

5th SBSE International Meeting - Paris
Aroma Analysis of Cocoa and Chocolate at Barry Callebaut
Isabelle Van Leuven



5TH SBSE INTERNATIONAL MEETING

23 & 24 SEPTEMBRE 2019 - NOVOTEL PARIS-SUD

SBSE 
Technical Meeting

Agenda

- ▶ Presentation of company Barry Callebaut (BC)
- ▶ From Cocoa to Chocolate flavor
- ▶ Aroma Chemistry of Cocoa and Chocolate
- ▶ Aroma analysis at BC
- ▶ Case studies of Aroma analysis at BC
- ▶ Conclusions

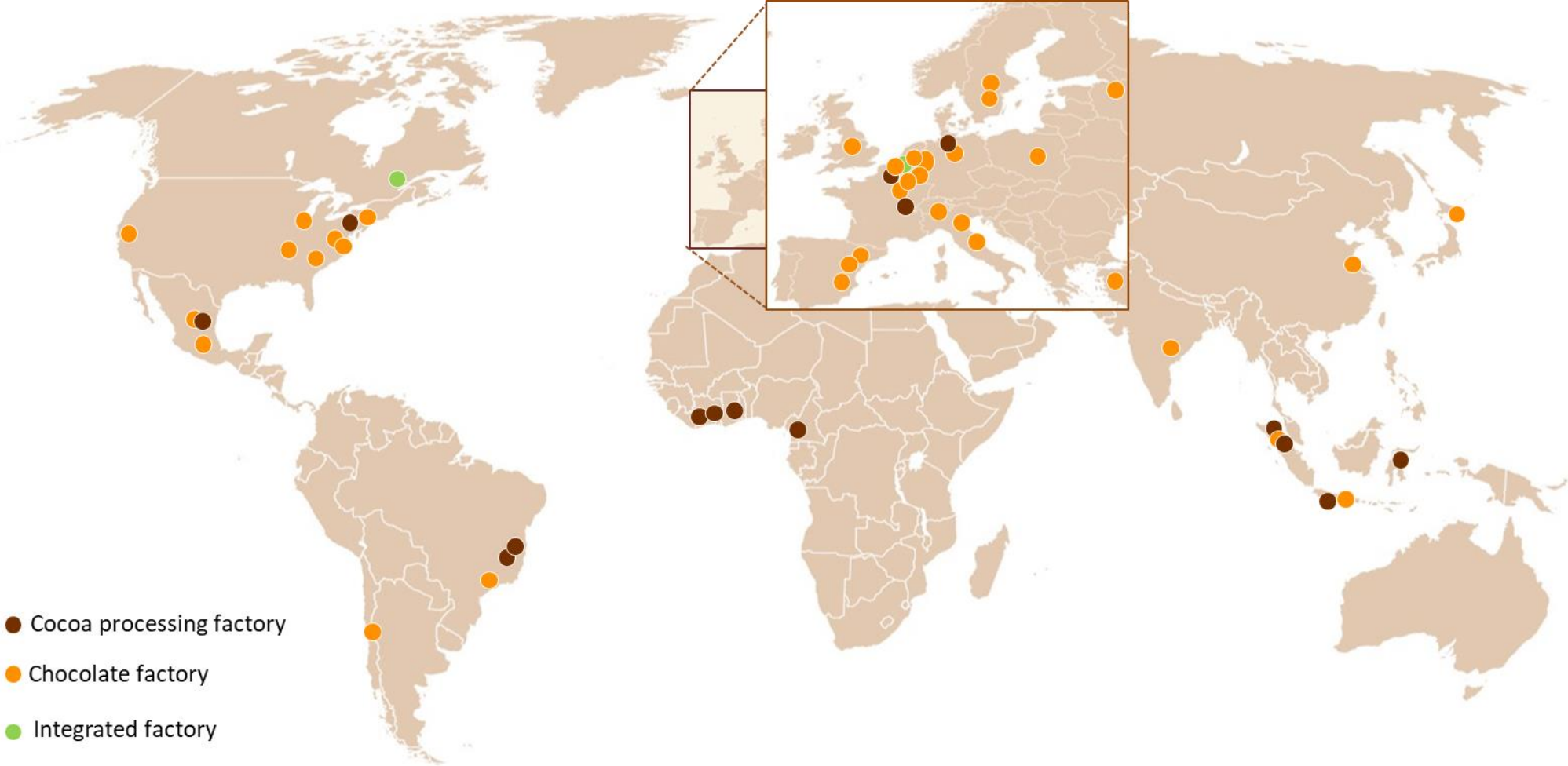
Aroma Analysis of Cocoa and Chocolate at Barry Callebaut



- ▶ The world's leading manufacturer of high-quality chocolate and cocoa products
- ▶ Headquartered in Zurich, Switzerland
- ▶ A business-to-business company
- ▶ Founded in July 1996 as a merger between French Cacao Barry and Belgian Callebaut
- ▶ Some of our brands date back to the early 19th century
- ▶ Chocolate and cocoa products - from sourcing and processing cocoa beans to producing the finest chocolates, including chocolate fillings, decorations and compounds
- ▶ More than 11.000 employees



Aroma Analysis of Cocoa and Chocolate at Barry Callebaut

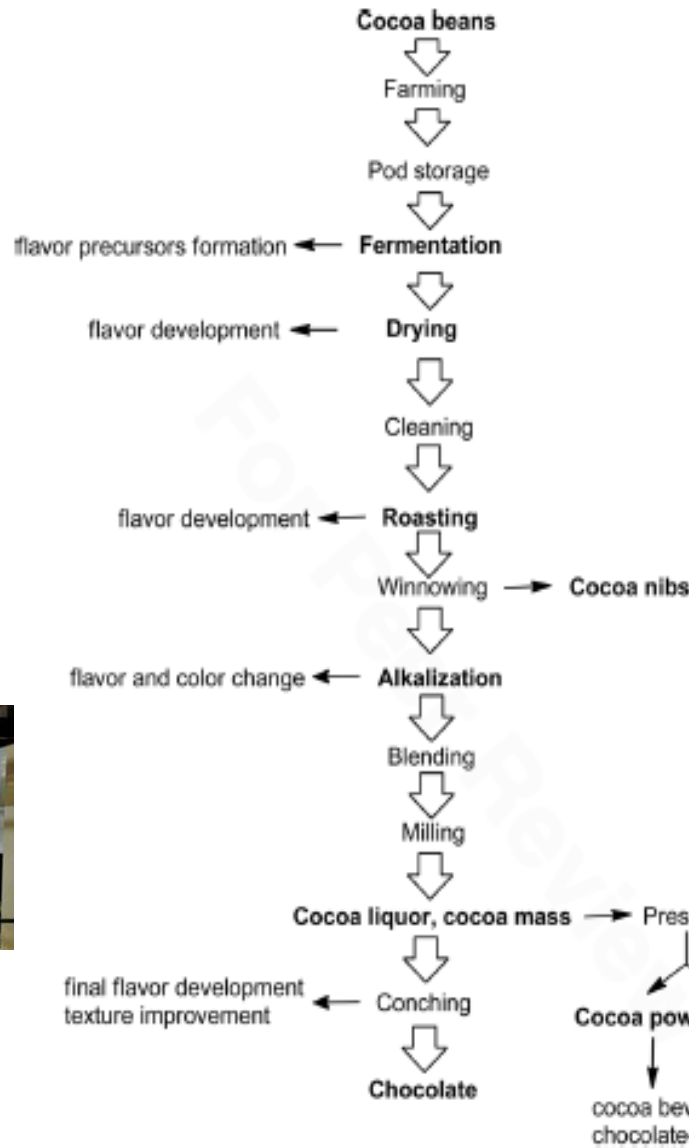


Aroma Analysis of Cocoa and Chocolate at Barry Callebaut

From Cocoa to Chocolate flavor



Theobroma cacao



Cocoa Liquor

Cocoa Butter

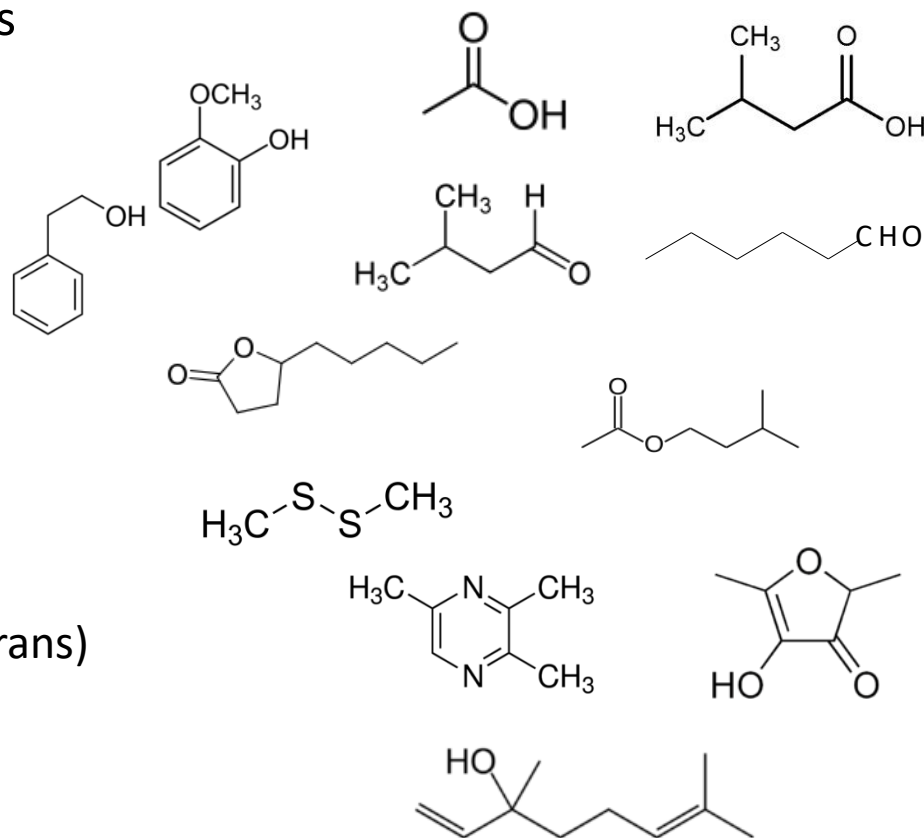
Cocoa Powder



Aroma Chemistry of Cocoa and Chocolate

▶ Chemical classes of aroma compounds

- ▶ Volatile acids
- ▶ Volatile phenols
- ▶ Alcohols
- ▶ Aldehydes
- ▶ Ketones
- ▶ Lactones
- ▶ Esters
- ▶ Sulfur compounds
- ▶ Heterocyclic compounds (pyrazines, furans)
- ▶ Terpenes



→ odor characteristic and threshold value!!

Aroma Chemistry of Cocoa and Chocolate

► Aroma Compounds and their sensory descriptions

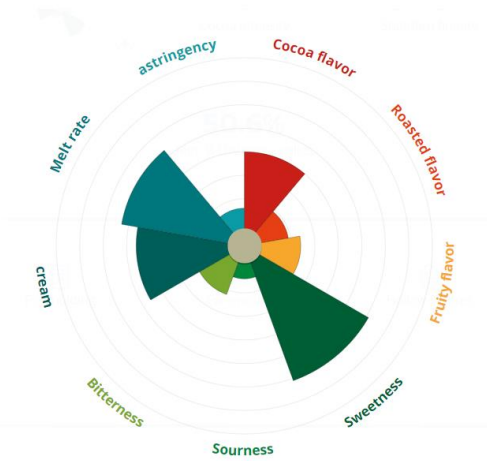
Aroma Compounds	Sensory description
Cocoa notes	
2-methylbutanal	malty, chocolate-like
3-methylbutanal	malty, cocoa
5-methyl-2-phenyl-2-hexenal	cocoa, coffee-like
Roasted notes	
trimethylpyrazine	roasted, earthy
2-ethyl-3,6-dimethylpyrazine	roasted, earthy
2-ethyl-3,5-dimethylpyrazine	roasted, earthy
Volatile acidity	
acetic acid	vinegar-like, sour
Fruity notes	
ethyl 2-methylpropanoate	fruity
2,3-butanedione	buttery
methyl 3-methylbutanoate	fruity, pineapple, apple
ethyl 2-methylbutanoate	fruity
ethyl 3-methylbutanoate	fruity
g-nonolactone	coconut-like
2-octen-d-lactone	coconut-like, fruity
massoya lactone	coconut-like, fruity
furaneol	caramel-like, strawberry-like
ethyl-3-phenylpropanoate	floral, fruity
Floral notes	
linalool	floral
phenylacetaldehyde	honey-like, rose-like
ethyl phenylacetate	honey-like, rose-like
2-phenylethyl acetate	honey-like, rose-like
b-damascenone	floral
2-phenylethanol	honey-like, rose-like
Cinnamon/spicy notes	
ethylcinnamate	cinnamon-like

Aroma Compounds	Sensory description
Vanillin	
vanillin	vanilla-like
Cabbage, meaty notes	
dimethyl disulfide	cabbage-like
dimethyltrisulfide	cabbage-like
2-methyl-3-(methyldithio)-furan	cooked meat-like
Smoky/hammy notes	
2-methoxyphenol	medicinal, smoky
4-methyl-2-methoxyphenol	woody, smoky
3-methylphenol	medicinal, woody
4-methylphenol	medicinal, woody
4-ethylphenol	smoky, bacon-like
2,6-dimethoxyphenol	hammy, bacon-like
Musty, earthy notes	
3-isopropyl-2-methoxypyrazine	green, bell-pepper
3-isobutyl-2-methoxypyrazine	earthy, bell-pepper
Paper/cardboard notes	
E-2-nonenal	cucumber-like, cardboard
Sweaty, cheesy notes	
2-methylpropanoic acid	rancid, buttery
3-methylbutanoic acid	sweaty
2-methylbutanoic acid	sweaty
butanoic acid	rancid, cheese-like
Animal, fecal-like	
skatol	fecal-like

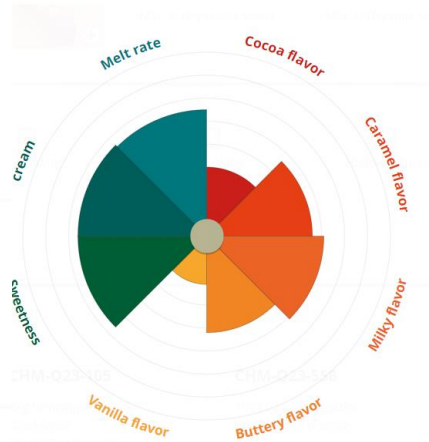
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Aroma Chemistry of Cocoa and Chocolate

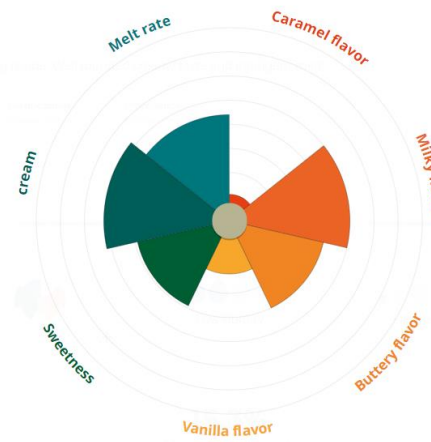
► Sensory evaluation of chocolates



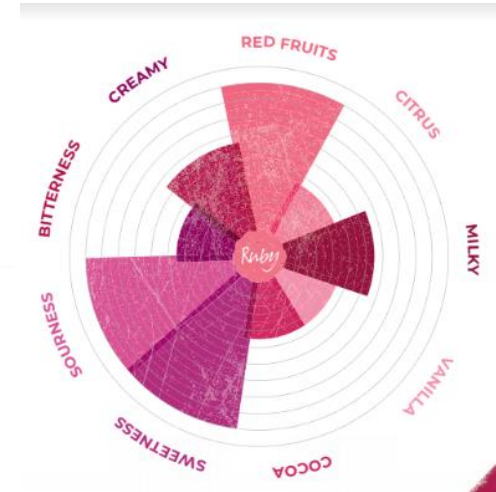
dark chocolate



milk chocolate



white chocolate



ruby chocolate

Source: <https://www.barry-callebaut.com>

Aroma analysis at BC

- ▶ Introduction

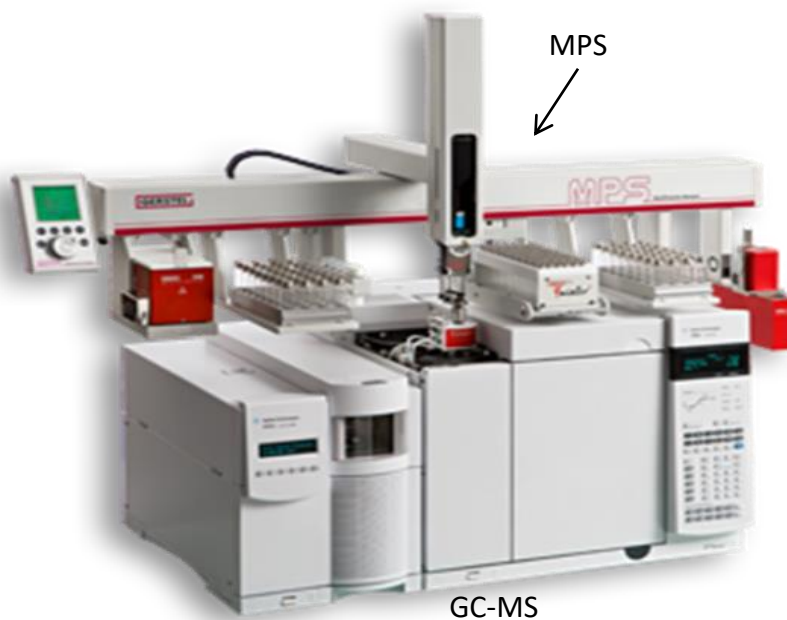
- ▶ Flavor

- ▶ Aroma: 80% responsible for final flavor

=> gas chromatography-mass spectrometry (GC-MS)

- ▶ Taste

- ▶ Texture



Aroma analysis at BC

- ▶ Configuration of GC-MS system (1)
 - ▶ MultiPurpose Sampler (MPS, Gerstel®)
 - ▶ Static Headspace Unit (SHS)
 - ▶ Direct Liquid extraction
 - ▶ Thermal Desorption Unit (TDU)
 - ▶ Twister (device for Stir Bar Sorptive Extraction (SBSE))
 - ▶ Direct thermal desorption of liquids or solids in micro-vials
 - ▶ Agitator and heater (heated conditioning and mixing/stirring)



Static Headspace Unit



Twister bar



Agitator-Stirrer



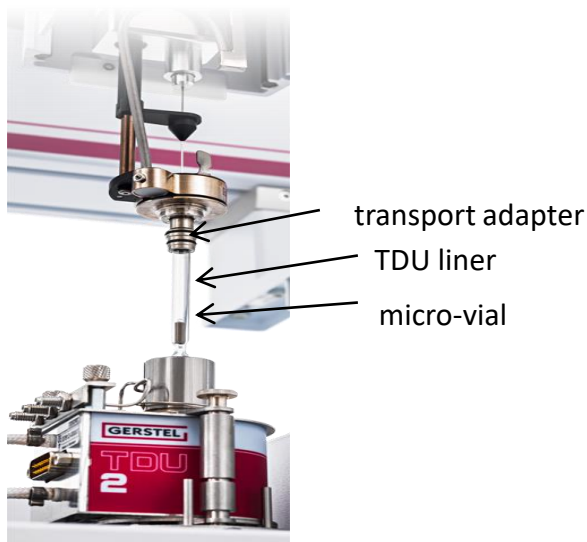
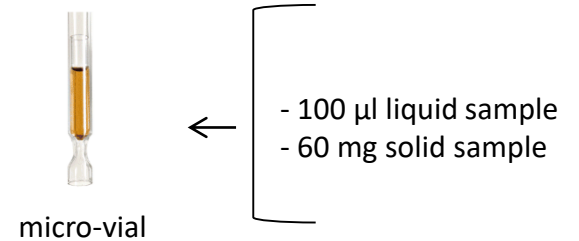
TDU with
Twister desorption



micro-vial
in TDU liner

Aroma analysis at BC

- ▶ Configuration of GC-MS system (2)
 - ▶ MultiPurpose Sampler (MPS, Gerstel®)
 - ▶ Thermal Desorption Unit (TDU)
 - ▶ TDU liner and transport adapter
 - ▶ Micro-vials (for liquid or solid samples)
 - ▶ Twister
 - ▶ Cooled Injection System (CIS, Gerstel®)



TDU (30 min desorption@70°C)



CIS (-10°C (1 min); 12°C/min to 280°C (20 min))

Aroma analysis at BC

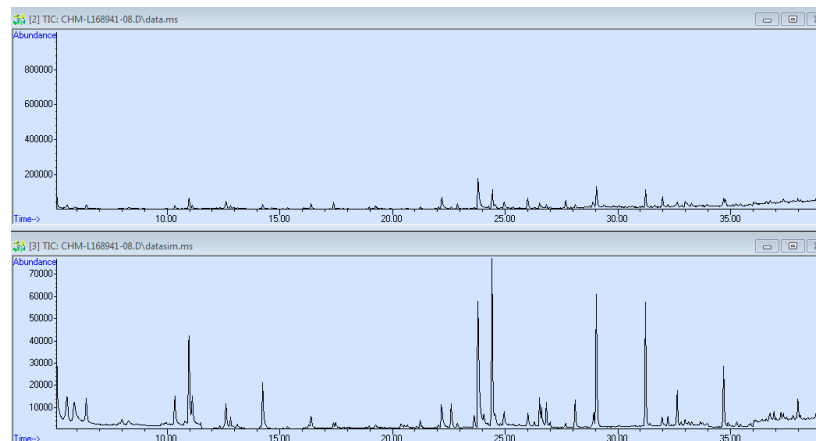
► Configuration of GC-MS system (3)

► ¹D GC-MS (7890A-5975C Agilent)

- Precolumn CS Fused Silica (2 m x 0.53 mm ID)
- Agilent J&W DB-FFAP GC Column (50 m x 0.32 mm x 0.50 μm)
- Agilent Ultimate Union Kits
- Single quadrupole MS
- EI source
- MassHunter software
- SCAN and SIM mode



Ultimate Union kit



SCAN and SIM mode simultaneous

Aroma analysis at BC

- ▶ GC-MS analysis of raw materials
 - ▶ Cocoa beans: of different geographical origins, of different qualities, screening of smokiness
 - ▶ Cocoa liquor: batch variability
 - ▶ Cocoa butter: batch variability
 - ▶ Cocoa powder: natural vs alkalized
 - ▶ Fats and cocoa butter: oxidation status
 - ▶ Milk powders: oxidation status
 - ▶ Additives: e.g. nut paste, flavors: different suppliers
 - ▶ Food contact materials

Aroma analysis at BC

- ▶ Impact of processing on the final flavor
 - ▶ Fermentation of cocoa beans
 - ▶ Roasting of cocoa beans/nibs: different roasting parameters (time, temperature, water)
 - ▶ Conching of chocolate: different conching parameters (time, temperature)
 - ▶ Batch variability of chocolate
- ▶ Impact of sugar reduction on final chocolate flavour
- ▶ Countertyping and Benchmarking
- ▶ Shelf life study: Impact of packaging material and storage conditions on flavor of raw materials and chocolate
- ▶ Off-odors and taints: identification of responsible off-flavor molecule(s) and characterization of possible cause of off-flavor

Aroma analysis at BC

- ▶ GC-MS-Olfactometry (GC-MS-O)
 - ▶ Identification of odor-active molecules
 - ▶ Identification of off-flavor molecules
 - ▶ Additional information for sensory analysis
 - ▶ Valuable information for GC-MS analysis on which compounds to focus on (GC-O simultaneous with GC-MS)

Conclusions

- ▶ Benefits of GC-MS analysis
 - ▶ Molecular insight in the aroma of food ingredients, flavors and food products
 - ▶ Molecular insight in the odor of food contact materials
 - ▶ Characterization of possible cause of off-flavor by identification of responsible off-flavor molecule(s)
 - ▶ Validation with sensory data (correlation)



Thank you for your attention!

