



CENTRAL LABORATORY

R&D Education and Measurement Center METU, Ankara

Polymer Analysis Laboratory (PAL)

The measurement of properties such as the elemental composition of materials synthesized in both university and industrial laboratories, the molecular weight distribution of polymers, and their hydrodynamic sizes is crucial for the progress of research. In our laboratory, we have the capability to perform these measurements using LECO TruSpec Micro Elemental Analysis (EAC), Malvern OmniSEC Size Exclusion Chromatography, and Malvern ALV/CGS-3 Dynamic Light Scattering (DLS) instruments.

BASIC PRINCIPLES

ELEMENTAL ANALYSER (EAC): The working principle of the device can be defined in three separate steps. In the first stage, the sample is placed into a tin (Sn) capsule and oxidized by oxygen (O₂) gas. The resulting gas mixture is then separated in a section containing carrier inert gas, helium (He), and special reagents, where elemental carbon, hydrogen, nitrogen, and sulfur are converted into CO₂, H₂O, N₂, and SO₂, respectively. The resulting and separated gas mixture is then passed through copper (Cu) initially to remove excess oxygen and then directed to different detectors.



Elemental Analysis

- Polymers
- Environmental samples (soil, sediment, etc.)
- Coal samples
- Pharmaceuticals
- Proteins
- Industrial products
- Organic compounds

Device Usage Settings:

- Furnace Temperature: 950°C
- Sample Amount: 2-2.5 mg
- Analysis Time: ~200 seconds

After standard calibration, 2-3 consecutive measurements are taken, and the average is reported as weight percentage.

EDTA (std)	C%	H%	N%
1. Measurement	40,96	5,48	9,50
2. Measurement	40,87	5,53	9,55
Standart Measurement	40,94	5,54	9,57

Sülfametazin (std)	C%	H%	N%	S%
1. Measurement	51,73	5,10	20,10	11,55
2. Measurement	51,80	5,09	20,12	11,52
Standart Measurement	51,78	5,07	20,13	11,52

DYNAMIC LIGHT SCATTERING SPECTROMETER (DLS): It is based on measuring the intensity and changes in the scattered light from small particles in a dilute solution. The variation in the intensity of the scattered light depends on the movement of the particles, and consequently, on the size of the particles, the viscosity of the medium, and the temperature. Colloidal suspensions are characterized using two fundamental light scattering methods.

The Static Light Scattering (SLS) method is used to determine the molecular weight, gyration radius, and second virial coefficient of polymers.

The Dynamic Light Scattering method is used to obtain hydrodynamic size, diffusion coefficient, distribution index, and particle size distribution. The combination of these two techniques provides information for determining the structure of particles in solution.

In this method, high-intensity monochromatic light is passed through a solution containing macromolecules, and the intensity of scattering is detected from one or more points.



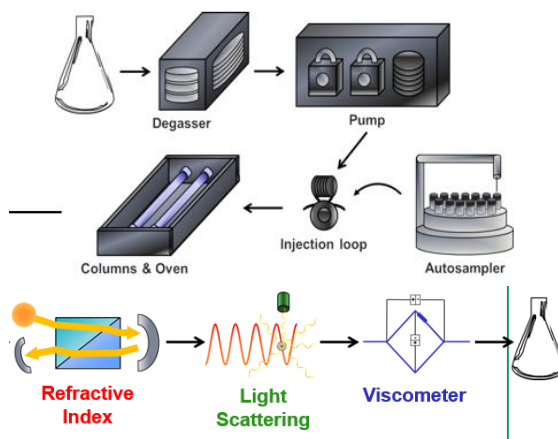
used as the solvent, and samples need to be homogeneously dissolved in THF. The flow rate is set at 1 mL/minute.

In the Reveal section, there is a three-detector module to characterize synthetic and natural polymers and proteins:

- Highly sensitive light scattering (RALS and LALS) detector: It operates at 90 degrees for RALS and 7 degrees for LALS, using a laser with a wavelength of 640 nm.
- Viscosity detector: It operates with a 4-capillary Wheatstone bridge.
- Refractive index detector.

The temperature of the module containing detectors is fixed at 35°C. Molecular weight can be measured in the range of 200-2x10⁶ Da using Triple Detection. When light scattering detector is not suitable for the samples, Universal Calibration (UC) method is used with RI and viscosity detectors instead of Triple Detection for comparison with polystyrene. Molecular weight can be measured in the range of 1200-4.2x10⁶ Da using UC method.

SEC Instrument Schematic

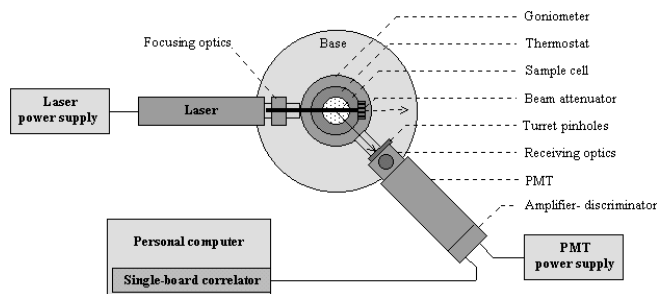


Size Exclusion Chromatography

- Colloidal Systems
- Macromolecules
- Proteins

For Application:

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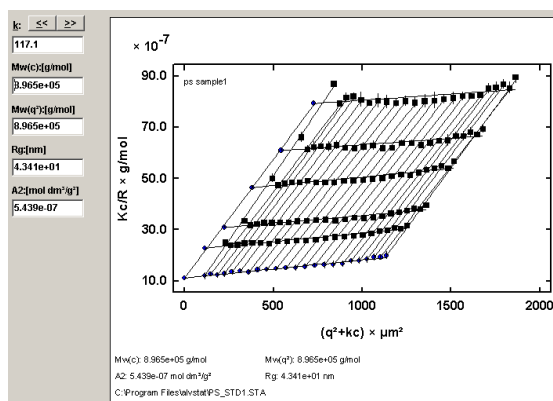


This method's greatest advantage is its applicability to a wide range of macromolecules without requiring rigorous pretreatment. However, it is highly sensitive to factors such as dust, air bubbles, etc., and may necessitate centrifugation, filtration, and dilution.

Dynamic Light Scattering:

- Polymers
- Biopolymers
- Samples with particle sizes ranging from 1 nm to 3 μm

Static Light Scattering Spectrometer (SLS)



GEL PERMEATION CHROMATOGRAPHY (GPC): It is a chromatographic method that separates molecules based on their sizes. This method is used to determine the molecular weight and molecular weight distribution of polymers quickly and accurately.

The device consists of two sections: Resolve and Reveal. In the Resolve section, there is a pump, degasser, sample stand, and column oven where separation takes place. The column oven temperature is fixed at 35°C, with a guard column and 2 Tx6000M columns. Tetrahydrofuran (THF) is