Sample preparation procedures for use in near infrared analysis of fruit and vegetable crops

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Introduction

Over the last decade, research has been conducted at the Yanco Agricultural Institute into the use of near infrared (NIR) reflectance spectroscopy to determine the nitrogen concentration in various crop tissues.[1–3] Here we report the extension of this technology to the viticultural and horticultural industries.

The Australian grape and wine industry is mainly located in the states of New South Wales, Victoria and South Australia with plantings in Southern Queensland, Tasmania and the Perth area of Western Australia. Fertilizer recommendations for grapes are based on an interpretation of soil and tissue analyses, with tissue nitrogen being one of the most critical with regard to the production of optimum yields of high quality fruit for wine making or dried fruit.[4,5] At present, tissue nitrogen is determined by chemical laboratory procedures. Horticultural industries are widely distributed in Australia but well represented near our Institute.

Materials and methods

Grape leaf and petiole samples of 15 cultivars were collected from a number of wine growing areas that included vineyards in the Hunter Valley, Griffith, Wagga Wagga and Leeton areas in New South Wales; the Barossa Valley, Riverland and Adelaide Hills in South Australia; near Rutherglen and Merbein in Victoria; the Perth area in Western Australia and the Hobart and Pipers Brook areas of Tasmania. Samples of citrus, potato and tomato leaves were collected at our institute and surrounding farms.

Samples were dried in plastic mesh baskets using a microwave oven (domestic 700 Watts). The base of the plastic basket was raised from the oven turntable by placing it inside another plastic basket. This allowed the movement of water in all directions away from the sample during drying. Samples of most crops are dried using full power. Drying time is only a few minutes but varies with the amount of tissue. Samples were turned half way through the drying period to prevent charring or burning. Leafy vegetables such as cabbage and lettuce required cutting and shredding prior to microwave drying to ensure that hot spots did not develop in the sample and charring did not occur. A domestic kitchen shredder was used to pre-process these samples prior to microwaving. A sheet of fibreglass mesh was used to cover the bottom and sides of the plastic basket to ensure that small leaf particles were not lost.

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Dried samples were ground to pass a 0.5 mm screen in a Cyclotec mill. These were then analysed for total nitrogen using a Kjeldahl digestion of sulphuric acid, potassium sulphate and selenium,[6] followed by spectrophotometric determination of ammonium using a flow injection analyser.[7]

NIR spectra of the dried ground samples were recorded as log(1/*R*) with a scanning spectrometer (NIRSystems model 6500). Calibrations were developed with multiple linear (MLR) and partial least squares (PLS) regression using NSAS software.

Results and discussion

Grape vine leaf and petiole calibrations

A universal NIR calibration was developed by combining leaf and petiole calibration sets containing samples from all cultivars and geographic regions (Figures 1 and 2).

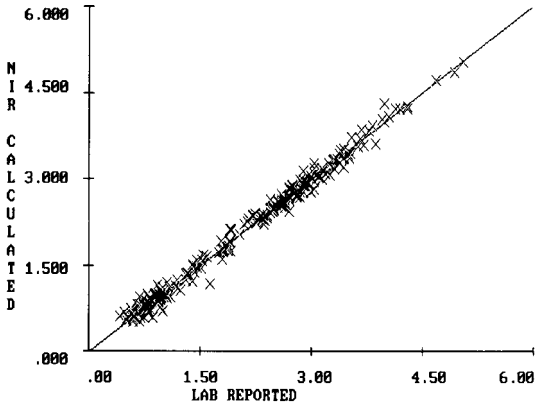

Figure 1. NIR calibration equation developed for nitrogen using both grape leaf samples and petiole samples. (*n* = 213; R^2 = 0.99; *SEC* = 0.11%N).

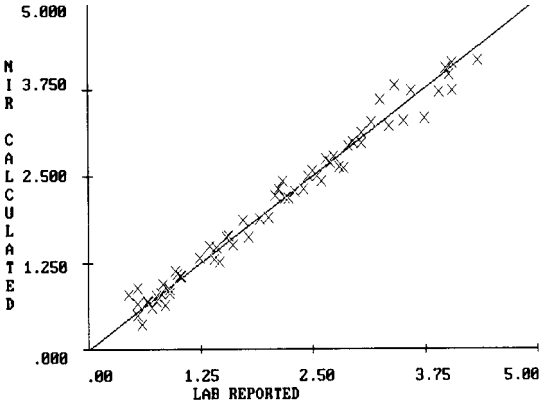

Figure 2. Verification equation for nitrogen in leaf samples and petiole samples. (*n* = 64; R^2 = 0.98; *SEP* = 0.16%N).

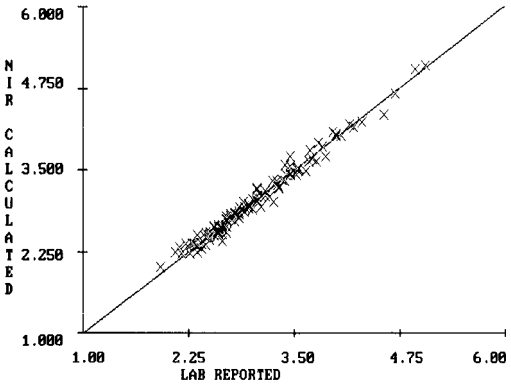

Figure 3. NIR calibration equation developed for nitrogen using leaf samples from the entire database (*n* = 108; R^2 = 0.98; *SEC* = 0.10%N; *SEP* = 0.17%N).

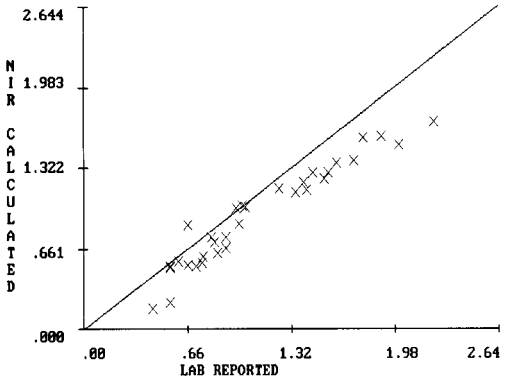

Figure 4. Verification equation for nitrogen in petiole samples using the leaf calibration equation (*n* = 64; R^2 = 0.96; *SEP* = 0.14%N).

Calibrations were also developed for leaf samples and petiole samples, e.g. Figure 3. However, the leaf calibration was not a good predictor of petiole nitrogen (Figure 4).

Calibrations were developed using leaf or petiole samples from only one region, from one cultivar across all regions, and from many cultivars across all regions at the flowering stage. The accuracy of these calibrations was no better than those illustrated in Figures 1 and 2. Preliminary calibrations have also been developed by Hutton *et al.*[3] for a number of elements, including phosphorus and potassium in petioles. The current set of samples will be used to improve and verify these calibrations.

Vegetable crop calibrations

Potato leaves gave a good nitrogen calibration, as did three leafy brassica vegetables (cabbage, broccoli and cauliflower) combined [Figure 5(a)–5(b)].

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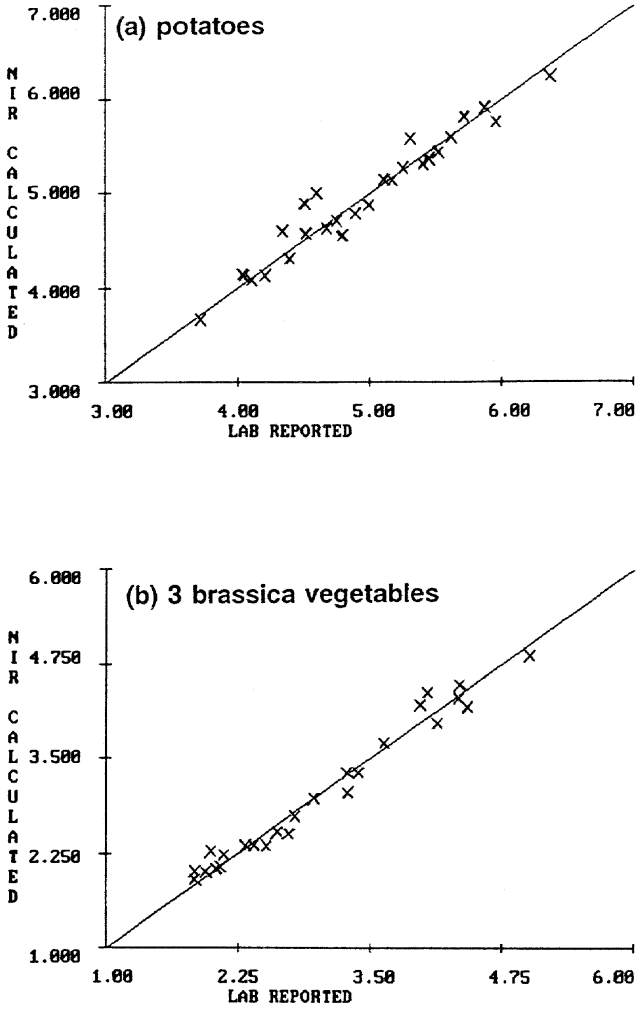

Figure 5. NIR calibration equations developed for nitrogen in (a) potatoes and (b) three brassica vegetables.

Conclusions

This paper demonstrates that:

- By using chopped leaf tissue, where necessary, the microwave drying technique developed for cereal crops can be adapted to the preparation of samples from horticultural crops;
- NIR can be used to determine nitrogen and mineral nutrients in horticultural crops; and
- A tissue testing service of the type outlined in Figure 6 could be established to assist horticulturists.

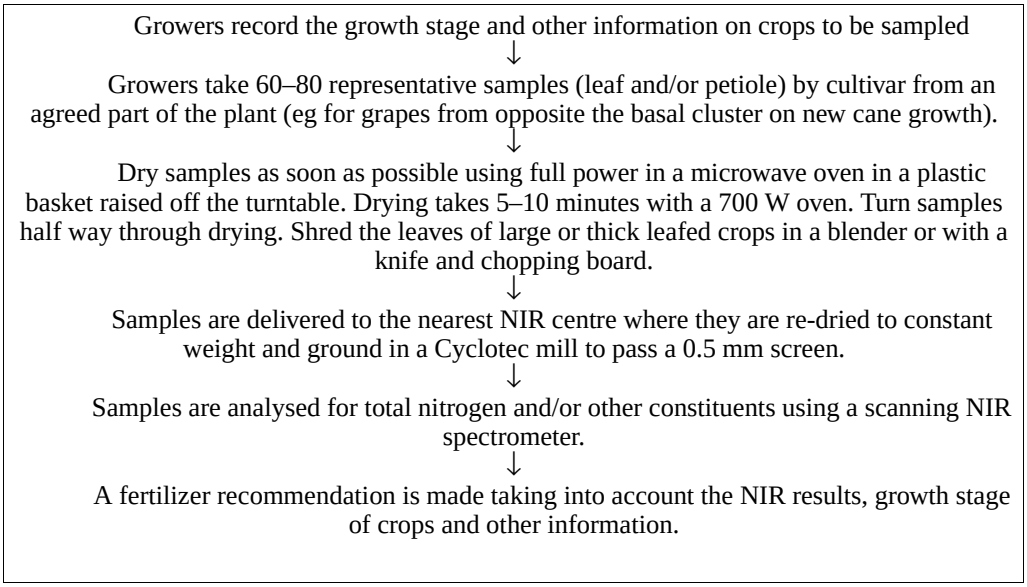

Figure 6. Flow diagram showing possible NIR tissue tests for horticultural crops.

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References

1. G.D. Batten and A.B. Blakeney, *NIR news* **2(4),** 10 (1991).
2. A.B. Blakeney, G.D. Batten and S. Ciavarella, "An Interactive Database for Use with the Rice Tissue Test Service", in *Temperate Rice—Achievements and Potential*, Ed by E. Humphreys, E.A. Murray, W.S. Clampett and L.G. Lewin. NSW Agriculture, Griffith, pp. 477–484 (1994).
3. R.J. Hutton, P.J. Sinclair, A.B. Blakeney and G.D. Batten, "Development of NIR-based Analysis for Fruit and Vegetable Crops", in *Leaping Ahead with Near Infrared Spectroscopy* Ed by G.D. Batten, P.C. Flinn, L.A. Welsh and A.B. Blakeney. Near Infrared Spectroscopy Group, RACI, North Melbourne, pp. 256–260 (1995).
4. A. Loué, J. Gagnard and P. Morand, "Vines", in *Plant Analysis as a Guide to the Nutrient Requirements of Temperate and Tropical Crops*, Ed by P. Martin-Prevel, J. Gagnard and P. Gautier. Lavoisier Publications Inc., New York, pp. 175–206 (1987).
5. J.B. Robinson, "Grapevine Nutrition", in *Viticulture, Vol. 2: Practices*, Ed by B.G. Coombe and P.R. Dry. Winetitles, Adelaide, pp. 178–208 (1992).
6. R.B. Bradstreet, *The Kjeldahl Method for Organic Nitrogen.* Academic Press, New York (1965) .
7. M. Van Son, R.C. Schothorst and G. Den Boef, *Analytica Chimica Acta* **153,** 271 (1983).