

**Validation of the TD-GC/MS method for the determination of decabromo diphenylether (DeBDE) in brominated fire retardants in polymeric materials**

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Obtaining accurate data on the levels of polybrominated diphenylether (PBDE) in consumer products is a challenging task. The difficulty can be attributed to two factors. First of all, PBDEs are normally solvent extracted from the matrix, a process which is time consuming, expensive and often leads to poor recoveries. The second source of error is especially due to the nature of very high boiling points of DeBDE in the PBDEs of which retention index (RI) is more than 4,200. These two factors often result in unduly low analytical precision and force the analyst to use a number of surrogates, internal standards and calibration check samples, in order to achieve the desired data quality.

In 2006, the National Institute of Advanced Industrial Science and Technology (AIST) in Japan sponsored a "round robin" survey of laboratories routinely analyzing polymeric samples for Decabromodiphenyl ether (DeBDE). The DeBDE concentration in the polystyrene test sample was 317ppm. Samples were provided to a number of analytical laboratories performing the determination of PBDEs using the prescribed RoHS method which is based on Soxhlet extraction and GC/MS analysis. The results of the study clearly demonstrated the shortcomings of the RoHS method. As practiced, the results obtained using the RoHS method were inaccurate and the precision was greater than 50%. The sample was also analyzed using a method based upon thermal desorption (TD) of the DeBDE from the matrix followed by GC/MS analysis<sup>1,2)</sup>.

The TD-GC/MS method requires no sample preparation. The sample is analyzed as received. This eliminates the tedious extraction step and avoids errors normally associated with solvent extraction. A special column is used for the analysis which is designed to elute DeBDE under 300°C eliminates thermal degradation during the separation step...

This work will present a summary of the TD-GC/MS method, the calibration curve, and the AIST standard results<sup>3)</sup>. Factors influencing the accuracy and precision of the DeBDE analysis will be discussed.

1) A.Hosaka, C.Watanabe and S.Tsuge, *Analytical Sciences*, 21, p1145, 2007

2) C.Watanabe, A.Hosaka and S.Tsuge, 17<sup>th</sup> Analytical Pyrolysis Symposium in Hungary, 2006

3) T.Yuzawa, C.Watanabe and S.Tsuge, under contribution