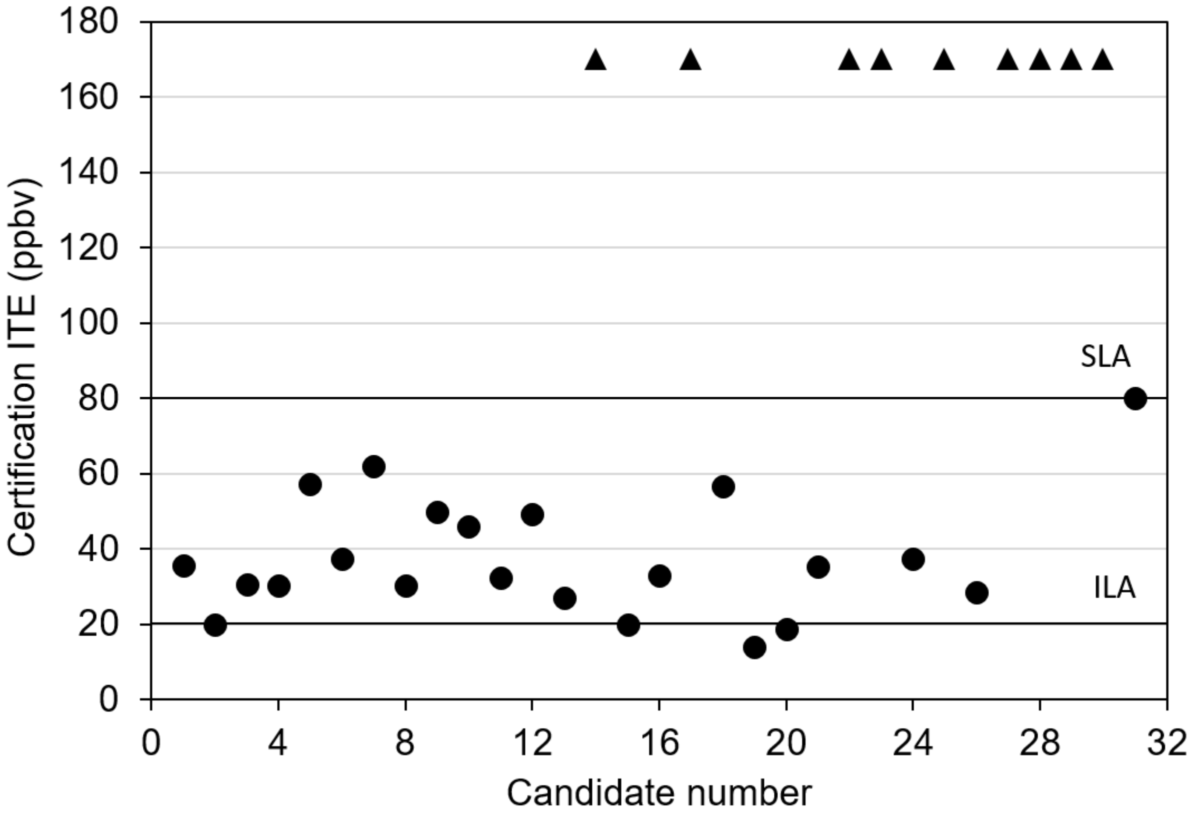
Flask
Certified
Non certified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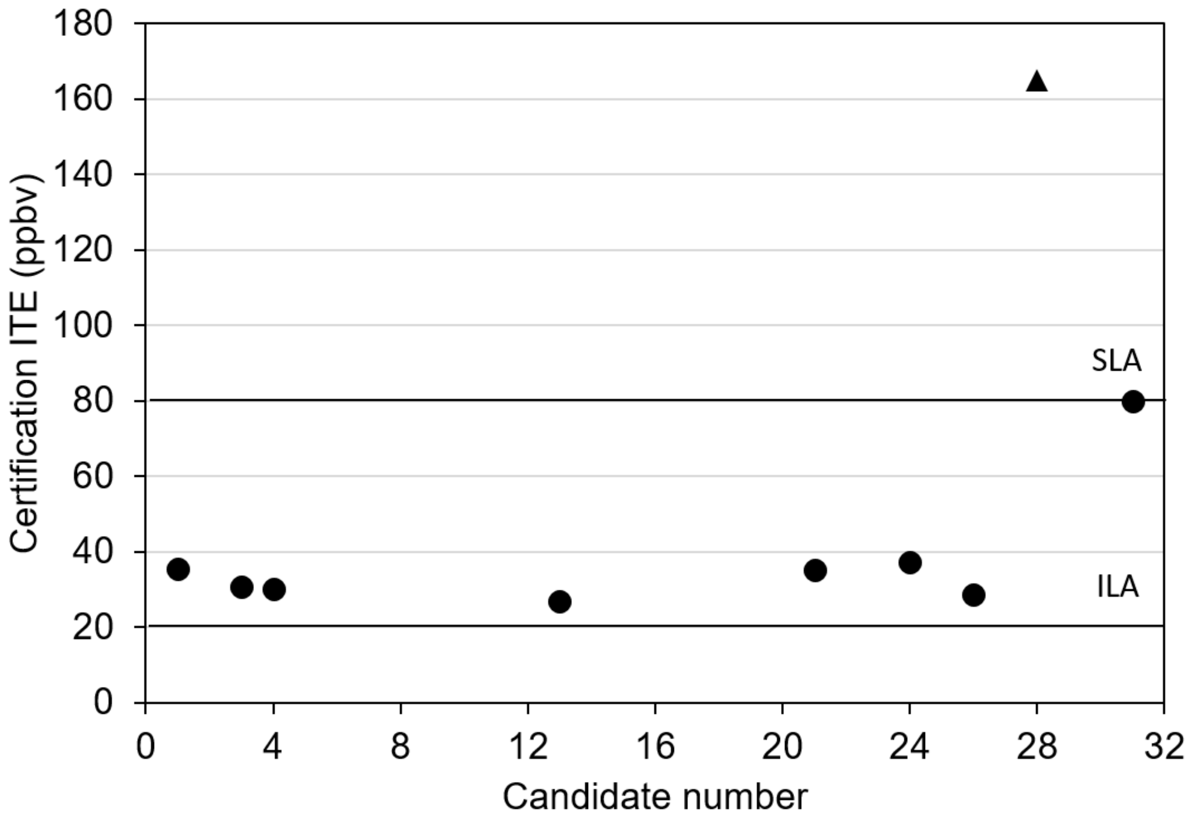
Candidate number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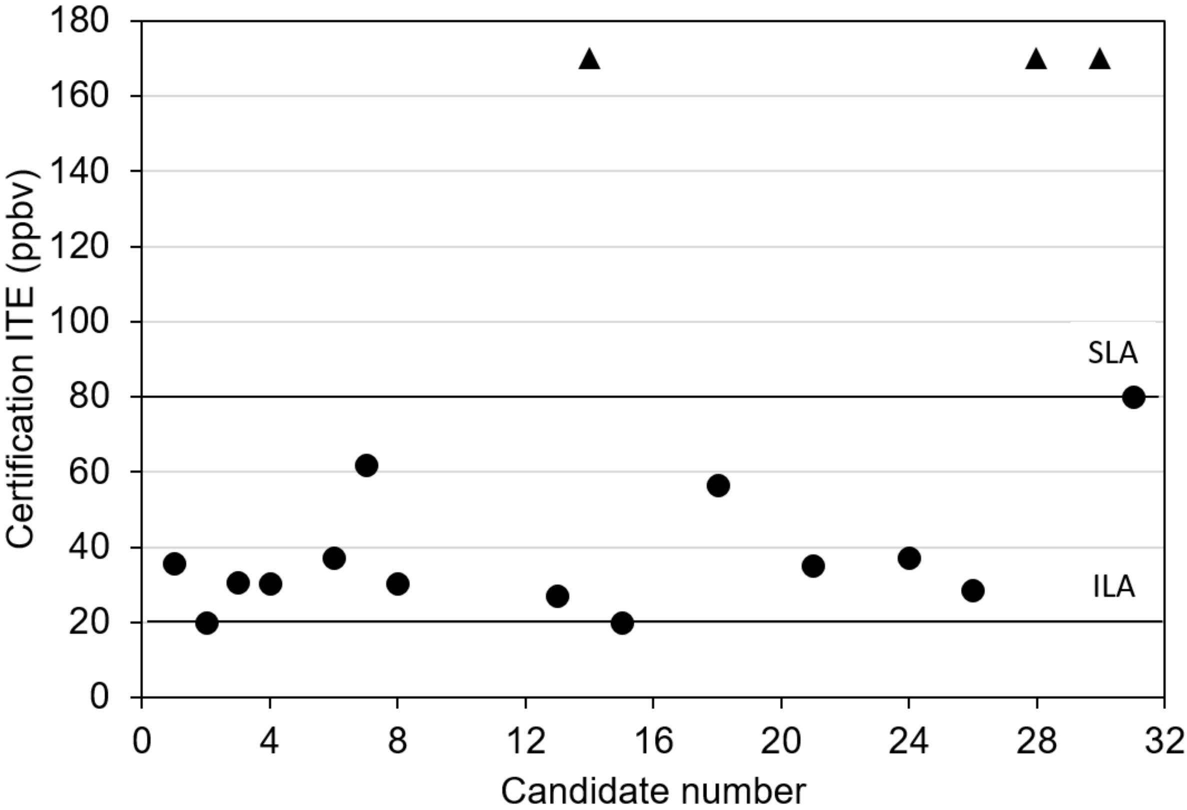
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