Food preparation, composite formation and chemical analysis

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WP4 OBJECTIVES

- O4.1: To ensure food preparation methods reflect consumer practice since this can influence contaminant levels;
- O4.2: To ensure composite formation and storage maintains the integrity of food samples;
- O4.3: To establish requirements and capabilities of analytical methods to perform adequately at the low concentrations expected for diluted composites;
- O4.4: To evaluate the impact of analytical method performance (measurement uncertainty) on uncertainties in the final exposure assessment using TDS samples.

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FOOD PREPARATION

The Joint Guidance document on TDS from EFSA, FAO and WHO (2011) describes why consumer practice should be mimicked but it gives only brief clues on how consumer practice can best be captured and described.

Qualitative and quantitative information:-

- recipe books, on-pack instructions, ...

- consumer surveys as part or (preferably) separate from the Food Consumption (Dietary) Surveys.

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FOOD PREPARATION

As part of Del.4.1 we evaluated the physical and chemical effects of cooking on the top-20 chemicals identified as a priority in this project.

Conclusions and Recommendation. Prior to undertaking a TDS, an evaluation should be conducted for each chemical covered, describing its physical and chemical fate during normal food preparation methods. This evaluation report should guide the formation of the food list. Any assumptions and consequent uncertainties along with their impact on the resulting exposure estimates, should be recorded in an Uncertainty Analysis Table.

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FOOD PREPARATION

Thereafter:

- Record what is intended to be done: means detailed instructions provided in the food list
- Record what actually was done: means keeping records of times, temperatures, weights, photographs etc.

COMPOSITE FORMATION – FOCUS ON HOMOGENEITY

The requirement is two-fold.

- First, before the individual food items are combined into a single composite, they need to be blended separately and then a defined mass taken for pooling. Then portions taken from that composite aredistributed for analysis.

- Second, when portions are received by the analytical lab or when portions are withdrawn from archive, they should be properly re-mixed (or re-homogenised if needed) before sub-sampling for the analysis in question.

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COMPOSITE FORMATION – FOCUS ON HOMOGENEITY

It may be desirable or even necessary to test for homogeneity by using surrogates.

For water-soluble substances (e.g. heavy metals, acrylamide) the elemental composition of the pooled food composite can be tested because the concentrations are high (e.g. sodium, calcium, magnesium) and the analytical method (e.g. acid-digestion followed by ICP-MS) is quick and precise.

Surrogates for other classes of contaminants were not well established.

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COMPOSITE FORMATION – FOCUS ON HOMOGENEITY

A consideration of food types and the composite:

- solids and powders (including granulates and crumbs)
- pastes
- slurries / dispersions / suspensions (i.e. 2- or more phases)
- true liquids

A consideration of analyte types:

- polar / water-soluble analytes
- non-polar / fat-soluble analytes
- intermediate-polarity substances

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COMPOSITE FORMATION – FOCUS ON HOMOGENEITY

As a pragmatic test for homogeneity, it can be recommended to use the procedure of FAPAS®.

Duplicate specimens are taken from each of 10 portions, and these are analysed in random order.

Te difference between duplicates is taken to represent the variability of the analytical method used and then any residual difference between the 10 portions (after allowing for the variability of the analytical method) is taken to represent the (in) homogeneity of the test material.

Fearn, T. and Thompson, M., 2001, A new test for sufficient homogeneity, Analyst, 126, 1414-1417

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COMPOSITE FORMATION – FOCUS ON HOMOGENEITY

Homogeneity tests in every case or SOPs proven to work?

Conclusion and Recommendation:

- For a new chemical (new or just new to that TDS laboratory) demonstrate homogeneity by the preparation of composite samples and analysis of replicate portions.
- If this is not feasible, then use chemical surrogate(s) in place of the target chemical(s).
- At the same time as the above, record the sample preparation process as an SoP such that following that SOP will in future ensure that homogeneity is achieved.

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CHEMICAL ANALYSIS

We dealt with the availability and suitability of methods applied to TDS, identification criteria, LoDs, LoQs and MU.

WPs 4/5 prepared reviews of the analytical methods available for TDS work.

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CHEMICAL ANALYSIS - MU

The nature and origin of analytical measurement uncertainty (MU) and how MU carries through into an estimate of consumer exposure.

Two main features of TDS:-

1. Normally, two or more food composites are analysed for each food group and the results are used to indicate the concentration of the chemical in that food group.
2. In a well-designed TDS with a well-considered food list and with proper pooling, then a (preferably large) number of different food groups will contribute to the estimate of exposure.

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CHEMICAL ANALYSIS - MU

Part 1. Two or more composites of each food group are analysed. Normally under repeatability conditions - i.e. in the same batch

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- taking the relation between the concentration of analyte and the reproducibility relative standard deviation as being described by the modified Horwitz relation
- if we assume “expected analytical performance” rather than “only just fit for purpose”

Then the uncertainty associated with analysis, can be expected to make the following contributions to the total relative standard uncertainty of the mean concentration of analyte in that food group.

Mean concentration	Analytical relative standard uncertainty (%) for means based on: 1 sample	many samples
1 ppb	22	17
10 ppb	22	17
100 ppb	22	17
1 ppm	16	12
10 ppm	22	8.5
100 ppm	8.0	6.0
1000 ppm	5.7	4.2

The effect of MU is not greatly attenuated because, even with multiple measurements of different pooled samples, in a single batch much of the MU is not random but it is linked to e.g. a single analyst, a single instrument, a single set of calibration standards, use of a single set of volumetric glassware etc.

CHEMICAL ANALYSIS - MU

Part 2. If only one food group contributes to consumer exposure, then the analytical MU goes directly into that exposure estimate (to be joined by other uncertainties).

But if a number of food groups contribute to exposure - as should be the case – then the effect of MU is attenuated. If the different food groups are analysed in different analytical batches (as is usually the case) then since MU is random (uncertainty has a probabilistic basis) the MU starts to cancel-out.

The number of food groups does not affect the uncertainty very much unless the exposure contribution made by each group is approximately equal. This is because, simply, if just one or a few food groups dominate then the attenuation effect is similarly limited.

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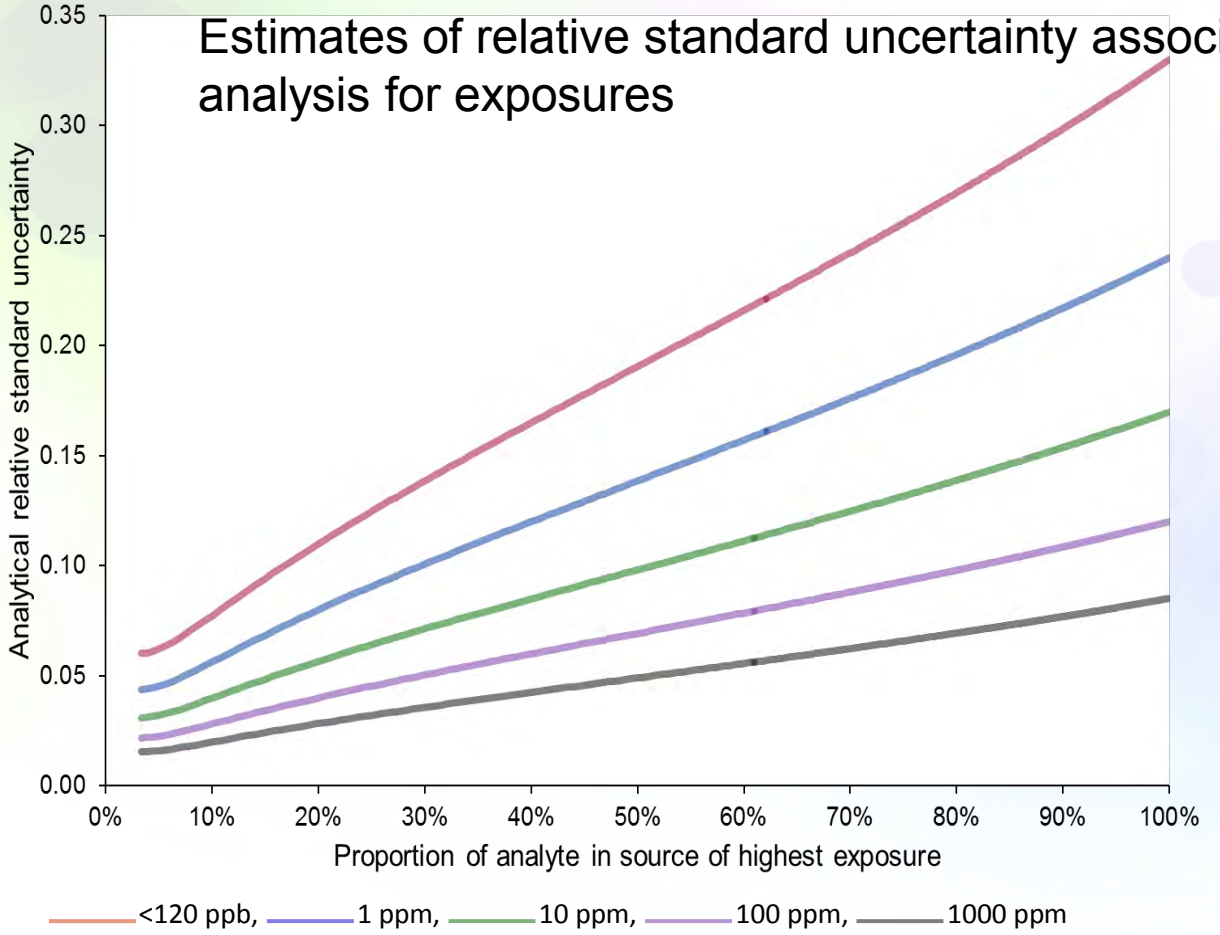

The mathematics of this are described fully in WP4 documents.

CHEMICAL ANALYSIS - MU

Conclusion and recommendation. When setting a target value for MU as a performance criteria, consideration should be given to how many food composites and how many food groups are expected to contribute to the overall estimate of consumer exposure and if they are included in just one or in multiple analytical batches.

In fact, MU is relatively unimportant in TDS work, whereas accuracy (actually, the trueness component of accuracy) along with considerations of LOD and LOQ are far more important.

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CHEMICAL ANALYSIS - ACCURACY

Conclusion and recommendation.

The analytical laboratory should include the analysis of Certified Reference Materials or other traceable reference materials. The availability of CRMs and other quality control tools was summarised in Deliverable D5.8.

If CRMs are not available or are limited in their relevance, then a laboratory should participate in proficiency test exercises analysing test materials containing the target analyte in the most closely-related food matrix. In that case, usually not a 'true' but at least an 'assigned' value is arrived at by consensus

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CHEMICAL ANALYSIS – LODS AND LOQS

There is a clear need to reduce the uncertainty in estimates of exposure brought about by upper-bound and lower-bound estimates resulting from 'non-detects'. So careful consideration is needed when pooling foods, to be sure not to dilute too much.

Conclusion and Recommendation. The tools used in TDS to take concentration data and combine with food consumption data to estimate exposure, should be pre-run but inputting the LoD/LoQ values to calculate the impact of LODs/LOQs on the exposure estimates. The outcome should inform the decision of how much or how little pooling to conduct, and should help to specify if and what improvements in the analytical method performance are needed.

WP4 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 LoD / LoQ on exposure estimate

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