

## IDENTIFICATION OF PURE AND BLENDED FOOD SPICES

Fourier Transform Near Infrared Spectroscopy (FT-NIR)  
(Fast, Accurate, Reliable, and Non-destructive)



The objective of this study was to evaluate the effectiveness of FT-NIR technology in the identification of a variety of pure and blended food spices. Initially, an FT-NIR reference library was created using four different spices consisting of Onion Powder, Paprika, Black Pepper (ground) and Nutmeg. In a follow up study, several mixtures of Onion powder and Paprika of different percentages and Paprika and Nutmeg, again, of different percentages were included in the reference library. This study demonstrates that the FT-NIR technology is not only capable of identifying pure spices but also the blends of spices.

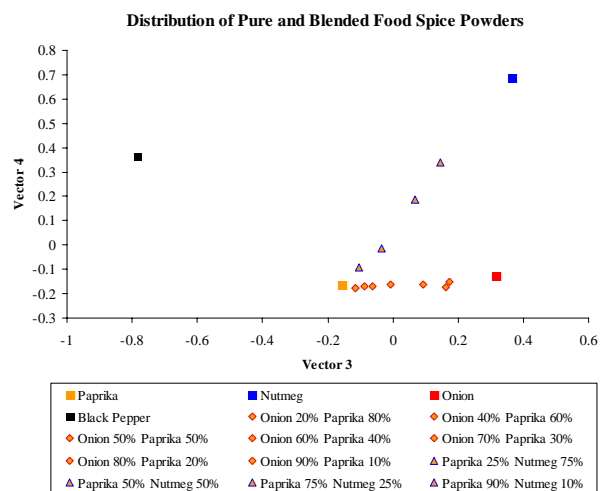
This technical note describes the application of FT-NIR technology in the easy identification of food spices. We hope that the readers will be able to see the simplicity and speed with which the FT-NIR technology can help in the identification of food spices and enhancement of Quality Control and Quality Assurance programs. Samples were tested as received with no sample preparation. A more general description of the FT-NIR technology can be found in Technical Note (TN 001 - Materials Identification).

**Scanning:** All spices were scanned using a fibre optic probe and the Bruker Vector 22/N or Matrix-F Fourier Transform Near Infrared Spectrometers.

**Scanning time:** Each measurement took about 5 seconds, and in most cases, five to ten measurements were taken for spectral averaging and identification purposes.

**Identification Process:** The outcome of the scanning process is an absorption spectrum. The NIR absorption and its characteristics are described in the Technical Note (TN 001 - Materials Identification). The focus of this technical note is the identification of food spices and the development of a spectral model for this purpose. In this particular study, all scanned powders of interest were used in developing an FT-NIR spectral reference model for the follow up identification purposes. Like many other spectroscopic techniques, the FT-NIR technique is a comparative technology, so, **once a reference spectral model is developed, identification of a test sample can be achieved within seconds!**

All absorption spectra for different spices and their blends were averaged and a Factorized Analysis of the averaged spectra was then carried out. The following graph shows the scatter of the four pure spices and several blends.



As can be seen from the above scatter graph, the four pure spices show up at four different coordinates (Vector 3 and Vector 4). Furthermore, the scatter graph also shows that as the concentration of Onion or Nutmeg are increased the coordinates of the blended powder move from pure Paprika towards the pure Onion or Nutmeg.

**Identification of test samples:** An individual Identity Test Report for each powder composed of four sections; 1) Sample Information, 2) Average Report, 3), Second Derivative Spectrum comparison to the respective reference spices, and 4) Identity Report was then generated. Several Identity Test Reports are shown in the following pages for different spices or their blends.

The first part of the Identity Test Report shows the sample and test information, i.e., what method file was used, the time and date of the test, and the result (see below for more details). The second part of the Identity Test Report is the average report. As mentioned above and in Technical Note (TN 001), on average, each spice powder was scanned at least five times and the resulting spectra were averaged. The average report contains the 'Hit Quality' value and the file name. Hit Quality is defined as a measure of the spectral distance between the average spectrum and the individual spectrum. The individual spectra are then sorted according to spectral distances in ascending order. Hit No. 1 is the sample spectrum that is the most similar to the average spectrum while the last spectrum in the list shows the greatest difference to the average spectrum.

Two parameters are derived from the spectral distance to define the confidence region for the average spectrum: Mean Distance,  $D_m$ ; and Standard Deviation,  $S_0$ . The closer the Hit Quality values the more consistent are the spectral measurements. These calculations are automatically carried out within the OPUS/IDENT software.

The third part of the Identity Test Report is the comparison of the second derivative spectrum of the average file and the matching reference i.e., Nutmeg, Onion, Paprika and Black Pepper. Depending on the chemical composition of each spice, the FT-NIR response would be different, as can be seen in the second derivative spectra.

The final and most important part of the report is the identity report and, in addition to Hit Quality values, it also contains threshold values for the references. The threshold values for each spice reference were automatically calculated within the OPUS/IDENT method development function. The threshold value is calculated from the worst hit (the greatest distance from  $D_{max}$  in the average report), the standard deviation  $S_0$ , and a probability factor in this case a 99% confidence interval. These parameters were also set during the method development using OPUS/IDENT software.

The Identity Report section is the result of the comparison between the spectral differences of the test spice powder with those of the references (in this case, four different spices and their blends). As a result of this comparison, OPUS software provides three possible outcomes:

- 1 **Identical:** If the Hit Quality for the test sample is lower than the threshold value of the reference sample and no other Hits are found to match this criterion, the result is reported as IDENTICAL; that is, the test sample is identical to the reference material.

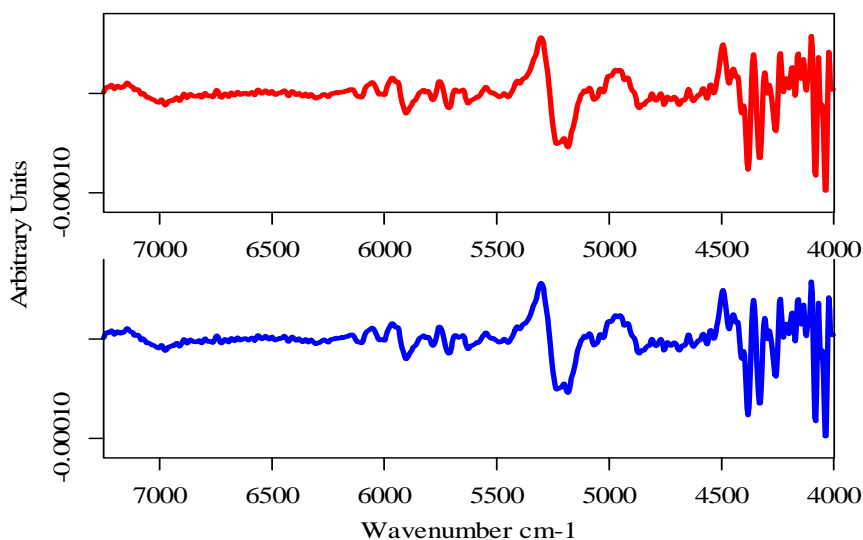
- 2 **It can be confused with <N> samples:** A second possible result of an Identity Test is CAN BE CONFUSED WITH <N> OTHER HITS. This result appears if Hit No. 1 is smaller than the threshold of the expected reference, but the Hit Quality of one or more other reference spectra are also found to be below this limit value.
- 3 **Not identical:** The third outcome is NOT IDENTICAL. In this case no match has been found for the test sample, therefore, the test sample is not identical to any of the references in the library. This situation may also arise if the sample is not scanned properly or the batch to batch variation is so different that the Hit Quality is greater than the threshold, therefore, it is not statistically identical even though chemically it may be a similar material.

Black Pepper (Ground)

Method file:	SPICES6NT.FAA
from (date):	4/06/03
(time):	16:36:26
Description:	
IDENTICAL TO: Black Pepper:	1
Hit quality with expected reference:	0.000000
Threshold for expected reference:	0.013308

Average Report

Hit No.	Hit Quality		File Name
1	0.271180	0.812311 * S.Dev	BLPEPPER.104
2	0.281230	0.842415 * S.Dev	BLPEPPER.101
3	0.295730	0.885848 * S.Dev	BLPEPPER.102
4	0.319002	0.955559 * S.Dev	BLPEPPER.103
5	0.322403	0.965747 * S.Dev	BLPEPPER.105



Hit No.	Hit Quality	Sample Name	File Name	Threshold
1	0.000000	Black Pepper   Av. of 5	BLPEPPER.100	0.013308
2	0.900150	Onion 60% Paprika 40%   Av. of 5	ONI6PAP4.100	0.018977
3	0.909365	Onion 50% Paprika 50%   Av. of 5	ONI5PAP5.100	0.010723
4	0.917703	Onion 40% Paprika 60%   Av. of 5	ONI4PAP6.200	0.010471
5	0.924332	Onion 70% Paprika 30%   Av. of 5	ONI7PAP3.100	0.015184

Identity Report

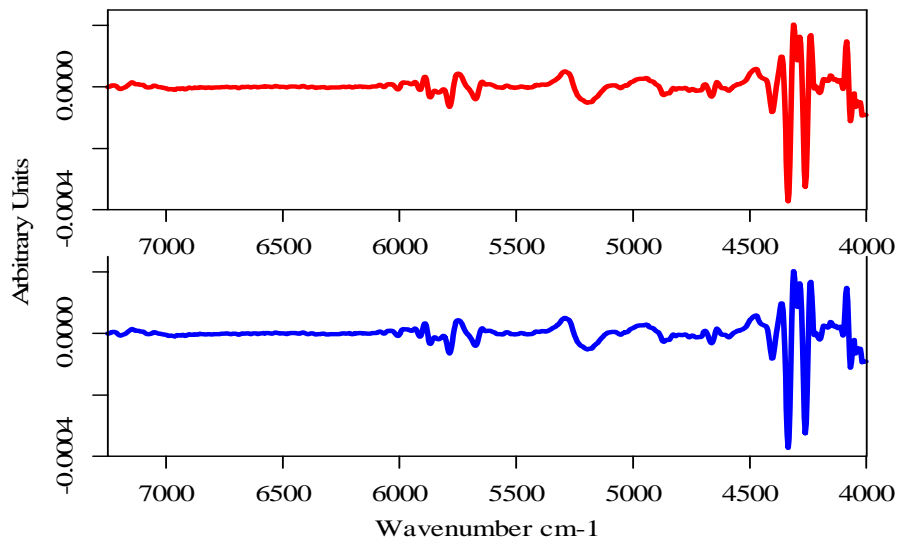
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Paprika

Method file:	SPICES6NT.FAA
from (date):	4/06/03
(time):	16:36:26
Description:	
IDENTICAL TO: Paprika:	1
Hit quality with expected reference:	0.000000
Threshold for expected reference:	0.005419

Average Report

Hit No.	Hit Quality		File Name
1	0.143714	0.791731 * S.Dev	PAPRIKA.105
2	0.148175	0.816308 * S.Dev	PAPRIKA.101
3	0.157583	0.868138 * S.Dev	PAPRIKA.104
4	0.167598	0.923312 * S.Dev	PAPRIKA.102
5	0.190433	1.04911 * S.Dev	PAPRIKA.103



Hit No.	Hit Quality	Sample Name	File Name	Threshold
1	0.000000	Paprika   Av. of 5	PAPRIKA.100	0.005419
2	0.054943	Paprika 90% Nutmeg 10%   Av. of 5	PPR9NUT.100	0.007074
3	0.064994	Onion 20% Paprika 80%   Av. of 5	ONI2PAP8.200	0.007795
4	0.127809	Onion 40% Paprika 60%   Av. of 5	ONI4PAP6.200	0.010471
5	0.128381	Paprika 75% Nutmeg 25%   Av. of 5	PR75NU25.100	0.009568

Identity Report

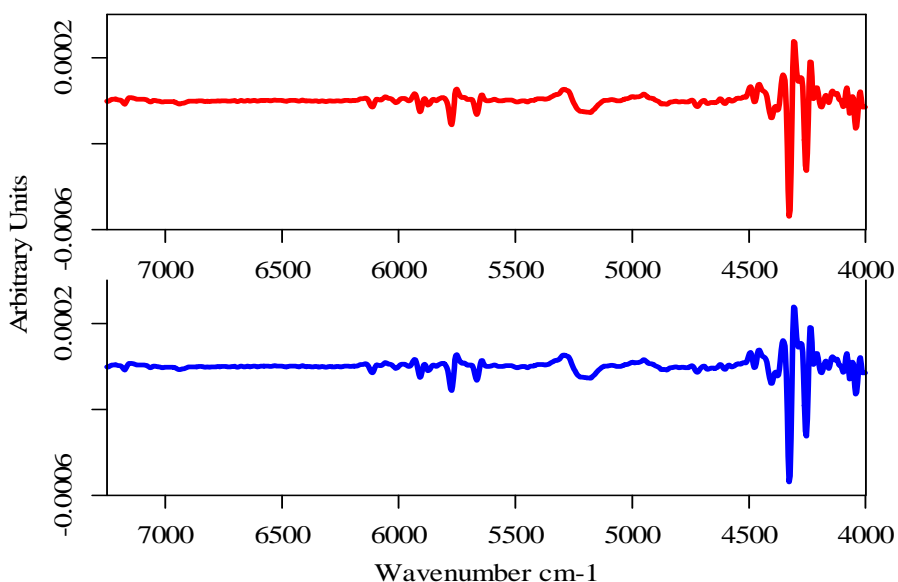
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# Nutmeg

Method file:	SPICES6NT.FAA
from (date):	4/06/03
(time):	16:36:26
Description:	
IDENTICAL TO: Nutmeg:	1
Hit quality with expected reference:	0.000000
Threshold for expected reference:	0.007003

## Average Report

Hit No.	Hit Quality		File Name
1	0.106161	0.766992 * S.Dev	RNUTMEG.101
2	0.112916	0.815793 * S.Dev	RNUTMEG.102
3	0.113250	0.818206 * S.Dev	RNUTMEG.103
4	0.131533	0.950301 * S.Dev	RNUTMEG.105
5	0.149951	1.08336 * S.Dev	RNUTMEG.104



Hit No.	Hit Quality	Sample Name	File Name	Threshold
1	0.000000	Nutmeg   Av. of 5	RNUTMEG.100	0.007003
2	0.223508	Paprika 25% Nutmeg 75%   Av. of 5	PR25NU75.200	0.006039
3	0.312812	Paprika 50% Nutmeg 50%   Av. of 5	PR50NU50.100	0.007030
4	0.357836	Onion 70% Paprika 30%   Av. of 5	ONI7PAP3.100	0.015184
5	0.379020	Onion 60% Paprika 40%   Av. of 5	ONI6PAP4.100	0.018977

## Identity Report

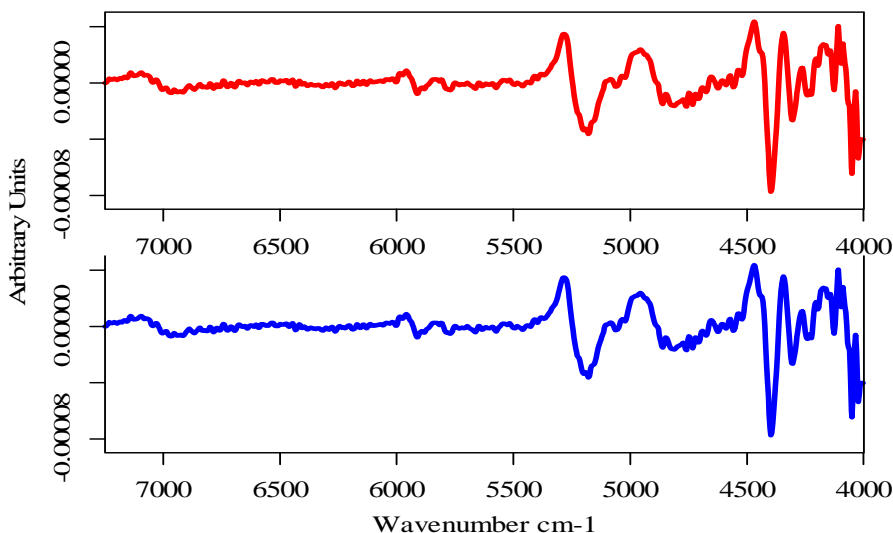
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Onion Powder

Method file:	SPICES6NT.FAA
from (date):	4/06/03
(time):	16:36:26
Description:	
IDENTICAL TO: Onion Powder:	1
Hit quality with expected reference:	0.000000
Threshold for expected reference:	0.022828

Average Report

Hit No.	Hit Quality		File Name
1	0.127728	0.680879 * S.Dev	RONION.105
2	0.155862	0.830852 * S.Dev	RONION.103
3	0.177283	0.945041 * S.Dev	RONION.101
4	0.181453	0.967272 * S.Dev	RONION.102
5	0.189215	1.00865 * S.Dev	RONION.104



Hit No.	Hit Quality	Sample Name	File Name	Threshold
1	0.000000	Onion   Av. of 5	RONION.100	0.022828
2	0.300927	Onion 90% Paprika 10%   Av. of 5	ONI9PAP1.100	0.013608
3	0.330528	Onion 80% Paprika 20%   Av. of 5	ONI8PAP2.100	0.015098
4	0.461823	Onion 70% Paprika 30%   Av. of 5	ONI7PAP3.100	0.015184
5	0.640818	Nutmeg   Av. of 5	RNUTMEG.100	0.007003

Identity Report

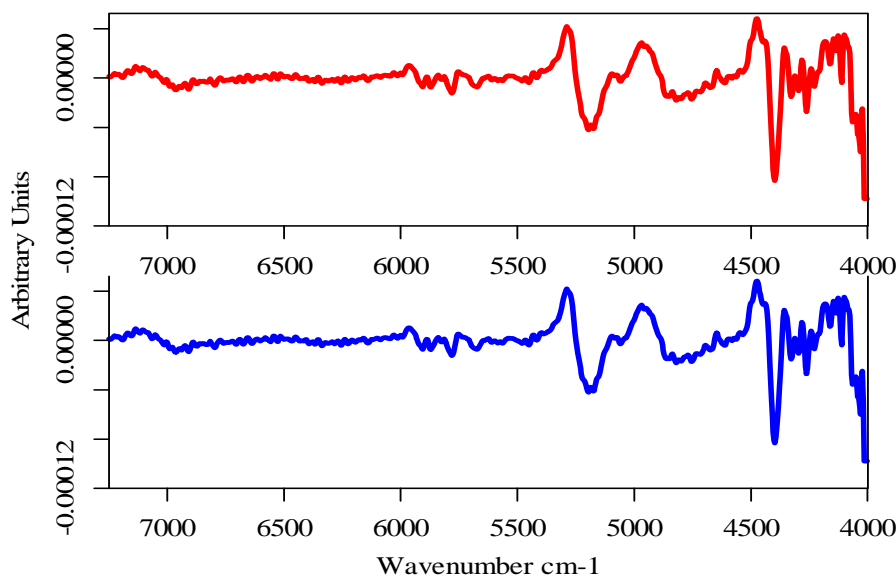
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Onion Powder 90% - Paprika 10%

Method file:	SPICES6NT.FAA
from (date):	4/06/03
(time):	16:36:26
Description:	
IDENTICAL TO: Onion Powder 90% Paprika 10%:	1
Hit quality with expected reference:	0.000000
Threshold for expected reference:	0.013608

Average Report

Hit No.	Hit Quality		File Name
1	0.253904	0.787632 * S.Dev	ONI9PAP1.104
2	0.253934	0.787725 * S.Dev	ONI9PAP1.102
3	0.271405	0.841919 * S.Dev	ONI9PAP1.103
4	0.297431	0.922655 * S.Dev	ONI9PAP1.101
5	0.352986	1.09499 * S.Dev	ONI9PAP1.105



Hit No.	Hit Quality	Sample Name	File Name	Threshold
1	0.000000	Onion 90% Paprika 10%   Av. of 5	ONI9PAP1.100	0.013608
2	0.030079	Onion 80% Paprika 20%   Av. of 5	ONI8PAP2.100	0.015098
3	0.160957	Onion 70% Paprika 30%   Av. of 5	ONI7PAP3.100	0.015184
4	0.300926	Onion   Av. of 5	RONION.100	0.022828
5	0.358402	Onion 60% Paprika 40%   Av. of 5	ONI6PAP4.100	0.018977

Identity Report

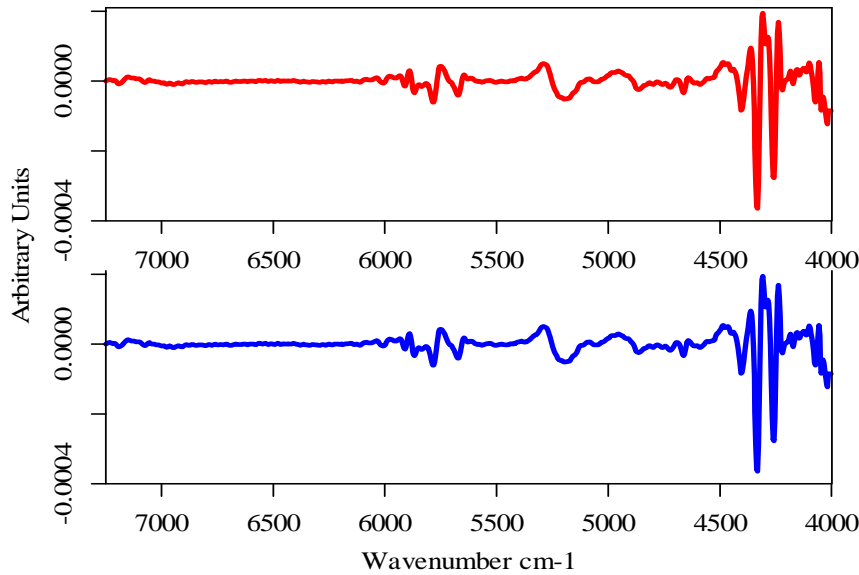
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Paprika 90% - Nutmeg 10%

Method file:	SPICES6NT.FAA
from (date):	4/06/03
(time):	16:36:26
Description:	
IDENTICAL TO:	Paprika 90% Nutmeg 10%: 1
Hit quality with expected reference:	0.000000
Threshold for expected reference:	0.007074

Average Report

Hit No.	Hit Quality		File Name
1	0.051039	0.745379 * S.Dev	PPR9NUT1.104
2	0.056310	0.822353 * S.Dev	PPR9NUT1.103
3	0.064182	0.937326 * S.Dev	PPR9NUT1.101
4	0.064398	0.940478 * S.Dev	PPR9NUT1.102
5	0.068647	1.00253 * S.Dev	PPR9NUT1.105



Hit No.	Hit Quality	Sample Name	File Name	Threshold
1	0.000000	Paprika 90% Nutmeg 10%   Av. of 5	PPR9NUT.100	0.007074
2	0.032200	Onion 20% Paprika 80%   Av. of 5	ONI2PAP8.200	0.007795
3	0.054944	Paprika   Av. of 5	PAPRIKA.100	0.005419
4	0.073500	Paprika 75% Nutmeg 25%   Av. of 5	PR75NU25.100	0.009568
5	0.090477	Onion 40% Paprika 60%   Av. of 5	ONI4PAP6.200	0.010471

Identity Report

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