**Testing of food contact materials**

Types of testing food contact materials:
- Migration test
- Simple testing for identification types of packaging polymers
- Permeability

**Migration tests**

Migration of components or compound from food contact material to food is very important property of this type of materials. This property related with safety and stability of material. Migration of compounds is not one-way but two-way process, because compounds or components of food contact materials can migrate from materials into food equally compound of food can migrate into material.

Generally there are two types of migration:
- global migration (there is monitored transmission level of all compounds/components of polymers into food/food simulants)
- specific migration (there is monitored transmission level of specific compounds/components (e.g. primary aromatic amines) of polymers into food/food simulants)

There is a problem, because many types of food material have very complex matrices, which is not chemically uniform and determination of migrant substance is very complicated. For better determination of compound migration are used food simulants. There are four basic food simulants:
- A – distilled water (substitution of neutral food)
- B – diluted acid solution (e.g. 3% (w/v) acetic acid solution, substitution of acidic food)
- C – Ethanol/water mixtures (e.g. 10% (v/v) ethanol solution, substitution of alcoholic food)
- D – e.g. Olive oil or isooctane (substitution of fatty food)

**Laboratory work:**

**Determination of migration of primary aromatic amines from food contact materials**

**Principle:**
Aromatic amines in extract from tested samples (extract = specific volume of food simulant which is in contact with tested material the specific time and the specific temperature) reacts in presence of hydrochloric acid (HCl), sodium nitrite (NaNO₂) and ammonium sulfamate with \( N-(1\text{-naphthyl})\text{ethylenediamine dihydrochloride} \) to violet product whose intensity is measured by spectrophotometer at wavelength 550 nm.

**Materials and reagents:**
- Distilled water
- food simulant extracts which was in contact with tested material for specific time in specific temperature
- 3 % (w/v) solution of acetic acid in distilled water (optional)
- 10 % (v/v) solution of ethanol in distilled water (optional)
- 0.0725 M solution of sodium nitrite in distilled water
- 0.22 M solution of ammonium sulfamate in distilled water
- 0.0385 M solution of \( N-(1\text{-naphthyl})\text{ethylenediamine dihydrochloride} \) in distilled water
- 1 M solution of hydrochloric acid in distilled water
- ΘZ = 1.1 mM solution of aniline hydrochloride in distilled water
- ΘA = working solution of aniline hydrochloride – Preparation: 5 ml of 1.1 mM solution of aniline hydrochloride put into volumetric flask (500 ml) and fill up to mark by distilled water (final concentration of aniline is 1 µg/ml)
- ΘR3 = Control chart solution of aniline hydrochloride – Preparation: 5 ml of working solution of aniline hydrochloride put into volumetric flask (100 ml) and fill up to mark by food simulant (distilled water or 3% solution of acetic acid)

Preparation of calibration curve:
- Prepare six test tubes and add into each one test tube 0.00 (blank), 0.25, 0.50, 1.00, 2.00 and 4.00 ml of solution ΘA and fill up to final volume 20 ml by food simulant (e.g. to test tube with 0.5 ml of solution ΘA add 19.5 ml of food stimulant, etc.)
- Add into each test tube 2.5 ml of 1 M hydrochloric acid and 0.5 ml of sodium nitrite, after addition mix with solution in test tubes
- After 10 minutes add into each test tube 1 ml of 0.22 M solution of ammonium sulfamate, after addition mix with solution in test tubes
- After 10 minutes add into each test tube 1 ml of 0.0385 M solution of N-(1-naphthyl)ethylenediamine dihydrochloride, after addition mix with solution in test tubes
- After 2 hours measure absorbance by spectrophotometer at wavelength 550 nm

Control chart measurement:
- Add into each of three test tube 20 ml of solution ΘR3
- Next steps are the same like in preparation of calibration curve: add into each test tube 2.5 ml of 1 M hydrochloric acid, after addition mix with solution in test tubes etc.

Sample measurement:
- Procedure is the same like control chart measurement, only don’t add 20 ml of solution ΘR3, but 20 ml of food simulant which was in contact with tested sample the specific time and the specific temperature
- Do one parallel measurement for each sample

Calculation:
- From calibration curve deduct amount of aniline (amines) extracted from tested sample to aliquot volume of food simulant in µg of aniline
- For calculation migration of aromatic amines from food contact material into food simulant use this formula:

\[ M_i = \frac{V_{ci} \times m_i}{P_i \times V_i} \]

where:
- \( P_i \) means area of tested sample which was in contact with food stimulant in dm²
- \( m_i \) means amount of aniline in aliquot volume of food stimulant which was deduct from calibration curve in µg
- \( V_i \) means aliquot volume which was added into test tube in ml (commonly 20 ml)
- \( V_{ci} \) means total volume of food simulant which was in contact with tested sample in ml
- \( M_i \) means amount of aromatic amines which was transferred from food contact material into food simulant in µg of aniline/dm² of tested material
Laboratory work

Determination of packaging materials tensile profile

Principle:
Tensile strength (and other tensile properties) of packaging material are obtained by Instron device. This device measure for example required force to break the strips which were cut out from packaging material.

Devices, software and other materials:
- Instron 5544 - laboratory device
- Evaluation software Series IX
- Cutting device for making strips from packaging material
- Micrometer L&W SE D 051 for determination of thickness of tested packaging material
- Tested packaging material

Instruction:
- Cut strips from packaging material by cutting device
- Measure thickness of strips
- Determinate a tensile profile of packaging material by measuring device Instron

Calculation:
- Software program itself will assess tensile profile and will calculate four basic parameters: maximal force at break moment [N], tensile strength [MPa], elongation at break moment [mm] and mechanical work [J] required for irreversible deformation

Laboratory work

Determination of water vapor permeability

Principle:
Determination of water vapor permeability is gravimetric analysis. Dry silica gel in boxes with cap from packaging material changes own weight, because silica gel receive water vapor through packaging material in environment with known relative humidity and temperature.

Materials:
- Boxes for the location of the silica gel
- Dry silica gel
- Packaging material
- Mixture from wax and paraffin for sealing the space between packaging material and box

Instruction:
- Cut circle with specific proportions from tested packaging material
- Put dry silica gel into boxes (approximately same amount silica gel in each box) and cover open space over silica gel by circle from packaging material
- Seal the space between packaging material and box by molten mixture from wax and paraffin
- Weigh boxes immediately after sealing (this weight is at time 0 hour)
- Hourly weigh boxes (with silica gel inside and cap from packaging material) and make note of the weight and with the time of weighing
Calculation:
- Make a graph where x-axis (horizontal) represents total time of measuring of weight in hours and y-axis (vertical) represents actual total difference of weight in milligrams calculated from increment of weight between two measuring of weight
- Use linear regression to determine a slope or gradient of a graph
- For determination water vapor permeability in \( \text{g H}_2\text{O}/(\text{m}^2 \times \text{day}) \) can use formula:
  \[
  q = \frac{240 \times \Delta m}{S \times \Delta t},
  \]
  where:
  - \( \Delta t \) means difference of time (h) between two measuring of weight
  - \( \Delta m \) means difference of box weight (mg) between two measuring of weight
  - \( S \) means area (cm\(^2\)) of cap from packaging materials
  - \( q \) means water vapor permeability in \( \text{g H}_2\text{O}/(\text{m}^2 \times \text{day}) \)
- \( \frac{\Delta m}{\Delta t} \) means slope or gradient of a line (graph) which was determined in the previous procedure
- If we known that area for all polymer caps are same \( S=50,2 \text{ cm}^2 \), we can simplify previous formula:
  \[
  q = 4.78 \times \alpha,
  \]
  where:
  - \( \alpha \) means slope or gradient of a line (from graph)
  - \( q \) means water vapor permeability in \( \text{g H}_2\text{O}/(\text{m}^2 \times \text{day}) \)