



# Agilent 1260 Infinity II HT-ELSD (G7826A) - For Molecular Weight Determination

## Technical Note

The 1260 Infinity II HT-ELSD (G7826A) can provide a linearized analogue output which allows the detector to be used as a linear concentration detector replacing refractive index (DRI) or InfraRed (IR) in HT-GPC applications. The main advantage of using this detector for this application is the significant increased sensitivity of the high temperature evaporative light scattering detector (ELSD) compared with the DRI and IR; typically, the ELSD is 30 times more sensitivity than a DRI. This increased sensitivity enables detection of much lower concentration which is essential for the analysis of UHMWPE samples.

## Determination of the Power Function

The ELSD signal in the function of concentration is linearized, based on a power law function:

$$ELSD \text{ signal intensity} = k_{ELSD} * \text{concentration}^{(\alpha_{ELSD})}$$

The exponent,  $\alpha_{ELSD}$ , defined by LinF in the instrument interface is solvent dependent but NOT polymer type dependent.

In [Table 1](#) on page 2 below are predetermined Power function for common high temperature GPC solvents.



**Table 1** ELSD parameters for different solvents for HT-GPC of polyolefins

Solvent	Polymers	Power	References
TCB	PS, PE, PP	1.61	[ <sup>1</sup> , <sup>2</sup> ]
o-DCB	PS, PE, PP	1.20	
butylal	PS, PE, PP	1.35	[ <sup>3</sup> ]
xylene	PS, PE, PP	1.40	[ <sup>4</sup> ]

If a new solvent is used that doesn't have a predetermined Power function then you'll need to measure the area response for several concentrations of accurately prepared solutions in the range 5 – 500 µg/ml [<sup>5</sup>]. Plot the response against concentration and fit the data to a power function as described in reference [<sup>2</sup>]. The power coefficient which best fits the data is the one to be used as LinF parameter for the respective solvent and test conditions.

- <sup>1</sup> A. Boborodea, S. O'Donohue, New evaporative light scattering detector for high temperature gel permeation chromatography, , Int. J. Polym. Anal. Charact., vol 22, no 7, 631-638 (2017)
- <sup>2</sup> A. Boborodea, S. O'Donohue, Linearization of evaporative light scattering detector signal, Int. J. Polym. Anal. Charact., vol 22, no 8, 685-689 (2017)
- <sup>3</sup> A. Boborodea, A. Brookes, Polyolefin characterization in dibutoxymethane by high temperature gel permeation chromatography with a new evaporative light scattering detector, Polym. Test., vol 64, 217-220 (2017)
- <sup>4</sup> A. Boborodea, F. Mirabella, S. O'Donohue, Polyolefin Characterization in Xylene by High-Temperature Gel Permeation Chromatography with a New Evaporative Light Scattering Detector, Chromatographia, vol 81, no 3, 419-424 (2018)
- <sup>5</sup> A. Boborodea, A. Brookes, Investigation of sample preparation for high temperature gel permeation chromatography using a low solvent consumption method, Polym. Test., vol 63, 210-213 (2017)

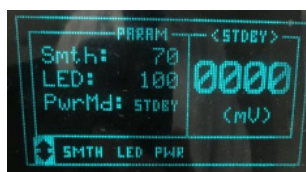
## Setting Up the Instrument

Once the power function has been determined for the operating solvent it can be entered on the instrument via the second sub-screen as shown below:

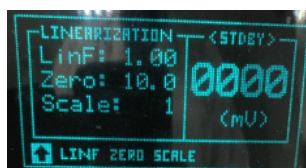
Main Screen



Sub-screen 1



Sub-screen 2



Navigation between the screens is achieved by the arrows on the keypad when the cursor is on the arrow.

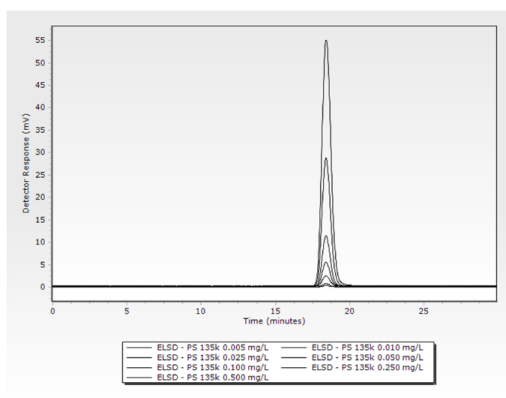
Sub-screen 2 allows changes to the following electronic parameters:

		Default	Range
LinF	Linearization function	1.0	1.00 – 2.00
ZERO	Baseline position the instrument is zeroed to	10	0.1 – 10
SCALE	Scaling factor to increase peak height after linearization function applied	1.0	1 – 35

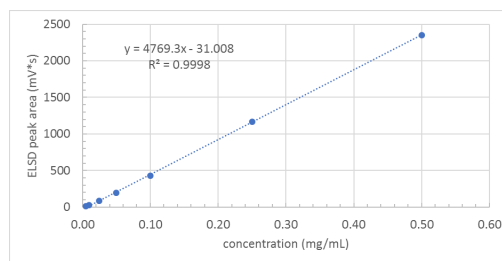
Once the LinF has been set for the solvent being used, each data point is corrected by the following equation:

$$\text{Corrected ELSD} = (\text{ELSD}_{\text{Signal}})^{(1/\text{LinF})}$$

The net effect is that signal is linear (see figure below), but the apparent signal sensitivity is reduced and therefore to compensate for this loss in sensitivity the Scale can be applied to amplify the signal again.



**Figure 1** Overlay of PS 135k chromatograms, concentrations: 5 to 500 µg/ml



**Figure 2** Peak area versus concentration linear correlation over two orders of magnitude

To obtain correct molecular weights the zero offset value applied needs to set the baseline as close to zero as practically possible without the data be at or below zero or the calculation will just return zero and thus loss of data. For TCB, the recommended Zero value is 0.1, but this value can be slightly increased up to 1 for solvents presenting a higher level of baseline noise.

To ensure that the baseline is set to the zero offset value before each run we advise using Cable (G7826-60015) with the Agilent 1260 Infinity II HT GPC System which is connected between the Inj Sync at the back of the control drawer and the Aux I/O port at the rear of the HT-ELSD prior to each run. The same can be achieved for third party users by using Cable (0890-0055) with both Inject Sync and Autozero lines connected to the injector.

## Example – Analysis of SRM1475a

### Experimental Conditions

Agilent 1260 Infinity II High Temperature GPC System (G7820B) interfaced to an Agilent 1260 Infinity II HT-ELSD (G7826A).

**Table 2** Chromatography Conditions

Mobile phase	1,2,4 Tricholobenzene + 250 ppm BHT
Columns	3 x PL-Olexis Columns, 7.5 mm x 300 mm
Column Oven temperature	160 °C
Flow Rate	1.0 mL/min
Injection volume	200 µL
Concentration	1 mg/ml for SRM PE 1475a
Calibration Standards	EasiVials PS standards PS-H

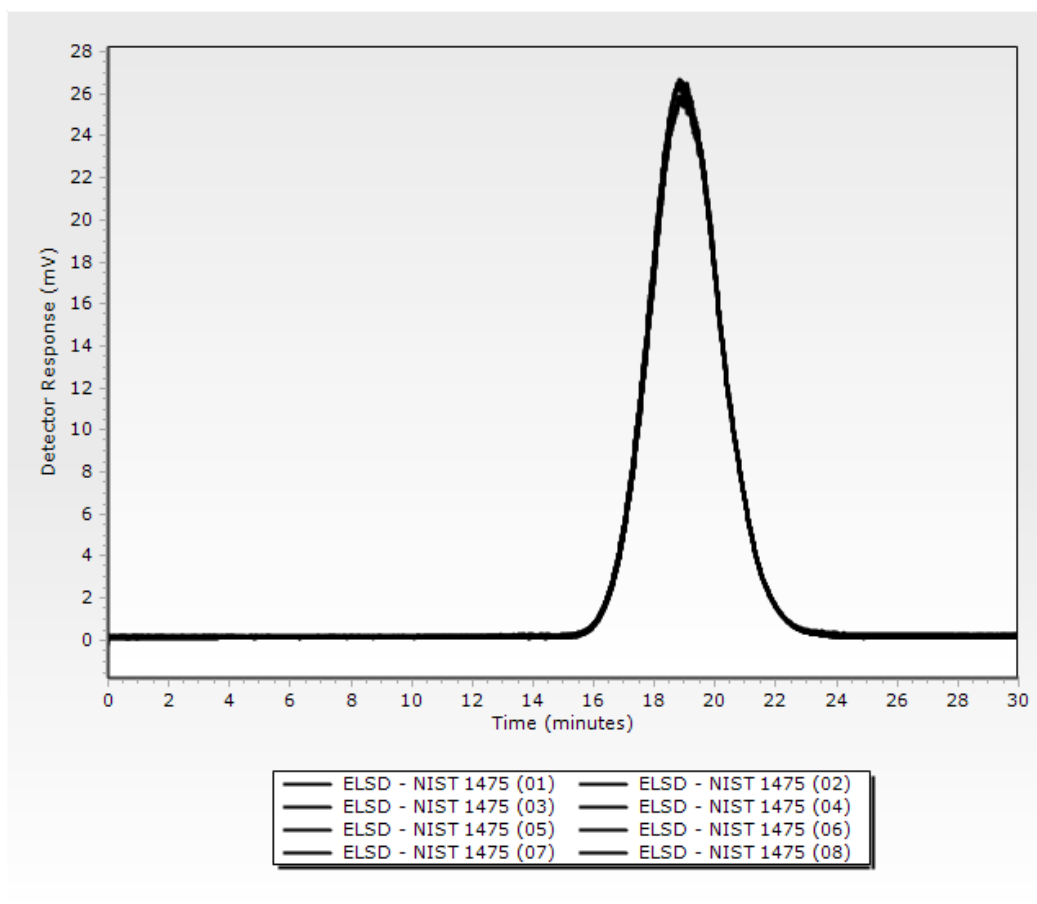
**Table 3** Detector Conditions

Xfer (Transfer line temperature)	160 °C
Neb (Nebuliser temperature)	160 °C
Evap (Evaporation temperature)	120 °C
Gas (Gas flow rate)	0.4SLM
Smth (smoothing factor)	60
LED (Light source power)	100 %
LinF	1.61
Zero	0.3
Scale	1

## Analysis of PE SRM 1475 sample

Eight analyses were performed on PE SRM 1475 sample during several days, using the same setting for the power coefficient for ELSD,  $\text{LinF} = 1.61$ .

The overlay of the obtained chromatograms in [Figure 3](#) on page 6 shows an excellent reproducibility:



**Figure 3** Overlay of chromatograms of PE SRM 1475 sample

The average molecular weights, presented in Table 4 on page 7 are in excellent agreement with the expected values provided by PE SRM 1475 certificate (Mn = 18310, Mw = 53070 g/mol, Mz = 138000 g/mol).

**Table 4** Average molecular weights determined from the HT-ELSD chromatograms using conventional GPC and polystyrene calibration standards (Easivial PS-H)

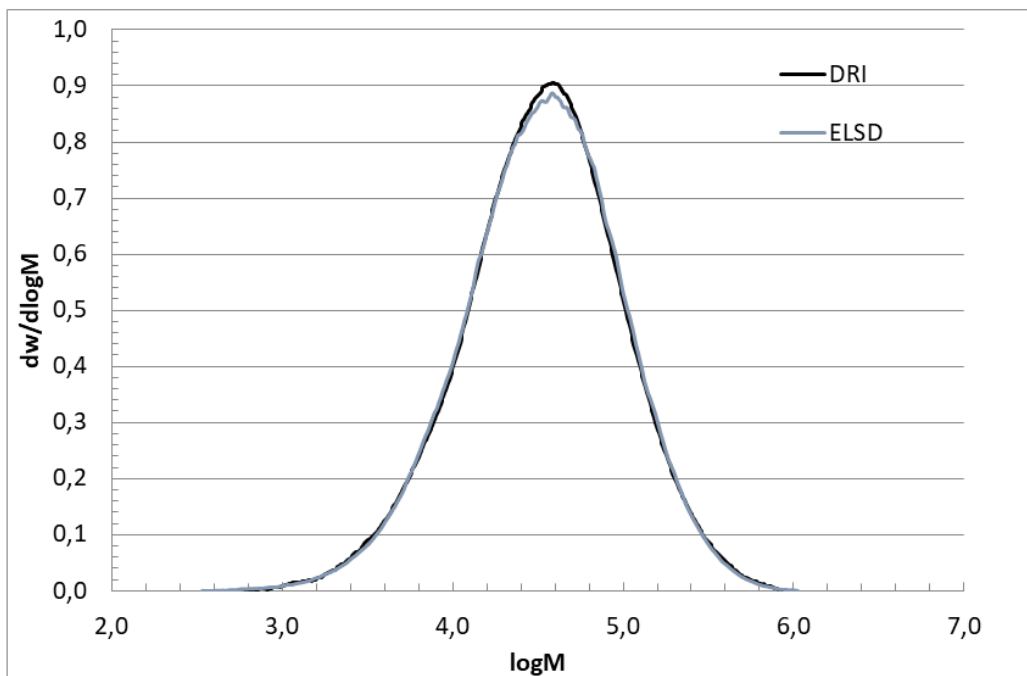
Injection	Mn (g/mol)	Mw (g/mol)	Mz (g/mol)
NIST 1475 (01)	18242	57925	145027
NIST 1475 (02)	18353	57648	144716
NIST 1475 (03)	18314	57537	143238
NIST 1475 (04)	18411	57434	142142
NIST 1475 (05)	17960	56259	138325
NIST 1475 (06)	17950	56369	140288
NIST 1475 (07)	18044	56323	139180
NIST 1475 (08)	18170	56434	139052

The following Mark Houwink-Sakurada parameters were used in the calculation to convert the Polystyrene equivalent molecular weight values to Polyethylene values:

- $KPS = 19 \cdot 10^{-3} \text{ mL/g}$ ;  $\alpha_{PS} = 0.655$
- $KPE = 39 \cdot 10^{-3} \text{ mL/g}$ ;  $\alpha_{PE} = 0.725$

**Example – Analysis of SRM1475a**  
Analysis of PE SRM 1475 sample

The linearized ELSD signal is independent of molecular weight and this is demonstrated by the excellent agreement between the MWD obtained with classical DRI detector and the one given by ELSD, as presented in [Figure 4](#) on page 8:



**Figure 4** Comparison between MWD obtained with DRI detector and ELSD in TCB at 160°C for PE SRM 1475 sample



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