Electronic devices – Investigation of the direct thermal extraction properties of the e-liquid

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1. Context

The mode of action of a wide range of e-devices on the market is based on the transfer of e-liquid compounds through a heated air flow.

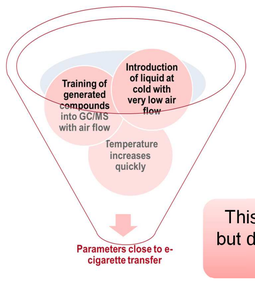
The **aim** of this work is to provide a method to assess the e-liquid for qualitative screening of the vapour without using a smoking machine. This poster describes:

- the investigation of the liquid used in the e-device by focusing on the properties of the liquid itself using Gerstel Thermal Desorption (TDS3) or Pyrolysis Module (PM1)- Mass Spectrometer equipment
- the comparison on carbonyls and alpha-carbonyls emissions with an aerosol generated on a VC10 smoking machine.

Tests were carried out with both e-vapour devices and e-liduids prototypes.
The liquid mixtures tested were only base liquids without flavour.

2. Analytical parameters optimisation

The key parameters of TDS3- and PM1-GS/MS methods are:



- Gas flow and pressure
- Running time
- Temperatures (TDS oven, cryotrap CIS, transfer capillary, GC oven)
- Sampling mode of the solution
- Splitless or Split mode for TDS and CIS…

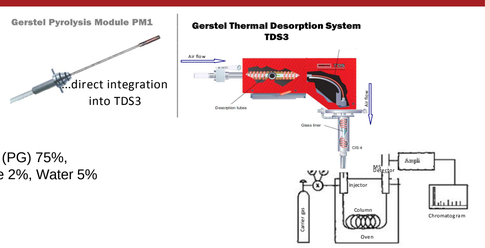
This method does not exactly simulate e-cigarette testing but during the optimisation, the conditions were oriented to be as close to the transfer system of e-cigarette

3. TDS/PM operating conditions

Sampling
2 liquids tested

MixA: 100% PG

MixB: Propylene Glycol (PG) 75%, Glycerol 18%, Nicotine 2%, Water 5%



Sampling	300 nL of liquid sample placed in the capillary tube e-liquid **without preparation**
Thermodesorption TDS3 (oxydative mode)	*Indirect heating of the sample: the oven heats both the desorption and the capillary tube containing the liquid sample* From 150°C to 400°C for 1 min (ramp rate: 60°C/min) Transfer line T°: TDS t° + 20°C. Splitless
Pyrolysis PM1 (oxydative mode)	*Direct heating of the capillary containing the liquid sample by the heating wire* From 500°C to 1000°C for 1 min (wait for 2min before and after) TDS T°: 50°C Transfer line T°: 200°C. Splitless
CIS4 – injection (switch to Helium)	Initial t°: -50°C for 2min - Ramp rate: 12°C/s - Final t°: 280°C (for 10min) Split 1:200
GC column	DB-5ms (30mx250µmx1µm, low polarity) 1,1mL/min with Helium
Oven T° GC	Initial t°: 32°C (for 5min) - Ramp rate: 10°C/min - Final t°: 200°C (for 2min) Analyse time: 24min
Detection	MS – EI source - Mode SCAN (29 à 450 amu) Results expressed in peak area or relative peak area (=compound area/total area in %)

Reference

[1] Determination of carbonyl compounds generated from the electronic cigarette using coupled silica cartridges impregnated with hydroquinone and 2,4-dinitrophenylhydrazine. Ohta, Kazushi Uchiyama, Shigehisa Inaba, Yohei Nakagome, Hideki Kunugita, Naoki ref: 2011 JOUR : Bunseki Kagaku volume: 60 issue: 10 pages: 791-797 written in Japanese.

4. Results and discussion

TDS-GC/MS profiles with MixB e-liquid

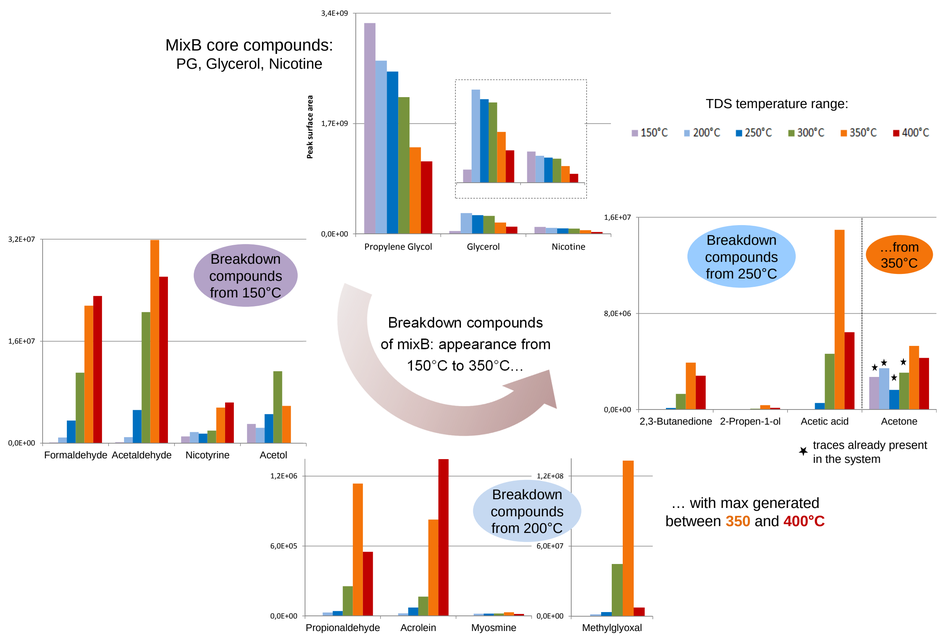


Figure 1: Profile of the degradation of MixB e-liquid with TDS-GC/MS (150°C≤T°≤400°C) in surface area. Average peak surface areas of 2 replicates. All identified compounds were confirmed with pure chemical products

- 12 breakdown compounds are identified (carbonyls, α-carbonyls and others compounds) in addition to the 3 core compounds contained in the original e-liquid (PG, glycerol and nicotine).
- By increasing the TDS temperature, we observe a decrease of the original compounds area (PG, glycerol and nicotine) *vs.* an appearance of breakdown compounds from 150°C and an increase of their areas.
- At 350°C all products produced after decomposition of the liquid are already identified.
- No additional by-products up to 1000°C using the PM1-pyrolysis system (T° between 500°C and 1000°C), profiles not represented here.

TDS-GC/MS *vs.* VC10 smoking machine

Comparison of the carbonyls emission

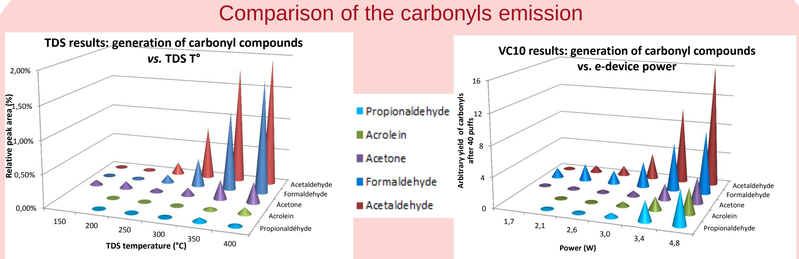


Figure 5: Profiles of the carbonyls emissions of MixB e-liquid with TDS-GC/MS (150°C≤T°≤400°C) in relative area

Figure 6: Yields of the carbonyls emissions of MixB e-liquid with VC10 smoking machine after 40 puffs at different powers (between 1,7 and 4,8 W)

Conditions:

- Same solution tested: MixB (PG 75%, Glycerol 18%, Nicotine 2%, Water 5%)
- VC10 (Vitrocell) smoking machine (bell shape profile, 55 ml puff volume, 4 sec puff duration, 30 sec interpuff duration), use of prototype e-devices, analysis of trapped vapour after 40 puffs by HPLC-UV.

✓ Appearance of formaldehyde and acetaldehyde first, then higher yields compared to acrolein and propionaldehyde

► Common point: energy supplied to the liquid either by increasing the TDS temperature or by increasing the power given to the e-device (related to voltage and resistance of the e-device)

Zoom on methylglyoxal

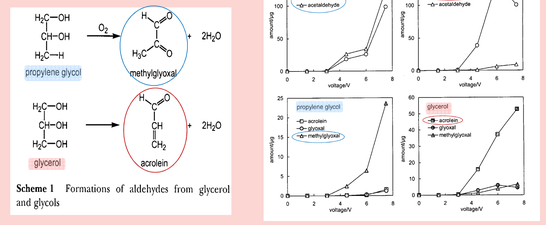


Figure 2: Extract from [1]. Generation of carbonyl compounds from PG and Glycerol heated by using a Ni-Chrome Wire

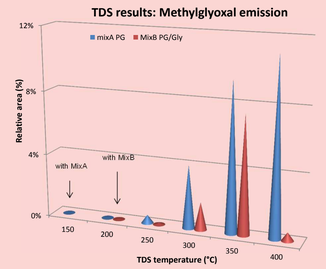


Figure 3: Profiles of the degradation of e-liquids containing mixA and mixB with TDS-GC/MS (150°C ≤T° ≤ 400°C) in relative area

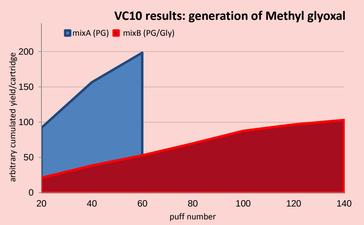


Figure 4: Cumulated yields of Methylglyoxal quantified on an e-device prototype using 2 different e-liquid cartridges (mixA and mixB) with VC10 smoking machine (analysis of trapped vapour by HPLC-UV)

- The main degradation compounds of PG and glycerol are methylglyoxal and acrolein respectively (fig.2. Scheme1).
- Focus on methylglyoxal emission, comparable trend between TDS and VC10:
  - with TDS-GC/MS, methylglyoxal appears first with the mixA (PG) and the relative areas stay higher compared to mixB (PG/Gly) (fig.3).
  - with VC10 smoking machine, methylglyoxal is much higher with the mixA (PG) than with the mixB (PG/Gly) and from the first 20 puffs (fig.4).

Conclusions

- An optimised TDS-GC/MS method has been developed to assess the qualitative profiles of the thermal decomposition of different solutions (temperature from 150 to 400°C) without smoking and without sample preparation.
- Thermal extraction and pyrolysis are complementary: although no additional compounds are identified with PM1-pyrolysis compared to TDS3, the PM1-pyrolysis profiles give complementary information about the yields of these compounds.
- TDS *vs.* smoking machine:

Similar decomposition process of the e-liquid have been observed: the generation of carbonyl and methylglyoxal compounds depends either on the TDS temperature or the e-device puff number or the thermal energy delivered by the e-device to the e-liquid.

Perspectives

- To confirm the TDS-GC/MS results by testing additional liquids.
- To confirm TDS *vs.* smoking machine results on squared wave profile.