update: October 2020

PTG EINDHOVEN

MATERIAL INNOVATORS

ANALYSIS TECHNIQUES

Overview of available techniques
mechanical, chemical, physical, viscoelastic

SERVICE ANALYSIS AT PTG/e

Following info cards show (part of) the analysis equipment and techniques available at PTG/e.

INDEX

LEGEND

- Identification
- Thermal properties
- Mechanical properties
- Viscoelastic properties
- Optical analysis
- Molecular structure
- Water content
- Surface analysis

ABOUT PTG/e

Polymer Technology Group Eindhoven BV (PTG/e) is an independent applied research and knowledge partner. We specialise in and are fully equipped (staff and infrastructure) in the field of (polymer)chemistry, polymers and hybrid materials. PTG/e-labs are located at the TU/e Campus in the middle of Brainport Eindhoven.

PTG/e offers (since 2004) further improvement of your in-house innovation. Whether it is contract research or material analysis, you can count on our professional support. We take a pragmatic to-the-point approach, which translates into short communication lines and regular contact between our multi-disciplinary staff and you.

www.ptgeindhoven.nl

CONFOCAL RAMAN SPECTROSCOPY

Brand	WITec Alpha 300R
Possible laser wavelengths	532nm, 633nm and 785nm
Typical sample size	1 – 1000 µm
Sample types	Most organic and inorganic samples
Identification	Bio-Rad database containing over 25.000 reference spectra
Output	Molecular fingerprint

Raman spectroscopy is similar to infrared spectroscopy in a way that both techniques are used to identify unknown substances. Raman spectroscopy uses a laser to interact with an unknown substance. Confocal Raman microscopy combines the Raman spectroscopy with an optical microscope, which provides extra spatial (vertical and horizontal) resolution of samples. Therefore, this technique is especially useful for microscopic defect analysis. Analyses can be performed in 1D, 2D and 3D with spot sizes of less than 1,0 µm.

CONTACT ANGLE MEASUREMENT

Brand	OCA-20 Contact Angle Measuring Instrument (DataPhysics Instruments GmbH)
Liquid	Water, hexadecane
Output	Contact angle

Contact angle measurements can be done on flat surfaces to determine its hydrophobic or hydrophillic behavior. A total of 10 drops will be placed on the to be measured surface which are then imaged by a camera. Via the software the contact angle at the left and right size is than calculated, and gives an avarage over the 10 droplets. With contact angle measurements a variation of +/- 3° in between measurements is within the tolerance limits.

DEKTAK

Brand	Bruker DektakXT Stylus profiler
Scan Length Range	55 mm or 200 mm with scan stitching capability
Vertical resolution	1Å (@ 6.55 µm range)
Max sample dimensions	200x200x50 mm (WxLxT)
Output	Two-dimensional surface profile measurements; Optional three-dimensional measurement/analyses

Surface profilers are commonly used for measuring thin film thickness, and surface roughness and form in applications ranging from educational research verification to semiconductor process control.
The tip of a stylus gently moves over the surface of a flat surface in one line, leading to a two-dimensional surface profile. When multiple lines next to eachother are combined a three-dimensional plot can be derived and analysed.

DIFFERENTIAL SCANNING CALORIMETRY (DSC)

Brand	TA Instruments Q2000
Temperature range	-80 to 300 °C
Maximum Heating rate	50 K/min
Typical sample size	3 mg
Type of DSC pans	Aluminum hermetic
Output	Melting point Glass transition point (Tg) Crystallization Heat capacity

DSC is used to examine the thermal transitions in a polymeric material (e.g. melting point, Tg and crystallization).
Measurements are carried out with a TA Instruments Q2000, which has a temperature range of -80 to 300 °C.
A material sample of as little as 3 mg is sufficient for a measurement.
The results of a DSC measurement can for example be used to determine if a material melts and if so, when.
It can also be used to distinguish between a homopolymer, a copolymer and a blend.

DYNAMIC MECHANICAL THERMAL ANALYSIS (DMTA)

Brand	TA Instruments Q800 DMTA
Temperature range	-140 to 600 °C
Frequency range	0.01 – 200 Hz
Maximum force	18 N
Modes & Sample Size (LxWxT [mm])	Dual / single cantilever: *35/17.5 x <15 x <5* 3 point bending: *50 x <15 x < 7* Tension film: *10 – 30 x <8 mm x <2* Compression: *15 and 40 mm dia., <10 mm (T)*
Output	Storage and loss modulus, Tan Delta, Frequency, Static/Dynamic force, Discplacement, Shrinkage, Creep, Relaxation, Stress-strain, CTE, TTS.

DMTA is a technique used to analyse the viscoelastic behaviour of a material as a function of temperature or frequency. The DMTA measurement results are used for determining thermal transisions suchs as the glass transition temperature (Tg). A DMTA diagram shows the stifness (storage and loss modulus) as a function of temperature.

GAS CHROMATOGRAPHY MASS SPECTROMETRY (GC-MS)

Brand	PerkinElmer Clarus 690 GC & PerkinElmer SQ8T MS
Carrier Gas	Helium
Output	Mass spectrum Material identification

Gas Chromatography combined with Mass Spectrometry is a way to identify or quantify (semi) volatile compounds. The sample is injected into the GC-MS and seperated based on boiling point and affinity with the column. The separated components are than detected with a Mass Spectrometer resulting in a mass spectrum unique for a material. The obtained mass spectra are run through the NIST database to identify the detected components.

IMPACT TESTING (IZOD & CHARPY)

Brand	Zwick/Roell HIT5.5P
Temperature range	Room temperature
Configuration	IZOD (ISO 180 & and ASTM D 256) and Charpy (ISO 179)
Notcher	Karch Manual notching machine
Sample geometry	IZOD ISO 80x10x4 mm / ASTM 64x12.7x3.2 mm
Output	Impact energy, impact strength

The impact test is a standardised method for analysing the toughness or brittle-fracture sensitivity of a material.

Measurements are carried out with a Zwick/Roell HIT5.5P impact tester equipped with all hammers suitable for use with plastics. Measurements can be carried out at room temperature as per IZOD (ISO 180) or Charpy (ISO 179). The specimens can be notched with the help of a standardised notch-cutting machine.

The (notched) impact value is defined as the energy lost per unit of specimen thickness (at the notch).

INFRARED (IR) SPECTROSCOPY

Brand	PerkinElmer Frontier IR
IR range	600 to 4.000 cm^{-1}
Typical sample size	1x1cm or 10mg
IR modes	ATR (reflection) Transmission
Output	Molecular fingerprint

IR spectroscopy is used to record an IR spectrum of a material that is unique to that type of material, in other words a fingerprint of the material.
Measurements are carried out with a PerkinElmer Frontier FT-IR spectrometer.
IR spectroscopy can be used to identify an unknown organic material. Samples may vary from a small piece of a product (1x1cm), a powder (10mg), or even contaminants of up to 50 µm diameter. For this kind of identification, PTG/e has an extensive database of IR-spectra. IR analysis is also an excellent method for comparing different material batches as identical materials have an identical IR spectrum.

KARL-FISCHER TITRATION (KF)

Brand	- Metrohm 831 KF Coulometer - Metrohm Thermoprep 832 oven
Sample type	Any: solid, liquid, organic, inorganic
Typical sample size	10 mg to 5 g
Output	Water content

Karl Fischer titration is a technique used for measuring the water content of a sample. The sample can be an organic solvent or a solid. Measurements are carried out with a Metrohm 831 KF Coulometer in combination with a Thermoprep 832 oven. Solid samples can be measured over a temperature range of 30 to 250 °C. Karl Fischer titration allows accurate measurement of water content from ppb-level to 100%. Being a selective method, it reacts to water only.

MELT FLOW INDEX (MFI) TESTER

Brand	Karg MeltFlow Basic
According norm	ISO 1133
Temperature range	30 to 400 °C
Weights	2.160 or 5.000 g
Typical sample size	100 g (granulate)
Materials	PE, PP, PS
Output	MFI in g/10 min

The MFI is a measure of the flow rate of a polymer melt (expressed in grams per 10 minutes) at a standard temperature and a standard load, in conformity with ISO Standard 1133.

Measurements are carried out with a Karg MeltFlow Basic tester equipped with a range of loads.

An MFI measurement can be used to check if a material is on-spec with regards to the MFI specified in the data sheet. To gain more insight into the rheology of a polymer, a rheometer can be used.

NUCLEAR MAGNETIC RESONANCE (NMR) SPECTROSCOPY

Brand	Bruker Avance III HD
^{1}H Frequency	400 MHz
Typical sample size	10 mg
Nuclei	^{1}H, ^{2}H, ^{11}B, ^{13}C, ^{19}F, ^{29}Si, ^{31}P
Sample tube	Standard 5 mm diameter
Output	Structure elucidation Identification unknown samples Sample comparison

Solution NMR spectroscopy is a non-destructive analytical technique used for identification and quantification of a sample. The technique gives detailed structural information about the structure of organic compounds, including polymers. Chemical transformations can conveniently be monitored using NMR spectroscopy as well.

Measurements are carried out using a Bruker Avance III HD 400 MHz spectrometer. The most commonly used technique is proton NMR (^{1}H NMR), but other elements (see table) can also provide valuable structural information. For an accurate measurement, the sample must be dissolved in a suitable NMR solvent. PTG/e offers a variety of deuterated solvents for this purpose.

OPTICAL MICROSCOPY

Brand	Motic Stereo SMZ-168T-LED
Magnification range	7.5x to 50x
Lighting options	Transmitted/incident LED illumination Crossed polars
Output	Magnified image of the sample

A stereo microscope is a microscope with two objective lenses and two eyepieces, which makes it possible to visualise a sample in 3D. Measurements are carried out with a Motic Stereo SMZ-168T-LED microscope with a magnification range of 7.5x to 50x and the option to save the images.
A stereo microscope can be used to obtain a first impression of the surface of a sample and for sample preparation.

RHEOLOGY

Brand	DHR-2 TA Instruments
Geometries	8 – 60 mm parallel plate 60 mm cone-plate Torsion DMTA (for solids)
Temperature range	-140 – 600 °C (ETC oven) -20 – 200 °C (Peltier plate)
Sample types	(Viscous) fluids, polymer melts Solids (rectangular bars)
Sample size	5 g – 10 mL
Output	Viscoelastic behaviour of materials

Rheology measurements are used to gain insight into the viscoelastic behaviour of a material under different temperatures and shear velocities. A variety of measurement modes can be used, depending on the property of interest:

- Frequency sweep: storage and loss modulus, zero-shear viscosity
- Flow sweep (fluids only): viscosity as function of shear rate
- Time sweep: curing behaviour, gelation, drying, thermal stability
- Strain sweep: determination of Linear Viscoelastic Regime (LVR), material robustness
- Creep and/or relaxation behaviour
- Extension of measurement range by Time-Temperature Superposition (TTS)

SCANNING ELECTRON MICROSCOPY (SEM)

Brand	Quanta 3D FEG
Magnification	30x to 1.000.000x
Combined with	Energy Dispersive X-Ray (EDX) for elemental analysis.
Typical Sample size	<1 µm - 2 cm
Output	- High resolution images - Present elements

An electron microscope produces images of the surface of a sample with the help of an electron beam. This technique enables much larger magnifications than are possible with an optical microscope. Measurements are carried out with a FEI Quanta 3D FEG SEM, which enables magnifications of 30x to 1,000.000x with a resolution of 2 nm. In addition, an EDX (Energy Dispersive X-ray) probe is available for identifying the elements present in the sample. SEM analysis makes it possible to visualise sample surfaces in great detail. Furthermore, (very) small impurities or foreign elements in a sample can be visualised and identified by means of elemental analysis.

SHORE HARDNESS

Brand	PCE instruments PCE-DD
Possible scales	Shore A and Shore D
Typical sample size	> 6 mm thick > 33x33 mm wide Flat surface
Hardness range	0-100
Output	Hardness (Shore A or D)

The Shore Hardness scale indicates the resistance of a material to indentation. A Shore durometer has a needle on a spring which is placed against the material and pressure is applied. Once the durometer is pressed firmly against the material and the needle has penetrated as far as it can go, the corresponding shore hardness can be read from the display.

Shore A is used for softer materials like elastomers, soft rubbers, polyesters, resins and leather.
Shore D is used for harder materials like thermoplastics, fibers and hard rubbers.

SIZE EXCLUSION CHROMATOGRAPHY (SEC)

Brand	SEC Waters 2695
Eluentia	Tetrahydrofurane (THF) or Hexafluoroisopropanol (HFIP)
Detectors	Refractive index / UV-VIS
Typical sample size	2 mg
Output	Average molecular weight Polydispersity index

GPC or SEC is used to determine the average molecular weight (Mn and Mw) and the polydispersity index (Mw/Mn) of a polymer material. These properties of a polymeric material have a strong influence on the material properties (physical, mechanical and processing properties). The results of a SEC measurement can therefore be useful in explaining certain physical or mechanical properties of a material specimen. Complementary to NMR and Infrared spectroscopy, SEC analysis is an excellent technique for comparing different material batches.

HIGH TEMPERATURE
SIZE EXCLUSION CHROMATOGRAPHY (HT-SEC)

Brand	Polymer Char GPC-IR® HT-SEC
Eluentia	1,2,4-trichlorobenzene (TCB)
Detectors	IR-4 dual wavelength infrared
Typical sample size	20 mg
Output	Average molecular weight Polydispersity index

HT-GPC or HT-SEC is employed to determine of the average molecular weight (Mn and Mw) and the polydispersity index (Mw/Mn) of highly crystalline polymers which show low solubilities in common organic solvents at ambient temperature.

Therefore, HT-SEC is routinely applied to polyolefins including polyethylenes (HDPE, LDPE, and LLDPE), polypropylene (PP), ethylene-Propylene (EP) copolymers, poly(ethylene-vinyl acetate) (EVA) and poly(ethylene-butyl acrylate) (EBA)).

Measurements are carried out in 1,2,4-trichlorobenzene (TCB) at 160 °C.

TENSILE TESTING

Brand	Zwick Zmart.Pro
Temperature range	Room temperature
Loadcel	100 N and 10 kN
Speed	Max 500 mm/min
Modes	Tensile, compression, 3 point bending
Sample preparation	Dumbel cutting press (ISO 527-2 A & B)
Output	Stress-strain, E-modulus (stiffness), strength, yield, creep, hysteresis, ...

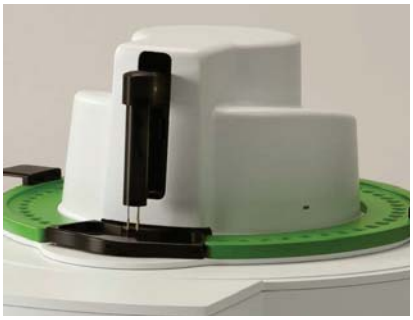
A tensile tester is used to determine the elongation, compression and bending characteristics of a material.

Measurements are carried out with a Zwick Zmart.Pro 10 kN tensile tester, among others. Customers can provide their own sample specimens, but PTG/e has a cutting press for producing the dumb-bell shaped specimens from a film or coating. These specimens are tested as specified in ISO 527.

The stress-strain curve as well as all raw data can be supplied.

THERMOGRAVIMETRIC ANALYSIS (TGA)

Brand	PerkinElmer TGA4000
Temperature range	35 to 1.000 °C
Typical sample size	10 mg
Type of TGA pans	Ceramic sample pans
Atmosphere	Nitrogen or Air
Output	Mass loss Ash content Volatile content Degradation onset



TGA is used to determine mass loss of a material as a function of temperature.
Measurements are carried out with a PerkinElmer TGA 4000, which allows measurements over a temperature range of 35 to 1,000 °C in nitrogen or in air. A material sample of just 10 mg is sufficient for a measurement.
TGA measurements can, among other things, be used to determine mass loss in a material due to evaporation or degradation. It can also be used to determine the amount of inorganic filler in a material.

TGA - IR HYPHENATION

Brand	PerkinElmer TGA4000 & PerkinElmer Frontier IR
Temperature range	35 to 1.000 °C
Typical sample size	10 mg
Type of TGA pans	Ceramic sample pans
Atmosphere	Nitrogen or Air
Output	Identification of gasses during TGA measurement

The two techniques of TGA and IR spectroscopy can be combined to obtain additional information on a material.
Measurements are carried out with a PerkinElmer TGA 4000 coupled to a gas cell in a PerkinElmer Frontier FT-IR spectrometer. Samples can be measured over a temperature range of 35 to 1,000 °C in nitrogen or air, with the gases released being measured online in the IR spectrometer.
The method can be used to determine whether mass loss in a sample has to do with evaporation of specific components or is the result of degradation. In the case of multi-component systems, the order of evaporation can also be established.

TGA – IR – GCMS HYPHENATION

Brand	PerkinElmer TGA4000 & PerkinElmer Frontier IR & PerkinElmer Clarus 690 GC & PerkinElmer SQ8T MS
Parameters	See cards individual technique
Obtained results	Identification of gasses during TGA measurement via IR GCMS measurement at selected temperatures

With this setup, it is possible to combine the analysis techniques TGA, FT-IR and GC-MS via coupling of the devices with a transfer line. The TGA – IR – GC-MS 'Hyphenation setup' is a powerful analysis technique to identify complex and unknown materials. Evolving gasses from the TGA are transported via a transfer line through an IR chamber. The gasses can be collected at any given time during the measurement to be injected in the GC-MS. The coupling can be varied to use the setup partially to improve it's applications. Some of the applications of the hyphenation technique are:

- The identification of additives, like plasticizers, in plastics
- Determination of the primary components of a material
- Analysis of unknown contaminations in a material, like fragrances or solvents

X-RAY FLUORESCENCE (XRF)

Brand	Malvern Panalytical - Epsilon 3 XLE
Principle	Energy dispersive X-ray fluorescence (EDXRF)
X-Ray Tube	High-performance ceramic tube, 50KV 3mA
Elemental range	Beryllium (Be) to uranium (U)
Limit of detection	1 ppm - 100%
Output	Elemental analysis, Contaminant detection and analysis, Elemental quantification.

X-ray fluorescence (XRF) is an analytical technique that can be used to determine the chemical composition of a wide variety of sample types including solids, liquids, slurries and loose powders. X-ray fluorescence is also used to determine the thickness and composition of layers and coatings. It can analyze elements from beryllium (Be) to uranium (U) in concentration ranges from ppm to 100% levels.

X-RAY PHOTOELECTRON SPECTROSCOPY (XPS)

Brand	Thermo Scientific K-Alpha
Characteristics	Analysis of top 0-10 nm of the surface; Equiped with depth profiling by means of argon ion sputtering and stage tilting
Specimen	Max. specimen size: Regular XPS 60 (w) x 60 (l) x 20 (d) mm; AR-XPS 8 (w) x 28 (l) x 5 (d) mm
Output	Elemental composition of surface

XPS analysis is a surface-sensitive quantitative spectroscopic technique that typically measures the top 0-10 nm of a sample surface. Elements from Lithium (Li) upwards in the periodic table can be measured.

Measurements are carried out with a Thermo K-Alpha XPS. For the measurements, the sample has to be placed in ultra-high vacuum.

XPS technique can be used to measure elemental composition of surface. The atomic concentrations of the sample are determined. This information is then used to derive the empirical formula of pure materials. It can be used to determine the chemical states of each element in the material. Line profiling and mapping across the top surface can also be realized.

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