Moving from TCB to oDCB in CRYSTEX QC

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CRYSTEX QC is a technique for determination of soluble fraction in polypropylene, both homo polymers and copolymers (also applicable to other polyolefins), which has been recently included as alternative method in the ISO 16152. This technique is implemented in manufacturing facilities to support the process control and also for QC of the product, as well as in R&D and central laboratories supporting product development activities and pilot plant or lab scale reactor monitoring and control.

A separation by crystallinity is realized in a column, following a simplified TREF approach, for which polymer solutions are prepared and handled within the automated instrument. Due to its good properties as solvent for polyolefins and the good IR transparency 1,2,4-trichlorobencene (TCB) was the solvent of choice for use with CRYSTEX QC, even if other solvents are also possible. However, TCB is known to be a hazardous solvent for the environment besides being toxic for humans and therefore it was restricted for most applications under the REACH listing of the European Chemicals Agency. For those reasons both the supply and the disposal of TCB is becoming more difficult and expensive, bringing the laboratories to a critical situation, unable to proceed with their work.

One feasible and practical solution to supply shortages is the use of ortho-dichlorobencene (o-DCB) which is already considered a standard solvent for chemical composition distribution methods (based on crystallization or interaction chromatography), in addition to be already the solvent of choice for HT-GPC of polyolefins in many countries. This makes the replacement of TCB with o-DCB very convenient, although some important implications must be understood.

One important consideration is the impact of the solvent change on the detection after separation. When using o-DCB as the mobile phase, interferences of chemical composition and concentration level in the IR detector response are found which can be compensated for by applying proper calibration procedures in the software. In addition to that, lower sensitivity in the detection of ethylene content in ethylene-propylene copolymers was observed with o-DCB, and a new enhanced IR detector was developed to allow reliable and accurate quantification.

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