

**ADDENDUM  
TO  
FINAL PROGRESS REPORT  
MARCH 2007**

**PROJECT TITLE**

**Quantification of Fumonisin in South African Maize by means of Near Infrared Spectroscopy**

**Collaborators:**

Medical Research Council:

Programme on Mycotoxins and Experimental Carcinogenesis (PROMEC Unit), Medical Research Council, PO Box 19070, Tygerberg 7505.

Tel: 021-938-0279

Fax: 021-938-0260

Stellenbosch University

Department of Food Science, Faculty of AgriSciences, Private Bag X1, Matieland 7602, South Africa

Tel: 021-808-3511

Fax: 021-808-3510

**Project Participants:**

Dr GS Shephard (PROMEC; e-mail: [gordon.shephard@mrc.ac.za](mailto:gordon.shephard@mrc.ac.za))

Dr M Manley (Department of Food Science, US; email: [mman@sun.ac.za](mailto:mman@sun.ac.za))

## Introduction

Due to the fact that both Evan Springfield and Pumza Gatyeni, who were project leader and responsible for the HPLC analyses, respectively, resigned from their positions at the MRC last year it was not possible to report back on the final results obtained. The final results obtained are reported in this addendum.

## Materials and Methods

### *Samples and sample preparation*

Ground maize samples (n = 71), with known fumonisin content (Crop Quality Survey Samples 2004/2005), were obtained from the Southern African Grain Laboratory (SAGL).

### *Reference methods for NIRS calibration model development*

#### Quantification of fumonisin using the VICAM Fluorometer

Fumonisin content was determined at the SAGL by means of the VICAM Fluorometer test (n = 71) according to the VICAM Fumoni Test Instruction Manual (November 2002). Results of single analyses have been reported in ppm ( $\text{mg}\cdot\text{kg}^{-1}$ ).

#### Quantification of fumonisin using HPLC

Total fumonisin content was additionally determined using HPLC (n = 71) for verification purposes, according to the method as described by Sydenham *et al.* (1996). Results of single analyses have been reported in ppm ( $\text{mg}\cdot\text{kg}^{-1}$ ).

### *Near infrared spectroscopy measurements*

#### Collection of spectra

A Büchi NIRLab N-200 Fourier transform near infrared (FT-NIR) spectrophotometer with NIRLabWare (version 3.0) near infrared (NIR) measurement software was used to perform the NIRS measurements in diffuse reflectance mode. The ground maize samples were presented to the instrument in rotating glass petri-dishes and the NIR spectra collected from 1100-2500 nm.

#### NIRS calibration model development

Unscrambler v9.2 was used for calibration model development on spectra (n = 71) pre-treated with MSC using the VICAM Fluorometer as well as HPLC test results as reference data. The calibration model was validated by means of full cross-validation (leave-one-out) and no outliers were removed.

## Results and Discussion

### *NIRS calibration model development*

A summary of the reference data for fumonisin content is given in **Table 1** and the NIRS calibration model and full cross-validation results for the prediction of fumonisin in ground maize in **Table 2**. Detailed reference data are listed in Attachment 1. It is clear from the results in **Table 2** that it is not possible to determine fumonisin in ground maize by NIRS with the calibration model developed and the data currently available. **Figure 1** illustrates the correlation between the two reference methods.

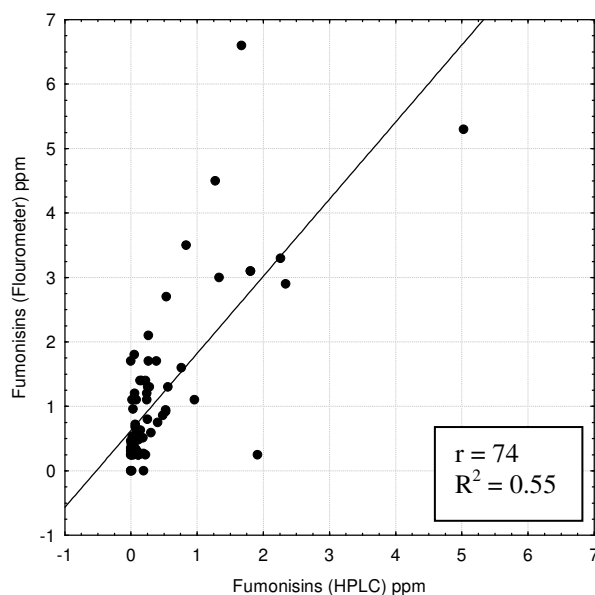
**Table 1** Summary of reference data for prediction of fumonisin content.

	Fumonisin VICAM Fluorometer	Fumonisin HPLC
<b>n</b>	73	73
<b>Range (ppm)</b>	0 – 6.600	0 – 5.300
<b>Mean (ppm)</b>	1.16	0.43
<b>Standard deviation (SD)</b>	1.27	0.79

**Table 2** Summary of the NIRS calibration and full cross-validation results for the prediction of fumonisin content in ground maize.

	Fumonisin (n=73) VICAM Fluorometer	Fumonisin (n=73) HPLC
<b>SEC (ppm)</b>	1.249	0.770
<b>R<sup>2</sup></b>	0.020	0.028
<b>RMSECV (ppm)</b>	1.290	0.759
<b>R<sup>2</sup></b>	0.010	0.040
<b>Bias</b>	-0.002	-0.001
<b>PLS factors<sup>a</sup></b>	1	1
<b>RPD</b>	1.017	0.991

<sup>a</sup> Number of PLS factors used.



**Figure 1** Correlation plot between fumonisin data as determined by HPLC and the Fluorometer test.

## Conclusions

After having analysed all the available samples results still do not look promising. With this project it has been attempted to quantify very low levels (ppm) not normally attempted by using NIRS and the small range in fumonisin content was an additional limiting factor.

**Attachment 1.**

Reference data (fumonisin content) as determined by HPLC and VICAM Fluorometer.

<b>Fumonisin (HPLC) (ppm)</b>	<b>Fumonisin (Fluorometer) (ppm)</b>
0.240	1.100
0.065	0.720
0.000	0.250
0.055	0.440
0.759	1.600
0.262	2.100
0.049	0.280
0.180	0.510
0.000	0.250
0.000	0.350
0.039	0.250
0.000	0.310
0.190	0.000
0.000	0.000
0.042	0.310
0.103	0.290
5.028	5.300
0.241	1.200
0.063	0.560
0.835	3.500
0.057	1.200
0.071	0.330
0.000	0.000
0.019	0.340
0.524	0.940
0.190	0.270
0.118	0.250
0.116	0.250
0.023	0.530
2.262	3.300
0.075	0.330
1.671	6.600
1.914	0.250
0.052	1.800
0.000	1.700
0.117	0.480
0.000	0.460
0.079	1.100
0.483	0.860
0.092	0.600
0.017	1.100
0.053	0.330
0.382	1.700
0.006	0.410
0.160	1.400
0.013	0.000
0.105	0.250
0.015	0.250

	0.533	2.700
	0.278	1.300
	0.255	1.300
	0.303	0.590
	0.137	1.400
	0.525	0.920
	2.339	2.900
	0.116	0.250
	1.276	4.500
	0.559	1.300
	0.020	0.250
	0.959	1.100
	0.405	0.750
	0.252	0.800
	0.068	0.680
	0.222	1.400
	0.222	0.250
	1.331	3.000
	1.807	3.100
	1.807	3.100
	0.264	1.700
	0.029	0.960
	0.142	0.630
<b>Mean</b>	<b>0.43</b>	<b>1.14</b>
<b>SD</b>	<b>0.79</b>	<b>1.26</b>
<b>Minimum</b>	<b>0.000</b>	<b>0.000</b>
<b>Maximum</b>	<b>5.028</b>	<b>6.600</b>