Chemometrics

Application Note

Chemometric Analysis of Chocolate

Headspace Mass Spectrometry

Abstract

In many manufacturing processes, one of the most critical quality control issues is to determine the consistency of the product from day-to-day or from plant-to-plant. Not only is this an issue of preserving integrity, but also if there is a minor shift in product attributes, it is important to determine its cause as quickly as possible so that waste is minimized.

Headspace mass spectrometry is able to detect differences among classes of chocolates (milk *vs.* dark *vs.* white) allowing us to see distinctions from manufacturer to manufacturer that reflects variation in process or ingredients. As implemented in the GERSTEL Headspace ChemSensor System, this technique is both rapid and reliable for screening samples in a routine quality control setting.

To demonstrate how batch-to-batch quality control can be exercised, we examined five manufacturing sources. Examples of white, milk and dark chocolates were collected from Ghirardelli, Guittard, Heritage Farms, Hershey's and Nestlé, with 4 chips (approximately 1.75 g) placed in 10 mL headspace vials and sealed. Runs were with 3 (45°C and 80°C) or 6 (125°C) replicates.

The first analysis was conducted at 45°C to look at low temperature volatiles. Although the sample repeatability was reasonable, there was a low signal-to-noise ratio (Figure 1).
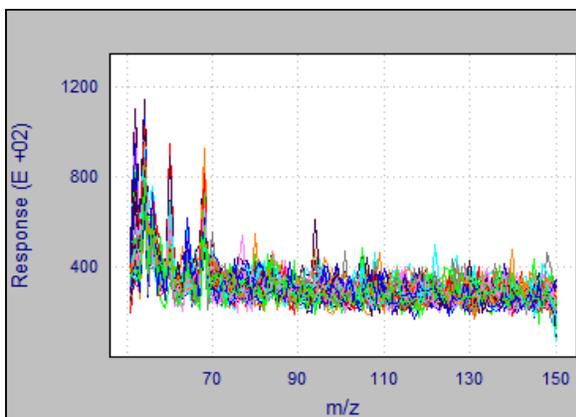

Figure 1. Chocolate samples show very little variation and low signal in mass spectra with heating at 45°C

Increasing the analytical temperature to 80°C provided complete separation of the chocolate types, as shown in Figure 2.
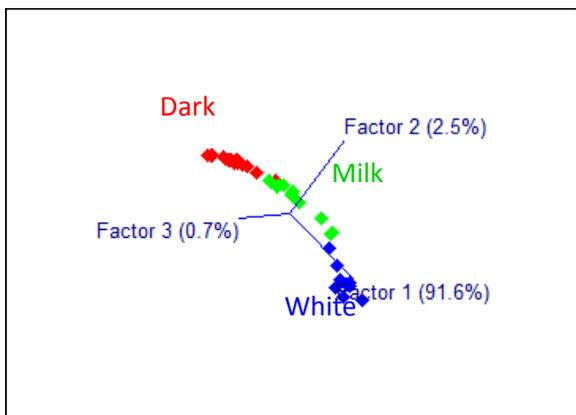

Figure 2. Chocolate samples differentiate slightly at moderate temperature (80°C) as shown in this PCA scores plot

At 125°C, the groups start to break out from the linear trend (white to milk to dark) and the samples cluster more strongly by manufacturer (Figure 3). Some thermal degradation of the white chocolate was in evidence.
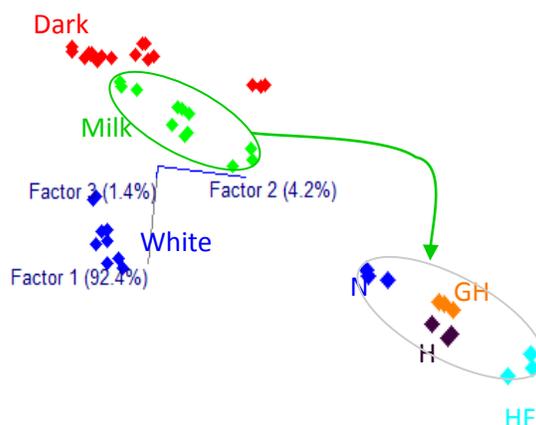

Figure 3. Increasing the temperature of heating to 125°C is better able to distinguish, not only between classes of chocolates, but also among the various manufacturers
N = Nestle
GH = Ghirardelli
H = Hershey's
HF = Heritage Farms

Mass Spectrometer Conditions	
Scan Mode	EI (BFBtune.u)
Run Time	0.75 min
Inlet Temperature	125°C
Mass Range	51-150 da

Headspace Conditions		
Temperatures	Oven	80°C
	Loop	85°C
	Transfer line	100°C
Time Events	Heating	30 min
	Interval between samples	5 min
	Pressurize	0.30 min
	Loop fill	0.15 min
	Loop Equil	0.02 min
	Inject	0.30 min
Pressures	Carrier	5.5 psig
	Vial pressurization	16.5 psig
Shaking	Low	