

Polybrominated biphenylethers in plastics: analytical state of the art

Thomas PJ Linsinger, Almuth Liebich

The Directive 2002/95/EC on the "Reduction of the use of certain hazardous substances in electrical and electronic equipment" (RoHS) bans from 1st July 2006 the use of Pb, Cd, Hg, Cr(VI) and certain polybrominated flame retardants in electric and electronic devices unless no technical substitutes exist. At present an upper limit of 1 g/kg has been set for PBBs and PBDEs per compound.

Certified reference materials (CRMs) are a prerequisite for quality assurance in analytical chemistry. CRMs exist for most of the relevant analyte/matrix combinations of the RoHS substances, with the notable exception of polybrominated flame retardants. The European Commission's Institute for Reference Materials and Measurements (IRMM) has therefore decided to develop a set of two certified reference materials for PBBs and PBDEs in polymers.

An intercomparison was organised to evaluate the analytical state of the art for the determination of pentaBDE, octaBDE, decaBDE and octaBB in polymers. Commercially available poly(ethyleneterephthalate) (PET) was spiked with technical mixtures of the analytes as well as Sb₂O₃ and homogenised by several extrusion cycles. The spiking level chosen was close to the legal limit for each technical mixture. The granules were mixed again and filled into glass vials. Homogeneity of the materials was tested using XRF. Between-bottle heterogeneity was quantified as 0.32 % and therefore negligible.

Samples were distributed to 63 laboratories. Each laboratory received two bottles of the materials. Three replicate determinations per bottle had to be performed. In addition, participants in Europe received a solution of technical PBB and PBDEs to see if a common standard would improve results. Participants were supposed to report on individual congeners. In addition, laboratories were asked to give details of their analytical method. Results will still be accepted until the end of this year.

This contribution will present the result of this intercomparison. It shall focus on the number of outliers as well as within and between-laboratory variation. Other factors for evaluation will be the agreement on congener identification as well as a check whether a common calibrant would improve the result. Last but not least, an attempt will be made to identify the most important factor influencing the accuracy of results and the results of this intercomparison will be compared to the one organised by the German Federal Environment Agency for the validation of the method for penta- and octaBDE [1].

1 S. Kemmlin, M. Bergmann, O. Jann (2005) Probenaufbereitungs- und Analyseverfahren für Flammschutzmittel (Pentabromodiphenylether, Octabromodiphenylether) in Erzeugnissen, Umweltbundesamt, Dessau, 2005, ISBN0722-186x