



Dark Chocolate... The Real Dark Secret

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2015

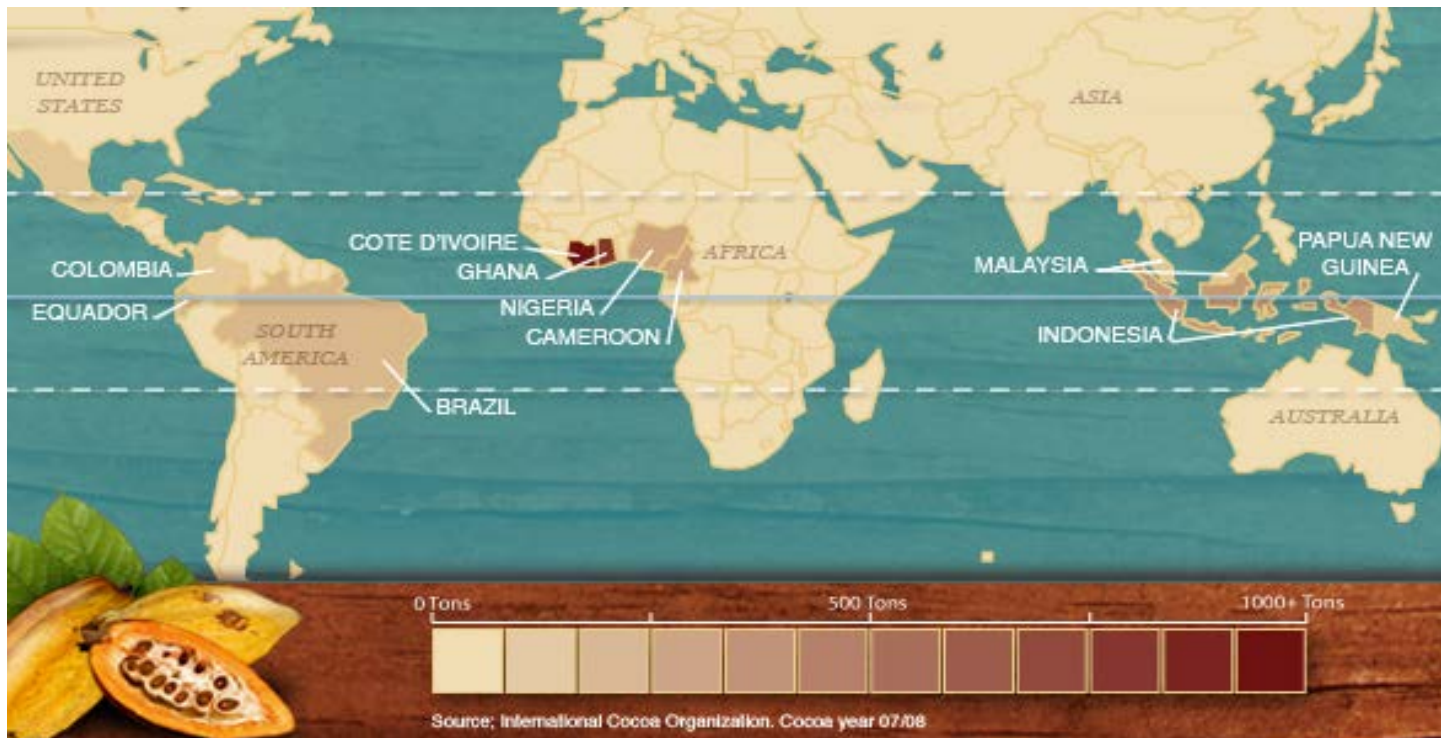


Presentation Outline :

- General facts
- Method of Extraction
- GC-TOFMS Methodology
- Results
- Conclusion

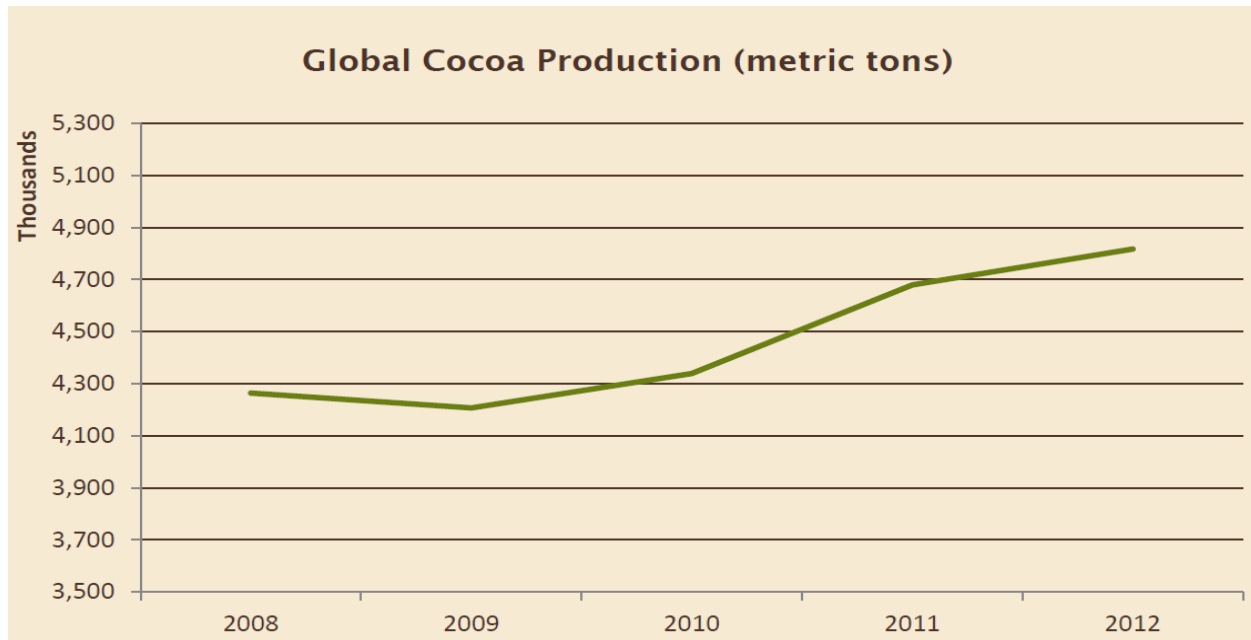


Where is Cocoa Grown ?





Global Production :





Bean to Bar :

- Pods are collected and split open
- A farmer can expect 20-50 beans per pod, depending upon variety
- Takes approximately 400 beans to produce 500g of Chocolate
- Harvested beans are fermented for 3-7 days
- Fermented beans are then dried, sold, packed and transported
- Roasting and Grinding- Cocoa liquor produced
- Cocoa liquor pressed ready for Chocolate manufacture



Dark Chocolate...

- More cocoa solids than sugar
- Greater health benefits
- More intense flavours





LECO GC-TOFMS :

Fastest Type of Mass Analyzer

- Requires fast data acquisition system
- Leco Pegasus HT: Capable of 500 Full Mass Range Spectra/s
- Ability to properly define narrow chromatographic peaks (Hi Speed GC)
- Delivers the data density needed for True Signal Deconvolution®
- Full range mass spectrum is collected at all times without sacrificing speed or sensitivity....no need for SIM for best sensitivity
- Spectral Continuity, no spectral skewing



Method of Extraction :

- 2 grams of each sample weighed out into headspace vial
- Each sample incubated at 37°C for 15 minutes
- Fibre used for SPME was PDMS-DVB-Carboxen
- Sampled with SPME for 20 minutes
- Thereafter sample was desorbed in GC inlet and analysed



GC-TOFMS Method :

GC: Agilent 7890 Gas Chromatograph

Column: 30m x 0.25mm x 0.25 μ m Restek Stabilwax

Inlet: Splitless @ 220°C

Carrier Gas: 1.50 mL/min, Constant Flow

GC Oven: 40°C (5 min hold), 7°C/min to 220°C, (5 min hold)

MS Transfer Line: 200°C

MS: LECO Pegasus HT

Ion Source: EI at -70 eV

Ion Source Temperature: 220°C

Spectral Acquisition Rate: 20 spectra/s

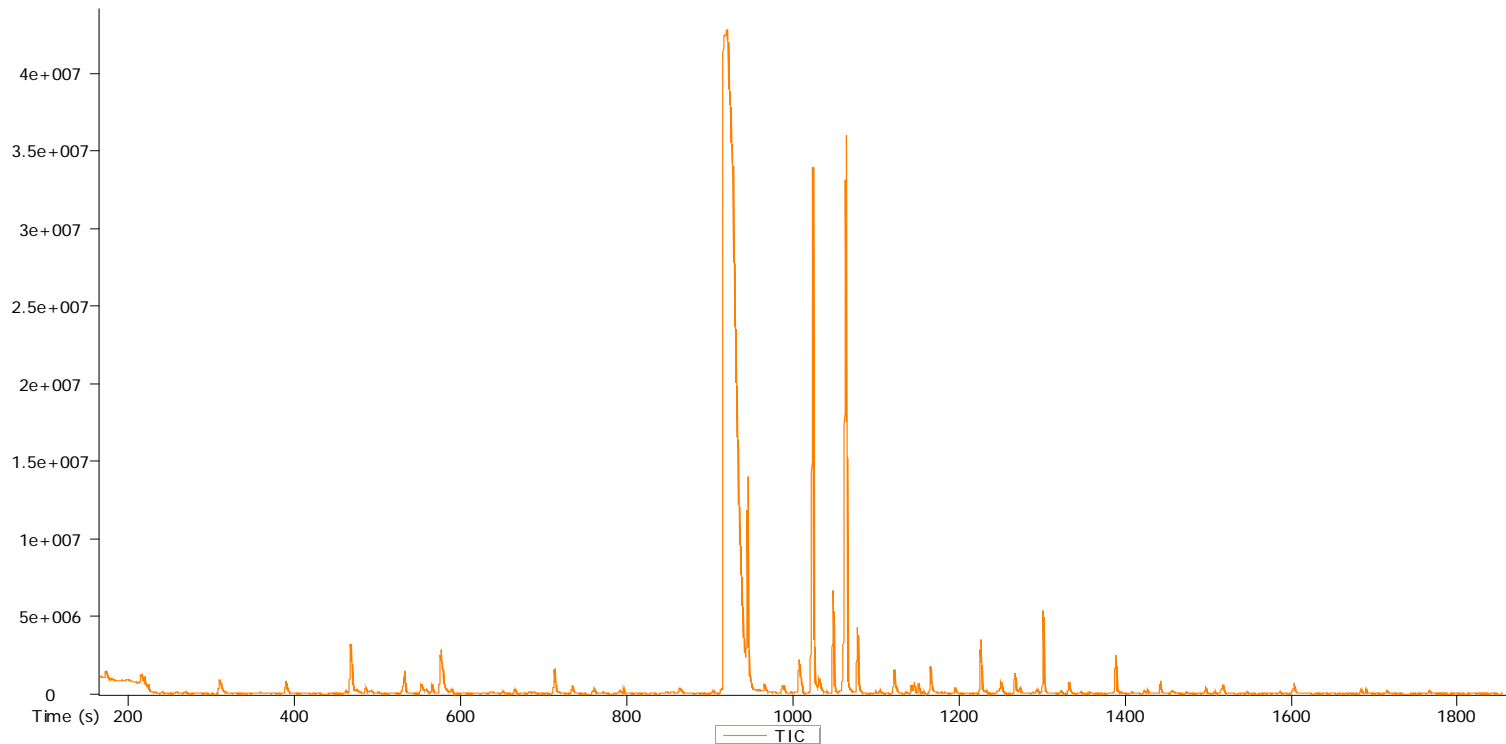
Acquired Mass Range: 30-550 m/z

Instrument Control and Data Review: ChromaTOF

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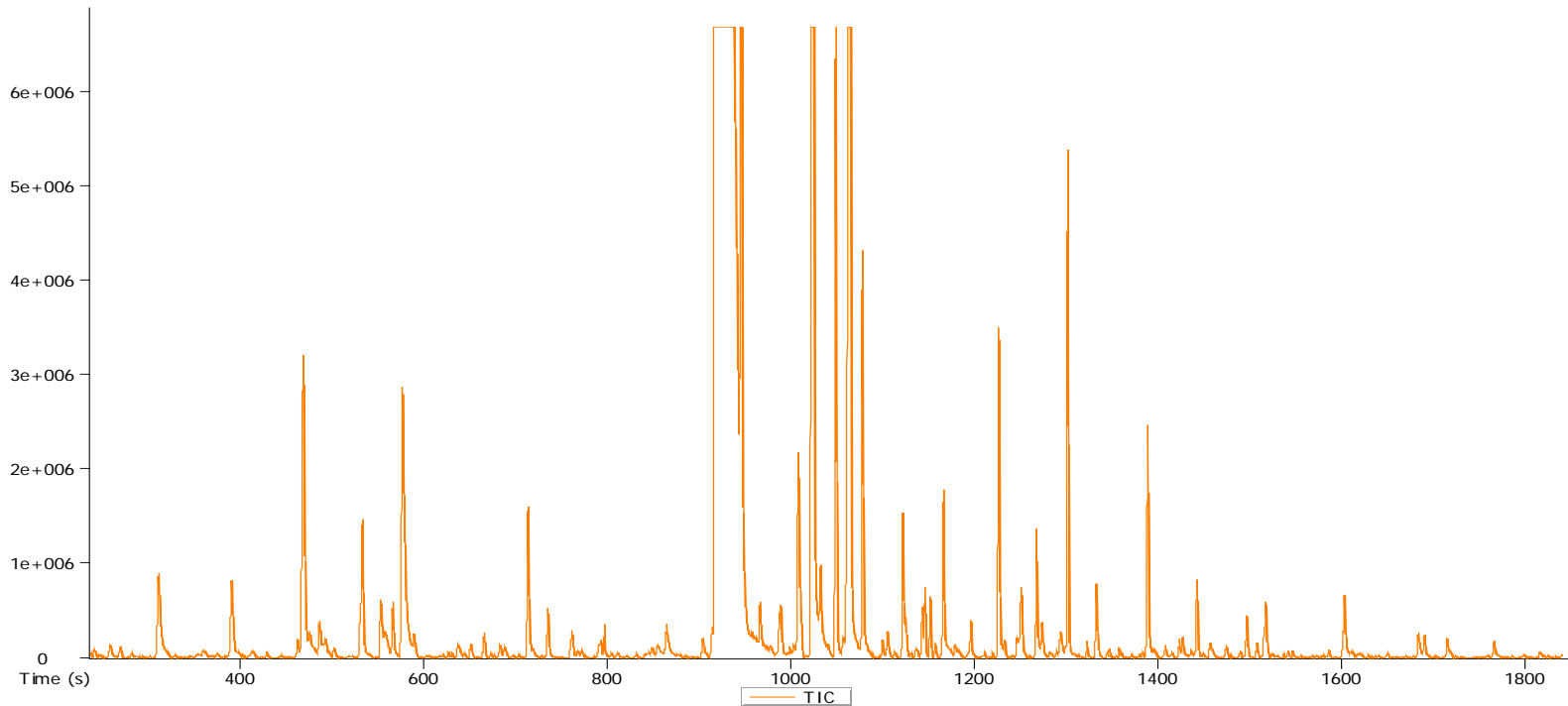


Typical Dark Chocolate Chromatogram:





Does Chromatographic Peak Size Count ?



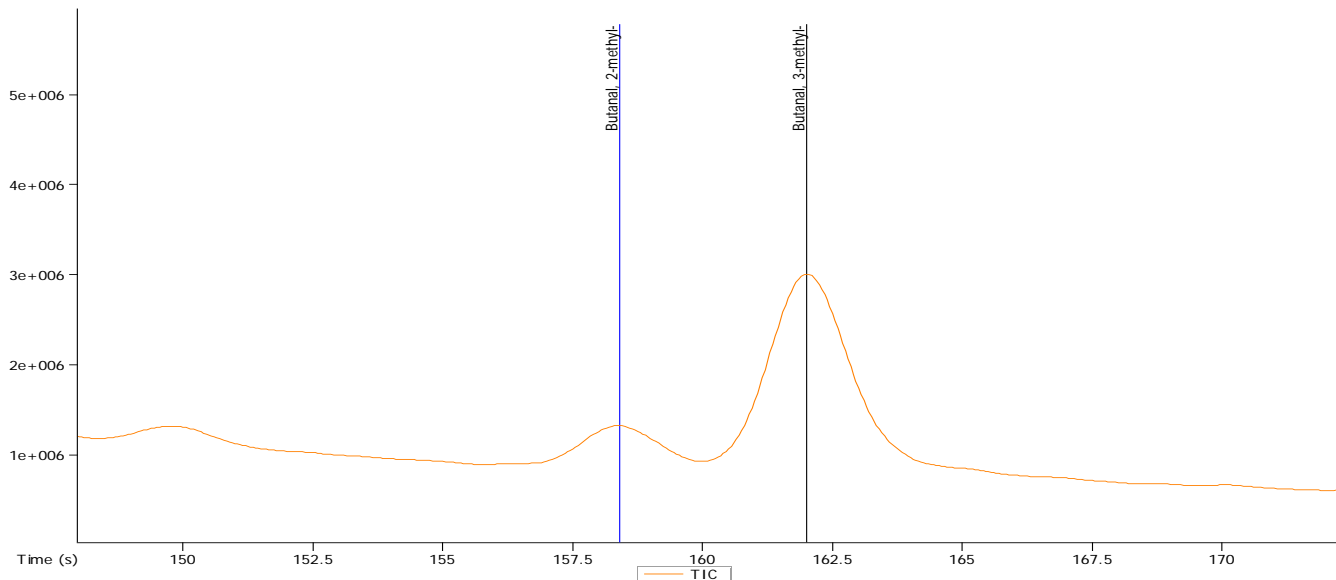


Marks & Spencer Dark Chocolate 72% : Mix of Flavours

- Aldehydes impart chocolate flavour :
2-Methylbutanal and 3-Methylbutanal
- Furanoid : Flowery, fruity taste, : **trans-Linalool oxide**
- Pyrazines generally a roasted odour or flavour :
Methylpyrazine

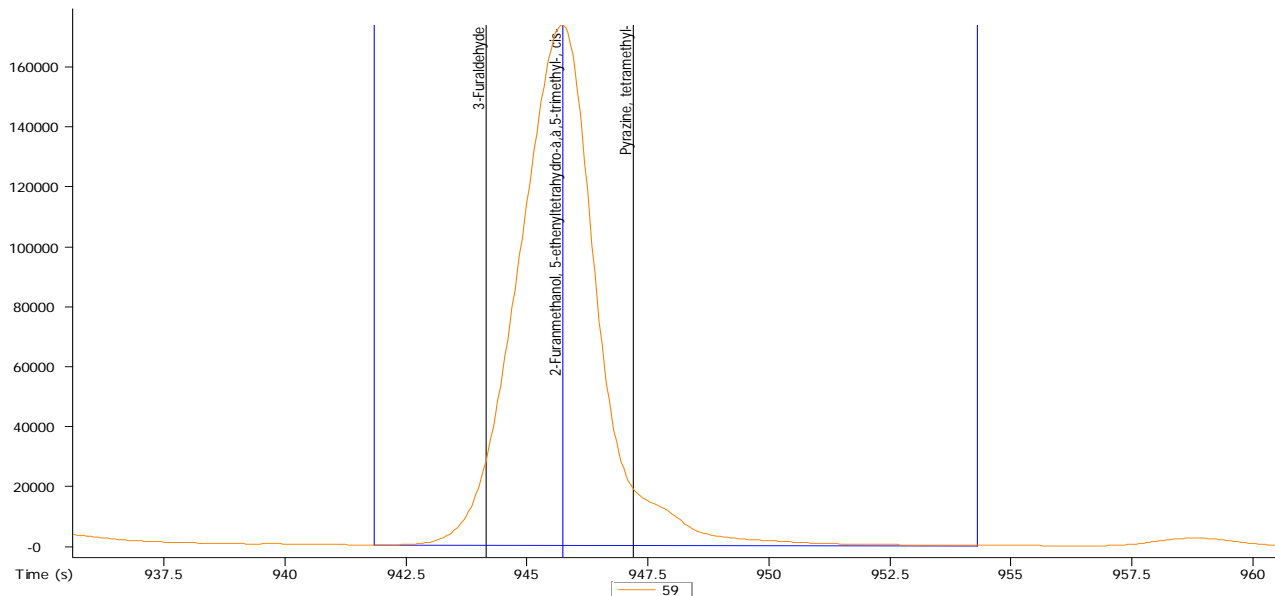


Aldehydes impart chocolate flavour : **2-Methylbutanal and 3-Methylbutanal**



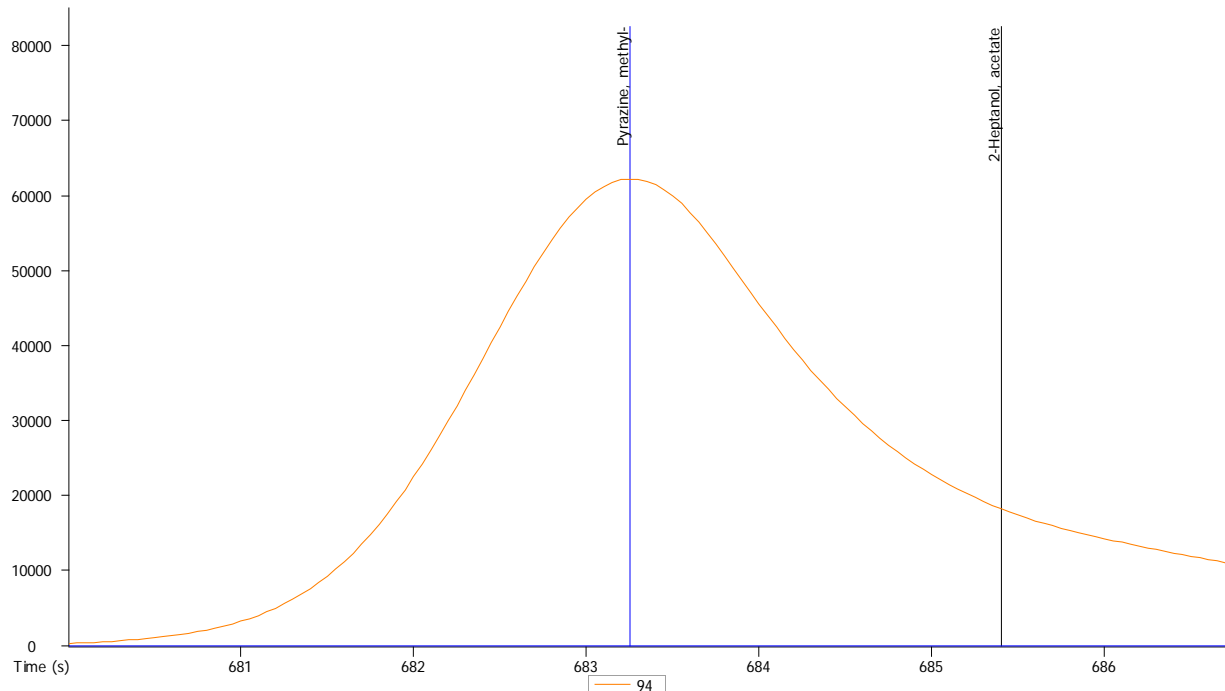


Furanoid Flowery, fruity taste: **trans-Linalool oxide**





Pyrazines Roasted : Methylpyrazine



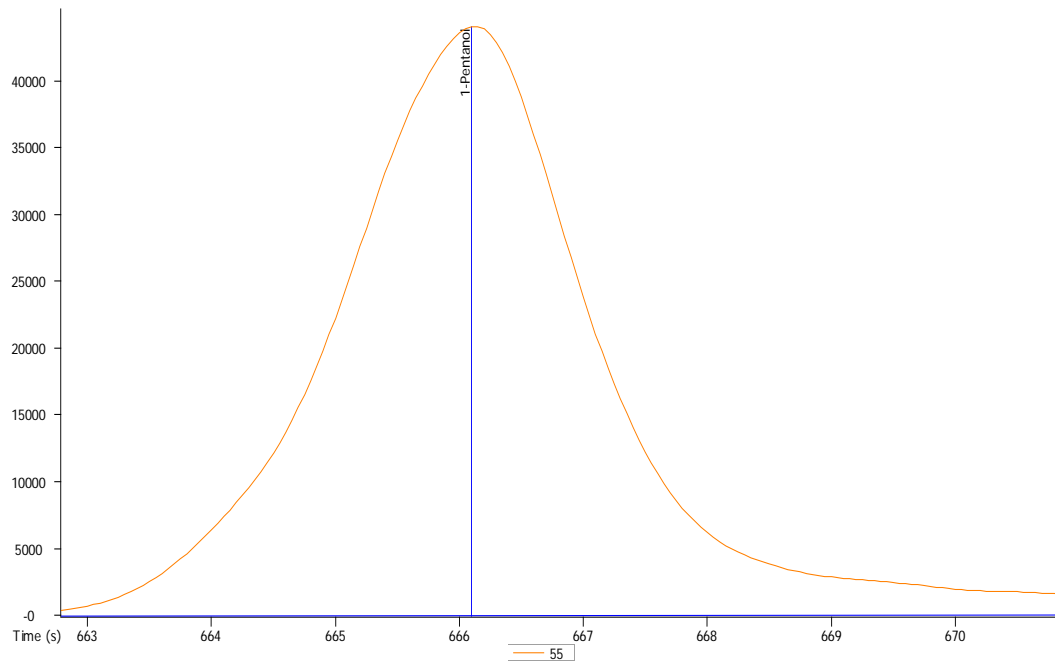


Willie's Cacao Madagascan Gold, 71% - Notes of Summer fruits :

- Alcohols – Flowery, Floral and Fruity:
1-Pentanol
- Esters – Honey, Sweet, Fruity and Floral :
Acetic acid, 2-phenylethyl ester



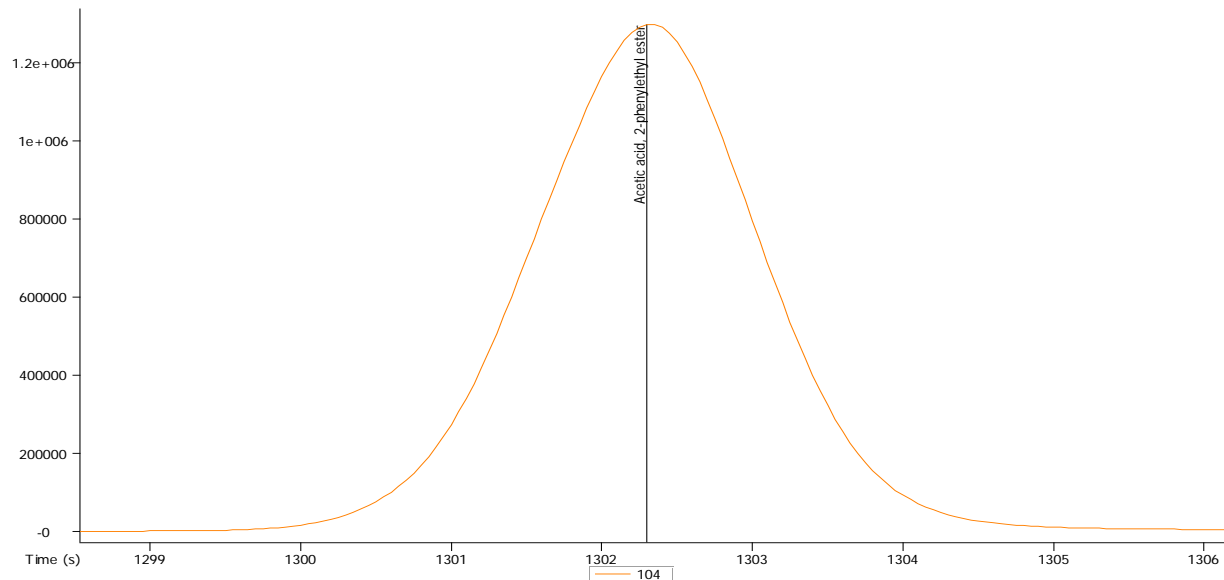
Alcohols – Flowery, Floral and Fruity: **1-Pentanol**





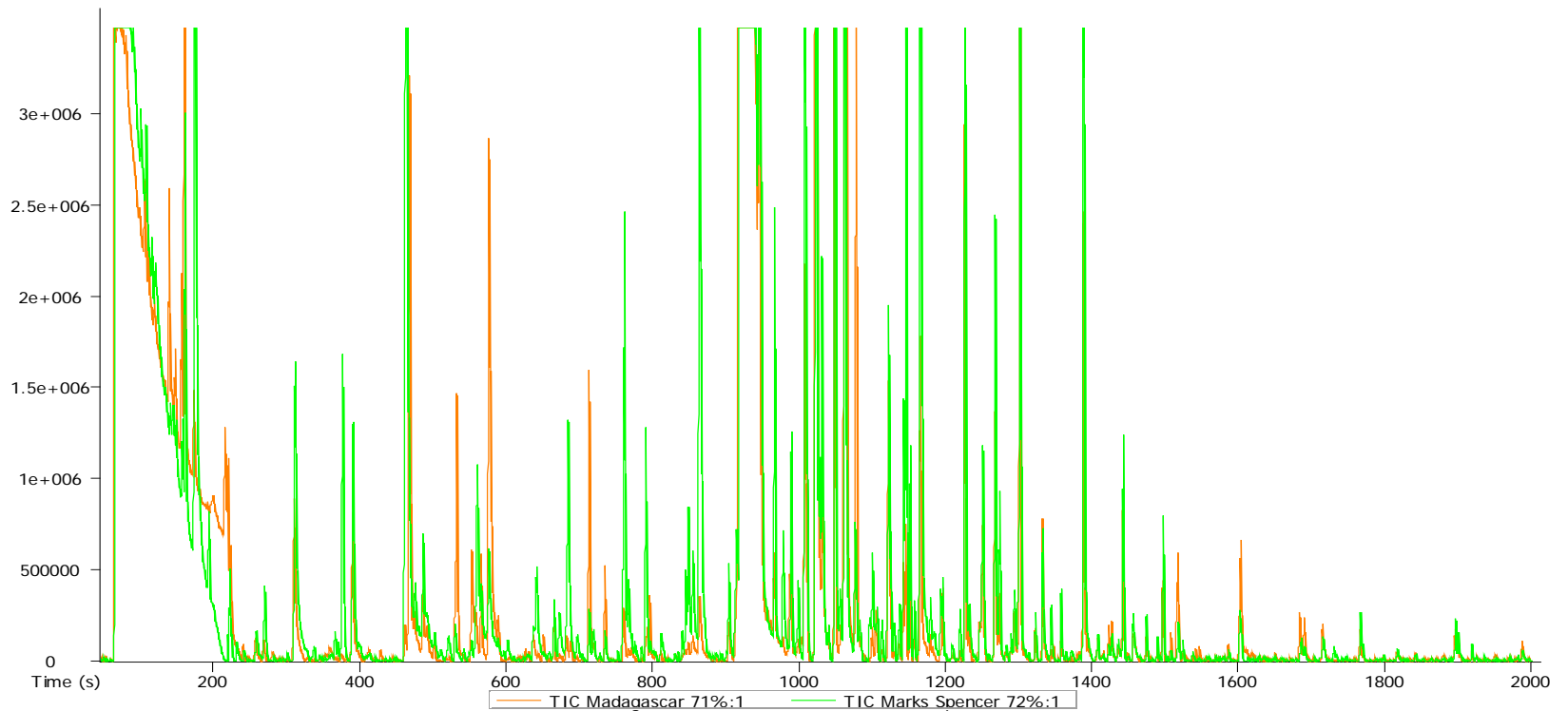
Esters – Honey, Sweet, Fruity and Floral :

2-phenylethyl ester Acetic acid



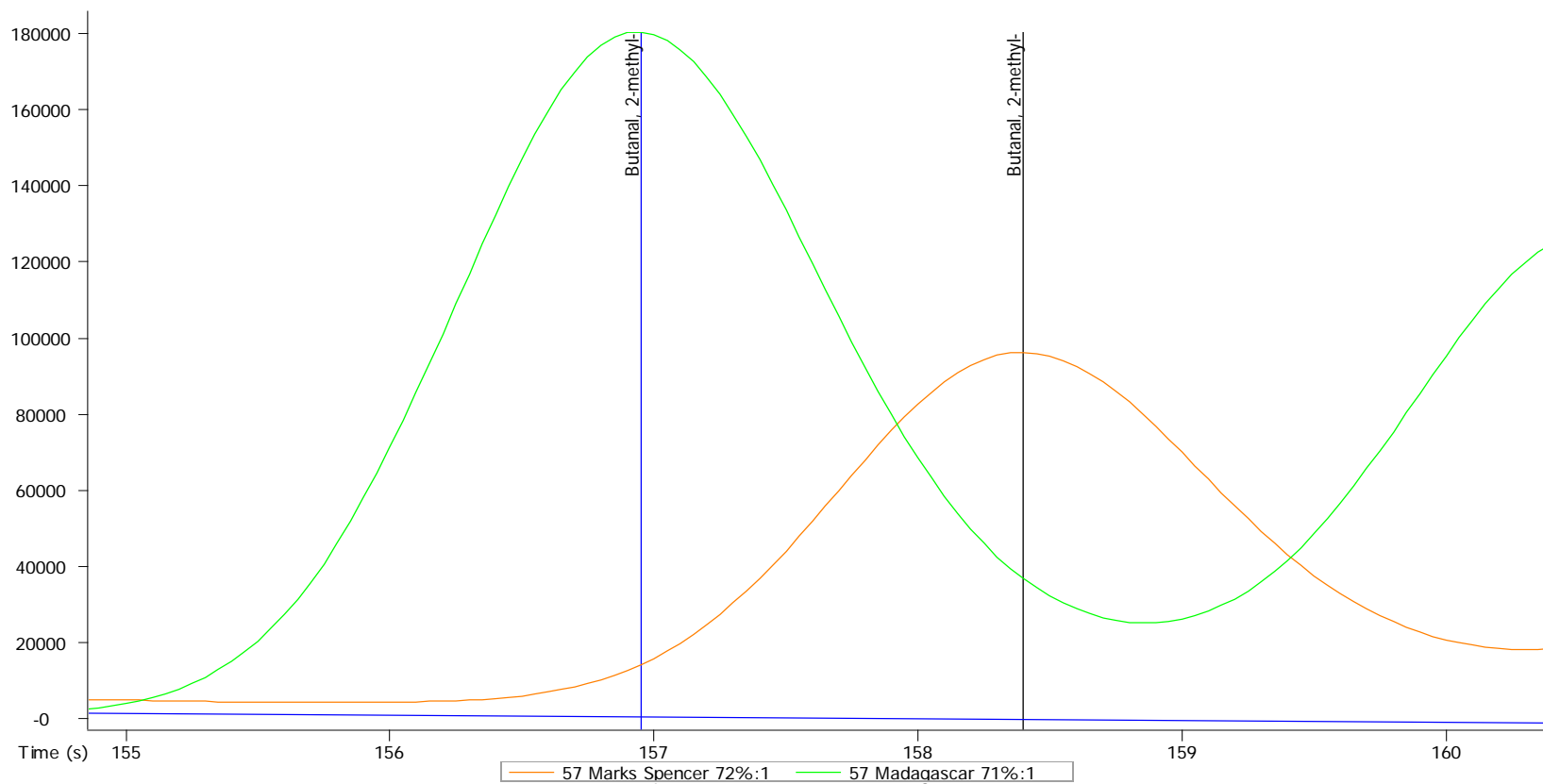


Visual Difference :



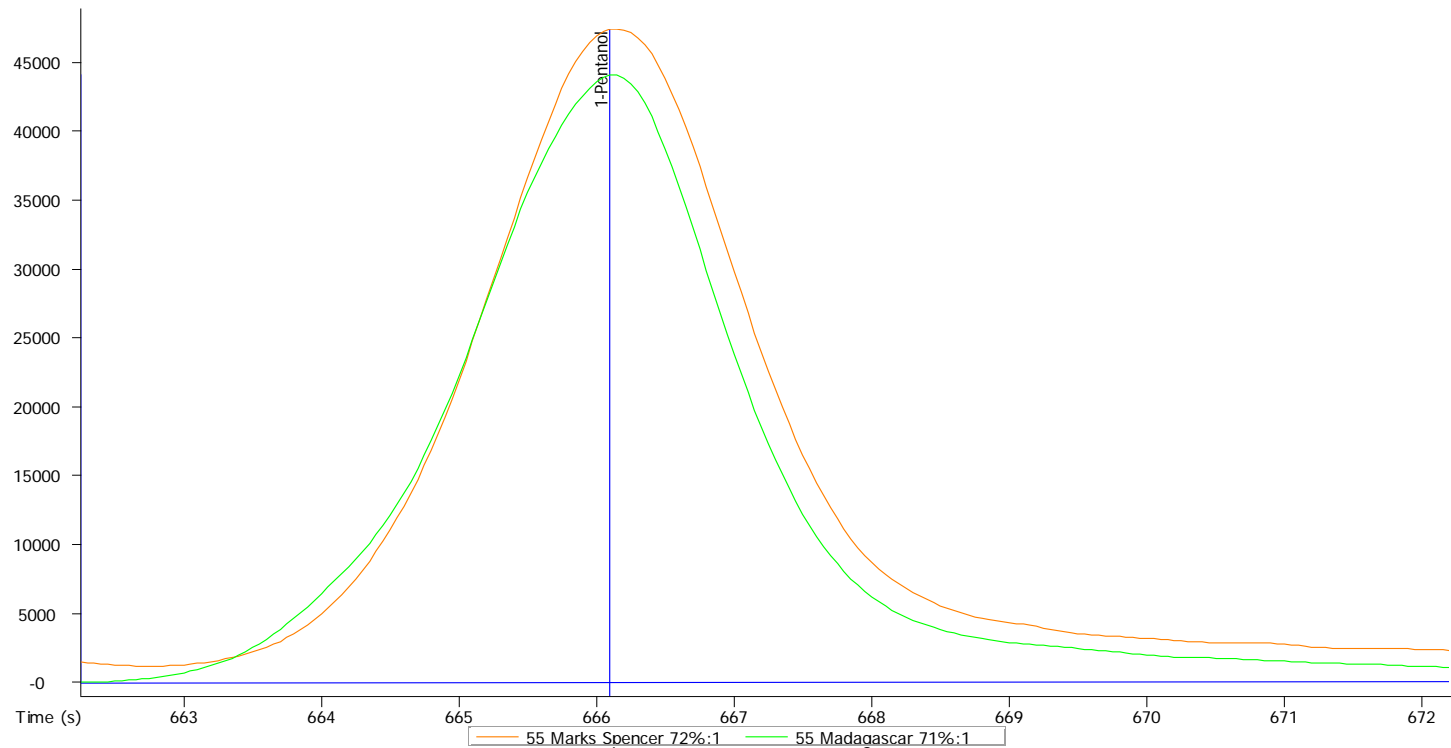


Aldehydes : 2-Methyl Butanal



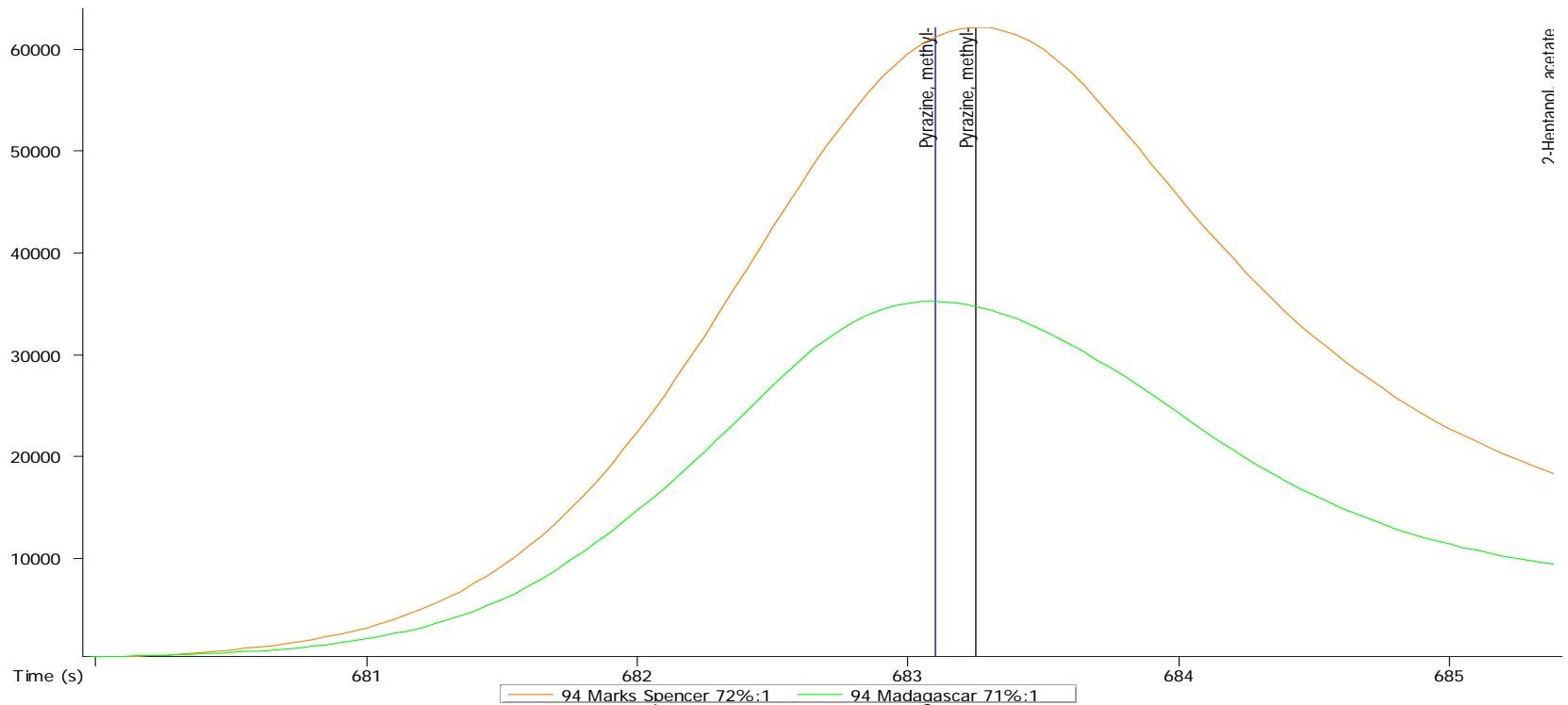


Alcohols: 1- Pentanol



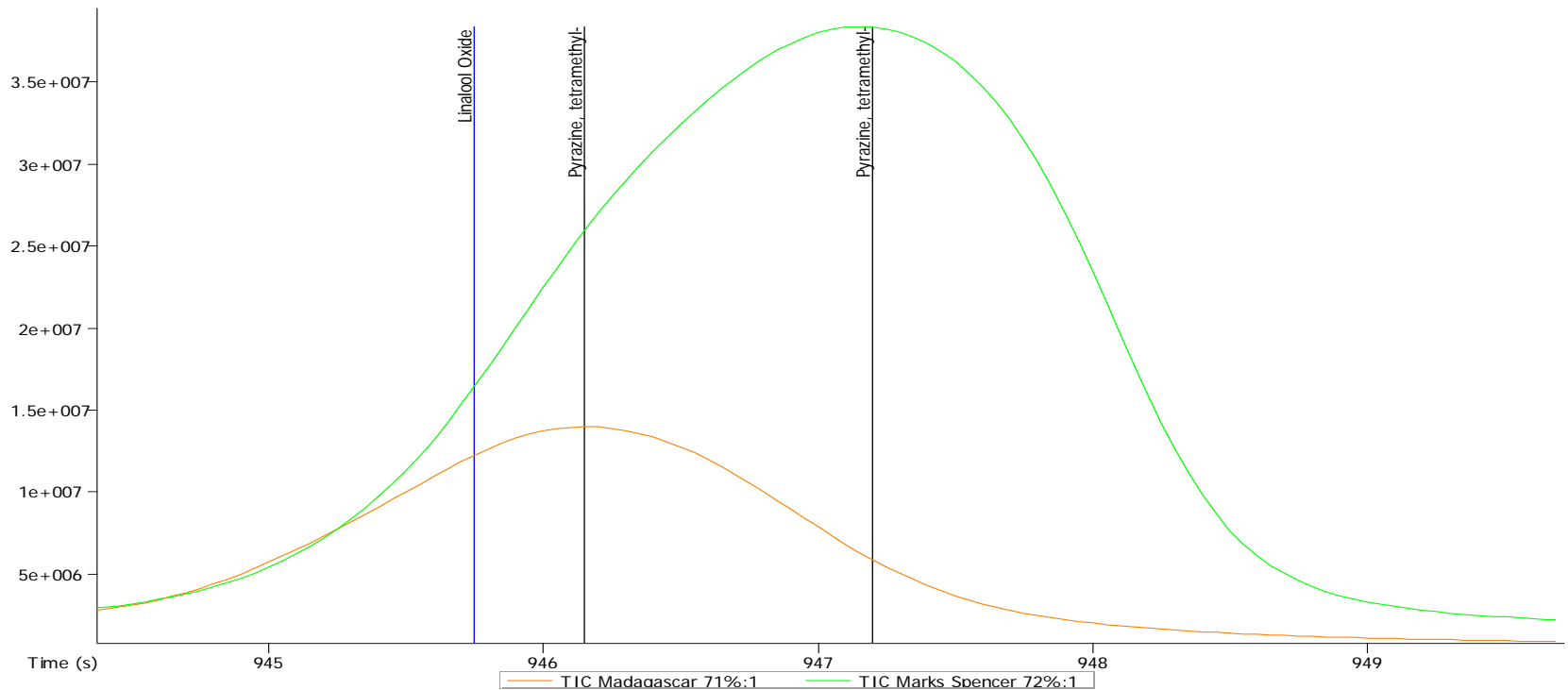


Pyrazines: Methyl-Pyrazine





The Difference : Linalool Oxide, Marks Spencer





Conclusion :

- Chocolate is a very complex sample, not as simple as it tastes
- SPME provides a snapshot
- GC-TOFMS can find a multitude of components
- We have only seen the tip of the iceberg
- Due to mass spectra produced fingerprinting of compounds is even more beneficial
- Provides more accurate results



Acknowledgements :

- SENSORY FX
- KERRY Flavours
- SAAFoST

Thank you!