

ANALYSIS OF POLYCHLORINATED DIBENZOFURANS AND POLYCHLORINATED
DIBENZODIOXINS: II - AN INTER- AND INTRA-LABORATORY
EVALUATION OF MATERIALS OF CONSTRUCTION

by

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An old, multi building manufacturing facility was targeted for remediation. Given the cost for remediation and disposal of the potentially contaminated materials of construction subsequent to the demolition, a detailed sampling program was designed to determine the extent of contamination of the site. This study addressed many media for which official analytical protocols did not exist. Based upon a prior study of residue and sediment in raceways associated with this manufacturing facility, the protocol devised for that effort was used in the study of the buildings themselves. Specifically, this methodology included the Soxhlet extraction (16 hours) of the total sample taken. The extracting solvent was toluene. Benzene was not selected by the primary analytical laboratory and the primary QA/QC laboratory because of toxicity. According to protocol, large size samples were taken and coupled with enhanced low resolution GC/MS to achieve low limits of detection for the polychlorinated dibenzofurans and polychlorinated dibenzodioxins (PCDF/PCDD) analyses. This approach enabled the analytical phase to maintain a realistic schedule within the overall program. The QA/QC laboratory designated as "II" used benzene for the Soxhlet extraction and performed the analyses using high resolution GC/MS. If there were any questions on the PCDF/PCDD results because of matrix interference, both the primary laboratory and secondary QA/QC laboratory (III) used high resolution GC/MS to confirm results.

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The matrices that were analyzed included wood, stone, granite, brick, concrete, as well as hexane-wetted wipes of impermeable surfaces such as metal beams and ceilings. The matrices sampled were frequently painted, dusty, oily, varnished, or otherwise sealed. All effort was extended to obtain samples which were replicated as closely as possible for use in the QA/QC program. Adjacent samples of similar appearance were taken. These were sampled to the same depth. In fact, a major part of the sampling program investigated the penetration of the PCDF/PCDD into the materials of construction. Because of the large amount of surface area involved in the study, a procedure for compositing samples was developed. The procedure included composite confirmation samples which could be used to evaluate potential "hot spots". Samples were taken from floors, walls, ceilings, and several miscellaneous structures and surfaces. The results of these studies are summarized in Tables 1 and 2.

Table 1 summarizes the intra-laboratory analyses of replicates. It is believed that actual sample duplication is impossible since the media of construction are heterogeneous in the distribution of the constituents of interest. This heterogeneity results not only from inhomogeneities in the matrices themselves, but also differences in tracking/disposition mechanisms for a given surface, i.e., airborne, footsteps, etc. Rather than compare the complicated congener specific data, a reduced form is used for the summary tables. The reduced data are based upon the toxicity equivalency (TE) of the PCDF/PCDD results correlated to an equivalent concentration of 2,3,7,8 tetrachlorodibenzodioxin. While the actual weighting factors to arrive at the TE value for a sample can be controversial, the toxicity equivalency reported in this paper is based upon the most recent USEPA factors (March 1989). As can be seen for the media used in these replicate studies, comparison is very good on an intra-lab basis employing consistent protocols. We feel there is merit to looking at the variations between 0.001 and 0.002 less as a factor of 2 but rather as a difference of 0.001 ppb. (See the last column of Table 1.)

Table 2 is a comparison of the data from the inter-laboratory QA/QC program. The one primary laboratory was complemented by two QA/QC laboratories. As indicated earlier, laboratory II chose to employ benzene and high resolution GC/MS in its preparation and analysis. Once again, the agreement, given the possible heterogeneity in the samples, as well as the differences in laboratories, is very good. Sources of differences in results will be discussed in the full paper. This component of the QA/QC program included both blind replicates to the primary laboratory, blind replicates to the QA/QC laboratories and cross-checking replicates among all three.

TABLE 2
 INTERLABORATORY COMPARISON
 OF TOXICITY EQUIVALENTS FROM VARIOUS REPLICATE
 WOOD SAMPLE ANALYSES

Medium	Laboratory No ^a	TE Values, ppb
Wood Floor	I	0.011
	I	0.002
	II	0.014
	III	0.011
	III	0.006
Wood Floor (aged)	I	ND
	III	0.02
Wood Floor	I	0.0090
	II	0.075
Wood Floor (varnished)	I	0.010
	III	0.087
Wood Floor	I	0.0038
	III	0.0019
Wood Floor (white paint)	I	0.004
	II	0.011
Wood Ceiling (white paint)	I	0.0067
	III	0.0051
Wood Ceiling (white paint)	I	0.017
	II	0.19
Wood Ceiling (white paint)	I	0.0011
	II	0.019
Wood Floor (dark)	I	0.16
	I	0.10
	II	0.21
	III	0.28

^aLaboratory I is the primary laboratory.

TABLE 1
 COMPARISON OF TOXICITY EQUIVALENTS
 FROM REPLICATE SAMPLE ANALYSES OF
 VARIOUS MEDIA OF CONSTRUCTION
 (PRIMARY LABORATORY ONLY)

Medium	TE #1, ppb	TE #2, ppb	ΔTE, ppb
Concrete Floor	0.014	0.004	0.010
Concrete Floor	0.02	0.04	0.02
Concrete Floor (oil stained)	0.12	0.13	0.01
Concrete Wall	ND	ND	--
Brick Wall	0.001	0.002	0.001
Granite Wall (white paint)	0.004	0.006	0.002
Granite Wall (green paint)	1.6	1.0	0.6
Granite Wall (light green paint)	0.004	ND	--
Stone Wall (painted)	0.06	0.08	0.02
Wood Ceiling (white paint)	0.09	0.09	0.0
Wood Ceiling	0.12	ND	--
Wood Ceiling (dark varnish)	0.0012	0.0018	0.0006
Wood Ceiling (white paint)	0.0006	ND	--
Metal Ceiling (brown paint; wipe)	0.015	0.009	0.006
Carpet and Pad	0.24	0.16	0.08

TE - toxicity equivalent to 2,3,7,8 tetrachlorodibenzodioxin.