

## **Analytical characteristics of odour concentration measure by dynamic olfactometry: preliminary results**

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The EN 13725 represents a common reference for all European countries for the determination of odour concentration by dynamic olfactometry. In this law, in particular, the necessary requisites for the standardization of the methodology and the sensitivity, uncertainty and accuracy are defined. All the laboratories that intend to adopt this analytical method must respect the requisites expressed in the Normative mentioned. The aim of this work is to study the reliability and robustness of the method in order to establish the uncertainty associable to the results of the measures. In fact a deeper knowledge of the analytical characteristics could allow a more opportune use of the olfactometric technique.

As dynamic olfactometry is a methodology linked to more variables, not directly verifiable, because human nose is used as sensor, it is not possible to know a priori the correlations between the variables and the synergic effects, that is those effects deriving from the role of two or more variables. It could be useful to recur to chemiometrics studies, because it is a system of high complexity. With this approach the repeatability of the measures varying the exposition time of the sensorial system and the typology of the odour will be studied.

### **1. Introduction**

Dynamic olfactometry, the reference technique acknowledged by the EN 13725 at a European level, is a sensorial technique which uses the human nose of a group of standardized panellists. The standardization is performed employing a reference substance, the n-butanol, and evaluating the mean perception, which must be representative of the population. The answers towards this reference substance must be as constant as possible in order to guarantee the repeatability of the results. The olfactory sensitivity of the panellists must vary in a range narrower than the one of common people. The selection is based on the hypothesis that the answers of the panellists and their characteristics defined on the reference substance can be transferred to other substances, represented by real samples. The olfactory perception, however, because of its intrinsic physiologic nature, is linked to a complex system, made up of physical and psychological factors of the receptor and it depends on the chemical-physical characteristics of the odorous substances. The mechanism that leads to the olfactory perception rises from the nose, in the olfactory epithelium, where the sensorial cells (neurons), responsible of the interaction with odorous molecule, are located. The sensation of odour is generated only after the information, determined by the stimulus,

has been transmitted to the brain, which works out the answer, both in terms of recognition and evaluation of the sensation, and introducing emotional and instinctive components. The nose represents the direct interface between the central olfactory system and the external environment, from which it receives the stimulus. It is fundamental the concept that it is impossible to determine the origin of an odour, if it has never been filed in a “data base” obtained by the experience of the subject.

The mechanism by which odours are perceived is constituted by two levels:

- Perception level (olfactory system);
- Recognition level (olfactory system + brain).

It is evident that the “analyzer”, the human nose, which the olfactometric technique is based on, is characterized by subjective components. Despite of the standardization provided by the European Normative, it is necessary to deep those aspects and to verify how they can influence the quality of the measure. In fact in the EN 13725 are expressed the quality requisites of the performances, in terms of accuracy (precision and exactness). In particular, when the real value is known, it is possible to determine the exactness of the measure, which is expressed in terms of systematic error. The precision can be evaluated in terms of repeatability and reproducibility: the former is referred to measures performed in a singular laboratory, instead the latter to more laboratories. Besides, in dynamic olfactometry it is not possible to reach conditions of repeatability in a strict sense, as defined in the ISO 5725. This study aims to give a measure of the uncertainty linkable to an olfactometric result, which considers the variables that influence it. This uncertainty can be determined through an opportune evaluation of the precision. In particular the attention will be focused on two variables: the exposition time of the panellist to a particular odour and the type of odour (chosen considering the pleasantness and unpleasantness of the odour, the hedonic tone). In the definition of those variables, the contribute of the eventual uncertainty introduced by the dilution system, for the different intervals of dilution, has been considered unimportant.

## 2. Objectives

The purpose of this study is the investigation of the analytical characteristics of the olfactometric method, its reliability and robustness, trying to establish the uncertainty that can be associated to obtained measures.

In particular, it intends to realize an experimental design that permits to verify the way in which different conditions vary and influence final uncertainty associated to the measure. This aim can be executed through the study of the repeatability of results that each panellist gives during a certain measure session, varying the variable time of exposure and type of odour. The statistical treatment of results will permit to obtain an estimation of precision relative to the measure and can allow the introduction of further indicators of reliability for panellists.

To execute this type of study, it's necessary considering the fact that the analysis duration depends of physiological tiredness of panellists, because dynamic olfactometry is a method based on human sensors. For this reason, it has been estimated a mean duration for the employment of analysts in a measure session equal to two hours. This time range has been established on the bases of the answers given by panellists in a questionnaire.

To evaluate the variations of the parameters considered, that is time of exposure and type of odour, it's necessary to vary them in alternative way (fixing one and varying the other). In particular, the use of certified bottles, containing a particular odour substance with a defined concentration, from which samples are collected, permits to have same samples for different analysis. In this way, the introduction of further variables in measures is avoided. In fact, samples collected in different moments, even though by the same matrix, can't be considered exactly the same because, during the sampling phase, new variables can be introduced and they can be added to those determined during analysis phase.

On the other hand, if certified bottle containing particular odour substances are not available, it's possible using a sample bag with a volume such that it can be used during a complete measure session.

To analyse the considered variables, eight panellists have been examined, each of them has executed two measure sessions with a determined odour sample in two different days.

### **3. Methodology and materials**

The olfactometric analysis have been preformed considering substances with olfactory characteristics easily recognizable and well defined, such as aromatic essences, like the orange essence, tea tree essence, menthol essence, rosemary essence, vinegar,...

The kind of odour has been chosen in relation to the hedonic scale, trying to consider essences with different pleasantness grade.

The sampling has been done withdrawing a sample from the head space of the bottle containing the essence, by means of a depression pump. In particular, the analysis have been realized considering odour concentrations, that could be perceived in the range of the dilution of the olfactometer. The olfactometer employed is the TO8 by ECOMA GmbH, with four positions, whose dilution range is  $2^2$ -  $2^{16}$ . For each session 4 panelists have been considered, to whom a sample has been submitted almost continuously for two hours.

### **4. Results**

Actually the data of the analysis performed on samples of tea tree essence and orange essence are available.

For each member of panel have been recorded the estimates of individual odour threshold of which the decimal logarithms have been calculated because it's necessary to consider the decimal logarithms of odour concentrations for calculus of statistical parameters.

As example, graphs relative to three different panellists are reported in figg. 1, 2, 3; in these graphs decimal logarithms of individual odour threshold are presented for each cycle.

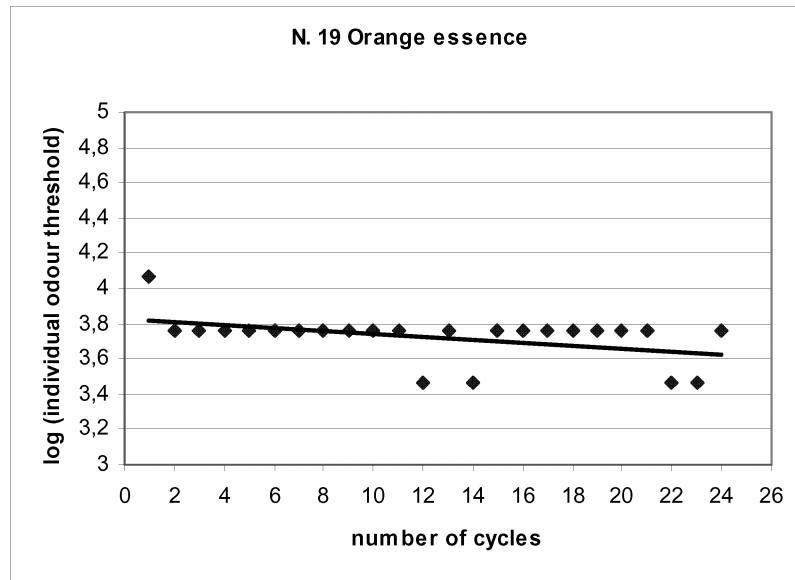


Fig. 1 Graph of the value of individual threshold of panellist n. 19 for the orange essence analysis.

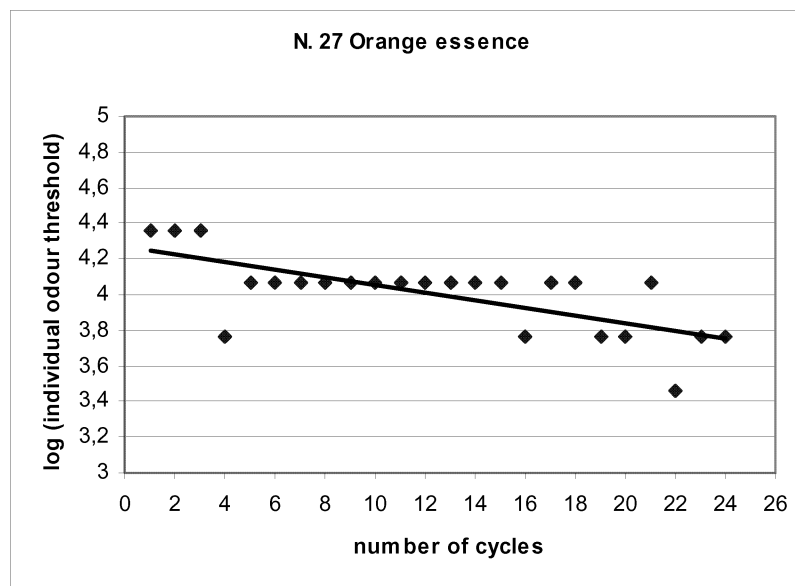


Fig. 2 Graph of the value of individual threshold of panellist n. 27 for the orange essence analysis.

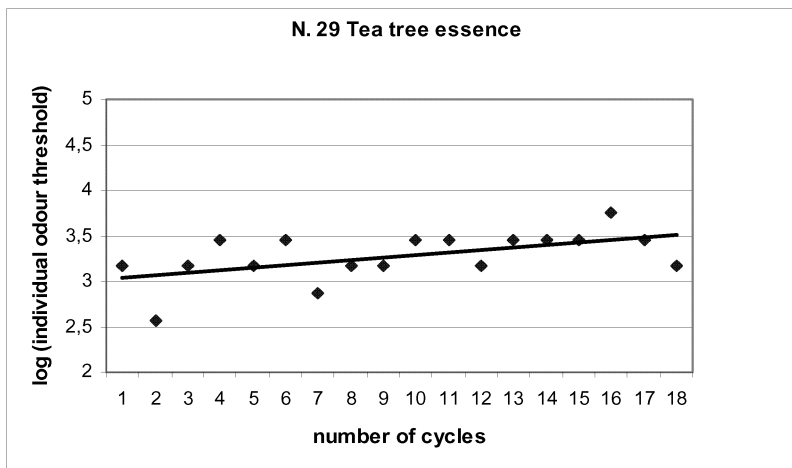


Fig. 3 Graph of the value of individual threshold of panellist n. 29 for the tea tree essence analysis.

The graphs reported are referred to three panellists with the different performance: a trend nearly constant in time (fig.1), a trend that tends to a decrease (fig.2) and a trend that tends, on the contrary, to an increase of the answers (fig. 3).

## 5. Conclusions

The present work is actually at a preliminary state of in-depth examination, because the experimental design foresees the analysis of several samples characterized by different grades of pleasantness (hedonic tone). Once the survey has been completed, proper evaluations can be done on the basis of the different conditions tuned. In particular, useful information will be available for linking a more proper uncertainty on the results. The two fundamental aspects on which the uncertainty will be defined are:

- the time in which:
  - a) The answer could be considered not significantly different from the mean of measures;
  - b) The variability is kept almost constant in a range considered acceptable;
- the variability of the global uncertainty in relation to the time.

In this way the olfactometric technique will be adopted in a proper way thanks to a deeper knowledge of the analytical characteristic of the measure.

## 6. References

- Apat "Metodi di misura delle emissioni olfattive – quadro normativo e campagne di misura" 2003
- "Air Quality – Determination of Odour Concentration by Dynamic Olfactometry" EN 13725, 2003
- "Olfactometry - Odour Threshold Determination - Fundamentals" VDI 3881 Blatt 1/Part1.

