

A comparison to assess the preparative and analytical capabilities of laboratories for the measurement of semi-volatile organic compounds in indoor air

Marivon Corbel^{1*}, N. Allen¹, M. Nohr², M. Richter², D. Heikens³, J. Li³

¹National Physical Laboratory, Analytical Science Division, United Kingdom

²BAM Federal Institute for Materials Research and Testing, Berlin, Germany

³VSL Dutch Metrology Institute, Delft, The Netherlands

Corresponding e-mail address: marivon.corbel@npl.co.uk

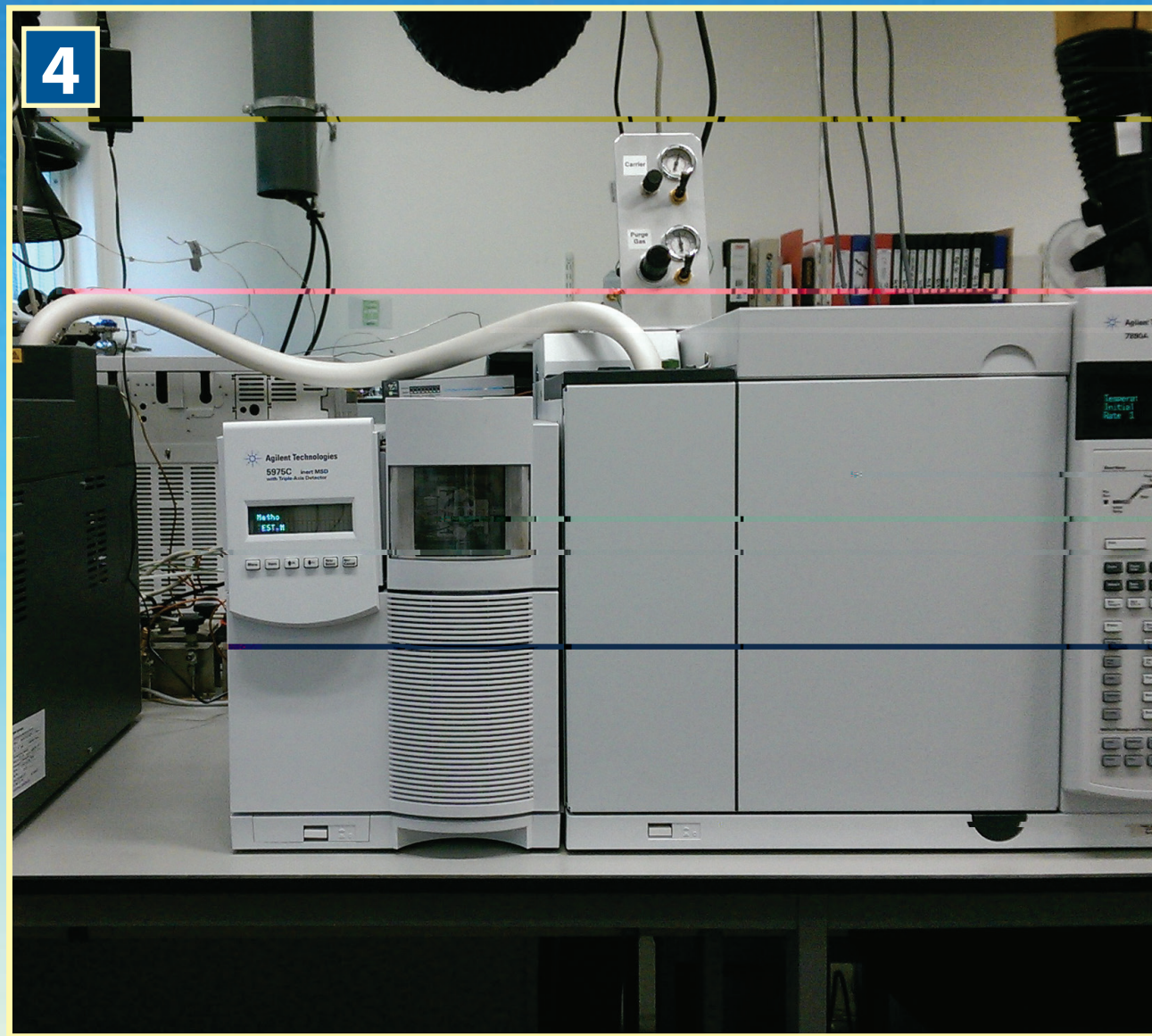
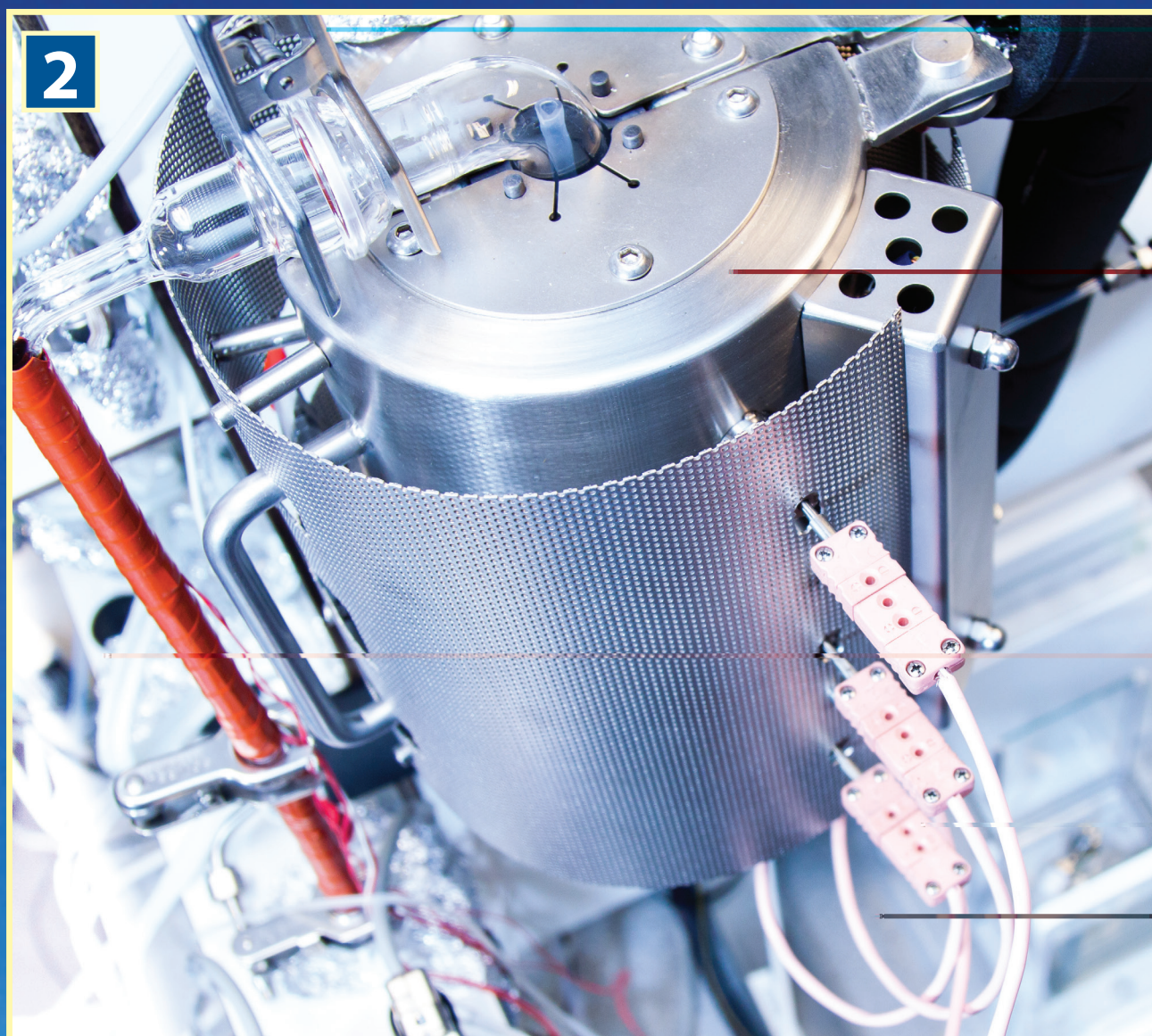
Abstract

Indoor air quality is fast becoming an area of concern due to its impact on human health and the environment. The EU ambient air quality directives set limits and data quality objectives for various pollutants. However these directives are wholly focussed on outdoor air quality. A European Metrology Research Programme project entitled “Metrology for chemical pollutants in air” aims to provide a metrological infrastructure for the measurement of key compounds in indoor air emitted from building materials so that standardisation committees, regulatory bodies, environmental monitoring networks and testing laboratories will benefit. One of the project objectives is to determine the reliability of the developed measurement approaches and the preparation of reference materials.

The study presented here focusses on the use of different approaches for preparing transfer standards. Five semi-volatile organic compounds, typically found in indoor air, were loaded onto sorbent tubes. The mass of each compound was determined using thermal desorption gas chromatography mass spectrometry. Calibration standards were prepared on sorbent tubes from a primary reference gas mixture or from a direct liquid injection. A statistical evaluation of the results obtained from these measurements is presented and discussed.

Transfer standards

Transfer standards were prepared in Tenax®TA tubes (figure 1). Sets of tubes were charged with 30 ng and 100 ng of a reference material containing the compounds listed below.



Preparation of transfer standards

Two different approaches were used to prepare transfer standards.

The first approach consists of loading the sorbent tubes with a reference gas mixture generated by dynamic dilution method. The system used is based on the delivery of a constant mass flow of liquid through a capillary. The liquid can be one pure compound or a gravimetric mixture containing several compounds. The constant mass flow is obtained by applying a constant pressure to the liquid (mixture), thereby forcing it through the capillary. The mass flow is measured by weighing the loss of liquid (mixture) in the reservoir. The delivered liquid flow is evaporated by means of a specially designed oven (figure 2) and the vapours are purged with clean nitrogen into a purified air gas stream.

The second approach consists of spiking the sorbent tubes with a reference liquid solution. A methanolic solution containing all the compounds was prepared. An aliquot of this solution was then accurately injected onto each sorbent tube using an automated system. (figure 3) The dispensing syringe was calibrated using gravimetry. After each injection, nitrogen was passed over the sorbent tube.

Analytical methods

The analysis of the sorbent tubes was carried out by each laboratory using thermal desorption systems coupled with gas chromatography (TD-GC(FID)) or gas chromatography-mass spectrometry (TD-GC-MS) (figure 4). Each laboratory measured the samples using their own analytical method, the comparability of which was investigated in this cross-check study.

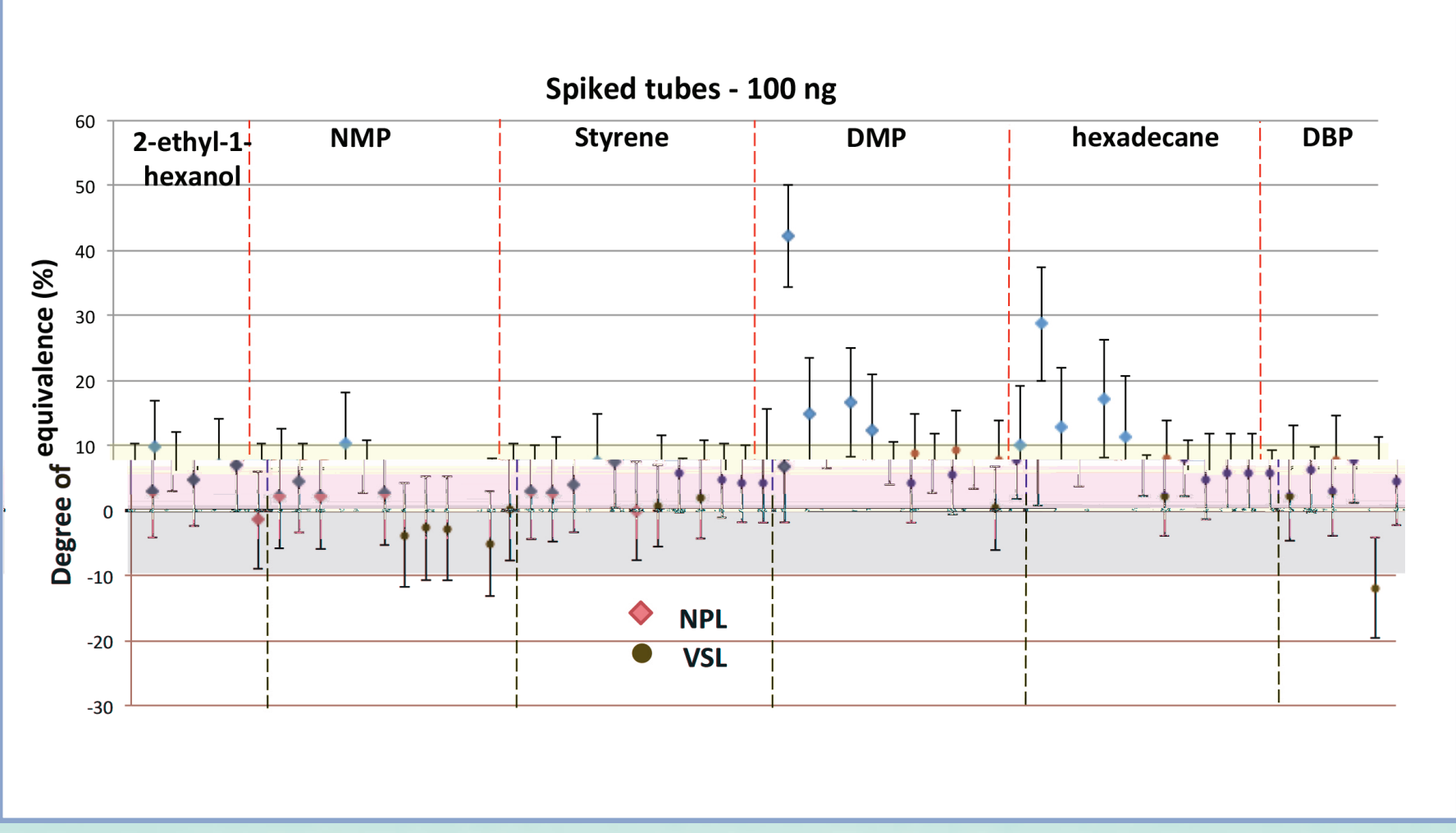
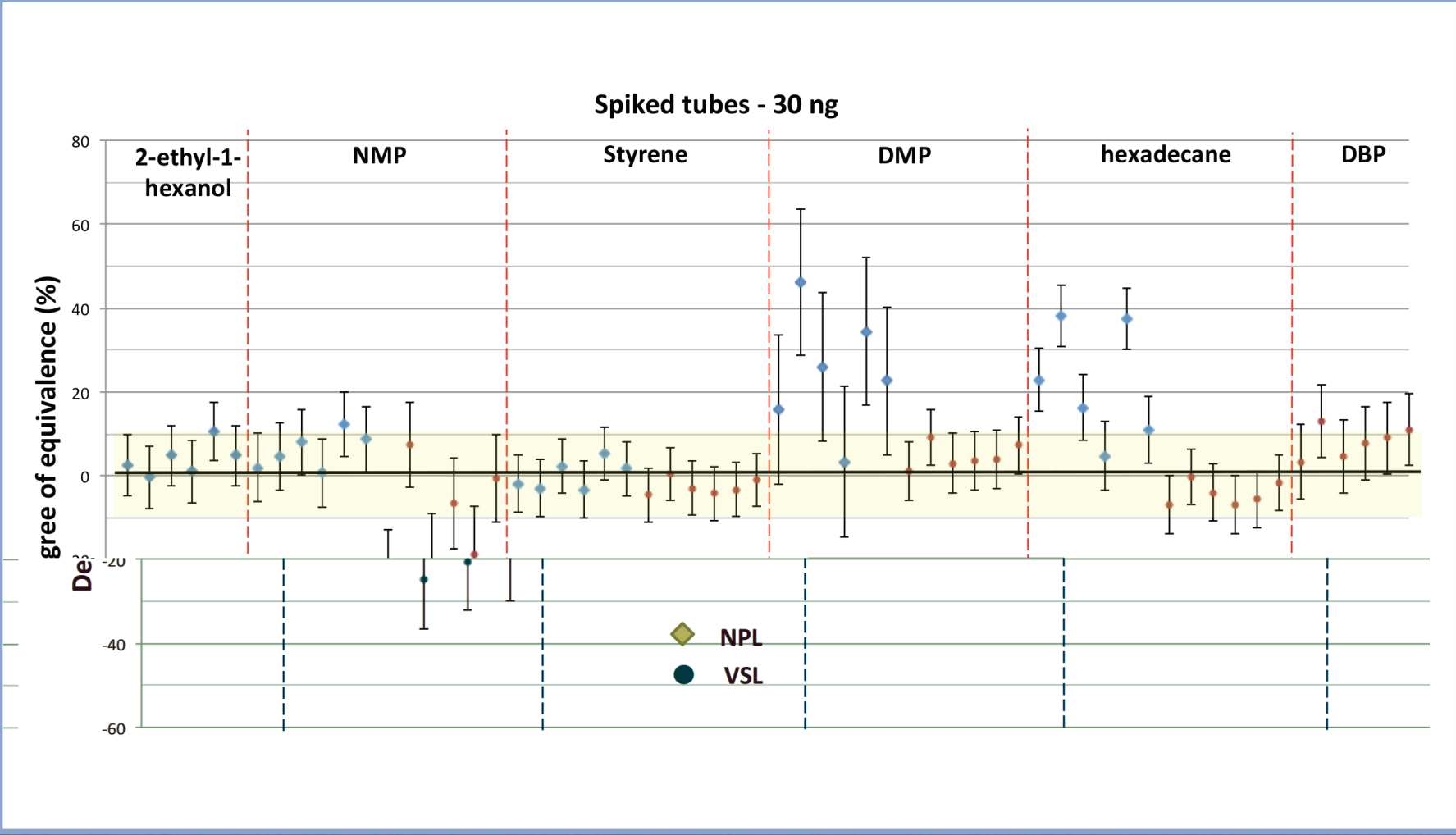
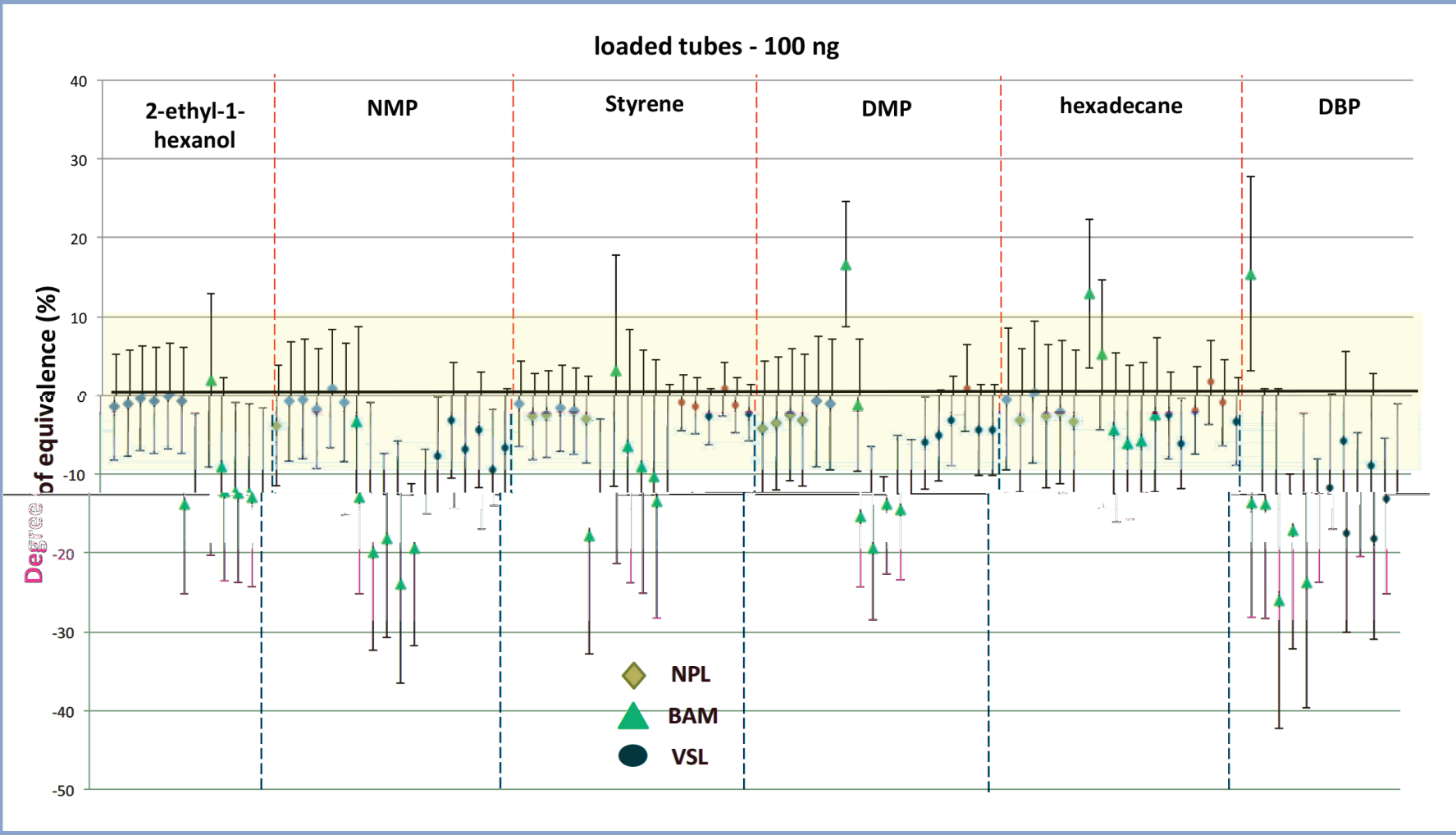
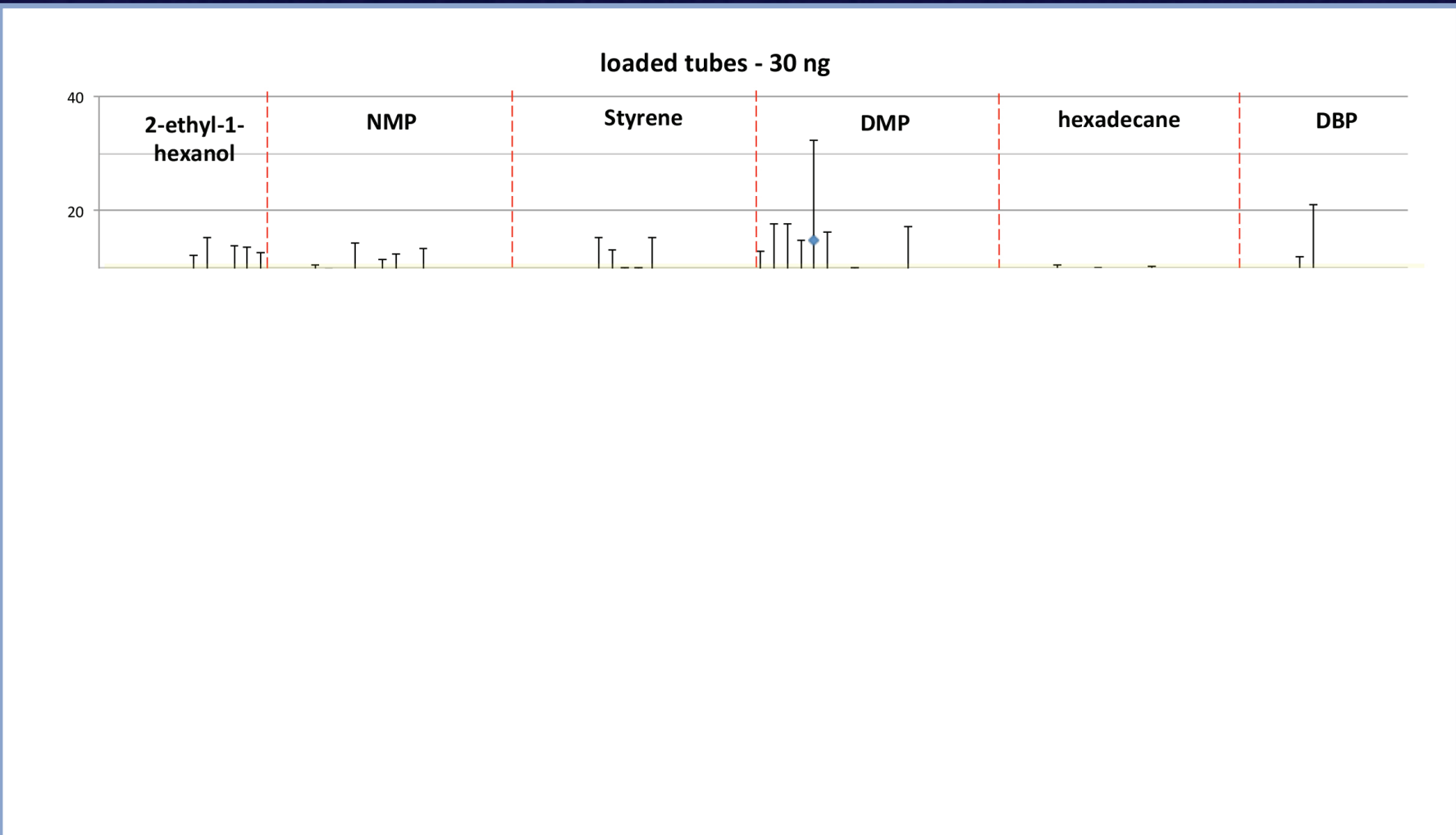
Results

The graphs show a comparison of the results for each compound for the four sets of tubes:

The degree of equivalence of each laboratory is expressed quantitatively by two terms: its deviation from the reference value and the uncertainty of this deviation (at a 95 % level of confidence). The degree of equivalence between measurement standards is expressed by the difference of their deviations from the reference value and the uncertainty of this difference (at a 95 % level of confidence) as follows:

$$\Delta x_{lab} = x_{lab} - x_{ref}$$
$$u^2(\Delta x_{lab}) = u_{lab}^2 + u_{ref}^2$$

- Δx_{lab} difference between laboratory result and reference value (ng)
- x_{lab} result of laboratory (ng)
- x_{ref} amount of loaded or spiked compound (ng)
- U_{ref} expanded uncertainty of reference value, at a 95% level of confidence (ng)
- U_{lab} expanded uncertainty of laboratory, at a 95% level of confidence (ng)



Component name	Formula	Structure
Styrene	C ₈ H ₈	
1-Methyl-2-pyrrolidone (NMP)	C ₅ H ₉ NO	
2-Ethyl-1-hexanol	C ₈ H ₁₈ O	

Globally, the preparation and the measurement methods for the measurement of volatile and semi-volatile organic compounds in indoor air are in agreement between the three laboratories (BAM, VSL and NPL). The large majority of the results agree within 10% (or 10 ng) of the reference value. This study also highlights the importance of the physical properties of the compounds selected. © Queen's Printer and Controller of HMSO, 2014.

Formulas: Dimethyl phthalate (DMP), Hexadecane, Dibutyl phthalate (DBP)