

## Enhanced Results with a Reduced Amount of Sample: Improved Performance on a Magnetic Sector Mass Spectrometer

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### Introduction

Polychlorinated dibenzo-p-dioxins and polychlorinated dibenzofurans (PCDD/F) are environmental contaminants, from which the 2,3,7,8 substituted congeners are assumed to be the most toxic<sup>1,2</sup>. In order to determine the PCDD/Fs in a sensitive and reliable manner, high resolution gas chromatography (HRGC) coupled to high resolution mass spectrometry (HRMS) is employed. Even with highly selective HRGC/HRMS methods, several compound classes found in common matrices will cause interference in the analyte channel. In order to eliminate these interferences a thorough sample cleanup, which is a very time consuming and costly task, is employed. The cleanup procedure is a multi-step liquid chromatography protocol, which involves different stationary phases. For low detection limits, the highest sample concentration is subjected to the cleanup procedure. By nature, PCDD/Fs are lipophilic compounds, which means they are enriched in the fat component of the sample. One major step in the cleanup procedure is the separation of the analytes from the fat fraction by gel permeation chromatography (GPC). The separation efficiency of this step is concentration dependent. To facilitate femtogram level detection limits of PCDD/Fs, it is often required to split the sample extract into several GPC runs, resulting in increased cleanup time and cost. To eliminate these extra cleanup steps it is desirable for laboratories to operate with the most sensitive HRGC/HRMS instrument available.

### Materials and Methods

All measurements were performed on a high resolution magnetic sector mass spectrometer with BE geometry (Thermo Electron, Bremen, Germany), running under the Xcalibur (Thermo Electron) data system. Calibration standards of the polychlorinated dioxins and furans were obtained from Wellington Laboratories.

### Results and Discussion

It could be shown that hardware modifications on the high resolution magnetic sector instrument facilitated higher signal-to-noise in the analysis of PCDD/Fs, resulting in decreased detection limits. On extracts prepared from different sample (i.e. fat fraction) amounts, significantly lowered detection limits were found. This resulted in the same data quality from less sample material, and thus less time and cost involved per analysis.

Measurements on calibration standards and real-life samples showed both, lower detection limits and improved reproducibility.

### References

1. North Atlantic Treaty Organisation (NATO)-CCMS report No. 176. (1988)
2. Berg, M.V., Birnbaum, L., Bosveld, A.T.C., Brunstom, B., Cook, P., Feeley, M., Giesy, J.P., Hanberg, A., Hasegawa, R., Kennedy, S.W., Kubiak, T., Larsen, J.C., Leeuwen, F.X.R., Liem, A.K.D., Nolt, C., Peterson, R.E., Poellinger, L., Safe, S., Schrenk, D., Tillitt, D., Tysklind, M., Younes, M., Waern, F., Zacharewsky, T.; (1998) Environmental Health Perspectives, 36, 775.