Report ref.	REPGET22-010	Center of analysis	Biodesiv
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Category	External		67200 Strasbourg
			FRANCE
Subject	Performance assessment: Getxent tubes impregnated with dead body odor		

Background

The purpose of this paper is to evaluate the:

- ability of the Getxent tubes to capture and release the odor of dead body
- identify some specific VOC from dead body
- evaluate the evolution of concentration at two impregnation durations: 24h and 72h

Identification of samples

- 1 empty vial
- 1 non impregnated Getxent tube
- 3 samples of Getxent tubes impregnated with a piece of dead body (hand) for 24h
- 3 samples of Getxent tubes impregnated with a piece of dead body (hand) for 72h

Sample preparation

The samples, the non-impregnated Getxent tube and the empty vial have been directly analyzed.

Analysis - Procedure

All samples were analyzed by SPME GC-MS on Getxent GC-MS with method "Tube40RD v2" (see Annex 1).
One empty vial has been analyzed in between each duplicate.

Only peaks integrated by the software are considered.
The number of peaks and the identification of the compounds have been generated by the NIST database. Only identifications with a probability of more than 75% were considered.

For analysis with 40°C extraction temperature, the baseline of the chromatograms has been corrected by the software to focus on main molecules.
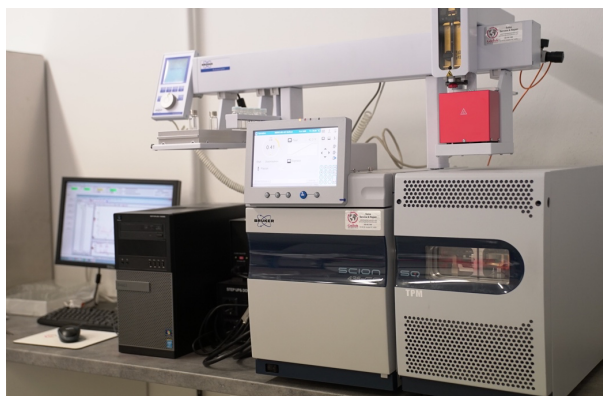
Analysis - Machine

Brand: Scion

Model: 436-GC, single quadrupole, injector split/splitless

Accessories:

- SPME
- Incubator
- Combi-PAL
- Column: DB-WAX 60m, 0.25mm, 0.25µm (Agilent)
- SPME fiber: DVB/CAR/PDMS 2cm (Sigma-Aldrich 57299-U)

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Analysis - Consumables

Gas: 6.0 quality Helium filtered over an Oxygen, Moisture & Hydrocarbon Trap (Restek, RE21982).

Vials:

- Body: clear glass
- Cap: magnetic metal cap with hole + septum PTFE/Silicone
- Volume: 20ml
- Closure diameter: ND18
- Height: 75mm

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Results

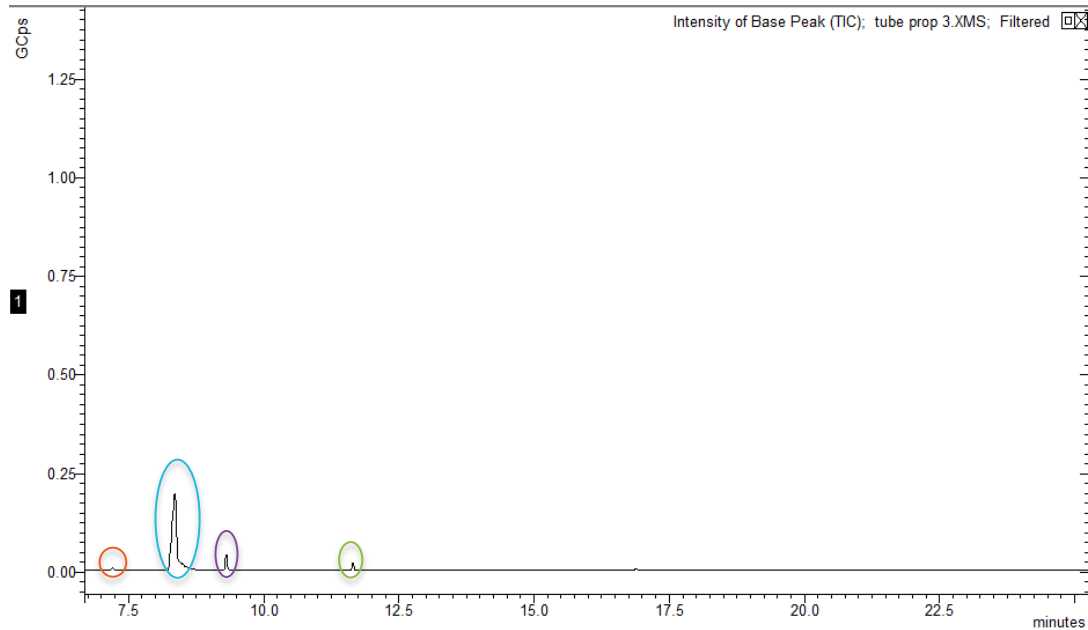The analytical cleanliness of a not impregnated Getxent tube has been checked (see Figure 1).

Figure 1: Chromatograms of not impregnated Getxent tube

The chromatogram of the not impregnated Getxent tube shows 4 peaks:
- 7,20 min: octamethylcyclotetrasiloxane
- 8,30 min: water
- 9,30 min: decamethylcyclopentasiloxane
- 11,60 min: dodecamethylcyclohexasiloxane

Same peaks are also found when the analysis is performed with blank vials, without any sample. These signals come from the SPME fiber which is composed of siloxanes. The Getxent tube not impregnated is therefore clean.

The odor profile of a Getxent tube impregnated with a piece of dead body (more specifically a hand) is shown in Figure 2 and compared at two different impregnation durations: 24h and 72h.

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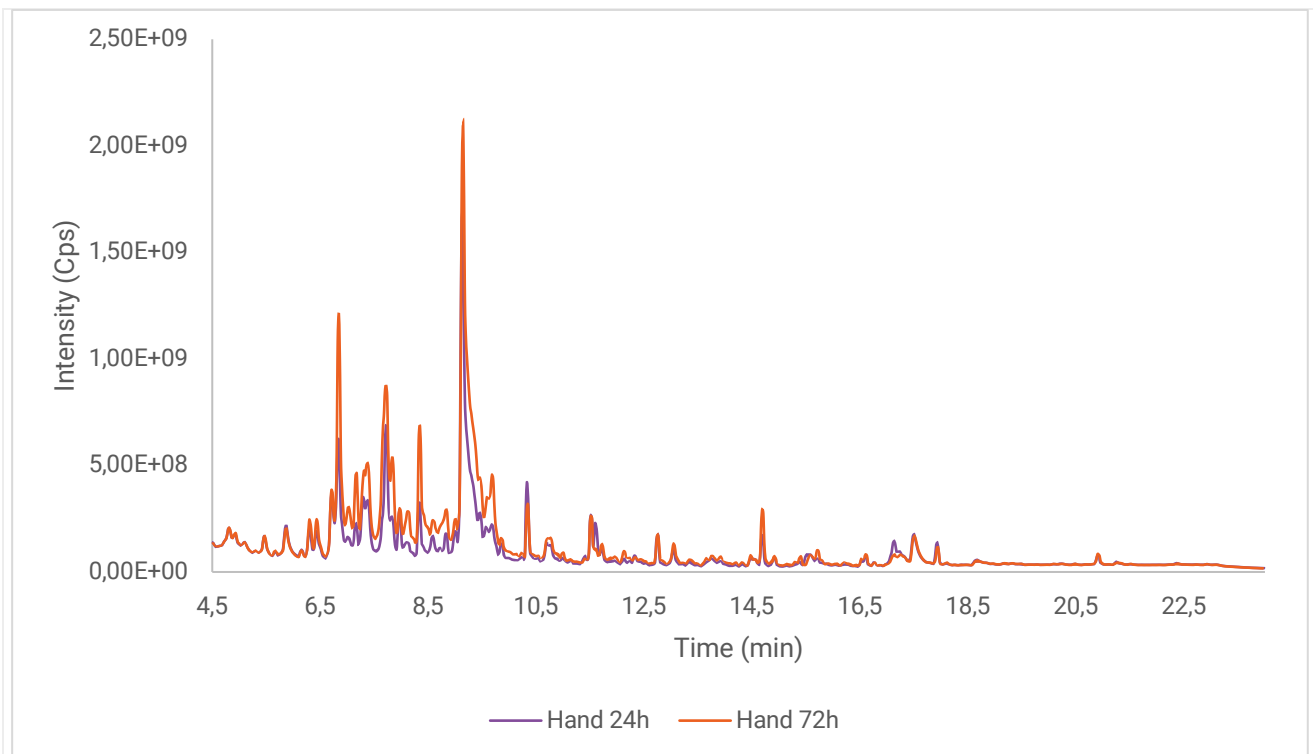
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Figure 2: GC-MS chromatograms of a Getxent tube impregnated 24h (purple) and 72h (orange)

On each chromatogram, 167 peaks have been detected by the NIST database.

7 compounds have been identified:

Retention time (min)	Compound	Probability (%)
5,85	Acetone	85,4
6,69	Ethyl acetate	90,2
9,67	Disulfide, dimethyl	97,5
11,95	Eucalyptol	77,6
14,68	Ethanol, 2-butoxy	79
15,53	acetic acid	80,3
15,78	Pyrazine, tetramethyl	90

Figure 3: VOCs identified by the NIST database

Their area (proportional to the concentration of molecules released by the impregnated Getxent tubes) are plotted in Figure 3, as a function of time of impregnation.
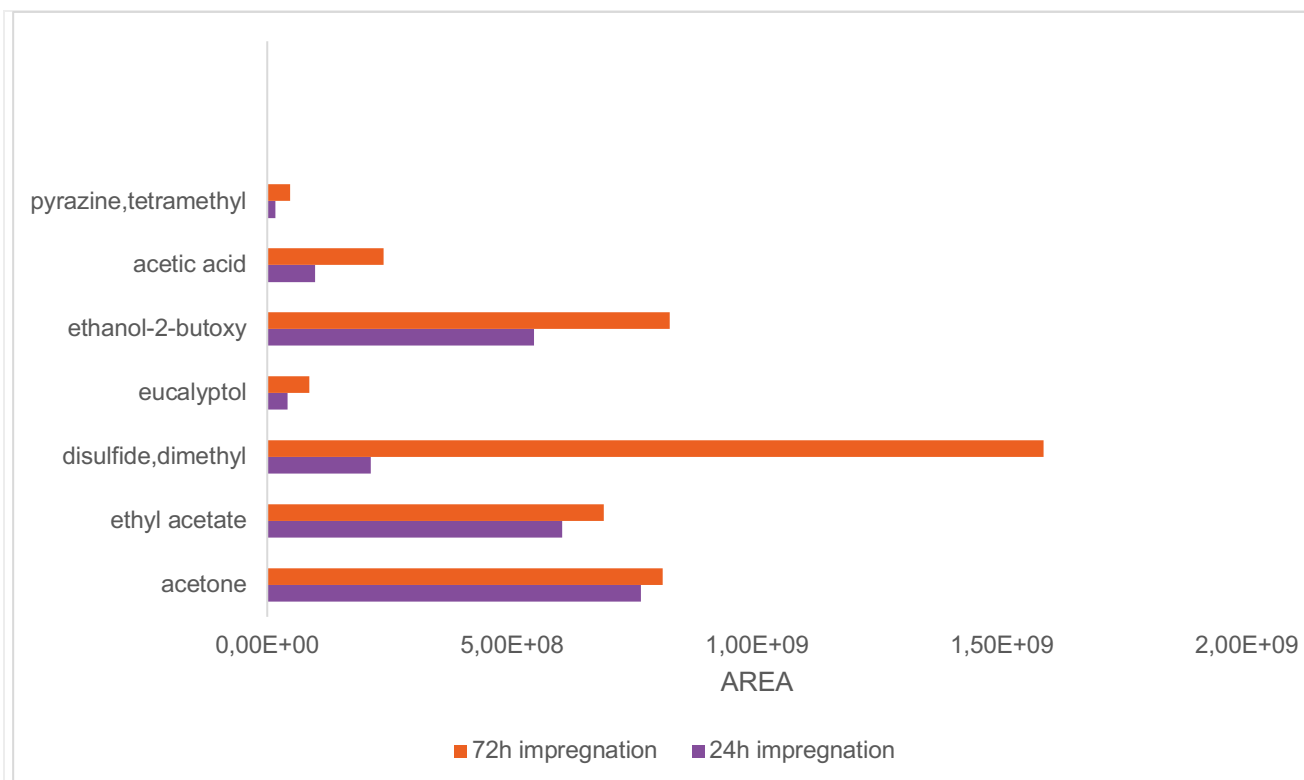
Figure 3: Amounts of compounds released by impregnated Getxent tubes after 24h (purple) and 72h (orange) of impregnation of dead body.

For the seven compounds identified, the concentration increases between 24h and 72h. The increasing percentage have been calculated in figure 4. The values have been calculated according to the formula:

$$\% \ increasing = \left(\frac{area \ peak \ 72h}{area \ peak \ 24h} \times 100\right) - 100\%$$

Compound	Area at 24h	Area at 72h	Increasing percentage between 24h and 72h impregnation (%)
Acetone	7,63E+08	8,07E+08	+6%
Ethyl acetate	6,03E+08	6,87E+08	+14%
Disulfide, dimethyl	2,12E+08	1,59E+09	+649%
Eucalyptol	4,14E+07	8,55E+07	+107%
Ethanol, 2-butoxy	5,45E+08	8,22E+08	+51%
Acetic acid	9,82E+07	2,38E+08	+142%
Pyrazine, tetramethyl	1,65E+07	4,63E+07	+181%

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Figure 4: Increasing percentage of VOCs concentrations from 24h to 72h

Discussion

As siloxane peaks belong to the chromatograms of empty vials and not impregnated Getxent tubes, they are not released by the Getxent tubes. Getxent tubes are thus analytically clean. Due to the presence of hydrophilic blocks in the polymers composing the Getxent tubes, Getxent tubes can release water captured during impregnation (humidity from air or target odor). As siloxanes and water have no impact on the study, they will not be considered.

After impregnation of the tubes at 24 and 72 hours, the analysis have shown an identical number of peaks (167 signals). The profiles are therefore comparable.

Seven molecules have been identified and measured. The analysis shows that the concentrations of these compounds released by the Getxent tube increase over the time of impregnation. The increasing of the signal is faster for dimethyl disulfide (+649% in 48h) than for acetone for example (+6% in 48h), however the signals were always very intense for each component (always over 10^7).

Given the sensitivity of the dog to detect odors and the intensity of the compounds detected by GC-MS (> 10^8 cps), the dog should be able to detect the odor of a cadaver by an impregnated Getxent tube.

Conclusion

Getxent tubes are analytically clean.

They can accurately capture and release the odor of dead body. Over time, the Getxent tubes captured and released the same number of compounds (167 peaks).

The concentration of molecules released by the impregnated Getxent tubes increases over time. However, the evolution of the concentration is not the same for each compound, since disulfide dimethyl increases much faster than acetone for example.

The high concentrations of molecules and the sensitivity of the dog should promote the detection of a dead body.

Author	Reviewer
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Annex 1: GC-MS method

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**************************************************************************

MSWS 8.0.1 for SCION — Method Listing   Mon May 02 10:58:02 2022

Method: Tube40RD v2.mth

**************************************************************************

**************************************************************************

***********************
    CombiPAL AutoSampler
***********************
Module Address: 24

CPAL Method:

                    Injection Mode: GC SPME
                    Read Bar Codes: Never
                  Required Syringe: SPME Fiber
               Agitator Temperature: 40.0 C
          Sample Pre-Incubation Time: 5 min. 0 sec.
       Pre-Incubation Agitator Speed: 250 rpm
      Pre-Incubation Agitation Cycle: 2 sec On, 4 sec Off.
          Extraction Agitator Speed: 250 rpm
            Fiber Depth From Bottom: 10 mm
                    Extraction Time: 25 min. 0 sec.
                           Injector: Front
                        Desorb Time: 2 min. 0 sec.
                 Use Bakeout Station: Yes
                 Fiber Bakeout Time: 5 min. 0 sec.
           Fiber Clean Temperature: 225.0 C
     GC Cycle Time (for Prep Ahead): 36 min. 0 sec.

***********************
    SCION Mass Spec
***********************
Module Address: 40

Acquisition Method =================

Acquisition delay 4.50 min.
No pre run macro.
No post run macro.
CID Gas off
Ion Source: EI
Data Type: Centroid

******************************
    43X-GC  — Model 436-GC
******************************
Module Address: 44

Valve Table
-----------
    No Valves Used

 Front Injector Type S/SL
----------------------------------
            Oven Power: On
               Coolant: Off
      Enable Coolant at: 250.0 C
        Coolant Timeout: 20.00 min

    Temp      Rate       Hold       Total
    (C)      (C/min)    (min)      (min)
    --------------------------------
    250.0     0.0       20.00      20.00

     Time      Split     Split
     (min)     State     Ratio
     ---------------------
    Initial     On         10
      0.01      Off        Off
      2.00      On         50

 Front Injector EFC Type 21: Enabled
--------------------------
      Constant Column Flow: 1.00 ml/min
            Pressure Pulse: none

            No Backflush.

Column Oven
-----------
                Coolant: Off
      Enable Coolant at: 50.0 C
        Coolant Timeout: 20.00 min
     Stabilization Time: 2.00 min
```

```
    Temp     Rate      Hold      Total
    (C)     (C/min)    (min)     (min)
    --------------------------------
    40.0      0.0       2.10      2.10
   200.0     10.0       5.00     23.10
```

No Auxiliary Heaters installed

Data Acquisition

```
    Acquisition Frequency : 25.0 Hz
          Monitor Length : 64 points (2.560 sec)
     Front FID/NPD Scale: 10 Volts
    Middle FID/NPD Scale: 10 Volts
      Rear FID/NPD Scale: 10 Volts
```

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