

Which McCleary Method is recommended for Dietary Fiber Measurement in Foods?



Advantages of the Rapid integrated TDF method (AOAC 2017.16) over the integrated TDF method (AOAC 2009.01)

As the integrated TDF (**K-INTDF**) method was adopted within industry, Megazyme's ongoing research identified scope for further improvements to the procedure with benefits in terms of accuracy, convenience and safety. Adaptions were made to create the RINTDF method (**K-RINTDF**) which addresses all of the reported limitations in the integrated TDF method. RINTDF was accepted as AOAC Method 2017.16 in 2018 and is consistent with the CODEX Alimentarius definition of dietary fiber.

A summary of the benefits of using K-RINTDF over K-INTDF to measure Dietary Fiber in foods.

| | K-RINTDF | K-INTDF |
|---------------------------------------------------------|----------------------------|------------------------------------------------------|
| Speed of Measurement | 4 h incubation | 16 h incubation |
| Represents ' <i>in vivo</i> ' conditions | Yes | No |
| Resistant Maltodextrin (artifact) | No (accurate TDF value) | Yes (overestimated TDF value) For some samples |
| Resistant Starch (RS ₄ and RS ₂) | Accurately measured | Underestimated |
| Fructooligosaccharides | Accurately measured | Underestimated |
| Validated | Yes | Yes |

In addition, K-RINTDF follows a simplified process removing the requirement for the hazardous reagent, Sodium Azide.

The adaptations made to create K-RINTDF and the advantages over K-INTDF

1. More representative of '*in vivo*' conditions

The pancreatic α -amylase (PAA)/amyloglucosidase (AMG) incubation time of 16 h used in K-INTDF does not represent appropriate '*in vivo*' conditions given that a more likely time of residence of food in the small intestine is ~ 4 h. K-RINTDF employs a shorter incubation time of 4 h in order to reflect the physiological conditions.

With the longer incubation conditions of K-INTDF, together with the lower levels of AMG and PAA employed, it was shown that for samples with high levels of starch, small amounts of resistant maltodextrins were produced as an artefact of the method. This material was then incorrectly included in the SDFS component of dietary fiber and therefore resulted in an overestimation of total dietary fiber content by 0.5-2%.

2. Shorter incubation time with PAA/AMG

The incubation time has been shortened by 75% resulting in a significant reduction in indirect laboratory costs.



3. Improved Accuracy

| Overall Dietary Fiber | Resistant Starch | Fructooligosaccharides |
|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------|----------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| The higher enzyme loadings and shorter incubation time applied in K-RINTDF solve the problem of overestimation of dietary fiber due to the production of resistant maltodextrins in the SDFS fraction that was identified in K-INTDF. | The incubation conditions of K-RINTDF give more physiologically relevant values for samples such as Fibersym® (RS ₄) and Hylon VII® (RS ₂). | FOS is more accurately measured due to the use of a TSKgel® G2500PW _{XL} column to achieve complete separation of fructotriose (in FOS) from disaccharides. |

| Comprehensive Total Dietary Fiber | | | | | |
|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|---|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-------------------------|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|---|
| High Molecular Weight DF | | | Low Molecular Weight DF | | |
| Classic Fibers (examples) | | Resistant Starch | | Non-digestible Oligosaccharides (NDOs) (examples) | |
| <ul style="list-style-type: none"> • Cellulose • β-Glucan • Galactomannan • Arabinoxylan • Pectin • Arabinogalactan | | <ul style="list-style-type: none"> • RS₁ (physically inaccessible starch) • RS₂ (resistant starch granules) • RS₃ (retrograded starch) • RS₄ (phosphate-crosslinked starch) | | <ul style="list-style-type: none"> • Fructooligosaccharides (FOS) • Galactooligosaccharides (GOS) • Polydextrose • Resistant Maltodextrins (RMD) • Xylooligosaccharides (XOS) | |
| Which methods accurately measure these components? | | | | | |
| RINTDF | ✓ | RINTDF | ✓ | RINTDF | ✓ |
| Prosky/Lee | ✓ | Prosky/Lee | ✗ | Prosky/Lee | ✗ |
| Matsutani | ✓ | Matsutani | ✗ | Matsutani | ✓ |

4. Safer Process

The use of the toxic chemical, sodium azide, in the incubation buffer is no longer necessary due to the shortened incubation time of 4 h. This makes the process safer for the analyst.

5. Simpler Process

Sample preparation for HPLC in K-RINTDF is simplified by removing most of the salt by deionisation with Ambersep and Amberlite resins in a tube, followed by complete deionisation using Bio-Rad HPLC deionisation pre-column cartridges. This avoids the necessity to desalt by column chromatography followed by sample concentration.

6. Validation

Megazyme developed K-RINTDF to provide the most accurate measurement possible of dietary fiber as defined by Codex Alimentarius. This procedure has been fully validated as AOAC Method 2017.16 and ICC Standard No. 185.