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.

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María Lorente
INTRODUCTION

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

CASE STUDY: “CALIBSENSORY” project

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

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**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”.


**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”.

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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:

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>no perceptible off-odour / no perceptible off-taste</td>
</tr>
<tr>
<td>1</td>
<td>off-odour just perceptible / off-taste just perceptible</td>
</tr>
<tr>
<td>2</td>
<td>weak off-odour / weak off-taste</td>
</tr>
<tr>
<td>3</td>
<td>clear off-odour / clear off-taste</td>
</tr>
<tr>
<td>4</td>
<td>strong off-odour / strong off-taste</td>
</tr>
</tbody>
</table>

(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)
"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.
Stage 1: to create the formulas for calibration samples
(Scores 0, 1, 2, 3 and 4)

- Odour test calibration: odour samples
- Off-flavour test calibration: Fatty food samples
- Off-flavour test calibration: Dry food

Stage 2: Validation of panel calibration
To determine the viability of the calibration samples by another laboratory, give feedback and agree on possible adjustments

Stage 3: Intercomparison test
To determine the feasibility of the calibration procedure in practise by an intercomparison test
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Creating formulas for calibration samples: matrix + spiking compound

1. Select appropriate spiking compounds to be used in the calibration samples and selection of the matrix
   - Which odorants / flavourings?

2. Develop the spiking methods
   (and the chemical methods to determine the actual concentrations of the spiked compounds in the calibration samples)
   - How do I spike the samples?

3. Find the correct concentrations for scores (1-4)
   - Which concentrations?
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**selection of appropriate matrix**

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

<table>
<thead>
<tr>
<th>Test</th>
<th>Matrix</th>
</tr>
</thead>
<tbody>
<tr>
<td>odour test</td>
<td>paper</td>
</tr>
<tr>
<td>off-flavour test</td>
<td>fatty food</td>
</tr>
<tr>
<td>off-flavour test</td>
<td>dry food</td>
</tr>
</tbody>
</table>

- **Coffee filter paper**: Quite neutral
- **Coconut fat**: Routinely analysed in off-flavour context
- **Icing sugar**: Two different media

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

Additives: spiking compounds.

<table>
<thead>
<tr>
<th>matrix</th>
<th>spiking compound</th>
</tr>
</thead>
<tbody>
<tr>
<td>odour test</td>
<td>coffee filter paper</td>
</tr>
<tr>
<td>off-flavour test</td>
<td>coconut fat</td>
</tr>
<tr>
<td>off-flavour test</td>
<td>icing sugar</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th></th>
<th>+ triacetin</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hexanal</td>
<td></td>
</tr>
<tr>
<td>Benzaldehyde</td>
<td></td>
</tr>
<tr>
<td>Benzaldehyde</td>
<td></td>
</tr>
</tbody>
</table>

Conditions for selection of spiking compounds:

- Flavour related to paper and board, safety (for alimentary use EU).
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### Selection of the **spiking method**

<table>
<thead>
<tr>
<th>matrix</th>
<th>spiking compound</th>
<th>Spiking method</th>
</tr>
</thead>
<tbody>
<tr>
<td>odour test</td>
<td>coffee filter paper</td>
<td>Hexanal</td>
</tr>
<tr>
<td>off-flavour test</td>
<td>coconut fat</td>
<td>Benzaldehyde</td>
</tr>
<tr>
<td>off-flavour test</td>
<td>icing sugar</td>
<td>Benzaldehyde</td>
</tr>
</tbody>
</table>
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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

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**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
- Before assessment of icing sugar add mineral water and stir
- Close and remove spiked icing sugar test portions

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

Batch 1
Batch 2
Batch 3

Are samples similar?
Are samples similar?

Homogeneity within batches
Homogeneity between batches

CHEMICAL ANALYSIS
SENSORY ANALYSIS

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Fresh samples

Time 0  Time 1  Time 2  Time 3

Are samples similar?

Stability of samples

If samples are stable:
Possibility of being prepared in one lab and send to others
More practical: no need to assess immediately

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### CALIBRATION SAMPLES

<table>
<thead>
<tr>
<th></th>
<th>HOMOGENEITY OF SAMPLES WITHIN BATCHES</th>
<th>HOMOGENEITY OF SAMPLES BETWEEN BATCHES</th>
<th>STABILITY</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>FATTY FOOD</strong></td>
<td>Very homogeneous</td>
<td>Very homogeneous</td>
<td>Very stable during 4 weeks after preparation</td>
</tr>
<tr>
<td><strong>DRY FOOD</strong></td>
<td>Homogeneous</td>
<td>Homogeneous</td>
<td>Should be assessed within 2 hours after preparation</td>
</tr>
<tr>
<td><strong>ODOUR</strong></td>
<td>Homogeneous</td>
<td>Homogeneous</td>
<td>Should be assessed immediately after preparation</td>
</tr>
</tbody>
</table>
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.

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

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

DIN scale has been used

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FINDING CONCENTRATIONS FOR CALIBRATION SAMPLES

Detection threshold oversized to the double app.

Identification threshold oversized to the double app.

DIN scale has been used

<table>
<thead>
<tr>
<th>Score</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>no perceptible off...</td>
</tr>
<tr>
<td>1</td>
<td>Off...just perceptible</td>
</tr>
<tr>
<td>2</td>
<td>Weak off...</td>
</tr>
<tr>
<td>3</td>
<td>Clear off...</td>
</tr>
<tr>
<td>4</td>
<td>strong off...</td>
</tr>
</tbody>
</table>

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

- odour
  - Lab 1

Validation of samples by another lab

- odour
  - Lab 4

Validation of samples by many labs

- Inter-laboratory Odour trial
- Inter-laboratory dry food trial
- Inter-laboratory fatty food trial

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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 1\textsuperscript{st} lab and 2\textsuperscript{nd}.

- 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, ...

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

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

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

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

VTT Biotecnology, Food Industry R&D
Finland
panel calibration

UNEW University of Newcastle Upon Tyne
United Kingdom
Statistics

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)
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 !!!