Development of an all-inclusive method for the measurement of total dietary fibre

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Abstract

Over the past 8 years, the CODEX Committee on Nutrition and Foods for Special Dietary Uses (CCNFSDU) has been deliberating on a definition for dietary fibre that correctly reflects the current consensus thinking on what should be included in this definition. It became evident that no currently available, single method could meet the requirements of this definition. Consequently, we have developed an 'all-inclusive' procedure, based upon the principles of AOAC Official Methods 991.43, 2001.03, and 2002.02 that is compliant with the emerging CODEX definition. This procedure quantitates high and low molecular weight dietary fibre as defined, giving an accurate estimate of resistant starch and non-digestible oligosaccharides (NDO). In 2008/2009, CODEX produced a clarifying definition of dietary fibre that reflects the scientific findings of the past 5 plus decades in a single, concise definition. The 'all-inclusive' method is, in fact compliant with the final definition, and is currently the subject of an AOAC International interlaboratory evaluation. The method is discussed in this paper, together with modifications that include a recommendation for an improved internal standard as well as an incubation format that should help simplify the assay.

Keywords: total dietary fibre, resistant starch, 1,5-pentanediol, diethylene glycol, non-digestible oligosaccharides, integrated procedure, CODEX-compliant

Introduction

At the 30th session of CCNFSDU, the Committee agreed on a definition for dietary fibre (Box 1 on page 16).

The Committee also agreed on the establishment of an electronic Working Group (eWG) led by the Delegation of France, open to all Codex members. The specific role of this eWG was to: (a) review and update, as appropriate, the list of methods available for dietary fibre analysis, taking into account the new provisions in the draft definition of dietary fibre that would require the selection of methods of analysis, and possible information of new available methods; (b) consider how the results from different methods specific to different types of dietary fibre could be combined together to arrive at the total dietary fibre content in a food; (c) evaluate the performance of methods in measuring different

types of dietary fibre; (d) make recommendations for methods of analysis for dietary fibre in different food matrices; (e) consider the footnote in the accepted definition that relates to oligosaccharides of degree of polymerisation (DP) of 3-9, and to prepare a recommendation as to its revision with regard to the methods of analysis, if necessary.

In their draft document (Alinorm, 2009), the eWG noted that the Official AOAC methods (AOAC, 2002) are widely accepted globally for general labelling of nutrient content in foods as well as for health and nutrition claims. The AOAC methods are designed to be accurate, cost effective, and reproducible in various analytical environments on which industry relies. They are the most studied and validated methods available for the quantification of food components. Their use in routine analysis presents no insurmountable difficulty. These methods have passed the rigor of scientific substantiation to achieve the status of reference methods. The eWG also noted that no single AOAC validated method can measure all non-digestible carbohydrates in foods. AOAC 991.43 (Lee *et al.*, 1992) is one of the most widely used 'total' dietary fibre methods. Both this method and AOAC 985.29 (Prosky *et al.*, 1985) will measure insoluble polysaccharides and soluble high molecular weight components i.e. those that are precipitated by alcohol. However, neither fully measures the resistant starch fraction, nor do they recover the non-digestible oligosaccharide components included in the definition of dietary fibre. They quantify only part of the total resistant starch, inulin, polydextrose (Craig *et al.*, 2000), fructooligosaccharides and resistant maltodextrin, all of which have relevant physiological functions. Furthermore, some oligosaccharides are not measured at all. The eWG also noted that due to the complexity of the molecular structure of fibres, additional AOAC methods were subsequently developed to validate labelling declarations and claims by measuring specific dietary fibre components in foods that have been shown to exert physiological benefit. Maintaining these methods [e.g. AOAC 999.03, (McCleary *et al.*, 2000), for fructans] has a number of advantages. By focusing on one component the method is more specific, resulting in higher specificity and accuracy needed to detect fibre present in food products. Equally important, these component-specific methods facilitate routine, cost-effective analysis.

The eWG concluded (draft document) that the NSP method does not accurately quantify total dietary fibre. It is inappropriate as a routine technique given its inability to support the now agreed upon Codex definition of dietary fibre. Methods measuring NSP alone (Englyst and Hudson, 1996) give lower estimates than methods for total dietary fibre in foods containing resistant starch, resistant oligosaccharides and/or lignin. The eWG did not recommend the inclusion of methods where there is yet no publication about protocol and relevant validation data.

The eWG also noted that the definition encompasses a range of different types of carbohydrate polymers which are recovered to varying extents by different analytical methods. This creates potential problems of double accounting when a carbohydrate

fraction is partially or completely measured by more than one method. E this are high molecular weight inulin, which in addition to being measured by enzymatic-chemical fructan methods are also partially recovered in the enzymatic-gravimetric methods (Quemener *et al.*, 1994, 1997). The enzymatic-g methods AOAC 991.43 and 985.29 also recover some, but not all, resist (McCleary and Rossiter, 2004), which can create a double accounting prob data is then combined with that obtained by a separate specific determination starch. There is also the potential for obtaining a lower than expected value under recovery of a specific fraction by particular methods. The high degree of associated with most direct chemical methods generally means that the pr combining results from different methods are diminished.

The eWG noted (draft document) that the lack of a validated procedure tc AOAC methods to determine total fibre content has repeatedly raised conce the lengthy process to finalise the definition of dietary fibre. It also noted that i to this gap in methodology, a new integrated method of analysis of total dietar been developed by McCleary (2007) which measures total dietary fibre (includin starch), non-digestible oligosaccharides and available carbohydrates. This new i method is based principally on existing official AOAC methods (AOAC 991.4 2002.02 and AOAC 2001.03) (Gordon and Okuma, 2002) and uses conditio to those described in AOAC Official Method 2002.02 (resistant starch) to qua molecular weight resistant polysaccharides (including resistant starch). A furth similar to that described in AOAC Official Method 2001.03 allows for the mea of those non-digestible oligosaccharides in the range of DP 3 to DP 10.

The eWG concluded that 'this new integrated method provides a path fo analysing the full range of dietary fibres included in the scope of the Codex defi a manner that better reflects overall the fibre that is physiologically relevant. Thi is in the stage of collaborative study analysis and is likely to achieve AOAC ap addition, the eWG suggested that the Committee should consider the inclusi new method of analysis for total dietary fibre (McCleary, 2007), once AOAC v has been completed.

In this paper, the integrated procedure for the measurement of total dietar described, as well as some potential improvements to the method.

Materials and methods

Materials

D/L-maleic Acid (cat. no. M-0375), BSA (cat. no. A-2153), dimethyl su (DMSO; cat. no. D-8779) and sodium azide (cat. no. S-8032) were from Sigma

Ireland Ltd., Dublin 24. Acetic acid (glacial) GR, sodium hydroxide and calcium chloride ($CaCl_2.2H_2O$) were from Merck, Darmstadt, Germany. Amyloglucosidase (cat. no. E-AMGDF), thermostable α-amylase (cat. no. E-BLAAM), protease (cat. no. E-BSPRT) and purified pancreatic α-amylase (E-PANAA) were obtained from Megazyme International Ireland Limited, Bray, Ireland. Barley β-glucan (medium viscosity; cat. no. P-BGBM), citrus pectin (cat. no. P-CITPN) and wheat arabinoxylan (cat. no. P-WAXM) were also from Megazyme. Partially degraded chicory inulin (Raftilose P-95®) was a kind gift from Raffinerie Tirlemontoise S.A. Tienen, Belgium. Polydextrose® (Litesse®) was from Danisco, N.Y., USA and Fibersol 2® was from Matsutani Chemical Company, Hyogo, Japan. Regular Maize Starch (Lot 60401; RMS), High Amylose Maize Starch (Lot 60107; HAMS) was from Penford Australasia, Lane Cove, NSW, Australia. Hylon VII® (Ref. 98GH8401), Novelose 330® (Ref. AH17529) and Novelose 240® (Ref. 96LF10063) were from National Starch and Chemical Company, Bridgewater, USA. Native potato starch was from Avebe, Foxhol, the Netherlands. ActiStar® (enzyme modified tapioca/cassava starch; US Patent 6,043,229) was from Cerestar, Vilvoorde, Belgium. Potato amylose (cat. no. A-9262) and ACS Soluble starch (cat. no. S-9765) were from Sigma Chemical Company. D-Fructose/D-Glucose assay kit (cat. no. K-FRUGL), α-Amylase assay kit (Ceralpha; cat. no. K-CERA), Total starch assay kit (cat. no. K-TSTA), Total dietary fibre assay kit (cat. no. K-TSTA), D-Sorbitol/xylose assay kit (cat. no. K-SORB) and Resistant Starch assay kit (cat. no. K-RSTAR) were obtained from Megazyme International Ireland Limited, Bray, Ireland.

Methods

The method under consideration (McCleary, 2007) is modelled on AOAC Method 2002.02 for measurement of resistant starch. The enzymes used are essentially devoid of activity on dietary fibre components and non-digestible oligosaccharides (NDO). The amyloglucosidase (AMG) and pancreatic α-amylase enzyme preparations are devoid of activity on pectin and fructooligosaccharides (FOS) and have negligible activity on β-glucan. Activity on resistant maltodextrins (RMD) and Polydextrose® is consistent with reported information on partial hydrolysis of the oligosaccharides by α-amylase and AMG (Craig *et al.*, 2000; Gordon and Okuma, 2002). The protease employed is devoid of α-amylase (an essential requirement in this assay format). This procedure measures insoluble dietary fibre (IDF), high molecular weight soluble dietary fibre (HMWSDF), resistant starch (RS), and low molecular weight non-digestible oligosaccharides (NDO).

Sample treatment and analysis of high molecular weight dietary fibre

Duplicate one gram amounts of the sample being analysed are incubated in 250 ml Duran® bottles in a shaking incubation bath at 150 rev/min in orbital mode, with pancreatic α-amylase and amyloglucosidase (AMG) for exactly 16 hours at 37 °C. During this time, non-resistant starch is solubilised and hydrolysed to D-glucose by the combined action of

the two enzymes. The pH is adjusted to approx. 8 and the reaction solutions are incubated at 100 °C to inactivate α-amylase and amyloglucosidase and to denature protein. Denatured protein is digested at 60 °C with protease. The pH is adjusted to approx. 4.5 with acetic acid. One ml of sorbitol or glycerol (now replaced by diethylene glycol) internal standard (100 mg/ml) is added followed by four volumes of ethanol with mixing to precipitate soluble, polymeric dietary fibre (including resistant starch that is solubilised, but not depolymerised, in the 100 °C incubation step). The suspension is filtered and the residue is washed sequentially with 76% ethanol, 95% ethanol and acetone; dried, and weighed. One duplicate is analysed for protein and the other is incubated at 525 °C to determine ash. The TDF is the weight of the filtered and dried residue less the weight of the protein and ash.

Analysis of low molecular weight non-digestible oligosaccharides

The pooled ethanolic wash solution is concentrated, desalted, reconcentrated by rotary evaporation (at 60 °C) and analysed by HPLC to determine NDO. The method employed is modelled on AOAC Method 2001.03, 'Total dietary fibre in foods containing resistant maltodextrins' (Gordon *et al.*, 2002). In that method, the low molecular weight resistant maltodextrins (LMWRMD) that are soluble in 78% v/v ethanol are recovered and analysed by HPLC. In the current method, the same principle is used to measure all of the NDO likely to be in the food product or to have been added. The aqueous ethanol filtrate is concentrated by rotary evaporation, desalted through ion exchange resins (25 g Amberlite FPA53 (OH^-) and 25 g Amberlite 200 C (H^+) or equivalent, (Rohm and Haas, France, Chauny, S.A.S.), concentrated and analysed by HPLC. This can be done using either gel permeation chromatography according to AOAC Method 2001.03 [using two TSK-GEL® G2500 PWXL, 7.8 mm × 30 cm (Tosoh Corp, Tokyo, Japan) gel permeation columns in series with a TSK® guard column PWXL 6.0 mm id × 4 cm (Tosoh Corp)] with glycerol (now replaced by diethylene glycol) as internal standard (Gordon and Okuma, 2002), or alternatively using a Waters Sugar-Pak® 6.5 × 30 cm (part no. WAT085188) column, with D-sorbitol as the internal standard. It should be noted that Amberlite FPA53 (OH^-) should not be replaced by Amberlite IRA 900 (OH^-), as the latter resin tends to bind some of the oligosaccharides in the sample.

Apparatus and reagents used in this analysis, together with exact details of the analytical procedures and the calculations, can be seen in McCleary (2007).

Results and discussion

A range of starches, milled grain and some food samples were assayed for resistant starch (RS) using the procedure described here as well as AOAC Method 2002.02 (RS), and the results are shown in Table 1. Clearly, there is a good agreement in values for all samples except for native potato starch, ActiStar® and green bananas. Native potato starch and

Table 1. Resistant starch values determined for a number of samples using AOAC Method 2002.02 and the current TDF/RS procedure.

Sample details	Resistant starch % w/w (as is basis)	
	AOAC Method 2002.02	New TDF/RS Method
Native potato starch	64.9	56.8
Actistar®	58.0	48.8
Green banana	51.0	38.0
Hylon VII®	50.0	48.6
Novelose 240®	48.4	44.2
Novelose 330®	38.8	38.7
Hi Maize 1043®	41.0	41.7
CrystaLean®	39.8	37.9
Amylose (potato)	38.2	36.6
Regular maize starch	0.5	0.8
Pinto beans (dry milled)	39.4	35.6
Haricot beans (dry milled)	36.9	31.2
Red kidney beans[1]	5.0	5.3
Red lentils (dry milled)	7.6	6.1
Flageolet beans (freeze-dried) [1]	5.3	4.5
Cooked/cooled potato	4.0	3.2
Corn flakes	2.2	2.4

[1] Samples were freeze-dried with a final moisture content of approx. 2-3%.
Hylon VII® is native high amylose maize starch. Novelose 240®, Novelose 330®, Hi Maize 1043® and Crystalean® are retrograded high amylose maize starches.

ActiStar® completely dissolve in the 100 °C incubation step (as does most of the banana starch), and subsequent precipitation by ethanol is apparently not complete.

While the RS values for these two samples are underestimated by the current method, it should be noted that with AOAC Method 985.29 or 991.43 the TDF (and thus RS) value is essentially zero. From ileostomy studies, native potato starch has been shown to contain high levels of RS. However, this is a very fragile starch, with the granule structure being readily destroyed by heat or physical treatment. Thus it is unlikely that native potato starch will ever be used as a source of RS in food products. Actistar®, which is prepared by partial hydrolysis of tapioca starch with α-amylase and isoamylase, is also very susceptible to heat

treatment. Consequently, ActiStar® can only be used in foods designed for consumption without cooking.

TDF values for samples traditionally used to check the efficacy and purity of enzymes used in DF analysis (e.g. β-glucan, larch arabinogalactan, wheat arabinoxylan, pectin and casein) were essentially the same with the new method and AOAC Method 991.43 (McCleary, 2007). However, for high amylose maize starch (HAMS), a value of 46.5% was obtained with the new method compared to 29.3% with AOAC Method 991.43. The much higher DF value for HAMS reflects a true measure of the RS content of this sample. With wheat starch, a TDF value of 0.8% was obtained, compared to a value of 0.1% by AOAC Method 991.43, demonstrating that even wheat starch contains a small percentage of RS.

The TDF values for a range of resistant starch containing samples, determined with AOAC Method 991.43 and the new method, are shown in Table 2. In general, the TDF values determined with the new method are much higher than those obtained with AOAC Method 991.43. Just two samples, Novelose 240 and Novelose 330, showed similar values

Table 2. TDF values determined for a range of RS containing samples by AOAC Method 991.43 and the current TDF/RS method.

Sample details	Total dietary fibre, % w/w (as is basis)	
	AOAC Method 991.43	New TDF/RS Method
Hylon VII®	25.6	49.3
Novelose 240®	47.1	44.3
Novelose 330®	35.0	39.9
Actistar®	0.5	47.3
Green banana (freeze-dried)	7.5	37.6
Native potato starch	0.9	64.6
Red kidney beans (freeze-dried)	20.4	21.8
Cooked/cooled potato (freeze-dried)	7.1	9.6
Red lentils	11.3	14.8
Pinto beans (dry milled)	17.3	54.9
Haricot beans (dry milled)	23.3	51.9
Regular maize starch	0.1	0.7

All data is reported on an as is basis. Some of the samples were freeze-dried before analysis. The moisture content of these samples was approx. 2-3%.

with the two methods. Both samples are retrograded high amylose maize starches. The new procedure for TDF/RS gives a more accurate measure of DF in samples containing RS.

The procedure described in this paper for the measurement of LMWNDO is based on AOAC Method 2001.03 for measurement of low molecular weight resistant maltodextrins (LMWRMD). Since in the current procedure, the samples are subjected to incubation with pancreatic α-amylase plus AMG for 16 h, followed by heat treatment and incubation with protease, it was important to demonstrate that there is no degradation of the NDO during this process. HPLC traces for Raftilose® (fructooligosaccharides; FOS) dissolved in water and analysed, compared to the same material subjected to the full enzymic incubation sequence are shown in Figure 1. The traces are essentially identical, showing that no degradation has occurred. A glycerol peak is evident in the material subjected to enzymic treatment, which was due to the presence of glycerol as a stabiliser in the AMG and protease enzyme preparations. With Neosugars® (also FOS), similar results were obtained showing no enzymic degradation. With Fibersol 2® and Polydextrose®, there is some degradation, however this is consistent with information supplied by the manufacturers.

In conclusion, the incubation steps in the new TDF procedure cause insignificant degradation of FOS and only the expected degradation of resistant maltodextrins and Polydextrose®, meaning that analysis of the aqueous ethanolic filtrate will give a true measure of the NDO in the original sample.

In AOAC Method 2001.03 (resistant maltodextrins), glycerol is used as the internal standard. However, many enzyme preparations used in TDF assay procedures contain glycerol as a stabiliser. Consequently, we evaluated a range of other sugars and sugar alcohols as a potential replacement for glycerol. Of these, D-sorbitol had the best chromatographic properties on the Waters Sugar-Pac column, but was not suitable for the gel permeation columns as it chromatographs at the same point as D-glucose. In this case, glycerol is the internal standard of choice.

The method described here is currently the subject of an interlaboratory evaluation through AOAC International. Results of the first stage of this study have been presented to the Association.

Further developments

Since it would be useful for the internal standard for HPLC to be appropriate for both the gel permeation chromatographic format as well as for the ion exchange system with the Waters Sugar-Pak® column, several potential internal standards were evaluated. Of these 1,2-pentanediol and 1,5-pentanediol showed potential. The chromatographic patterns of these compounds against D-glucose and glycerol in the gel permeation system, is shown

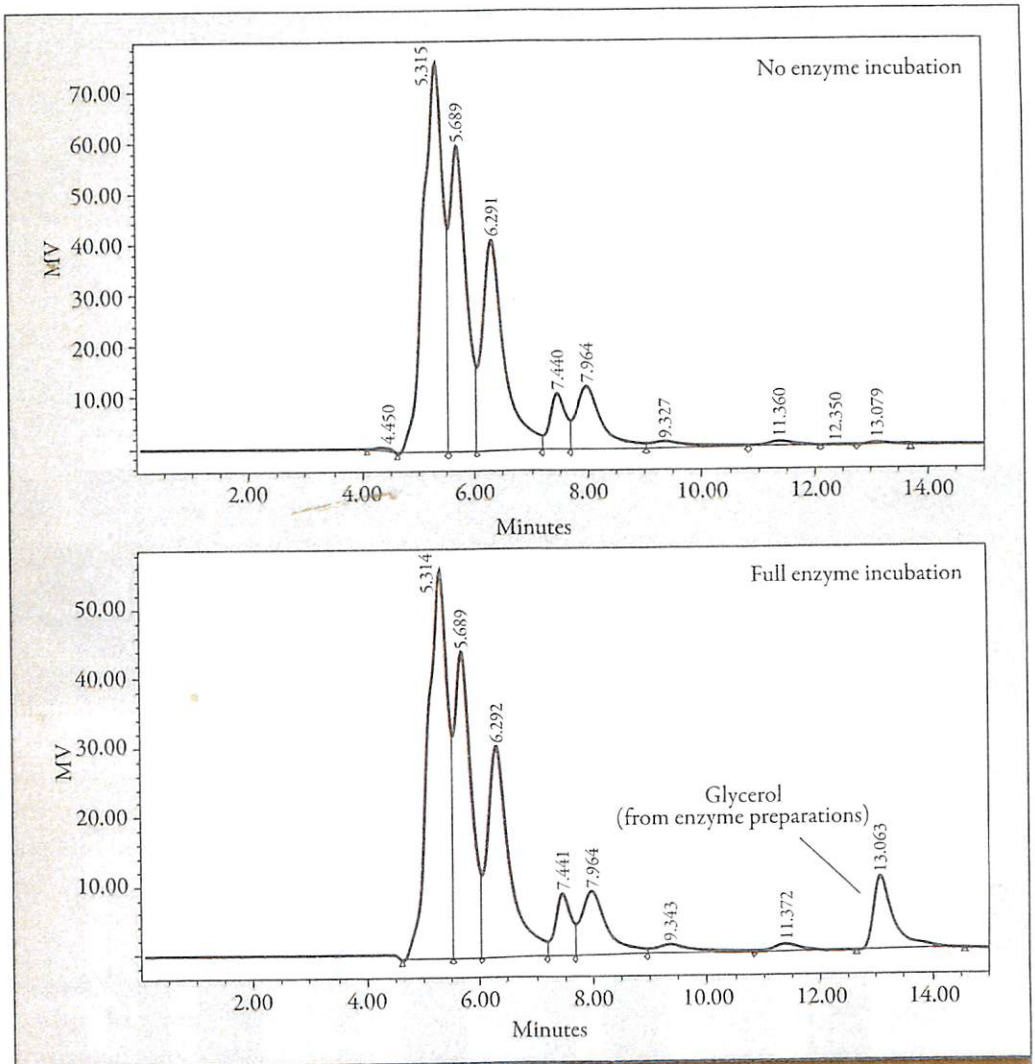

Figure 1. HPLC trace for Raftilose® dissolved in water and analysed directly, compared to Raftilose® recovered as NDO after running through the current TDF/RS procedure. Column: Waters Sugar-Pak® (6.5 × 300 mm). Solvent: distilled water containing EDTA (50 mg/l). Flow rate: 0.5 ml/min. Temperature 90 °C.

in Figure 2. Clearly, both are well separated from both D-glucose and glycerol. To be of use, these compounds must not be lost on concentration of the eluate from the desalting resins. Losses of 1,2-pentanediol and 1,5-pentanediol (as well as of glycerol) compared to D-glucose on rotary evaporation, are shown in Figure 3a,b. Clearly, compared to D-glucose, there was no loss of glycerol even on running the evaporator up to one hour after the water had been evaporated. The loss in 1,5-pentanediol is insignificant, whereas there

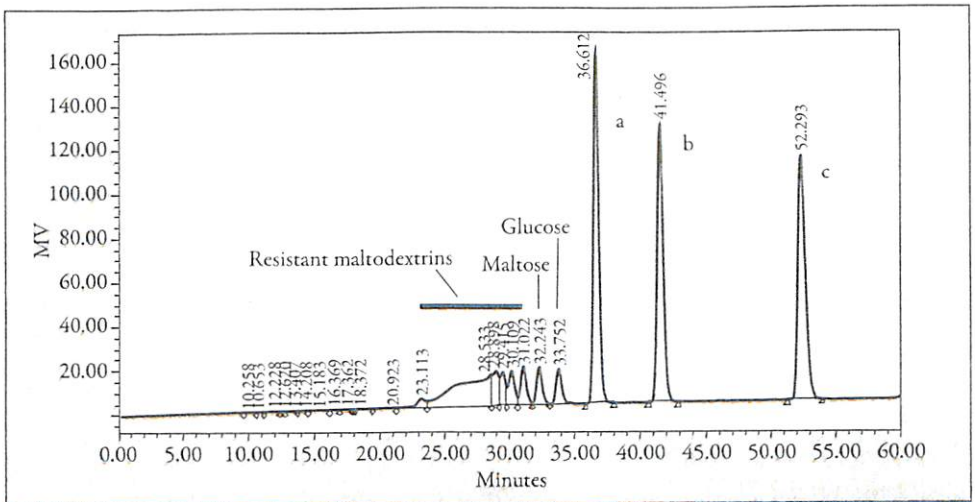

Figure 2. HPLC of DE-25 corn syrup plus glycerol, 1,2-pentanediol and 1,5-pentanediol on two TSK-GEL G2500 PWXL, 7.8 mm × 30 cm (Tosoh Corp) gel permeation columns connected in series.
(a) glycerol; (b) 1,2-pentanediol; and (c) 1,5-pentanediol.

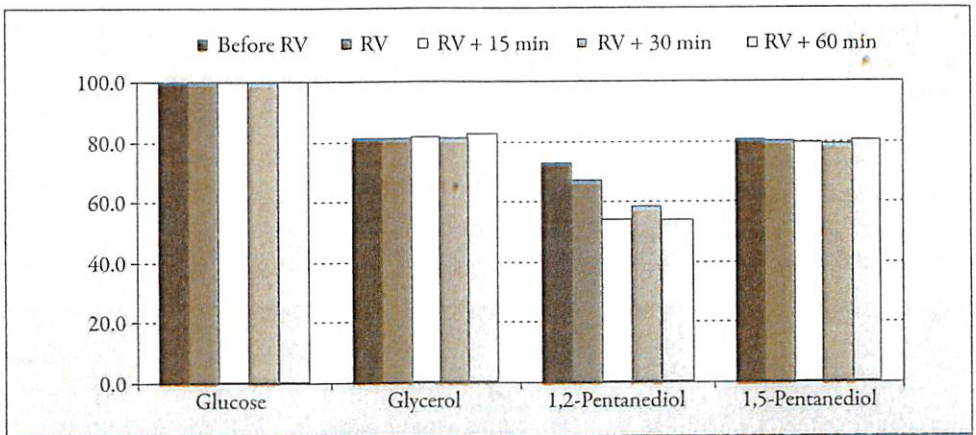

Figure 3a. Recovery of glycerol, 1,2-pentanediol and 1,5-pentanediol relative to D-glucose on rotary evaporation of 200 ml of solution containing 100 mg of each component at 60 °C. The graph shows original solution mix; concentration to near dryness; further evaporation for 15 min; 30 min; and 60 min. All re-dissolved to a final volume of 10 ml (RV=rotary evaporation).

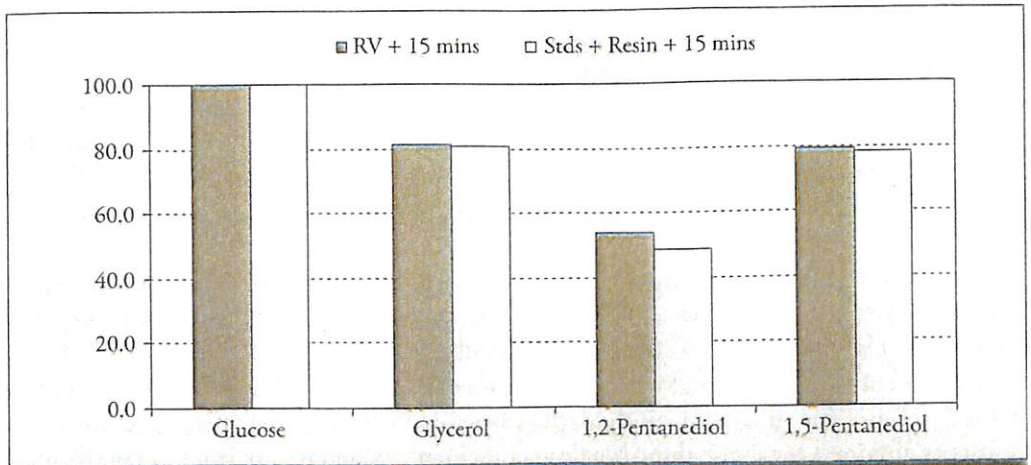

Figure 3b. Recovery of glycerol, 1,2-pentanediol and 1,5-pentanediol relative to D-glucose on desalting of solutions through ion exchange resins and rotary evaporation at 60 °C to dryness and further evaporation for 15 min.

was considerable loss of 1,2-pentanediol. 1,5-Pentanediol would appear to be an excellent internal standard. It chromatographs quite distinctly from sugars, oligosaccharides and glycerol, there is no loss on evaporation at 60 °C, there is a good peak shape (Figure 3b) and no adsorption to the desalting resins, and the peak shows no significant broadening even though it elutes well after the other components. Glycerol is not an ideal internal standard because this compound is widely used as an ingredient in the food industry.

Dr. Okuma, Matsutani Chemical Company, recently recommended the use of either diethylene glycol or triethylene glycol as internal standard. We have confirmed that both of these compounds are interesting and neither compound is lost on evaporation during concentration, nor by adsorption to resins. Comparisons of these compounds and 1,5-pentanediol are continuing.

An in-line desalting of samples in preparation for liquid chromatography is very time consuming. Fortunately, an in-line desalting system has been evaluated and appears to be suitable to this application (DeVries, personal communication).

In the incubation of sample with α-amylase plus amyloglucosidase, a shaking water bath is employed. In the development of the resistant starch method (AOAC 2002.02) it was found that if samples were stirred in glass tubes using a magnetic stirrer the resistant starch granules were ground between the stirrer bar and the glass base of the tube resulting in significant solubilisation, and subsequent underestimation of the resistant starch content for some samples. Since it was considered that incubations involving stirring would have advantages over those in which the incubation containers are shaken (as in McCleary,

2007), the effect of shaking and stirring of samples in bottles during the incubation with enzymes was evaluated. The three incubation arrangements were:

1. shaking of reaction solution in a 250 ml Duran® bottle in rotary motion (150 rpm);
2. stirring employing a suspended stirrer with no contact between the stirrer bar and the bottom of the bottle (Figure 4); and
3. conventional magnetic stirring with the stirrer bar added to the reaction container.

In the latter two cases, stirring was achieved with a 2mag Mixdrive 15 submersible magnetic stirrer (http://www.2mag.de/english/stirrer/multiple/stirrer_multiple_04_mixdrive6_15.html) set at 170 rpm. All incubations were performed at 37 °C. A comparison of results obtained with the three mixing systems is shown in Figure 5 and in Table 3. Clearly, for all samples studied, the suspended stirrer arrangement gives the same results as obtained for the shaking bottle arrangement. When incubations are performed in bottles with conventional stirring with a magnetic stirrer (not suspended) results obtained were the same for all samples except native potato starch. Native potato starch is known to be a very fragile starch and thus is unlikely ever to be used as a source of resistant starch in processed foods. On the basis of these results, and for the sake of convenience and simplicity, we recommend the use of conventional magnetic stirring at 37 °C for 16 h for the incubations.

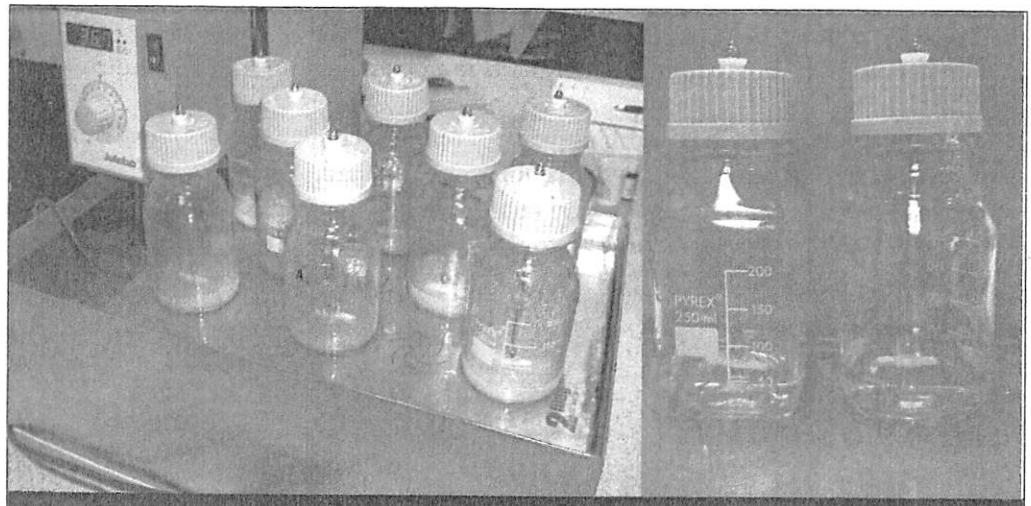

Figure 4. Arrangement for mixing suspensions of resistant starch containing samples using a suspended magnetic stirrer (to avoid grinding of sample between the stirrer bar and the glass bottle).

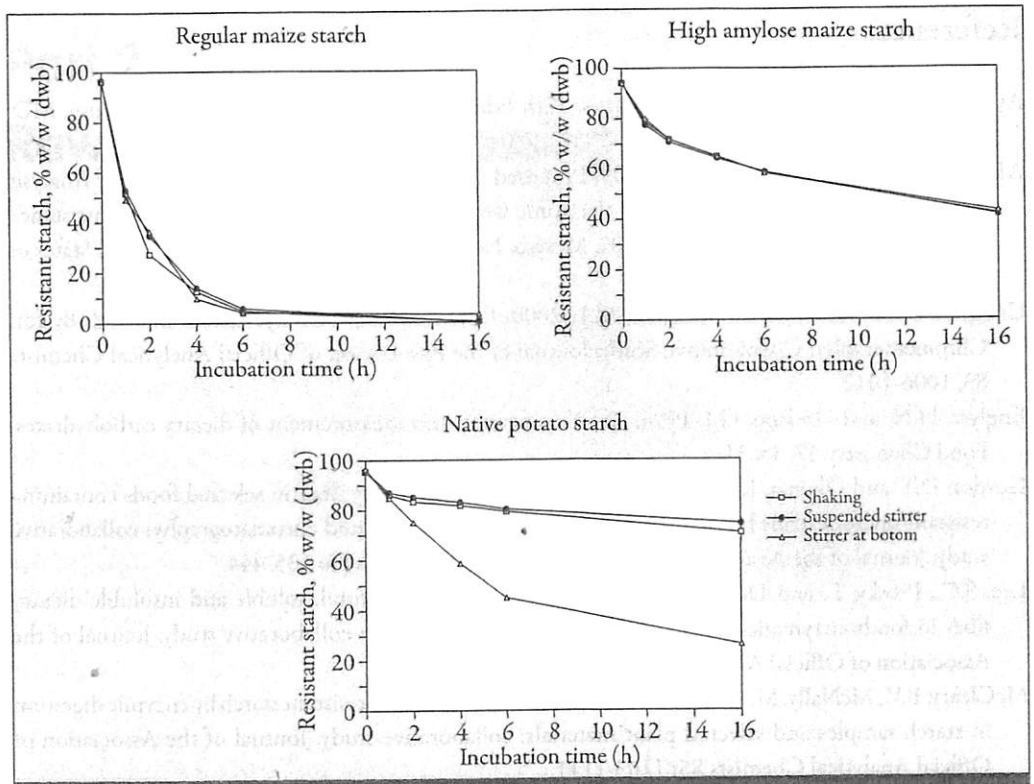

Figure 5. Effect of shaking, suspended stirring and magnetic stirring on the time course of hydrolysis of regular maize starch, high amylose maize starch and native potato starch.

Table 3. The effect of the shaking or stirring conditions on the measured level of resistant starch in a range of samples.

Sample	Resistant starch % w/w (dry weight basis)		
	Shaking	Suspended stirring	Conventional magnetic stirring
Regular maize starch	0.3	0.1	0.3
Kidney beans	5.3	5.6	5.7
Green banana	51.0	48.4	46.6
High amylose maize starch	41.7	41.4	42.9
Hylon VII (high amylose maize starch)	52.6	-	49.7
Native potato starch	70.6	74.4	26.3

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