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1 Preparation of thermodesorption tube 2 standards: comparison of usual methods 3 using accuracy profile evaluation 4

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11 Abstract

12 In order to quantify organic impurities into gas produced from renewable sources, thermal desorption
13 coupled with GC-MS or GCxGC-MS is very useful. However, the preparation of the standard tubes
14 appears not to be trivial. For that, different strategies, based on commercial setups, have been
15 developed. The goal of this study is to compare the classical manual deposit of a liquid standard
16 solution with other commercial methods: the gas stream assisted deposit and the vaporization
17 followed by adsorption assisted by gas stream. A standard mixture of 48 compounds from different
18 families was used for the comparison of the performances of the three strategies using the accuracy
19 profile methodology. Global validation score was attributed to each strategy as well as a score by family
20 of compounds and by boiling point range, in order to have a detailed comparison of the techniques.
21 On the set of molecules studied, commercial setups have shown to be more efficient than the manual
22 deposit.

23 Keywords

24 Thermal desorption, Quantification, Accuracy profile, GCxGC-TOFMS

25 1. Introduction

26 The direct injection of a gas sample into GC-FID or GC-MS is often not sensitive enough to analyze low
27 concentration compounds inside gaseous matrices. Thus, a preconcentration step appears necessary
28 and it could be achieved by means of sorption tubes. Trapped compounds are then released and
29 injected into the GC via a Thermal desorption (TD) process [1, 2]. Another advantage of sampling on
30 tubes is the possibility to carry out the sampling on site and to send very simply the tubes to the
31 laboratory for further analysis [3]. Some examples of this strategy could be found in the case of biogas
32 [1, 4], of natural products [5], or to monitor industrial processes like CO₂ capture [6], etc.

33 Quantitative analysis by TD coupled with GC can be done using gaseous or liquid calibration samples
34 [7]. In this study the focus was made on the preparation of standards using liquid calibration samples.
35 Three main techniques can be used. The first one, the simplest, is the manual deposit (MD) of the
36 calibration solution directly on the head of the tube. The two other techniques considered in the

37 present study were the gas stream assisted deposit (GSAD) [1, 8, 9], and the vaporization followed by
38 adsorption assisted by gas stream (VGSD) [10–13].

39 The evaluation and comparison of the performances of the three calibration methods were carried out
40 using the accuracy profile methodology, a statistical tool developed for method validation at the
41 beginning of the 21st century [10, 14–19]. Recently, it has been applied successfully with a multi-
42 response combination strategy, to compare the quantitative performances of different mass
43 spectrometers for the determination of allergens in cosmetics [20]. Another implementation of
44 accuracy profile methodology to compare the performance of preparation techniques of
45 thermodesorption standard tubes is described in this paper.

46 2. Materials and Methods

47 2.1. Standard and tubes

48 The reference air indoor standard solution (48 VOC, reference 40353-U) and Tenax TA tubes (Poly(2,6-
49 diphenylphenylene oxide)), reference 30131-U were supplied by Sigma-Aldrich (Saint-Quentin-
50 Fallavier, France). The standard mixture was composed of: Acetone, Benzene, Bromodichloromethane,
51 1-Butanol, 2-Butanone, Chloroform, Dibromochloromethane, Decane, Decanal, 1,4-Dichlorobenzene,
52 1,2-Dichloroethane, Dichloromethane, 1,2-Dichloropropane, 2,4-Dimethylpentane, Dodecane,
53 Ethanol, Ethylbenzene, 2-Ethyltoluene, 3-Ethyltoluene, 4-Ethyltoluene, Heptane, Hexane,
54 Hexadecane, Limonene, 4-Methyl-2-pentanone, Nonanal, Nonane, Octane, Pentadecane, α -Pinene, β -
55 Pinene, 1-Propanol, 2-Propanol, Styrene, Tetrachloroethylene, Tetradecane, 1,2,4,5-
56 Tetramethylbenzene, Toluene, Trichloroethylene, Tridecane, 1,2,3-Trimethylbenzene, 1,2,4-
57 Trimethylbenzene, 1,3,5-Trimethylbenzene, 2,2,4-Trimethylpentane, Undecane, o-Xylene, m-Xylene,
58 p-Xylene, at 1000 $\mu\text{g}/\text{mL}$ in Methanol/Water (19:1 v:v).

59 A 2 μL gas tight syringe supplied by Hamilton (Villebon sur Yvette, France) was used to collect and
60 dispense the liquid samples.

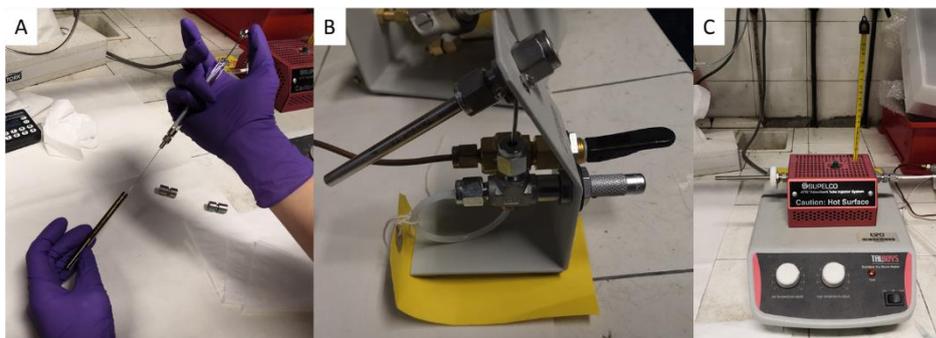
61 2.2. Tube preparation

62

63 To apply the accuracy profile evaluation methodology, several series, i.e. combinations of calibration
64 and validation tubes, were required. Calibration tubes were loaded using three different protocols
65 corresponding to the three techniques to be compared. For the three protocols, the same syringe was
66 used for sampling: 1 μL sample volume was taken between 0.2 μL volumes of air (sandwich injection)

- 67 i) Manual deposit (MD) (Figure 1-A) was performed by directly delivering the syringe content
68 to the sorbent at the head of the tube. The needle was in direct contact with the retaining
69 gauze.
- 70 ii) Gas stream assisted deposit (GSAD) (Figure 1-B) was performed using the Calibration
71 Solution Loading Rig (CSLR) from Markes International (Bridgend, United Kingdom). The
72 tube was locked with a ferrule to the system and the solution was added by direct contact
73 with the sorbent retaining gauze at the head of the tube through a septum. Helium, used
74 as carrier gas at 50 mL/min flowrate was started at the same time as the introduction of
75 the syringe. The syringe remained in place during 0.25 min and the helium was flown
76 during 3 min after the start of the deposit for a total volume of 150 mL of gas.
- 77 iii) Vaporization followed by adsorption assisted by gas stream (VGSD) (Figure 1-C) was
78 performed using the Adsorbent Tube Injector System from Sigma-Aldrich (Saint-Quentin-
79 Fallavier, France). The tube was locked with a ferrule to the system and the solution was

80 injected in the vaporization chamber heated at 140°C (stabilized at least 1 hour before
81 experiments). Helium was used as carrier gas at 50 mL/min flowrate and started at the
82 same time as the injection. The syringe remained in place during 0.25 min and the helium
83 was flown during 3 min after injection for a total volume of 150 mL of gas.



84
85 *Figure 1 – Illustrations for Manual deposit (MD) (A), Gas stream assisted deposit (GSAD) (B), Vaporization followed by*
86 *adsorption assisted by gas stream (VGSD) (C)*

87 For validation tubes, methane was used as carrier gas instead of helium. MD validation tubes were
88 loaded with 150 mL of methane before the deposit.

89 For each series, 12 calibration tubes and 9 validation tubes were loaded [21]. The four calibration levels
90 10, 7, 4 and 1 ng for each compound deposited on the tube were loaded with helium or without gas
91 (MD). The three validation levels 8.5, 5.5 and 2.5 ng for each compound deposited on the tube were
92 loaded with methane. The objective of using methane as gas for the validation tube was to mimic a
93 matrix of renewable gases. Since 21 tubes were loaded for each series, 4 series performed for each
94 technique and 3 loading techniques tested, 252 tubes were prepared for this study.

95 2.3. TD-GCxGC-MS method

96 All tubes were analyzed using a system composed of a TD100-xr from Markes International (Bridgend,
97 United Kingdom), hyphenated with a GCxGC-TOFMS Pegasus BT4D from LECO (Villepinte, France).

98 Tubes were desorbed at 270 °C for 10 min into a cold trap “General Purpose” U-T11GPC-2S (Markes
99 International, Bridgend, United Kingdom) at -30°C containing graphitized carbon at a flowrate of 50
100 mL/min. Then the cold trap was desorbed at 300 °C for 5 min with a split flow of 20 mL/min, and an
101 inlet flow of 1.2 mL/min with helium as carrier gas.

102 The separation was performed using a column set composed of an apolar column in the first dimension
103 30 m x 0.25 mm x 0.25 µm Rxi-5ms (Restek, Lisses, France) and a medium polar column in the second
104 dimension 1.1 m x 0.1 mm x 0.1 µm DB 1701 (Agilent, Les Ulis, France). The GCxGC was used with a
105 temperature program starting at 40 °C, held for 2 min then heated up to 250 °C at 3 °C/min. The
106 temperature of the secondary oven was set 5°C higher than the primary oven, and the modulator
107 temperature was set 5 °C higher than the secondary oven. Using the quad jet modulator, the
108 modulation period was set at 4 s (consisting of 2 cycles with 1.5 s of hot jet and 0.5 s of cold jet).

109 The mass spectrometer was used with electron ionization at 70eV, a scan range of m/z 45 – 300 at a
110 scan frequency of 200 Hz.

111 Data processing was handled using ChromaTOF software 5.51 from LECO (Villepinte, France). All
112 integrations were made on the most abundant m/z for each compound. The ChromaTOF software
113 automatically integrated peaks on the 1D chromatogram with a “Target Analyte Finding” method
114 (TAF), followed by a manual checking.

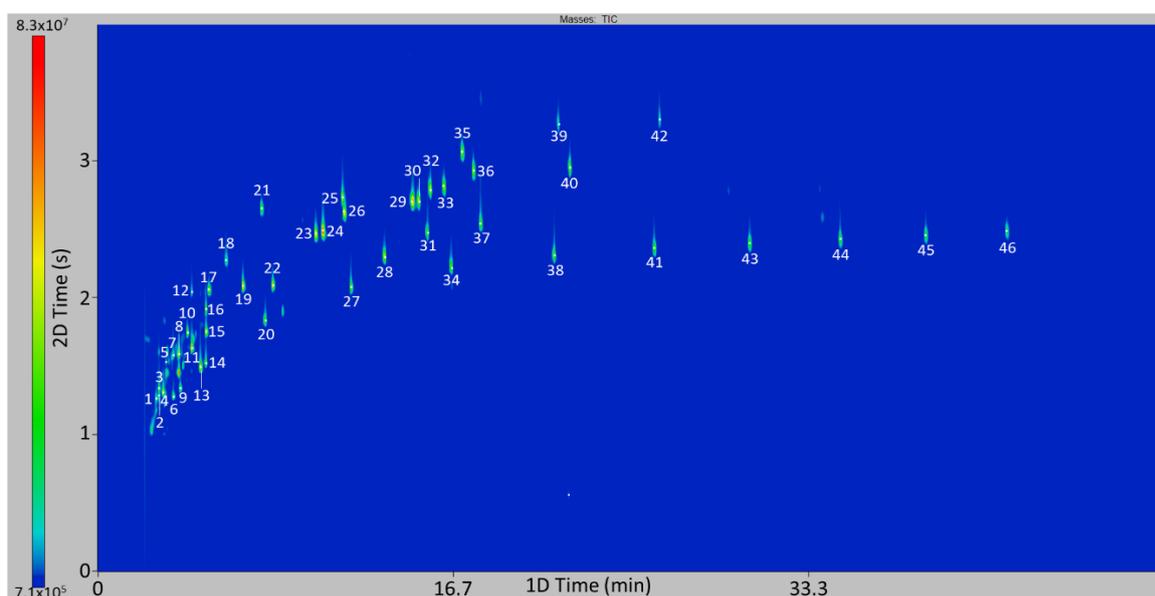
115 2.4. Accuracy Profile evaluation

116 For each calibration technique, accuracy profiles were built for each compound with a homemade
117 Excel 2019 (Microsoft, Redmond, USA) spreadsheet containing macros to avoid losing time on
118 repetitive calculation tasks.

119 3. Results and Discussion

120 3.1. Standard separation

121 The column set and the modulation period were set using the air indoor standard solution as a
122 reference. This reference standard contains 48 compounds with boiling point ranging from 40 to 287°C,
123 and various families of compounds with various polarities (*i.e.*, Alkanes, Aromatics, Alcohols, Ketones,
124 Aldehydes, Halogenated, Terpenes). After method development and optimization, all compounds
125 were separated except two pairs of isomers, m-xylene and p-xylene, and 1-ethyl-3-methylbenzene and
126 1-ethyl-4-methylbenzene. The 2D chromatogram of the standard mix is given in the Figure 2.

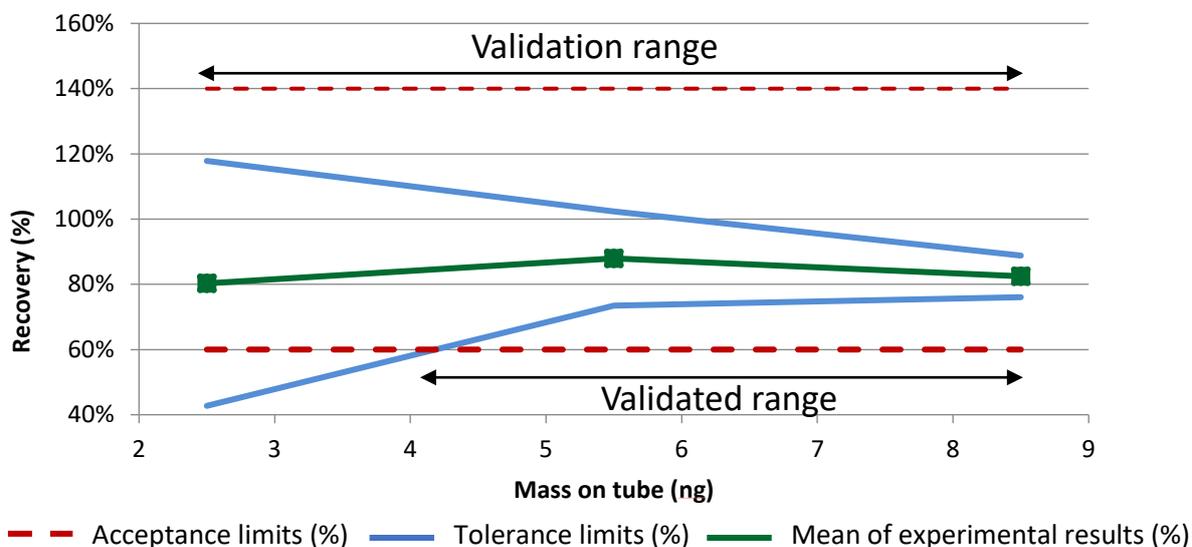


127
128 *Figure 2 - TD-GC×GC-MS Chromatogram of 48 VOC standard: 1 Ethanol; 2 Acetone; 3 2-Propanol; 4 Methylene Chloride; 5 1-*
129 *Propanol; 6 n-Hexane; 7 2-Butanone; 8 Chloroform; 9 2,4-Dimethylpentane; 10 1,2-Dichloroethene; 11 Benzene; 12 1-Butanol;*
130 *13 Isooctane; 14 Heptane; 15 Trichloroethene; 16 1,2-Dichloropropane; 17 Bromodichloromethane; 18 4-Methyl-2-pentanone;*
131 *19 Toluene; 20 Octane; 21 Dibromochloromethane; 22 Tetrachloroethene; 23 Ethylbenzene; 24 p/m-Xylene; 25 Styrene; 26 o-*
132 *Xylene; 27 Nonane; 28 α-Pinene; 29 1-Ethyl-3/4-methyl-benzene; 30 Mesitylene; 31 β-Pinene; 32 1-Ethyl-2-methyl-benzene;*
133 *33 1,2,4 Trimethylbenzene; 34 Decane; 35 1,4 Dichlorobenzene; 36 1,2,3-Trimethylbenzene; 37 Limonene; 38 Undecane; 39*
134 *Nonanal; 40 1,2,4,5 Tetramethylbenzene; 41 Dodecane; 42 Decanal; 43 Tridecane; 44 Tetradecane; 45 Pentadecane; 46*
135 *Hexadecane. Analytical conditions are described in the experimental part.*

136 3.2. Accuracy Profile determinations

137 In the present study, for each of the three calibration techniques, four series of calibration and
138 validation sample analyses were performed with various days and operators for each series. Each
139 series comprised four levels for calibration samples and three levels for validation samples. For each
140 level three tubes were used. As a consequence, one series represented 21 tubes. For each calibration
141 technique, accuracy profiles were determined individually for 46 compounds, since two critical pairs
142 were not separated m/p-xylene and 3/4-ethyltoluene. The acceptance limit (λ) was set at 40 %
143 considering the intrinsic variability of the TD-GC×GC-MS methods, and the tolerance limit (β) was set
144 at 80 %, which represents 4 future values out of 5 inside the tolerance interval in average. Then, the
145 validated range, corresponding to the zone where the tolerance interval was inside the acceptance

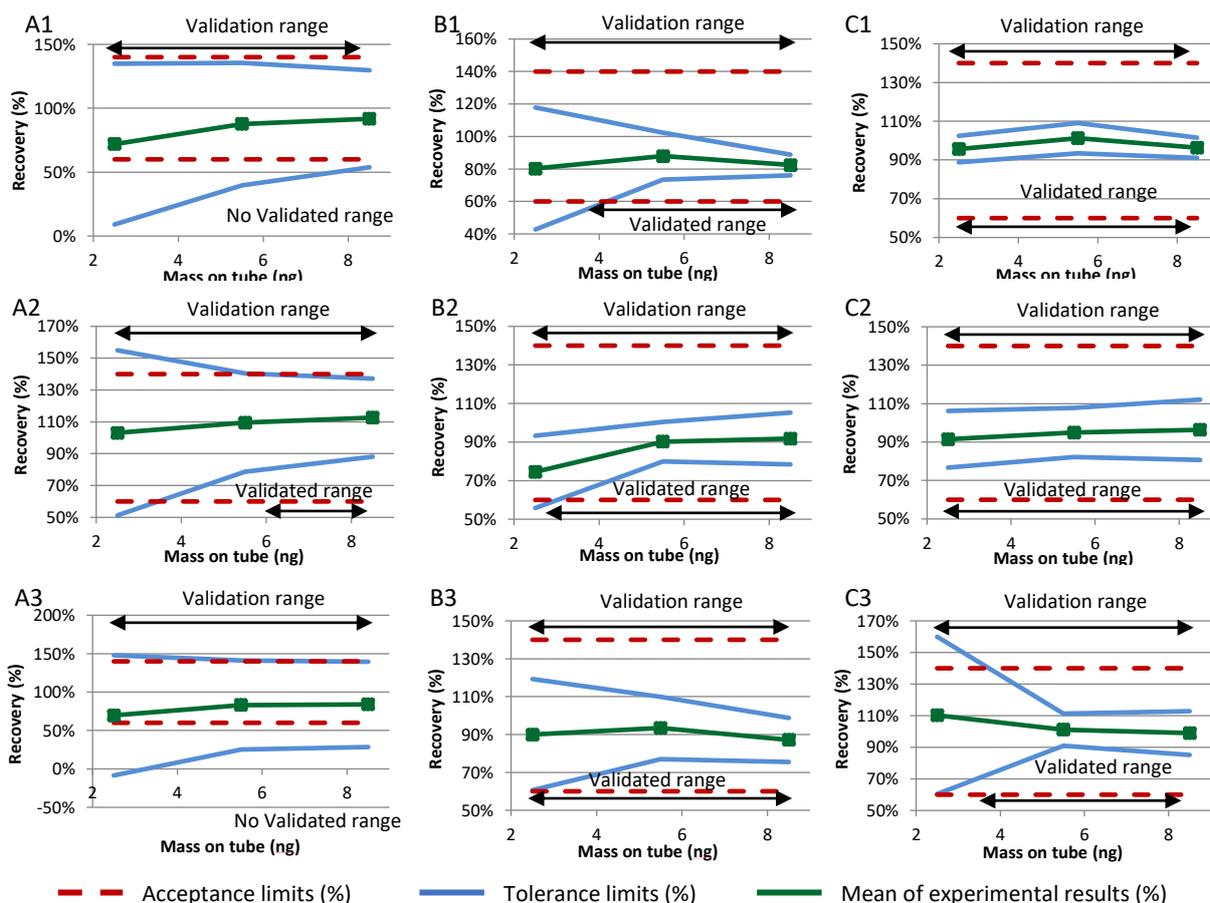
146 limits, was determined for each compound. The score obtained for this profile corresponded to the
147 percentage of the validated range by reference to the validation range (Figure 3).



148

149 *Figure 3 - Accuracy Profile example (Mesitylene (30)), validated range 72/100*

150 In a first instance, the comparison was possible between techniques for each compound. Different
151 situations could occur, with a validation over all the range of concentration or only for a part as
152 illustrated in Figure 4. The technique which presented a full validated range (score 100) for the highest
153 number of compounds was the VGSD with 24 compounds validated out of 46. This behavior is
154 illustrated for example by the case of mesitylene (Figure 4 A1-C1). However, for five compounds (1-
155 propanol, p-xylene, dodecane, tridecane and pentadecane) the validated range was larger for the
156 GSAD technique than for the VGSD as illustrated by propan-1-ol, Figure 4 A3-C3. For the MD, there was
157 only a partial validation for 9 compounds as illustrated by hexadecane, Figure 4 A2-C2.



158

— Acceptance limits (%) — Tolerance limits (%) — Mean of experimental results (%)

159

Figure 4 – Examples of Accuracy Profiles with the corresponding validation score for Mesitylene (30), A1 MD score 0, B1 GSAD score 72, C1 VGSD score 100; Hexadecane (46), A2 MD score 45, B2 GSAD score 91, C2 VGSD score 100; 1-Propanol (5), A3 MD score 0, B3 GSAD score 100, C3 VGSD score 80

160

161

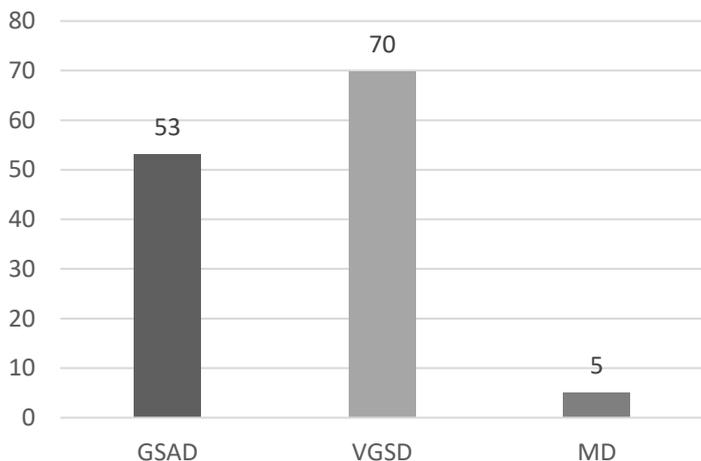
162 It can also be noted that 26 compounds out of 46 had a fully validated range in at least one technique;
 163 among these compounds, seven presented a 100 score both in the VGSD and the GSAD techniques
 164 (1,2 dichloro-ethane, Trichloroethane, Bromodichloromethane, Dibromochloromethane,
 165 Tetrachloroethylene, 1-ethyl-4-methylbenzene, 1,4-dichlorobenzene). These compounds belonged to
 166 the halogenated family meaning that this type of compounds seemed to be well deposited when there
 167 was a gas stream.

168

3.3. Score calculations

169

After determining the validated range for each compound (Percentage of validated range are
 170 presented for each compound in Table S1), global mean scores, over all the compounds, were
 171 calculated for each technique according to the methodology proposed by Remy *et al.* [20] A
 172 comparison of the global scores between the different calibration techniques is presented in Figure 5.

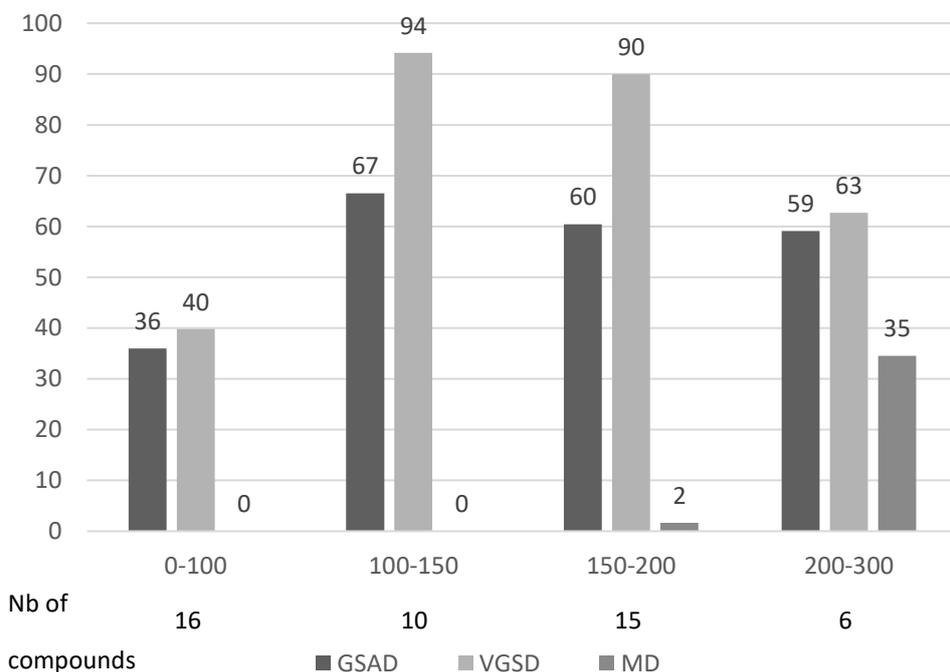


173

174 *Figure 5 - Global score of the three calibration techniques*

175 VGSD had the highest mean score of validation with 70 %. The addition of a gas stream on the MD (*i.e.*,
 176 GSAD) allowed the validation range to rise from 5 to 53 %. This meant that a simple addition of gas
 177 stream can help the deposition of compounds compared to the classic MD.

178 Then, compounds were classified according to their range of boiling temperature (16 compounds
 179 between 0 and 100 °C, 10 between 100 and 150°C, 15 between 150 and 200 °C, 6 between 200 and
 180 300 °C) and mean scores were calculated for each class and each technique (Figure 6).



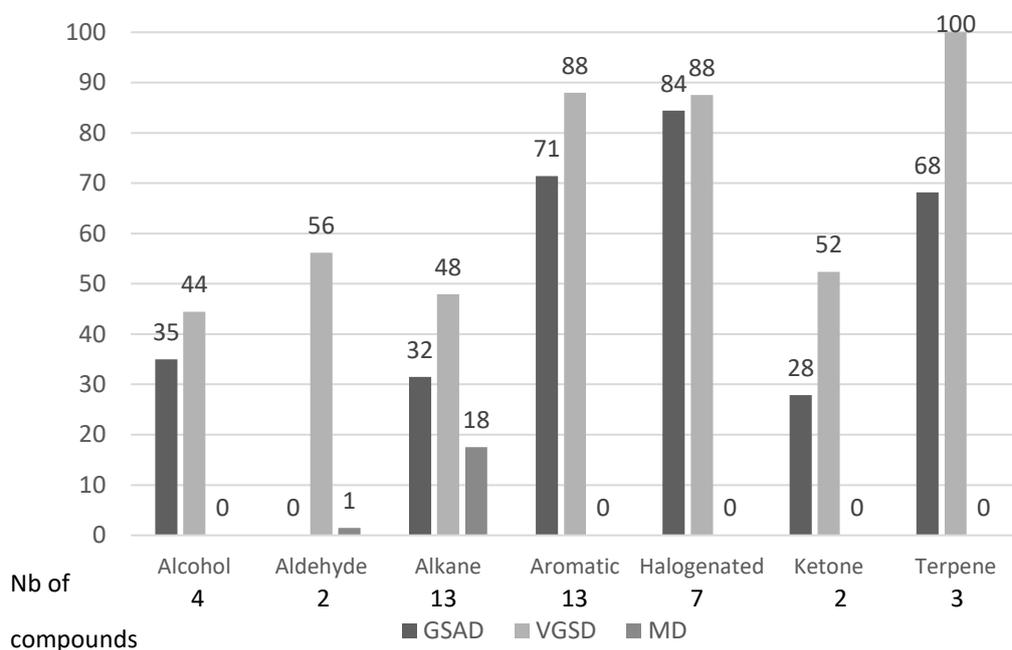
181

182 *Figure 6 - Score by boiling temperature range*

183 An observation could be made that the VGSD had its higher validation results (mean validation score
 184 of 94 and 90 %) with compounds having a boiling temperature close to the temperature of the VGSD
 185 apparatus, 140°C. For compounds having boiling temperature below 100 °C or higher than 200 °C the
 186 VGSD and the GSAD had similar mean validation scores. It can also be noted that the MD had mean
 187 validation score of 35 %, its highest score, only for compounds having a boiling point higher than 200°C.

188

189 Results were also studied by chemical families (*i.e.*, Alcohol, Aldehyde, Alkane, Aromatic, Halogenated,
190 Ketone, Terpene) and their mean validation score were calculated (Figure 7).



191

192 *Figure 7 - Score by chemical family*

193 In almost every family, the mean scores were slightly higher for VGSD than for GSAD, except for
194 aldehydes where there was no validated range for GSAD. The MD only provided a validation score for
195 the alkanes having more than 11 carbon atoms. One should notice that the aldehyde family was only
196 constituted of high boiling point molecules (nonanal, decanal), which can explain why there was no
197 validated range for GSAD.

198 Despite being the most efficient technique, VGSD presents two main drawbacks compared to the two
199 other techniques, i) the vaporization chamber needs to be heated 1 hour before experiments, ii) Due
200 to all three, the heating of the chamber, the presence of a septum, and the need for cleaning the
201 chamber, there is a risk of pollution and carry over (examples are provided in supplementary material
202 Figure S1 to S3), which involves more regular monitoring and maintenance. So, the GSAD technique
203 appeared as the best compromise in terms of reliability and time investment to prepare standard tubes
204 for the calibration in TD-GC-MS.

205 4. Conclusion

206 Gas stream assistance implemented in the GSAD significantly improved the performance of MD with
207 global scores of respectively 53 and 5 % on mean validation range. From a practical point of view, it
208 appeared to be the best compromise for a lab who needs to implement quantitative analysis in TD-GC-
209 MS.

210 Using the accuracy profile evaluation, VGSD has shown to be the most efficient among the tested
211 techniques for the preparation of standards for quantitative analysis in TD-GCxGC-TOFMS in term of
212 accuracy, with a global score of 70 % on mean validation range.

213 MD should be avoided if accurate quantification is required. Its use must be restricted to qualitative
214 analysis.

215 Some families like the most polar amine, acid, were not represented in the standard mixture.
216 Complementary tests should be performed in order to investigate the behavior of these more polar
217 compounds. Possible future developments are an automatization of the GSAD, or the use of preheated
218 GSAD without vaporization.

219 This study showed, one more time, the possibility to use accuracy profile methodology as a powerful
220 tool for the comparison of quantitative performances of analytical methods.

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