

## Developments in Glucosinolate Analysis in the UK

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The most pressing problem in relation to glucosinolate analysis is the need to have a method which is consistently reproducible between laboratories and which gives results as close as possible to those obtained by the EC-reference method, currently temperature programmed gas-liquid chromatography (TPGLC). It is also vital in the UK that the method is rapid so that it can be applied to samples arriving at the crushing mill before the load is tipped. Following experience of the 1987-harvest, the UK Rapeseed Liaison Committee felt that it was desirable to compare some of the "rapid" methods available with particular regard to reproducibility within and between laboratories. Accordingly a collaborative experiment was organised by the National Institute of Agricultural Botany (NIAB) in which 6 methods were included. These were as follows:

- a) TPGLC - the current EC-reference method.
- b) High performance liquid chromatography (HPLC) - the reference method proposed by a "Committee of Experts" set up by the Commission.
- c) A glucose release method developed at the Food Research Institute (FRI), Norwich, UK, - a UK registered method in 1987.
- d) A glucose release method developed at the UK Unilever research facility - a UK registered method in 1987.
- e) A glucose release method developed at the NIAB - the UK National Variety testing method since 1978.
- f) X-ray fluorescence (XRF) - a German registered method in 1987.

The results showed that XRF was the best method for reproducibility within laboratories and repeatability between laboratories. The NIAB-

method was also better in these respects than the chromatographic methods whereas the Unilever and FRI-methods were worse. Some difficulties in the chromatographic techniques were highlighted relating to the absence of pure reference samples of individual glucosinolates and the subsequent problems of interpretation of data. This is an area still outstanding in relation to the protocol of the EC-reference method and needs urgent attention to maximize consistency across member-states.

In terms of speed of analysis XRF was again best with an analysis time of about 4-5 minutes. The Unilever method took 15-30 minutes and the NIAB and FRI-methods require at least 2 hours.

As a result of this exercise, the UK decided to register the XRF-method as a "rapid" method for harvest 1988. It was also felt desirable to register an alternative "wet" chemistry method for those organizations not able to adopt XRF in view of the high cost of equipment. A major criterion in the choice of method was speed of analysis and despite the fact that it was not the best method technically, as shown in the collaborative experiment, the Unilever method was felt to be the only acceptable alternative for logistical reasons. Hence, the UK registered the Unilever glucose release method again for harvest 1988. In order to promote the maximum level of agreement between laboratories, it was agreed that testing laboratories should use a set of reference samples, previously analysed by TPGLC, to standardize their results. These would be supplied under the auspices of the Intervention Board for Agricultural Produce.

It is too early to comment in detail on the scheme this harvest, but initial experience suggests that the introduction of the XRF-method has been successful although it is apparent that the protocol must be carefully defined to obtain the maximum level of repeatability between laboratories.