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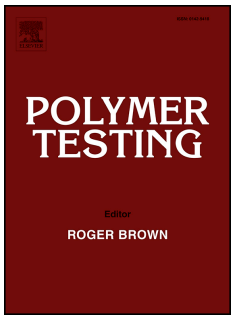


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# Characterization of broad molecular weight distribution polyethylene with multi-detection gel permeation chromatograph

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**Abstract:** High temperature gel permeation chromatograph (GPC) equipped with differential refractive index, light scattering and viscometer is used to characterize polyethylene with broad molecular weight distribution (MWD). Methods are discussed through the measurement of broad MWD polyethylene PE52K and 4731B, including Mark-Houwink calibration, universal calibration, triple-detectors and light scattering dual-detectors calibrations. As the broadness of MWD increases, the two fitting curve calibrations with first order equations are chosen, despite a slight inaccuracy within molecular weight range of standards. More reliable polymer dispersity indexes (PDI) are obtained by triple-detectors and light scattering dual-detectors calibrations. From the Mark-Houwink plot of 4731B, branching information can only be obtained from triple-detectors calibration, indicating the validity of triple-detectors calibration on the characterization of branched polyethylene with broad MWD. The triple detectors calibration upon 4731B provides a weight average molecular weight of 313 kg/mol, a PDI of 47 and a branching of 0.30 per chain.

**Key words:** multi-detectors gel permeation chromatograph, calibration, polyethylene, broad molecular weight distribution

## 1. Introduction

Due to low production costs, remarkable physical properties and wide range of applications, polyolefins become the most important commodity plastics and rubbers [1]. The molecular weight distribution (MWD) influences the physical properties of polyolefins directly and is a prime parameter to characterize a polyolefin [2]. When compared to narrow MWD polyethylene, polyethylene with broad MWD has excellent processing and mechanical properties. The molecules in the range of larger molecular weights are responsible for mechanical strength and those in the range of lower molecular weights for rheological properties [3]. For example, studies claimed that polymer dispersity index (PDI) was related to the zero shear viscosity and shear thinning effect [4, 5]. Thus, it is important to accurately characterize the MWD of polyethylene with broad MWD.

Multi-detectors gel permeation chromatography (GPC) is commonly used to characterize MWD of polyethylene. Hyphenated detectors are differential refractive index (RI), viscometric detection (Visco) and light scattering detection (LS, right angle light scattering and low angle light scattering). The method to extract MWD information from the elution curves of GPC is to establish a calibration. Common GPC calibrations involve conventional calibration, Mark-Houwink calibration, universal calibration, triple-detectors calibration and light scattering dual-detectors calibration [6]. The accuracy and precision of MWD measurement is significantly correlated to GPC calibration. Netopilik [7] analyzed GPC elution curves of polystyrene-*graft*-polystyrene (PS-*g*-PS) with conventional and triple-detectors calibrations respectively, resulting in different weight average molecular weights ($M_w$). This occurs because PS-*g*-PS has a different chemistry and architecture with respect to the standard polystyrene (PS), leading to some inaccuracy of conventional calibration. Hazlitt [8] noticed that MWD determined by various calibrations presented changes, which were eliminated by the systematic approach for offset determination coupled with a robust axial dispersion correction. Baudilio [9] used GPC with four detectors to characterize ethylene/propylene copolymers and obtained MWD by universal calibration. Langston [10]

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