Application Note 14
Discrimination of Black Peppers

By Olivia Koper and Ted Zhang December 2000

1. Introduction

The customer is interested in determining the country of origin of black pepper. In the spice industry, Malabar pepper is a premium spice while Vietnamese pepper is less costly. The goal in developing this method was to determine if the Cyranose 320 could be used to identify incoming shipments of peppercorns. The customer provided samples of Malabar and Vietnamese black peppercorns.

2. Experimental

Sample preparation:
The samples of pepper were sent in large sealed glass jars. One jar was sent of each pepper. Samples of Malabar and Vietnamese peppercorns were filled into 40-mL headspace vials fitted with a Teflon-lined septum. Each vial was half full. Five replicates were prepared for each sample. Fresh samples were prepared for prediction and all samples were kept in a laboratory environment at ambient conditions. The samples were analyzed at room temperature. The total sampling time was 3 minutes. A 45 second sample exposure time was chosen to ensure that all sensors reached equilibrium. Random sampling sequence was used to build the training set. The humidity was kept low by using syringes filled with drierite both on the purge inlet and sample venting needle as shown in Figure 1. To maintain a dry air system we purged the sample inlet with dry air using drierite in a 40-mL vial as shown in Figure 2.

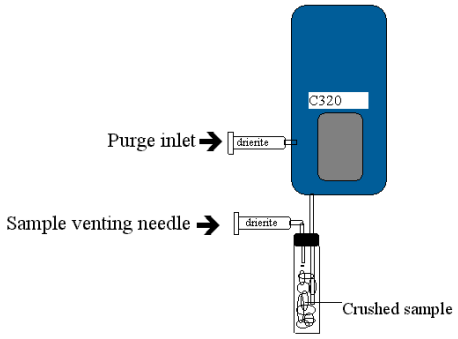

Figure 1. Drying air through purge inlet to establish baseline and drying air for sample vent.

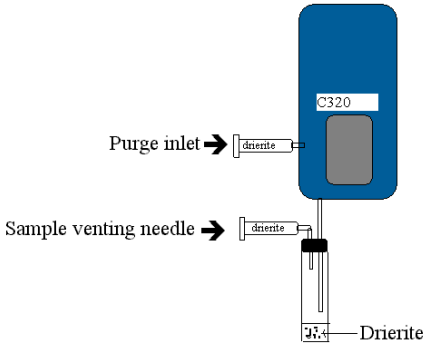

Figure 2. Purging the sample inlet with dry air.

Testing Conditions:
The Cyranose 320 with a 32 sensor array was used to test the peppercorns. Sensors 5, 6, 23 and 31, were deselected due to their sensitivity to polar compounds. The method settings used are in Table 2.

Data handling:
Data was recorded with the digital filter on. The sensor responses were calculated as $(R_{max}-R_{min})/R_{min}$ where R_{min} is the minimum of the resistance reading during the baseline purge and R_{max} is the maximum resistance reading during the vapor exposure. Canonical discriminant analysis (CDA), an algorithm for pattern recognition, with auto-scaling and 1-normalization was used for model making and predictions. Identification quality for predictions was set at medium.

3. Results

The principal components analysis (Figure 4) and canonical plot (Figure 5) clearly illustrate the discrimination of the two samples. The interclass distance and internal cross validation results of the two black peppers are shown in Figure 3. The first table in Figure 3 shows that all ten exposures to Malabar black pepper (BPM) were identified as Malabar black pepper using the CDA model. Similarly, the ten samples trained as Vietnamese black pepper (BPV) were correctly identified as Vietnamese black pepper. This led to 100% correct cross validation. The second table in Figure 3 gives an indication of how far apart the two pepper classes are in the model. The interclass distance was 6.1, which indicated good discrimination between the two black pepper samples.

The unit was used in Identify mode over a period of 13 days. The results are listed in Table 1. Identification quality was set at Medium.

Table 1. Identification Results of two black peppers over 13 days testing

	Malabar	Vietnamese
1st Day	5 sniffs. All correctly identified with ***** rating.	4 sniffs correctly identified with ***** rating 1 sniff incorrect
2nd Day	6 sniffs correctly identified with ***** rating 1 sniff correctly identified with *** rating 1 sniff (the 1st sniff of the day) incorrect	5 sniffs. All correctly identified with ***** rating
9th Day	5 sniffs. All correctly identified with ***** rating.	5 sniffs. All correctly identified with ***** rating.
13th Day	5 sniffs. All correctly identified with ***** rating.	5 sniffs. All correctly identified with ***** rating.

The black pepper samples were correctly classified 95% of the time. One sample of Malabar black pepper was incorrectly predicted due to the instrument not being warmed up. This was the first sniff of the day. One sample of Vietnamese pepper was also incorrectly predicted. All other samples were correctly predicted with high confidence suggesting that the incorrect prediction may have been a one-time event such as a

sampling error. The training set was externally validated during the 13 day period and was shown to be robust and reliable over this time. The experiment was terminated after 13 days, although the training set still remained robust.

4. Conclusions

A method for the incoming inspection of Malabar black pepper and Vietnamese black pepper was successfully created using the Cyranose 320. The method was used over a period of 13 days.

Table 2. Method setting used in the experiments.

Method name	Seasonings	
Class 1	Malabar Black Pepper (BPM)	
Class 2	Vietnamese Black Pepper (BPV)	
Baseline purge	20	Medium
Sample draw	45	Medium
Sample draw 2	0	
Snout removal	0	
1st sample gas purge	0	
1st air intake purge	5	High
2nd sample gas purge	90	High
2nd air intake purge	20	High
Digital filtering	On	
Substrate heater	On	32 °C
Training repeat count	1	
Identifying repeat count	1	
Active sensors	All except 5, 6, 23, 31	
Algorithm	Canonical	
Preprocessing	Autoscaling	
Normalization	Normalization 1	
Identification Quality	Medium	

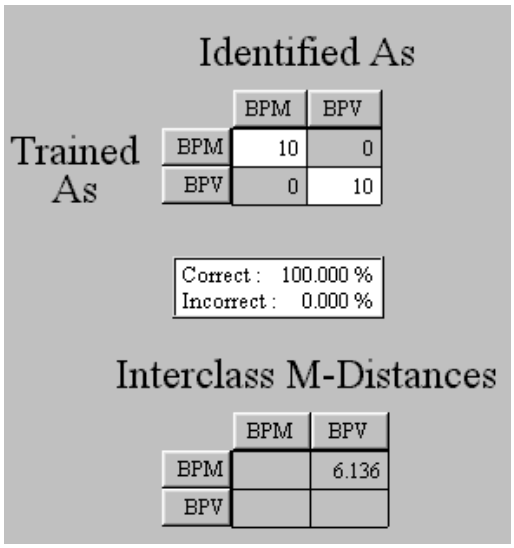

Figure 3. The internal cross validation of the training set of black peppers CDA, autoscale with 1-normalization.

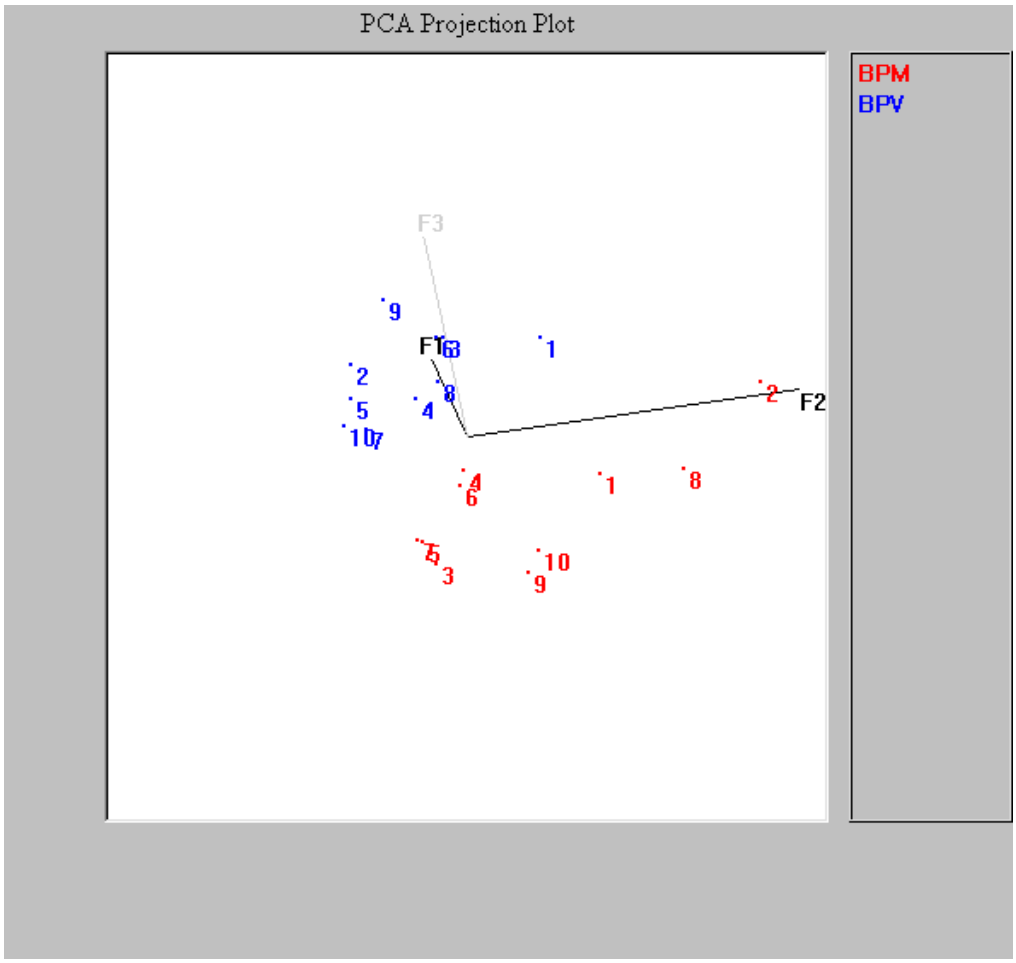

Figure 4. The PCA plot of the training set of black pepper using CDA, autoscale with 1-normalization.

Cyrano Sciences Inc, 73 N. Vinedo Avenue, Pasadena, CA 91107
Tel. 626-744-1700, Fax 626-744-1777
www.cyranosciences.com, info@cyranosciences.com

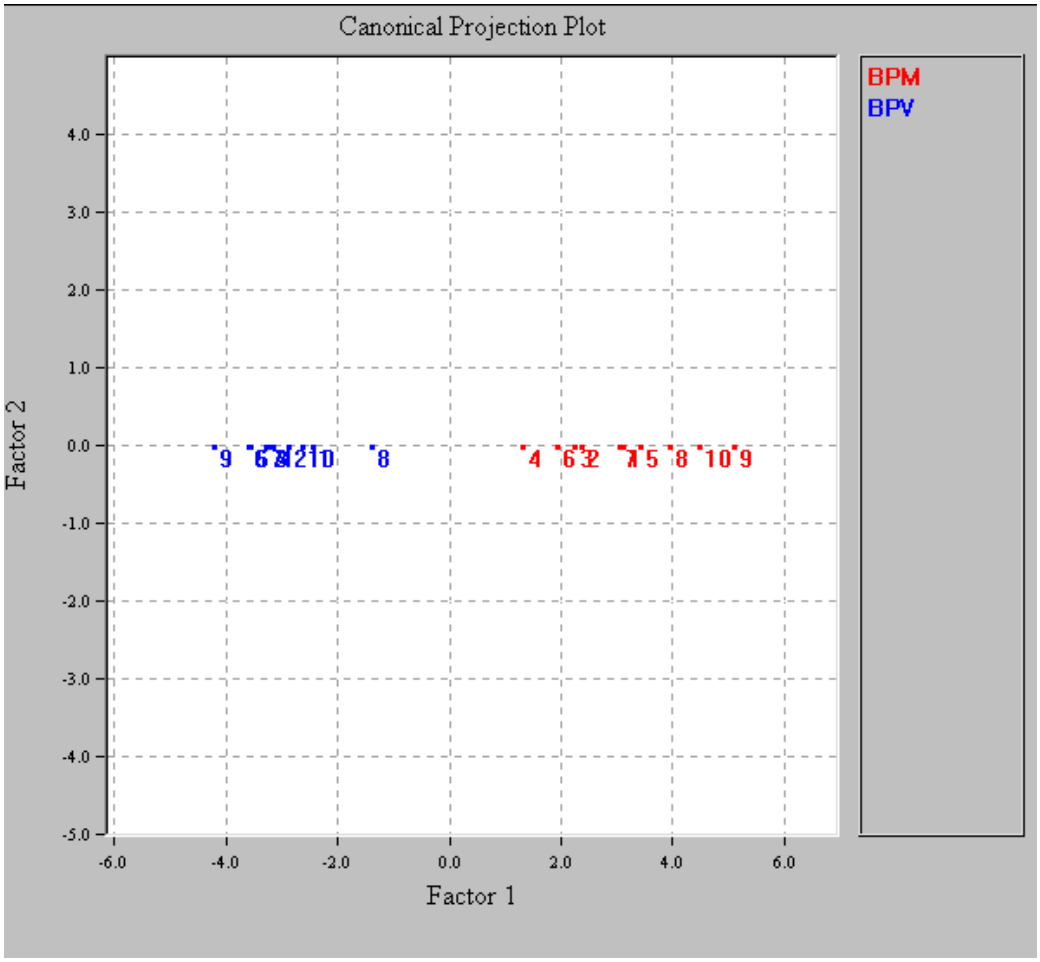

Figure 5. The CDA plot of the training set of black pepper using CDA, autoscale with 1-normalization.

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