Supplementary Information

Surface Acoustic Wave sensing of linear alcohols using *para*-acylcalix[n]arenes

Steven Revel,[a] Ludovic Doudet,[a] Ludovic Alonsoa and Anthony W. Coleman[*a] and Patrick Shahgaldian[b]

[a] LMI UMR 5615, Universite de Lyon 1, Villeurbanne, F69622 France. Tel : +33 4 7243 1027; E-mail: antony.coleman@adm.univ-lyon1.fr

[b] University of Applied Science Northwestern Switzerland, Department of Chemistry and Bioanalytics, Gründenstrasse 40, Muttenz, CH-4132, Switzerland.; E-mail:Patrick.shahgaldian@fhnw.ch

Synthesis

The three *para*-acylcalix[n]arenes were prepared according the literature, *para*-acylcalix[4]arene, S1, *para*-acylcalix[6]arene, S2, and *para*-acylcalix[8]arene, S3. All physical data, mpt, ^{1}H and ^{13}C NMR, mass spectra, are in full agreement with those published.

Surface Acoustic Wave detector

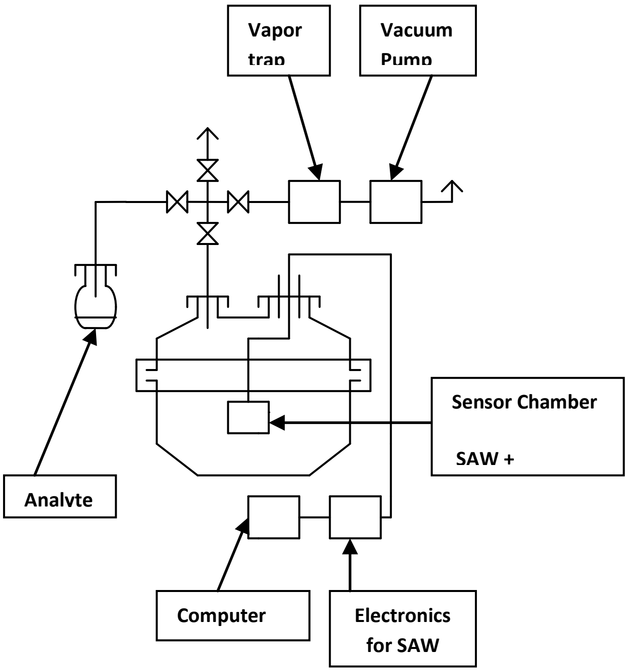
The Surface Acoustic Wave sensor platform was purchased from SenSeor S.A., Besancon, as were the dual channel detector chips.

The active sensing layer of **1**, **2** or **3** was deposited on only one of the chambers in the sensor chip, the other one being used as the reference. To prepare the sensing layer 2 mgs of the compounds were dissolved in 2mL of chloroform and then 1 µL was placed on the sensor area and allowed to evaporate.

The sensing layers are robust and can be used over 10 cycles, the chips can be cleaned by washing with tetrahydrofuran.

Vapour Handling System

The vapour handling system was built at the Laboratoire de Multimatériaux et Interfaces, UMR 5615 Université de Lyon 1. The system is shown in the Scheme 1, below.



Scheme S1 Representation of the vapour handling system

The vapours are applied using vacuum-vapour cycles, the timing of the cycles is dependent on the arrival at a constant value both for the vacuum and vapour cycles.

Atomic Force Microscopy

Samples for AFM were prepared by depositing a solution, at the same concentration as for the sensing experiments, of the relevant calix[n]arene onto a flat gold surface.

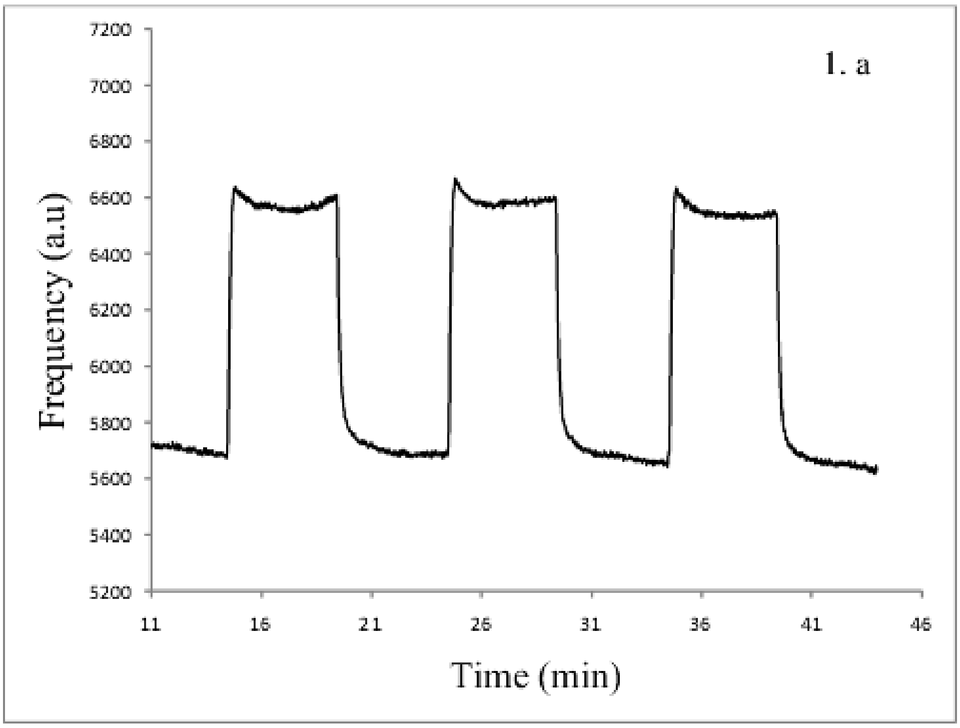
AFM imaging was carried out with a NT-MDT (Moscow, Russia) NTegra system using Silicon NSG10 (NT-MDT) tips
(cantilever length 95 um, width 30 um, force constant 3-37 N/m, resonant frequency: 200 kHz)
Imaging was carried out in non-contact mode at a scanning speed of 1 Hz.

Results

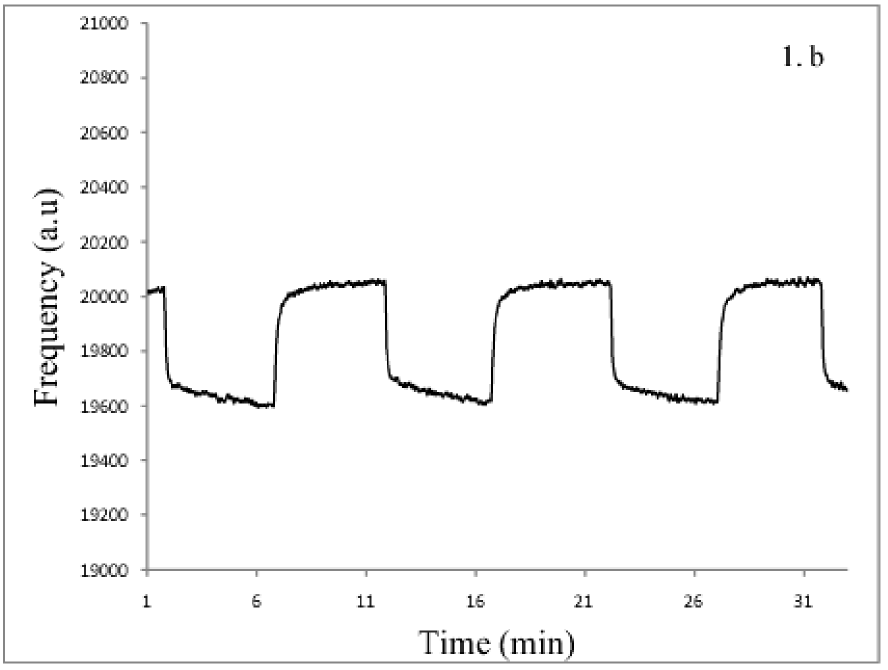
The sensing curves are given below in Figures S1, S2 and S3. A=methanol, b=ethanol, c=propanol, e=butanol, f=pentanol, h=hexanol, j=decanol

C6C4

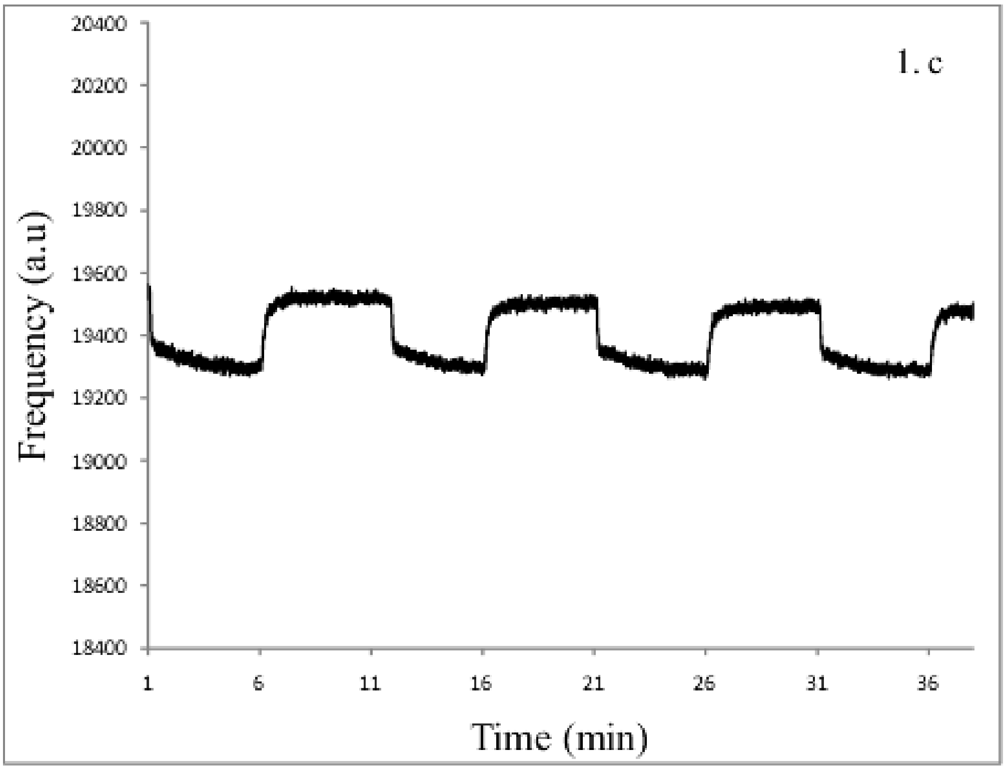
Methanol



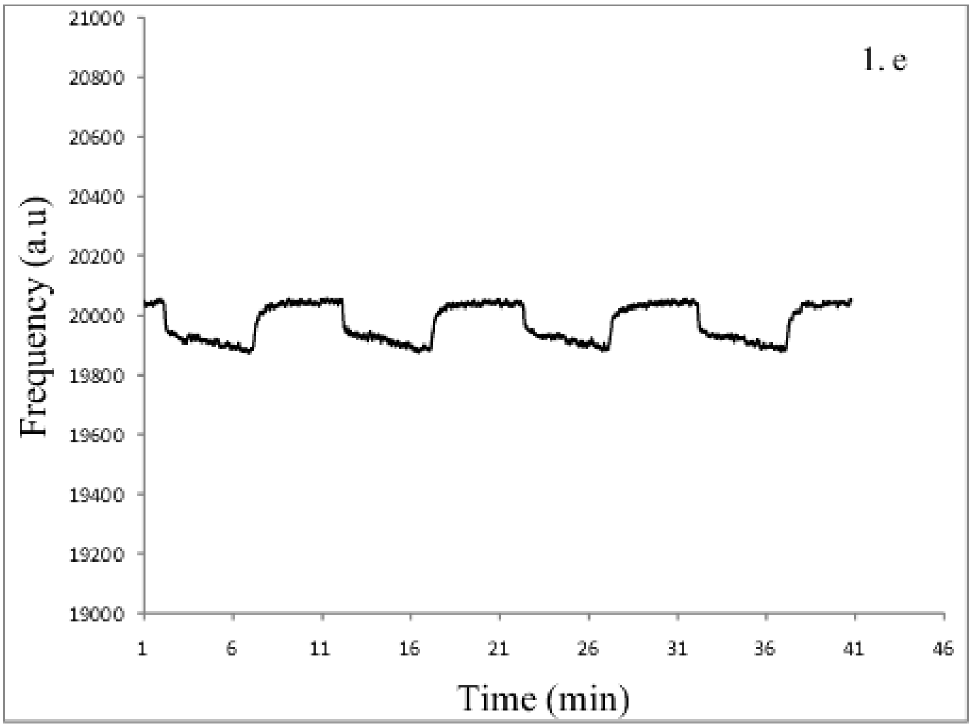
Ethanol


1. b
Frequency (a.u)
21000
20800
20600
20400
20200
20000
19800
19600
19400
19200
19000
1
6
11
16
21
26
31
Time (min)

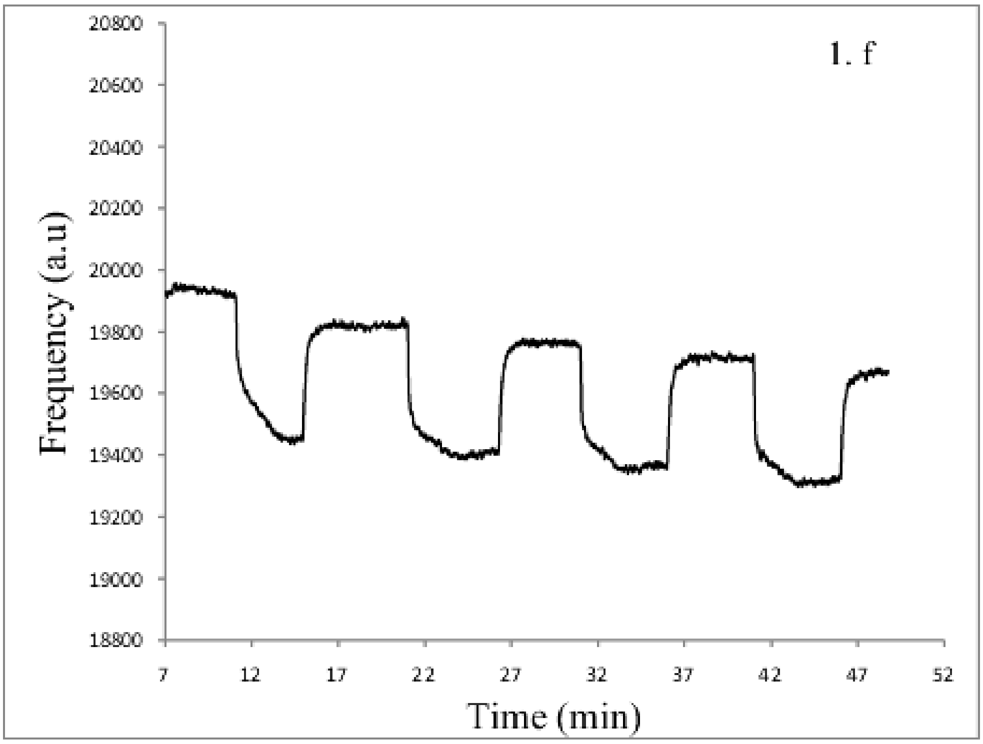
Propan-1-ol



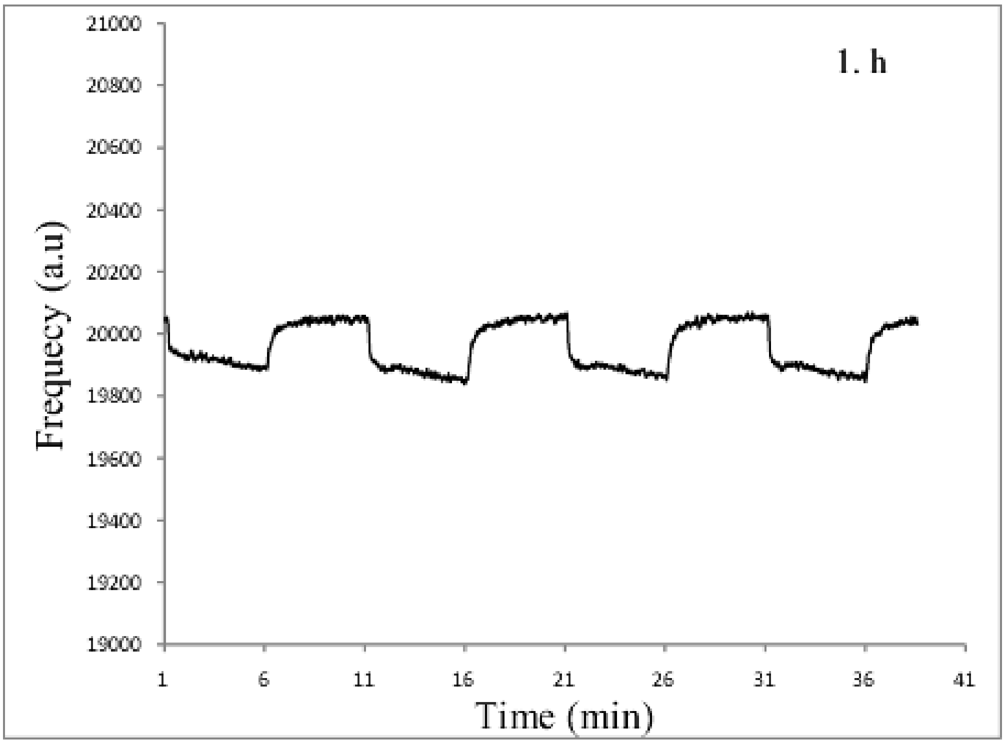
Butan-1-ol



Pentan-1-ol



Hexan-1-ol



Electronic Supplementary Material (ESI) for Chemical Communications

Decan-1-ol

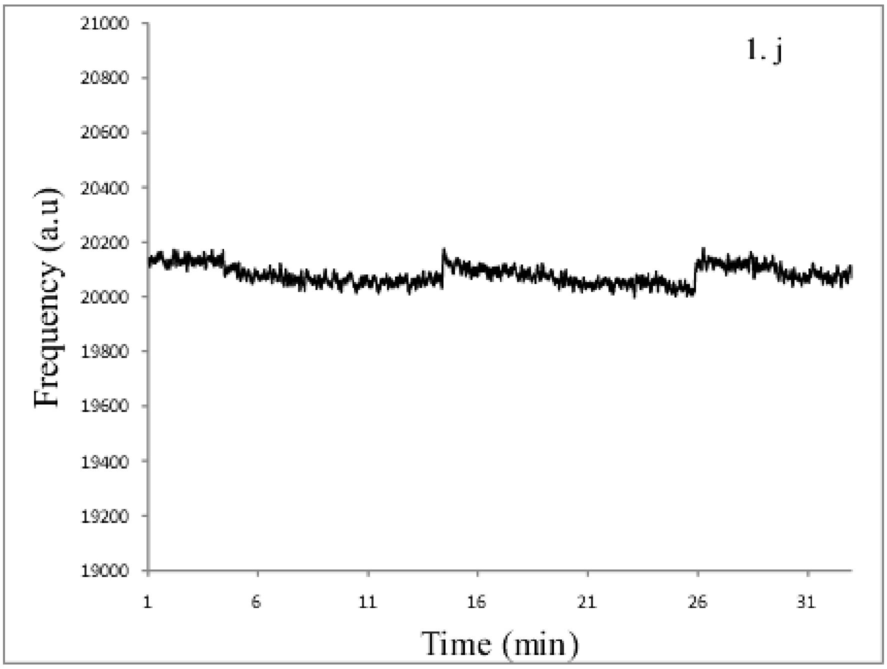
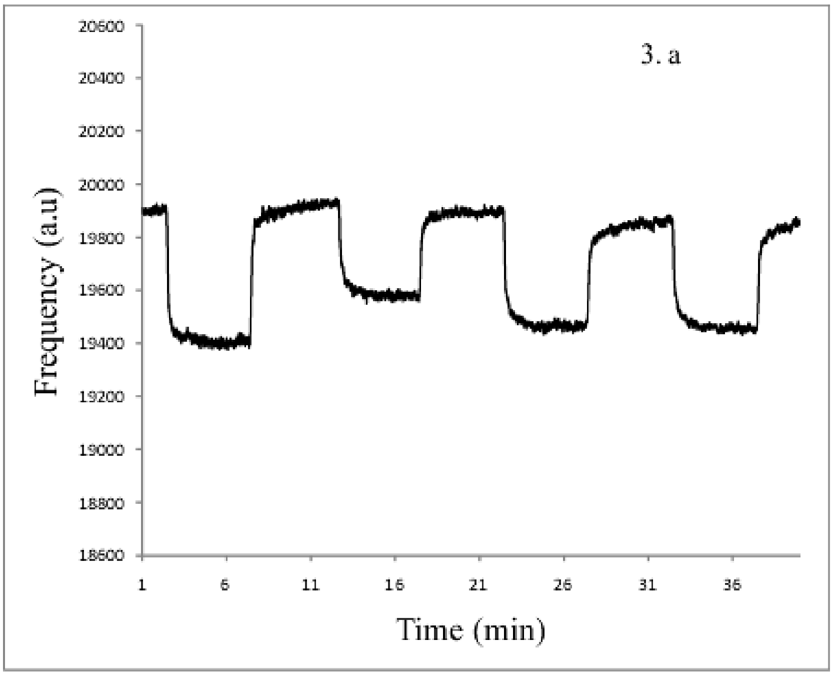
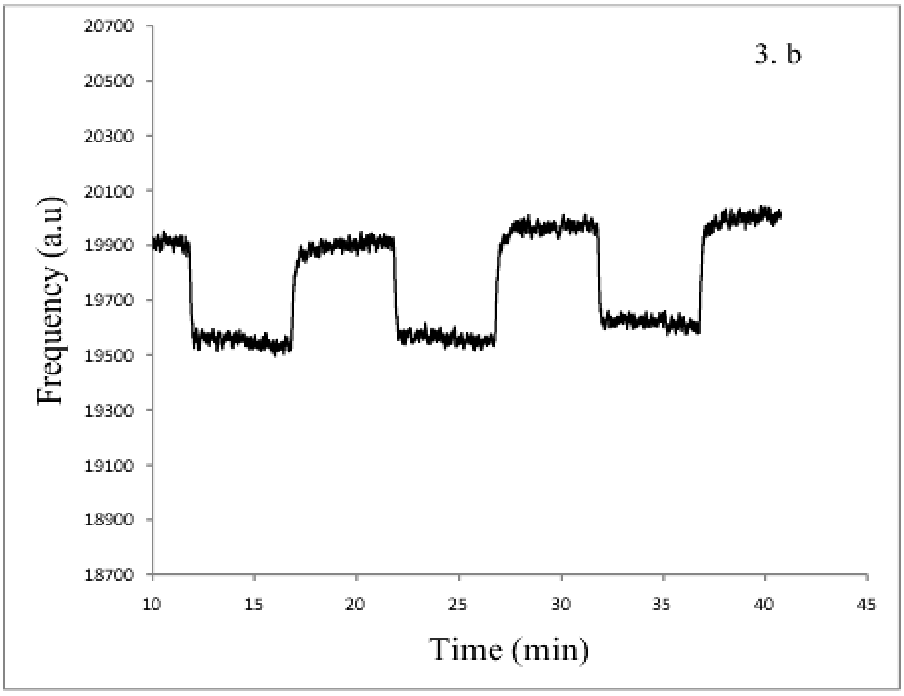


Figure S1 Response curves for a C6C4 capture layer

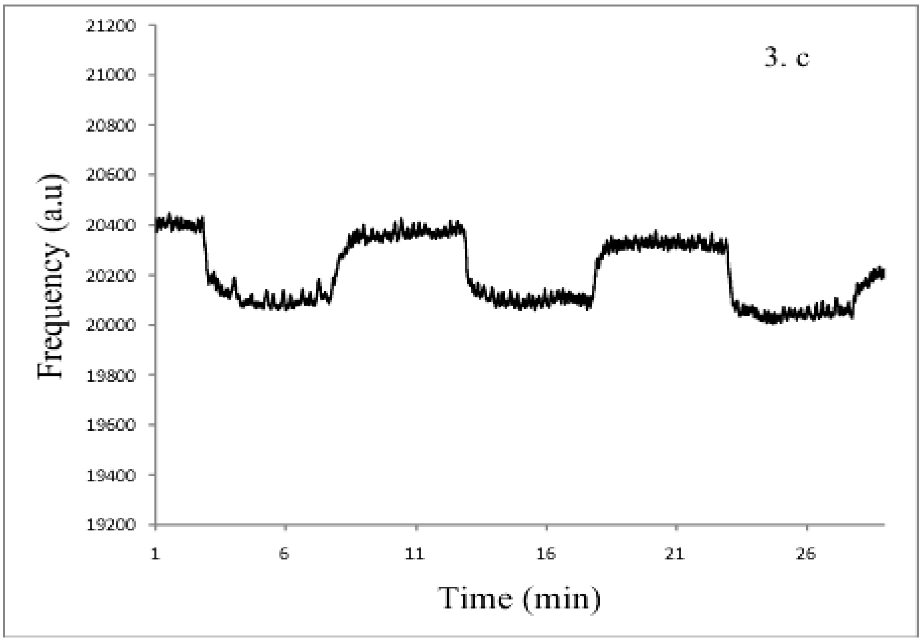
Methanol



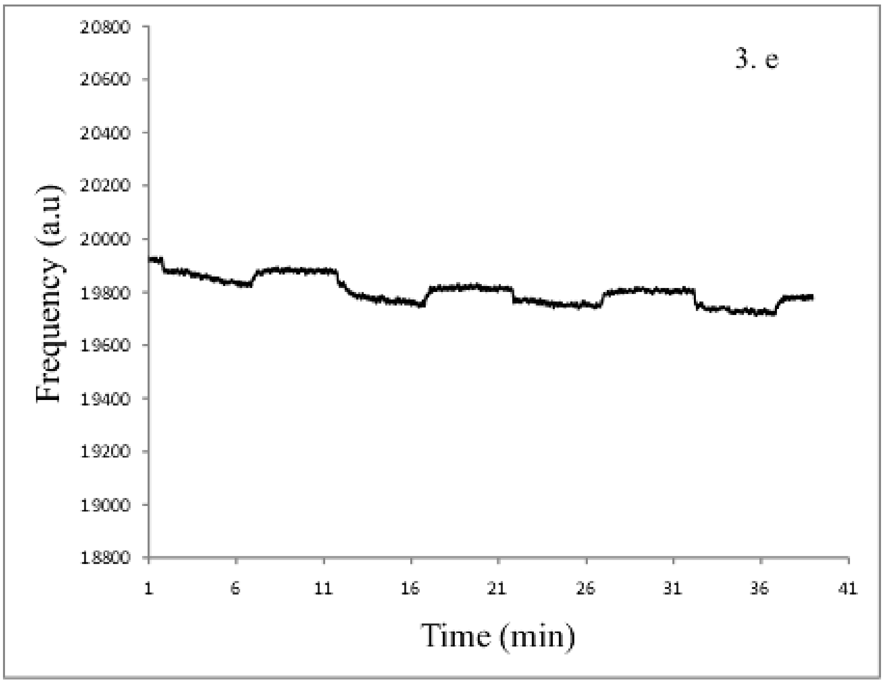
Ethanol



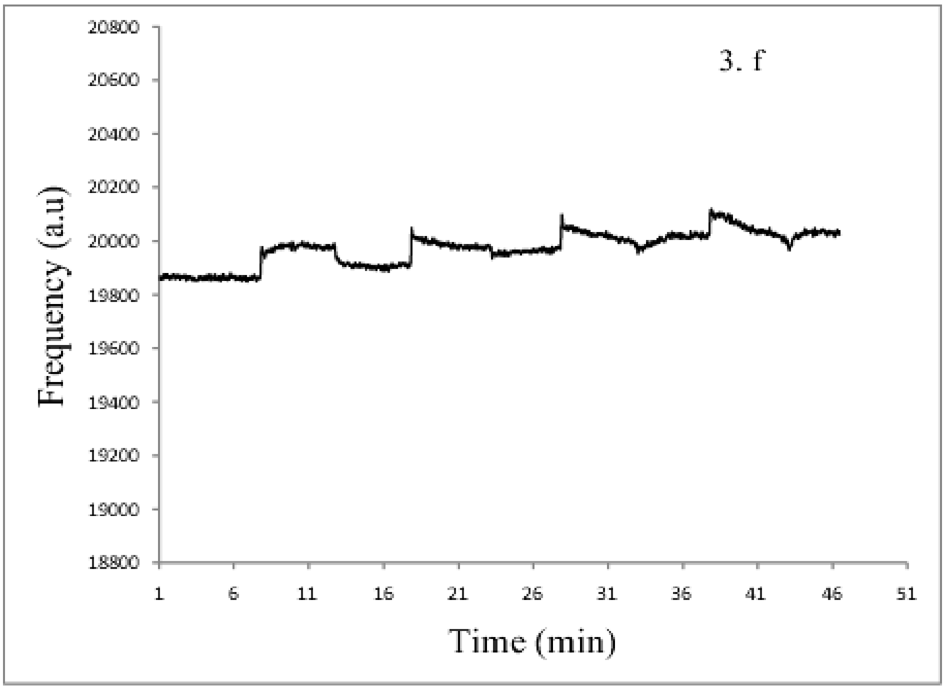
Propan-1-ol



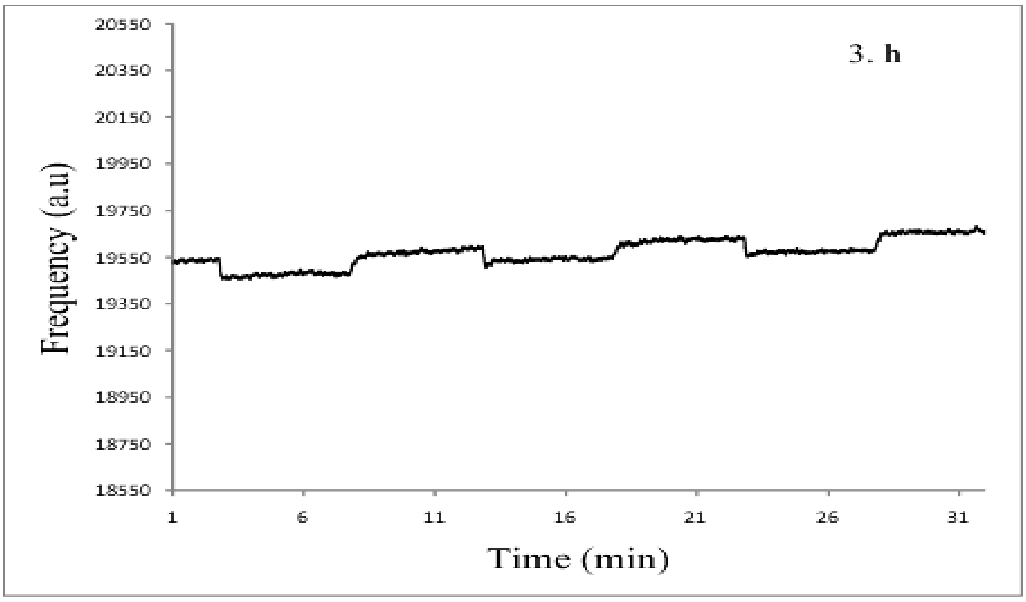
Butan-1-ol



Pentan-1-ol



Hexan-1-ol



Decan-1-ol

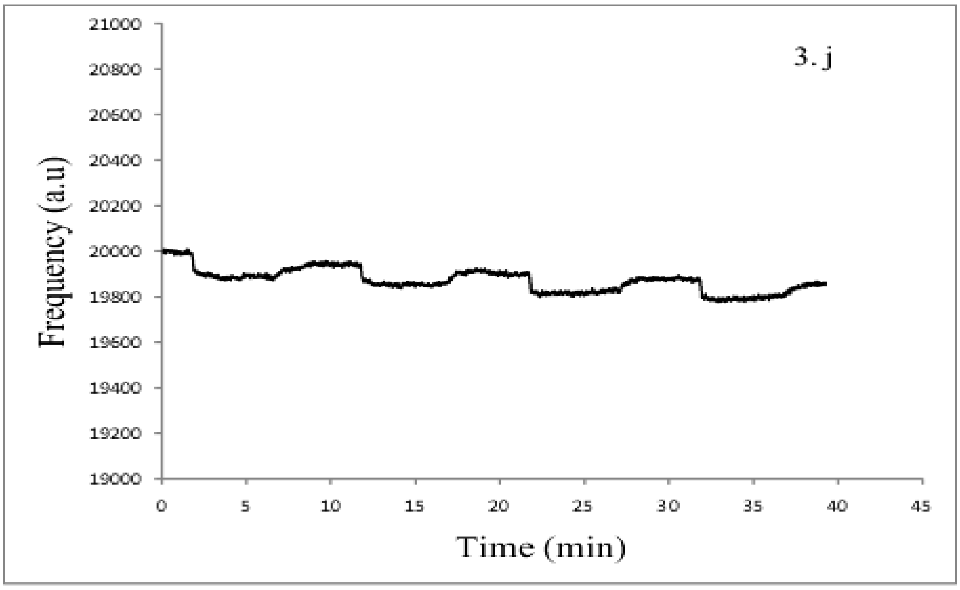
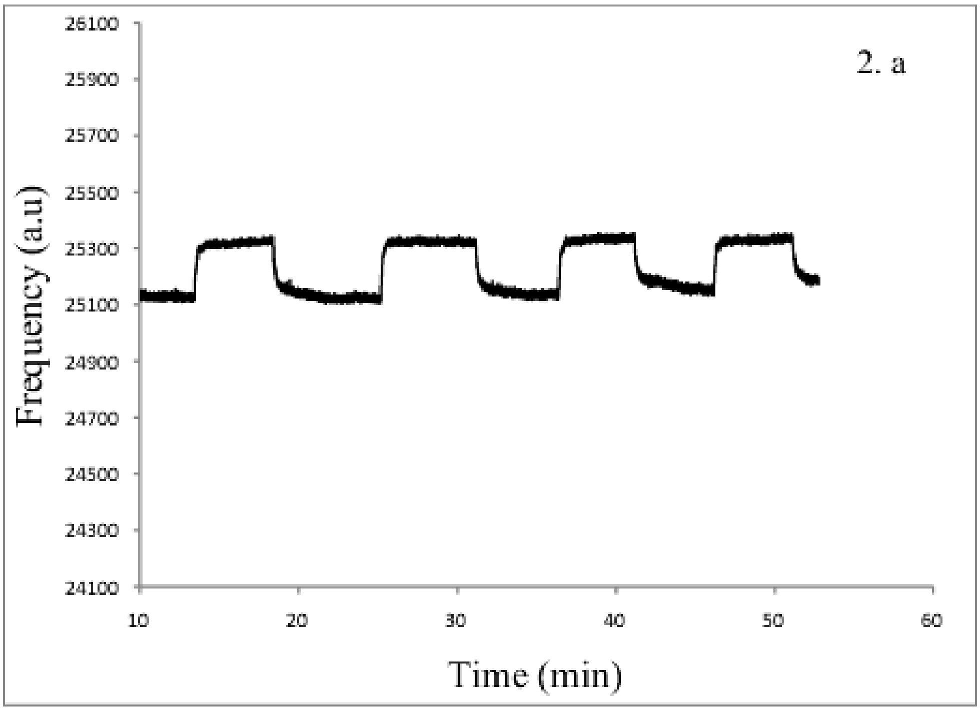
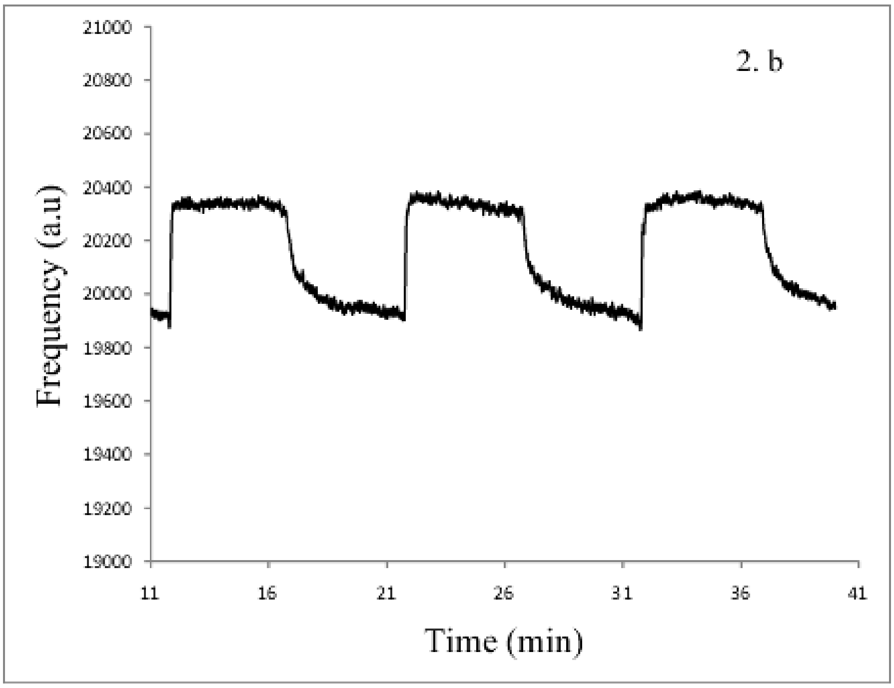


Figure S2 Response curves for a C6C6 capture layer

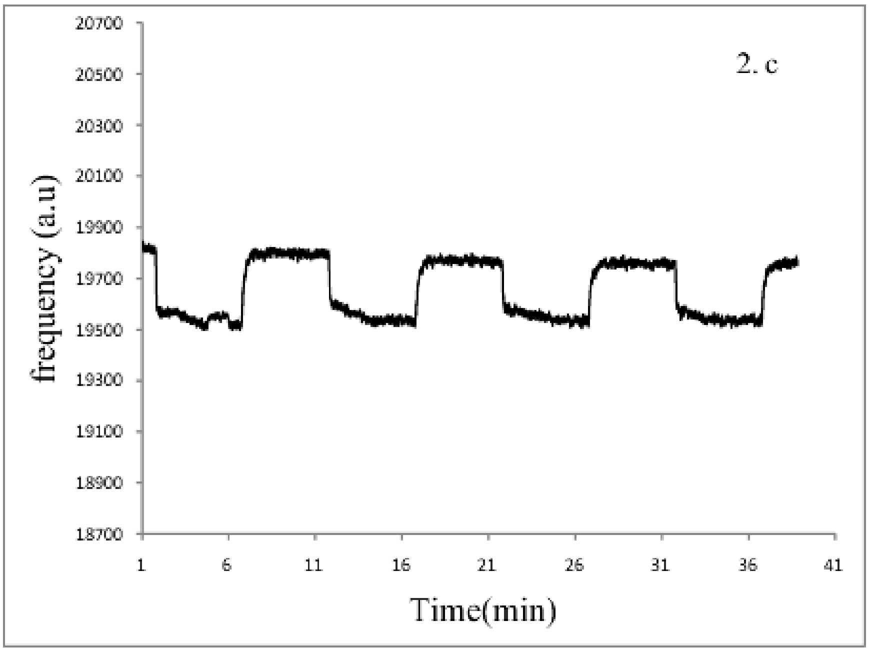
Methanol



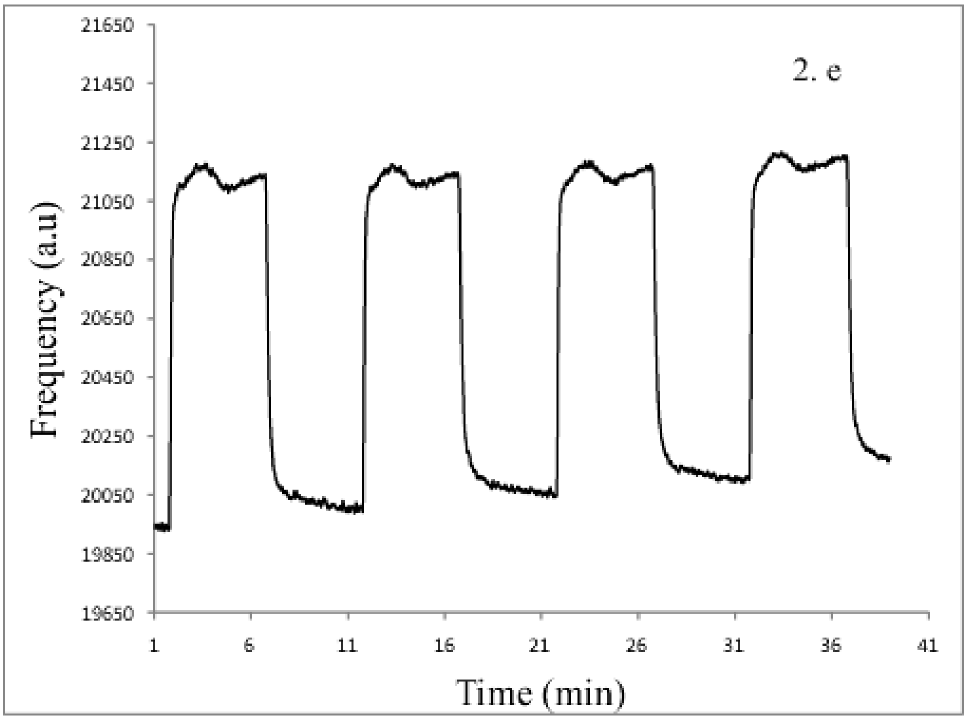
Ethanol



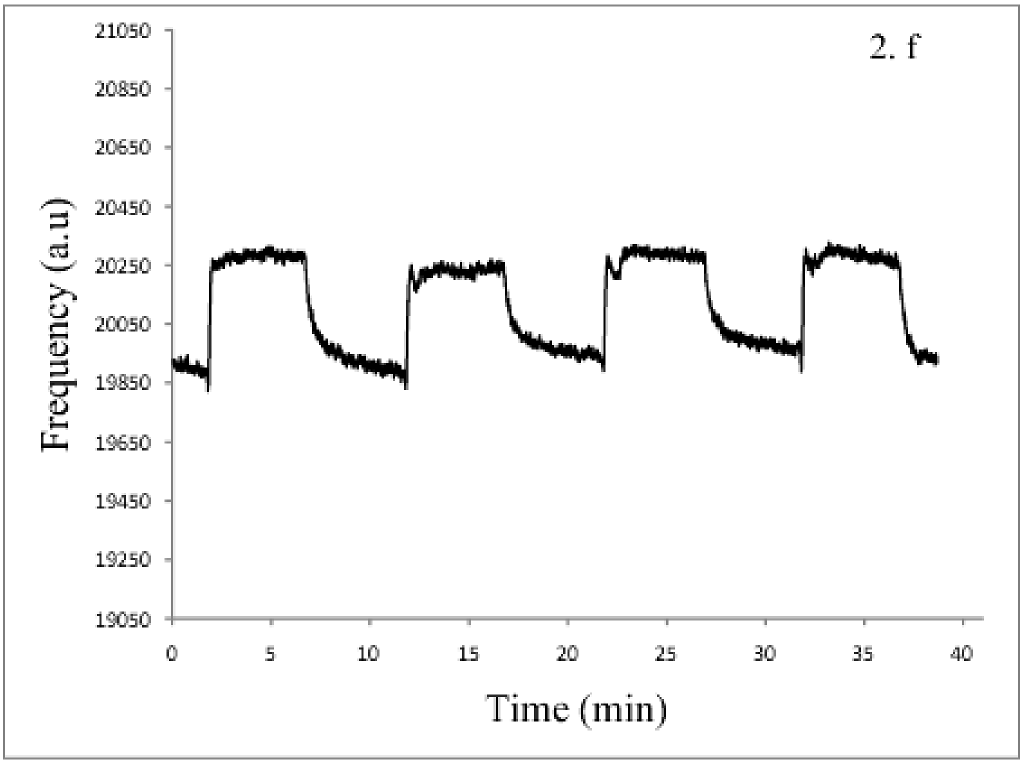
Propan-1-ol



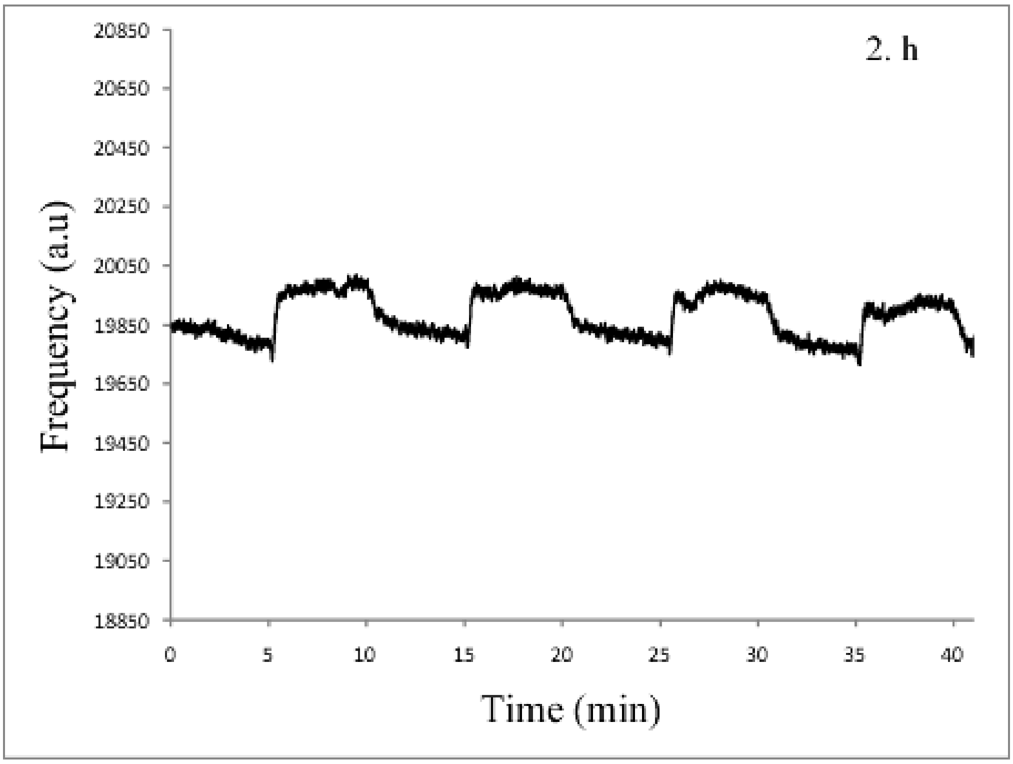
Butan-1-ol



Pentan-1-ol



Hexan-1-ol



Decan-1-ol

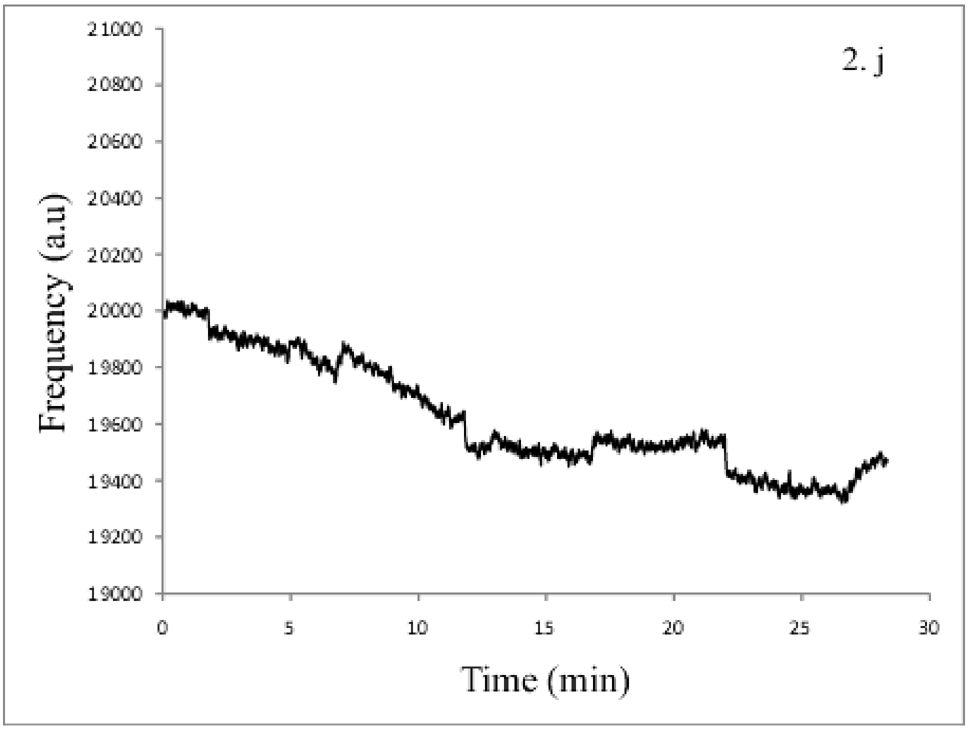
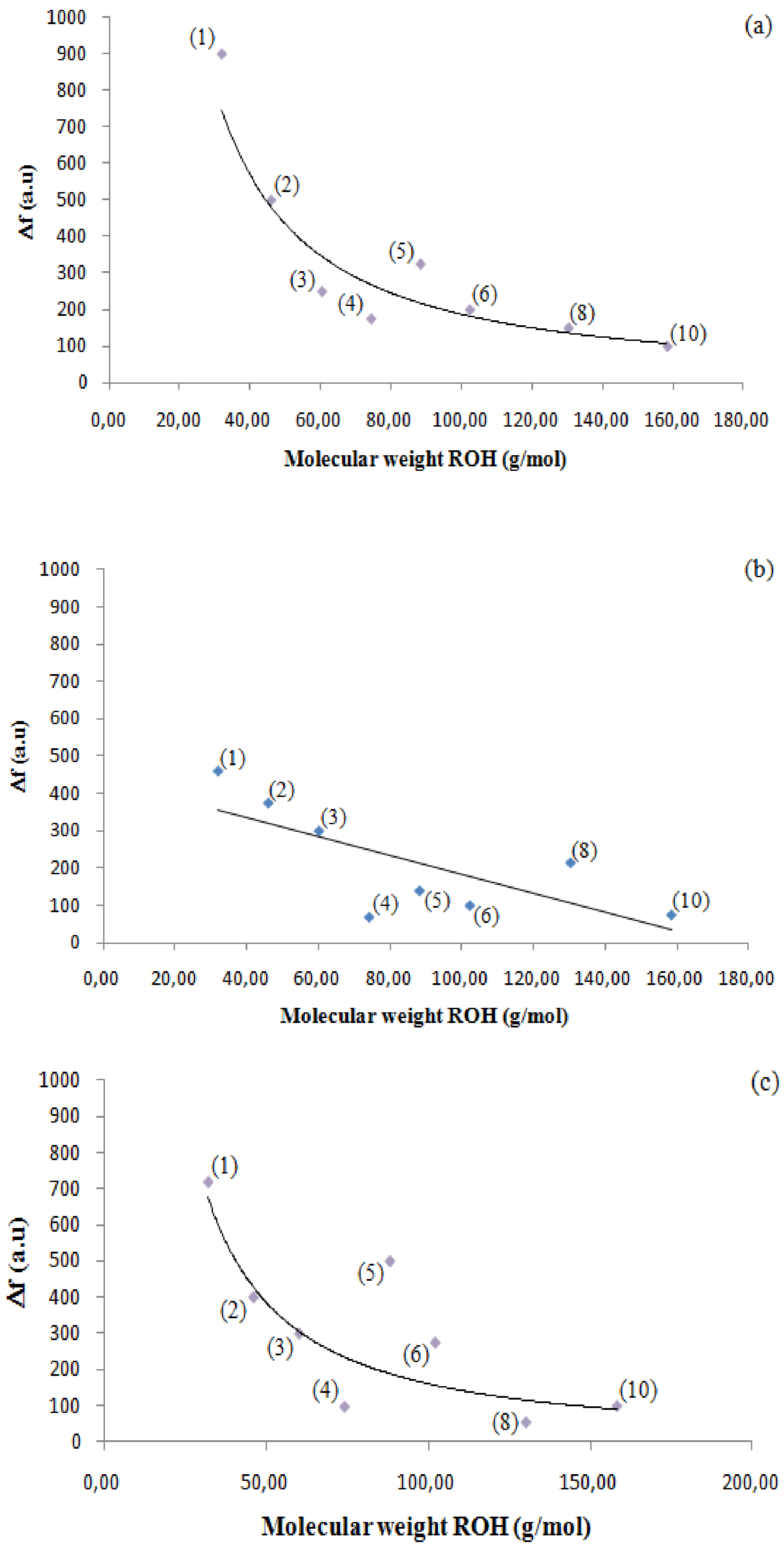


Figure S3 Response curves for a C6C8 capture layer



Figiure S4 Response versus Molecular mass, a) for a C6C4 capture layer, b) for a C6C6 capture layer, c) foe a C6C8 capture layer, the numbers on the curve refer to the alkyl chain length.

Data treatment

The data obtained was treated using Excel 2010, in particular the noise in the data was removed using the curve smoothing option in the graphics interface.

References

S1 Shahgaldian, P.; Coleman, A. W.; Kalchenko, V. I. **Synthesis and properties of novel amphiphilic calix-[4]-arene derivatives.** Tetrahedron Letters (2001), 42(4), 577-579.

S2 Said Jebors, Barbara Leśniewska, Oleksandr Shkurenko, Kinga Suwińska and Anthony W. Coleman, **Para-acylcalix[6]arenes: their synthesis, per-O-functionalisation,solid-state structures and interfacial assembly properties,** J. Inclu. Phenom, (2010), 68, 207-17

S3 Said Jebors, Fabienne Fache, Sylvain Balme, Floriane Devoge, Melany Monachino, Sebastien Cecillon and A. W. Coleman, **Designer amphiphiles based on *para*-acyl-calix[8]arenes**, Org. Biomol. Chem., 2008, **6**, 319 - 329