

Food Analysis and Quality Control

A Practical Manual



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6.2 FOOD ANALYSIS AND QUALITY CONTROL

LIST OF PRACTICALS

1. Proximate analysis of marketed food products
 - 1.1. Moisture
 - 1.2. Ash
 - 1.3. Crude Fat
 - 1.4. Crude Protein
 - 1.5. Crude Fibre
 - 1.6. Carbohydrates
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 - 2.4. spices,
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 - 2.6. oils,
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Experiment- 1- Moisture Content- Lab Oven Method

Objective:

To find out the moisture content from a given food sample by lab oven method.

Theory:

Upon heating water evaporates and loss of weight is equal to the moisture content of material.

Materials:

1. Enough food sample to supply three 20 gram samples.
2. Oven (almost any oven will do, provided the temperature can be set reliably at 130°C for an extended period of time).
3. Analytical Balance.
4. Tray (to place the paddy grain samples on before drying in the oven).
5. Simple plastic containers or small paper bags to hold grain.
6. Pen/pencil, paper and calculator to compute the average of three samples and record the moisture content.

Procedure:

1. Set the oven temperature to 130°C.
2. From your paddy grain supply, use your scale to measure three 20-gram paddy grain samples.
3. Place the three samples inside the oven and leave for 16-24 hours.
4. Measure the final weight of each sample after the 16 to 24 hours.
5. Compute the moisture content for each sample using the equation.
6. You now have three separate moisture content results. Compute the average of these results by adding them together and dividing by 3.

Calculation:

$$MC = \frac{\text{Initial Weight} - \text{Final Weight}}{\text{Initial Weight}} \times 100\%$$

Moisture Content (%) = $\{(W2 - W1) / (W1 - W)\} \times 100$

Where,

W = Weight of empty moisture dish

W1 = Weight of empty moisture dish + Sample

W2 = Weight of empty moisture dish + Dried Sample

Observations:

Sample Name	Wheat Flour		
Sample Number	Weight of Empty Moisture Dish (W) (in gms.)	Weight of Moisture Dish+Sample (W1) (in gms.)	Weight of Moisture Dish+Dried Sample (W2) (in gms.)

Weight of Sample (W1-W) (in gms.)	Weight of Moisture (W2-W1) (in gms.)	Moisture %	Average Moisture %	% RSD

Conclusions:

1. Did each of your samples contain approximately the same amount of moisture content?
2. What was the average moisture of all three samples?
3. Based on what you know about correct moisture content for milling (14%), is the paddy rice you sampled ready for milling? Why or why not?

Sample Name	Wheat Flour		
Sample Number	Weight of Empty Moisture Dish (W) (in gms.)	Weight of Moisture Dish+Sample (W1) (in gms.)	Weight of Moisture Dish+Dried Sample (W2) (in gms.)
A	34.5679	39.6384	39.5173
B	34.3369	39.7865	39.6652
C	34.1256	39.5596	39.4396

Weight of Sample (W1-W) (in gms.)	Weight of Moisture (W2-W1) (in gms.)	Moisture %	Average Moisture %	% RSD
5.0705	0.1211	2.39	2.28	4.327
5.4496	0.1213	2.23		
5.4340	0.1200	2.21		

Space for Observations and Calculations

Experiment- 2- Moisture Content- Using Moisture Meter

Objective:

To find out the moisture content from a given sample by using moisture meter.

Materials:

1. Moisture meter and instructions for use
2. Paddy rice – enough to provide three samples for the moisture meter (approximately one handful)
3. Simple plastic containers or small paper bags to hold grain
4. Pen/pencil, paper and calculator to compute the average of three samples and record the moisture content

Procedure:

1. Read the operator’s instructions.
2. Turn the moisture meter on.
3. Ensure the machine is set for paddy rice.
4. Fill the tray or bowl of the moisture tester with a sample of the paddy rice to be tested
5. Turn or press the knob until the moisture reading is displayed.
6. Test at least three samples and calculate the average of the three readings.

Observations & Calculations:

Sample	
Volume	
Thickness	
Sample No.	Moisture %
1	
2	
3	
Average Moisture Content	
% RSD	

Conclusions:

1. Did each of your samples contain approximately the same amount of moisture content?
2. What was the average moisture content of all three samples?
3. Is the paddy rice you sampled ready for milling? Why or why not?

Sample	Paddy
Volume	C
Thickness	3.25
Sample No.	Moisture %
1	10.30
2	10.40
3	10.80
Average Moisture Content	10.50
% RSD	2.52

Space for Observations and Calculations

Experiment- 3- Ash - Total

Objective:

To find out the ash in the given food sample.

Theory:

Organic matter is burnt off at as low temperature as possible. Heating is done in stages, first to char the product thoroughly and finally to ash at 550° C in a muffle furnace. The inorganic matter left after burning organic matter is cooled and weighed.

Apparatus:

1. Crucible
2. Heating Plate
3. Muffle Furnace
4. Dessicator
5. Analytical Balance

Procedure:

1. Place the crucibles in muffle furnace to heat at 550° C for 15 minutes.
2. Remove the crucibles, cool in a dessicator for one hour and weigh the crucible (W).
3. Weigh 2 g of sample in the crucible (W1).
4. Keep the sample on a hot plate till smoking ceases and sample become thoroughly charred.
5. Place the crucibles inside the muffle furnace and heat to 550° C for 5 to 6 hours.
6. Let the furnace cool and take out crucibles containing ash, clean and white in appearance.
7. If traces of carbon are still evident, cool the crucible, add 1 – 2 ml of water and stir with a glass rod to break up the ash. Dry on steam bath and place in muffle furnace and again heat at 550° C.
8. Cool the crucible in a dessicator and reweigh (W2) the crucible containing ash.

Calculation:

$$\text{Ash \%} = \frac{(W_2 - W)}{(W_1 - W)} \times 100$$

Where,

W = Weight of empty crucible

W1 = Weight of empty crucible + Sample

W2 = Weight of empty crucible + Ashed Sample

Observations:

Sample Name	_____		
Sample Number	Weight of Empty Silica Crucible (W) (in gms.)	Weight of Silica Crucible+Sample (W1) (in gms.)	Weight of Silica Crucible+Ashed Sample (W2) (in gms.)
A			
B			

C				
Weight of Sample (W1-W) (in gms.)	Weight of Ash (W2-W) (in gms.)	Ash %	Average Ash %	% RSD

Sample Name	Wheat Flour		
Sample Number	Weight of Empty Silica Crucible (W) (in gms.)	Weight of Silica Crucible+Sample (W1) (in gms.)	Weight of Silica Crucible+Ashed Sample (W2) (in gms.)
A	10.2569	12.2698	10.2762
B	10.2444	12.3009	10.2642
C	10.3597	12.4001	10.3801

Weight of Sample (W1-W) (in gms.)	Weight of Ash (W2-W) (in gms.)	Ash %	Average Ash %	% RSD
2.0129	0.0193	0.96	0.97	2.381
2.0565	0.0198	0.96		
2.0404	0.0204	1.00		

Space for Observations and Calculations

Experiment- 4- Ash- Acid Insoluble

Objective:

To find out the acid insoluble ash from a given food sample.

Reagents:

Dil HCL approximately 0.5 N prepared from conc.HCl

Procedure:

To the ash contained in a dish, add 25 ml of dil, HCl. Cover with a watch glass and heat on a water bath for 10 minutes. Allow to cool and filter the contents of the dish through a Watman filter paper No.42 or its equivalent. Wash the filter paper with water until the washing are free from the acid and return it to the dish. Keep it in an electric oven maintained at $135 \pm 2^{\circ}\text{C}$ for about 3 hrs. Ignite in a muffle furnace at $550-600^{\circ}\text{C}$ for 1hr.

Cool the dish in a dessicator and weigh. Repeat the process of igniting in muffle furnace, cooling and weighing at half hours interval until the difference in mass between 2 successive weighings is less than 1 mg. Note: the lowest mass.

Calculation

% Ash Insoluble ash (on dry basis)

$$\frac{100(W_2 - W)}{(W_1 - W)}$$

% by mass =

Where

W_2 - Mass in g of dish + acid insoluble

W - Mass in g of empty dish

W_1 - Mass in g of dish with the dried material

Observation and Result

Acid insoluble ash % of a given sample

$$\frac{100(37.7816 - 37.7786)}{(45.7786 - 37.7786)} \\ = \frac{100 \times 0.0030}{8} = 0.037\%$$

Where

W_2 = 37.7816 g

W_1 = 45.7786 g

W = 37.7786 g

Sample Name	Wheat Flour		
Sample Number	Weight of Empty Silica Crucible (W) (in gms.)	Weight of Silica Crucible+Sample (W1) (in gms.)	Weight of Silica Crucible+Acid Insoluble Ashed Sample (W2) (in gms.)
A	10.2569	12.2698	10.2641
B	10.2444	12.3009	10.2521
C	10.3597	12.4001	10.3671

Weight of Sample (W1-W) (in gms.)	Weight of Acid Insoluble Ash (W2-W) (in gms.)	Acid Insoluble Ash %	Average Acid Insoluble Ash %	% RSD
2.0129	0.0072	0.36	0.36	1.604
2.0565	0.0077	0.37		
2.0404	0.0074	0.36		

Space for Observations and Calculations

Experiment- 5- Crude Protein- Kjeldahl Method

Objective:

To find out the amount of crude protein in a given food sample.

Introduction

The protein content of foods can be determined by numerous methods. The Kjeldahl, nitrogen combustion (Dumas) and infrared spectroscopy methods for protein analysis are based on nitrogen determination. The methods are from the Official Methods of Analysis of AOAC International (1), and are used commonly in research laboratories working on proteins.

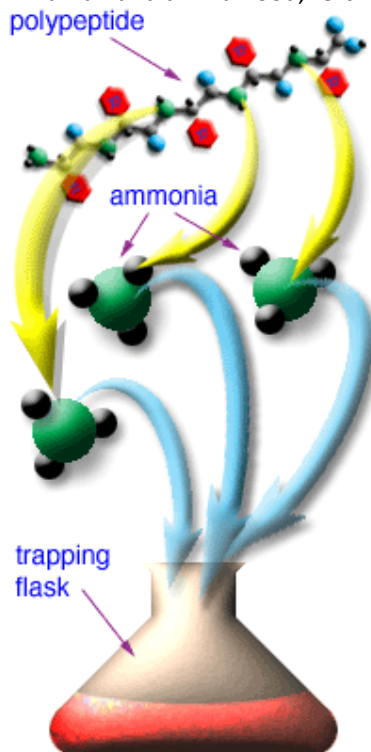
Theory

Nitrogen is one of the five major elements found in organic materials such as protein. This fact was recognized by a Danish chemist, Johan Kjeldahl, who used it as a method of determining the amount of protein in samples taken from a wide variety of organisms. In 1883 Kjeldahl presented to the Danish Chemical Society a method (much revised since his day) for determining the amount of nitrogen in mixtures of substances containing ammonium salts, nitrate, or organic nitrogen compounds.

The central basis used in this procedure is the oxidation of the organic compound using strong sulfuric acid. As the organic material is oxidized the carbon it contains is converted to carbon dioxide and the hydrogen is converted into water.

The **nitrogen**, from the amine groups found in the peptide bonds of the polypeptide chains, is converted to ammonium ion, which dissolves in the oxidizing solution, and can later be converted to ammonia gas.

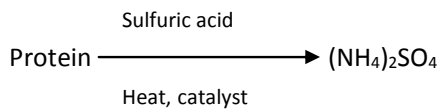
The Kjeldahl method of nitrogen analysis is the worldwide standard for calculating the protein content in a wide variety of materials ranging from human and animal food, fertilizer, waste water and fossil fuels.



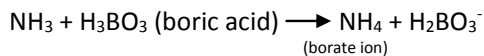
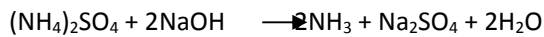
Principle

The Kjeldahl procedure can be basically divided into three parts: (1) digestion, (2) distillation, (3) titration. In the digestion step, organic nitrogen is converted to an ammonium in the presence of a catalyst at approximately 370°C. In this experiment, the sample is digested in H₂SO₄, using Copper-based catalyst,

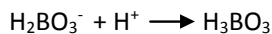
converting N to NH_3 which is distilled and titrated.



In the distillation step the digested sample is made alkaline with NaOH and the nitrogen is distilled off as NH_3 . This NH_3 is trapped in a boric acid solution.



The amount of ammonia nitrogen in this solution is quantified by titration with a standard HCl solution. A reagent blank is carried through the analysis and the volume of HCl titrant required for this blank is subtracted from each determination.

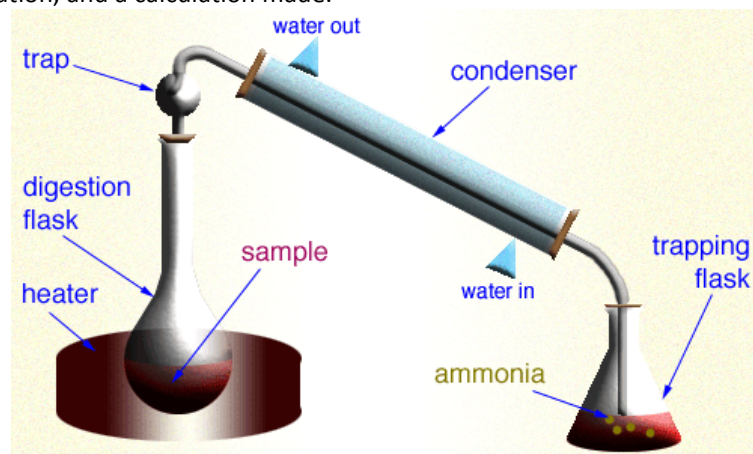


This analysis determines total nitrogen and not usable nitrogen and this is the reason it is called a crude protein analysis.

A three step procedure

The Kjeldahl method consists of three steps, which have to be carefully carried out in sequence:

1. the sample is first digested in strong sulfuric acid in the presence of a catalyst, which helps in the conversion of the amine nitrogen to ammonium ions,
2. the ammonium ions are then converted into ammonia gas, heated and distilled. The ammonia gas is led into a trapping solution where it dissolves and becomes an ammonium ion once again,
3. finally the amount of the ammonia that has been trapped is determined by titration with a standard solution, and a calculation made.

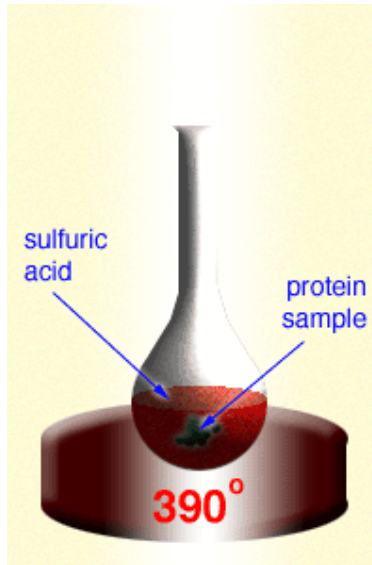


Step One: Digestion of the Sample

This is the most time-consuming step in the analysis. The purpose of this step is to break down the bonds that hold the polypeptides together, and convert them to simpler chemicals such as water, carbon dioxide and, of course, ammonia.

Such reactions can be considerably speeded up by the presence of a catalyst and by a neutral substance, such as potassium sulfate (K_2SO_4), which raises the boiling point of the digesting acid and thus the temperature of the reaction.

Catalysts are also used to help in the digestion process; many different one have been tried including selenium, mercury, copper, or ions of mercury or copper.



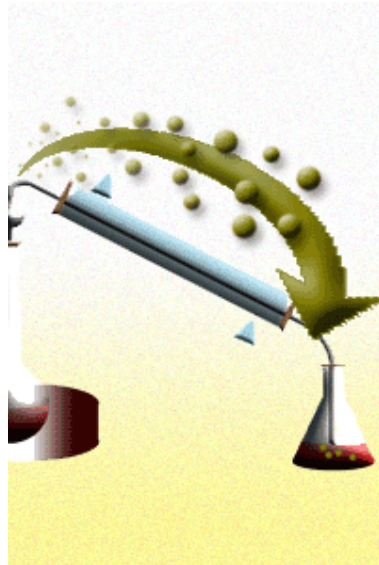
Digestion is accomplished by:

1. Weighing out approximately 1 gm of the sample containing protein, making a note of the weight, and placing the sample into a digestion flask, along with 12-15 ml of concentrated sulfuric acid (H_2SO_4).
2. Adding seven grams of potassium sulfate and a catalyst, usually copper.
3. Bringing the digestion tube/flask and mixture to a "rolling boil" (about $370^{\circ}C$ to $400^{\circ}C$) using a heating a block.
4. Heating the mixture in the tube/flask until white fumes can be seen, and then continuing the heating for about 60-90 mins.
5. Cooling the tube/flask and cautiously adding 250 mls of water.

Step Two: Distillation

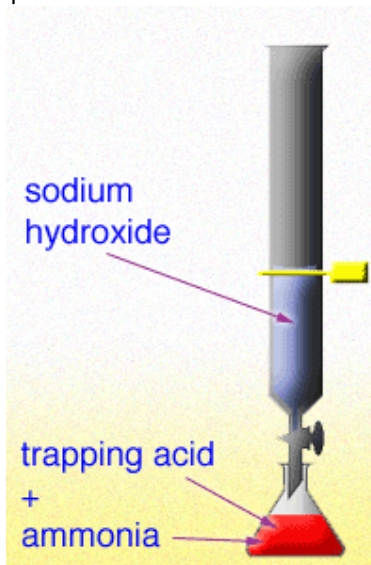
The purpose of the next step, distillation, is to separate the ammonia (that is, the nitrogen) from the digestion mixture. This is done by,

1. raising the pH of the mixture using sodium hydroxide (45% NaOH solution). This has the effect of changing the ammonium (NH_4^+) ions (which are dissolved in the liquid) to ammonia (NH_3), which is a gas.
2. separating the nitrogen away from the digestion mixture by distilling the ammonia (converting it to a volatile gas, by raising the temperature to boiling point) and then trapping the distilled vapors in a special trapping solution of about 15 ml HCl (hydrochloric acid) in 70 ml of water.
3. removing the trapping flask and rinsing the condenser with water so as to make sure that all the ammonia has been dissolved.



Step Three: Titration

As the ammonia dissolves in the acid trapping solution, it neutralizes some of the HCl it finds there. What acid is left can then be "back titrated", that is titrated with a standard, known solution of base (usually NaOH). In this way the amount of ammonia distilled off from the digestive solution can be calculated, and hence the amount of nitrogen in the protein determined.



The quantities of acid, and hence ammonia are determined by,

1. adding an indicator dye to the acid/ammonia trapping solution. This dye should turn a strong color, indicating that a significant amount of the original trapping acid is still present.
2. putting a standard solution of NaOH (sodium hydroxide) into the buret (a long tube with a tap at the end), and slowly, slowly adding small amounts of the sodium hydroxide solution to the acid solution with the dye.
3. watching for the point at which the dye turns orange, indicating that the "endpoint" has been reached and that now all the acid has been neutralized by the base.
4. recording the volume of the neutralizing base (sodium hydroxide solution) that was necessary to reach the endpoint.
5. performing a calculation to find the amount of ammonia, and thus nitrogen, that came from the original sample.

Calculations

One mole of ammonia coming from the digestion mixture (and hence from the original protein) will neutralize exactly one mole of the acid in the trapping flask.

The first calculation, therefore, is to find the number of moles of ammonia that have been produced and then trapped from your sample(s).

This is done by,

- calculating the number of moles of acid in the trapping flask originally (before any ammonia was trapped) by multiplying the molarity of the acid solution by the volume of the trapping solution

moles of acid = molarity of acid x volume used in flask

(moles_A = M x V)

- calculating the number of moles of base (NaOH) that were added from the buret to neutralize the remaining acid (that NOT neutralized by the ammonia).

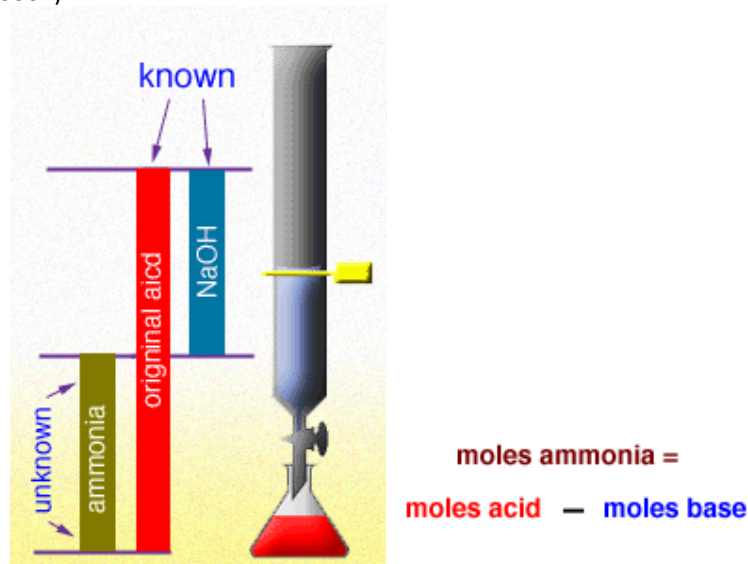
moles of base = molarity of base x volume added from buret

(moles_B = M x V)

- subtracting the "moles of base" added from the "moles of acid" present at the beginning, to get,
- the number of "moles of ammonia" coming from the protein,
- the number of "moles of ammonia" is the same as the "moles of nitrogen",
- so ... to calculate the number of grams of nitrogen in the original sample of protein, multiply the "moles of nitrogen" by the atomic mass of nitrogen (mass of atoms of nitrogen),

gms nitrogen = moles nitrogen x atomic mass

(g_N = moles_N x 14.0067)



percent Nitrogen

The percentage of nitrogen found in the original sample can now be calculated by:

%nitrogen = (gms nitrogen / gms sample) x 100

%N = (g_N / g_S) x 100

It is also possible to calculate the amount of crude protein in the sample. Although there are differences between different samples, the amount of "crude protein" (**CP**) can be found by multiplying the percent Nitrogen by a factor (usually 6.25).

CP = %N x 6.25

Apparatus/Instrument/Chemicals/Samples

- Sulfuric acid 98% min.
- Catalyst tablets to be used: Kjeltabs CX
- Caustic soda 32%
- Boric acid solution 2%

- Indicator Solution M5 (Merck) or similar
- Standard acid 0.1N or $c = 0.1 \text{ mol/l}$, alternatively sulfuric acid 0.1N or $c = 0.05 \text{ mol/l}$
- Mechanical comminuting instrument
- Analytical balance (0.001 g)
- Kjeldahl digestion block Kjeldatherm, Turbotherm, flask heater for Kjeldahl flask with wide neck opening
- Vapodest distillation System
- Burette, 50 ml nominal capacity, with a scale on 0.05 ml or titration system (not with the Vap 50) or pH meter with combined electrode

Procedure

1. Sample Preparation

1. Weigh accurately 2.00g comminuted sample as a start on a piece of a filter paper.
2. Store the sample air tight so that any changes or decay of the composition is avoided. Prior to the analysis the sample should be at room temperature. The examination of the thus prepared sample has to be done within the following 24 h.

2. Digestion chemicals

1. The chemicals are added. Sulfuric acid is used to wash down any sample residue, which might remain at the glass walls.

Chemicals	
Sulfuric acid	20 ml
Kjeltabs	2
Indicator solution	
M5	
Standard acid 0.1N or $c=0.1\text{mol/L}$; alternatively sulfuric acid 0.1N or $c=0.05\text{mol/L}$	

3. Digestion with Kjeldatherm

- When working with a **Kjeldatherm-System** with 250 ml Kjeldatherm-digestion tubes, the following digestion parameters are recommended:

Time in min	Temperature in ° C	Comments
40	400	Digestion tubes are put into the preheated block and time it takes for the sample to become translucent
30	400	Dehydrate the sample

- Foaming during the digestion has to be expected, however, the foaming should not go higher than 2/3 of the glass.
- If excessive reactions should occur, take out the insert rack.
- During the digestion black particles remaining at the glass wall are washed back with condensing sulfuric acid.
- The sample glass has to be translucent after the digestion in order to obtain good results.

4. Digestion with Turbotherm

- When working with a Turbotherm-System with 250 ml Kjeldatherm-digestion tubes, the following program parameter are recommended:

Time in min	Power in %	Comment
10 to 15	100	Heating up of the system to bring digestion solution to boiling
60	70 to 80	Digestion solution is turning translucent after ca 20 to 30 min

5. Digestion with Flask heater

- For Serial Flask Heater_ with 500 or 750 ml Kjeldahl flask with wide neck opening the following procedure is recommended:

Time in min	Power	Comments
20	3	Heating up till the digestion solution is boiling
50	1,5	After 20-30 min. the sample should turn translucent. Wash down remaining sample particles with condensing sulfuric acid into the flask.

6. Suction

- During the entire digestion period the scrubber should be on. About 1200 ml of a 15% caustic soda is recommended for the washing bottle; this amount is sufficient to neutralize digestion gases of about 60 digestions.
- The cooling off period after the lifting of the insert rack or the cooling off period after turning off the heating is about 30 minutes; during this time the scrubber should be working as well.

7. Distillation

After the digested sample has cooled off the water steam distillation is done according to the following program:

Program parameter	Vap 50
Water addition in s	9
NaOH addition in s	8
Reaction time in s	0
Distillation time in s	240
Steam output in %	100
Suction sample in s	25
Boric acid addition in s	6 s
Suction receiver in s	25
Titration	Auto
Calculation	Auto

8. Titration (is done automatically when using the Vap 50)

- 3 - 4 drops of an indicator mixture M 5 are added to the receiving solution and it is then titrated with 0.1 N titration acid till the color changes from green to grey/violet.
- If the determination of the endpoint is done with a pH-meter or a titrator, the addition of the indicator mixture is obsolete.

9. Blank Value

- For the determination of the blank value the analysis (digestion and distillation) is run just using the given chemicals.
- The consumption of those chemicals has then to be taken into account when the calculation is done.

10. Calculation

$$\% N = \frac{1.4007 * c * (V - V_b)}{\text{Sample weight (g)}}$$

c : Concentration of the standard-acid solution: Hydrochloric acid 0.1N or c = 0.1 mol/l

Alternative: sulfuric acid 0.1N or c = 0.05 mol/l

V: Consumption of the standard acid in ml (Sample)

V_b: Consumption of the standard acid in ml (Blank Sample)

% raw protein = % N * 6.25

Nitrogen to Protein Conversion Factors for Various Foods

Product	Factor
Egg or meat	6.25
Dairy products	6.38
Wheat	5.70
Other cereal grains or oilseeds	6.25
Almonds	5.18
Peanut and Brazil nuts	5.46
Other tree nuts and coconut	5.30
Soybean products	6.08

Protein by Kjeltec

Principle

All nitrogen in the sample is converted to NH₄⁺ by digestion with concentrated H₂SO₄ and H₂O₂ using inorganic salt catalysts at a high temperature. The solution is then made alkaline (with NaOH) and steam is passed through and the ammonia is distilled into a solution of boric acid that contains an indicator (methyl orange). Finally the distilled ammonia, now dissolved in the boric acid, is titrated against 0.1 M HCl.

The Kjeldahl method described above is the standard method for determining protein content. The Kjeltec is a semi-automated version of this method.

1 Digestion

Wear Gloves and Safety Glasses throughout this Procedure

1. Collect two Kjeldahl digestion tubes and label them with a permanent marker close to the top. Always carry them in a tube rack (share with your colleagues).
2. Accurately weigh out (to nearest mg) in duplicate approximately 0.1 g of dry sample on to a filter paper (W1).
3. If using a wet sample, pipette about 1 g (to nearest mg) directly into the bottom of a labelled digestion tube.
4. Fold up the paper containing the dry sample and carefully transfer the whole parcel to a labelled digestion tube.
5. Place a filter paper in a further digestion tube to act as a blank.
6. Add 2 catalyst tablets (copper sulphate + potassium sulphate) to each tube. Then add 10 cm³ concentrated H₂SO₄ and mix by gentle swirling. Take care. See the demonstrator before making these additions.
7. Now add 10 cm³ H₂O₂ in a fume cupboard and beware of frothing. Add the peroxide slowly at first.
8. Place the tubes in the digestion block and digest until clear (30 minutes to 2 hours). Check that there are no black carbon particles visible.

9. Allow the tubes to cool until barely warm but still liquid. Then add approximately 70 cm³ distilled water.

CARE - add the water slowly in the early stages and beware of spitting. If necessary the digestion tubes may be left at this stage by covering the top of the tubes with Parafilm.

2 Distillation and Titration

- In turn place the tubes into the distillation unit and twist to seal. If this is not done properly the tube contents may leak out and the results be useless.
- Close the door on the apparatus and start the cycle.
- The exact sequence will depend on which instrument you are using.
- The apparatus will automatically add alkali to the tube and then bubble steam to distil off the ammonia. This is collected in boric acid. The Foss Kjeltec machine used boric containing indicator but the Buchi machine measure pH. The titration is automatic. The volume of acid used is displayed on the front of the apparatus. With both instruments the results are saved and can be accessed later if necessary.
- When the titration is finished the tubes will be emptied automatically. Record the volume of acid used.
- Insert the next tube and continue.

Weight of food sample = W1
Volume of acid in sample titration = V1
Volume of acid in blank titration = BL
Concentration of acid, M = 0.1 M

See below for calculation of total and percentage values

Total Nitrogen and Protein in the food, measured in dry sample:

$$\text{Total Nitrogen} = \frac{(V1 - BL) \times 0.1 \times 0.0014 \times \text{Total Dry Matter of Food}}{W1}$$

where 0.1 is molar concentration of the HCl and 0.0014 is factor for equivalence of HCl to NH₃

To convert to protein multiply by the appropriate factor:

- x 6.25 for meat and general protein
- x 6.38 for milk protein
- x 5.70 for cereal protein.

NB These are not perfect and you must choose according to the major type of food in the sample. In most cases this will be the first one.

Percentage Nitrogen and Protein

In original, wet sample:

$$\text{Nitrogen, \%} = \frac{(V1 - BL) \times 0.1 \times 0.0014 \times 100 \times \% \text{Dry Matter}}{W1 \quad 100}$$

To convert to protein multiply by the appropriate factor:

x 6.25 for meat and general protein
x 6.38 for milk protein
x 5.70 for cereal protein.

NB These are not perfect and you must choose according to the major type of food in the sample. In most cases this will be the first one.

Space for Observations and Calculations

Experiment- 6- Crude Fat- Soxhlet Apparatus Method

Objective:

To find out the amount of crude fat in a given food sample.

Principle:

A **Soxhlet extractor** is a piece of laboratory apparatus invented in 1879 by Franz von Soxhlet. It is a procedure to remove lipids (fats) from food. A solvent is used to wash the solid using a reflux apparatus. The sample is dried and ground and placed in a tube above the extraction solvent. When heated, the solvent evaporates into a gas, then cools into a liquid in a condenser. It then leaks into the sample tube. This continues several hours until the lipid is removed from the sample. The solvent is evaporated off, and the amount of lipid is determined.

Apparatus

- *Soxhlet extraction apparatus* - A glass Soxhlet extraction apparatus of suitable size (100 mL) for containing the sample and a 250 mL collection flask is required for the conventional Soxhlet procedure. An automated extraction apparatus (Brinkmann Buchi B-810 or equivalent) with circulating oil bath and associated glassware is required for the automated Soxhlet procedure.
- *Alundum extraction thimbles* - Medium porosity (10 - 15 mm pore), sized to fit the Soxhlet extractor.
- *Analytical balance* - Sensitive to 0.1 mg.
- *Rotary evaporator with vacuum and water bath* - Rotary evaporator equipped with a "bump" trap, condenser, receiving vessel, and vacuum source sufficient to pull a vacuum of less than 150 torr.
- *Vacuum oven or drying oven* - Vacuum oven should be controllable to a temperature of $40 \pm 1^\circ\text{C}$ and vacuum of between 75 to 100 torr. If drying oven is used in place of the vacuum oven, the drying oven must be able to maintain $45 \pm 2^\circ\text{C}$

Reagents and Materials

- Ethyl alcohol, 95% in water (190 proof), USP grade.
- Boiling chips.
- Glass wool.
- Buchner funnel.
- Desiccator.

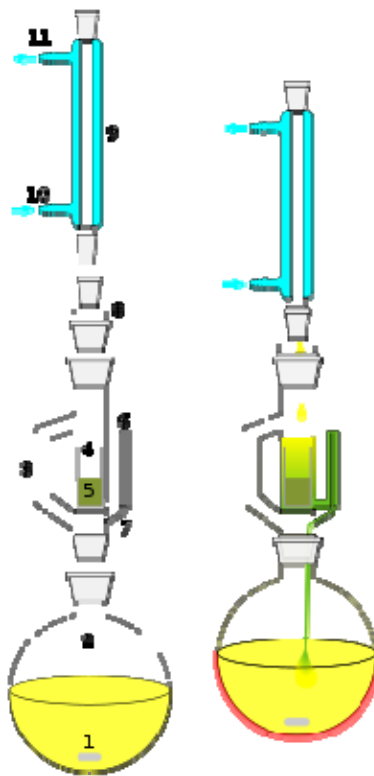
Procedure

1. Dry the Soxhlet extraction thimble at 105°C to constant weight. Remove, cool to room temperature in a desiccator, and weigh to the nearest 0.1 mg.
2. Carefully add the sample to the extraction thimble. Do not overfill the thimble, leave at least a 1 cm gap between the sample and the top of the thimble. Weigh the filled thimble to the nearest 0.1 mg. Place a plug of glass wool on top of the sample to prevent sample loss during the extraction.

Note: Samples for total solids determination (following Laboratory Analytical Procedure #001, Determination of Total Solids and Moisture in Biomass) must be weighed out at the same time as the samples for the extractives determination. If this determination is done at a later time, an error in the calculation of the amount of extractives will be introduced, since the moisture content of a biomass sample can change rapidly when exposed to air.

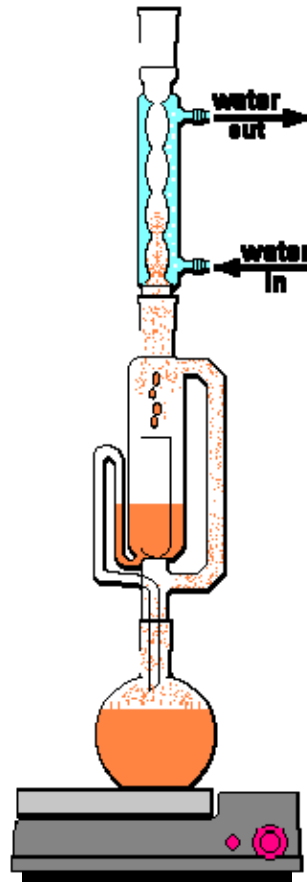
3. Place several boiling chips into a clean, dry receiving flask or beaker. Weigh the container, with chips, to the nearest 0.1 mg and record as the tare weight of the container.
4. For a conventional Soxhlet extraction (this procedure was reproduced from the Chemical Technologies Research Branch Procedure #001c, Determination of Extractives Content):
 - 4.1. Assemble the Soxhlet apparatus using at least 160 mL of 95% ethanol. Insert the thimble and heat at reflux for 24 hours. Periodically check the reflux rate and adjust the heating rate to give four to five solvent exchanges per hour in the Soxhlet thimble. Approximately 100-120 solvent exchanges are required during the 24 hour period.

- 4.2. When the extraction time is complete, remove the thimble and carefully transfer the sample to a Buchner funnel. Remove any residual solvent by vacuum filtration and wash the sample thoroughly with 95% ethanol, collecting all of the filtrate. Allow the biomass to air dry in the Buchner funnel while it is still attached to the vacuum system.
- 4.3. Combine the filtrate from the previous step and any solvent from the upper section of the Soxhlet apparatus with the solvent in the 250 mL flask. Place the flask on the rotary evaporator and remove the solvent under vacuum. Use a water bath temperature of $45 \pm 5^\circ\text{C}$ to heat the flask during evaporation.
- 4.4. After all of the visible solvent is removed by the rotary evaporator, place the flask in a vacuum oven ($75\text{-}100$ torr) at $40 \pm 1^\circ\text{C}$ for 24 ± 1 hour. Remove the flask at this time and allow to cool to room temperature in a desiccator. Weigh the flask and record this total weight to the nearest 0.1 mg.



A schematic representation of a Soxhlet extractor

- 1:** Stirrer bar **2:** Still pot (the still pot should not be overfilled and the volume of solvent in the still pot should be 3 to 4 times the volume of the soxhlet chamber) **3:** Distillation path **4:** Thimble **5:** Solid **6:** Siphon top **7:** Siphon exit **8:** Expansion adapter **9:** Condensor **10:** Cooling water in **11:** Cooling water out




Mechanism of Soxhlet extractor

Calculations

1. Calculate the oven dry weight of the sample, using the average total solids content.

$$\% \text{ Extractives} = \frac{\text{Weight container plus residue} - \text{Tare wt. container}}{\text{ODW}} \times 100$$

2. Calculate the amount of extractives in the sample, on a percent dry weight basis.

$$\text{ODW} = \frac{(\text{Weight, thimble plus sample} - \text{Weight, thimble}) \times \% \text{ Total solids}}{100}$$

Space for Observations and Calculations

Experiment- 7- Total Carbohydrates

Objective

To find out the amount of total carbohydrates in a given food sample.

Theory

Carbohydrates are the important components of storage and structural materials in the plants. They exist as free sugars and polysaccharides. The basic units of carbohydrates are the monosaccharides which cannot be split by hydrolysis into more simpler sugars. The carbohydrate content can be measured by hydrolyzing the polysaccharides into simple sugars by acid hydrolysis and estimating the resultant monosaccharides.

Principle

Carbohydrates are first hydrolysed into simple sugars using dilute hydrochloric acid. In hot acidic medium glucose is dehydrated to hydroxymethyl furfural. This compound forms with anthrone a green colored product with an absorption maximum at 630nm.

Materials

- 2.5 N-HCl
- *Anthrone Reagent*: Dissolve 200mg anthrone in 100mL of ice cold 95% H₂SO₄. Prepare fresh before use.
- *Standard Glucose*: Stock – Dissolve 100mg in 100mL water. Working standard – 10mL of stock diluted to 100mL with distilled water. Store refrigerated after adding a few drops of toluene.

Procedure

1. Weigh 100mg of the sample into a boiling tube.
2. Hydrolyse by keeping it in boiling water bath for 3 hours with 5mL of 2.5 N-HCl and cool to room temperature.
3. Neutralise it with solid sodium carbonate until the effervescence ceases.
4. Make up the volume to 100mL and centrifuge.
5. Collect the supernatant and take 0.5 and 1mL aliquots for analysis.
6. Prepare the standards by taking 0, 0.2, 0.4, 0.6, 0.8 and 1mL of the working standard. '0' serves as blank.
7. Make up the volume to 1mL in all the tubes including the sample tubes by adding distilled water.
8. Then add 4mL of anthrone reagent.
9. Heat for eight minutes in a boiling water bath.
10. Cool rapidly and read the green to dark green color at 630nm.
11. Draw a standard graph by plotting concentration of the standard on the X-axis versus absorbance on the Y-axis.
12. From the graph calculate the amount of carbohydrate present in the sample tube.

Calculation

$$\text{Amount of carbohydrate present in 100mg of the sample} = \frac{\text{mg of glucose}}{\text{Volume of test sample}} \times 100$$

Note

Cool the contents of all the tubes on ice before adding ice-cold anthrone reagent.

Space for Observations and Calculations

Experiment- 8- Crude Fiber

Objective

To find out the amount of crude fiber in a given food sample.

Theory

Crude fiber consists largely of cellulose and lignin (97%) plus some mineral matter. It represents only 60% to 80% of the cellulose and 4% to 6% of the lignin. The crude fiber content is commonly used as a measure of the nutritive value of poultry and livestock feeds and also in the analysis of various foods and food products to detect adulteration, quality and quantity.

Principle

During the acid and subsequent alkali treatment, oxidative hydrolytic degradation of the native cellulose and considerable degradation of lignin occur. The residue obtained after final filtration is weighed, incinerated, cooled and weighed again. The loss in weight gives the crude fiber content.

Materials

1. Sulphuric acid solution (0.255 ±0.005N) : 1.25g concentrated sulphuric acid diluted to 100mL (concentration must be checked by titration)
2. Sodium hydroxide solution (0.313 ±0.005N) : 1.25g sodium hydroxide in 100mL distilled water (concentration must be checked by titration with standard acid)

Procedure

1. Extract 2g of ground material with ether or petroleum ether to remove fat (Initial boiling temperature 35 -38°C and final temperature 52°C). if fat content is below 1%, extraction may be omitted.
2. After extraction with ether boil 2g of dried material with 200mL of sulphuric acid for 30min with bumping chips.
3. Filter through muslin and wash with boiling water until washing are no longer acidic.
4. Boil with 200mL of sodium hydroxide solution for 30min.
5. Filter through muslin cloth again and wash with 25mL of boiling 1.25% H₂SO₄, three 50mL portions of water and 25mL alcohol.
6. Remove the residue and transfer to ashing dish (preweighed dish W₁).
7. Dry the residue for 2h at 130 ±2°C. Cool the dish in a desiccator and weigh (W₂).
8. Ignite for 30min at 600 ±15°C.
9. Cool in a desiccator and reweigh (W₃).

Calculation

$$\% \text{ crude fiber in ground sample} = \frac{\text{Loss in weight on ignition } (W_2 - W_1) - (W_3 - W_1)}{\text{Weight of the sample}} \times 100$$

Determination of Crude Fibre Using Fibrebag (Gerhardt Method)

Objective

To determine fibre content in whole bran bread and spinach.

Apparatus/Instrument/Chemicals/Samples

- Hotplate
- 1L Beaker glass without spout and glass condenser with riffle
- FibreBag – Carousel for 6 FibreBags with bayonet coupling
- Bag with 100 FibreBags
- Glass spacer
- Accessory: crucible for incineration
- Drying Chamber, Temp. 105°C
- Muffle furnace, Temp. 600°C
- Water heater
- Desiccator
- Timer or alarm clock
- Analytical balance
- Fume cabinet
- Sulfuric acid c (H_2SO_4) = 0.13 mol/l
- Potassium hydroxide solution c (KOH) = 0.23 mol/l
- Petroleum ether, boiling range 40 to 60
- Water distilled or demineralized
- Sodium Hydroxide c (NaOH) = 0.313 mol/l
- Hydrochloride acid c (HCl) = 0.1 mol/l

System Description:

1. Preparation

- a. FibreBag is dried at $105 \pm 1^\circ\text{C}$ for 1h in the drying chamber. The weight of the FibreBag is the **value A** for the balance protocol. When storing the FibreBags in a desiccator they only have to be dried once and then, can be weighted directly.
- b. Put 1g sample into the FibreBag and weight with 1 mg preciseness; this gives **value B** for the weighing protocol. A determination of the blank value should be done parallel to the regular analysis. The value should be < 1 mg/FibreBag. The dry matter of the sample should be determined separately and is important for the calculation of the content (result related to the dry matter).
- c. Put the glass spacer into the FibreBag and insert the bag in carousel.
- d. De-fatting of the sample, especially important for samples with a fat content of $> 5\%$: Immerse the carousel three times in a row into 100ml 40/60 petroleum ether. By turning it as well as moving it up and down the sample is defatted. This facilitates the washing and filtration, which will follow. Furthermore, no crude fibre content is lost. Throw away the first petroleum ether fraction but the following can be reused. After a short drying process in the fume cupboard (about 2 minutes), immerse the carousel in the first washing solution.

2. Washing - Phase I (Instrument method)

- a. Measure 360 ml $\text{H}_2\text{SO}_4 = 0.13\text{mol/l}$ into the first beaker.
- b. Attach handling tool to the carousel and lower it gently into the beaker.
- c. Mix it by rotating the carousel for about 1 minute so that the sample is entirely soaked and make sure that the FibreBag is filled with washing solution.
- d. Place the beaker on the hotplate, which has been preheated for about 5 minutes.
- e. Bring it to a boil by setting it full (takes about 3-5 minutes); reduce the hotplate setting when it starts to boil (at about 90°C).
- f. Adjust the hotplate setting to obtain a very gentle simmering for about 30 minutes. During this boiling stage the sample should float freely in the FibreBag.

- g. This can be helped by gently rotating the carousel with the handling tool or by softly swirling the beaker.
- h. After exactly 30 minutes from the boiling point remove the beaker from the hotplate. Also, take the carousel out of the beaker using the handling tool thus draining the acid from the FibreBag.
- i. Washing out of the acid:
 1. Discard the acid and solubles within the beaker.
 2. Rinse the carousel several times with hot water.

3. Washing – Phase II (Instrument Method)

- a. Measure 360 ml potassium hydroxide solution c (KOH) = 0.23 mol/l into the beaker.
- b. Attach handling tool to the carousel and lower it gently into the beaker of solution. Mix it by rotating the carousel for about 1 minute so that the FibreBag is filled completely with the solution.
- c. Place again the extraction beaker on a preheated hotplate.
- d. Again, bring it to a boil by setting it full (takes about 3-5 minutes); reduce the hotplate setting when it starts to boil.
- e. Adjust the hotplate setting to obtain a very gentle simmering for about 30 minutes.
- f. During this boiling stage the sample should float freely in the FibreBag. This can be helped by gently rotating the carousel with the handling tool or by softly swirling the beaker.
- g. After exactly 30 minutes from the boiling point remove the beaker from the hotplate.
- h. Also, take out the carousel from the beaker using the handling tool thus draining the solution from the FibreBags.
- i. Washing out the alkalis:
 - Discard the alkali and soluble within the beaker.
 - Rinse the carousel several times with hot water. (Check by using pH-indicator paper)
 - Dry the FibreBags by wiping them with a paper towel or by rotating the carousel in an empty beaker,

4. Drying of the FibreBags

- a. Take out the drained FibreBags of the carousel and put into a crucible, which has to be preashed at 600°C and weighted (value F for the balance protocol). Place it into a drying chamber overnight at 105°C. FibreBag after digestion and crucible is **value C**.

5. Incineration of Samples

- a. Incinerate the FibreBags at 600°C for at least 4 hours or overnight. The resulting vapours are not hazardous!
- b. After the incineration, weight the crucible, which was left, to cool off in the desiccator and obtain value δ for the weighing protocol. – **value D**.

6. Calculation:

The crude fibre is the non-solubles which remain after digestion with acids and alkalis minus the content of ash and is calculated as follows:

$$\% \text{ Crude Fibre} = \frac{((C - A) - (D - E)) \times 100}{B}$$

$$\text{Blank Value E} = D - F$$

Meaning:

A = Mass FibreBag in g

B = Mass Sample weight in g (has to be adjusted according to dry content)

C = Mass Crucible and dried FibreBag after digestion in g

D = Mass Crucible and Ash in g

E = Blank Value of the empty FibreBag in g

F = Mass Crucible in g

Results/ Weighing protocol: Blank value E in g:

FibreBag Number	Sample-number	A in g	F in g	B in g	C in g	D in g *	Crude Fibre (%)
1							
1							
3							
4							
4							
5							
6							

* minus blank value E in g

Comment: A small beaker can also be used instead of a crucible.

7. Validation

The development of the FibreBags analysis has shown that it is of vital importance that certain parameters have to be strictly observed. Thus, it is recommended to strictly observing the times given for cooling, heating and boiling.

This also goes for:

- Amount of Sample
- Concentration of Acids and Alkalis
- Times for drying and incineration and temperatures
- No other solvents than Petroleum ether

References

1. Nielsen, S.S. 1994. Introduction to the Chemical Analysis of Foods. Boston: Jones and Bartlett Publishers, Inc.
2. Gerhardt manual training.

Space for Observations and Calculations

Experiment- 9- Cut out test for Canned Fishery Products

Objective

To perform the cut out test for the given sample of canned food product.

Theory

Cut out test is done to evaluate the general quality of a canned food. In this test, the condition of the food contents, the external and internal conditions of the Can and other characteristics of the product are examined by certain organoleptic, physical and chemical tests.

Materials and Equipments

1. Canned food : 4-6 nos. Cans
2. Tone tester
3. Physical balance
4. Vacuum gauge
5. Can opener
6. Brix refractometer
7. Scale
8. pH paper near neutral ranges

Procedure

1. If the Cans are labeled, note the particular of the label.
2. Record the embossed code mark on the lid.
3. Observe the external condition of the cans such as rusting, dents, physical damage, seam defects etc.
4. Test the tone and get an idea about the fill and vacuum.
5. Determine the gross weight.
6. Measure the vacuum.
7. Cut the lid almost completely, open the observe the food surface and inside the lid. Measure the head space.
8. Drain the contents for 5 min. collect the liquid in a measuring jar.
9. Note the volume, turbidity, colour, texture, flavour etc. Also look for foreign matter.
10. Observe the bottom and inside the Can, looking for settled curds.
11. Wash, dry and weigh the empty Can.

Evaluation Sheet for Cut-Out Test

Can No./Particulars	1	2	3	4	5
Product :					
Code :					
Manufacturer :					
Date of production :					
Date of Testing :					
Can size & Type :					

Can No./Particulars	1	2	3	4	5
Std. Net wt. / Solid wt. :					
Vacuum :					
Gross Weight :					
Solid + Can wt :					
Empty Can wt :					
Solid wt :					
Liquid wt :					
Net wt :					
Solid wt :					
Net wt :					
Pack wt :					
Colour :					
Tecture :					
Flavour :					
Appearance (Style) :					
No. of pieces :					
Salt/ Sugar degree :					
Turbidity :					
Acidity :					
PH :					
Size of pieces :					
Broken of flakes :					
Adhesion :					

Can No./Particulars	1	2	3	4	5
Curds :					
Remarks :					

Space for Observations and Calculations

Experiment- 10- Detection of adulterants in different food products

Objective

To test different given food samples for adulteration.

Procedure

Sl No.	Name of Food Article	Adulterant	Simple Method for detection of Common Adulterants	Remarks
A	Milk and Milk Products			
I	Milk	Water	i. The lactometer reading shall not ordinarily be less than 26. ii. The presence of water can be by putting a drop of milk on a polished slanting surface. The drop of pure milk either or flows lowly leaving a white trail behind it, whereas milk adulterated water will flow immediately without leaving a mark	Lactometer is marked in degrees ranging from 0 – 40. The test is not valid if skimmed milk or other thickening material is added.
	Milk	Starch	Add a few drops of tincture of Iodine or Iodine solution. Formation of blue colour indicates the of starch.	
	Milk	Removal of Fat	The lactometer reading will go above 26 while the milk apparently remains thick	
II	Khoa and its products	Starch	Boil a small quantity of sample with some water, cool and add -a few drops of Iodine solution. Formation of blue colour indicates the presence of starch	
III	Chhana or Paneer	Starch	Boil a small quantity of sample with some water, cool and add a few drops of Iodine solution. Formation of blue colour indicates the of starch.	

SI No.	Name of Food Article	Adulterant	Simple Method for detection of Common Adulterants	Remarks
IV	Ghee	Vanaspati or Margarine	Take about one tea spoon full of melted sample of Ghee with equal quantity of concentrated Hydrochloric acid in a stoppered test tube and add to it a pinch of sugar. Shake for one minute and let it for five minutes. Appearance of crimson colour in lower (acid) of Vanaspati or Margarine.	The test is specific for sesame oil which is compulsorily added to Vanaspati and Mrgarine. Some coal tar colours also give a positive test. If the test is positive i.e. red colour develops only by adding strong Hydrochloric acid (without adding crystals of sugar) then the sample is adulterated with coal tar dye. If the crimson or red colour develops after adding and shaking with sugar, then alone Vanaspati or Margarine is present
	Ghee	Mashed Potatoes, Sweet Potatoes and other starches.	The presence of mashed potatoes and sweet potatoes in a sample of Butter can easily be detected by adding a few drops of Iodine, which is brownish in colour turns to blue if mashed potatoes/sweet potatoes/other starches are present.	
V	Butter	Vanaspati or Margarine	Take about one teaspoon full of melted sample of Ghee with equal quantity of concentrated Hydrochloric acid in a stoppered test tube and add to it a pinch of sugar. Shake for one minute and let it for five minutes. Appearance of crimson colour in lower (acid) of Vanaspati or Margarine.	The test is specific for sesame oil which is compulsorily added to Vanaspati and Mrgarine. Some coal tar colours also give a positive test. If the test is positive i.e. red colour develops only by adding strong Hydrochloric acid (without adding crystals of sugar) then the sample is adulterated with coal tar dye. If the crimson or red colour develops after adding and shaking with sugar, then alone Vanaspati or Margarine is present

SI No.	Name of Food Article	Adulterant	Simple Method for detection of Common Adulterants	Remarks
	Butter	Mashed Potatoes, Sweet Potatoes and other starches.	The presence of mashed potatoes and sweet potatoes in a sample of Butter can easily be detected by adding a few drops of Iodine, which is brownish in colour turns to blue if mashed potatoes/sweet potatoes/other starches are present.	
VI	Oils and Fats	Argemone oil	Take small quantity of oil in a test tube. Add equal quantity of concentrated Nitric acid and shake carefully. Red to reddish brown colour in lower (Acid) layer would indicate the presence of Argemone oil	Colourless (not yellowish) Nitric acid may be used. Artificial colour if present will usually be a bright shade of colour, generally red or pink. The test may sometimes give misleading result. The test may not respond if the Argemone oil is present in small quantity.
	Oils and Fats	Mineral oil	Take 2 ml of the oil sample and add an equal quantity of N12 Alcoholic potash. Heat in boiling water bath (dip in boiling water) for about 15 minutes and add 10 ml of water. Any turbidity shows presence of mineral oil.	If mineral oil is present in small quantity this test may not be positive.
	Oils and Fats	Castor oil	Take about one ml of the oil, add 10 ml of acidified petroleum ether and mix well, Add a few drops of ammonium molybdate reagent. Immediate appearance of white turbidity indicates the presence of castor oil.	If castor oil is present in small quantity, this test may be positive
B	Sweetening Agents			
I	Sugar	Chalk powder	Dissolve 10 gm of sample in a glass of water, allow to settle, Chalk will settle down at the bottom.	
II	Pithi Sugar	Washing Soda	Add few drops of Hydrochloric acid, effervescence (give off bubbles) will indicate the presence of washing soda.	
	Pithi Sugar	Chalk powder	Dissolve 10 gm of sample in a glass of water, allow to settle, chalk will settle down at the bottom.	
III	Honey	Sugar solution	A cotton wick dipped in pure honey when lighted with a match stick burns and shows the purity of honey. If adulterated, the presence of water will not allow the honey to burn, If it does, it will produce a cracking sound.	This test is only for added water.

SI No.	Name of Food Article	Adulterant	Simple Method for detection of Common Adulterants	Remarks
IV	Sweetmeats, Ice-cream and beverages	Metanil yellow (a non-permitted coal tar colour)	Extract colour with luke-warm from food articles. Add few drops of concentrated Hydrochloric acid. If magenta red colour develops the presence of metanil yellow is indicated.	
	Sweetmeats, Ice-cream and beverages	Saccharin	<p>i. Taste a small quantity. Saccharin leaves a lingering sweetness on tongue for a considerable time and leaves a bitter taste at the end.</p> <p>ii. Take two spoons of liquid sample or about 5 to 10 gins of solid sample with little quantity of water in a test tube, add few drops of Hydrochloric acid and 10 ml of solvent ether. Shake well. Decant the ether layer into a test tube or a beaker, evaporate the ether spontaneously. Add one drop of water (warm) to the residue and taste. Sweet taste will indicate the presence of saccharin</p>	See Appendix-II.
	Sweetmeats, Ice-cream and beverages	Aluminium foil	Aluminium foil is whitish grey in colour and is readily soluble in concentrated Hydrochloric acid while pure silver foil is not,	
C	Foodorains and their Products			
I	Wheat, Rice, Maize, Jawar, Bajra, Ghana, Barley etc.	Dust, pebble, stone, straw, weed seeds, damaged grain, weevilled grain, insects, rodent hair and excreta.	These may be examined visually to see foreign matter, damaged grains, discoloured grains, insect, rodent contamination etc.	Damaged / discoloured grains should be as low as possible since they may be affected by fungal toxins, argemone seeds, Dhatura seeds etc. In moderately excessive amount can result in risk to health, Discard the damaged undesirable grains before use

SI No.	Name of Food Article	Adulterant	Simple Method for detection of Common Adulterants	Remarks
	Wheat, Rice, Maize, Jawar, Bajra, Ghana, Barley etc.	Ergot (a fungus containing poisonous substance)	(i) Purple black longer sized grains in Bajra show the presence of Ergots. (ii) Put some grains in a glass tumbler containing 20 per cent salt solution Ergot floats over the surface while sound grains settle down	
	Wheat, Rice, Maize, Jawar, Bajra, Ghana, Barley etc.	Dhatura	Dhatura seeds are flat with edges with blackish brown colour which can be separated out by close examination.	
	Wheat, Rice, Maize, Jawar, Bajra, Ghana, Barley etc.	Karnel Bunt	The affected wheat kernel have a dull appearance, blackish in colour and rotten fish smell.	
	Wheat, Rice, Maize, Jawar, Bajra, Ghana, Barley etc.	Argemone seed	Assemble mustard seed but show a protrusion on close examination. The surface of Argemone seed is grainy and rough while that of mustard seed is smooth. When Mustard seed is pressed in side, it is yellow whereas Argemone seed is white.	
II	Sella Rice (Parboiled Rice)	Metanil yellow (a non-permitted coal tar colour)	Rub a few grains in the palms of two hands. Yellow would get reduced or disappear. Add a few drops of dilute Hydrochloric acid to a few rice grains mixed with little water, presence of pink colour indicates presence of Metanil yellow	
	Sella Rice (Parboiled Rice)	Turmeric (colouring for golden appearance)	Take a small amount of sample in a test tube, add some water and shake. Dip Boric acid paper (filter paper dipped in Boric acid solution) If it turns pink turmeric is present	See Appendix-I
III	Dal whole and spilt	Khesari Dal	(i) Khesari dal has edged type appearance showing a slant on one side and square in appearance in contrast to other dals.	
	Dal whole and spilt	Khesari Dal	(ii) Add 50 ml of dilute Hydrochloric acid to the sample and keep on simmering water for about 15 minutes. The pink colour developed indicates the presence of Khesari dal.	The test is only for Khesari dal. (Metanil yellow if present will give a similar colour immediately even without simmering).

SI No.	Name of Food Article	Adulterant	Simple Method for detection of Common Adulterants	Remarks
	Dal whole and spilt	Clay, stone, gravels, webs, insects, rodent hair and excreta	Visual examination will detect these adulterants.	Reject if the number of Insects is large or if the odour is unpleasant and taste bitter or gritty
	Dal whole and spilt	Metanil yellow (a non permitted coaltar colour)	Take 5 gins of the sample with 5 ml. of water in a test tube and add a few drops of concentrated Hydrochloric acid. A pink colour shows presence of Metanil yellow	
IV	Atta, Maida Suji (Rawa)	Sand. soil, insects, webs, lumps. rodent hair and excrete	These can be identified by visual examination.	
	Atta, Maida Suji (Rawa)	Iron filings	By moving a magnet through the sample, iron filings can be separated.	
V	Besan	Khesari Flour	Add 50 ml of dilute Hydrochloric acid to 10 gins. of sample and keep on simmering water for about 15 minutes. The pink colour, if developed, indicates, the presence of Khesari flour	The test is only for Khesari del (Metanil yellow, if present will give a similar colour even without simmering).
D	Spices and Condiments			
I	Whole spices	Dirt, dust, straw, insect, damaged seeds, other seeds, rodent hair and excrete	These can be examined visually	
(a)	Black pepper	Papaya seeds	Papaya seeds can be separated out from pepper as they are shrunken, oval in shape and greenish brown or brownish black in colour.	
	Black pepper	Light black pepper	Float the sample of black pepper in alcohol (rectified spirit). The black pepper berries sink while the papaya seeds and light black pepper float.	
	Black pepper	Coated with mineral oil	Black pepper coated with mineral oil gives Kerosene like smell.	

SI No.	Name of Food Article	Adulterant	Simple Method for detection of Common Adulterants	Remarks
(b)	Cloves	Volatile oil extracted (exhausted cloves)	Exhausted cloves can be identified by its small size and shrunken appearance. The characteristic pungent of genuine cloves is less pronounced in exhausted cloves.	
(c)	Mustard seed	Argemone seed	Mustard seeds have a smooth surface The argemone seed have grainy and rough surface and are black and hence can be separated out by close examination. When Mustard seed is pressed inside it is yellow while for Argemone seed it is white	Use magnifying glass for identification.
II	Powdered spices	Added starch	Add a few drops of tincture of Iodine or Iodine solution. Indication of blue colour shows the presence of starch.	Iodine test for added starch is not applicable for turmeric powder.
	Powdered spices	Common salt	Taste for addition of common salt	
(a)	Turmeric powder	Coloured saw dust	Take a tea spoon full of turmeric powder in a test tube. Add a few drops of concentrated Hydrochloric acid. Instant appearance of pink colour which disappears on dilution with water shows the presence of turmeric If the colour persists, metanil yellow (an artificial colour) a now permitted coal tar colour is present.	This test is only for Metanil yellow
	Turmeric powder	Chalk powder or yellow soap stone powder	Take a small quantity of turmeric powder in a test tube containing small quantity of water. Add a few drops of concentrated Hydrochloric acid, effervescence (give off bubbles) will indicate the presence of chalk or yellow soap stone powder	
(b)	Chillies powder	Brick powder, salt powder or talc. powder	Take a tea spoon full of chillies powder in a glass of water. Coloured water extract will show the presence of artificial colour. Any grittiness that may be felt on rubbing the sediment at the bottom of glass confirms the presence of brick powder/sand, soapy and smooth touch of the white residue at the bottom indicates the presence of soap stone.	This test is only for earthy material.

SI No.	Name of Food Article	Adulterant	Simple Method for detection of Common Adulterants	Remarks
	Chillies powder	Water soluble coal tar colour	Water soluble artificial colour can be detected by sprinkling a small quantity of chillies or turmeric powder on the surface of water contained in a glass tumbler. The water soluble colour will immediately start descending in colour streaks	
	Chillies powder	Oil soluble coal tar colour	Take 2 gins of the sample in a test tube, add few ml of solvent ether and shake. Decant ether layer into a test tube containing 2 ml of dilute Hydrochloric acid (1 ml HOL plus 1 ml of warer). Shake it, the lower acid layer will be coloured distinct pink to red indicating presence of oil soluble colour	See also Appendix-I
III	Hing	Soap stone or other earthy material	Shake little portion of the sample with water and allow to settle. Soap stone or other earthy material will settle down at the bottom.	In compounded hing due to presence of starch, a slight turbid solution may be produced. However, this will settle down after keeping
IV	Saffron	Dried tendrils of maizecob	Genuine saffron will not break easily like artificial. Artificial saffron is prepared by soaking maize cob in sugar and colouring it with coal tar colour. The colour dissolves in water if artificially coloured. A bit of pure saffron when allowed to dissolved in water will continue to give its saffron colour so long as it lasts	
E	Miscellaneous Foods			
I	Common salt	White powdered Stone	Stir a spoonful of sample of salt in a glass of water. The presence of chalk will make solution white and other insoluble impurities will settle down.	

SI No.	Name of Food Article	Adulterant	Simple Method for detection of Common Adulterants	Remarks
II	Tea leaves	Exhausted tea or tur or gram dal husk with colour	Take a filter paper and spread a few tea leaves. Sprinkle with water to wet the filter paper. If coal tar colour is present it would immediately stain the filter paper. Wash the filter paper under tap water and observe the stains against light Spread a little slaked lime on white procelain tile or glass plate, sprinkle a little tea dust on the lime. Red, orange or other shades of colour spreading on the lime will show the presence of coal tar colour. In case of genuine tea, there will be only a slight greenish yellow colour due to chlorophyll, which appear after some time.	
	Tea leaves	Iron filings	By moving a magnet through the sample, iron filings can be separated	
III	Coffee	Chicory	Gently sprinkle the coffee powder sample on the surface of water in a glass. The coffee floats over the water but chicory begins to sink down within a few seconds. The falling chicory powder particles leave behind them a trail of colour, due to large amount of caramel	
	Coffee	Tamarind seeds powder and date seed powder	Sprinkle the suspected coffee powder on white filter/blotting paper and spray 1 per cent sodium carbonate solution on it. Tamarind and date seed powder will, if present, stain blotting paper/filter paper red.	
IV	Supari Pan Masala	Colour	Colour dissolves in water.	
	Supari Pan Masala	Saccharin	Saccharin gives excessive and lingering sweet taste and leaves bitter taste at the end.	
V	Catachu powder	Chalk	Chalk gives effervescence (gives off bubbles) with concentrated Hydrochloric acid	This test is only for Chalk.

Sl No.	Name of Food Article	Adulterant	Simple Method for detection of Common Adulterants	Remarks
VI	Silver leaves	Aluminium leaves	<p>(i). On ignition, genuine silver leaves burn away completely, leaving glistening white spherical ball of the same mass whereas aluminium leaves are reduced to ashes of dark grey blackish colour.</p> <p>(ii), Take silver leaves in test tube, add diluted Hydrochloric acid. Appearance of turbidity to white precipitate indicates the presence of silver leaves. Aluminium leaves do not give any turbidity or precipitate.</p> <p>(iii) Take a small portion of metal leaves and add a few drops of concentrated Nitric acid. Silver leaves will completely dissolve whereas aluminium leaves will remain undissolved.</p>	
VII	Vinegar	Mineral acid	Test with the Metanil yellow indicator paper, in case, the colour changes from yellow to pink, mineral acid is present	See Appendix -I

Appendix I

Method for Test

1. **Test for Metanil Yellow:** Take some sample in a test tube and add some amount of water, shake well. Add few drops of diluted hydrochloric acid, violet colour in the water portion indicates the presence of Metanil yellow.
2. **Test for Starch:** Boil the sample with some water in a test tube, cool and add a few drops of iodine solution. Appearance of blue colour indicates the presence of starch.
3. **Baudouin Test :** Take about one tea spoon full of melted ghee or butter with equal quantity of concentrated hydrochloric acid in a test tube and add to it a pinch of sugar. Shake well and allow to stand. Appearance of crimson red colour shows the presence of vanaspati or Margarine.
4. **Boric Acid Test for Turmeric :** Take a small amount of sample in a test tube, add some water and shake. Dip Boric acid paper. If it turns pink, Metanil Yellow (Coal Tar Dye) is present. Boric acid paper, can be prepared by dipping a strip of filter paper in the Boric acid solution provided in the kit. Boric Acid solution can be prepared by dissolving 5 gms. of boric acid in 100 ml concentrated Hydrochloric acid.
5. **Metanil Yellow Indicator Paper:** Metanil yellow indicator paper can be prepared by dipping a strip of filter paper in metanil yellow solution (1 gm Metanil yellow coal tar colour dissolved in 100 ml of water).
6. **Oil Soluble Coal Tar Colour :** Take a small quantity of chillies powder in a beaker and add 5 ml of rectified spirit (alcohol). Dip a small piece of white silk for two minutes. Remove the silk piece and wash with water, If the silk cloth is permanently dyed, it indicates the presence of oil soluble coal tar colour.

Appendix-II
List of Apparatus and Reagents for Developing a Simple Kit

Apparatus:

1. Magnifying Glass
2. Spatula
3. Magnet
4. Forcep
5. Lactometer
6. Beaker
7. Petri dishes
8. Dropper
9. Reagent Bottles
- 10 Spirit lamp

Reagents:

1. Hydrochloric acid
2. Nitric acid
3. Petroleum ether
4. Solvent ether
5. Rectified sprit
6. Iodine/Tincture of iodine
7. Potassium Hydroxide
8. Ammonium Molybdate
9. Boric acid
10. Sodium Carbonate
11. Metanil yellow powder
12. Test tube ordinary
13. Test tube stoppered
14. Glass rod
15. Test tube stand
16. Small plastic tray white
17. Porcelain tile white
18. Glass Cylinder
19. Glass Marking Pencil
20. Filter Paper
21. White silk cloth
22. Cotton

Precautions to be taken

Caution

1. The testing kit should be kept beyond the reach of the children as it contains harmful chemicals
2. Solvent ether is highly inflammable. Kept it away from fire.
3. Acids are high corrosive. In case of acid burn, wash immediately with cold water containing sodium bicarbonate (Meetha soda)
4. Use gloves while performing the tests.

Space for Observations and Calculations

Experiment- 11- Organochlorine Pesticides in Water by Gas Chromatographic (GC) Method

Objective

To find out the amount of organochlorine pesticides in water by gas chromatographic (GC) method

Theory

The analytical method describes determination of aldrin, α -BHC, β -BHC, γ -BHC, δ -BHC, chlordane, 4,4-DDD, 4,4'-DDE, 4,4'-DDT, dieldrin, endosulfan I, endosulfan II, endosulfan sulfate, endrin, endrin aldehyde, heptachlor, and heptachlor epoxide in water.

Principle

Measured volume of test portion (1L) is extracted with CH_2Cl_2 by shaking in separatory funnel or mechanical tumbling in bottle. CH_2Cl_2 extract is separated, dried with anhydrous Na_2SO_4 , solvent exchanged with methyl tert-butyl ether (MTBE), and concentrated to 5 ml. Pesticides are separated and measured by capillary column gas chromatography with electron-capture detection.

Apparatus

- *Separatory funnel* - 2000 ml, with TFE - fluorocarbon stopcock and ground-glass or TFE fluorocarbon stopper.
- *Tumber bottle* - 1.7 L, with TFE-fluorocarbon lined screw cap. Cut liners to fit screw cap from sheets and extract overnight with methane before use.
- *Kuderna Danish (K-D) apparatus*
 - (1) Concentrator tube -10 or 25 ml, graduated (Kontes 570050 - 1025 or 570050-2525, or equivalent). Check calibration of concentrator tube at volumes used in method. Use ground-glass stoppers to prevent evaporation of extracts
 - (2) *Evaporation flask* - 500 ml (Kontes 570001-0500, or equivalent). Attach to concentrator tube with springs.
- *Snyder columns* - Three-ball macro (Kontes 503000-0121, or equivalent); 2-ball micro (Kontes 569001-0219, or equivalent).
- *Vials* - Glass, 5-10 ml capacity, with TFE-fluorocarbon lined screw caps.
- *Separatory funnel shaker* - capable of holding 2 L separatory funnels and shaking them with rocking motion to thoroughly mix funnel contents (Eberbach Co., Ann. Arbor, MI USA).
- *Tumbler* - Capable of holding and tumbling bottles, (b), end-over-end at 30 turns/ min.
- *Boiling stones* - Carborundum, No. 12 granules (Thomas Scientific No. 1590-033), Heat 30 min. at 400°C before use. Cool and store in desiccator.
- *Water bath* - Heated, capable of control $\pm 2^\circ\text{C}$. Use bath in hood.
- *Balance* -Analytical, capable of accurately weighing to nearest 0.0001 g.
- *Gas chromatography* -Temperature programmable system suitable for use with capillary columns, including syringes, analytical columns, gases, detector, and strip chart recorder. Data system is recommended for measuring peak areas. Primary column; 30 m x 0.25 mm id DB-5 fused-silica capillary column, 0.25 μm film thickness (J&W Scientific, Inc.). Conformation column; 30 m x 0.25 mm id DB-1701 fused silica capillary column, 0.25 μm film thickness (J&W Scientific Inc.) Operating conditions; injection volume 2 μl ; He carrier gas at 30 cm/s linear velocity; injector 250°C; detector 320°C; oven programmed from 60-300°C at 4°C/min; electron capture detector.

Reagents

- *Standard solutions* - Use standards of test compounds with purity > 96% to prepare stock solutions at 1 mg/mL in methyl tert-butyl ether (MTBE). Commercially prepared stock standards may be used at any concentration if they are certified by manufacturer or independent source. Store solutions at room temperature and protect from light. Replace stock standard solutions after 2 months, or sooner if comparison with laboratory control standards indicates degradation.

- *Internal standard solution* - Prepare pentachloronitrobenzene (Purity > 98%) stock solution at 0.1 mg/mL in MTBE. Add 5 µl stock solution to 5 ml test portion extract to give final internal standard concentration of 0.1 µl pentachloronitrobenzene/ ml of extract.
- *Surrogate solution* - Prepare 4,4'-dichlorobiphenyl (purity >96%) stock solution at 0.5 mg/mL in MTBE. Add 50 µl stock solution to 1 L test sample prior to extraction to produce surrogate concentration of 25 g]4,4'-dichlorobiphenyl/L in test sample and, assuming quantitative recovery, 5.0 g/mL in extract.
- *Instrument performance solution* - Prepare individual stock standard solutions containing chlorothalonil, chlorpyrifos, DCPA, and d -BHC at 0.10 µl/mL in MTBE. For assessing instrument performance, combine 50 µl chlorothalonil stock solution, 2 µl chlorpyrifos stock solution, 50 µl DCPA stock solution, and 40 µl d -BHC stock solution in 100 mL volumetric flask and dilute to volume with MTBE.
- *Solvents* - Acetone, methylene chloride, and MTBE Distilled-in-glass quality, or equivalent.
- *Phosphate buffer*- pH 7 Mix 29.6 mL 0.1 M HCl and 50 mL 0.1 M dipotassium hydrogen phosphate.
- *Sodium sulfate* - Granular, anhydrous, ACS grade. Heat in shallow tray for > 4 h at 450°C to remove interfering organic substances.
- *Sodium chloride* - Crystals ACS grade. Heat in shallow tray for > 24 h at 450°C to remove interfering organic substances.
- *Reagent water* - Water reasonably free of contamination that would prevent determination of any analyte of interest.
- *Preservative* - Mercuric chloride solution. 10 mg HgCl (ACS grade) /mL reagent water
- *Sodium thiosulfate* - Na₂S₂O₃, Granular, anhydrous, ACS grade.

Procedure

Preparation of laboratory sample bottles

Add 1 ml_ preservative, to glass laboratory sample bottle. If residual chlorine is expected to be present in laboratory samples, add 80 mg Na₂S₂O₃ to laboratory sample bottle before collection.

Laboratory Sample Collection

Collect 1 L grab laboratory samples in glass bottles by conventional sampling practices. Since bottles contain preservative and Na₂S₂O₃, do not preinse bottles with laboratory sample before collection. Add laboratory sample to bottle containing preservative, seal laboratory sample bottle, and shake vigorously 1 min. Refrigerate laboratory samples at 4°C from time of collection until extracted. Protect from light. Extract laboratory samples within 7 days of sample collection.

Laboratory Sample Preparation

Automated extraction method - Add preservative, to any laboratory samples not previously preserved. Mark water meniscus on side of laboratory sample bottle for later determination of volume. Add 50 µl surrogate stock solution, to laboratory sample. If mechanical separatory funnel shaker is used, pour entire laboratory sample into 2 L separatory funnel. If mechanical tumbler is used, pour entire laboratory sample into tumbler bottle. Adjust sample to pH 7 by adding 50 ml_ phosphate buffer, C. check pH and add H₂SO₄ or NaOH if necessary.

Add 100 g NaCl, seal, and shake to dissolve salt. Add 300 ml CH₂Cl₂ to tumbler bottle or separatory funnel, seal, and shake 30s to rinse inner walls. Transfer rinse to test portion contained in separatory funnel or tumbler bottle, seal, and shake 10s, venting periodically. Repeat shaking and venting until pressure release is not observed during venting. Reseal and place test portion container in appropriate mechanical mixing device (separatory funnel shaker or tumbler). Shake or tumble test portion for 1 h. After extraction, pour contents of tumbler bottle into 2 L separatory funnel if tumbler bottle as used. Let organic layer separate from water phase for > 10 min. If emulsion interface between layers is more than one-third volume of solvent layer, complete phase separation mechanically. Collect CH₂Cl₂ extract in 500 ml_ Erlenmeyer flask containing ca 5 g anhydrous Na₂SO₄. Swirl flask to dry extract; let flask sit 15 min. Determine original laboratory sample volume by refilling laboratory sample bottle to mark and transferring water to 1000 ml_ graduated cylinder. Record sample volume to nearest 5 ml.

Manual extraction method - Add preservative to laboratory samples not previously preserved. Mark water meniscus on side of tumbler bottle for later determination of laboratory sample volume. Add 50 µl surrogate stock solution, to laboratory sample, pour entire contents into 2 L separatory funnel. Adjust to pH 7 by adding 50 ml phosphate buffer. Check pH and add H₂SO₄ or NaOH if necessary. Add 100 g NaCl to contents, seal, and shake to dissolve salt. Add 60ml CH₂Cl₂ to tumbler bottle, seal and shake bottle 30s to rinse inner walls.

Transfer rinse to separately funnel and extract laboratory sample by vigorously shaking funnel for 2 min with periodic venting to release excess pressure. Let organic layer separate from water phase for > 10 min. If emulsion interface between layers is more than 1/3 volume of solvent layer, complete phase separation mechanically. Collect CH₂Cl₂ extract in 500 ml Erlenmeyer flask containing ca 5 g anhydrous Na₂SO₄. Add second 60 ml portion of CH₂Cl₂ to separately funnel and repeat extraction procedure a second time, combining extracts in Erlenmeyer flask. Perform third extraction in same manner. Swirl flask to dry extract; let flask sit for 15 min. Determine original laboratory sample volume by refilling laboratory sample bottle to mark and transferring water to 1000 ml graduated cylinder. Record laboratory sample volume to nearest 5 ml.

Extract concentration

Assemble K-D concentrator by attaching 25 ml concentrator tube to 500 ml evaporation flask. Decant CH₂Cl₂ extract into concentrator. Rinse remaining Na₂SO₄ with two 25 ml portions of CH₂Cl₂ and decant rinses into concentrator.

Add 1 or 2 clean boiling stones to evaporation flask and attach macro-Snyder column. Prewet column by adding ca 1 ml CH₂Cl₂ to top. Place K-D apparatus on 65-75°C water bath so that concentrator tube is partially immersed in hot water and entire lower, rounded surface of flask is bathed with hot vapour. Adjust vertical position of apparatus and water temperature as required to complete concentration in 15-20 min. At proper rate of distillation, balls of column will actively chatter, but chambers will not flood. When apparent volume of liquid reaches 2 ml remove KD apparatus and let it drain and cool > 10 min. Remove Snyder column and rinse flask and its lower joint with 1-2 ml MTBE, collecting rinse in concentrator tube. Add 5-10 ml MTBE and fresh boiling stone.

Attach micro-Snyder column to concentrator tube and prewet column by adding ca 0.5 ml MTBE to top. Place micro K-D apparatus on water bath so that concentrator tube is partially immersed in hot water. Adjust vertical position of apparatus and water temperature as required to complete concentration in 5-10 min. When apparent volume of liquid reaches 2 ml, remove apparatus from bath and let it drain and cool.

Add 5-10 ml MTBE and boiling stone and reconcentrate to 2 ml. Remove micro KD apparatus from bath and let it drain and cool. Remove micro-Snyder column, and rinse walls of concentrator tube while adjusting volume to 5.0 ml with MTBE.

Add 5 µl internal standard stock solution, C to laboratory sample extract, seal, and shake to distribute internal standard. Transfer extract to appropriate-size TFE fluorocarbon-sealed, screw cap vial and store at 4°C until analysis. A 14-day maximum extract storage time is recommended.

Calibration of Gas Chromatograph with Electron-Capture Detector

Table 1 summarizes retention times and detection limits observed using this method of analysis of chlorinated pesticides.

Table 1. Relative retention times and estimated method detection limits for chlorinated pesticides

Analyte	Relative retention time		Estimated MDL
	Primary	Confirmation	
Aldrin	1.18	1.12	0.0750
α -BHC	0.93	0.97	0.0200
β -BHC	0.98	1.187	0.0700
γ -BHC	1.03	1.22	0.0100
δ -BHC	0.99	1.04	0.0150
α -chlordane	1.31	1.31	0.0075
γ -chlordane	1.28	1.29	0.0015

4,4' ODD	1.42	1.38	0.0025
4,4' DDE	1.35	1.32	0.0100
4,4 DDT	1.48	1.48	0.0600
Dieldrin	1.35	1.35	0.0200
Endosulfan I	1.30	1.28	0.0150
Endosulfan II	1.40	1.45	0.0150
Endosulfan sulphate	1.47	-	0.0150
Heptachlor	1.11	1.04	-
Heptachlor expoxide	1.24	1.24	0.0150

Calculations

The concentration (C) in the sample can be calculated from the following equation :

$$C (\mu\text{g/L}) = (A) (V) / (V_t) (V_s)$$

where,

A = Amount of the material injected (ng)

V_i = Volume of extract injected (μ l)

V_t = Volume of the total extract (μ l)

V_s = Volume of the water extract (ml)

Reference

AOAC Official method. 17th Edition. 990.06 (2000).

Space for Observations and Calculations

Experiment- 12- N-Methylcarbamoyloximes and N-Methylcarbamates in Finished Drinking Water by High Performance Liquid Chromatography (HPLC)

Objective

To find out the N-methylcarbamoyloximes and N-methylcarbamates in the given samples of finished drinking water by high performance liquid chromatography (HPLC).

Theory

The analytical method prescribes HPLC method for determination of aldicarb, aldicarb sulfone, aldicarb sulfoxide, baygon, carbaryl, carbofuron, 3-hydroxy carbofuron, methiocarb, methomyl, and oxamyl in drinking water.

Principle

Water sample is filtered and measured volume is directly injected onto reversephase LC column. Analytes are separated by gradient elution chromatography. After elution from LC column, analytes are hydrolyzed with NaOH at 95°C. Methylamine formed during hydrolysis is reacted with O-phthalaldehyde and 2-mercaptoethanol to form highly fluorescent derivative, which is detected by fluorescence detector. Estimated method detection limits range from 0.5µg/L for methomyl to 4.0µg/L for methiocarb; estimated method detection limits for 8 other compounds range from 1.0 to 2.0 µg/L.

Apparatus

- a. Grab sample bottle. 60-ml, borosilicate glass, screw-cap vials (Pierce No. 13075 meets these specifications) and caps with PTFE-faced silicone septa (Pierce No. 12722 meets these specifications). Before use, wash vials and septa with soap and water, followed by 3 tap water rinses and 3 deionized water rinses.
- b. Balance- Analytical, capable of accurately weighing to nearest 0.0001 g.
- c. Macrofilters.- 47 mm 0.45µm, nongridded, cellulose acetate filters for water phases; 47 mm, 0.5µm nongridded, PTFE filters for organic phases.
- d. Microfilters.- 13 mm stainless steel filter holder and 13 mm diameter, 0.2 µm polyster filters (Nuclepore No. 180406 meets these requirements).
- e. Hypodermic syringe.- 10µL, glass, with Luer-Lok tip.
- f. Syringe valve.- 3 way.
- g. Syringe needle-7-10 cm long, 17 gauge, with blunt tip.
- h. Microsyringes. - Various sizes.
- i. Solution storage bottles.- Amber glass, 10-15 mL capacity with TFEfluorocarbon lined screw cap.
- j. LC system.- Capable of injecting 200-400 µl aliquots and performing binary linear gradients at constant flow rate. Data system is recommended for measuring peak areas. Primary column: 250 mm X 4.6 mm id stainless steel packed with 5 µm Beckman Ultrasphere ODS. Mobile phase linear gradient from methanol water (15+85) to methanol in 32 min at 1.0 mL/min. Confirmation column: 250 mm X 4.6 mm id stainless steel packed with 5 µm supelco LC-1. Mobile phase linear gradient from methanol water (15+85) to methanol in 32 min at 1.0 mL/ min.
- k. Postcolumn reactor, -Reactor constructed with PTFE tubing and equipped with pumps capable of mixing 0.5 ml/min OPA reaction solution, C(i), and 0.5 ml/min NaOH, C(f), into mobile phase. Reactor must contain mixing tees and two 1.0 ml delay coils, one thermostated at 95°C.
- l. Fluorescence detector. Capable of excitation at 230 nm and detection of emission energies > 418 nm.

Reagents

- a. Standard solutions.- Use standards of test compounds with purity >96% to prepare stock solutions at 1.00 µg/mL in methanol. Commercially prepared stock standards may be used at any concentration if they are certified by manufacturer or independent source. Transfer stock standard solutions into TFE fluorocarbonsealed screw-cap vials. Store at room temperature protected from light. Replace stock standard solutions after 2 months, or sooner if comparison with laboratory control standards indicates degradation.

- b. Instrument performance solution.- Combine 20 μ l 3-hydroxycarbofuran stock solution, (a), and 1.0mL Aldicarb sulfoxide stock solution, (a) in 10 mL volumetric flask and dilute to volume with methanol.
- c. Reagent water. Distilled Water reasonably free of contamination that would prevent determination of analytes.
- d. Water.- LC grade.
- e. Methanol.- LC grade. Filter and degas with helium before use.
- f. Sodium hydroxide.-0.05M. 2.0g NaOH/1.0L reagent water Filter, B and degas with helium before use.
- g. Mercaptoethanol-acetonitrile.- (1+1). Mix 10.0 mL 2-mercaptoethanol and 10.0 mL CH₃CN. Store in borosilicate glass vial or bottle with PTFE-lined cap. (Caution: Strong odor. Store in hood.)
- h. Sodium borate.-0.05N. 19.1 g Na₂B₄O₇.10H₂O/1.0 L reagent water Sodium borate dissolves completely at room temperature if prepared day before use.
- i. OPA reaction solution.- 100 \pm 10 mg o-phthalaldehyde (mp 55-58 $^{\circ}$)/10 μ L CH₃OH Add to 1 L 0.05M Na₂B₄O₇ solution, (h). mix, filter, B (c), and degas with helium. Add 100 μ l 2-mercaptoethanol, (g), and mix. Prepare solution fresh daily.
- j. Helium.- For degassing solutions and solvents
- k. Monochloroacetic acid buffer.- pH 3. mix 156 mL 2.5M monochloroacetic acid and 100mL 2.5M potassium acetate.
- l. Sodium thiosulfate.- Na₂S₂O₃. granular, anhydrous. ACS grade.
- m. Buffered reagent water.- Mix 10mL monochloroacetic acid buffer, (k), and 1L reagent water, (c).
- n. Internal standard solution.- Prepare 4-bromo-3,5 dimethylphenyl N-methylcarbamate (BDMC) (Purity 98%, Aldrich Chemical Co.) stock solution at 0.1 mg/mL in methanol.

Procedure

Preparation sample Bottles

Add 1.8 mL monochloroacetic acid buffer, C(k), to sample bottle, B(a). if residual chlorine is expected, add 5 mg Na₂S₂O₃ to bottle before sample collection.

Sample collection

Collect 60mL grab samples in glass bottles by conventional sampling practices. Because bottles contain buffer and Na₂S₂O₃, do not prerinse with sample before collection. Add sample to bottle, seal, and shake vigorously 1 min. Refrigerate samples at 4 $^{\circ}$ from time of collection until storage. Store at -10 $^{\circ}$ until analyzed. Analyze samples within 28 days of collection.

Sample Preparation

Adjust pH of sample or standard to pH 3 \pm 0.2 by adding 1.5mL 2.5M monochloroacetic acid buffer, C (k), to 50mL sample. This step should not be necessary if sample pH was adjusted during sample collection. Fill 50mL volumetric flask to mark with sample. Add 5 μ l internal standard stock solution, C(n), to the 50mL of sample (final concentration 10 μ g/L). affix 3 -way valve to 10 μ l syringe. Place clean filter in filter holder, B(d), and affix filter holder and 7-10 cm syringe needle to syringe valve. Rinse needle and syringe with reagent water, C(C). Prewet filter by passing 5mL reagent water through filter. Empty syringe and check for leaks. Draw 10mL sample into syringe and expel through filter. Draw another 10mL sample into syringe, expel through filter, and collect last 5mL for analysis. Rinse syringe with reagent water. Discard filter. Inject 400 μ l of collected sample into LC system under conditions in B (j).

Calibration of LC system

Table 1 presents retention times and estimated method detection of 10 carbamate pesticides.

Analyte	Retention time	EDL
Aldicarb	21.4	1.6
Aldicarb sulfone	12.2	2.0
Aldicarb sulfoxide	17.5	2.0
Baygon	23.4	1.0
Carbaryl	25.4	2.0

Analyte	Retention time	EDL
Carboftiron	24.4	1.5
3 -hydroxycarboruron	19.0	2.0
Methiocarb	28.6	4.0
Methomyl	14.8	0.50
Oxamyl	14.6	2.0

Calculation

$$C (\mu\text{g/L}) = (A) (V) / (V_t) (V_s)$$

where,

A = Amount of the material injected (ng)

V = Volume of extract injected (μl)

V_t = Volume of the total extract (μl)

V_s = Volume of the water extract (ml)

Space for Observations and Calculations

Experiment- 13- Acesulfame K Detection and Determination in Sweets

Objective

To find out the Acesulfame K in the given sample of sweets.

Method

Qualitative Method (Thin-layer Chromatographic detection of acesulfame saccharin and cyclamate)

Apparatus:

- UV lamp (360 nm);
- Ion-exchange resin: Amberlite LA-2.

Reagents:

- Polyamide
- 2,7-dichlorofluorescein
- Bromine
- Formic acid
- Ammonia 5%
- Xylol
- Propanol
- Methanol.

Procedure:

Extract the sweetener from acidified food product with water or take acidified aqueous extract and pass through the ion-exchanger and wash with water. Elute the sweeteners with dilute ammonia solution. Evaporate the ammoniacal solution under vacuum to dryness and take up the residue in 1 ml of 50% methanol (alternatively extract these sweeteners from acidified sample, pH 0.6, with ethyl acetate and use concentrated ethyl acetate for TLC).

Apply 2-10 μ l of sample solution along with standards on TLC plates coated with polyamide. Develop the plate to about 15 cm height with a developing solvent consisting of xylol: n-propanol: formic acid (5:5:1). Dry the plates in a current of air and spray with 0.2% solution of dichlorofluorescein and after being dried, examine under UV light. To identify the spots in day light, place the plate in chamber containing bromine and then expose to ammonia vapour. Spots appear on a reddish background.

Quantitative method: (Analysis of acesulfame by high pressure liquid chromatography)

Apparatus:

Beaker, pipette, flasks,
HPLC instrument of any suitable make with a UV detector at 227 nm,
column; Lichrosorb-RP 18 (10 μ m).

Reagents:

- (i) Mobile phase: methanol : water (10 : 90) : Adjust this mixture to 0.01M using tetrabutylammonium sulphate,
- (ii) Standard solution of acesulfame: 0.1 mg/ml in distilled water.

Operating Conditions:

(1) Pressure: 160 bar (2) Flow rate: 40 ml/hr (3) Temperature (ambient) (4) Sample Volume: 10-20 μ l.

Preparation of sample:

(a) Liquid samples such as juices: filter through 0.45 μ m filter (Millipore Inc.) and inject 10-20 μ l.

(b) Solid samples: Stir 10 gm of sample vigorously with 100 ml distilled water for 30 minutes and centrifuge. Pass an aliquot of this solution through 0.45 μm filter, discard the first few drops of filtrate and collect the filtrate and chromatograph.

Procedure:

Inject standard solution ranging from 5-20 μl and record the peaks. Calculate the peak area and draw a calibration graph using μg of substance vs peak area. Inject samples solution ranging from 10-20 μl and record the peak area for sample. Calculate the acesulfame content of the sample from its peak area and the calibration graph.

Determination of Acesulphame – K, Aspartame and Saccharin by High Performance Liquid Chromatography

Principle

Extraction of sample with water or eluent, if necessary, clarification on solid phase extraction column or with Carrez reagent, chromatography at an HPLC reversed phase column and spectrophotometrical determination at a wavelength of 220 nm.

Reagents

1. Acetonitrile for HPLC
2. Methanol for HPLC
3. Pot. dihydrogen phosphate
4. Dipotassium hydrogen phosphate
5. Tetrabutyl ammonium hydrogen sulphate
6. Phosphoric acid 85% (w/w)
7. Phosphoric acid 5% (w/w). Carefully pipette 6 ml of Phosphoric acid at (6) above into a 100 ml volumetric flask which already contains 80 ml water. Dilute to mark with water.
8. Hydrochloric acid 25 % (w/w)
9. Carrez Solution 1 – Dissolve 15 gm Potassium hexacyanoferrate ($\text{K}_4[\text{Fe}(\text{CN})_6] - 3 \text{H}_2\text{O}$) in water and dilute to 100 ml
10. Carrez Solution 2 – Dissolve 30 gm Zinc Sulphate ($\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$) in water and dilute to 100 ml
11. Phosphate Buffer solution II – (KH_2PO_4 0.0125 mol / litre, pH = 3.5. Dissolve 1.70 gm potassium dihydrogen phosphate in 800 ml of water in a 1000 ml beaker. adjust to pH 3.5 with phosphoric acid. Transfer the solutions to a 1000 ml vol. flask and dilute to mark with water.
12. Phosphate Buffer solution III – pH 6.5 Dissolve 5.46 gm of potassium dihydrogen phosphate in 500 ml water in a 1000 ml beaker. adjust to pH 6.5 with dry dipotassium hydrogen phosphate. Add to the solution 3.4 gm of tetra butyl ammonium hydrogen sulphate and stir to dissolve. Adjust the pH to 6.5 by addition of more dipotassium hydrogen phosphate. Add 250 ml of methanol and adjust the pH to 4.0 by drop wise addition of HCl (8). Transfer this solution into a 1000 volumetric flask and dilute to the mark with water.
13. Mobile Phase – Phosphate buffer and either acetonitrile or methanol. Filter the phosphate buffer used for the mobile phase and either acetonitrile or methanol separately through suitable membrane filters, of pore size 0.45 μm and de gas for 5 minutes in an ultrasonic bath. Add carefully measured the required amounts of phosphate buffer and acetonitrile as given in A,5 and mix. Prepare the mobile phase freshly on the day of use
14. Control solution - containing acesulphame – K, Sodium saccharin and aspartame (and optionally diketopiperazine, aspartylphenyl alanine, phenylalanine, caffeine, benzoic acid, theobromine, hydroxyl methyl furfural, and vanillin) In a 100 ml volumetric flask, weigh to the nearest 0.1 mg, 30 mg of acesulphame – K, 20 mg of sodium saccharin, 220 mg of aspartame (and optionally 60 mg caffeine, 100 mg benzoic acid, 100 mg vanillin, 10 mg diketopiperazine, 10 mg of phenylalanine, 10 mg of aspartylphenylalanine, 20 mg of hydroxyl methyl furfural and 70 mg of theobromine). Dissolve and dilute to mark with water. Pipette 20 ml of the solution into a 100 ml vol. flask and dilute to mark with water.

15. Stock solution – Weigh to the nearest 0.1 mg, 100 mg of Acesulphame – K, 100 mg of sodium saccharin and 100 mg of aspartame in the same 100 ml volumetric flask. Dissolve and dilute to mark with water.
16. Standard Solution 1 – Pipette 10 ml of the stock solution (15) into a 100 ml vol. flask and dilute to mark with water.
17. Standard solution 2 – Pipette 5 ml of the stock solution into a 100 ml vol flask and dilute to mark with water.
18. Standard Solution 3 – Pipette 1 ml of the stock solution into a 100 ml vol flask and dilute to mark with water.

Apparatus

1. Analytical Balance
2. High speed blender or homogenizer
3. Volumetric flasks – 100, 250, 500 and 1000 ml capacity.
4. Beaker 1000 ml
5. Pipettes – 1, 5, 10, 20, 25, 100 ml
6. Micropipette 1000 μ L
7. Graduated cylinder – 1000 ml
8. Funnel
9. Fluted filter papers, medium fast qualitative
10. Water bath
11. Ultrasonic bath
12. Centrifuge
13. Degassing system
14. Membrane filters – pore size 0.45 μ m or smaller with filter holders and suitable syringe.
15. Solid phase extraction column
16. High Performance Liquid Chromatograph – equipped with UV detector (capable of operating at a wavelength of 220 nm, preferably a diode array detector) and equipped with recorder or integrator which allows measurement of peak heights and peak areas)
17. Column, Reverse phase – a RPC Stationary phase of 5 μ m, a length of 250mm, internal dia 4 mm, a guard column, RP C 18 (optional but strongly recommended for all solid sample materials).
Performance criteria for suitable analytical columns is the baseline resolution of the respective analyte.

Procedure

Preparation of sample test solution

- (1) Clear liquid products (lemonades, cola, beverages)

Dilute 20 ml of the liquid in a 100 ml volumetric flask with water. Filter the solution through a membrane filter of pore size 0.2 μ m before injection.

- (2) Cloudy liquid samples (juices , flavoured milk drinks)

Dilute 20 ml sample with 50 ml water in a 100 ml volumetric flask. Add 2 ml Carrez solution 1, mix and 2 ml of Carrez solution 2, dilute to mark with water and filter through a fluted filter paper. Pass the filtrate through a membrane filter of pore size 0.45 μ m before injection. To make allowance for the volume of any precipitate, if the fat free insoluble matter in the initial sample mass exceeds approx 3 gm, it is advisable to centrifuge the clarified solution for 10 minutes before filtering it quantitatively into a 100 ml volumetric flask. Wash the settled matter twice with water and centrifuge again, collect each of the supernatant in the 100 ml vol flask and then dilute the solution to mark with water.

- (3) Jams, preserves, marmalade and related products

Weigh to the nearest 1 mg, 20 gm of homogenized sample in a 100 ml vol. flask, add about 60 ml water and place the flask in an ultrasonic bath at 40 C for 20 minutes. The temperature should not

exceed 40 C since aspartame can get degraded. Cool to room temp. Add 2 ml Carrez solution 1 , mix followed by 2 ml carrez solution 2 . Shake vigorously and allow to stand for 10 minutes. Dilute to mark with water. Filter the solution through a fluted filter paper. Pass the filtrate through a membrane filter of pore size 0.45 um before injection. To make allowance for any precipitate, if the fat free insoluble matter in the initial mass exceeds 3 gm, it is advisable to centrifuge the clarified sample solution for 10 minutes at 1400 r.p.m before filtering it quantitatively into 100 ml vol flask. Wash with water and centrifuge again as in case of cloudy liquid samples.

(4) Semisolid and solid products

Weigh 10 – 20 gm of thoroughly homogenized sample in a 100 ml vol flask. Add about 50 ml water and place the vol flask in an ultra sonic bath at 40 0 C for 20 minutes. Cool to room temperature, add 2 ml Carrez solution 1 , mix and add 2 ml of Carrez solution 2, dilute to mark with water and filter through a fluted filter paper. In case of very complex matrices, additional purification using the solid phase extraction column may be necessary to protect the separating column, since colouring, flavouring and fat can not be separated by Carrez solution. In this case add 2 ml of clarified filtrate to the cartridge, previously activated with 3 ml of methanol and 20 ml water and elute with about 20 ml of mobile phase. Pass the filtrate through a membrane filter of pore size 0.45 um before injection. To make allowance for the volume of any precipitate follow procedure mentioned above.

(5) Custard Powder

Weigh 10 gm sample in a 500 volumetric flask. Add about 400 ml of water and proceed as described above. Add 6 ml of Carrez solution 1 and 2 for clarification

Identification

Identify the intense sweeteners by comparing the retention times of the analyte concerned in the sample solution with that of the standard substance or by simultaneous injection of the standard solution and the sample solution.

Determination

Integrate the peak areas or determine the peak heights and compare the results with the corresponding values for the standard substance with the nearest peak area / height or use a calibration graph. Check the linearity of the calibration graph.

Chromatographic conditions

Type – reversed phase (RP)

Stationary phase and column lengths – spherical particles of 3 um, for column lengths of 100mm, upto 10 um for column lengths of 300 mm

Internal diameter – 4.0 mm

Guard column – recommended (optional) – bondapak C 18 or partisil ODS3, or superspher60 RP select B

Flow rate – 0.8 ml /min to 1 ml / min

Injection volume – 10 µl upto 20 µl

Detection – Photometrical (UV) at a wavelength of

217 nm for aspartame

227 for Acesulphame – K

265 for saccharin

220 nm for all intense sweeteners if the detector does not allow a wavelength switch in one run.

Mobile phase – The following proportions are satisfactory

Solution A – phosphate buffer = 0.02mol / l pH = 4.3

Solution B = phosphate buffer = 0.0125 mol / l pH = 3.5

Solution C phosphate buffer = pH 6.5

Solution D – acetonitrile

Solution E – Methanol

(a) solution A + solution D (90+10 v/v)

- (b) solution B + solution D (80 = 20 v/v)
- (c) solution B + solution D (85 + 15 v/v)
- (d) solution B + solution D (90 + 10 v/v)
- (e) solution B + solution D (95 + 5 v/v)
- (f) solution B + solution D (98 + 2 v/ v)
- (g) solution C + solution E (90 +10 v/v)

Calculation

Calculate the mass fraction w expressed in mg / kg or mass concentration p in mg / litre of the intense sweetener as under

$$w \text{ or } p = (A_1 \times V_1 \times m_1 \times F_1 \times 1000) / (A_2 \times V_2 \times m_0)$$

Where A₁ = peak area of the intense sweetener concerned obtained with sample test solution

A₂ = peak area of the intense sweetener concerned obtained with the standard test solution

V₁ = total volume of sample test solution in milliliters

V₂ = total volume of the standard test solution in milliliters

m₁ =mass of the intense sweetener concerned in standard test solution

m₀ = initial sample mass in gms or mls

F₁ = dilution factor for the purification method used (e.g Column Clarification =10, Carrez clarification = 1

Experiment- 14- Sensory Evaluation- General Concepts

Objective

To explore the general concepts of sensory evaluation in class activities.

Theory

What is sensory evaluation?

Sensory evaluation is a scientific discipline that analyses and measures human responses to the composition of food and drink, e.g. appearance, touch, odour, texture, temperature and taste. It provides an ideal opportunity to analyst to evaluate and give feedback on their dishes, test products and experimental designs.

Sensory Analysis in the Food Industry

Sensory analysis testing is used considerably in the food industry for product development, recipe modification and the evaluation of products. It also plays a key role in quality control and in the marketing of products. Many types of sensory analysis tests have been devised to fulfil a number of specific objectives. These tests are grouped into three categories.

Categories of Sensory Analysis Tests

1. Preference Tests
2. Difference Tests
3. Descriptive Tests

Within each category there are various sensory analysis tests that can be carried out. The tests which are suitable for use in the classroom are included below.

1. Preference Tests

Preference tests supply information about whether people like or dislike a product. Preference tests are used in the food industry to determine:

- if consumers like a product
- if one product is preferred over another
- if consumers intend to use a product.

Preference tests are often referred to as “acceptance” or “consumer” tests.

Preference Tests Suitable for Classroom Use

- Paired Preference Test
- Hedonic Rating Scale
- Food Action Rating Test
- Preference Ranking Test

2. Difference Tests

Difference tests are used to detect small differences in foods. Difference tests are used in the food industry to answer some of the following questions:

- does a difference exist?
- would people notice the difference?
- how would you describe the difference?

Difference tests are sometimes called “discrimination” tests.

Difference Tests Suitable for Classroom Use

- Simple Difference Paired Comparison Test
- Directional Paired Comparison Test
- Triangle Test
- Duo-Trio Test

3. Descriptive Tests

Descriptive tests are used to describe the perceived sensory characteristics of products. Descriptive tests can be used in the food industry to answer some of the following questions:

- what does the product taste like?
- what are its perceived sensory characteristics / attributes?
- how does a change in processing / packaging / storage conditions affect the sensory quality of this product?

Descriptive Tests Suitable for Classroom Use

- Descriptive Ranking Test
- Descriptive Rating Test – one product
- Descriptive Rating Test – two products

Uses of Sensory Analysis in the Food Industry

Sensory analysis testing has become an integral part of the food industry. It has many different purposes. It can be used to:

- evaluate a range of existing food products
- analyse a test kitchen sample for improvement
- gauge consumer response to a product
- check that a final product meets its original specifications
- evaluate differences in similar products
- analyse specific attributes e.g. shortness in biscuits.

It is important that the test chosen should suit the particular purpose. Very often more than one type of test will have to be carried out on products. Companies often develop products to taste like another, e.g. own label foods to taste like the brand leader. If a food is designed to taste like another, then a difference test is used. This may be followed by a preference test to find out the acceptability of the new product among consumers.

Preference tests can be used to research how a company's product compares to that of its competitors. A ranking test may be done and if the results of this are favourable to the company, this may be presented to retailers to persuade them to allocate more shelf space to the company's product.

Cost and quality are important factors in the food industry. A company may consider changing the supplier of one of the ingredients in a product for economic reasons. It is important that consumers do not detect that the product has been changed in any way. In this case the company may use a panel of trained testers to carry out difference tests to determine if the testers can detect a difference from the original product.

Companies may contemplate changes to their existing product based on consumer demand e.g. healthy eating, by replacing salt with a low sodium alternative. It is important that food companies are attentive to the demands of the consumer in order to retain their market share. As a result, sensory analysis testing is ongoing in industry.

Food companies may carry out their own sensory analysis testing or they may contract a specialist company to do this for them. Results of sensory analysis tests are calculated either manually or by computer programme. Statistical analysis is carried out to ensure reliability and validity of the results.

Product Development in the Food Industry

Increased competition in the food industry has led to the development of new products. There is also constant re-appraisal of existing products, leading to improvements in e.g. flavour or packaging.

Product development may involve:

- **Making a completely new food product** - developing ideas for a new product by drawing up the product profile e.g. shape, size
- **Modifying an existing food product** - making changes to an original recipe e.g. adding or removing an ingredient to improve flavour or changing the size or shape of a product
- **Matching an existing food product** - copying other popular branded products of similar types.

Stages of Product Development

The process of product development involves a series of complex stages, requiring the combined talents of many specialists to make it successful. The main stages are outlined below.

1. Development of ideas

Ideas are developed for the new product and a specification is produced.

2. Testing of ideas on a small scale

Ideas are tested on a small scale. Research is carried out to formulate a number of recipes and specify the ingredients to be used. Several versions are made, altering ingredients or processes. In other words the products are prototyped, often by a professional chef or food consultant.

3. Product modification

Trained testers evaluate the product being developed to ensure that it displays the desired characteristics. The recipe may need to be modified and further testing is carried out.

4. Consumer testing

The product is then tested to determine consumer acceptability.

5. Final product specification

The final product specification is then agreed detailing the exact ingredients and methods of production.

6. Large scale production trial

Food scientists work together in a pilot plant to determine the best method of producing large quantities of the product.

7. Large scale production

The product is then produced on a large scale. This is done under controlled conditions to maintain consistent product quality.

8. Packaging and labelling

Appropriate packaging is chosen bearing in mind shelf-life considerations. Labelling is designed to meet legal requirements.

9. Product launch

The product is advertised and then launched.

Sensory analysis testing is carried out at many stages as the product is being developed.

Sensory Analysis in Class Room

In the class, sensory analysis is used for the following activities:

- **evaluation of products / dishes**
- **product development and recipe modification.**

Evaluation of Products

When evaluating products students may:

- identify and describe the characteristics of a food e.g. flavour, appearance, shape
- rate the characteristics of a food e.g. colour - very pale to very dark
- compare foods e.g. in terms of taste.

Product Development and Recipe Modification

In product development and recipe modification students may:

- draw up a product profile that describes the desired characteristics of a food or product
- design a product e.g. a range of biscuits for a cake sale
- modify a recipe to suit the design e.g. change the ingredients, flavour, shape
- compare a modified recipe with an original recipe, or with a similar branded product
- test the end product for acceptability e.g. among their classmates
- assess quality assurance e.g. shelf life issues such as how the absorption of fat from a food or the retention of moisture may affect packaging.

Tasting and Testing in the Classroom

Before carrying out sensory analysis tests as required for an assignment, it would be important to have a certain amount of preparation done. This could include:

- the basic theory of sensory analysis
- a vocabulary of descriptive terms
- guidelines for testing in the classroom
- procedure for tests appropriate to the assignment.

Guidelines for Testing in the Classroom

Where to Test

Ideally testing takes place in special testing booths. However, a quiet area of the classroom, with adequate light and ventilation, could be used. Ensure adequate space between testers.

Testing Session

It is very important that silence is maintained throughout the session and that students should not discuss their results. Keep the testing session short to avoid fatigue.

Timing

Mid-morning is the best time before any aromas of cooking fill the air. If this is not possible, try and ensure that the room is odour free and well ventilated. Testers should not eat strongly flavoured food in the thirty minutes immediately prior to the test.

Number in Group

This will depend on the size of the class. When arranging tests in class, it is important that the people involved in testing the food samples should not be involved in the coding and arranging of samples, or in the collating of results for these samples. This is because they will be familiar with the coded samples on the trays that they have set up.

Special Dietary Conditions

Take into account students with special dietary conditions e.g. a coeliac should not test starchy foods.

Hygiene

Ensure that the general rules of hygiene apply for the handling of all food samples.

Equipment

The size and shape of containers should be standard. Polystyrene cups, paper plates and plastic spoons are useful equipment for testing food. These could either be disposable or a designated set of plastic equipment.

Quantity of Sample

The samples presented should be sufficient in amount. Ensure that all samples are uniform in colour, shape and size.

Temperature

It is important that all samples presented are at the same temperature.

Coding of Samples

One of the most important things about testing is making sure that testers are unaware of the identity of products, which means that coding is necessary. This is an essential part of every test carried out. Samples can be coded with geometric shapes e.g. triangle, square, circle. They can also be coded with three digit numbers. Codes used should not induce any bias among testers. For example if samples are coded A and B, testers might feel that sample A is the better sample. A record should be kept of the arrangement of samples presented to each tester.

Number of Samples

In industry large numbers of testers are used and tests are repeated a number of times to ensure validity and reliability. This is not required in the classroom situation.

Setting of Trays

Ensure that a glass of water and / or dry crackers are included on trays in order to cleanse the palate between the tasting of samples.

Tasting and Testing Word Bank

Appearance	Flavour	Smell	Texture	Sound
Appetising	Acidic	Aromatic	Adhesive	Bubbling
Attractive	Bitter	Astringent	Airy	Crackly
Brittle	Bland	Burnt	Brittle	Crunchy
Burnt	Burnt	Coffee	Bubbly	Grating
Cellular	Buttery	Fermented	Chewy	Fizzy
Clear	Creamy	Floral	Coarse	Percolating
Cloudy	Fatty	Fresh	Cohesive	Sizzling
Cold	Herby	Fruity	Cold	Snapping
Colourful	Hot	Musty	Crisp	
Colourless	Musty	Pungent	Crumbly	
Creamy	Piquant	Rancid	Crunchy	
Crumbly	Salty	Roasted	Crystalline	
Dark	Sharp	Smokey	Dry	
Dry	Smokey	Sour	Effervescent	
Foamy	Sour	Spicy	Elastic	
Fresh	Spicy	Stale	Fibrous	
Grained	Stale		Fine	
Greasy	Sweet		Firm	
Healthy	Tangy		Fizzy	
Moist	Tart		Flaky	
Mottled	Tasty		Flat	
Opaque	Tasteless		Foamy	
Pale	Undercooked		Grainy	
Powdery	Watery		Greasy	
Shiny			Gritty	
Slimy			Hard	
Smooth			Juicy	
Soggy			Lumpy	
Sticky			Moist	
Thick			Mushy	
Translucent			Powdery	
Watery			Rubbery	
			Slimy	
			Smooth	
			Soft	
			Spongy	
			Sticky	
			Tender	
			Tough	
			Watery	

Training for Tasting and Testing in Sensory Analysis

Panels of trained testers are used in the food industry to taste and test food products. In order to train students in tasting and testing, particularly in the descriptive and difference tests, it would be useful, as is done in industry, to do a few preliminary tests with them.

The most useful tests would be the taste identification test and taste intensity tests.

Space for Observations and Calculations

Experiment- 14- Sensory Evaluation- Taste Identification Test

Aim: To encourage students to develop an awareness of the basic tastes - sweet, sour, salt and bitter.

Materials required per student

- 4 cups. Code the cups 1, 2, 3 and 4.
- 1 glass of water (to rinse mouth)

Procedure

1. Prepare the cups as follows:

- Cup 1 250ml water + 1 teasp. sugar Sweet
- Cup 2 250ml water + ½ teasp. salt Salt
- Cup 3 250ml water + 2 teasp. lemon juice Sour
- Cup 4 250ml water + 100ml tonic water (decarbonated) Bitter

2. Instruct students to follow instructions on scorecard.

Scorecard		
Taste Identification Test		
Tray number		Name
<p>You are presented with 4 samples of solutions which represent the basic taste sensations of sweet, sour, salt and bitter. Starting in any order, choose a cup, take a sip from it, hold it in your mouth for 10 seconds and note the taste. Proceed through the other samples in a similar manner, rinsing your mouth between each. Fill in the taste identified in each case.</p>		
Solution	Taste Identified	Correct ✓ Incorrect ✕
1		
2		
3		
4		

3. Collect scorecards and correct results.

Templates

Scorecard Taste Identification Test		
Tray number Name		
You are presented with 4 samples of solutions which represent the basic taste sensations of sweet, sour, salt and bitter. Starting in any order, choose a cup, take a sip from it, hold it in your mouth for 10 seconds and note the taste. Proceed through the other samples in a similar manner, rinsing your mouth between each. Fill in the taste identified in each case.		
Solution	Taste Identified	Correct ✓ Incorrect ✗
1		
2		
3		
4		

Scorecard Taste Identification Test		
Tray number Name		
You are presented with 4 samples of solutions which represent the basic taste sensations of sweet, sour, salt and bitter. Starting in any order, choose a cup, take a sip from it, hold it in your mouth for 10 seconds and note the taste. Proceed through the other samples in a similar manner, rinsing your mouth between each. Fill in the taste identified in each case.		
Solution	Taste Identified	Correct ✓ Incorrect ✗
1		
2		
3		
4		

Space for Observations and Calculations

Experiment- 15- Sensory Evaluation- Taste Intensity Tests

Taste intensity tests are used to encourage students to discriminate between concentrations of particular tastes.

Taste Intensity Test

- Testers are presented with three coded samples
- Testers must indicate the order of the samples in terms of intensity of the specified taste

Salt Intensity Test

Aim: To discriminate between the taste intensity of three solutions.

Materials required based on six testers

- 6 trays
- 6 glasses of water
- 18 containers
- 6 scorecards

Procedure

1. Code 18 containers as follows:
 - 6 containers with symbol □
 - 6 containers with symbol ○
 - 6 containers with symbol △
2. Place the following solutions into coded containers:
 - 6 coded □ - 250ml water
 - 6 coded ○ - 250ml water + ½ teasp. salt
 - 6 coded △ - 250ml water + 1 teasp. saltWater can be slightly warm to aid the dissolving of salt.
3. Set up trays numbered 1 - 6. Place one container with symbol □, one with symbol ○ and one with symbol △ on each tray.
4. Instruct testers to follow instructions on scorecard.

Scorecard Salt Intensity Test

Tray number Name

Starting in any order, choose a cup, take a sip from it, hold it in your mouth for at least 10 seconds and note the taste.

Proceed through the other samples in a similar manner, rinsing your mouth between each.

Please indicate the order of the samples in terms of taste intensity i.e. 1 for the weakest solution and 3 for the strongest solution.

□ _____ ○ _____ △ _____

5. Collect and correct results.

Sour and sweet intensity tests can be carried out in a similar manner.

- Sour** 250ml water
250ml water + 1 teasp. lemon juice
250ml water + 1 tablesp. lemon juice

- Sweet** 250ml water
250ml water + 1 teasp. sugar
250ml water + 1 tablesp. sugar

Scorecard
Sour Intensity Test

Tray number Name

Starting in any order, choose a cup, take a sip from it, hold it in your mouth for at least 10 seconds and note the taste.

Proceed through the other samples in a similar manner, rinsing your mouth between each.

Please indicate the order of the samples in terms of taste intensity i.e. 1 for the weakest solution and 3 for the strongest solution.

_____ _____ _____

Scorecard
Sweet Intensity Test

Tray number Name

Starting in any order, choose a cup, take a sip from it, hold it in your mouth for at least 10 seconds and note the taste from it.

Proceed through all three samples in a similar manner, rinsing your mouth out between each.

Please indicate the order of the samples in terms of taste intensity i.e. 1 for the weakest solution and 3 for the strongest solution.

_____ _____ _____

Templates

Scorecard Salt Intensity Test

Tray number Name

Starting in any order, choose a cup, take a sip from it, hold it in your mouth for at least 10 seconds and note the taste.

Proceed through the other samples in a similar manner, rinsing your mouth between each.

Please indicate the order of the samples in terms of taste intensity i.e. 1 for the weakest solution and 3 for the strongest solution.

_____ _____ _____

Scorecard Sour Intensity Test

Tray number Name

Starting in any order, choose a cup, take a sip from it, hold it in your mouth for at least 10 seconds and note the taste.

Proceed through the other samples in a similar manner, rinsing your mouth between each.

Please indicate the order of the samples in terms of taste intensity i.e. 1 for the weakest solution and 3 for the strongest solution.

_____ _____ _____

Scorecard Sweet Intensity Test

Tray number Name

Starting in any order, choose a cup, take a sip from it, hold it in your mouth for at least 10 seconds and note the taste from it.

Proceed through all three samples in a similar manner, rinsing your mouth out between each.

Please indicate the order of the samples in terms of taste intensity i.e. 1 for the weakest solution and 3 for the strongest solution.

_____ _____ _____

Space for Observations and Calculations

Experiment- 16- Sensory Evaluation- Preference Test- Paired Preference Test

Paired Preference Test

A paired preference test is used to express a preference between two products.

Paired Preference Test

- Tester is presented with two coded samples
- Tester decides which one they prefer

Procedure for a Paired Preference Test

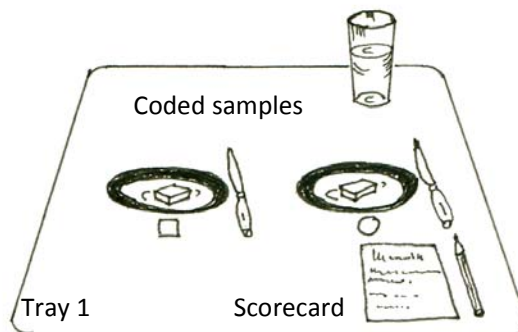
Aim: To determine which of two samples of Shortbread is preferred by testers.

Materials required based on six testers

- 6 trays
- 6 glasses of water
- 12 containers
- 6 samples of food A – Stewart’s Shortbread
- 6 samples of food B – Gordon’s Shortbread
- 6 scorecards
- 6 record sheets

Procedure

1. Code 12 containers as follows:
 - 6 containers with symbol □
 - 6 containers with symbol ○
2. Arrange shortbread in containers:
 - 6 coded □ - Stewart’s Shortbread
 - 6 coded ○ - Gordon’s Shortbread
3. Set up trays numbered 1 – 6. Place one container with symbol □ and one with symbol ○ on each tray.



4. Instruct testers to follow instructions on scorecard.

Scorecard
Paired Preference Test

Tray number Name

In front of you are two coded samples. Taste each sample and tick ✓ the sample that you prefer.

5. Collect scorecards and transfer results onto record sheet.

6. Count results.

7. Reveal codes and present results.

Record Sheet Paired Preference Test		
Food Product	Ticks	Total Number of Ticks
<input type="checkbox"/> Stewart's	✓✓✓✓	4
<input type="radio"/> Gordon's	✓✓	2

In the above record sheet four testers ticked Stewart's Shortbread coded and two testers ticked Gordon's Shortbread coded . Therefore the preferred product was Stewart's Shortbread.

8. Evaluate results.

Templates

Scorecard
Paired Preference Test

Tray number Name

In front of you are two coded samples. Taste each sample and tick ✓ the sample that you prefer.

Scorecard
Paired Preference Test

Tray number Name

In front of you are two coded samples. Taste each sample and tick ✓ the sample that you prefer.

Scorecard
Paired Preference Test

Tray number Name

In front of you are two coded samples. Taste each sample and tick ✓ the sample that you prefer.

Templates

Record Sheet Paired Preference Test		
Food Product	Ticks	Total Number of Ticks
<input type="checkbox"/>		
<input type="radio"/>		

Record Sheet Paired Preference Test		
Food Product	Ticks	Total Number of Ticks
<input type="checkbox"/>		
<input type="radio"/>		

Record Sheet Paired Preference Test		
Food Product	Ticks	Total Number of Ticks
<input type="checkbox"/>		
<input type="radio"/>		

Record Sheet Paired Preference Test		
Food Product	Ticks	Total Number of Ticks
<input type="checkbox"/>		
<input type="radio"/>		

Space for Observations and Calculations

Experiment- 17- Sensory Evaluation- Preference Test- Hedonic Rating Scale

Hedonic Rating Scale

Rating tests can be used to find out how much testers like or dislike a product. The term hedonic means having to do with pleasure so rating scales to do with likes or dislikes are called hedonic rating scales.

Hedonic Rating Scale

- Tester is presented with one or more coded samples
- Tester indicates their degree of liking for each product

Procedure for a Hedonic Rating Scale

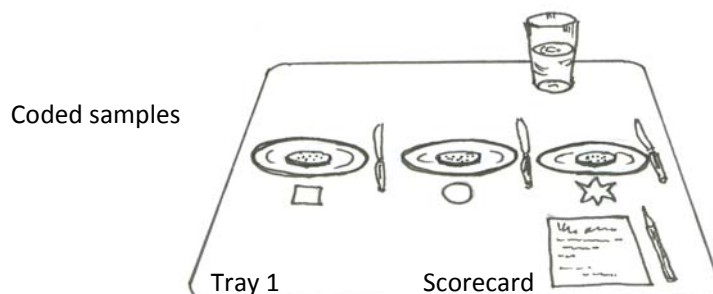
Aim: To determine the extent of liking for each of three brands of Digestive Biscuits.

Materials required based on five testers

- 5 trays
- 5 glasses of water
- 15 containers
- 5 samples food A - White Pack Digestive Biscuits
- 5 samples food B - Red Pack Digestive Biscuits
- 5 samples food C - Blue Pack Digestive Biscuits
- 5 scorecards
- 5 record sheets

Procedure

1. Code 15 food containers as follows:
 - 5 containers with symbol □
 - 5 containers with symbol ○
 - 5 containers with symbol *
2. Arrange biscuits in containers:
 - 5 containers coded □ - White Pack Digestive Biscuits
 - 5 containers coded ○ - Red Pack Digestive Biscuits
 - 5 containers coded * - Blue Pack Digestive Biscuits
3. Set up trays numbered 1 - 5. Place one container with symbol □, one with symbol ○ and one with symbol * on each tray.



4. Instruct testers to follow instructions on scorecard.

Scorecard
Hedonic Rating Scale

Tray number Name

In front of you are three coded samples. Taste each sample and tick ✓ how much you like or dislike it.

□ ○ *

Like a lot _____ _____ _____

Like a little _____ _____ _____

Neither like nor dislike _____ _____ _____

Dislike a little _____ _____ _____

Dislike a lot _____ _____ _____

5. Collect scorecards and transfer results onto record sheet.

6. Calculate results.

To calculate the score for each product assign each descriptor a score value:

like a lot = 5 like a little = 4 neither like nor dislike = 3 dislike a little = 2 dislike a lot = 1.

Work out the average score for each product.

Record Sheet Hedonic Rating Scale							
Food Product □ White Pack Digestive Biscuits							
Food Product ○ Red Pack Digestive Biscuits							
Food Product * Blue Pack Digestive Biscuits							
Score Value Assigned: like a lot = 5 like a little = 4 neither like nor dislike = 3 dislike a little = 2 dislike a lot = 1							
Food Product	Tester					Total Score	Average Score (total score ÷ number of testers)
	1	2	3	4	5		
□	5 pts	5 pts	4 pts	4 pts	5 pts	$\frac{23}{5} = 4.6$	5 points
○	5 pts	2 pts	3 pts	5 pts	3 pts	$\frac{18}{5} = 3.6$	4 points
*	1 pt	1 pt	2pts	3 pts	1 pt	$\frac{8}{5} = 1.6$	2 points

7. Reveal codes and present results.

Product □ - White Pack Digestive Biscuits were liked a lot (5 pts).

Product ○ - Red Pack Digestive Biscuits were liked a little (4 pts).

Product * - Blue Pack Digestive Biscuits were disliked a little (2 pts).

Therefore White Pack Digestive Biscuits was the preferred product.

8. Evaluate results.

Templates

**Scorecard
Hedonic Rating Scale**

Tray number Name

In front of you are three coded samples. Taste each sample and tick ✓ how much you like or dislike it.

□	○	*			
Like a lot			_____	_____	_____
Like a little	_____	_____	_____	_____	_____
Neither like nor dislike			_____	_____	_____
Dislike a little	_____	_____	_____	_____	_____
Dislike a lot	_____	_____	_____	_____	_____

**Scorecard
Hedonic Rating Scale**

Tray number Name

In front of you are three coded samples. Taste each sample and tick ✓ how much you like or dislike it.

□	○	*			
Like a lot			_____	_____	_____
Like a little	_____	_____	_____	_____	_____
Neither like nor dislike			_____	_____	_____
Dislike a little	_____	_____	_____	_____	_____
Dislike a lot	_____	_____	_____	_____	_____

Templates

Record Sheet Hedonic Rating Scale							
Food Product <input type="checkbox"/> _____							
Food Product <input type="radio"/> _____							
Food Product * _____							
Score Value Assigned: like a lot = 5 like a little = 4 neither like nor dislike = 3 dislike a little = 2 dislike a lot = 1							
Food Product	Tester					Total Score	Average Score (total score ÷ number of testers)
	1	2	3	4	5		
<input type="checkbox"/>							
<input type="radio"/>							
*							

Record Sheet Hedonic Rating Scale							
Food Product <input type="checkbox"/> _____							
Food Product <input type="radio"/> _____							
Food Product * _____							
Score Value Assigned: like a lot = 5 like a little = 4 neither like nor dislike = 3 dislike a little = 2 dislike a lot = 1							
Food Product	Tester					Total Score	Average Score (total score ÷ number of testers)
	1	2	3	4	5		
<input type="checkbox"/>							
<input type="radio"/>							
*							

Space for Observations and Calculations

Experiment- 18- Sensory Evaluation- Preference Test- Food Action / Attitude Rating Test

Food Action / Attitude Rating Test

In a food action rating test a scale is used to determine the attitudes of testers to a food. It is often referred to as a "FACT Scale". The test can be carried out on one or more samples of food.

Food Action Rating Test

- Tester is presented with one or more food samples
- Tester indicates their attitude to the food on prepared scales

Procedure for a Food Action Rating Test

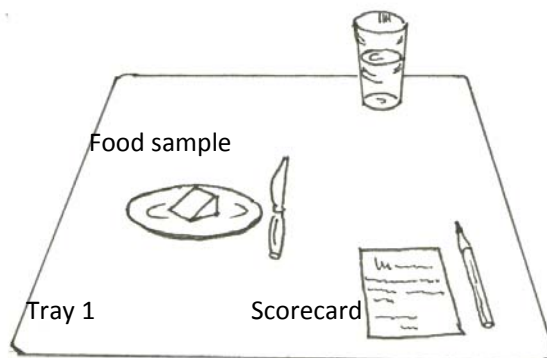
Aim: To determine the attitude of testers to one type of Cheddar Cheese.

Materials required based on six testers

- 6 trays
- 6 glasses of water
- 6 containers
- 6 samples of Cheddar Cheese
- 6 scorecards
- 6 record sheets

Procedure

1. Place the cheese samples in six containers.
2. Set up trays numbered 1 - 6. Place one container on each tray.



3. Instruct testers to follow instructions on scorecard.

**Scorecard
 Food Action Rating Test**

Tray number Name

You are presented with a food sample.
 Please taste the sample and tick ✓ the box that best describes how you feel about it.

- I would eat this every opportunity that I had
- I would eat this very often
- I like this and would eat it now and then
- I would eat this if available but would not go out of my way
- I don't like this but would eat it on occasion
- I would hardly ever eat this
- I would eat this only if forced to

4. Collect scorecards and transfer results onto record sheet.

Record Sheet Food Action Rating Test	
Action	Total Ticks
I would eat this every opportunity that I had	
I would eat this very often	✓ ✓
I like this and would eat it now and then	✓ ✓ ✓
I would eat this if available but would not go out of my way	✓
I don't like this but would eat it on occasion	
I would hardly ever eat this	
I would eat this only if forced to	

5. Count results.

6. Present results.

In this case, three testers would eat this food now and then; two would eat it very often and one would eat it if available but would not go out of their way to eat it.

7. Evaluate results.

Templates

Scorecard Food Action Rating Test

Tray number Name

You are presented with a food sample.
Please taste the sample and tick ✓ the box that best describes how you feel about it.

- I would eat this every opportunity that I had
- I would eat this very often
- I like this and would eat it now and then
- I would eat this if available but would not go out of my way
- I don't like this but would eat it on occasion
- I would hardly ever eat this
- I would eat this only if forced to

Scorecard Food Action Rating Test

Tray number Name

You are presented with a food sample.
Please taste the sample and tick ✓ the box that best describes how you feel about it.

- I would eat this every opportunity that I had
- I would eat this very often
- I like this and would eat it now and then
- I would eat this if available but would not go out of my way
- I don't like this but would eat it on occasion
- I would hardly ever eat this
- I would eat this only if forced to

Templates

Record Sheet Food Action Rating Test	
Action	Total Ticks
I would eat this every opportunity that I had	
I would eat this very often	
I like this and would eat it now and then	
I would eat this if available but would not go out of my way	
I don't like this but would eat it on occasion	
I would hardly ever eat this	
I would eat this only if forced to	

Record Sheet Food Action Rating Test	
Action	Total Ticks
I would eat this every opportunity that I had	
I would eat this very often	
I like this and would eat it now and then	
I would eat this if available but would not go out of my way	
I don't like this but would eat it on occasion	
I would hardly ever eat this	
I would eat this only if forced to	

Space for Observations and Calculations

Experiment- 19- Sensory Evaluation- Preference Test- Preference Ranking Test

Preference Ranking Test

Preference ranking tests are used to rank foods in order of preference. They are used when two or more samples are being tested. The number of samples used is dependent on the tester's attention span and memory. The tester is asked to assign an order to the samples according to his / her preference. Ranking tests do not determine the degree of liking / disliking for each of the samples.

Preference Ranking Test

- Tester is presented with a number of coded samples
- Tester ranks samples in order of preference

Procedure for a Preference Ranking Test

Aim: To determine which of three different brands of Chocolate Yoghurt is preferred by testers.

Materials required based on six testers

- 6 trays
- 6 glasses of water
- 18 containers
- 6 samples of food A – Yellow Pack Chocolate Yoghurt
- 6 samples of food B – Red Pack Chocolate Yoghurt
- 6 samples of food C – Blue Pack Chocolate Yoghurt
- 6 scorecards
- 6 record sheets

Procedure

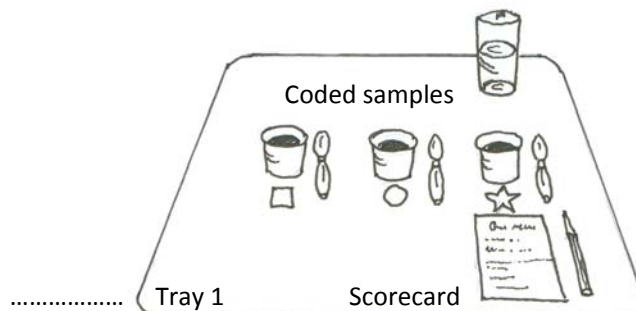
1. Code 18 containers as follows:

- 6 coded □ - Yellow Pack Chocolate Yoghurt
- 6 coded ○ - Red Pack Chocolate Yoghurt
- 6 coded * - Blue Pack Chocolate Yoghurt

2. Arrange yoghurt in containers:

- 6 coded □ - Yellow Pack Chocolate Yoghurt
- 6 coded ○ - Red Pack Chocolate Yoghurt
- 6 coded * - Blue Pack Chocolate Yoghurt

3. Set up trays numbered 1 - 6. Place one container with symbol □, one with symbol ○ and one with symbol * on each tray.



4. Instruct testers to follow instructions on scorecard.

Scorecard	
Preference Ranking Test	
Tray number	Name
In front of you are three coded samples. Taste each sample.	
Please indicate your preference by placing:	
1 st choice beside the sample that you prefer most	
2 nd choice beside your next preference	
3 rd choice beside the one you least prefer.	
□ _____	○ _____ * _____

5. Collect scorecards and transfer results onto record sheet.

6. Calculate results.

To calculate the results assign each choice a score value:

- 1st choice give 3 points
- 2nd choice give 2 points
- 3rd choice give 1 point

Calculate the score for each product by multiplying the number of ticks in each box by the score value assigned to that choice as in the record sheet below. The order of preference is determined from the score i.e. the product with the highest score is the preferred product.

Record Sheet					
Preference Ranking Test					
Food Product	□	Yellow Pack Chocolate Yoghurt			
Food Product	○	Red Pack Chocolate Yoghurt			
Food Product	*	Blue Pack Chocolate Yoghurt			
For each tester place a tick ✓ in the box that corresponds to their choice for that product.					
Score Value Assigned:					
1 st choice give 3 points					
2 nd choice give 2 points					
3 rd choice give 1 point					
Food Product	1 st choice	2 nd choice	3 rd choice	Score	Rank Order

□	✓✓ 2x3=6	✓✓ 2x2=4	✓✓ 2x1=2	12 points	2 nd
○	✓✓ 2x3=6	✓✓✓ 3x2=6	✓ 1x1=1	13 points	1 st
*	✓✓ 2x3=6	✓ 1x2=2	✓✓✓ 3x1=3	11 points	3 rd

7. Reveal codes and present results.

In the above case Red Pack Chocolate Yoghurt was the group preference, followed by Yellow Pack. Blue Pack Chocolate Yoghurt was the least preferred of the three samples.

8. Evaluate results.

Templates

Scorecard
Preference Ranking Test

Tray number Name

In front of you are three coded samples. Taste each sample.

Please indicate your preference by placing:
1st choice beside the sample that you prefer most
2nd choice beside your next preference
3rd choice beside the one you least prefer.

_____ _____ * _____

Scorecard
Preference Ranking Test

Tray number Name

In front of you are three coded samples. Taste each sample.

Please indicate your preference by placing:
1st choice beside the sample that you prefer most
2nd choice beside your next preference
3rd choice beside the one you least prefer.

_____ _____ * _____

Templates

Record Sheet Preference Ranking Test					
Food Product <input type="checkbox"/> _____ Food Product <input type="radio"/> _____ Food Product * _____					
For each tester place a tick ✓ in the box that corresponds to their choice for that product. Score Value Assigned: 1 st choice give 3 points 2 nd choice give 2 points 3 rd choice give 1 point					
Food Product	1 st choice	2 nd choice	3 rd choice	Score	Rank Order
<input type="checkbox"/>					
<input type="radio"/>					
*					

Record Sheet Preference Ranking Test					
Food Product <input type="checkbox"/> _____ Food Product <input type="radio"/> _____ Food Product * _____					
For each tester place a tick ✓ in the box that corresponds to their choice for that product. Score Value Assigned: 1 st choice give 3 points 2 nd choice give 2 points 3 rd choice give 1 point					
Food Product	1 st choice	2 nd choice	3 rd choice	Score	Rank Order
<input type="checkbox"/>					
<input type="radio"/>					
*					

Space for Observations and Calculations

Experiment- 20- Sensory Evaluation- Difference Test- Paired Comparison Test

Paired Comparison Test

This test is useful when comparing two types of the same food e.g. baked beans, yoghurt, juice etc. There are two different types of paired comparison test:

- Simple difference paired comparison test - are the samples different?
- Directional paired comparison test - which sample is sweeter / saltier?

Simple Difference Paired Comparison Test

Simple Difference Paired Comparison Test

- Tester is presented with two coded samples
- Tester is asked if there is a difference between the samples

Procedure for a Simple Difference Paired Comparison Test

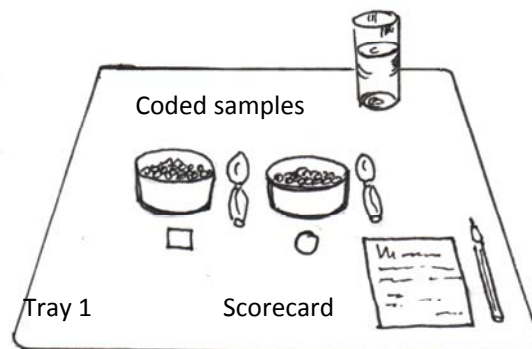
Aim: To determine if testers can detect a difference between two samples of Baked Beans.

Materials required based on six testers

- 6 trays
- 6 glasses of water
- 12 containers
- 6 samples of food A – Baked Beans
- 6 samples of food B – Reduced Sugar Baked Beans
- 6 scorecards
- 6 record sheets

Procedure

1. Code 12 containers as follows:
 - 6 containers with symbol □
 - 6 containers with symbol ○
2. Set up trays numbered 1 – 6. Place one container with symbol □ and one container with symbol ○ on each tray.



3. Arrange beans in containers. It is important to present the coded samples in random order on each tray. The samples on the trays can be the same or different. A possible presentation order for six testers is illustrated below.

Presentation Order for Six Trays

Tray	AB
1	<input type="checkbox"/> <input type="radio"/>

Tray	BA
2	<input type="checkbox"/> <input type="radio"/>

Tray	AB
3	<input type="checkbox"/> <input type="radio"/>

Tray	BA
4	<input type="checkbox"/> <input type="radio"/>

Tray	AB
5	<input type="checkbox"/> <input type="radio"/>

Tray	BA
6	<input type="checkbox"/> <input type="radio"/>

- A - Baked Beans
- B - Reduced Sugar Baked Beans

4. Instruct testers to follow instructions on scorecard.

Scorecard	
Simple Difference Paired Comparison Test	
Tray number	Name
You are presented with two coded samples. Please taste the samples in the order given. Can you detect a difference between the samples?	
Yes _____	No _____

Note: the taste order is always specified on the scorecard for a simple difference paired comparison test to ensure random tasting of food.

5. Collect scorecards and transfer results onto record sheet.

Record Sheet				
Simple Difference Paired Comparison Test				
Food Product A: Baked Beans				
Food Product B: Reduced Sugar Baked Beans				
When recording results, transfer responses from the scorecards by indicating whether testers answered Yes or No. Tick ✓ those that are correct.				
			Response	✓ If Correct
Tester 1				
Food product	A	B	Yes	✓
Code	<input type="checkbox"/>	<input type="radio"/>		
Tester 2				

Food product	B	A	No	
Code	<input type="checkbox"/>	<input type="radio"/>		
Tester 3				
Food product	A	B	Yes	✓
Code	<input type="checkbox"/>	<input type="radio"/>		
Tester 4				
Food product	B	A	Yes	✓
Code	<input type="checkbox"/>	<input type="radio"/>		
Tester 5				
Food sample	A	B	Yes	✓
Code	<input type="checkbox"/>	<input type="radio"/>		
Tester 6				
Food product	B	A	No	
Code	<input type="checkbox"/>	<input type="radio"/>		
Total number of correct responses				4

6. Count the correct responses.

7. Reveal codes and present results.

As you can see, four testers correctly detected a difference between the two samples.

8. Evaluate results.

Templates

Scorecard
Simple Difference Paired Comparison Test

Tray number Name

You are presented with two coded samples. Please taste the samples in the order given.
Can you detect a difference between the samples?

Yes _____ No _____

Scorecard
Simple Difference Paired Comparison Test

Tray number Name

You are presented with two coded samples. Please taste the samples in the order given.
Can you detect a difference between the samples?

Yes _____ No _____

Scorecard
Simple Difference Paired Comparison Test

Tray number Name

You are presented with two coded samples. Please taste the samples in the order given.
Can you detect a difference between the samples?

Yes _____ No _____

Templates

Record Sheet				
Simple Difference Paired Comparison Test				
Food Product A: _____				
Food Product B: _____				
When recording results, transfer responses from the scorecards by indicating whether testers answered Yes or No. Tick ✓ those that are correct.				
			Response	✓ If Correct
Tester 1				
Food product	A	B		
Code	<input type="checkbox"/>	<input type="radio"/>		
Tester 2				
Food product	B	A		
Code	<input type="checkbox"/>	<input type="radio"/>		
Tester 3				
Food product	A	B		
Code	<input type="checkbox"/>	<input type="radio"/>		
Tester 4				
Food product	B	A		
Code	<input type="checkbox"/>	<input type="radio"/>		
Tester 5				
Food sample	A	B		
Code	<input type="checkbox"/>	<input type="radio"/>		
Tester 6				
Food product	B	A		
Code	<input type="checkbox"/>	<input type="radio"/>		
Total number of correct responses				

Experiment- 21- Sensory Evaluation- Difference Test

Paired Comparison Test

This test is useful when comparing two types of the same food e.g. baked beans, yoghurt, juice etc. There are two different types of paired comparison test:

- Simple difference paired comparison test - are the samples different?
- Directional paired comparison test - which sample is sweeter / saltier?

Directional Paired Comparison Test

Directional Paired Comparison Test

- Tester is present with two coded samples
- Tester is asked to determine which of the samples has a greater degree of intensity in terms of a particular characteristic

Procedure for a Directional Paired Comparison Test

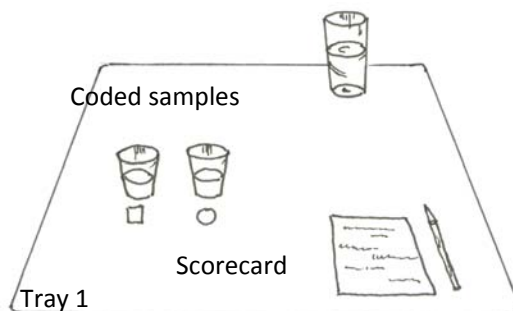
Aim: To determine which of two samples of Orange Juice is sweeter.

Materials required based on six testers

- 6 trays
- 6 glasses of water
- 12 containers
- 6 samples of food A – Unsweetened Orange Juice
- 6 samples of food B – Sweetened Orange Juice
- 6 scorecards
- 6 record sheets

Procedure

1. Code 12 containers as follows:
 - 6 containers with symbol □
 - 6 containers with symbol ○
2. Set up trays numbered 1 – 6. Place one container with symbol □ and one with symbol ○ on each tray.



3. Arrange orange juice in containers. It is important to present the coded samples in random order on each tray. A possible presentation order for six testers is illustrated below.

Presentation Order for Six Trays

Tray	AB
1	<input type="checkbox"/> <input type="radio"/>

Tray	BA
2	<input type="checkbox"/> <input type="radio"/>

Tray	AB
3	<input type="checkbox"/> <input type="radio"/>

Tray	BA
4	<input type="checkbox"/> <input type="radio"/>

Tray	AB
5	<input type="checkbox"/> <input type="radio"/>

Tray	BA
6	<input type="checkbox"/> <input type="radio"/>

A - Unsweetened Orange Juice
 B - Sweetened Orange Juice

4. Instruct testers to follow instructions on scorecard.

Scorecard	
Directional Paired Comparison Test	
Tray number	Name
In front of you are two coded samples. Starting with the sample on the left, taste each sample and circle the sample that is sweeter. You must make a choice. You may re-taste as often as you wish.	
<input type="checkbox"/>	<input type="radio"/>

Note: the taste order is always specified on the scorecard for a directional paired comparison test to ensure random tasting of food.

5. Collect scorecards and transfer results onto record sheet.

Record Sheet			
Directional Paired Comparison Test			
Food Product A: Unsweetened Orange Juice			
Food Product B: Sweetened Orange Juice			
When recording results, circle the letter that corresponds with the symbol selected on the scorecard. Tick ✓ the correct responses.			
			✓ If Correct
Tester 1			
Food product	A <input checked="" type="radio"/>	B	
Code	<input type="checkbox"/>	<input type="radio"/>	

Tester 2			
Food product	B <input type="radio"/>	A <input type="radio"/>	✓
Code	<input type="checkbox"/>	<input type="checkbox"/>	
Tester 3			
Food product	A <input type="radio"/>	B <input type="radio"/>	✓
Code	<input type="checkbox"/>	<input type="checkbox"/>	
Tester 4			
Food product	B <input type="radio"/>	A <input type="radio"/>	
Code	<input type="checkbox"/>	<input type="checkbox"/>	
Tester 5			
Food product	A <input type="radio"/>	B <input type="radio"/>	✓
Code	<input type="checkbox"/>	<input type="checkbox"/>	
Tester 6			
Food product	B <input type="radio"/>	A <input type="radio"/>	✓
Code	<input type="checkbox"/>	<input type="checkbox"/>	
Total number of correct responses			4

6. Count correct responses.

7. Reveal codes and present results.

Four people correctly identified the Sweetened Orange Juice as being the sweeter sample.

8. Evaluate results.

Templates

Scorecard
Directional Paired Comparison Test

Tray number Name

In front of you are two coded samples.
Starting with the sample on the left, taste each sample and circle the sample that is _____
You must make a choice. You may re-taste as often as you wish.

Scorecard
Directional Paired Comparison Test

Tray number Name

In front of you are two coded samples.
Starting with the sample on the left, taste each sample and circle the sample that is _____
You must make a choice. You may re-taste as often as you wish.

Scorecard
Directional Paired Comparison Test

Tray number Name

In front of you are two coded samples.
Starting with the sample on the left, taste each sample and circle the sample that is _____
You must make a choice. You may re-taste as often as you wish.

Template

Record Sheet			
Directional Paired Comparison Test			
Food Product A: _____			
Food Product B: _____			
When recording results, circle the letter that corresponds with the symbol selected on the scorecard. Tick ✓ the correct responses.			
			✓ If Correct
Tester 1			
Food product	A	B	
Code	<input type="checkbox"/>	<input type="radio"/>	
Tester 2			
Food product	B	A	
Code	<input type="checkbox"/>	<input type="radio"/>	
Tester 3			
Food product	A	B	
Code	<input type="checkbox"/>	<input type="radio"/>	
Tester 4			
Food product	B	A	
Code	<input type="checkbox"/>	<input type="radio"/>	
Tester 5			
Food product	A	B	
Code	<input type="checkbox"/>	<input type="radio"/>	
Tester 6			
Food product	B	A	
Code	<input type="checkbox"/>	<input type="radio"/>	
Total number of correct responses			

Space for Observations and Calculations

Experiment- 21- Sensory Evaluation- Difference Test- Triangle Test

Triangle Test

The triangle test is used to see if there is a detectable difference between two similar products.

Triangle Test

- Tester is presented with three coded samples
- Two samples are the same, one is different
- Tester is asked to identify the sample that is different

Procedure for a Triangle Test

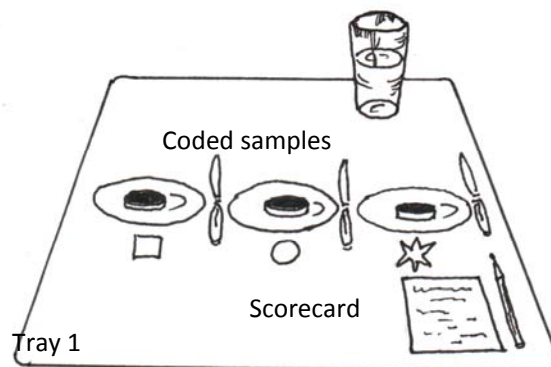
Aim: To find out if there is a detectable difference between two brands of Jaffa Cakes.

Materials required based on six testers

- 6 trays
- 6 glasses of water
- 18 containers
- 9 samples of food A - White Pack Jaffa Cakes
- 9 samples of food B - Blue Pack Jaffa Cakes
- 6 scorecards
- 6 record sheets

Procedure

1. Code 18 containers as follows:
 - 6 containers with symbol □
 - 6 containers with symbol ○
 - 6 containers with symbol *
2. Set up trays numbered 1 - 6. Place one container with symbol □, one with symbol ○ and one with symbol * on each tray.



3. Arrange the food samples in each container. In a triangle test “Balanced Presentation Order” is important. This means that:
 - (i) every possible combination of samples should be presented

(ii) each food being tested is offered an equal number of times.
 In a triangle test there are six possible combinations that can be presented. The six combinations are illustrated below.

Balanced Presentation Order for Six Trays

Tray	ABA
1	□○*

Tray	AAB
2	□○*

Tray	BBA
3	□○*

Tray	BAB
4	□○*

Tray	ABB
5	□○*

Tray	BAA
6	□○*

A - White Pack Jaffa Cakes
 B - Blue Pack Jaffa Cakes

On tray 1:
 food container □ contains White Pack Jaffa Cakes
 food container ○ contains Blue Pack Jaffa Cakes
 food container * contains White Pack Jaffa Cakes.

By setting up six trays one can ensure that every possible combination of samples is offered. The samples are also presented in **random order** and no tester gets the samples presented in the same sequence. Each food sample is offered an equal number of times i.e. nine times, so a **balanced presentation order** is achieved. It is important to note that the **codes on each tray remain the same**; it is the **food in the container that changes** each time.

4. Instruct testers to follow instructions on scorecard.

Scorecard	
Triangle Test	
Tray number	Name
In front of you are three coded samples, two are the same and one is different. Starting from the left, taste the samples and circle the one that is different from the other two. You may re-taste the samples. You must make a choice.	
□	○ *

Note: the taste order is always specified on the scorecard for a triangle test to ensure random tasting of foods.

5. Collect scorecards and transfer results onto the record sheet.

**Record Sheet
Triangle Test**

Food Product A: White Pack Jaffa Cakes

Food Product B: Blue Pack Jaffa Cakes

When recording the results circle the letter that corresponds with the symbol selected on each scorecard.

Tick ✓ the appropriate column if the tester correctly identified the sample that was different.

				✓	If
Tester 1					
Food product	A	B <input type="radio"/>	A	✓	
Code	<input type="checkbox"/>	○	*		
Tester 2					
Food product	A	A	B <input type="radio"/>	✓	
Code	<input type="checkbox"/>	○	*		
Tester 3					
Food product	B	B <input type="radio"/>	A		
Code	<input type="checkbox"/>	○	*		
Tester 4					
Food product	B	A <input type="radio"/>	B	✓	
Code	<input type="checkbox"/>	○	*		
Tester 5					
Food product	A <input type="radio"/>	B	B	✓	
Code	<input type="checkbox"/>	○	*		
Tester 6					
Food product	B <input type="radio"/>	A	A	✓	
Code	<input type="checkbox"/>	○	*		
Total number of correct responses				5	

6. Count correct responses.

7. Reveal codes and present results.

In this case five out of six people correctly identified the sample that was different.

8. Evaluate results.

Templates

**Scorecard
Triangle Test**

Tray number Name

In front of you are three coded samples, two are the same and one is different.
Starting from the left, taste the samples and circle the one that is different from the other two.
You may re-taste the samples. You must make a choice.

 *

**Scorecard
Triangle Test**

Tray number Name

In front of you are three coded samples, two are the same and one is different.
Starting from the left, taste the samples and circle the one that is different from the other two.
You may re-taste the samples. You must make a choice.

 *

**Scorecard
Triangle Test**

Tray number Name

In front of you are three coded samples, two are the same and one is different.
Starting from the left, taste the samples and circle the one that is different from the other two.
You may re-taste the samples. You must make a choice.

 *

Template

Record Sheet Triangle Test					
Food Product A: _____					
Food Product B: _____					
When recording the results circle the letter that corresponds with the symbol selected on each scorecard. Tick ✓ the appropriate column if the tester correctly identified the sample that was different.					
				✓	If
Tester 1					
Food product	A	B	A		
Code	<input type="checkbox"/>	<input type="radio"/>	*		
Tester 2					
Food product	A	A	B		
Code	<input type="checkbox"/>	<input type="radio"/>	*		
Tester 3					
Food product	B	B	A		
Code	<input type="checkbox"/>	<input type="radio"/>	*		
Tester 4					
Food product	B	A	B		
Code	<input type="checkbox"/>	<input type="radio"/>	*		
Tester 5					
Food product	A	B	B		
Code	<input type="checkbox"/>	<input type="radio"/>	*		
Tester 6					
Food product	B	A	A		
Code	<input type="checkbox"/>	<input type="radio"/>	*		
Total number of correct responses					

Space for Observations and Calculations

Experiment- 22- Sensory Evaluation- Difference Test- Duo Trio Test

Duo-Trio Test

The duo-trio test is an alternative to the triangle test. This test is used in the food industry when changes are contemplated in a product currently available. This test is particularly useful when the product concerned has an intense odour or taste.

Duo-Trio Test

- Tester is presented with three samples
- Two samples are coded and one is identified as the reference. In industry the reference is normally the product currently being manufactured
- Tester is asked to identify the sample that is different from the reference

Procedure for a Duo-Trio Test

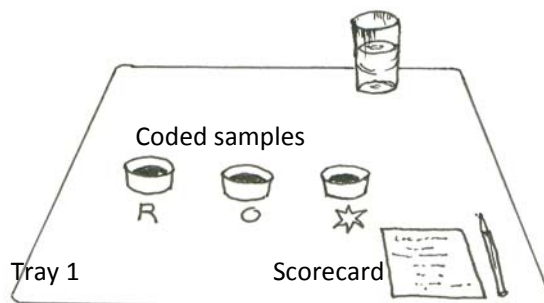
Aim: To find out if there is a detectable difference in taste between an Original Garlic Dip recipe and a Modified Garlic Dip recipe.

Materials required based on six testers

- 6 trays
- 6 glasses of water
- 18 containers
- 12 samples of food A – Original Garlic Dip recipe
- 6 samples of food B – Modified Garlic Dip recipe
- 6 scorecards
- 6 record sheets

Procedure

1. Code 18 containers as follows:
 - 6 containers with symbol R
 - 6 containers with symbol ○
 - 6 containers with symbol *
2. Set up trays numbered 1 - 6. Place one container with symbol R, one with symbol ○ and one with symbol * on each tray.



3. Arrange the food in the containers on each tray. It is important to present the food in a random order

on each tray. A possible presentation order for six testers is illustrated below.

Presentation Order for Six Trays

Tray	AAB
1	R○*

Tray	ABA
2	R○*

Tray	AAB
3	R○*

Tray	ABA
4	R○*

Tray	AAB
5	R○*

Tray	ABA
6	R○*

- R - Original recipe
- A - Original recipe
- B - Modified recipe

The codes on each tray remain the same. It is the food in the container that changes each time. The food placed in the container coded R is always the reference food, in this case the Original Garlic Dip recipe. Only the foods in the containers coded ○ and * change.

4. Instruct testers to follow instructions on scorecard.

Scorecard		
Duo-Trio Test		
Tray number	Name	
<p>You are presented with three samples, one marked R and two other coded samples. Starting from the left, taste the R sample followed by the two coded samples in the order given. Circle the sample that is different from R. You may retaste the samples. You must make a choice.</p>		
R	○	*

5. Collect scorecards and transfer results onto record sheet.

Record Sheet				
Duo-Trio Test				
Food Product A: Original Garlic Dip				
Food Product B: Modified Garlic Dip				
When recording the results circle the letter that corresponds with the symbol selected on each scorecard.				
Tick ✓ the appropriate column if the tester correctly identified the sample that was <i>different</i> from R.				
				✓ If Correct
Tester 1				
Food product	A	A	B	✓
Code	R	○	*	
Tester 2				
Food product	A	B	A	✓
Code	R	○	*	
Tester 3				
Food product	A	A	B	
Code	R	○	*	
Tester 4				
Food product	A	B	A	✓
Code	R	○	*	
Tester 5				
Food product	A	A	B	✓
Code	R	○	*	
Tester 6				
Food product	A	B	A	
Code	R	○	*	
Total number of correct responses				4

6. Count correct responses.

7. Reveal codes and present results.

In this case four testers could identify the sample that was different from the reference.

8. Evaluate results.

Templates

Scorecard
Duo-Trio Test

Tray number Name

You are presented with three samples, one marked R and two other coded samples.
Starting from the left, taste the R sample followed by the two coded samples in the order given.
Circle the sample that is **different** from R. You may retaste the samples. You must make a choice.

R ○ *

Scorecard
Duo-Trio Test

Tray number Name

You are presented with three samples, one marked R and two other coded samples.
Starting from the left, taste the R sample followed by the two coded samples in the order given.
Circle the sample that is **different** from R. You may retaste the samples. You must make a choice.

R ○ *

Scorecard
Duo-Trio Test

Tray number Name

You are presented with three samples, one marked R and two other coded samples.
Starting from the left, taste the R sample followed by the two coded samples in the order given.
Circle the sample that is **different** from R. You may retaste the samples. You must make a choice.

R ○ *

Template

Record Sheet Duo-Trio Test				
Food Product A: _____				
Food Product B: _____				
When recording the results circle the letter that corresponds with the symbol selected on each scorecard. Tick ✓ the appropriate column if the tester correctly identified the sample that was <i>different</i> from R.				
				✓ If Correct
Tester 1				
Food product	A	A	B	
Code	R	○	*	
Tester 2				
Food product	A	B	A	
Code	R	○	*	
Tester 3				
Food product	A	A	B	
Code	R	○	*	
Tester 4				
Food product	A	B	A	
Code	R	○	*	
Tester 5				
Food product	A	A	B	
Code	R	○	*	
Tester 6				
Food product	A	B	A	
Code	R	○	*	
Total number of correct responses				

Space for Observations and Calculations

Experiment- 23- Sensory Evaluation- Descriptive Test- Descriptive Ranking Test

Descriptive Ranking Test

A descriptive ranking test is used to rank foods in order of intensity of a specific sensory attribute. A sensory attribute is the term used to describe a key characteristic of a food product e.g. sweetness, saltiness, aroma / flavour, rancidity, viscosity.

Descriptive Ranking Test

- Tester is presented with a number of coded samples
- Tester ranks samples in order of intensity of specified attribute/s

Procedure for a Descriptive Ranking Test

Aim: To rank the perceived creaminess of three types of Milk.

Materials required based on six testers

6 trays
6 glasses of water
18 containers
6 samples of food A - Full Fat Milk
6 samples of food B - Low Fat Milk
6 samples of food C - Skimmed Milk
6 scorecards
6 record sheets

Procedure

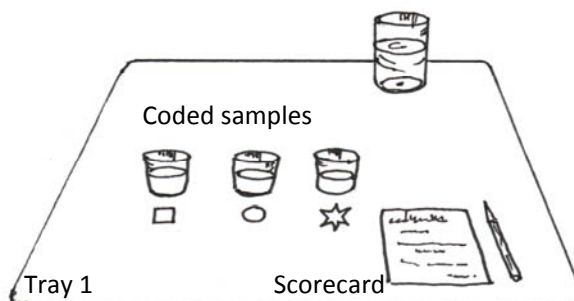
1. Code 18 containers as follows:

- 6 containers with symbol □
- 6 containers with symbol ○
- 6 containers with symbol *

2. Arrange milk in containers:

- 6 coded □ - Full Fat Milk
- 6 coded ○ - Low Fat Milk
- 6 coded * - Skimmed Milk

3. Set up trays numbered 1 - 6. Place one container with symbol □, one with symbol ○ and one with symbol * on each tray.



4. Instruct the testers to follow instructions on scorecard.

Scorecard
Descriptive Ranking Test

Tray number Name

In front of you are three coded samples. Taste each sample.
 Please rank the samples in order of creaminess by placing:
 1st choice beside the sample that you consider to be the creamiest
 2nd choice beside the next creamiest
 3rd choice beside the least creamy.

_____ _____ * _____

5. Collect scorecards and transfer results onto record sheet.

6. Calculate results.

To calculate the results assign each choice a score value:

1st choice give 3 points

2nd choice give 2 points

3rd choice give 1 point.

Calculate the score for each product by multiplying the number of ticks in each box by the value assigned to that choice as in the record sheet below. The rank order is determined from the score.

Record Sheet Descriptive Ranking Test					
Food Product <input type="checkbox"/> Full Fat Milk					
Food Product <input type="radio"/> Low Fat Milk					
Food Product * Skimmed milk					
For each tester place a tick ✓ in the box that corresponds to their choice for that product.					
Score Value Assigned:					
1 st choice give 3 points					
2 nd choice give 2 points					
3 rd choice give 1 point					
Food Product	1 st choice	2 nd choice	3 rd choice	Score	Rank Order
<input type="checkbox"/>	✓✓ 2x3=6	✓✓✓ 3x2=6	✓ 1x1=1	13 points	1 st
<input type="radio"/>	✓✓ 2x3=6	✓✓ 2x2=4	✓✓ 2x1=2	12 points	2 nd
*	✓✓ 2x3=6	✓ 1x2=2	✓✓✓ 3x1=3	11 points	3 rd

7. Reveal codes and present results.

In the above example the product coded was perceived to be the creamiest milk. This was the Full Fat Milk.

8. Evaluate results.

Templates

Scorecard
Descriptive Ranking Test

Tray number Name

In front of you are three coded samples. Taste each sample.
Please rank the samples in order of _____ by placing:
1st choice beside the sample that you consider to be the _____
2nd choice beside the next _____
3rd choice beside the least _____

_____ _____ * _____

Scorecard
Descriptive Ranking Test

Tray number Name

In front of you are three coded samples. Taste each sample.
Please rank the samples in order of _____ by placing:
1st choice beside the sample that you consider to be the _____
2nd choice beside the next _____
3rd choice beside the least _____

_____ _____ * _____

Scorecard
Descriptive Ranking Test

Tray number Name

In front of you are three coded samples. Taste each sample.
Please rank the samples in order of _____ by placing:
1st choice beside the sample that you consider to be the _____
2nd choice beside the next _____
3rd choice beside the least _____

_____ _____ * _____

Templates

Record Sheet					
Descriptive Ranking Test					
Food Product <input type="checkbox"/> _____					
Food Product <input type="radio"/> _____					
Food Product * _____					
For each tester place a tick ✓ in the box that corresponds to their choice for that product. Score Value Assigned: 1 st choice give 3 points 2 nd choice give 2 points 3 rd choice give 1 point					
Food Product	1 st choice	2 nd choice	3 rd choice	Score	Rank Order
<input type="checkbox"/>					
<input type="radio"/>					
*					

Record Sheet					
Descriptive Ranking Test					
Food Product <input type="checkbox"/> _____					
Food Product <input type="radio"/> _____					
Food Product * _____					
For each tester place a tick ✓ in the box that corresponds to their choice for that product. Score Value Assigned: 1 st choice give 3 points 2 nd choice give 2 points 3 rd choice give 1 point					
Food Product	1 st choice	2 nd choice	3 rd choice	Score	Rank Order
<input type="checkbox"/>					
<input type="radio"/>					
*					

Space for Observations and Calculations

Experiment- 24- Sensory Evaluation- Descriptive Test- Descriptive Rating Tests- Line Scales

Descriptive Rating Tests

Descriptive rating tests are used to evaluate and rate pre-selected sensory attributes in a food. A sensory attribute is the term used to describe a key characteristic of a food product.

The attributes can be rated on **line scales** or **star diagrams**.

A sensory profile is a written description of the sensory attributes of a food. This is compiled from the ratings obtained for the selected attributes.

Descriptive Rating Test – profiling one product using line scales.

Descriptive Rating Test

- Tester is presented with one food sample
- Tester is asked to rate the intensity of the pre-selected attributes for the sample

Procedure for a Descriptive Rating Test

Aim: To compile a sensory profile of one type of Tomato Soup using four pre-selected attributes.

Materials required based on six testers

- 6 trays
- 6 glasses of water
- 6 containers
- 6 samples of Tomato Soup
- 6 scorecards
- 6 record sheets

Procedure

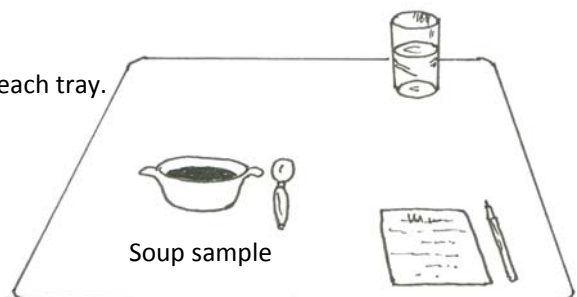
1. Agree four attributes to be rated. This is an important stage in the test and can be done by brainstorming / discussion within the class group. Each student should have a clear understanding of the meaning of each chosen attribute.

The four attributes agreed for the tomato soup are:

- aroma
- colour (tomato colour)
- flavour (tomato flavour)
- sweetness (sweet).

2. Label scorecard and record sheet with agreed attributes.

3. Set up trays numbered 1 – 6. Place a sample of soup on each tray.



Scorecard

Tray 1

4. Instruct testers to follow instructions on scorecard.

Scorecard
Descriptive Rating Test - one product

Tray number Name

You are presented with a sample of Tomato Soup.
Please evaluate and rate the sample for each attribute and mark the number that best describes your choice on the accompanying line scale.

1 = very weak 2 = weak 3 = neither weak nor strong 4 = strong 5 = very strong

Attributes

Aroma

1 2 3 4 5

Tomato Flavour

1 2 3 4 5

Tomato Colour

1 2 3 4 5

Sweetness

1 2 3 4 5

5. Collect scorecards and transfer results onto record sheet.

6. Calculate the average score for each attribute.

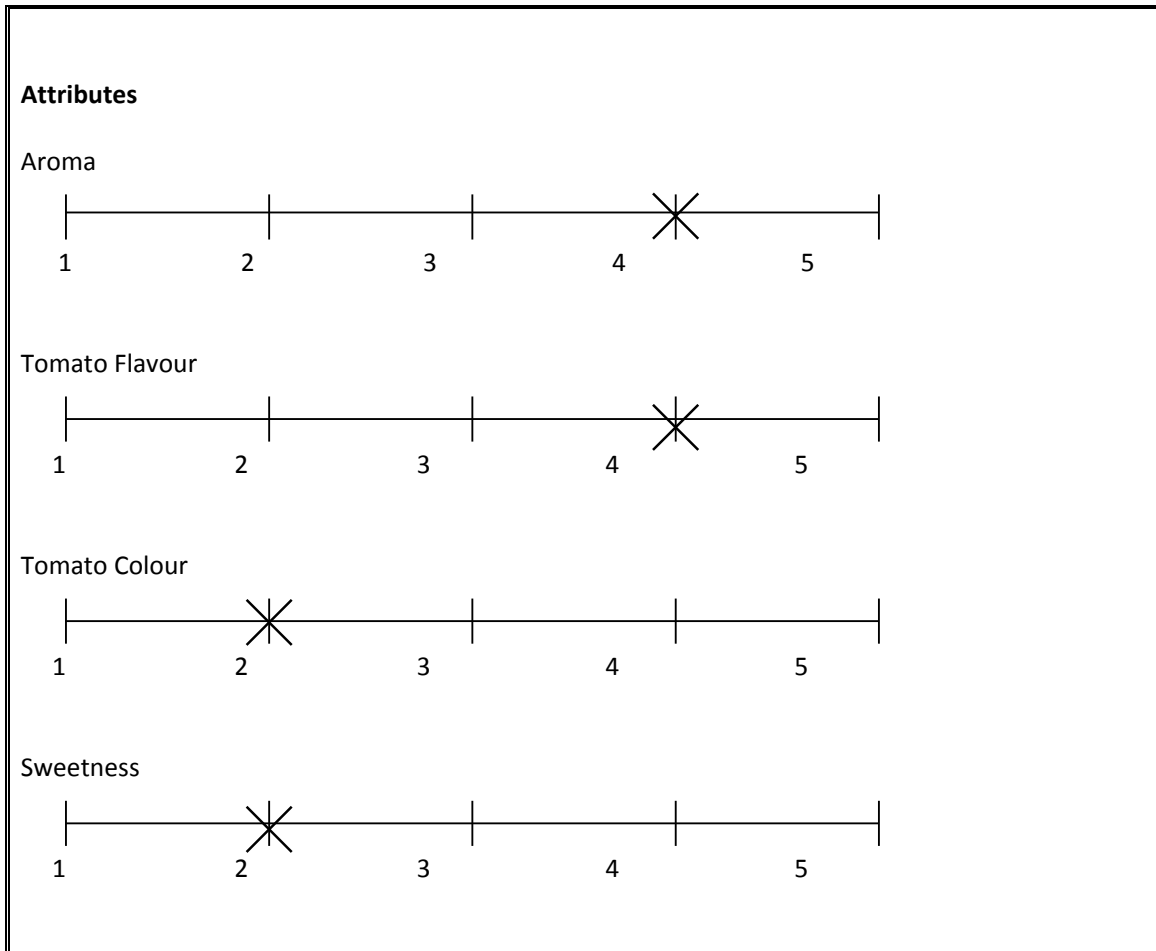
Record Sheet
Descriptive Rating Test - one product

Food Product: Tomato Soup

	Attributes Fill in attributes selected for profile			
	Aroma	Tomato Flavour	Tomato Colour	Sweetness
Tester 1 Results from scorecard	5	3	3	2
Tester 2 Results from scorecard	4	3	2	3
Tester 3 Results from scorecard	4	5	1	3
Tester 4 Results from scorecard	5	4	2	3
Tester 5 Results from scorecard	3	4	2	2
Tester 6 Results from scorecard	3	3	2	1
Total score	24	22	12	14
Average score (total score ÷ number of testers)	4	3.6	2	2.3

7. Present results by plotting the average score for each attribute on to the line scales. It is acceptable to round off average scores to the nearest whole number.

Presentation of Results	
Descriptive Rating Test - one product	
Tray number	Name
Food Product: Tomato Soup	
Please plot the average score for each attribute on the accompanying line scales.	
1 = very weak 2 = weak 3 = neither weak nor strong 4 = strong 5 = very strong	



8. Compile a sensory profile of the tomato soup based on the group result. For accurate profiling it is important to use the appropriate words for each number on the scales.

Profile of Tomato Soup

This soup has a strong aroma and a strong tomato flavour. However, it has a weak tomato colour and a weak sweet taste.

9. Evaluate results.

Template

Scorecard
Descriptive Rating Test - one product

Tray number Name

You are presented with a sample of _____

Please evaluate and rate the sample for each attribute and mark the number that best describes your choice on the accompanying line scale.

1 = very weak 2 = weak 3 = neither weak nor strong 4 = strong 5 = very strong

Attributes

.....



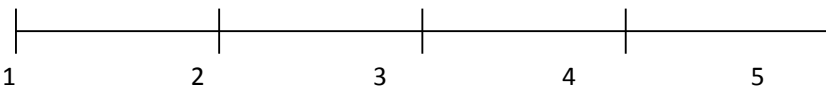
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.....



.....



Template

Record Sheet Descriptive Rating Test - one product				
Food Product: _____				
	Attributes Fill in attributes selected for profile			
Tester 1 Results from scorecard				
Tester 2 Results from scorecard				
Tester 3 Results from scorecard				
Tester 4 Results from scorecard				
Tester 5 Results from scorecard				
Tester 6 Results from scorecard				
Total score				
Average score (total score ÷ number of testers)				

Template

**Presentation of Results
Descriptive Rating Test - one product**

Tray number Name

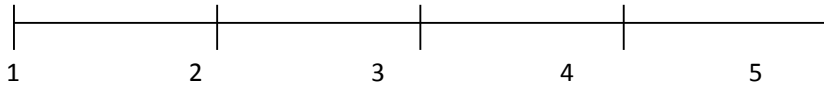
Food Product: _____

Please plot the average score for each attribute on the accompanying line scales.

1 = very weak 2 = weak 3 = neither weak nor strong 4 = strong 5 = very strong

Attributes

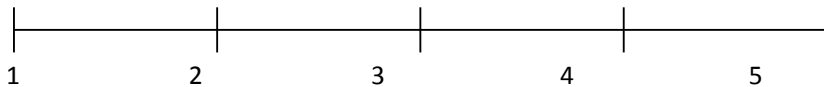
.....



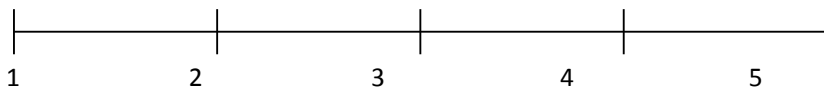
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.....



Space for Observations and Calculations

Experiment- 25- Sensory Evaluation- Descriptive Test- Descriptive Rating Test- Star Diagrams

Descriptive Rating Tests

Descriptive rating tests are used to evaluate and rate pre-selected sensory attributes in a food. A sensory attribute is the term used to describe a key characteristic of a food product.

The attributes can be rated on **line scales** or **star diagrams**.

A sensory profile is a written description of the sensory attributes of a food. This is compiled from the ratings obtained for the selected attributes.

Descriptive Rating Test – profiling two products using star diagrams.

Descriptive Rating Test

- Tester is presented with two coded samples
- Tester is asked to rate the intensity of the pre-selected attributes for each sample

Procedure for a Descriptive Rating Test using two products

Aim: To compile a sensory profile of each of two types of Fruit Scones using six pre-selected attributes.

Materials required based on four testers

- 4 trays
- 4 glasses of water
- 8 containers
- 4 samples of Fruit Scone A
- 4 samples of Fruit Scone B
- 4 scorecards
- 4 record sheets

Procedure

1. Agree six attributes. This is an important stage in the test and can be done by brainstorming or discussion within the class group. Each student should have a clear understanding of the meaning of the chosen attribute.

The six attributes agreed for the scones are:

- colour (golden brown)
- shape (even)
- lightness (light)
- sweetness (sweet)
- fruitiness (fruity)
- crumbliness (crumbly).

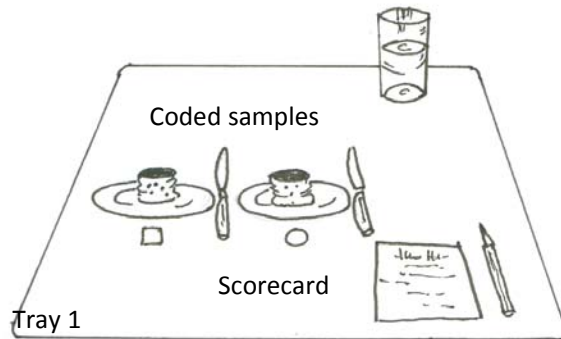
2. Label the scorecard and record sheet with the selected attributes. It is important that all scorecards have the attributes labelled at the exact same point on the star diagram. Begin labelling by writing the first attribute at the “12 o’clock position” and then move clockwise around the star by inserting the remaining attributes in sequence.
3. Code 8 containers as follows:
 - 4 containers with symbol □
 - 4 containers with symbol ○

4. Arrange food in the containers:

4 coded □ - Fruit Scone A

4 coded ○ - Fruit Scone B

5. Set up trays numbered 1 - 4. Place one container with symbol □ and one container with symbol ○ on each tray.



6. Instruct testers to follow instructions on scorecard.

Scorecard
Descriptive Rating Test – two products

Tray number Name

You are presented with two coded samples. Beginning with sample □, evaluate and rate the attributes from 0 - 5 using the star diagram coded □. Begin with the visual attributes. Then taste the product.

Repeat the same process with sample coded ○.

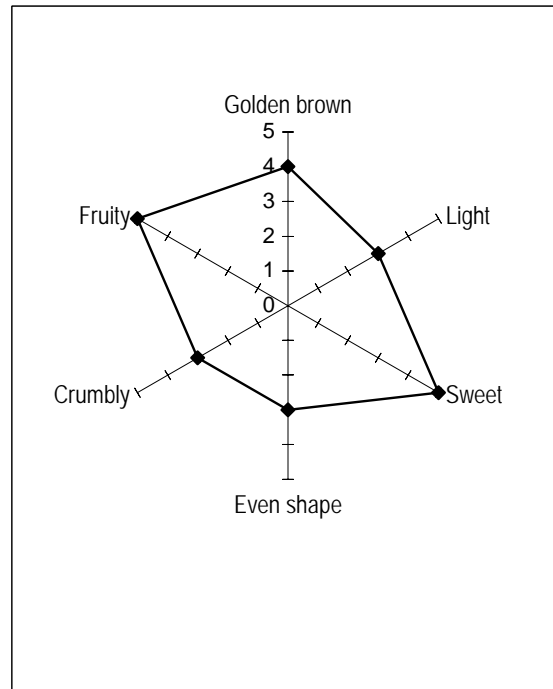
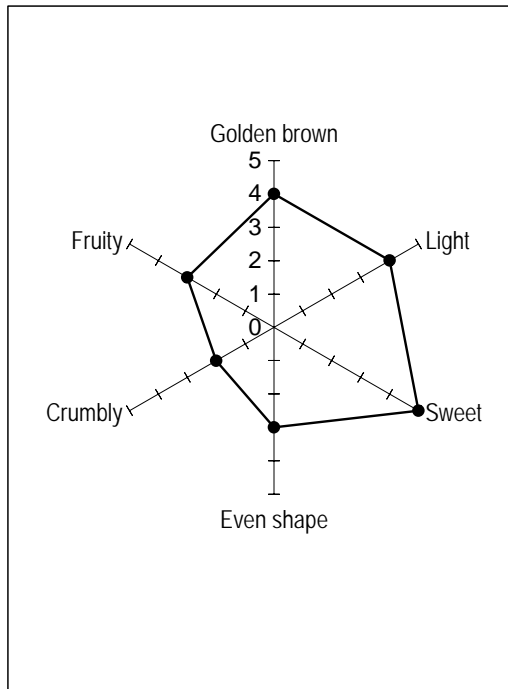
Join the dots to complete each star diagram.

Food Product □ _____

Food Product ○ _____

Star diagram coded □

Star diagram coded ○



0 = not at all 1 = weak 2 = fairly 3 = moderate 4 = quite 5 = very

7. Collect scorecards and transfer each tester's result onto the record sheet.
 Each tester should first of all transfer their own results from each star diagram on to the appropriate places on their own record sheet. They must then transfer the results of each tester in their group onto the record sheet. Average scores for each attribute are then calculated.

Record Sheet						
Descriptive Rating Test – two products						
Food Product <input type="checkbox"/> _____						
Collate the results from the scorecards in your group.						
	Attributes					
	Fill in attributes selected for profile					
	Golden Brown	Light	Sweet	Even Shape	Crumbly	Fruity
Tester 1 Results from star diagram	4	4	5	3	2	3
Tester 2 Results from star diagram	3	4	5	4	2	5
Tester 3 Results from star diagram	3	5	2	5	2	4
Tester 4 Results from star diagram	4	5	3	5	2	5
Total score	14	18	15	17	8	17
Average score (total score ÷ number of testers)	3.5	4.5	3.75	4.25	2	4.25
Food Product <input type="radio"/> _____						
Collate the results from the scorecards in your group.						
	Attributes					
	Fill in attributes selected for profile					
	Golden Brown	Light	Sweet	Even Shape	Crumbly	Fruity
Tester 1 Results from star diagram	4	3	5	3	3	5
Tester 2 Results from star diagram	4	3	4	4	2	5
Tester 3 Results from star diagram	3	3	5	3	3	4
Tester 4 Results from star diagram	4	2	4	2	2	5
Total score	15	11	18	12	10	19
Average score (total score ÷ number of testers)	3.75	2.75	4.5	3	2.5	4.75

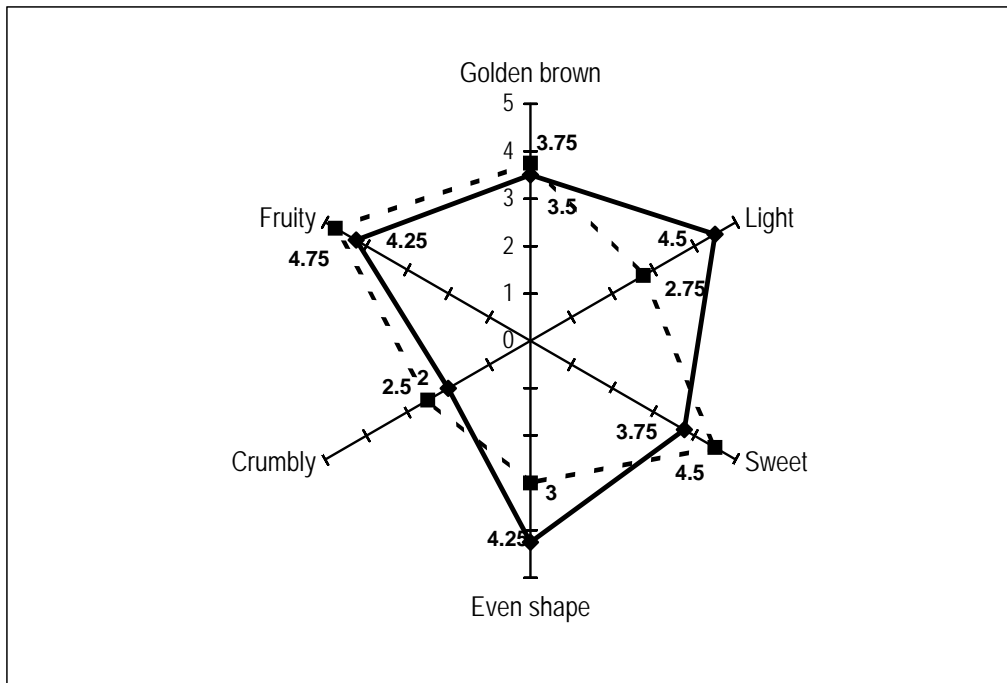
8. Present results by plotting the average scores from the record sheet onto the group star diagram. It is acceptable to round off average scores to the nearest whole number.

Presentation of Results
Descriptive Rating Test – two products

Tray number Name

For each product please plot the average score for each attribute on the star diagram.
 Use a different colour pen for each product.

Group Star Diagram



0 = not at all 1 = weak 2 = fairly 3 = moderate 4 = quite 5 = very

————— Food Product _____
 Food Product _____

9. Compile a sensory profile for each product based on the group results.
 For accurate profiling it is important to use the appropriate words for each number on the star diagram.

Profile of Fruit Score

This fruit scone is quite golden brown. It is very light and is quite sweet. It has quite an even shape, is fairly crumbly and quite fruity.

Profile of Fruit Scone ○

This fruit scone is also quite golden brown. It is moderately light and is very sweet. It has a moderately even shape, is moderately crumbly and very fruity.

10. Reveal codes.

11. Evaluate results.

Template

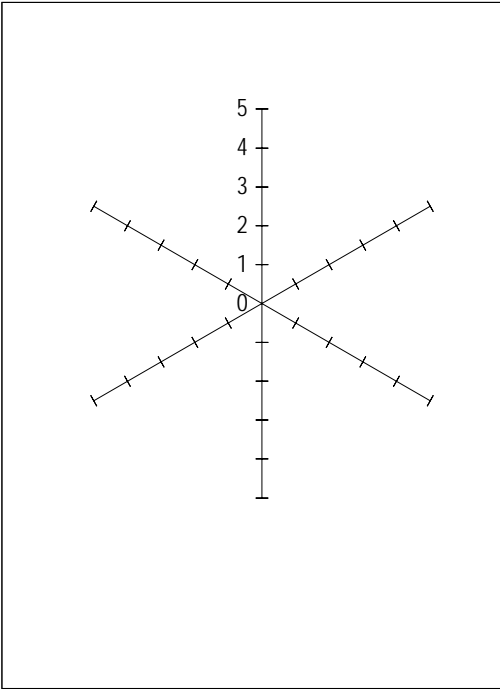
Scorecard
Descriptive Rating Test – two products

Tray number Name

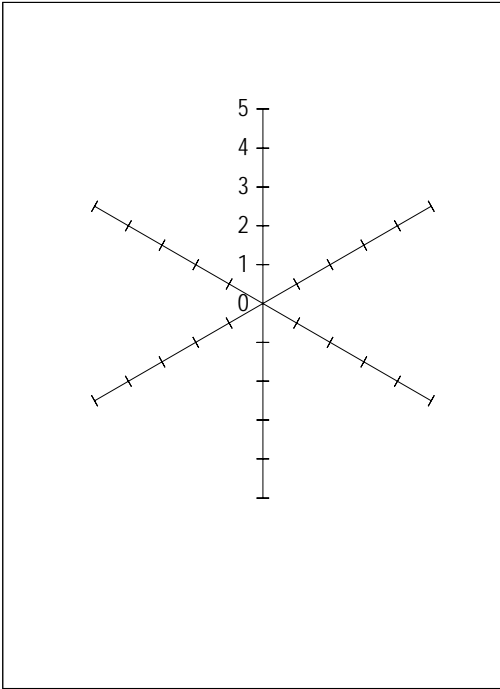
You are presented with two coded samples. Beginning with sample □, evaluate and rate the attributes from 0 - 5 using the star diagram coded □. Begin with the visual attributes. Then taste the product.
Repeat the same process with sample coded ○.
Join the dots to complete each star diagram.

Food Product □ _____ **Food Product ○** _____

Star diagram coded □



Star diagram coded ○



The form contains two identical star diagrams for rating. Each star diagram consists of a central point '0' with four arms extending outwards. The vertical arm is marked with numbers 1, 2, 3, 4, and 5. Each arm has tick marks corresponding to these numbers, with 5 being the furthest from the center. The star diagrams are intended to be filled in with dots and then connected to form a star shape.

0 = not at all 1 = weak 2 = fairly 3 = moderate 4 = quite 5 = very

Template

Record Sheet Descriptive Rating Test – two products						
Food Product <input type="checkbox"/> _____ Collate the results from the scorecards in your group.						
	Attributes Fill in attributes selected for profile					
Tester 1 Results from star diagram						
Tester 2 Results from star diagram						
Tester 3 Results from star diagram						
Tester 4 Results from star diagram						
Total score						
Average score (total score ÷ number of testers)						
Food Product <input type="radio"/> _____ Collate the results from the scorecards in your group.						
	Attributes Fill in attributes selected for profile					
Tester 1 Results from star diagram						
Tester 2 Results from star diagram						
Tester 3 Results from star diagram						
Tester 4 Results from star diagram						
Total score						
Average score (total score ÷ number of testers)						

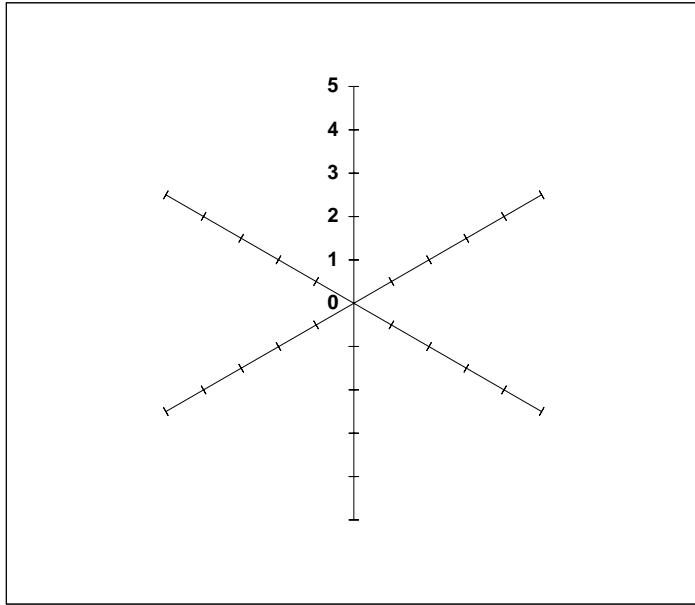
Template

**Presentation of Results
Descriptive Rating Test – two products**

Tray number Name

For each product please plot the average score for each attribute on the star diagram.
Use a different colour pen for each product.

Group Star Diagram



0 = not at all 1 = weak 2 = fairly 3 = moderate 4 = quite 5 = very

Food Product _____

Food Product _____

Profile of Food Product

Profile of Food Product

Space for Observations and Calculations

Appendix 1-Summary of Sensory Analysis Tests Suitable for the Classroom

Category	Tests	Number Samples	of	Aim
Preference Tests	Paired Preference Test	2		To determine the preferred product.
	Hedonic Rating Scale	1 or more		To find out how much a product is liked / disliked.
	Food Action Rating Test	1 or more		To determine attitude by indicating degree of liking / disliking for a product.
	Preference Ranking Test	2 or more		To rank products in order of preference.
Difference Tests	Simple Difference Paired Comparison Test	2		To determine if there is a difference between two samples.
	Directional Paired Comparison Test	2		To determine which of two samples has a greater degree of intensity in terms of a particular characteristic.
	Triangle Test	3 samples - 2 are the same and 1 is different.		To identify the sample that is different.
	Duo-Trio Test	3 samples - 2 are coded and 1 is identified as the control / reference.		To identify the sample that is different from the control / reference.
Descriptive Tests	Descriptive Ranking Test	2 or more		To rank samples of a product in order of intensity of specified attribute/s.
	Descriptive Rating Test - one product	1		To rate the intensity of pre-selected attributes for a food sample.
	Descriptive Rating Test – two products	2		To rate the intensity of pre-selected attributes for each food sample.

Appendix 2- Scales

In sensory analysis many different types of scales are used. However, the choice of scale should be considered in light of the test objective.

Target Groups

Product development is frequently aimed at particular target groups. Simple scales are used to identify and collate this information.

Example 1 – Gender

Male		Female	
------	--	--------	--

Example 2 – Age Group

15-25	26-35	36-45	46-55

Example 3 – Do you eat yoghurt?

Do you eat yoghurt?	Yes	No

Rating Scales

Many sensory analysis tests require products to be assessed and then rated on some form of scale. The scale chosen depends on the aim of the test and the possible outcome. It is essential to choose an appropriate scale for the test. There are many different rating scales and some of them are interchangeable.

The following are examples of rating scales where the categories lie in a specific order. The scales may contain numbers, words, or a combination of both. The lowest number on the scale denotes "less of" and the highest number denotes "more of".

Example 1 - Numeric

Not sweet

Extremely sweet

0	1	2	3	4	5	6	7	8	9
---	---	---	---	---	---	---	---	---	---

Example 2 - Verbal

Not sweet	Slightly sweet	Moderately sweet	Very sweet	Extremely sweet
-----------	----------------	------------------	------------	-----------------

Example 3 – Hedonic

Hedonic scales express degrees of like or dislike. The term hedonic means having to do with pleasure, so rating scales to do with likes or dislikes are called hedonic rating scales. Three commonly used hedonic scales are included below.

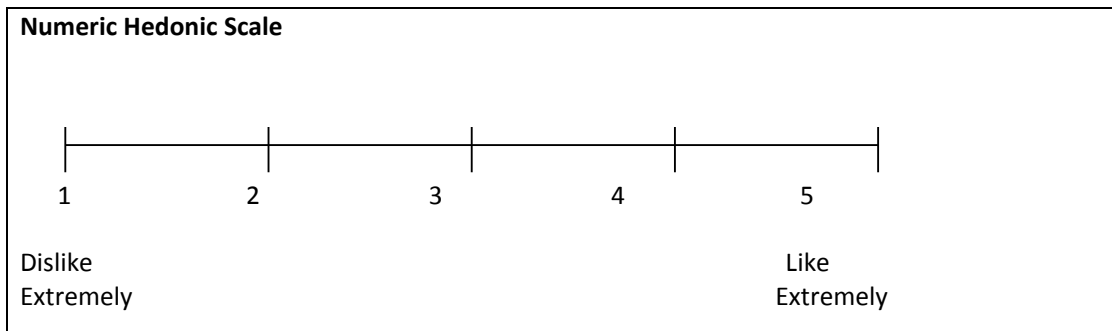
(a) Facial Hedonic Scale

Pictures are used to divide the scale.



(b) Numeric Hedonic Scale

Numbers are used to divide the scale. The scale is usually a five, seven or nine-point scale centred on a mid-point. In other words the scale always has an uneven number of points. When using this type of scale with students it would be important to keep the scale short. A five-point scale should be adequate.



(c) Verbal Hedonic Scale

Words or phrases are used to divide the scale. The words / phrases chosen are used to indicate the degree of liking for the product. The scale is usually a five, seven or nine-point scale. When designing verbal scales for classroom use it is important to use words that are easily understood by students.

Five-Point Verbal Hedonic Scale

Verbal Hedonic Scale			
	□	○	*
Like very much	_____	_____	_____
Like slightly	_____	_____	_____
Neither like nor dislike	_____	_____	_____
Dislike slightly	_____	_____	_____

Dislike very much

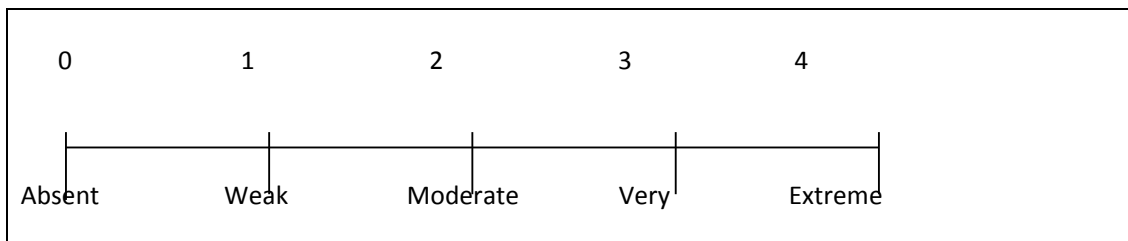
Nine-Point Verbal Hedonic Scale

Verbal Hedonic Scale			
	□	○	*
Like extremely	_____	_____	_____
Like very much	_____	_____	_____
Like moderately	_____	_____	_____
Like slightly	_____	_____	_____
Neither like nor dislike	_____	_____	_____
Dislike slightly	_____	_____	_____
Dislike moderately	_____	_____	_____
Dislike very much	_____	_____	_____
Dislike extremely	_____	_____	_____

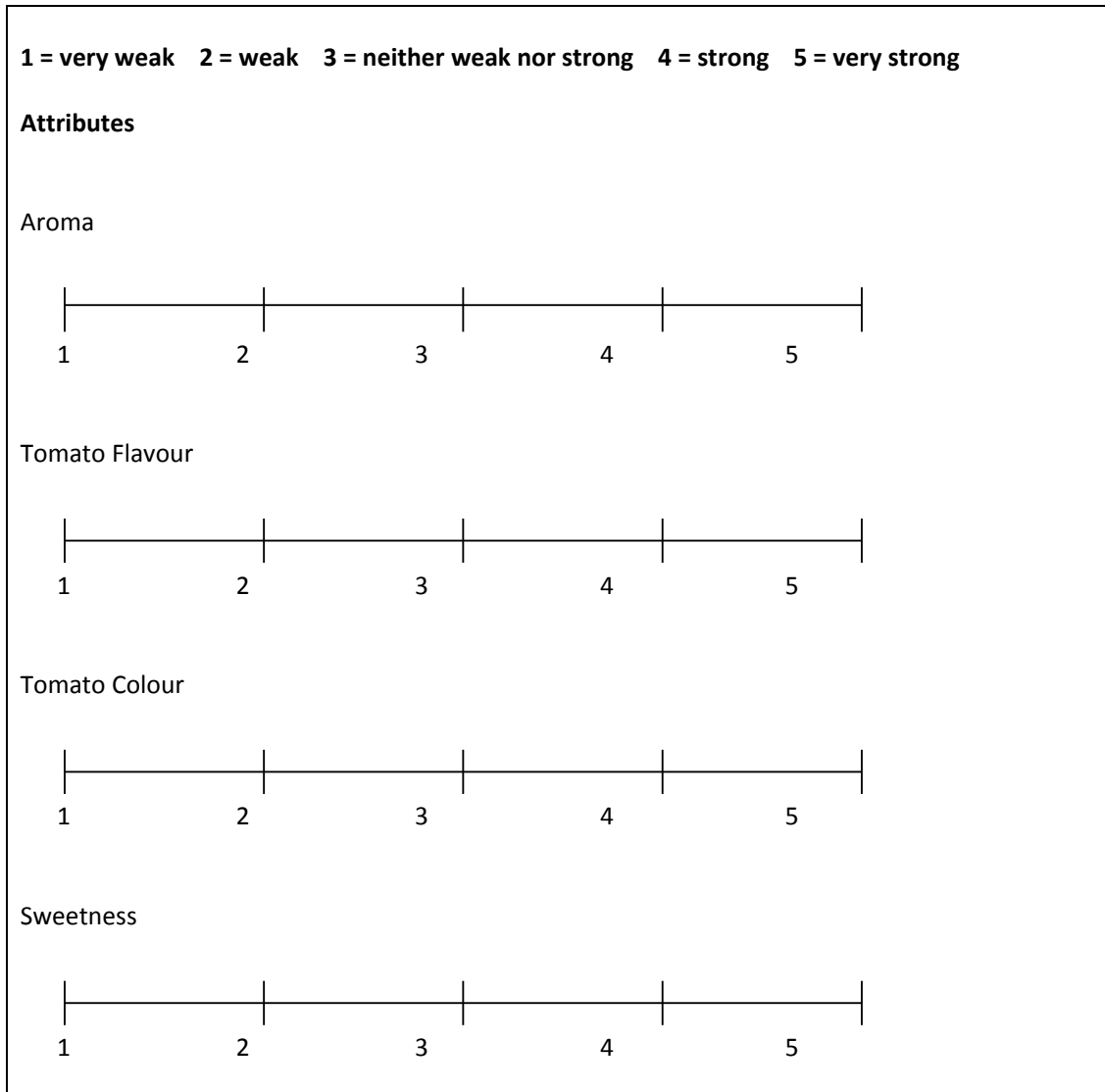
Example 4: Line Scales

Line scales are usually represented as a horizontal line, with a low rating at the left-hand end of the line and a high rating at the right-hand end of the line.

Single Line Scales



Series of Line Scales



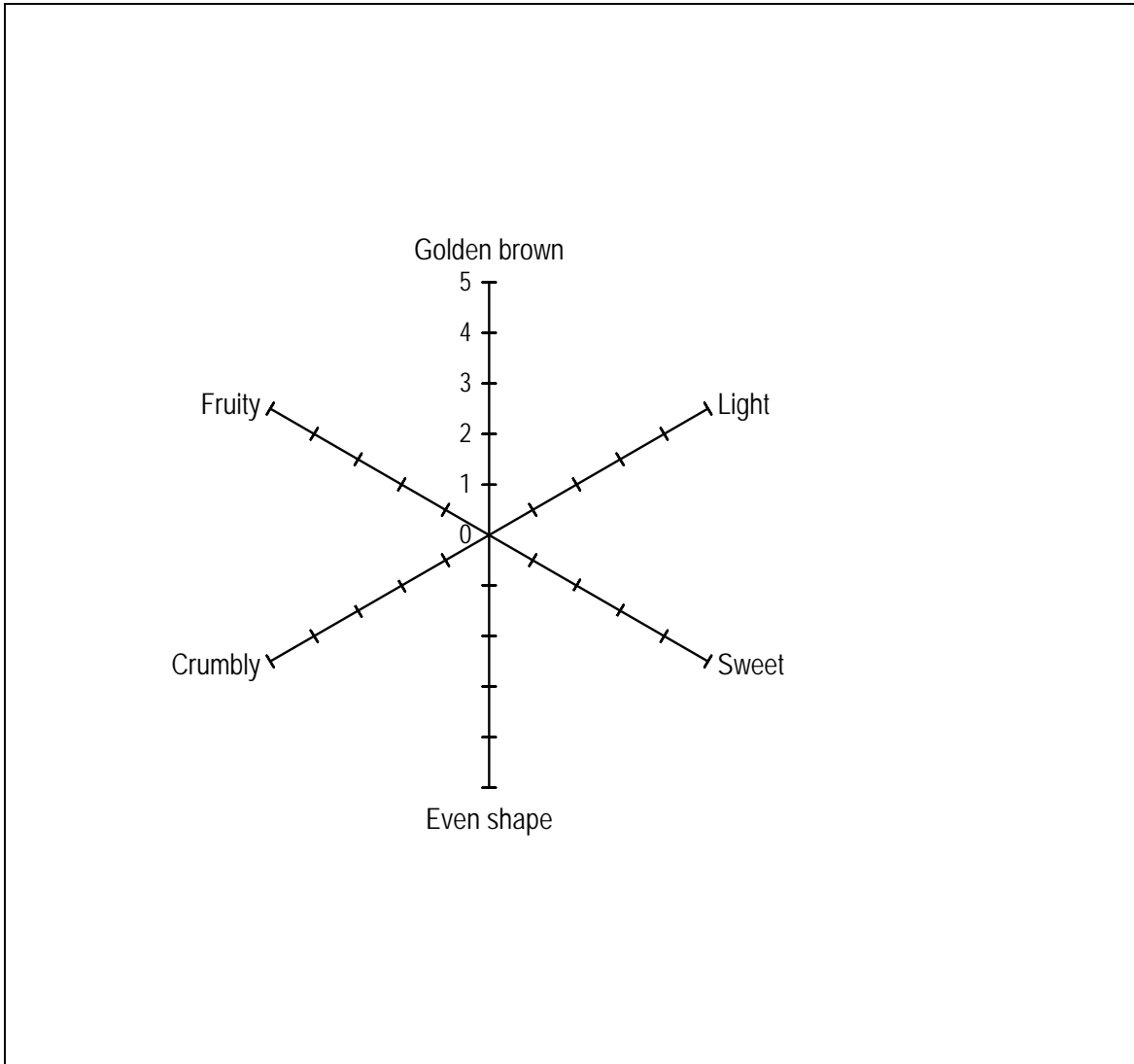
Example 5 - Star Diagrams

Star diagrams are used to rate particular attributes of a food.

How to draw a star diagram

- (i) Draw three or four lines (depending on the number of points required), intersecting at a central point.
- (ii) Label points of the scale on each line. Keep the scale short. A five-point scale should be sufficient. For each attribute the relative intensity increases as it goes further from the centre point.
- (iii) Label each line with the specific attribute being rated.
- (iv) Place a dot on the number, on the appropriate line, that best describe each attribute.
- (v) Join up the dots to make a star shape.

A Six-Point Star Diagram



Appendix 3- Presentation of Results

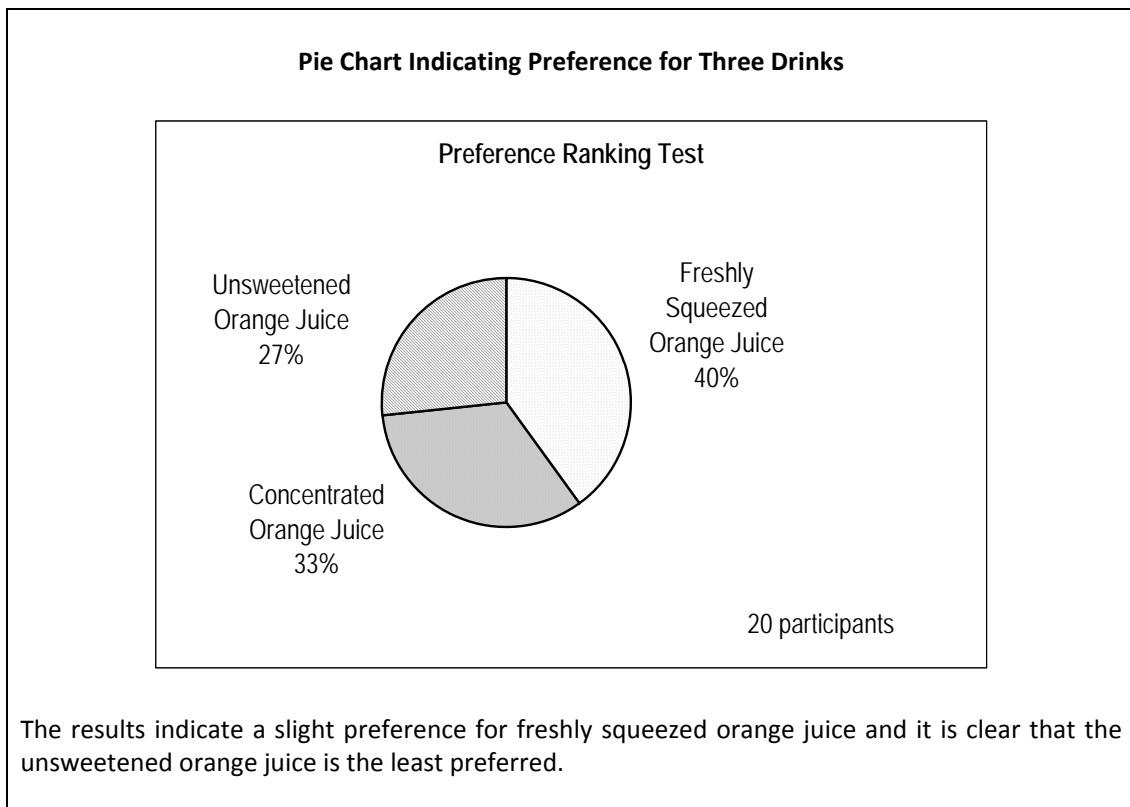
Sensory analysis test results can be presented on pie charts, bar charts and scales such as star diagrams. The method of presentation will depend on the nature of the data collected and the type of analysis required.

Pie Charts

Pie charts are best suited to simple tests where the tester is carrying out one instruction only such as:

- (i) which product is preferred
- (ii) ranking products in order of preference.

A pie chart can be drawn by hand or using a computer programme. It is important to label the pie chart indicating a key for each product, the percentage result for each segment and the number of testers involved in the test. An exemplar is illustrated below.

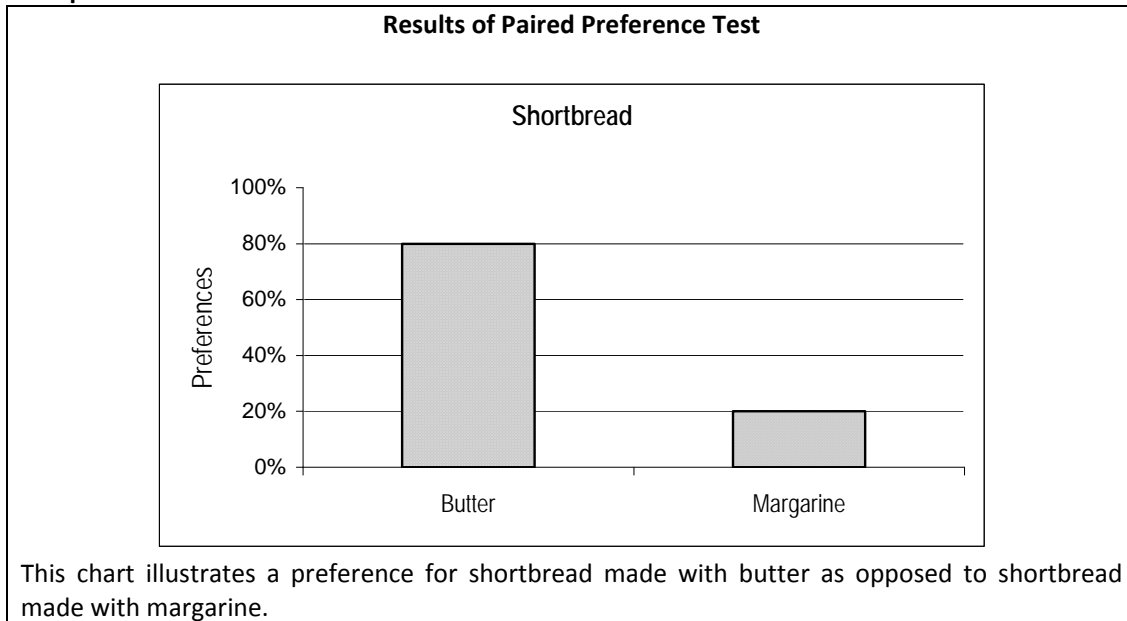


Bar Charts

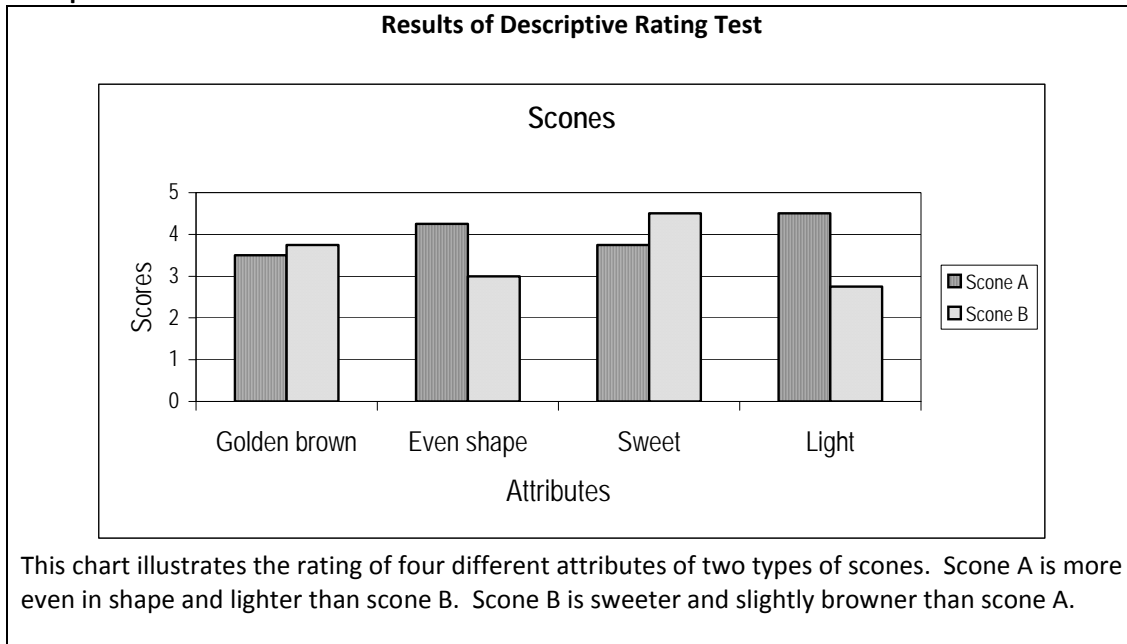
Bar charts can be used as an alternative to pie charts to present simple information. They can also be used to present more complex data.

A bar chart can be drawn by hand or using a computer programme. Both axes should be labelled. The number or percentage of testers should be shown clearly on the vertical axis. The products or characteristics of products being tested should be indicated on the horizontal axis. A key may be required to help identify the products. Exemplars are illustrated below.

Example 1



Example 2



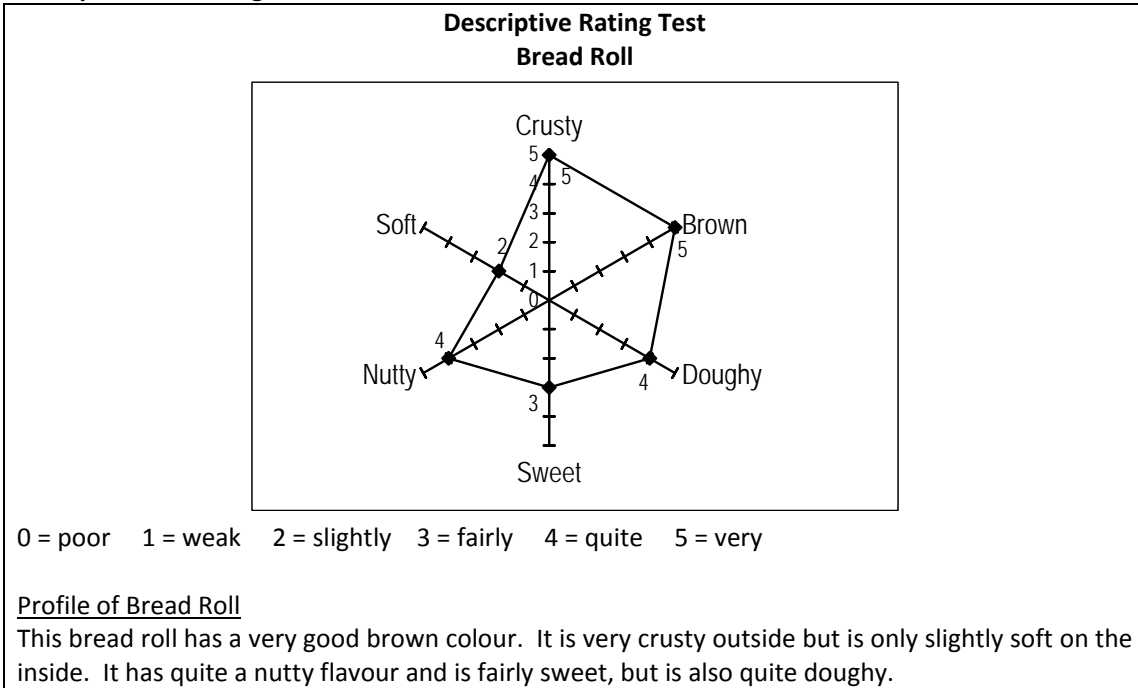
Star Diagrams

While scales are generally used for rating (Appendix 2) they can also be used to present results. Star diagrams are particularly useful.

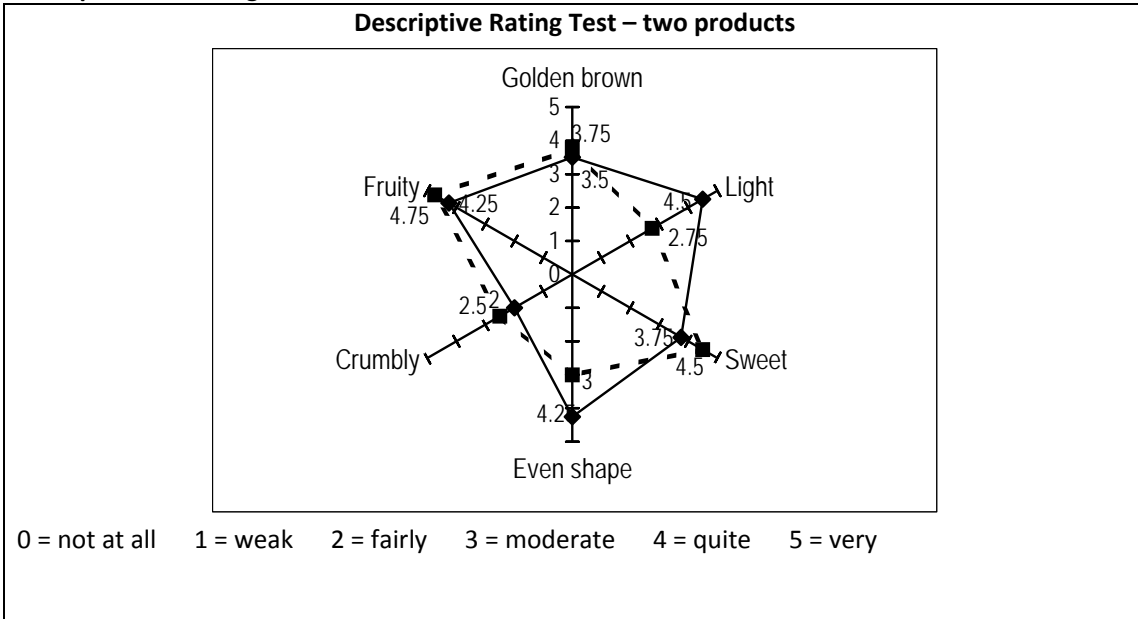
Star diagrams can be used to present data for one or more products. Further details on star diagrams are available on page 79. Exemplars are illustrated below.

A profile can be compiled from the information presented on the star diagram.

Example 1 – Star Diagram for One Product



Example 2 - Star Diagram for Two Products



Appendix 4- Glossary of Terms Used in Sensory Analysis

Appearance The visible attributes of a food.

Attribute A perceived sensory characteristic of a food.

Balanced Presentation Order Each food is presented an equal number of times and the samples are presented in random order.

Control Sample of the product being tested is chosen as a reference point against which all others are compared.

Hedonic Relating to like or dislike.

Hedonic Scale Scales expressing degrees of like or dislike.

Organoleptic Assessment Using the senses to evaluate food.

Profile Description of the perceived sensory attributes of a food.

Random Order The presentation order of samples on each tray is varied between testers.

Ranking Food samples are placed in order according to preference or intensity of a specified attribute.

Rating Food samples are scored according to the intensity of a specified attribute.

Record Sheet Document used to compile results from a sensory analysis test.

Reference Sample of the product being tested is chosen as a reference point against which all others are compared.

Scorecard Sheet that testers use to answer the question/s asked in a sensory analysis test.

Tester Person who evaluates foods and fills out a scorecard. This person may be trained or untrained.

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