



ANALYSIS

GPC/SEC

Size Exclusion Chromatography (SEC, or Gel Permeation Chromatography GPC) is the most common method for polymer analysis. In this process the polymer is solved in an eluent and fractionated in a separation column, which is filled with porous material. By means of different detectors the polymer can be analysed. The column material consists normally of highly cross-linked PS latex particles, whereupon the pores have a broad size distribution. The fractionation of the polymer only bases on geometric pore effects, i.e. for this process the separation parameter is the hydrodynamic volume of the polymer.



These are the specifications of our measuring system:

Aqueous SEC/GPC

- Analyte: Polyelectrolytes, non-ionic water soluble polymers, polysaccharides
- Sample amount ~100 mg
- Range of molar mass: 103 106 g/mol
- Eluent: aqueous salt solution, if necessary. MeOH/H2O-mixture
- Detector: UV-Detector, RI-Detector
- Conventional calibration with dextran
- \Rightarrow Molecular weight (Mw, Mn), polydispersity PDI
- Universal calibration
- \Rightarrow Mw, Mn and PDI for additional polymers accessible

Organic GPC/SEC

- Sample amount ~ 100 mg
- Range of molar mass: 103 106 g/mol
- Eluent: Tetrahydrofuran, Toluene
- Detectors: UV-Detector, RI-Detector
- Conventional calibration with polystyrene, polymethylmethacrylate
 ⇒ Molecular weight (Mw, Mn), polydispersity PDI
- Universal calibration
 - \Rightarrow Mw, Mn and PDI for additional polymers accessible







Membrane osmometry

Membrane osmometry is a technique for the determination of molecular masses of polymers by means of osmosis. The phenomenon of osmosis describes the attempt of solvent molecules to go through a semipermeable membrane into a solution. The detection of the so originated osmotic pressure can be determined into the number average molecular weight Mn of the solved polymer.

These are the specifications of our measuring system:

- Analyte: in water or in organic solvents soluble polymers, nanoparticle-dispersions
- Sample amount ~ 100 500 mg
- Range of molar mass: 104 106 g/mol
- Equipment: GONOTEC Membrane Osmometer OSMOMAT 090
- Solvent: Water, aqueous salt solutions, organic solvents
- Membrane: Cellulose-triacetate (cut-off 5.000, 10.000, 20.000 g/mol), regenerated cellulose (cut-off 20.000 g/mol)
- Measurement of the osmotic pressure depending on the polymer concentration
 - \Rightarrow Determination of the absolute molecular mass Mn
 - \Rightarrow Determination of the A2-value (2nd virial coefficient): Measure for solvent quality







Classification numbers

Chemical characteristic numbers are applied in many fields like quality control and research and development. Our range covers the volumetric characterization of polymers/plastics according to standardized methods.



Methods:

DIN EN ISO 4629	Binders for paints and varnishes: Determination of hydroxyl value - Titrimetric method
DIN EN ISO 3681	Binders for paints and varnishes: Determination of saponification value - Titrimetric method
DIN EN ISO 1061	Unplasticized cellulose acetate: Determination of free acidity
DIN EN ISO 3001	Epoxy compounds: Determination of epoxy equivalent
DIN EN ISO 2114	Plastics (polyester resins) and paints and varnishes (binders): Determination of partial acid value and total acid value
DIN EN ISO 1264	Homopolymer and copolymer resins of vinyl chloride: Determination of pH of aqueous extract
ISO 14900	Plastics (Polyols): Determination of hydroxyl number
	Determination of degree of substitution for cellulose acetate according to E. Samios
	Quantitative determination of chloride- and bromide ions (Mohr method)





Physical parameters

Physical parameters are needed in the fields quality control, security data, and research and development. We offer following methods:

Absorption spectra (UV/Vis)

We use a two-beam spectrometer with a wavelength range from 190 to 1,100 nm. We can offer following experiments:

- Absorption spectra
- Time depending testings (e.g. reaction kinetic)
- Wavelength depending testings (e.g. purity testing)
- Determination of concentration



Refractive index

For the determination of the refractive index, we use an **Abbe-Refractometer**.

Methods:

DIN EN ISO 489	Plastics – Determination of refractive index
ISO 1739	Butter – Determination of refractive index of the fat (Reference method)

Vapor pressure

The vapor pressure occurs when a gas in a closed system with the associated liquid is in thermodynamic equilibrium. It is determined in a double-jacketed glass vessel according to VERORDNUNG (EG) Nr. 440/2008 DER KOMMISSION from 30.05.2008. The determination can be carried out using both the static and dynamic methods.

- Sample volume: ca. 20 mL
- Temperature range: 5 ... 60°C (lower/higher temperatures on request)







Density

Density is used in various areas of application to identify material or product properties, such as concentration. The determination is one of the most frequently used gravimetric methods in the laboratory. Our repertoire includes measuring density using a pycnometer and a vibration measuring device.

Pycnometer:

- Volume calibrated glass bulb
- Very precise procedure
- Suitable for solids, powders, granules, liquids, and dispersions



Vibrometer:

- Measurement of the natural frequency of the sample
- (homogeneous) liquids
- Temperature range: -10 ... 80°C



Methods:

DIN EN ISO 2811/1	Paints and vanishes – Pycnometer method
DIN EN ISO 2811/3	Paints and vanishes – Oscillation method
DIN EN ISO 1183/1	Plastics – Pycnometer method
ISO 8130/3	Coating powders – Pycnometer method
ISO 1675	Plastics – Liquids resins – Pycnometer method
ISO 2781	Rubber, vulcanized of thermoplastic
ISO 845	Cellular plastics and rubbers – Determination of apparent density

Density under elevated pressure / temperature

Due to the compressibility of substances, the density depends not only on the temperature but also on the pressure. Our measuring system consists of a pressure-resistant and temperature-stable vibration measuring device and allows density measurements up to 140°C and 400 bar. Using the Tait equation, these values can generally be extrapolated reliably up to at least 1,000 bar.





Dipole moment

The dipole moment is a measure of the spatial separation of charges in the molecule (strength of the dipole character). To determine the dipole moment for organic liquids, we use the WTW model DM01 dipole meter with a measuring frequency of 2MHz. A dipole meter can be used to measure the dipole moment and dielectric constant of a liquid sample at a specific temperature.



Freezing point depression

Freezing point depression refers to the phenomenon that the freezing point / melting point of solutions is lower than that of the pure liquid. We have a double-jacketed glass vessel at our disposal to determine the freezing point depression. By slowly cooling the dissolved sample or the pure liquid, the freezing point and the resulting freezing point depression can be determined.

 Temperature range: ...-40°C (other temperatures after request)

Possible parameters that can be determined using freezing point depression:



- Molar mass
- Number averaged molar mass (polymer, oligomer)
- Osmololality





Surface tension

The surface tension of a liquid and the interfacial tension between two liquids are determined using the **Du Noüy ring method**. If requested, we measure according to the OECD GUIDELINES FOR THE TESTING OF CHEMICALS #115.

Methods:

When measuring the surface or interfacial tension, the force acting on a ring wetted with the liquid is measured, which is created when the ring moves from one phase to the other due to the tension of pulled-out liquid lamella. If the lamella is perpendicular to the plane of the ring when the ring moves out, a maximum force occurs, which correlates with the surface or interfacial tension. The maximum force is determined using an electronic force sensor.



Melting point

The melting point is determined according to OECD GUIDELINES FOR THE TESTING OF CHEMICALS #102.

Boiling point

The boiling point is determined according to OECD GUIDELINES FOR THE TESTING OF CHEMICALS #103.

In addition, the distillation process can be determined according to DIN EN ISO 3405 resp. AST D86.





OECD/EU methods:

OECD (Organisation for Economic Cooperation and Development) developed standardized analysis methods as part of chemical testing. We provide you with the following physical-chemical tests to characterize your substance or to create safety-relevant data:

Standard	Description
OECD 101	UV/Vis absorption spectra
OECD 102	Melting point
OECD 103	Boiling point
OECD 104	Vapor pressure
OECD 105	Water solubility
OECD 112	Dissociation constant in water
OECD 114	Viscosity
OECD 115	Surface tension
OECD 118	Number average molar mass and molar mass distribution of polymers by means of size exclusion chromatography
OECD 119	Low molar mass ratio of polymer by means of size exclusion chromatography
OECD 122	Acidity and Alkanity





In addition, we offer the following analyzes to determine the physico-chemical properties according to VO(EG)440/2008, Appendix A:

Standard	Description
A.1	Melting / freezing point
A.2	Boiling point
A.3	Relative density
A.4	Vapor pressure
A.5	Surface tension
A.6	Water solubility
A.8	Distribution coefficient
A.18	Number average molar mass and molar mass distribution of polymers
A.19	Low molar mass ratio of polymer by means of size exclusion chromatography
A.20	Dissolution / extraction behavior of polymers in water





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