

Determination of Total Glucosinolate in Rapeseed (colza) Activities of BCR-Programme

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I - BACKGROUND BCR Programme

A Community Research Programme with the broad aim of improving the accuracy and thereby the comparability of measurements of importance in the Member States.

The programme brings together specialists who compare the results of their measurements (inter-comparisons) using their usual "best" methodology. The projects are designed to identify and eliminate the major measurement errors. Particularly in the field of chemical analysis, the projects often lead to the issue of a Certified Reference Material (CRM) having a reference value for the property of interest. The reference value is obtained from the measurements of specialists and certified through the BCR programme.

Certified reference materials allow any other laboratory to verify its measurements.

In some cases, CRMs may be used to calibrate a procedure (e.g. calibration of XRF equipment with rapeseed CRMs of known S-content).

II - GLUCOSINOLATE MEASUREMENT

The choice of method is dictated by *inter alia*:

- legal considerations e.g. use of the Community Method (GC) for certain official purpose;
- speed and economy, where very large sample through-put is required (seed crushers, control stations);
- accuracy (closeness to the true value), especially where payment is involved or when the laboratory operates a quality control system;
- precision: the ability of the method to repeat the measured value;
- need for information of individual glucosinolates (seed producers, nutritional studies, confirmation of absence of non-rapeseed seeds in the analytical sample);
- historical practices and regional/personal preferences.

It is therefore not surprising that many methods have been developed and are in use in the Community.

In such a situation, it is essential that a means is provided that allows every laboratory, regardless of the method used, to show that its results are acceptably accurate or the intended purpose.

III - ACTIVITIES OF BCR PROGRAMME

1) Intercomparison

An intercomparison involving almost all methods used in the Community and 18 laboratories was carried out in 1988. The results showed that with the exception of the "Palladium colourimetric method", all the methods studied are capable of giving results in "reasonable agreement" if care is taken with their application and calibration.

This is a frequent conclusion of BCR studies: the accuracy of the results often depends more on the laboratory personnel than on the choice of method.

The results of the intercomparison are presented in bar-chart form in the attached Figure.

2) Total Glucosinolate (GSL) in Rapeseed CRMs. (with indicative values for individual glucosinolates)

A rapeseed (CRM 190) with a certified total glucosinolate content of 25,5 ± 0,9 micromol/g of whole seed was issued last year and is available from the Commission.

The certified value is based on measurements by a variety of HPLC, GLC and glucose release methods, carried out by 13 experienced European laboratories.

CRM 190 allows laboratories to verify the reliability of their total GSL measurements, particularly in the region of importance for Community legislation (20-35 micromol/g).

Indicative values are given for individual glucosinolates, the identity of which was confirmed by HPLC-MS and GC-MS.

In the future, rapeseed CRMs will be prepared in the range from 5 to 100 microm/g.

3) S in Rapeseed

The XRF procedure is now widely accepted and used as a rapid and very precise method for determining total GSL.

The method is an indirect one and relies on the high degree of correlation between S-content and total GSL. There are good theoretical reasons for the correlation (Schnug, et.al.)

Full benefit of the XRF-method can only be realized if:

- the method gives the same results as would have been obtained by an "official or reference" method;
- there is a common basis for the calibration of the instrument in all Member States.

After consultation with specialists, the BCR programme is undertaking the preparation of a further 2 rapeseed RMs.

These will be certified by highly specialized laboratories for total S-content and would provide the basis for a common Community calibration system.

Total glucosinolate will also be certified following the procedure used for CRM 190.

It is proposed to prepare rapeseed CRMs having the following approximate contents selected to cover the required calibration range: 0,3 to 1% S.

IV - PROGRESS

Batches of suitable material have been obtained (Dr. Wathelet, Gembloux).

Homogeneity testing and packaging was completed in June 89 with certification of the S-contents and stability measurement being completed by end of 1989.

For the 1989 harvest, interim batches of rapeseed RMs were prepared and made available by Dr. Schnug (Kiel), who provided provisional values for their S-contents.

