

Interlaboratory Comparison: Determination of Chlorinated Dibenzo-p-dioxins and Dibenzofurans by Methods EN 1948 and EPA 1613b

M. Wilken¹, Les Lamparski¹, G. D. Martin¹, T. L. Hescott¹, K. K. Mendyk¹, V. N. Fishman¹, W. J. Luksemburg², M. Maier², S. Hamm³, W. Sunderhauf³, M. Van Ryckeghem⁴, F. Neugebauer⁴, G. de Smet⁴

¹Dow Chemical Co.

²Alta Analytical Laboratories

³Eurofins GfA

⁴SGS Belgium

Introduction

Because of the global breadth of The Dow Chemical Company, environmental projects frequently span international borders; and data generated to demonstrate compliance with national regulations must conform to nationally-mandated analytical methods. Frequently, these compliance methods are designed to be rugged, reproducible, and expedient but may not always deliver absolutely accurate results due to some inherent biases. We have found that, occasionally, researchers wish to compare data generated according to the different compliance analytical methods. However, comparison of results of analyses performed according to similar compliance methods such as those in use in the United States and the European Union should not be attempted unless the correlation of the two methods has been established. For example, a previous comparison of three national compliance methods (US EPA Method 23 and German VDI 3499/2 and 3499/3) for the collection of incinerator stack gas emissions for the measurement of chlorinated dibenzo-p-dioxins and dibenzofurans (CDD/CDFs) demonstrated that there was a distinct bias in the sample collection procedures¹.

In this report, a variety of sample matrices (wastewater, biological sludge, and carbon adsorption media) were analyzed for CDD/CDFs according to US EPA Method 1613b² and following the principles of the European Standard Method EN 1948³ by 4 different laboratories skilled in the application of these methods. The results obtained from the laboratories have been compared and, in some cases, additional confirmatory analyses performed in order to minimize the differences between the methods.

Materials and Methods

The laboratories were selected to participate in the collaborative study based upon their experience in analyzing samples according to the methods defined in Table 1 below. Because of the experience of SGS in analysis by both EPA 1613b and EN 1948, this laboratory was chosen to perform the analyses by both of the methods in order to eliminate any interlaboratory variables. The participants were instructed to follow their standard laboratory protocols which are based on the defined methods. No special cleanup or separation steps were to be implemented unless they were standard practice for the laboratory or were allowed by the method (e.g., extended Soxhlet extraction time for samples containing activated carbon).

Table 1: Participating contract laboratories and methods used for the analysis

Laboratory	Analytical Method
Alta Analytical, El Dorado Hills, CA, USA	EPA 1613b
Dow Chemical Co., Midland, MI, USA	EPA 1613b (modified)
GfA, Münster, Germany	EN 1948 based
SGS, Antwerp, Belgium	EPA 1613b and EN 1948 based

The following sample matrices were chosen for the study based upon a variety of factors including expected analyte concentration (ppq to ppb), expected interferences (isomeric and non-CDD/CDF), analyte extractability from the