



# **TDS culinary operations, sample preparation and chemical analysis**

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## WHERE THIS FITS IN

sampling plan

sample collection

**culinary operations, sample preparation, and  
pooling**

**chemical analysis**

## FOCUS FOR TODAY:

- culinary operations - how to know what consumers do for food preparation and how to replicate this behaviour
- sample preparation - how to demonstrate adequate homogeneity of every sample / composite at every step
- analysis - analytical method performance and impact on overall uncertainty in TDS exposure estimates

## ANALYSED AS CONSUMED – NO MORE AND NO LESS

sampling plan

sample collection

**culinary operations, sample preparation, and pooling**

chemical analysis

"Eye of newt, and toe of frog,  
Wool of bat, and tongue of dog,  
Adder's fork, and blind-worm's sting,  
Lizard's leg, and howlet's wing,--  
For a charm of powerful trouble,  
Like a hell-broth boil and bubble."



## FOOD PREPARATION METHODS USED

How to ensure that consumer practice is mimicked in preparing the individual food samples that make-up the representative composite for the TDS food groups?

- washing, peeling cooking, discarding inedible portions, water used for reconstitution, use or discard of cooked-out fats and juices, .. etc.
- baking, boiling, grilling, or frying, ...etc
- time and temperature conditions.

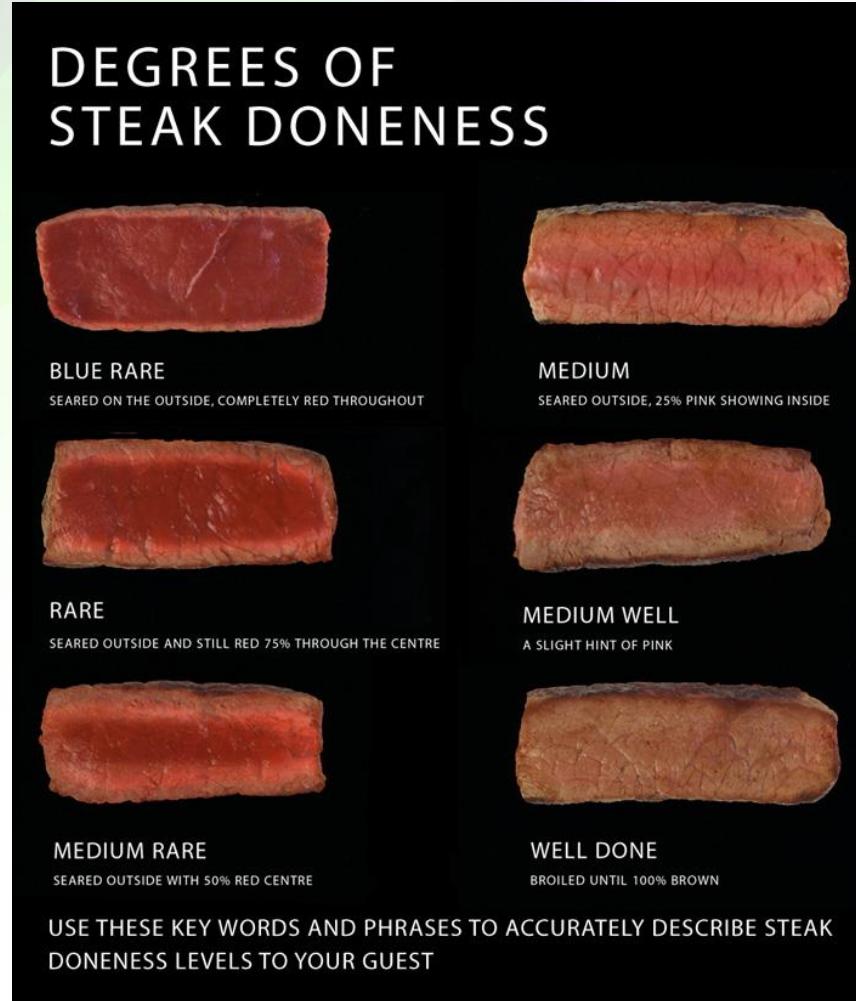
# FOOD PREPARATION METHODS

- take the information from the national food consumption surveys, if it is recorded there
- conduct a survey of consumers (e.g. CZ)
- take from recipe books (but no %age info)
- use instructions on pre-packaged foods (but get no %age preferences)

## EXAMPLE: COOKING TIMES – PROBABLY TOO LONG!



# USE OF A DONENESS COLOUR PALLETTE



# USE OF A BROWNING COLOUR PALLETTE



## FOOD PREPARATION METHODS USED

Yes, many parameters:-

washing, peeling cooking, discarding inedible portions, water used for reconstitution, use or discard of cooked-out parts, etc.  
baking, boiling, grilling, or frying, ...etc  
time and temperature conditions.

- Need to be pragmatic – must reflect general behaviour

## POOLING

- Foods prepared as consumed are pooled in predetermined combinations of food items prior to analysis.
- **The degree of pooling is driven by the purpose of the survey and the funding available.**
- It should consider the need to keep food groups separated, avoid combining foods with very different levels of the chemical substances to be analysed.
- Amounts of the each food item in the pooled sample should be proportional to its approximate share in the **typical diet**.

## EQUIPMENT NEEDED FOR POOLING & HOMOGENISATION

### TDS for screening

A TDS can be used for screening purposes, analysing a limited number of broadly pooled food samples.

Large samples numbers → limited pools = large sizes

### TDS for refined dietary exposure assessment

Includes analysis of a greater number of less pooled samples often separately covering different seasons and regions.

Less pooling = smaller sizes to be homogenised

## HIGH DEGREE OF POOLING: e.g. CLASSICAL UK TDS FOOD GROUPS

1. Bread
2. Miscellaneous cereals
3. Carcass meat
4. Offal
5. Meat products
6. Poultry
7. Fish
8. Oils & fats
9. Eggs
10. Sugars & preserves
11. Green vegetables
12. Potatoes
13. Other vegetables
14. Canned vegetables
15. Fresh fruit
16. Fruit products
17. Beverages
18. Milk
19. Dairy products
20. Nuts

## HOMOGENEITY - MIX A LOT AND JUST HOPE ?

- or demonstrate the procedures used really do achieve homogeneity
- Often not done.
- Analysis can be too imprecise and expensive
- Can use high level easy-to-measure surrogates (e.g. common metals by ICP-MS for water-solubles)
- Fat-soluble surrogates?

## CHEMICAL PROXIES FOR HOMOGENEITY TESTS

Prepared food sample -> pooled -> portions for transport or storage -> received and sampled -> sub-sampled for different tests -> archived -> tested later for some other purpose

### REQUIREMENT

- demonstrate homogeneity,  
and/or
- demonstrate capability of an SOP to make different sample types homogeneous

The target chemical itself often cannot be used – method too expensive, not precise enough

Chemical proxies for ionic, polar and non-polar contaminants: metals, fat, water-soluble or fat-soluble fluorescent dyes, electrical conductivity

# HOMOGENEITY TESTS

The target chemical itself

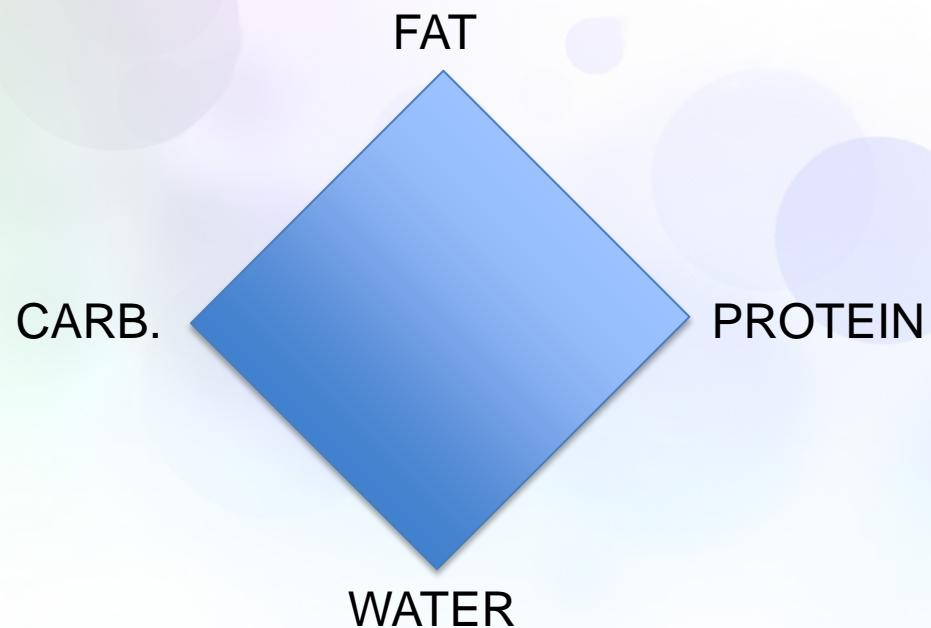
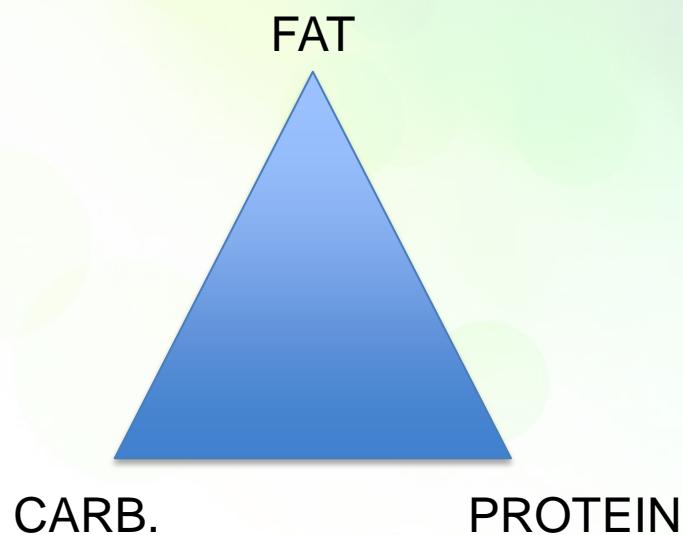
or

· Chemical proxy

Pragmatic test for homogeneity –  
the FAPAS test (duplicate  
specimens taken from each of 10  
portions, analysed in random  
order)

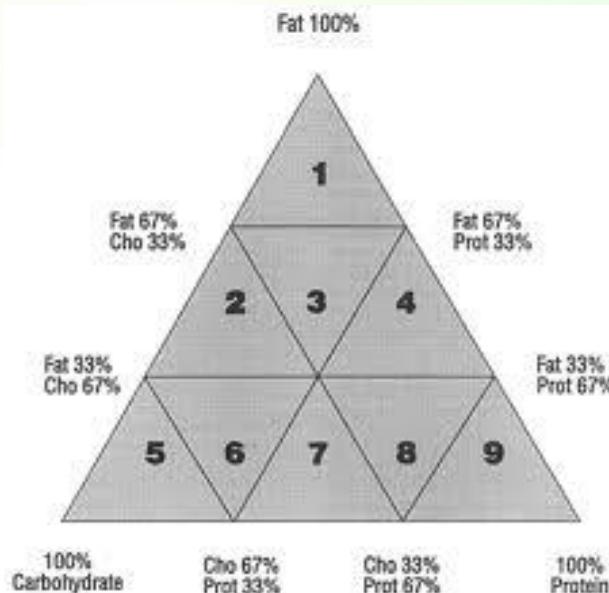
This material could then serve as  
an IHRM included in every batch,  
for a control chart.

# METHOD VALIDATION

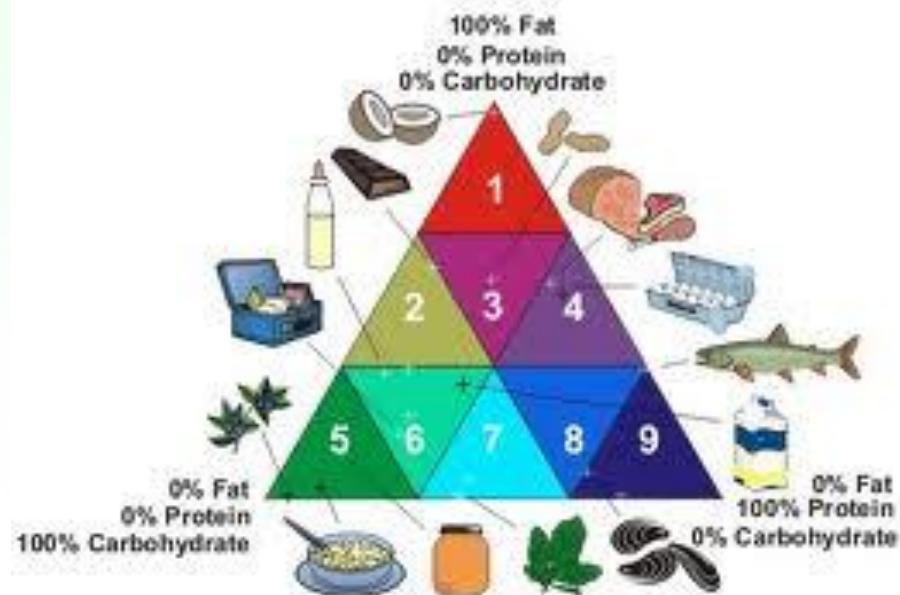


# METHOD VALIDATION

Are composites more complex than single food items?  
Is method validation significantly different ?



fao.org



nist.gov

# CONSIDERATIONS OF MU AND LoQ

## composite #1

## composite #2

## composite #3

## composite #4

## composite #5

## composite #6

## composite #7

## composite #8

## composite #9

## composite #10

## composite #etc

LoQ can be specified to reduce non-detects and uncertainty

Exposure Uncertainty = MU ÷ f [n]

Bias – tendency of a method to give results (concentrations) that are lower- or higher- than the true values

Pooling individual samples also  
loses information and adds  
some uncertainty – WP7

## REVIEW S OF (I) EFFECTS OF COOKING AND (II) ANALYTICAL METHODS

### #1-20 TDS Exposure priority number (ranking)

Class	category of the chemical
Occurrence	what typically are the affected foods
LoI	level of interest (for individual foods, not pooled)
Approach	analytical method(s) used most commonly & their capabilities
Phase	which food phase(s) does the chemical associate with (linked to polarity)
Cooking	is the concentration expected to be influenced by cooking
Speciation	is chemical speciation an issue in the analysis and reporting
TDS study	has the chemical been included successfully in published TDS work
CRMs	are certified reference materials or other AQA materials available
PTs	are proficiency tests routinely conducted for the chemical

## #7. ALUMINIUM

Class	Additive, FCM, general contaminant.
LoI	10-50 ppb
Approach	ICP-MS
Phase	Water
Cooking	Stable
Speciation	Yes (for reasons of bioavailability)
TDS study	Yes, many, so methods exist
CRM	Yes
PTs	Yes, frequent

- Occurrence: most food groups

## #8. ACRYLAMIDE

Class	Processing contaminant, FCM
LoI	5-10 ppb
Approach	LC-MS/MS; Derv.-GC-MS
Phase	Water
Cooking	Highly dependent concs in some foods
Speciation	No
TDS study	Yes, many, so methods exist
CRM	Yes
PTs	Yes, frequent

- ▶ Occurrence: foods with carbohydrate and asparagine that are baked, grilled or fried, e.g. potatoes, cereal products, coffee

## #9. BISPHENOL A

Class	Food Contact Material
LoI	1 ppb
Approach	LC-MS/MS (derv-GC-MS)
Phase	Water and fat (intermediate polarity)
Cooking	Stable (but ↑ if heated in contact)
Speciation	No (... chlorinated, other bis-phenols?)
TDS study	Yes, some, so methods exist
CRM	No
PTs	Yes, frequent

- Occurrence: most food groups, especially those canned and those in contact with polycarbonate. Why is it found in animal products with no known contacts post-mortem?

## #10. MINERAL OIL SATURATED HYDROCARBONS

Class	General contaminant, FCM, Additive, Processing aid.
LoI	100 ppb (not achieved)
Approach	LC-GC-FID; GC-FID
Phase	Fat
Cooking	Stable
Speciation	Yes (wrt MW and degree of branching)
TDS study	No, and methodology is inadequate
CRM	No
PTs	Very limited

- ▶ Occurrence: MOSH are present at different levels in nearly all foods.

# OUTPUTS

analytical methods and their performance  
availability of reference materials  
identification criteria  
capturing and mimicking consumer behaviour  
effects of cooking  
cooking, sample prep and storage utensils  
tests for homogeneity  
stability and storage aspects  
effect of MU and LoQ on uncertainty

TDS\_Exposure - Harmonised not standardised

- Resources
- Guidance documents
- Quality standards

Prepared and in further preparation

## ACKNOWLEDGEMENTS – WP4 PARTNERS

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VITO - Vlaamse Instelling Voor Technologisch Onderzoek NV, BE

# QUESTIONS ? DISCUSSION ?

