Electronic Supplementary Material (ESI) for RSC Advances.

Supporting Information

Improving chromatographic separation of polyolefins on porous graphitic carbon stationary phases: Effects of adsorption promoting solvent and column length

Anthony Ndiripo, Andreas Albrecht[a], Harald Pasch*

Department of Chemistry and Polymer Science, Stellenbosch University, Stellenbosch, South Africa.

[a]Borealis Polyolefine GmbH, St. Peter-Strasse 25, Linz 4021, Austria.

*Corresponding Author(s): hpasch@sun.ac.za

Important considerations when using the PL-ELS 1000 ELSD detector

From our experience, two factors that can affect resolution include detector age and cleanliness. Since the PL ELS 1000 detector uses nebulizing gas perpendicular to mobile phase flow as illustrated in Fig. S1, the alignment of the nebulizing outlet and eluent outlet (mobile phase) capillaries tends to decrease over time due to repeated heating and cooling (see direction indicated by blue arrows). As a result of the misalignment, a fraction of the particle plume will not reach the detection compartment and some material sticks onto the evaporator tube. Sometimes this can be identified by fumes leaking via the drain instead of the exhaust.

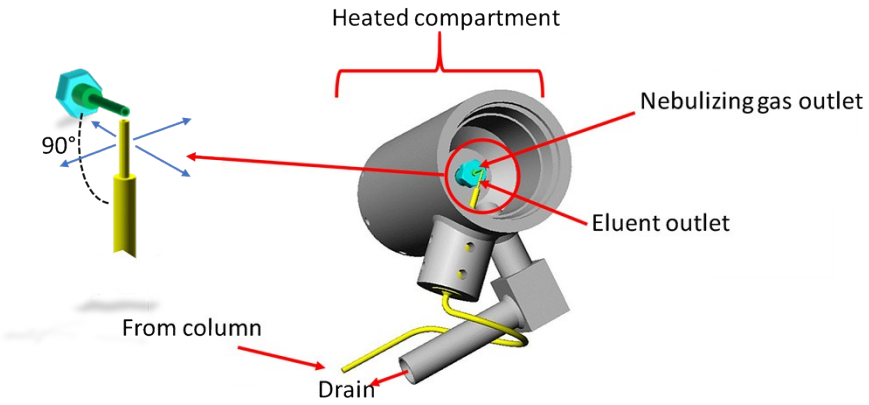

Figure S1. Diagram showing the ELSD nebulizer set-up.

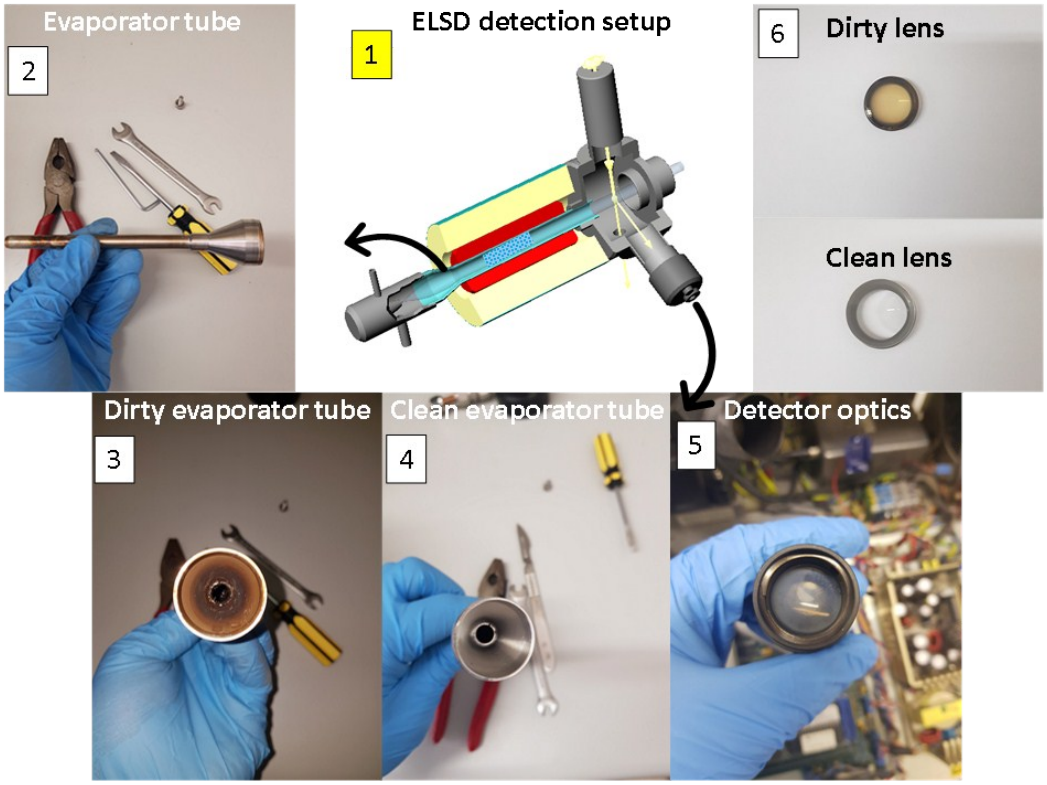

Figure S2. Diagram showing the ELSD setup (1), the evaporator tube (2), a dirty evaporator tube with accumulated polymer residue (3), a clean evaporator tube (4) the detector optics with a dirty lens (5), and a comparison of dirty and clean lenses (6).

Over time, polymer material accumulates at the head of the evaporator tube as well as at the diffuser. The material can create turbulence and promotes further sticking of polymer material onto the evaporator tube. Turbulence can result in dwelling of the particle plume in the detector resulting in poor resolution of eluting components. Again, polymer particles can stick to the detector lenses resulting in a loss of detector response over time. Fig. S2 shows the differences between the dirty and clean detector components which give rise to the mentioned challenges when using the ELSD for high temperature purposes. It is important to mention the loss of detector response over time since this affects the observed peak height and width which are essential for calculating resolution. Fig. S3 shows the changes in the detector response before and after cleaning. A decane signal was used to measure the detector response by injecting solvent from the first dimension into a PL Rapide column in the second dimension. A detailed description of the two-dimensional set-up is given in literature.[1-3]

In addition, the conditions in the detector can promote fraction mixing resulting in loss of resolution. In the present work, these issues were eliminated before commencing all experiments.

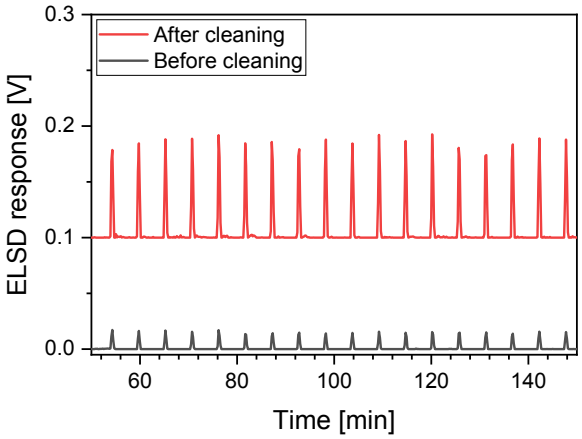

Figure S3. Comparison of ELSD signals obtained before and after cleaning the evaporator tube. 100 µL of decane was injected into the second dimension at 2 min intervals using an 8-port valve. 1,2-dichlorobenzene was used as the second-dimension mobile phase at a flow rate of 2.75 mL/min.

References

1. A. Ndiripo, M.-M. Pornwilard, T. Pathaweeisariyakul and H. Pasch, *Polym. Chem.*, 2019, **10**, 5859–5869.
2. A. Ndiripo and H. Pasch, *Anal. Chim. Acta*, 2018, **1027**, 137–148.
3. A. Ndiripo and H. Pasch, *Anal. Chem.*, 2018, **90**, 7626–7634.