

Development of reference materials:
a case study on packaging material as a source of taints in foods

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**ESN CONFERENCE:
SENSORY ANALYSIS. MORE THAN JUST FOOD**

Session 5: Proficiency testing in sensory analysis.

**Development of reference materials: a case study on
packaging material as a source of taints in foods.**

INTRODUCTION

- Proficiency testing. Test material.
- Sensory analysis of packaging

CASE STUDY: "CALIBSENSORY" project

- creation of calibration material
- feasibility of use of calibration material

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PROFICIENCY TESTING IN SENSORY ANALYSIS

Most of the sensory laboratories concern about validity of their work in an internal way..... but, what about checking validity of results externally?

Proficiency testing: based on the use of inter-laboratory test comparisons to determine the performance of individual laboratories for specific tests.

Gives a laboratory independent information about performance of its sensory panel compared to other panels.

SENSORY EVALUATION OF PACKAGING MATERIAL

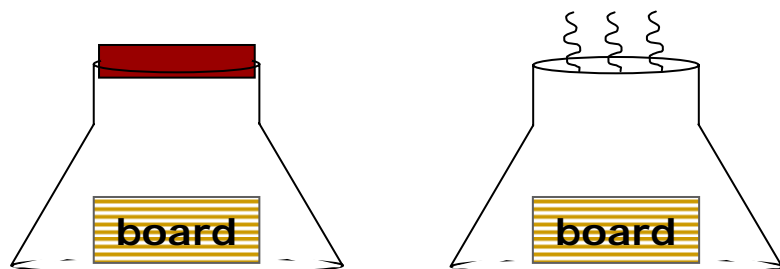
Food packaging materials should not smell or cause off-flavour in packed food.

The Regulation (EC) No 1935/2004 related to materials intended for contact with foodstuffs implies that materials must not transfer their constituents to foodstuffs in quantities which could bring about an unacceptable deterioration in organoleptic characteristics.

Available standards about sensory testing of packaging material related to transference of taints to foodstuff: DIN 10955, "Robinson test", EN 1230, ISO 13302, ASTM standards (E619-84, E460-88, E462-84,

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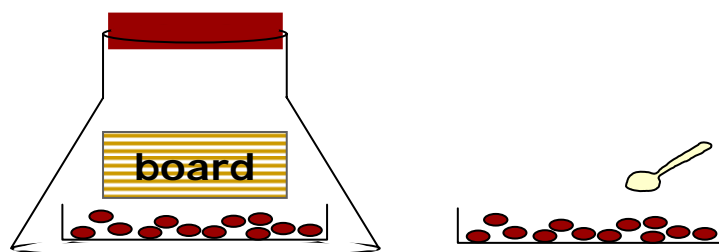
EN 1230-1: Paper and board intended for contact with foodstuffs – Sensory analysis – part 1: Odour.



ODOUR TEST

“Assessment of inherent odour of the packaging material: the packaging material under test is stored in a container under controlled conditions. The odour of the atmosphere developed upon confinement is assessed by means of sensory analysis methods”.

EN 1230-2: Paper and board intended for contact with foodstuffs – Sensory analysis – part 2: Off-flavour (taint).



OFF-FLAVOUR TEST “Assessment of the effect of the packaging material on the flavour of the foodstuff: The foodstuff and the packaging material under test, with direct or indirect contact, are stored in a container under controlled conditions. The flavour changes of the foodstuff are assessed by means of sensory analysis methods”.

Several sensory methods for testing packaging material (both odour test and off-flavour test): paired comparison test, triangle test, duo-trio test, extended triangle test, ranking test, scoring test (multiple comparison test).

Common sensory method: scoring the intensity of the odour or off-flavour according to a five-point scale:

0	no perceptible off-odour / no perceptible off-taste
1	off-odour just perceptible / off-taste just perceptible
2	weak off-odour / weak off-taste
3	clear off-odour / clear off-taste
4	strong off-odour / strong off-taste

(DIN 10 955 scale)

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Subjective interpretations of descriptions such as weak or strong.

Different results from panel to panel, and even to different individuals.

For helping in anchor the term slight or strong: specific reference standard that shows this lower level versus a stronger level.

Standards do not include instructions of how to calibrate the measurement tool and/or how to use any reference materials.

Need for common means for calibration of the testing method.

CALIBSENSORY

Calibration of sensory testing of food contact materials – paper and board

Project funded by the European Community under the
“Competitive and Sustainable Growth” Programme (1998-2002)

REREFERENCE MATERIAL

“Reference material is a material or substance one or more of whose property values are sufficiently homogeneous, stable, and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials” (ISO Guide 30:1992).

Reference material is used:

to help develop accurate methods of analysis

to calibrate measurements systems

to assure the long-term adequacy and integrity
of measurement quality assurance programs

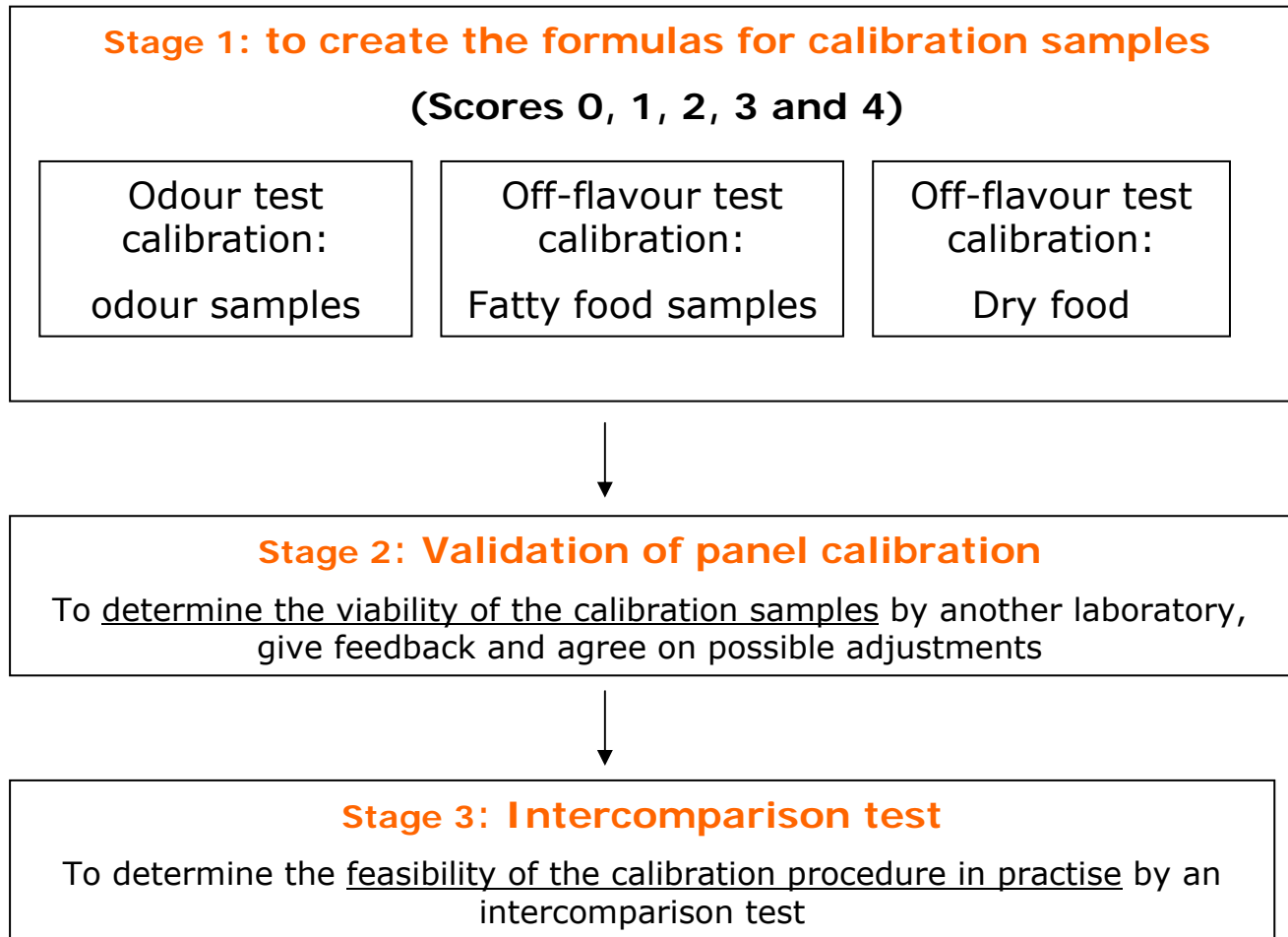
CASE STUDY: CALIBSENSORY PROJECT

AIM OF THE PROJECT: TO ESTABLISH A CALIBRATION PROCEDURE FOR SENSORY TESTING OF PAPER AND BOARD FOOD CONTACT MATERIALS.

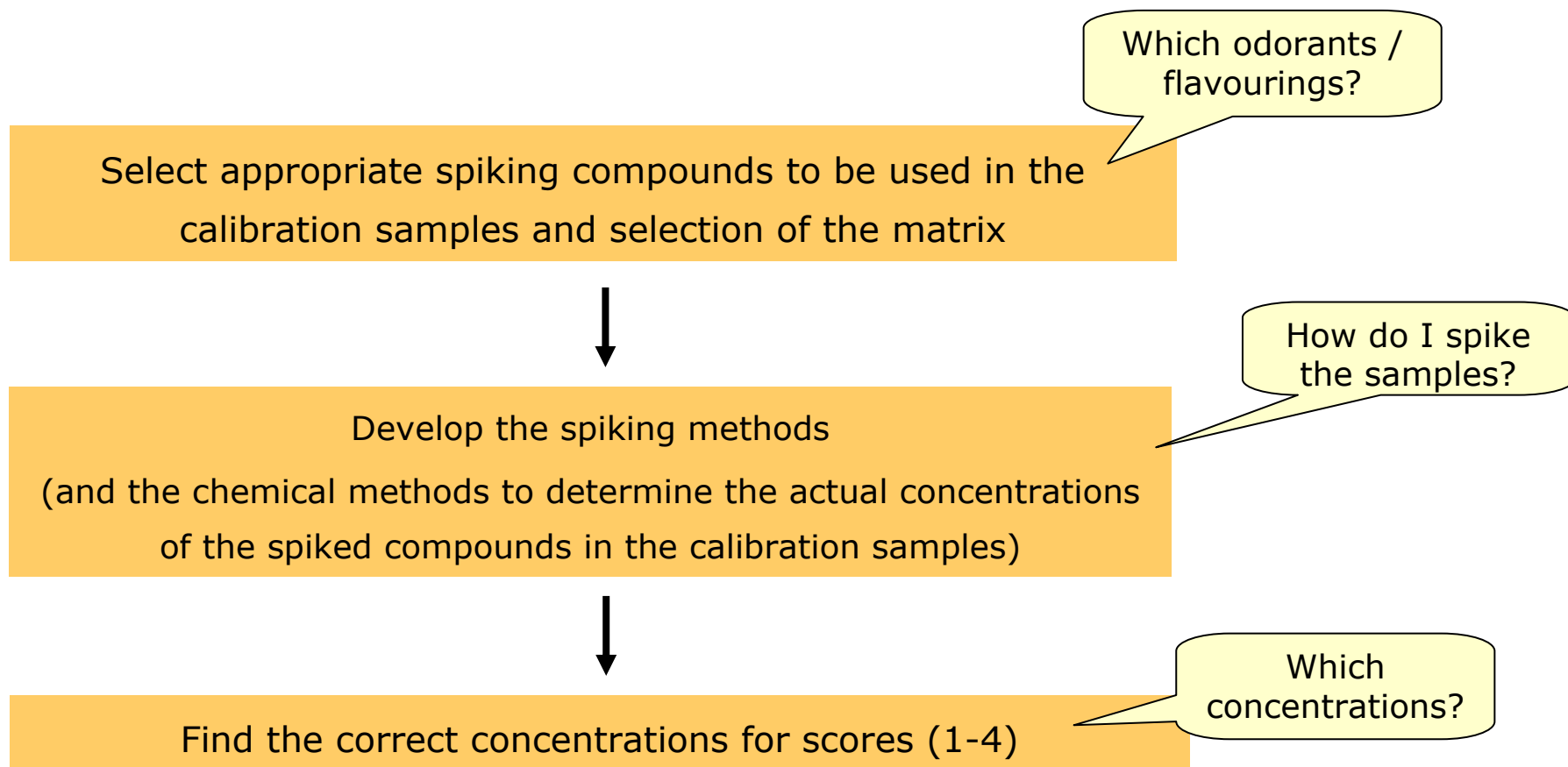
1st work objective: To develop procedures for the preparation of calibration samples:

- for odour test by spiking odourless paper with a defined compound
- for off-flavour test by spiking foods

2nd work objective: To establish a validated training procedure for sensory panels on how to use these calibration samples.

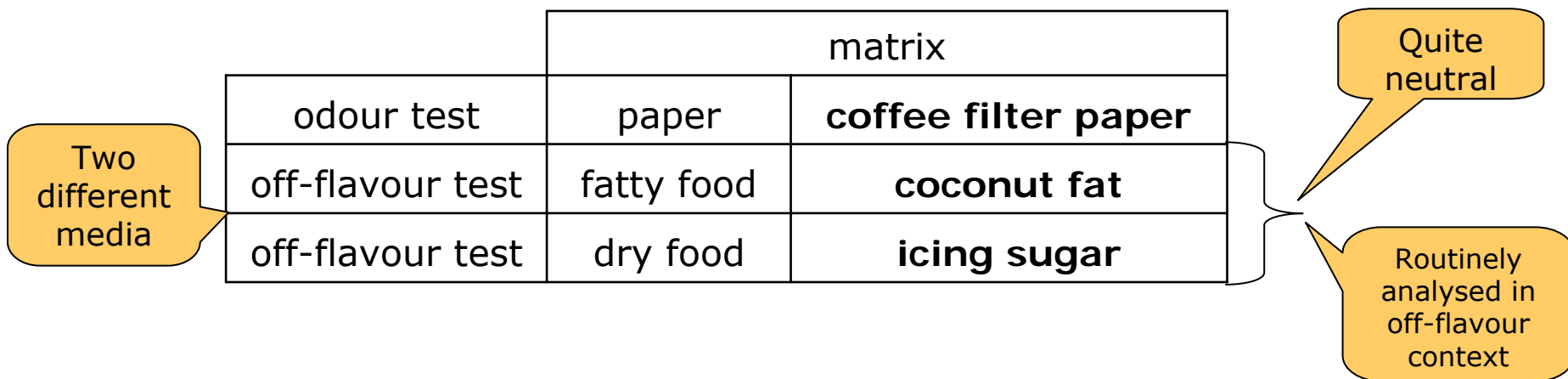


creating formulas for calibration samples: matrix + spiking compound



selection of appropriate **matrix**

Kind of reference material: a neutral matrix with addition of “additives”.



Conditions for selection of the provider for matrix:

- Paper: for alimentary use, odourless, high quality, easily available.
- Dry food and fatty food: food grade, free of off-flavours, high quality, easily available

selection of appropriate **spiking compounds**and a solvent

Additives: spiking compounds.

	matrix	spiking compound	
odour test	coffee filter paper	Hexanal	+ triacetin
off-flavour test	coconut fat	Benzaldehyde	
off-flavour test	icing sugar	Benzaldehyde	+ triacetin

Conditions for selection of spiking compounds:

- Flavour related to paper and board, safety (for alimentary use EU).

selection of the **spiking method**

	matrix	spiking compound	Spiking method
odour test	coffee filter paper	Hexanal	direct addition to paper
off-flavour test	coconut fat	Benzaldehyde	direct addition to fat
off-flavour test	icing sugar	Benzaldehyde	indirect contact / adsorption

Spiking method: odour



- Preparation of the hexanal dilutions
- Cutting and folding the paper
- Put paper inside the flask
- Spike paper with hexanal solution and close the flask
- Store the flasks for 3 hours



- Each assessor have their own flasks
- Turn the flask once up and down
- Open the flask and immediately sniff

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Spiking method: off-flavour, fatty food



Preparation of benzaldehyde dilutions in melted coconut oil



Pour spiked coconut oil onto holes of the mould

Freeze test portions



Drop down the test portions and serve them

Spiking method: off-flavour, dry food



Prepare benzaldehyde solutions in triacetin

Arrange icing sugar inside a dessicator

Place benzaldehyde solutions and close dessicator

Let benzaldehyde adsorb onto icing sugar for 24 hours

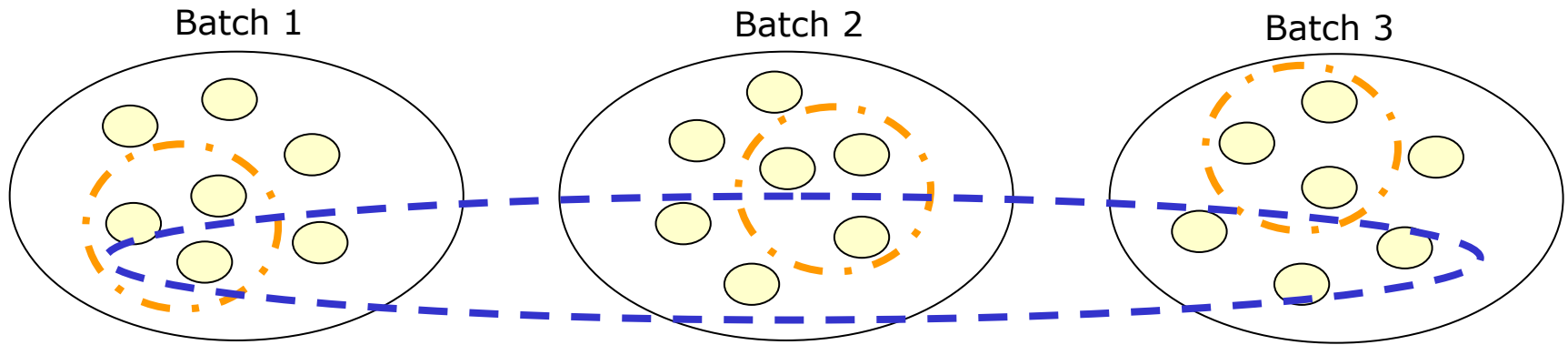


Close and remove spiked icing sugar test portions

Before assessment of icing sugar add mineral water and stir

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Validation of the spiking method...reproducibility of the spiking procedure



Are samples similar?

Homogeneity within batches

CHEMICAL ANALYSIS

FUNDAMENTAL!!!

Are samples similar?

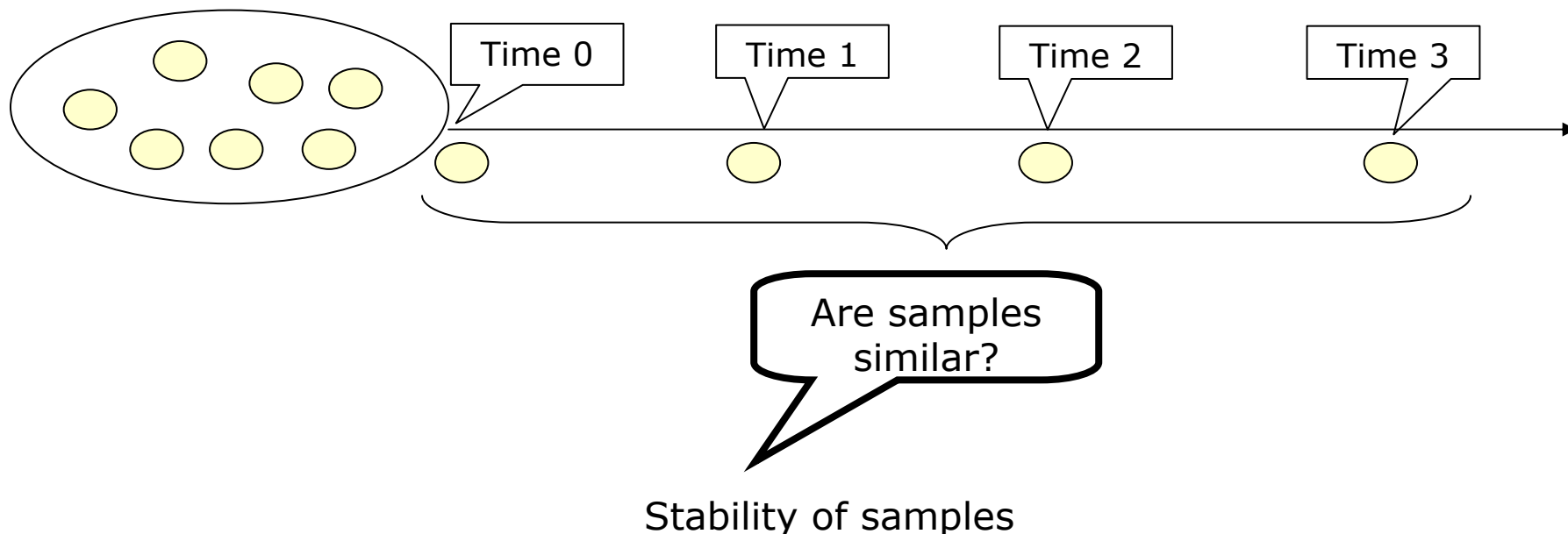
Homogeneity between batches

CHEMICAL ANALYSIS

SENSORY ANALYSIS

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Fresh samples



If samples are stable:

Possibility of being prepared in one lab and send to others

More practical: no need to assess immediately

CALIBRATION SAMPLES.....

	HOMOGENEITY OF SAMPLES WITHIN BATCHES	HOMOGENEITY OF SAMPLES BETWEEN BATCHES	STABILITY
FATTY FOOD	VERY HOMOGENEOUS	VERY HOMOGENEOUS	VERY STABLE DURING 4 WEEKS AFTER PREPARATION
DRY FOOD	HOMOGENEOUS	HOMOGENEOUS	SHOULD BE ASSESSED WITHIN 2 HOURS AFTER PREPARATION
ODOUR	HOMOGENEOUS	HOMOGENEOUS	SHOULD BE ASSESSED IMMEDIATELY AFTER PREPARATION

Other spiking compounds...

FATTY FOOD:

Diacetyl: good results, although not as good as benzaldehyde

Octanal: large variations in sensory thresholds between assessors

ODOUR:

Butyric acid: not full tested. Seemed to work

Diacetyl: problems of adaptation, large variations between and within assessors

FINDING CONCENTRATIONS FOR CALIBRATION SAMPLES

Concentrations for calibration samples for each medium were proposed by each partner / laboratory

Concentrations of spiking compound in calibration samples are adjusted specifically to match a score of 0 to 4 on the recommended sensory scale.

Score	Descriptions	Odour- Base paper	Off-flavour - Dry food	Off-flavour - Fatty food
		μg Hexanal/ g base paper	μg Benzaldehyde/ g icing sugar (ppm)	μg Benzaldehyde/ g coconut oil (ppm)
0	no perceptible	0	0	0
1	just perceptible	0.04	3.22	10
2	Weak	0.8	6.44	40
3	Clear	8	14.63	160
4	strong	-	30.32	660

Assessment: scoring. Multiple comparison test. Deviation from reference (score 0: no off-odour or off-flavour)

DIN scale has been used

FINDING CONCENTRATIONS FOR CALIBRATION SAMPLES

Detection
threshold
oversized to the
double app.

Identification
threshold
oversized to the
double app.

DIN scale has been used

0	no perceptible off...
1	Off...just perceptible
2	Weak off...
3	Clear off...
4	strong off...

Assessment: scoring. Multiple comparison test. Deviation from reference (score 0: no off-odour or off-flavour)

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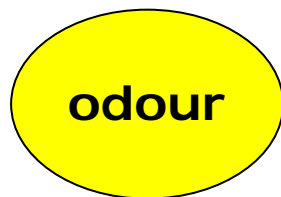
Creation of
samples by
one lab



Lab 1



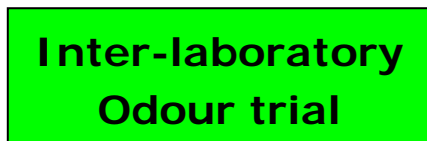
Validation of
samples by
another lab



Lab 4



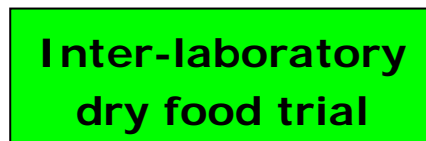
Validation of
samples by
many labs



Lab 2



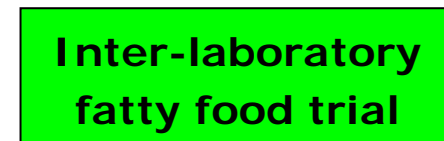
Lab 5



Lab 3



Lab 6



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Validation of calibration samples by another laboratory

Objectives

Establishment of a procedure on how to use the samples for panel calibration

Feedback procedure and possible adjustment if necessary

Procedure on how to use samples:

Assessment without formal training (2 sessions)

Training step (1 session)

Assessment after training (2 sessions)

Analysis of results. Comparison between results of 1st lab and 2nd.

Odour test: only scores 0, 1, 2 and 3 are reliable and should be used.

off-flavour test, for dry food: training had a positive effect.

Off-flavour test, fatty food: training had an extremely positive effect.

INTER-COMPARISON TRIALS

Final stage of the project:

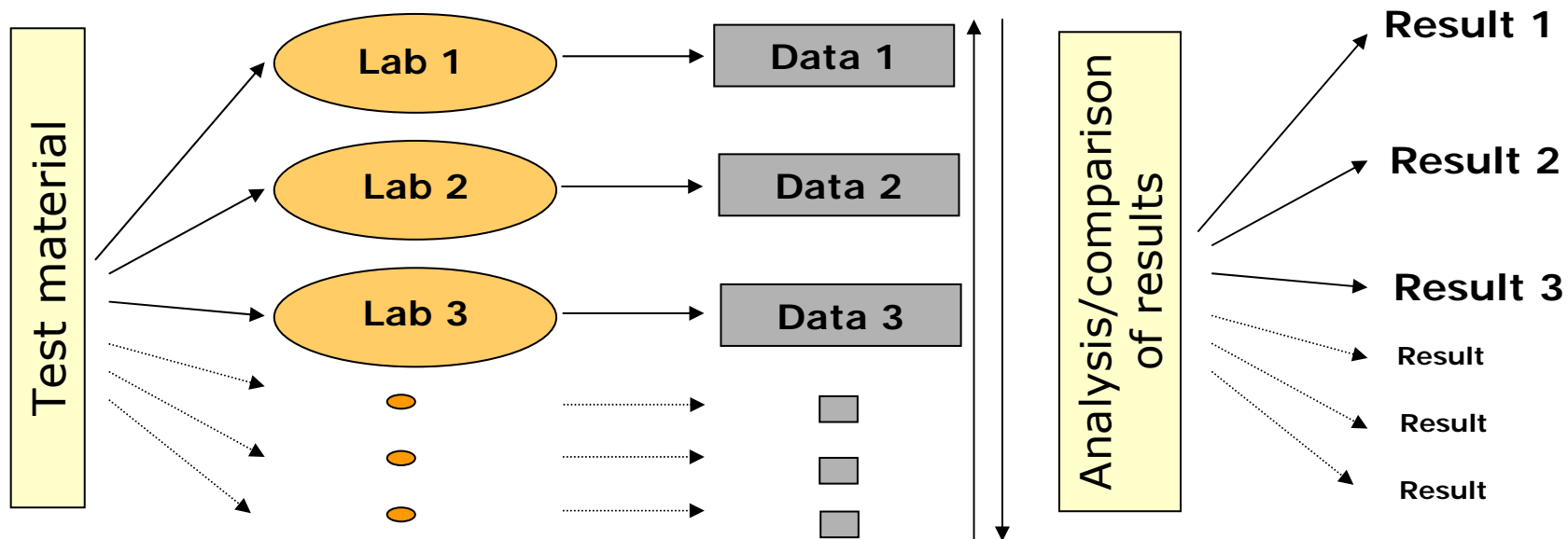
to determine feasibility of the calibration procedure in practise
by means of inter-laboratory test

One inter-laboratory for each kind of test samples: odour, dry and fatty food

working objectives:

- To evaluate the calibration samples (concentration and preparation instructions)
- To validate the calibration procedures (including panel training)
- To determine feasibility in practice of the overall procedures.

Basis of an inter-laboratory test schedule



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To participants: detailed instructions about materials and reagents needed, preparation of test materials and test portions, test procedure, sensory evaluation and scale, ...

Trial	odour	fatty food	dry food
Nº of participants	15	18	8
Material provided	Paper	Pre-spiked coconut fat	Icing sugar
Order in time	2 nd (Jan-Feb 04)	1 st (Nov-Dec 03)	3 rd (Mar-Apr 04)
Calibration samples	0, 1, 2 and 3	0, 1, 2, 3 and 4	0, 1, 2, 3 and 4

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Panel calibration procedure: {
assessment of samples before training (2 rep)
training session (1 rep)
assessment of samples after training (2 rep)

participants also complete:

a checklist to record any deviation from instructions (immediately after the trial)

a post-participation questionnaire (to help assess feasibility in practice)

REQUIREMENTS OF TEST MATERIAL

General requirements for test material:

Safety

stability

homogeneity

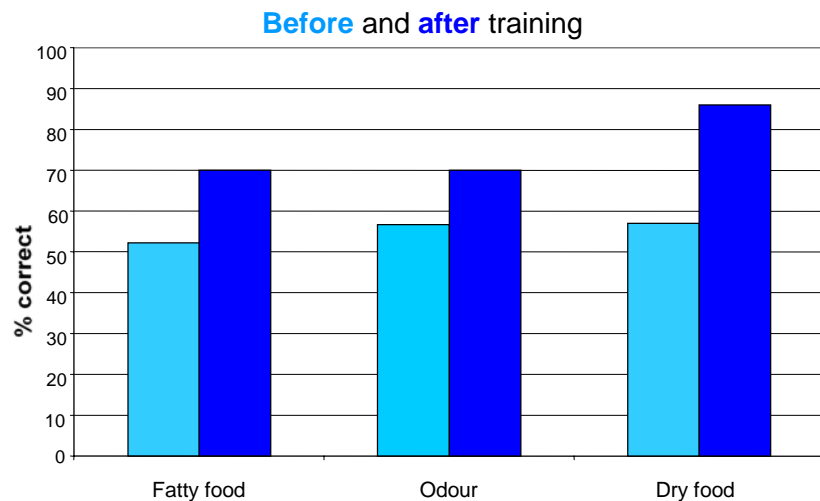
Special requirements for test material:

Similarity of test material to the type of products routinely analysed

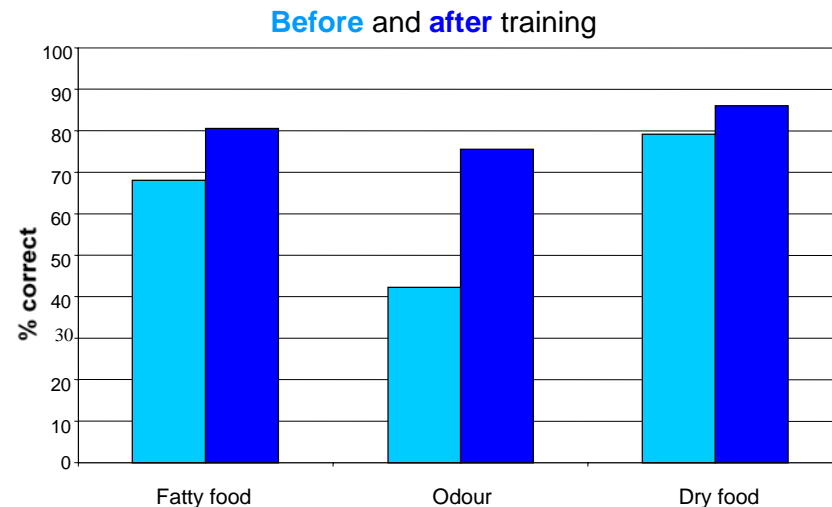
Sensory distances between test samples

Sample preparation, conditions of testing...

results of intercomparison trials



Overall percentages of correct median values.



Overall percentages of correct discrimination between sample pairs.

- For all the trials % of correct median increased after training
- Odour trial was the most difficult and the one with biggest improvement in discrimination between sample pairs

from post-participation questionnaire:

"feasibility of the trials seems to decrease with increasing difficulty and preparation time"

"labs have the feeling that the overall test procedure has helped raise the standard of the panel"

- Fatty food: very feasible in practice (simple and robust spiking method) and samples stable
- Dry food: no very practical (time and cost) but it works
too much dependence on very specific equipment
further research?
- Odour: validity?:
difficulties in finding differences between scores 0 and 1
no reference for score 4


CONCLUSIONS

Have been these procedures effective in calibrating the panels?

Panel performance has improve by training with the calibration samples

...but, will the participant panels produce more harmonized results when assessing a new and real set of samples???

Main output:
calibration methods for odour
and off-flavour



- Preparation of calibration samples
- Assessment before training
- Training
- Assessment after training
- Analysis of results

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European standardisation: “Paper and board intended for contact with foodstuffs – Sensory analysis – part 1: Odour & Part 2: Off-flavour”

will be revised in 2006 by the corresponding working group of the European Committee for standardization
(CEN for pulp, paper and board)

It will be proposed:

to include the calibration methods eg. as Technical documents

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CALIBSENSORY CONSORTIUM

KCL, Paper Industry R & D
Finland
Coordination



VTT Biotechnology, Food Industry R&D
Finland
panel calibration



CTP (Centre technique du papier)
Paper Industry R & D
France
Odour



UNEW University of Newcastle Upon Tyne
United Kingdom
Statistics



ainia centro tecnológico, Food Industry R & D
Spain
Dry food



CCFRA Campden & Chorleywood Food
Research Association Group
United Kingdom
Intercomparison



Iggesund Paperboard, Manufacturer of board
Sweden
Fatty food



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CALIBSENSORY PROJECT SPECIFICATIONS

Contract: G6RD-CT-2001-00514

Starting date: 1.9.2001

Duration: 36 months

Project funded by the European Community

under the 'Competitive and Sustainable Growth' Programme (1998-2002)

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Proficiency testing..... assessment of test material by panels, to compare lab results and check panel performance

In "CALIBSENSORY" project

creation of calibration material

feasibility has been checked by means of inter-laboratory trials (panel performance before and after training with calibration samples)

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THANK YOU FOR YOUR ATTENTION !!!