Testing for robusta in Arabica coffee in minutes using Pulsar

Application Note 11

Introduction

Coffee is one of the most widely traded commodities in the world. The trade is made up of two main varieties, commonly known as Arabica and robusta, with Arabica accounting for 60-70% of the world market and robusta most of the rest. Arabica is generally considered to be of higher quality, and sells on world commodity markets for about twice the price of robusta. There is therefore the potential for economic fraud, with unscrupulous traders adding robusta to Arabica and still labelling the product "100% Arabica". Analytical methods are therefore needed to detect the presence of robusta coffee in products labelled as Arabica.

Methods

The official method for testing for the presence of robusta in Arabica is DIN 10779, which is a complex HPLC method that takes over 7 hours and involves the use of acetonitrile (a known carcinogen). Faster and easier methods are therefore desirable.

The HPLC method looks for the presence of a diterpene compound known as 16-O-methylcafestol (16-OMC), which is present in both varieties of coffee but only at trace levels in Arabica. The trace levels of 16-OMC in Arabica are not detected by the HPLC method, so any detection is deemed to be a result of adulteration of the sample with robusta.

It has long been known that high-field NMR can detect the presence of 16-OMC in coffee, but the use of high-field NMR for such a test is also complicated and expensive. So, researchers at the Quadram Institute Bioscience (formerly the Institute of Food Research) decided to see whether low-field (benchtop) NMR could be used instead. They used a 60MHz **Pulsar** to do this work, and quickly discovered that 16-OMC could be detected at 60MHz sufficiently well to offer the possibility of a method to detect adulteration of Arabica by robusta.

The Pulsar method

16-OMC has a number of clear peaks in the NMR spectrum, but one in particular, at 3.16ppm, is in a region where there are no interfering peaks from other compounds present in the samples. Figure 1 illustrates where this peak appears in relation to spectra of various coffee samples. The peak at 3.16ppm is labelled (i) in the figure. The researchers at Quadram showed that the integrated area around this peak correlates very well with the amount of robusta in a sample of Arabica, as shown in Figure 2, with R^2 of 0.99 and RMSE of 0.6% w/w.

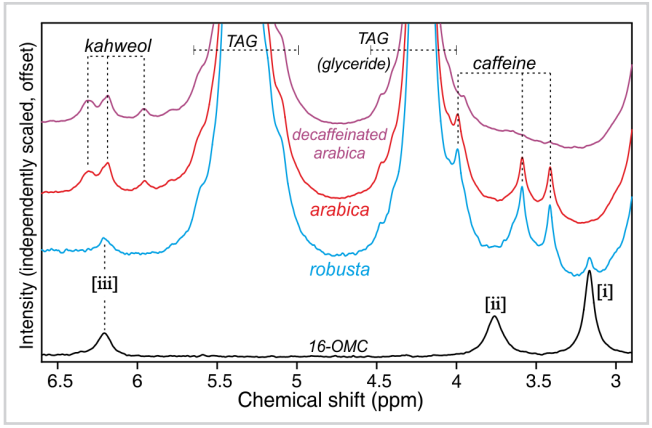

Figure 1: *NMR spectra of 16-OMC and various coffee samples*

As mentioned earlier, there are trace amounts of 16-OMC in Arabica, so allowance must be made for these naturally-occurring levels when trying to assess potential adulteration with robusta.

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Although the trace amounts vary from sample to sample, they are generally equivalent to an addition of approximately 1% of robusta into Arabica, so this is the lower limit of detection of adulteration. The complete statistical analysis generated by the Quadram researchers indicates that Arabicas adulterated with robusta at the 1% w/w level will be detected in about half of all cases. This rises to 90% at the 2% level, and at the 3% level it is unlikely that any adulterated sample will pass undetected.

Experimental method

To carry out this measurement using **Pulsar**, 10g of ground coffee beans are stirred with 30ml chloroform for 5 minutes, then filtered and dried in a vortex evaporator for 30 minutes. The dried extract is then re-dissolved in 800µl of deuterated chloroform and filtered directly into standard 5mm NMR tubes. Measurement time in **Pulsar** is approximately 40 minutes, making a total preparation and measurement time of approximately 90 minutes per sample.

An alternative method is available, dissolving 1g of sample directly into deuterated chloroform and then filtering into the NMR tube. Total measurement time for this method is about 45 minutes per sample, but because the concentration step is missed the detection limit for robusta in Arabica is higher. Samples containing at least 20% robusta are very likely to be detected, and a substantial proportion of samples containing only 10% robusta will be identified.

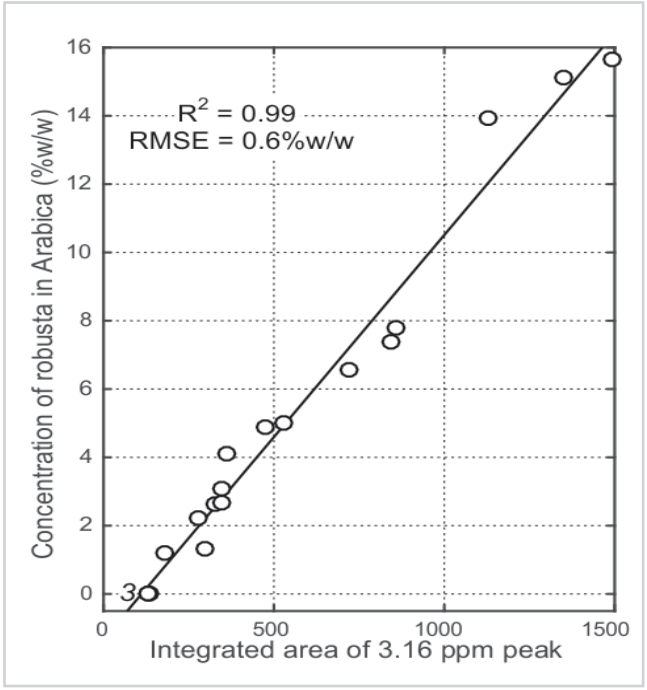

Figure 2: *Calibration of robusta in Arabica, using integrated area around 3.16ppm*

Acknowledgements

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