FOOD stars — Innovative Food Product Development Cycle: Frame for Stepping Up Research Excellence of FINS

European Commission

By-Product Characterisation Strategies

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Selection of Issues relating to By-product use since 1978

1981 Food oils contamination (Spain)
1998 Dioxins in citrus pulp (Germany, Netherlands)
1999 Dioxins, PCBs in feed (Belgium)
2001 Polycyclic Aromatic Hydrocarbons in olive oil derived from pomace (Spain)
2004 Iodine in soy milk fortified with Kelp (New Zealand)
2008 Dioxins in Pork due to oils in feed (Ireland)
2009 Iodine in soy milk fortified with Kombu (Australia)

Regulations regarding Testing of products for Food in EU

General Food Law 178/2002/EC

Assigns primary legal responsibility for the safety of products on market to business operator

EFSA oversees and enforces this regulation

Requirements for Additional Characterisation

Where Certain claims are made for a food further characterisation is required. This includes -

Novel Food (258/1997/EC)
Food for particular nutritional use (89/398/EC)
Food additives (89/107/EC)
Supplements (2002/46/EC)
Flavourings (91/71/EC, 88/388/EC)

Regulations on Health Claims

Regulated by 1924/2006/EC

Food or component should be well characterised

Characterisation methods are defined for components, mixtures and classes

Beyond Legal requirements

Determination of the Quality of Ingredients from By-Products

Characterisation of products during preliminary trials

Use of analysis to determine where methods can be improved

Review of Characterisation Strategies

TLC - Purity of product, useful for lab-scale checking
Infrared (IR) - Useful fingerprint of product, also functional groups, gross impurities
Ultraviolet (UV) - Useful fingerprint of product
Atomic Absorption (AA) Analysis of trace elements and metals
GC - Purity of product, useful for volatile compounds, impurities
LC - Purity of product, can be combined with other methods
Nuclear Magnetic Resonance spectroscopy (NMR) - Structure of product in solution
MS - Composition of product, determination of purity
Tandem methods GC-MS, LC-MS, LC-NMR
Electrochemical Methods: Sensors, Biosensors

Mass Spectrometry

Both Qualitative and Quantitative

Identifies molecular weight and primary structure of compounds

Highly sensitive method

Can fail to differentiate between different geometric isomers

Nuclear Magnetic Resonance Spectroscopy

Provides detailed information about structure, dynamics and environment of molecules in solution

Used to confirm identity of a substance

High quantity needed, in 2 - 50 mg range

Not a highly sensitive method

Requirement for deuterated solvents

Detailed Chemical Characterisation

LC or GC to show purity

MS to determine molecular weight and formula

NMR to determine detailed structural information

Other methods, IR, UV may complement this

Practical Examples of Methods

Nitrofurans Residue Testing

Nitrofurans are a class of antibiotics banned for use in the EU
Directive 96/23/EC requires programme of self-monitoring for these and other residues in food of animal origin
Teagasc Method uses Liquid Chromatography coupled with Tandem mass spectrometry (LC-MS/MS)
Method detects and quantifies in a single analysis metabolites of four of the main nitrofuran drugs
Validated for liver, muscle, fish, plasma, egg and honey according to 2002/657/EC guidelines
Limit of detection is below 0.10 ug/kg

teagasc
Agriculture and Food Development Authority

Apple Pomace

By-product of juice and cider industries

Contains high levels of antioxidants

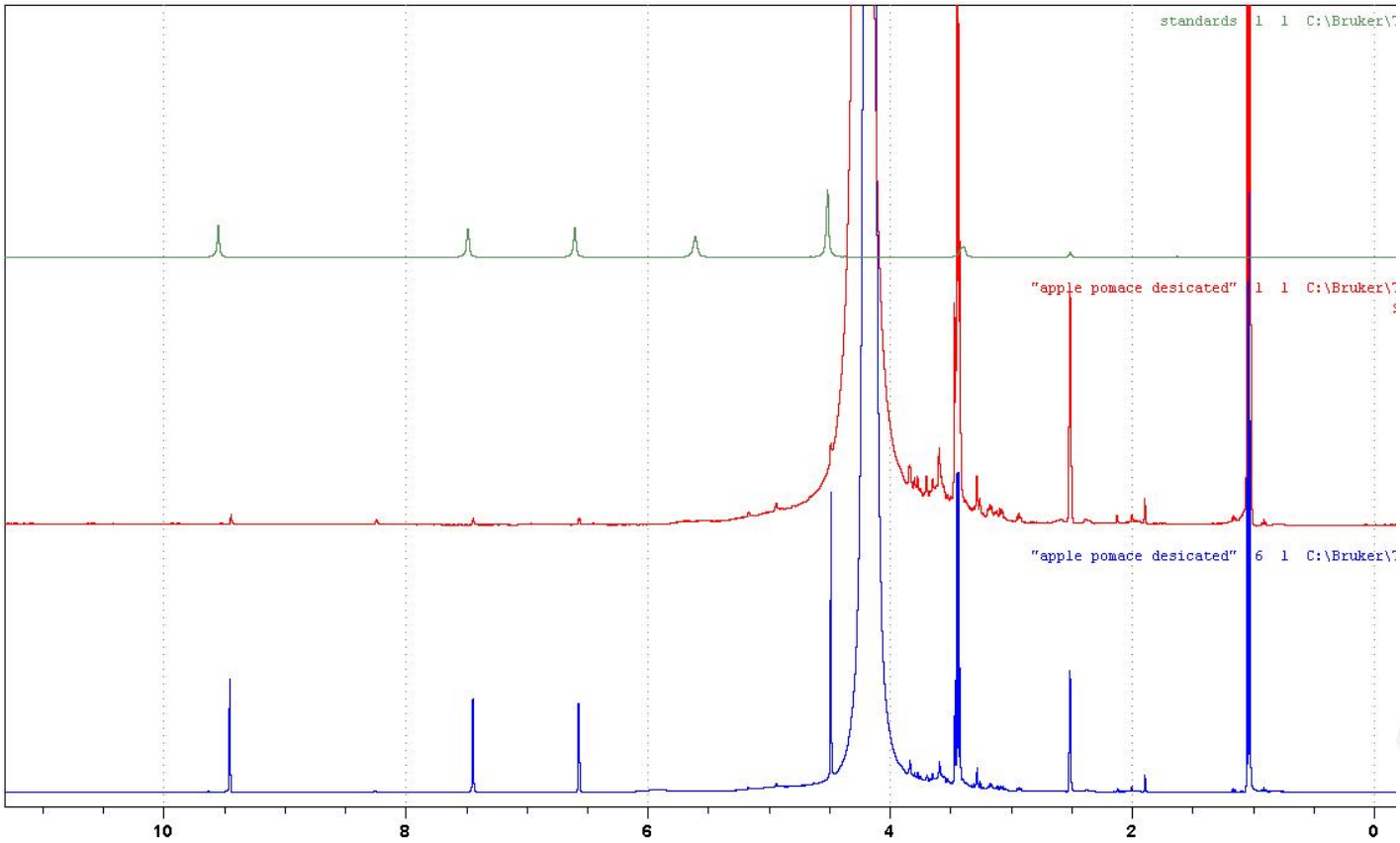
Acidic, High in fibre, High in sugars

Stabilised by oven drying before use

Powdered Pomace or extracts intended as food additive

Distinct off-odour noted

Analysis of crude extract of Pommace by NMR



Analysis of crude extract of Pomace by NMR

Hydroxymethyl Furfural identified as being present

Responsible for off-odour

Product of Maillard-type Reactions

Considered to be an indicator for heat processing of manufactured foods

Not a safety concern but potential to lower quality perceptions

Method altered to exclude oven drying, freeze dry instead

Pectin from pomace - Determination of quality via NMR

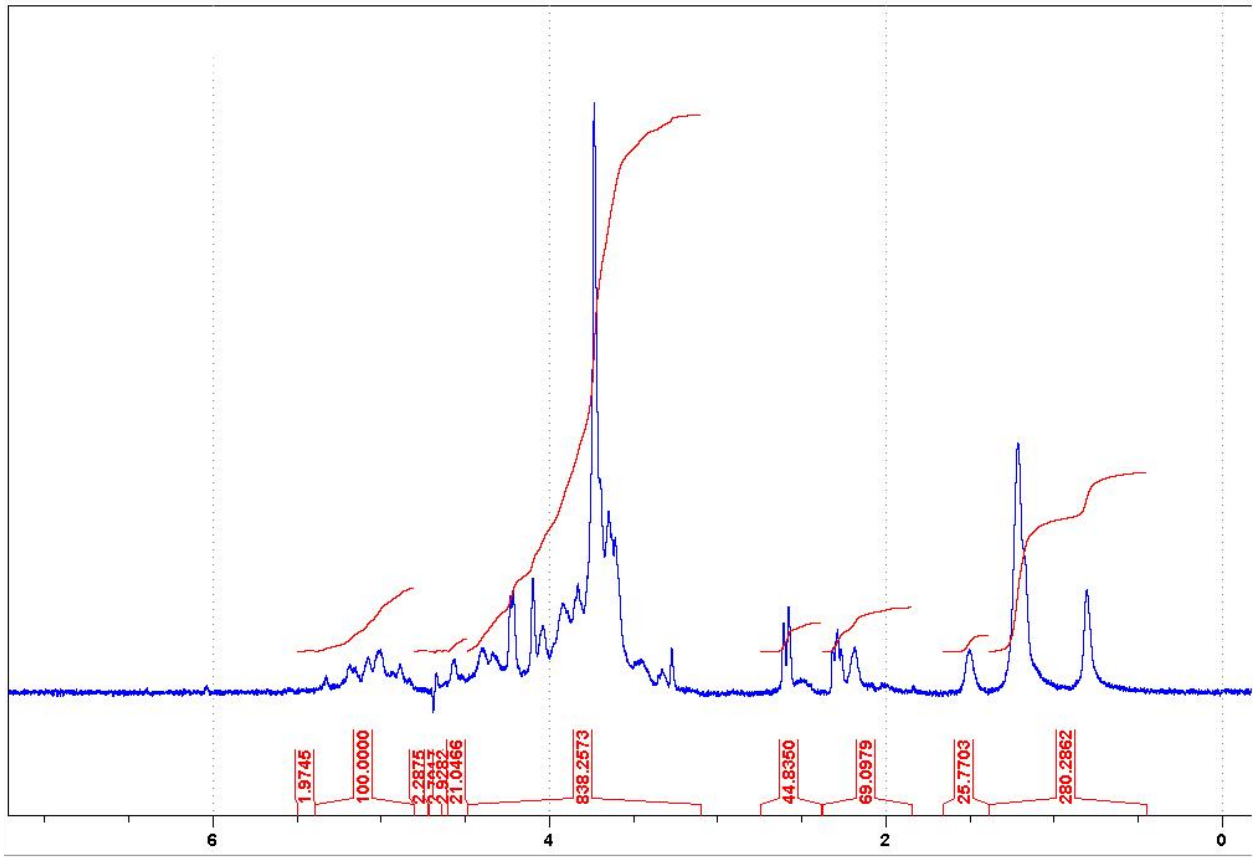
Pectin - Polysaccharide which tends to form gels

Gelling properties related to degree of methoxylation and methylation

Proton NMR used to determine these structural elements

Physicochemical assessment of two fruit by-products as functional ingredients: Apple and orange pomace.

Pectin from pomace - Determination of quality via NMR



N. O'Shea, A. Ktenioudaki, T. P. Smyth, P. McLoughlin, L. Doran, M.A.E. Auty, E. Arendt, E. Gallagher, Journal of Food Engineering, 153 May 2015, (89-95)

Characterisation of Chitosan NMR

Chitosan soluble polycationic derivative of insoluble chitin

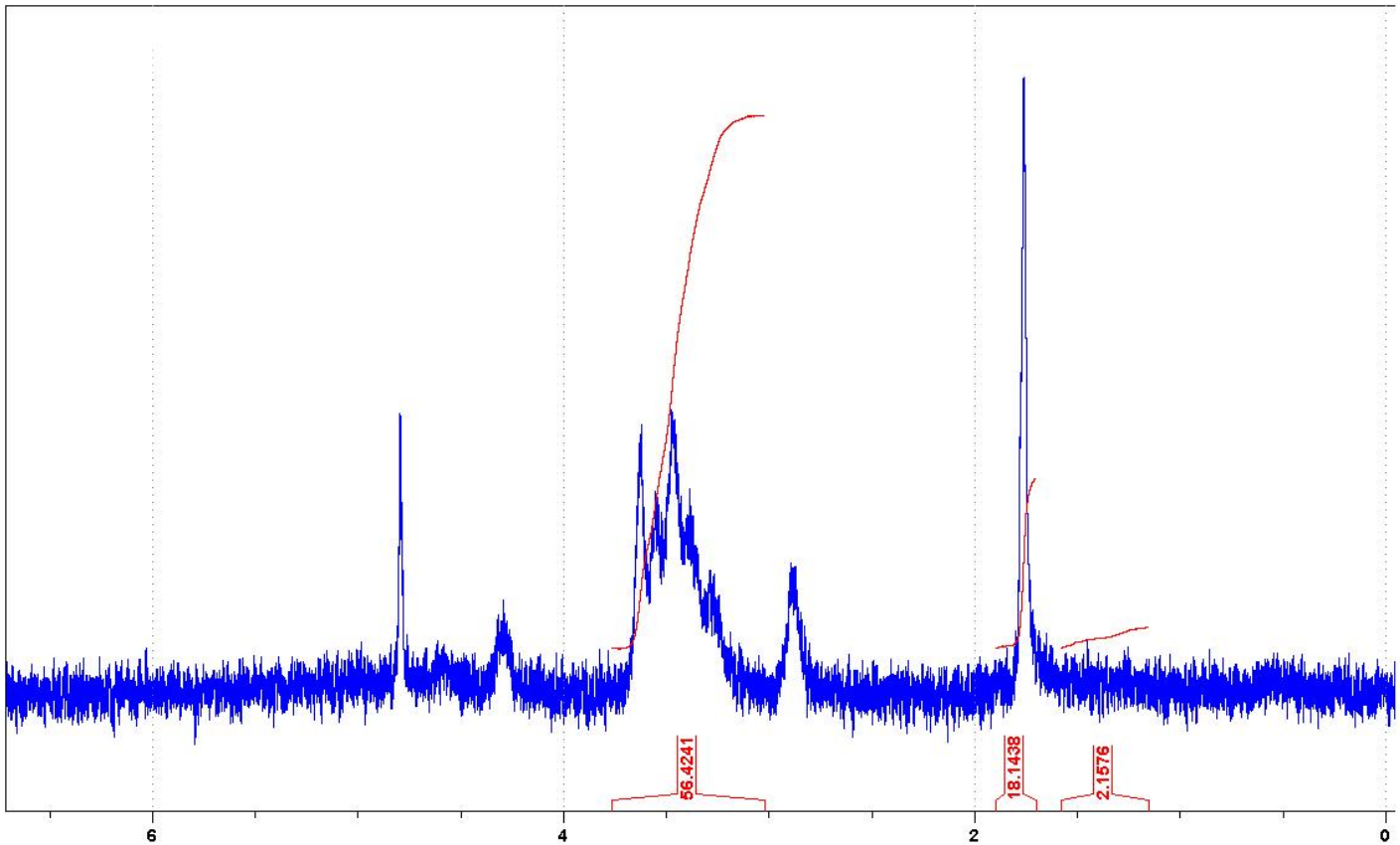
Sparingly soluble

Desired qualities depend on Degree of N-acetylation

Degree of N-acetylation can be determined by NMR

Hydrodynamic characterisation of chitosan and its interaction with two polyanions: DNA and xanthan, M. Hayes, F. M. Almutairii, Tayyibe Erten, G. G. Adams, P. McLoughlin, M. Samil Kik, A. R. Mackie, A. J. Dove, S. E. Harding, Carbohydrate Polymers, 122, 20 May 2015 (359 - 366)

Characterisation of Chitosan NMR



Characterisation of extract of Taraxacum Officinale

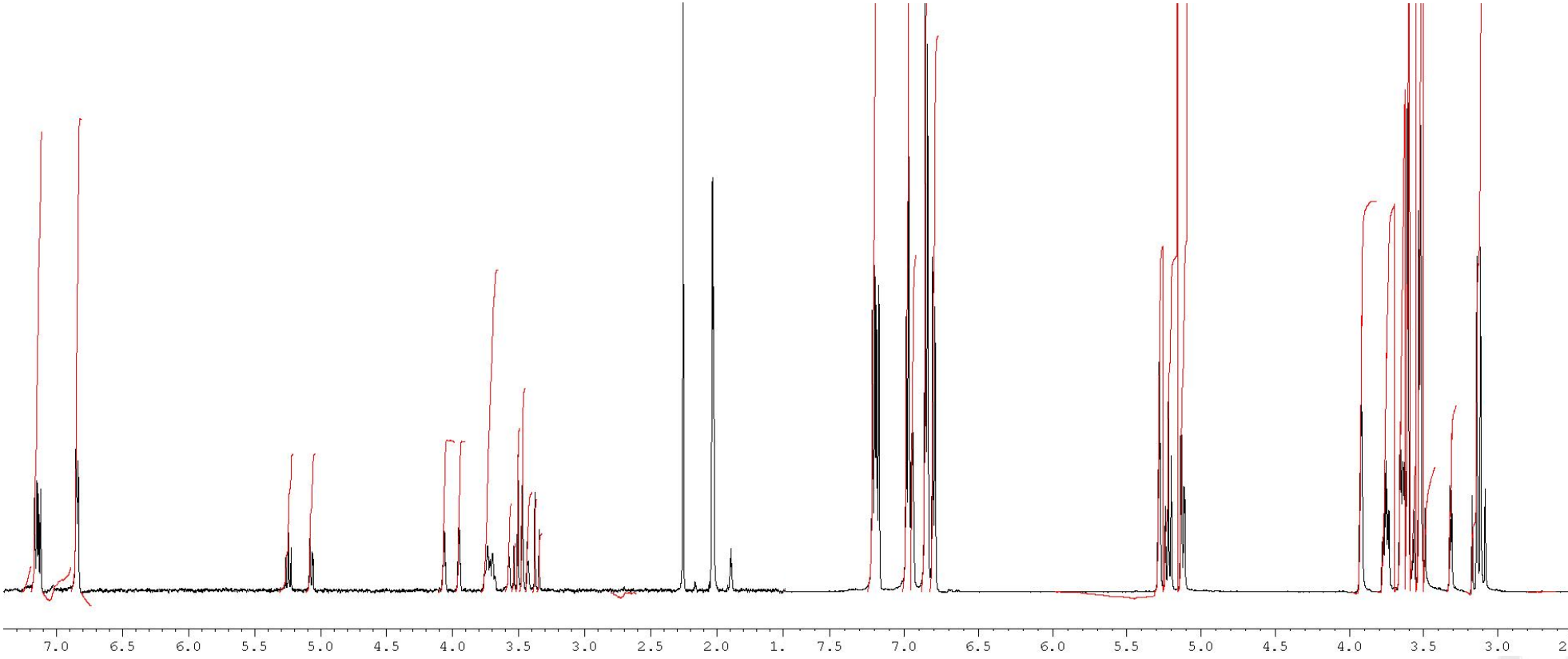
Combination of LC-NMR and LC-MS used on extract

Allowed identification of family of compounds present in extracts

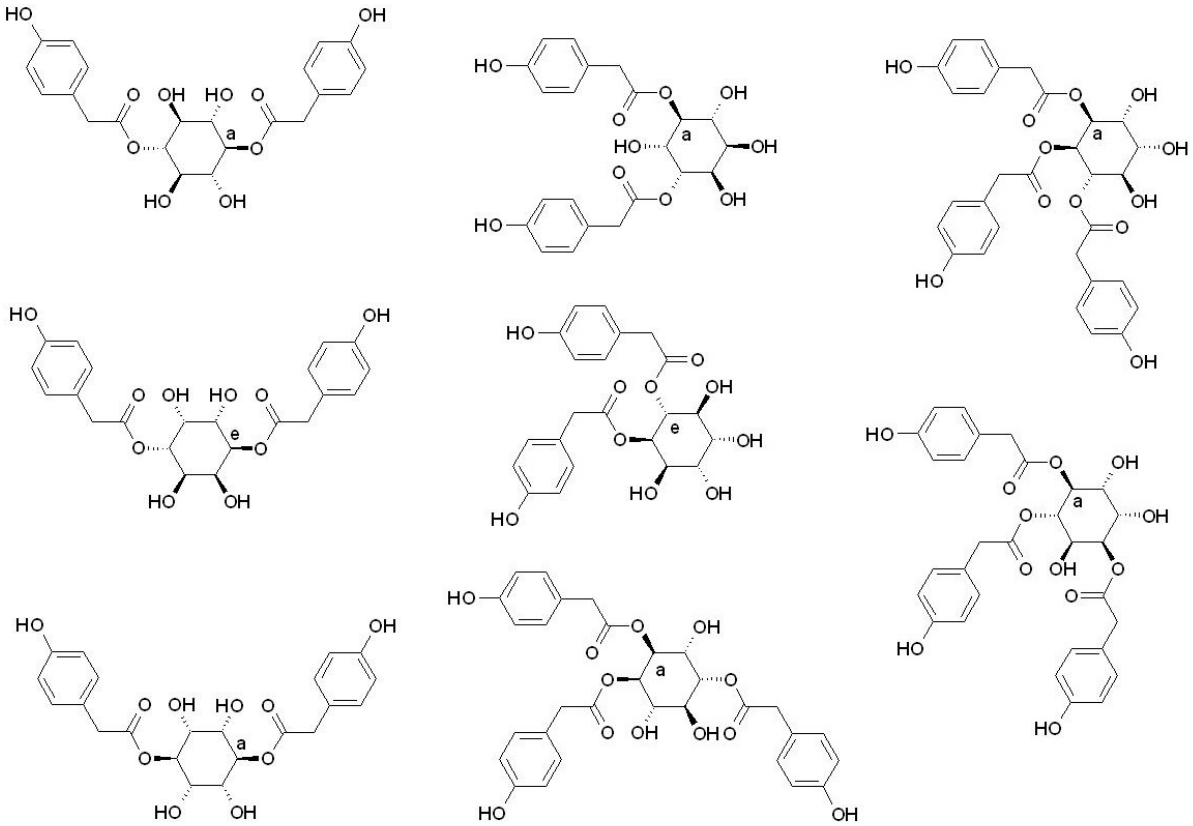
Typical of Strategy to be used for identifying new components.

Additional characterisation for Bioactivity can also be useful

Spectra



Compounds Identified



4-Hydroxyphenylacetic acid derivatives of inositol from dandelion (Taraxacum officinale) root characterised using LC-SPE-NMR and LC-MS techniques, O Kenny, T.J. Smyth, C.M. Hewage, N.P. Brunton, P McLoughlin, Phytochemistry, 98, February 2014, (197-203)

Challenges

Solubility

Sampling error

Matrix effects

Interference

Conclusions

Know regulations for particular product

Be aware of recommended analytical methods

Ensure that method is robust

Methods can be used together to complement each other

Analyse early and often

Any Questions?

Thank you

Hvala

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