## **Analysis of Dietary Fibre**





## **HK Regulation**

 Dietary fibre means any fibre analyzed by means of any official methods adopted by AOAC International.



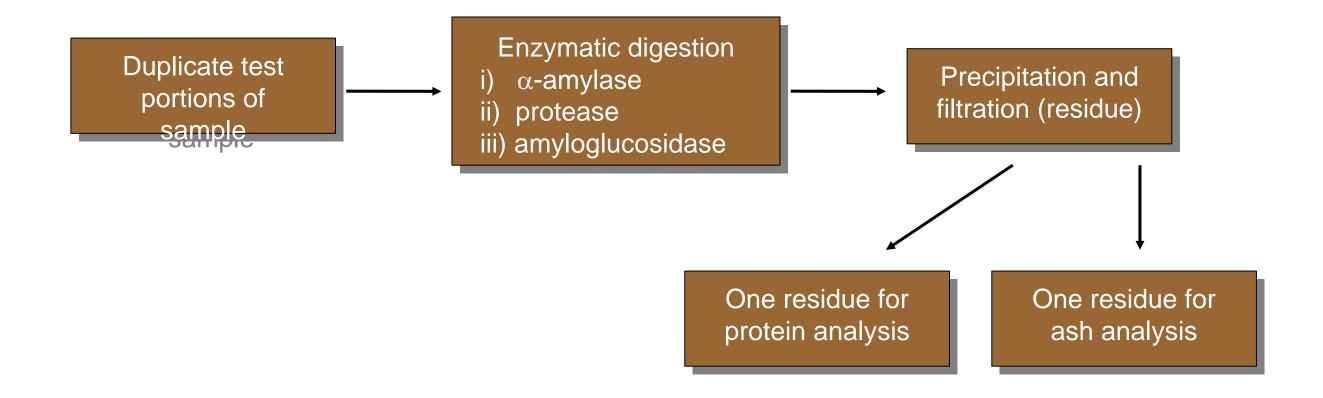


## **AOAC Official Methods**

AOAC 985.29	Total Dietary Fibre in Foods	Enzymatic-Gravimetric Method
AOAC 993.19	Soluble Dietary Fibre in Food and Food Products	Enzymatic-Gravimetric Method
AOAC 991.42	Insoluble Dietary Fibre in Foods and Food Products	Enzymatic-Gravimetric Method
AOAC 991.43	Total, Soluble, and Insoluble Deitary Fibre in Foods	Enzymatic-Gravimetric Method
AOAC 992.16	Total Dietary Fibre (Applicable to determination of total fibre in cereals, beans, vegetables and fruits)	Enzymatic-Gravimetric Method
etc		







Total dietary fibre = weight (residue) - weight (ash + protein)



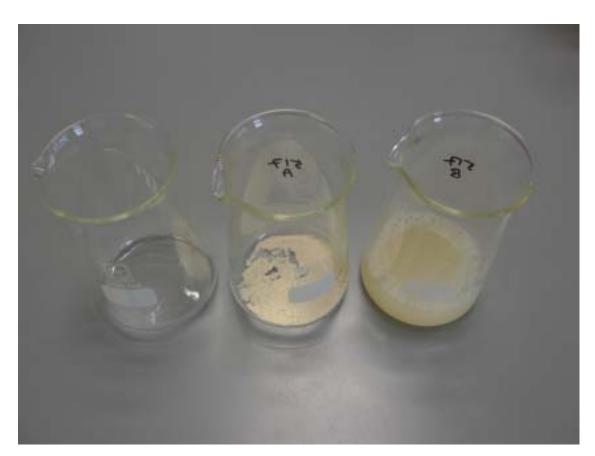


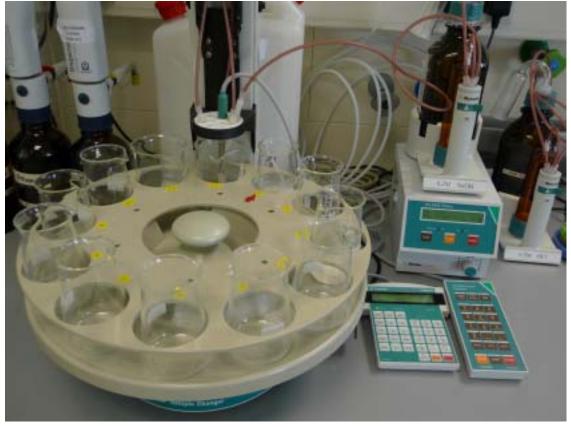
- Homogenize and dry sample (freeze-dry is recommended).
- Defat with petroleum ether if >10% fat content, otherwise false high results.
- Weigh duplicate test portions (difference in weight should not >20 mg).





## Preparing for digestion









#### Preparing for digestion









 $\alpha$ -Amylase: gelatinize

- Add phosphate buffer (pH 6.0, 50 mL) to sample.
- Adjust to pH  $6.0 \pm 0.2$ .
- Add enzyme, incubate at 95 100 °C.
- 30 minutes in water bath.





Protease: remove protein

- Cool to room temperature.
- Adjust to pH  $7.5 \pm 0.2$ .
- Add enzyme, incubate at 60 °C for 30 minutes.





Amyloglucosidase: remove starch

- Cool.
- Adjust to pH 4.0 4.6.
- Add enzyme, incubate at 60 °C for 30 minutes.

Note: final solution volume is about 70 mL.





#### **Summary**

- pH 6.0 ± 0.2, α-amylase, 95 100 °C, 15 - 30 min.
- pH 7.5 ± 0.2, protease, 60 °C, 30 min.
- pH 4.0 4.6, amyloglucosidase, 60 °C, 30 min.





- Add 280 mL 95 % ethyl alcohol (60 °C).
- Let precipitate form at room temperature for 60 minutes.
- Collect the residues (soluble fibre + insoluble fibre) in pre-weight crucibles.





## Precipitation









#### **Filtration**







- One test portion for protein, using N x 6.25 as conversion factor.
- Incinerate second test portion at 525 °C for 5 hours.

Total dietary fibre = [weight residue – protein – ash – blank] / weight test portion

Weight residue = average of duplicate
Weight test portion = average of duplicate





## Test for enzyme purity every half yearly

Test sample	Activity tested	Test portion (g)	Expected recovery (%)
Citrus pectin	Pectinase	0.1	95-100
Stractan (larch gum)	Hemicellulase	0.1	95-100
Wheat Starch	Amylase	1.0	0-1
Corn Starch	Amylase	1.0	0-2
Casein	Protease	0.3	0-2
β-Glucan (barley gum)	β-Glucanase	0.1	95-100





## **AOAC** Method for functional fibre

Functional fibre	Commercial name	Test Method	
Beta-glucan	Imprime PGG®	AOAC 995.16	
Oligofructose	Raftilose®, OliggoFiber <sup>TM</sup>	AOAC 997.08 or 999.03	
Fructooligosaccharides	Neosugar, Actilght®	AOAC 997.08 or 999.03	
Polydextrose	Litesse®	AOAC 2000.11	
Galactooligosaccharides	Yacult, Borculo Whey Products	AOAC 2001.02	
Resistant maltodextrin	Fibersol-2	AOAC 2001.03	
Resistant starch	C*Actistar	AOAC 2002.02	



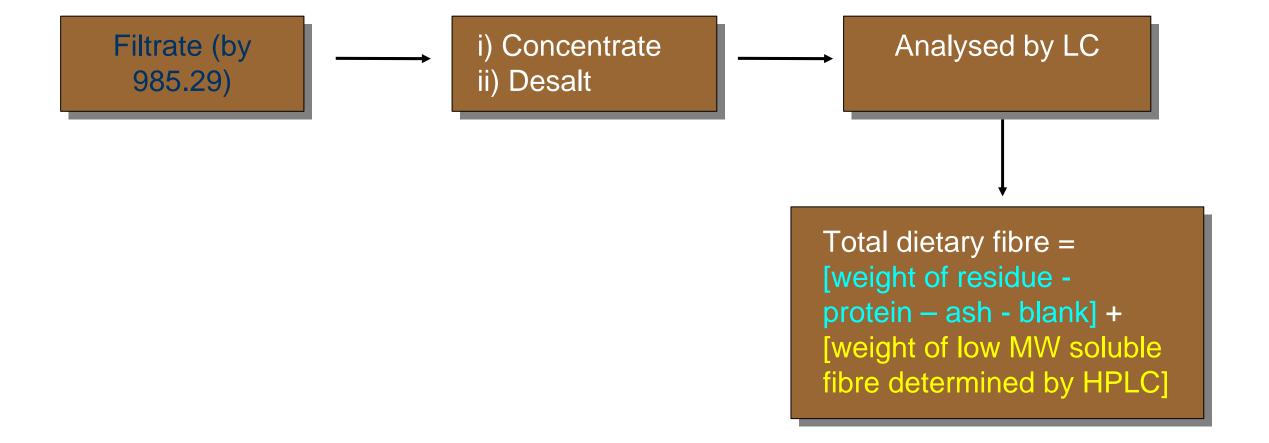


# Dietary Fiber Containing Supplemented Resistant Maltodextrin (RMD)

High MW RMD by Method 985.29 and Low MW RMD by HPLC











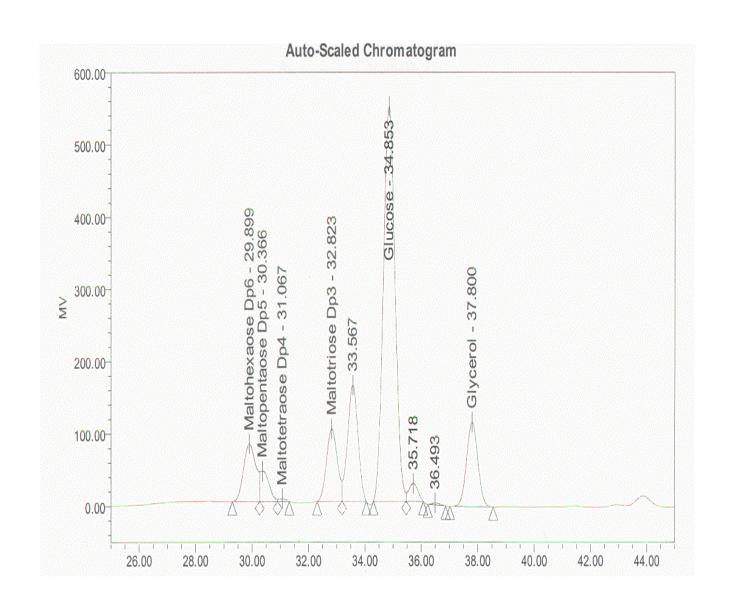
Only a portion of the resistant maltodextrin is precipitated in the aqueous ethanol when Method 985.29 is applied to foods containing resistant maltodextrin.







In Method 2001.03, resistant maltodextrin that are soluble in aqueous ethanol are desalted, concentrated and measured by liquid chromatography.







## Desalting

Ion exchange column(OH-type and H-type)





Total dietary fibre =

fibre as measured by *Method 985.29* plus
low molecular weight resistant maltodextrin

Note: low molecular weight resistant maltodextrin refers to resistant maltodextrin that are soluble in the aqueous ethanol in Method 985.29.





## Calculation of available carbohydrate when method 2001.03 is used

Available carbohydrate = 100 – [moisture + ash + protein + fat + alcohol + (soluble dietary fibre + insoluble dietary fibre) + low molecular weight resistant maltodextrin]

Note: low molecular weight resistant maltodextrin refers to resistant maltodextrin that is measured by AOAC 2001.03.





# Recoveries for other functional fibres when tested by AOAC 2001.03

Functional fibre	Recovery (%)
β-Glucan	101*
Fructooligosaccharides	98
Polydextrose	117
Galactooligosaccharides	111
Glucooligosaccharides	46
Resistant maltodextrin	88

\* Recovery of insoluble dietary fibre and soluble dietary fibre is particular high.





#### Points to note

- Definition of "0": ≤1 g/100 g.
- Results are method dependent.
- Functional fibre should be included in the calculation of available carbohydrate.
- AOAC 2001.03 may give higher TDF results but with higher testing cost.





## Available proficiency test

FAPAS

AOAC

■ LGC





## Thank You



