Amino Acid Profile – Method Summary

In order to determine the total amino acid content of a given sample, the protein chains must be broken down to its constituent parts. The samples are heated under acidic conditions 115°C to hydrolyse the protein chains. For the analysis of cystine and methionine, the samples are oxidised with performic acid prior to acid hydrolysis. The resulting hydrolysate is diluted, filtered and the pH adjusted.

The extracted amino acids are then derivatised prior to determination by gradient HPLC with fluorescence detection.

This method is suitable for the quantification of amino acids (listed below) in a wide range of samples including feeds and feed constituents and other protein containing samples. It is also suitable for products such as supplements and premixes where the amino acids are free as opposed to being bound in protein chains.
The method is not suitable for the analysis of the hydroxy analogue of Methionine, Taurine and Tryptophan. The method does not distinguish between the D and L forms of the amino acids.

Aspartic Acid	Threonine	Valine
Serine	Alanine	Lysine
Glutamic Acid	Methionine	Isoleucine
Glycine	Proline	Leucine
Histidine	Cystine	Phenylalanine
Arginine	Tyrosine	

The lower reporting limit is 0.02 g/100g

Samples with up to 100g/100g (100%) may be analysed by this method.

The method is UKAS accredited.

METHOD SUMMARY:
Determination of Dietary Fibre by the AOAC method using MES-TRIS Buffer

TEST METHOD NUMBER: C-TM-129

DATE: November 2015

Premier Analytical Services uses the UKAS accredited method C-TM-129 for the Determination of Dietary Fibre by the AOAC method using MES-TRIS Buffer in raw materials and food products.

Starch and protein are removed from the samples enzymatically and the resulting residue, which is insoluble in 78% ethanol, is determined gravimetrically. Corrections are applied to account for residual protein and inorganic components. Modifications are included for the determination of insoluble and soluble dietary fibre.

PERFORMANCE CRITERIA

Analysis

All analyses are carried out using validated methods which meet strict performance criteria and are valid for the analysis of dietary fibre by the AOAC method using MES-TRIS Buffer in raw materials and food products.

Recovery

All analyses are conducted with a control IQC sample.

For any further queries please visit our website at paslabs.co.uk
or contact Warren Jackson at warren.jackson@premierfoods.co.uk

METHOD SUMMARY:
Determination of Aluminium, Calcium, Copper, Iron, Magnesium, Manganese, Potassium, Sodium and Zinc by Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES)

TEST METHOD NUMBER: C-TM-206

DATE: June 2022

Premier Analytical Services uses the UKAS accredited method C-TM-206 for the Determination of Aluminium, Calcium, Copper, Iron, Magnesium, Manganese, Potassium, Sodium and Zinc by Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES) in raw materials and food products.

Samples are digested in concentrated nitric acid, which removes organic matter by oxidation. Elemental concentration in the resulting solution is measured using ICP-OES.

PERFORMANCE CRITERIA

Analysis

All analyses are carried out using validated methods which meet strict performance criteria and are valid for the analysis of Aluminium, Calcium, Copper, Iron, Magnesium, Manganese, Potassium, Sodium and Zinc in raw materials and food products.

Recovery

All analyses are conducted with a control IQC sample.

For any further queries please visit our website at paslabs.co.uk
or contact Warren Jackson at warren.jackson@premierfoods.co.uk

METHOD SUMMARY: Free Amino Acids

TEST METHOD NUMBER: C-TM-227

DATE: November 2015

Premier Analytical Services uses the UKAS accredited method C-TM-227 for the Determination of Free Amino Acids by HPLC in Cereal and Cereal Products.

Free amino acids are extracted into dilute hydrochloric acid and cleaned up by Carrez reagent. Sample extracts are derivatised with phthalic dicarboxaldehyde (OPA) and / or 3-mercaptoproprionic acid (3-MPA) and analysed by reverse-phase HPLC with fluorescence detection. Each amino acid is quantified by an internal standard method using norvaline and 2-amino adipic acid as internal standards.

This method is applicable to the analysis of asparagine and 13 other free, primary amino acids, in the following products:

Cereals including whole grains and flour fractions, raw dough, breads, biscuits, etc.

PERFORMANCE CRITERIA

Analysis

All analyses are carried out using validated methods which meet strict performance criteria.

Recovery

All analyses are conducted with a control IQC sample.

For any further queries please visit our website at paslabs.co.uk
or contact Dr Mike Jordan at mike.a.jordan@premierfoods.co.uk

METHOD SUMMARY:
Determination of Total Fat by NMR

TEST METHOD NUMBER: C-TM-267

DATE: November 2015

Premier Analytical Services uses the UKAS accredited method C-TM-267 for the Determination of Total Fat by NMR in raw materials and food products.

When hydrogen protons are placed in a magnetic field they acquire the ability to resonate when irradiated with radio waves of the correct frequency. When the magnetic field and the radio frequency are correctly matched, the protons absorb and re-emit the radio energy. By detecting the emitted energy and measuring its intensity, the resonating protons in the sample can be measured. Signals from protons in solids have short durations whereas signals from protons in liquids last much longer. As samples are dried prior to analysis to remove moisture and then equilibrated at elevated temperature to melt out any solid fats, the NMR can quantitatively measure the liquid portion and hence the fat content.

PERFORMANCE CRITERIA

Analysis

All analyses are carried out using validated methods which meet strict performance criteria and are valid for the analysis of Total Fat by NMR in raw materials and food products.

Recovery

All analyses are conducted with a control IQC sample.

For any further queries please visit our website at paslabs.co.uk
or contact Warren Jackson at warren.jackson@premierfoods.co.uk

Method Name: DETERMINATION OF CHOLESTEROL IN FOODS, FEEDINGSTUFFS, FATS AND OILS.

REF: The method is based upon JAOAC International, Vol. 76, No. 4, 1993, pp902 – 906.

Principle of method: The sample is saponified with alcoholic potassium hydroxide. The non-saponifiable fraction is extracted in hexane and then concentrated. Cholesterol is then quantified by gas chromatography.

List of parameters:

The procedure is applicable to foods, feedingstuffs, fats and oils. The range of application is up to 16g/100g as cholesterol, based upon 10g of sample used. The lowest reporting limit is 5 mg/kg based upon 10g sample extracted to 100ml with a 25ml → 10ml dilution step.

Quality

This method is UKAS accredited

Premier Analytical Services: Vitamin Methods (UKAS accredited)

Team Leader: David Hodgson (david.hodgson@premierfoods.co.uk)

Water Soluble Vitamins

Vitamin B1 (Thiamine) — C-TM-054

Thiamin is extracted by digestion with hydrochloric acid under autoclave conditions, followed by incubation with an α-amylase. Thiamine is separated by ion-pair HPLC and, after post-column reaction with sodium hydroxide/ferricyanide, its concentration is determined as thiochrome using fluorescence detection.

Accreditation scope: Food and food products

LOQ = 0.05 mg/100g

Rel. Uncertainty* = 12.2%

Vitamin B2 (Riboflavin) — C-TM-055

Riboflavin is extracted by digestion with hydrochloric acid under autoclave conditions, followed by incubation with an enzyme mixture containing both phosphatase and amylase activity. Riboflavin is separated by ion-pair HPLC and its concentration determined using fluorescence detection.

Accreditation scope: Food, food products and animal feeds

LOQ = 0.03 mg/100g

Rel. Uncertainty* = 9.5%

Vitamin B3 (Niacin) — C-TM-265

Total Vitamin B3 (Nicotinamide and Nicotinic acid) are extracted by digestion with hydrochloric acid under autoclave conditions. Nicotinamide and Nicotinic acid are separated by HPLC and their concentration determined by MS-MS using isotopic internal standards.

Accreditation scope: Food and food products

LOQ = 0.1 mg/100g

Rel. Uncertainty* = 7.7%

Vitamin B5 (Pantothenic acid) — C-TM-306

Pantothenic acid is extracted by heated digestion in a mildly acidic ammonium acetate buffer and heat-stable α-amylase, followed by dilution with water into the concentration range of the standards. Its concentration is determined by reverse-phase HPLC with MS/MS detection.

Accreditation scope: Food and food products

LOQ = 0.05 mg/100g

Rel. Uncertainty* = 13.7%

Vitamin B6	**C-TM-215**
Vitamin B6 (pyridoxine, pyridoxal and pyridoxamine) is extracted by digestion with hydrochloric acid under autoclave conditions, followed by incubation with an α-amylase. Vitamin B6 forms are separated by ion-pair HPLC and their concentration determined using fluorescence detection. **Accreditation scope:** Food, food products and animal feeds	**LOQ** = 0.05 mg/100g **Rel. Uncertainty*** = 4.0%

Folic Acid (Vitamin B9 – added synthetic form)	**C-TM-287**
Free folic acid is extracted with ammonium acetate buffer in the presence of mercaptoethanol as antioxidant and its concentration is determined by LC-MS/MS. **Accreditation scope:** Vitamin fortified food, food products and animal feeds	**LOQ** = 20 µg/100g **Rel. Uncertainty*** = 11.2%

Vitamin B12 (Cyanocobalamin)	**C-TM-285**
Total vitamin B_{12} is determined as cyanocobalamin, the form used for fortification of foods, using reaction with cyanide to convert naturally occurring cobalamins to the cyano form. Cyanocobalamin is determined by LC-MS/MS. **Accreditation scope:** Food, food products and animal feeds	**LOQ** = 0.1 µg/100g **Rel. Uncertainty*** = 14.3%

Vitamin C	**C-TM-023**
The vitamin C content is determined as the sum of the ascorbic acid (AA) and dehydroascorbic acid (DHAA) levels. These compounds are extracted into an EDTA/sulphuric acid solution, the AA oxidised to DHAA, the total DHAA derivatised and measured by high performance liquid chromatography with fluorescence detection. **Accreditation scope:** Food and food products	**LOQ** = 0.1 mg/100g **Rel. Uncertainty*** = 17.2% based on spike recovery

Fat Soluble Vitamins

Vitamin A (Retinol)	**C-TM-021**
Samples are saponified with alcoholic KOH and the retinol is extracted into hexane. After evaporation of the hexane, retinol is re-dissolved in ethanol and its concentration is determined by reverse phase HPLC with UV detection. **Accreditation scope:** Food, food products and animal feeds	**LOQ** = 10 µg/100g **Rel. Uncertainty*** = 11.5%

Vitamin A (Carotenes) *(not UKAS accredited)*	**C-TM-087**
Samples are saponified with alcoholic KOH and carotenes extracted into hexane. The alpha- and ß-carotenes are determined using reverse phase HPLC with visible detection.	**LOQ** = 10 µg/100g **Rel. Uncertainty*** = 8.1%

Vitamin D	**C-TM-273**
Vitamin D_3 is determined in the absence of vitamin D_2 using vitamin D_2 as internal standard. Alternatively, vitamin D_2 may be determined in the absence of vitamin D_3 using vitamin D_3 as internal standard. Samples are saponified with alcoholic potassium hydroxide and vitamin D_2 (ergocalciferol) and vitamin D_3 (cholecalciferol) are extracted into hexane. Their concentrations are determined using HPLC with UV detection. **Accreditation scope:** Food, food products and animal feeds	**LOQ** = 0.3 µg/100g **Rel. Uncertainty*** = 6.9%
Vitamin E (Tocopherols)	**C-TM-056**
Vitamin E activity is measured by determining α-, β- and γ- tocopherol content. Samples are saponified with alcoholic KOH and the tocopherols are extracted into hexane. After evaporation of the hexane, tocopherols are re-dissolved in ethanol and their concentration determined by reverse phase HPLC with fluorescence detection. **Accreditation scope:** Food, food products and animal feeds	**LOQ** = 0.5 mg/100g **Rel. Uncertainty*** = 8.9%

* **Relative uncertainty is based on QC test data (unless otherwise stated) and is 2x standard deviation/mean value expressed as percentage.**

Sub-contracted Methods

Total Folates

Vitamin B9 may be made up of natural folates of plant or animal origin, or added folic acid, which is a synthetic form of the vitamin. Our in-house method for folic acid only measures this added form and is appropriate for fortified foods. Where natural folates are present we sub-contract to a lab that uses LC-MS to determine both natural and added forms of folate.

Biotin (Vitamin B7)

The uncertainty of measurement for biotin, which is a microbiological turbidity assay, is 16.3%.