

# \*\*\*Qualification of Nutraceuticals

## Using Spectrum AssureID



### Introduction

Spectrum AssureID is a materials checking system which utilizes FT-IR and FT-NIR spectroscopy to generate sample specific 'fingerprints' of production materials. These fingerprints are compared using powerful classification algorithms to verify the identity and quality of each material.

For ease of implementation, AssureID mimics existing QA workflows, guiding users through method development, validation and trending and analysis of results. Expertise is built into the software to provide fast data modelling, troubleshooting and method validation without prior knowledge of chemometrics. AssureID also meets the stringent requirements of 21 CFR part 11 compliance.

This application describes the quality control of raw materials of a nutraceutical product using AssureID. Three components were qualified: acesulfame, aspartame and ascorbic acid. Two separate sets of ascorbic acid samples, from two different suppliers were analyzed.

### Creating a Method

Acceptance thresholds are established by measuring samples which define the potential quality limits of each raw material. This typically includes samples which are out of specification or at the limits of the specification, as well as previous batches of in-specification material. Samples of different materials which could be found in the same environment can also be usefully included.

### Modeling Algorithms

Once a database of typical samples has been created for each material AssureID can employ one of two algorithms, COMPARE™ and SIMCA (Soft Independent Modeling Class Algorithm), to classify new samples.

COMPARE determines the correlation between a sample spectrum and the average spectrum for the sample material. SIMCA is a chemometric approach which models the variation *within* the collection of reference spectra. For a given material, as well as the difference *between* spectra of different materials. This allows SIMCA to be sensitive to small spectral differences, for example impurities, even in the presence of normal batch-to-batch or sampling variations.

SIMCA is often the best initial choice when dealing with NIR spectra that are relatively featureless, and whenever there may be significant batch-to-batch variations within individual materials.

### Method

Fifteen spectra of each compound were collected using the Spectrum One NTS and NIRA accessory. Data was collected between 10,000 and 4000 $\text{cm}^{-1}$  at 16  $\text{cm}^{-1}$  spectral resolution. Six samples for acesulfame and aspartame and five each for the two ascorbic acid types were used for building the model, the others for validating the method. The SIMCA algorithm was chosen due to its compatibility with Near-IR spectral data.

Analysis of the overlaid spectra showed good overlap apart from the regions around 7300  $\text{cm}^{-1}$  and 5400  $\text{cm}^{-1}$ , where small traces of atmospheric absorption could be seen (Figure 1). However, a pre-processing filter function can be set to de-weight the influence of these absorptions on the model.

### Results

On modeling the data, two of the components were seen to overlap (Figure 2). Although classification performance was excellent, the inter-material distance for the two ascorbic acid components was quite small (1.99). This number provides a measure of the ability of the method to

Figure 1 Overlaid spectra of Acesulfame.

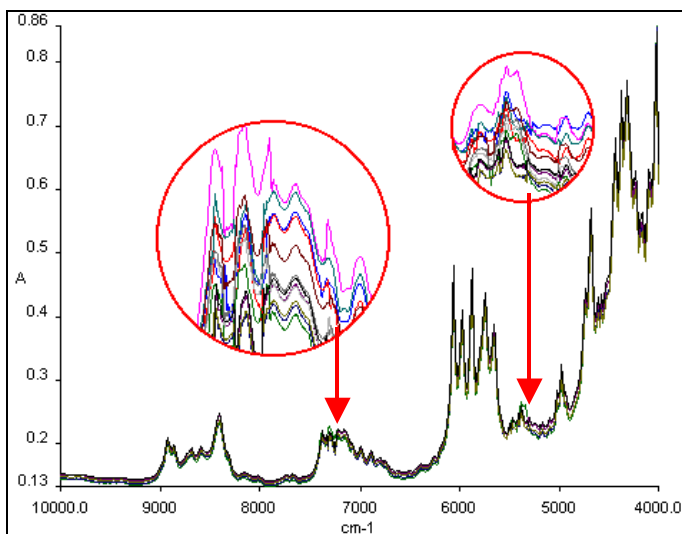
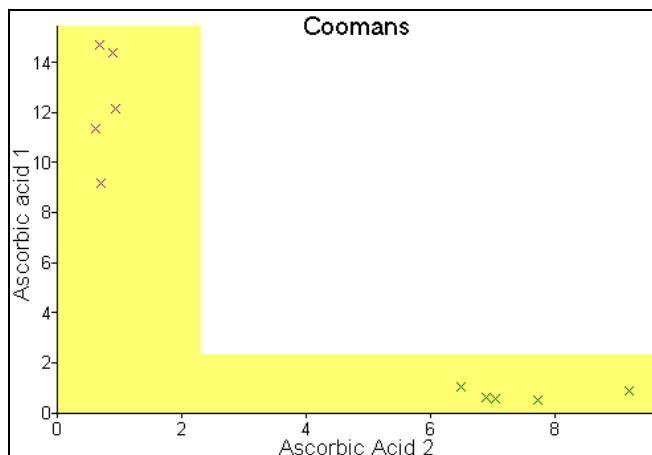


Figure 2 Model Summary.

Generate Model Summary				
Number of problem materials with extreme samples : 0				
Number of overlapped materials : 2				
Diagnostic Report				
Inter Material Distances				
	Acesulfame	Aspartame	Ascorbic acid 1	Ascorbic Acid 2
Acesulfame		51.4	48.2	51.1
Aspartame			21.8	26.6
Ascorbic acid 1				1.99
Classification Performance Report				
	% Recognition rate	% Rejection rate		
Acesulfame	100(6/6)	100(16/16)		
Aspartame	100(6/6)	100(16/16)		
Ascorbic acid 1	100(5/5)	100(17/17)		
Ascorbic Acid 2	100(5/5)	100(17/17)		

Figure 3 Coomans Plot.

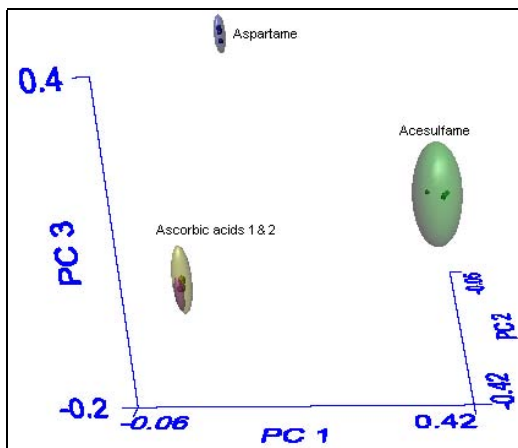


discriminate between materials. A figure over ten is normally seen as acceptable.

The Coomans plot (Figure 3) shows the degree of discrimination between spectra of individual materials in the model. This appears to show good separation between the two ascorbic acids, even though the model review showed that while the ascorbic acids were well separated from the other components (Figure 4a), they overlapped significantly with each other (Figure 4b). Model reviews indicate graphically how effective the model will be when used for the discrimination of new samples.

A series of samples for identification were then processed using the model. The results are shown in Figure 5. All samples were correctly identified.

Figure 4a Model Review for Components.



### Conclusion

AssureID provides rapid, unambiguous materials qualification without the need for expert knowledge of Chemometrics. Use of the SIMCA classification algorithm increases the robustness of methods by taking account of batch-to-batch and sampling variations in a model-based approach.

In the example given, all the different materials were clearly identified and qualified as suitable for use. As an extension of the work described, more samples should be included in the model to determine whether the ascorbic acids from the two supply sources can be differentiated more effectively.

Figure 5 Results for 5 'unknown' samples.

Sample View: My Views - Last 20 Results					
Database Results					
Sample ID	Analyst Name	Analysis Result	System Suitability	Method Name	Identified Material Name
AA72815_cs.sp	administrator	Pass	Pass	nutraceutical components check	Ascorbic acid 1
Asp79901019-20_cs.sp	administrator	Pass	Pass	nutraceutical components check	Aspartame
Asp7-990624-10_cs.sp	administrator	Pass	Pass	nutraceutical components check	Aspartame
Asp7990918-40_cs.sp	administrator	Pass	Pass	nutraceutical components check	Aspartame
Asp7991221-30_cs.sp	administrator	Pass	Pass	nutraceutical components check	Aspartame

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