# Evaluation of New Dual-Layer Carbon Reversible Column in Analysis of PCDDs/PCDFs and Co-Planar PCBs

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**Sample preparation for the analysis of co-planar PCBs/ PCDDs/ PCDFs often includes a classification procedure to separate similar compounds. Many analysts are using a carbon column among several other methods for this step due to its simple operation, ease of handling, and consistent lot-to-lot performance.**

**There is a problem of low and sometimes variable recovery using currently available carbons, however, which is further complicated by the requirement of a large elution volume.**

**With these factors in mind, we developed a new dual-layer carbon reversible column composed of two different adsorption strength carbons and tested the adsorption and elution behavior of 31 co-planar PCBs/PCDDs/PCDFs.**

**We discovered this column demonstrates an excellent recovery of dioxins. We now wish to describe our results which testify to the unique utility of the dual-layer carbon column for dioxin analysis.**

# Problems of a Mono-Layer Carbon Column
# Carboxen 1000 Reversible (100mg)

- Low recovery for hepta-octa CDD/DF
- Large volume of toluene is needed for elution
  of PCDDs/PCDFs
- Poor classification of co-planar PCBs

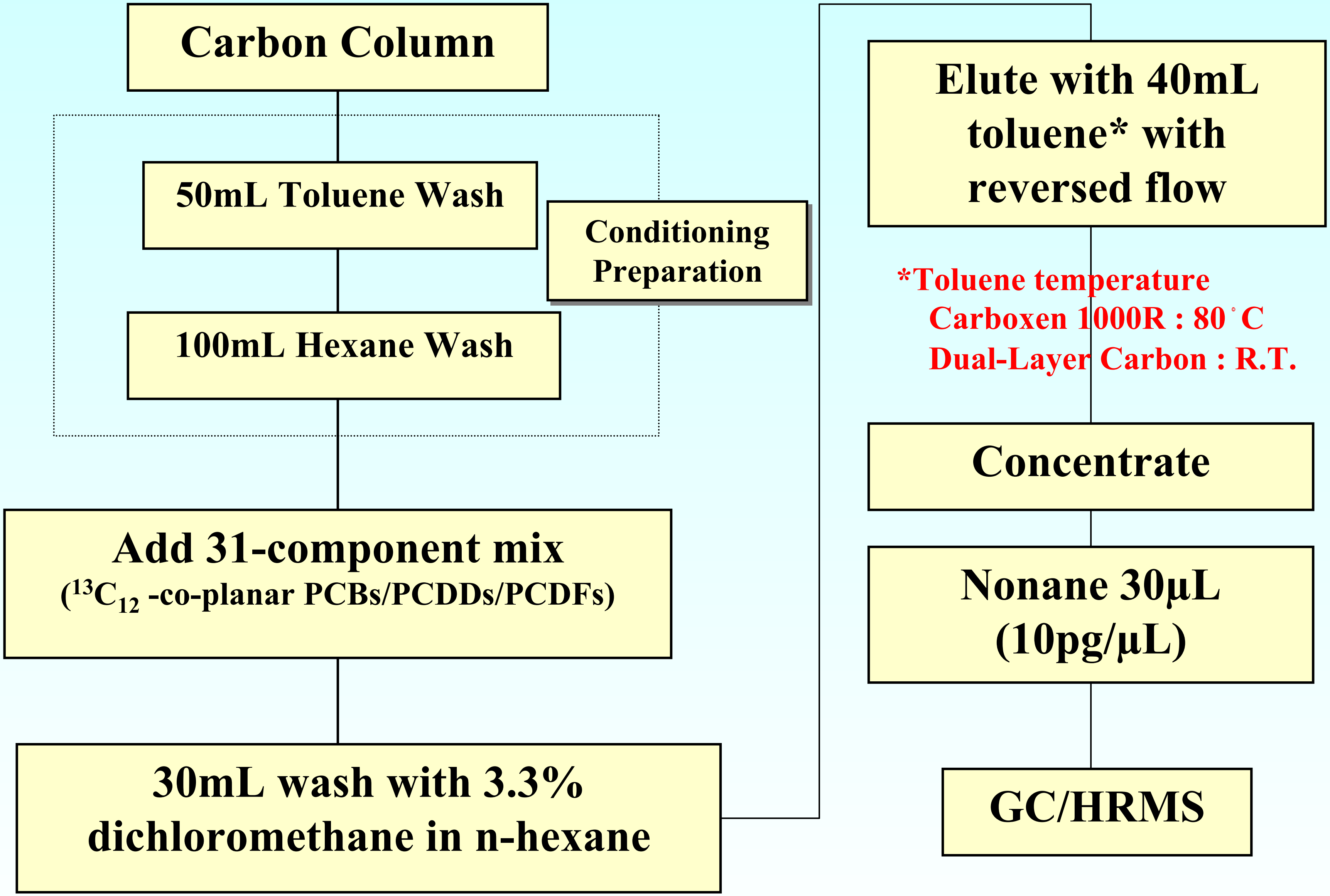
# *Recovery Comparison of the Dual-Layer Carbon Column versus the Carboxen 1000R Column*

**The dual-layer carbon column and Carboxen 1000R™ column were both pre-conditioned with toluene, followed by a rinse of n-hexane. A mix of 31 components of $^{13}C_{12}$- Co-planar PCBs and PCDDs/PCDFs(300pg in 30µL n-nonane) were loaded on both the dual-layer column and the Carboxen 1000R™ column. A 30mL rinse of 3.3% dichloromethane in n-hexane followed to flush from the columns any less strongly retained analyte interferences.**

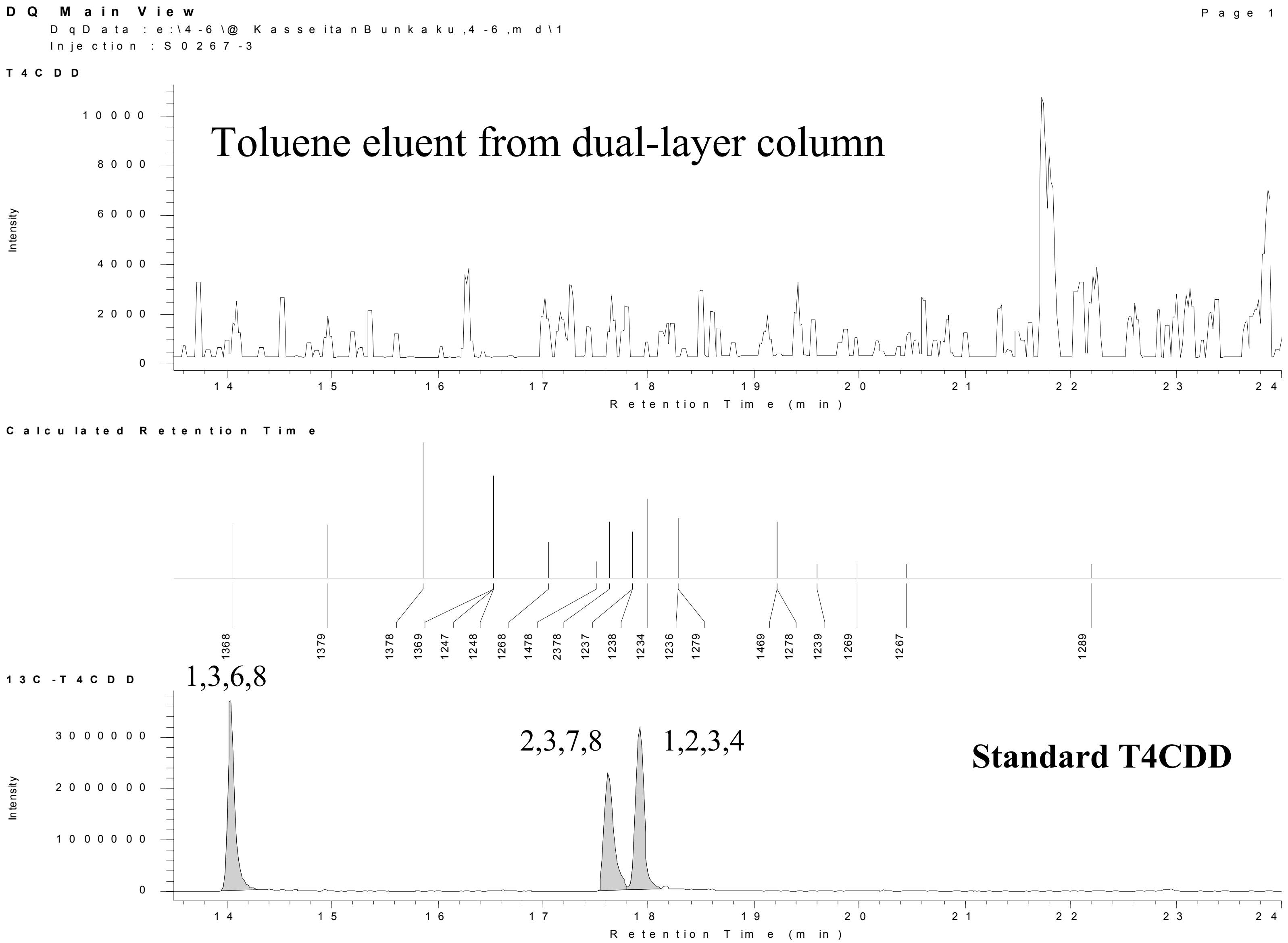
**The dual-layer carbon column was reversed and 50mL of toluene at room temperature was passed through the column to desorb the co-planar PCBs/PCDDs/PCDFs. The mono-layer carbon column was also reversed and 50mL of hot (80°C) toluene was used to desorb the analytes.**

**The eluants were concentrated and the dioxin recovery was determined using GC/HRMS (Micromass) with 10,000 or over.**

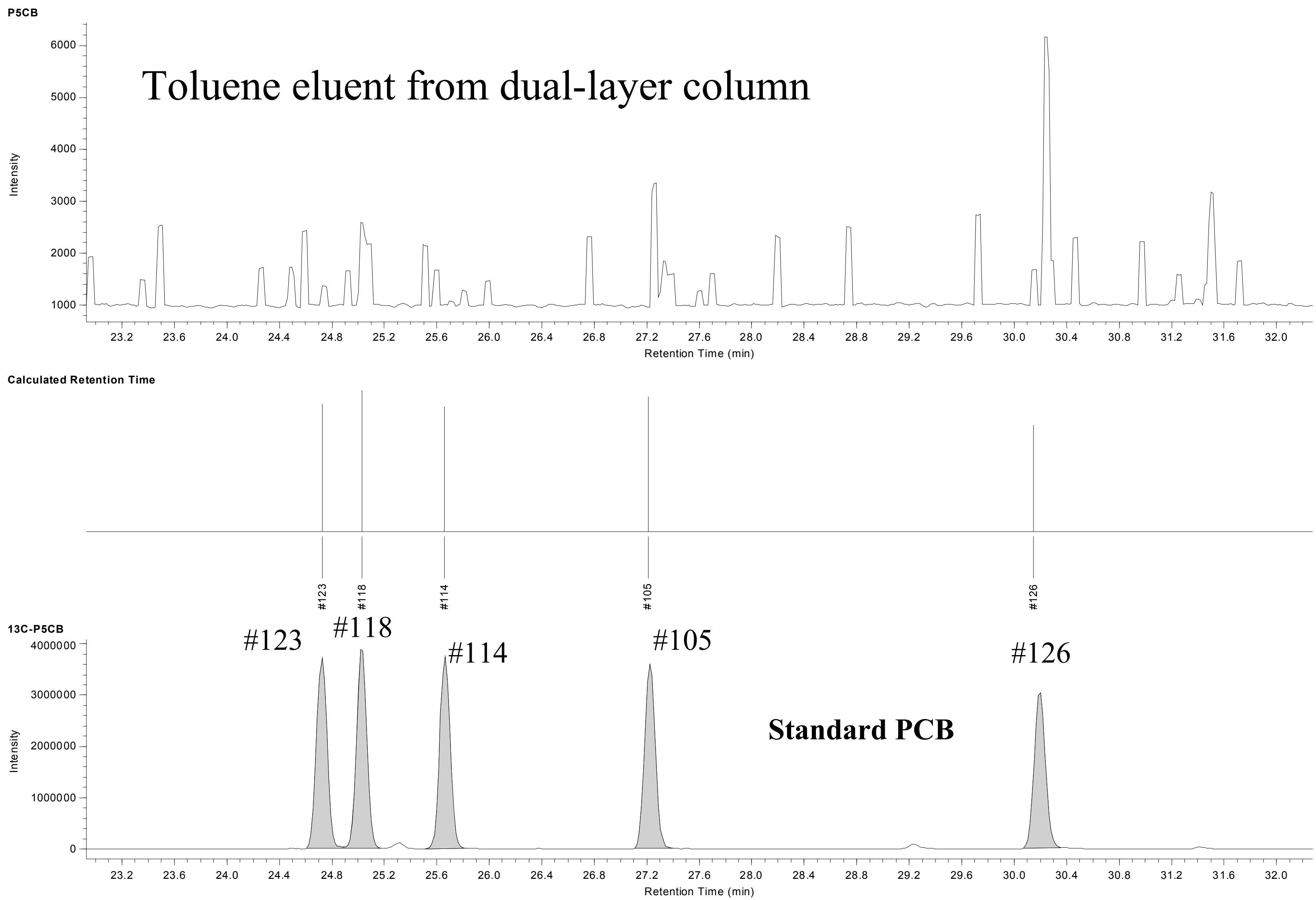
# Experimental Flow



# T4CDD Blank of Dual-Layer Tube



# PCBs Blank of Dual-Layer Tube



# *Dioxin Recovery and Distribution*

**The 31 analytes (300pg) were desorbed from the dual-layer carbon column with 50mL of room temperature toluene and the following fractions were collected: 0-5, 5-10, 10-20, 20-30, 30-40, 40-100mL. The recovery of dioxins for each fraction was determined.**

**The 31 analytes were also desorbed from the Carboxen 1000R column with 50mL of heated (80 °C) toluene and the recovery for each analyte was determined.**

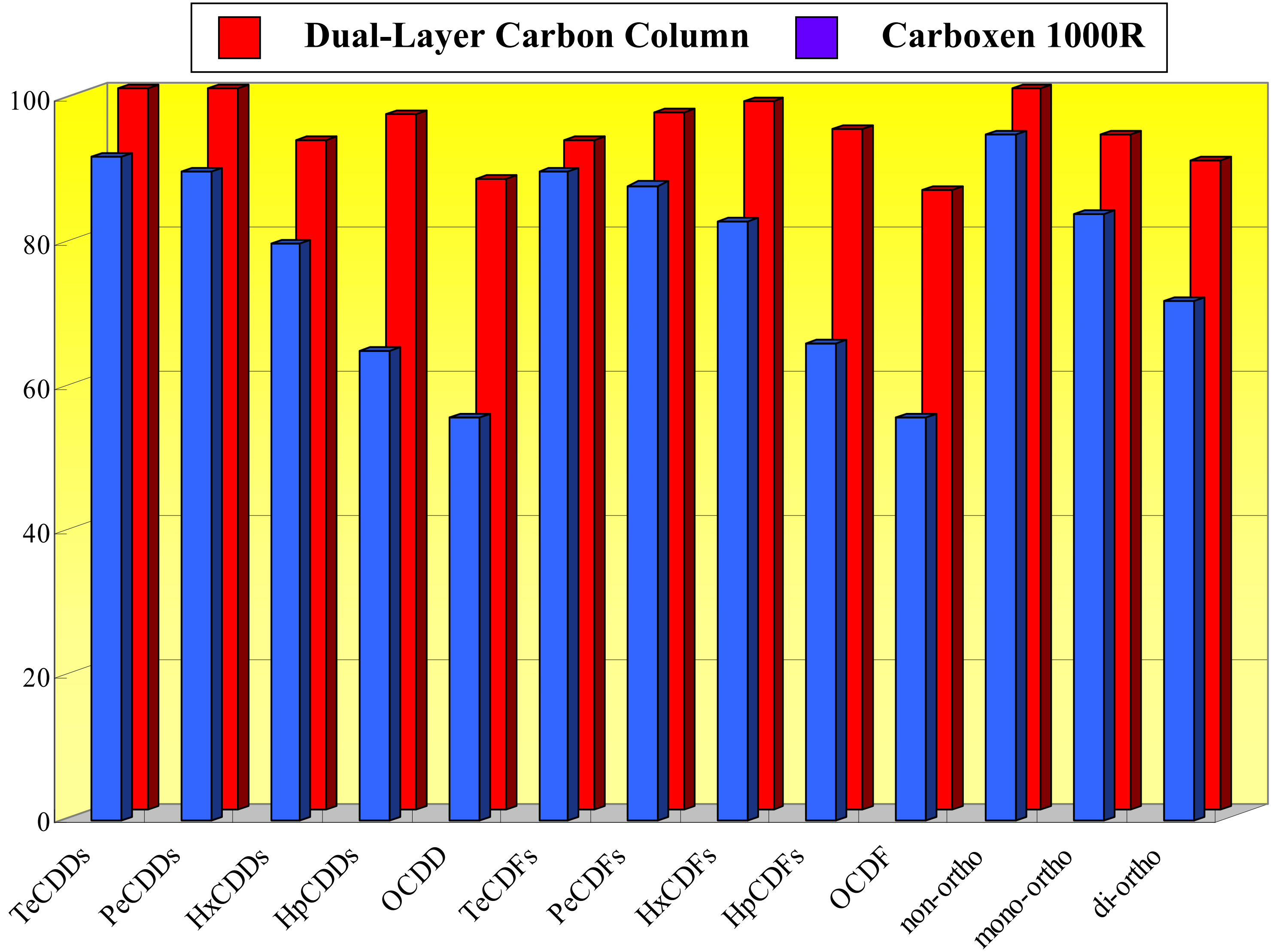
**Two reversible glass tubes were packed, one with Carboxen 1016 and the other with Carboxen 1000, respectively, then connected in series, and spiked with 300pg of dioxins in the same manner as dual-layer column.**

**After the 3.3% dichloromethane in hexane flush (30mL) the columns were disconnected. Each was reversed and desorbed with 40mL of room temperature toluene. The recoveries of the 31 analytes from each column were determined. These recoveries demonstrate how far each analyte was carried through the connected columns by the 30mL flush.**

**The analyte retention selectivity of each carbon allows the more strongly retained and difficult to desorb analytes to be trapped on the Carboxen 1016 where they can be desorbed easily. Likewise, the weakly retained and easily lost analytes from the Carboxen 1016 are trapped on the more retentive Carboxen 1000.**

**The two carbons in series complement each other and permit a higher overall recovery of the 31 analytes than either carbon could demonstrate separately.**

# Recovery of the New Dual-Layer Carbon Column versus Carboxen 1000R Column



# Discussion

**The recovery of the hepta- and octa-CDDs/DFs from the dual-layer carbon column is greatly improved in comparison to the Carboxen 1000R column. In addition, the recovery of co-planar PCBs from dual-layer carbon column was also better than from the Carboxen 1000R.**

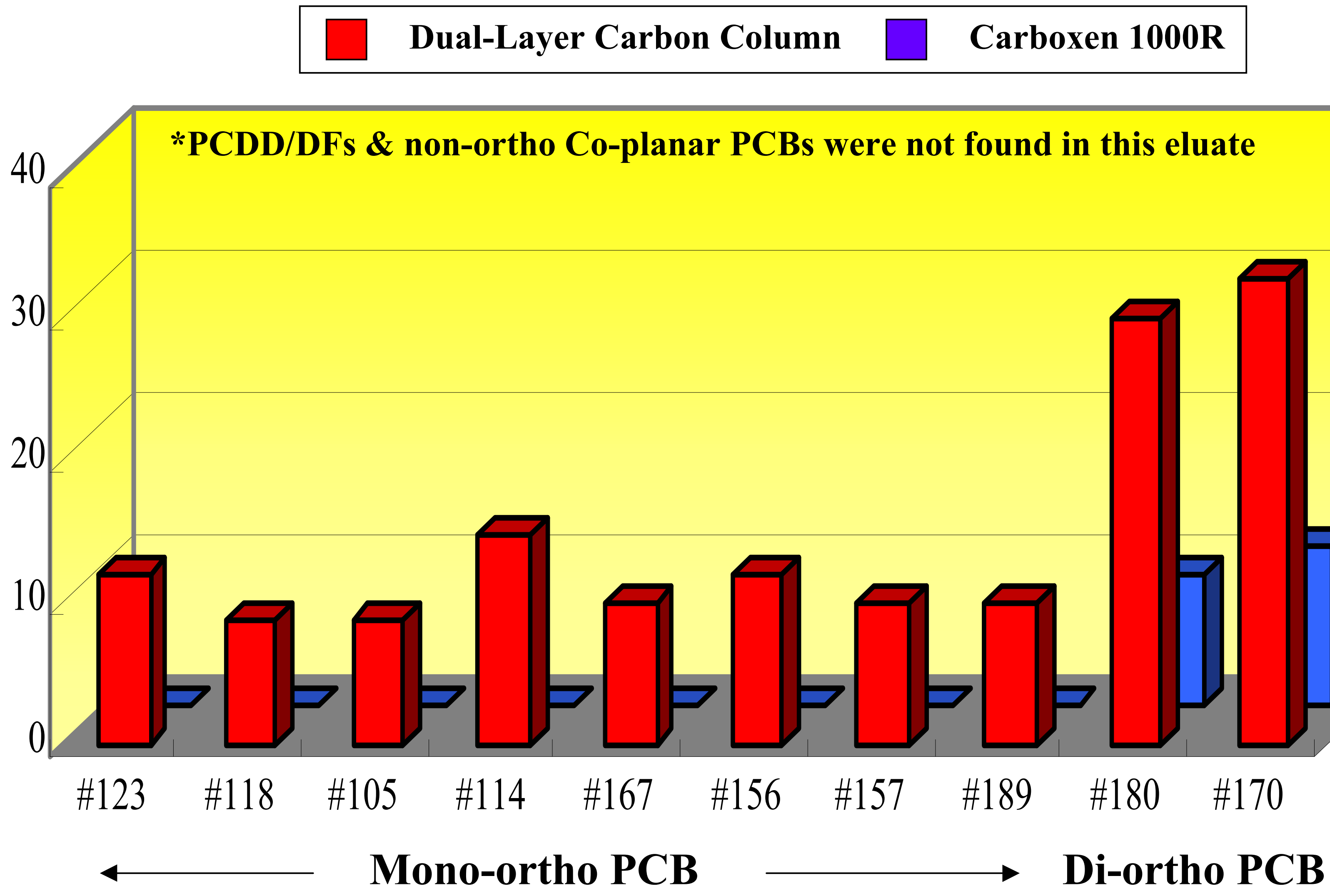
**We checked the 3.3% DCM/n-hexane eluant for the presence of co-planar PCBs/PCDDs/PCDFs. We found that 30% of Di-ortho Co-planar PCBs and 10% of Mono-ortho Co-planar PCBs were present in the 3.3% DCM/n-hexane eluent of Carboxen 1000R.**

**On the other hand, no Mono-ortho Co-planar PCBs and only 10% of Di-ortho Co-planar PCBs were present in 3.3% DCM/n-hexane eluant of Dual-layer Carbon column.**

**A smaller loss of the Di-ortho and Mono-ortho Co-planar PCBs in the washing step (3.3% DCM/n-hexane - 30mL) is another reason for the improved recovery from the dual-layer carbon column.**

# Di- & Mono-ortho PCBs in 3.3% DCM/hexane eluant*

## Dual-Layer Carbon - Carboxen 1000R



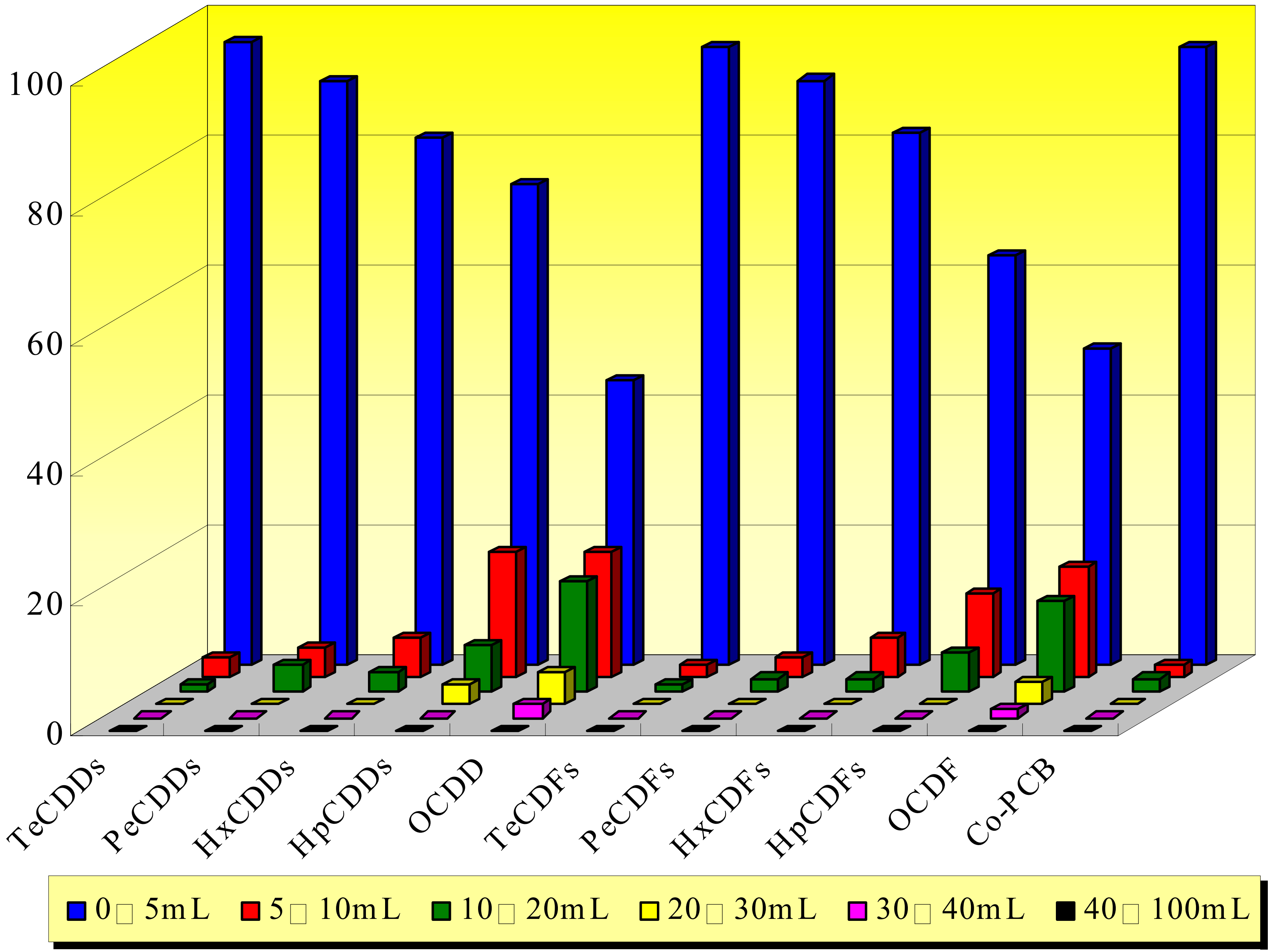
# *Recovery of Dioxins from the Dual-Layer Carbon Column*

**The distribution of dioxins from the dual-layer carbon column shown in the next figure shows an excellent recovery of dioxins with less solvent (40mL) than normally required (80mL).**

**Using only 5mL of unheated toluene over 40% of each of the octa-chloro DD/DFs were recovered while even higher percentages of the other dioxins were recovered.  Most of the dioxins can be recovered using only 20mL of toluene.**

**Only small amounts of these analytes were recovered using greater than 30mL of toluene, and almost none were recovered using more than 40mL of toluene.**

# Dual-Layer Carbon Column Dioxin Elution Pattern



# *Recovery of Dioxins from the Carbon Columns in Series*

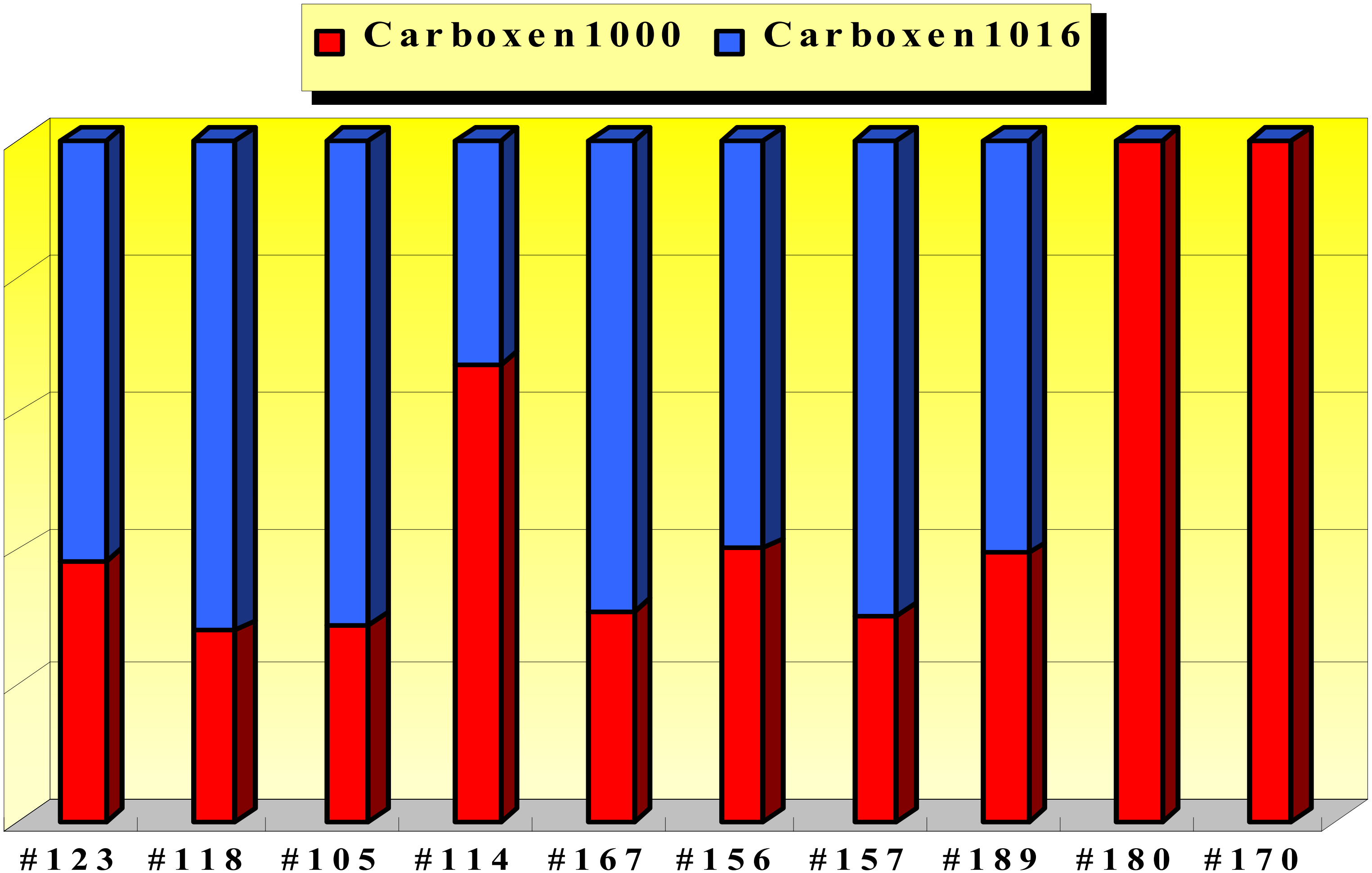
**About 30% to 65% of mono-ortho PCBs and all di-ortho-PCBs migrated through the Carboxen 1016 tube and were adsorbed on the Carboxen 1000 layer. The other non-ortho PCB and PCDDs/PCDFs were adsorbed on the Carboxen 1016 and were desorbed readily from it, unlike the Carboxen 1000 which tends to retain a fraction of these analytes.**

**The hepta and octa- chlorinated PCDD/PCDFs were desorbed readily from the Carboxen 1016 using unheated toluene. Thus, the dangerous and tedious procedure of using hot toluene is no longer necessary.**

**Based on these experiments, it is clear that the upper layer of Carboxen 1016 in the dual-layer carbon column contributes to the improvement in dioxin recovery and the reproducibility of the analysis.**

**From these results we hope to develop a better purification by changing the composition of washing solvent.**

# Mono- and Di- ortho PCB Distribution from Carboxen 1016 and Carboxen 1000 Columns in Series




**All PCDD/PCDFs and non-ortho co-planar PCBs were adsorbed on the Carboxen 1016**

# Summary

## Advantages of the Dual-Layer Carboxen Column

- Greatly improved recoveries of the PCDDs/PCDFs, especially the hepta- and octa- PCDDs/PCDFs

- Heated toluene (50mL @ 80 °C) is not needed. Toluene needed for adequate recovery is reduced by almost 50%.

- By changing the elution volume and the composition of washing solvent to remove interferences, we can isolate 31 co-planar PCBs/PCDDs/PCDFs with a good recovery.

- Classification of PCDDs/DFs and co-planar PCBs may be possible using the dual-layer Carboxen carbon column.