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DEVELOPMENT OF NEW CAPILLARY COLUMNS FOR DIOXIN ANALYSYS

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Introduction

In general, DB-5, DB-17, SP-2331, CP-Sil 88 are employed in various combinations as GC columns for dioxin analyses. However, these columns have several shortfalls, e.g., certain 2,3,7,8-substituted isomers are not separated by DB-5 or DB-17, sensitivities (especially for HxCDFs) are insufficient using SP-2331 or CP-Sil 88. There are no ideal columns for determination of dioxins. The authors have developed new columns for dioxin analysis. This is a report of all assignments of all dioxins/furans on these columns.

Methods and Materials

Several types (e.g., different liquid concentration, different bridging method for liquid phase on inner wall, different film thickness) of fused silica capillary column that have a methyl-phenyl silicone liquid phase were made by SGE International (Austria) and Inventx (USA). Full assignation was carried out using all of individual isomers of PCDDs/PCDFs and PCBs. After modifications to the liquid phase, in the end two columns, BPX-DXN (SGE International, 60m, 0.25mmID) and RH-12ms (Inventx, 60m, 0.25mmID) were selected for the analysis of dioxins and PCBs. Using two injections on this 2 column combination allows determination of all 2,3,7,8-substituted PCDDs/PCDFs isomers. A third injection on the RH-12ms column allows determination of 12 PCBs (with WHO-1998 assigned TEF's). Full assignment chromatograms of PCDDs/PCDFs and WHO-PCBs for BPX-DXN and RH-12ms are shown in Figures 1, 2 and 3. More detailed data is available at "http://www.metocean.co.jp/new/gijutu_panfu5.htm".

GC injection port temperature: 300DC; Column head pressure: 25.4psi; GC-MS interface temperature 290DC; Ion source temperature: 320DC; Trap current: 500μA; Electron energy: 40-50eV; Maximum acceleration voltage: 7500V; Ramp of oven temperature: 30DC(1min)-(15DC/min)-210DC(0min)-(3DC/min)-310DC(0min)-(5DC/min)-320DC(Hold)

Summary/Conclusion

Good separation and sensitivities were achieved using the two-column combination representing a marked improvement over currently available column combinations for PCDD/PCDF/PCB analysis. Characteristics of the two column combination are noted below.

1. High sensitivity (S/N ratio) caused by non cyanopropyl phase with low chemical background noise caused from liquid phase.
2. Two columns combination possible to separate all TEQ-related isomers.
3. Determination of Tetra-Octa PCDDs/PCDFs and 12 PCBs (WHO-1998) is possible using two columns and 3 injections.
4. As oven temperature programs are the same for all three injections, MS interruption is reduced..

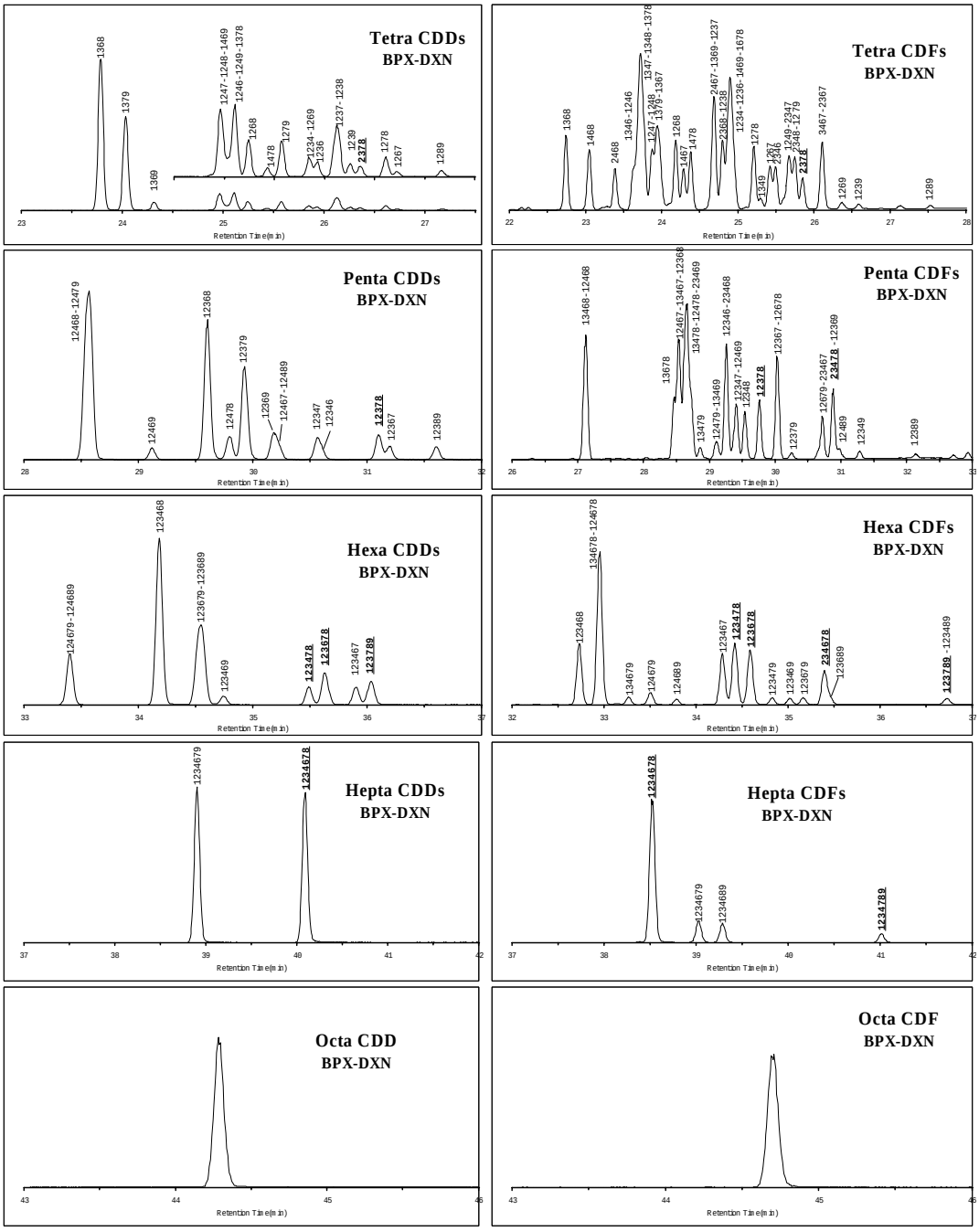

Figure 1. Elution order of all tetra-octa CDDs/CDFs isomers for BPX-DXN.

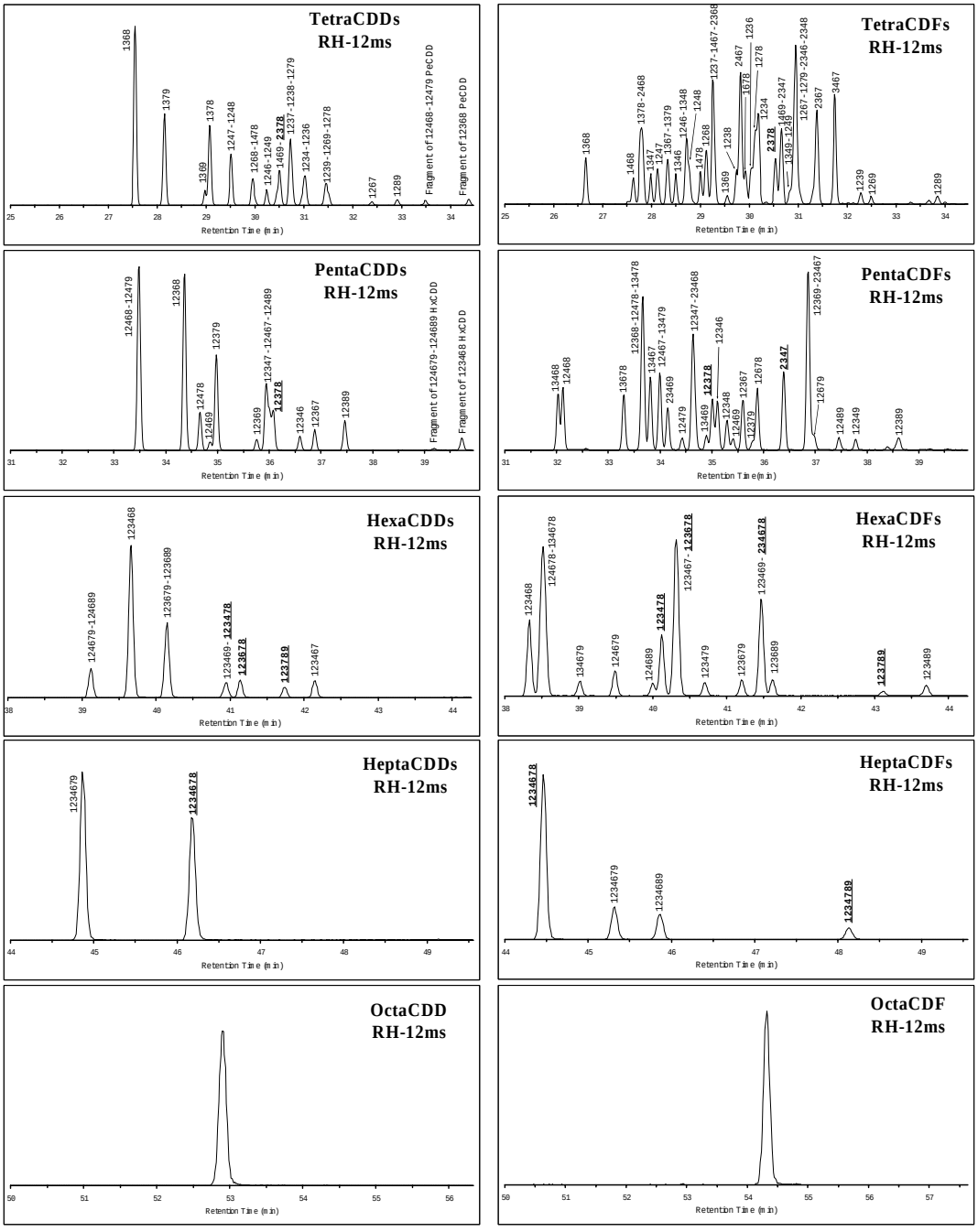

Figure 2. Elution order of all tetra-octa CDDs/CDFs isomers for RH-12ms.

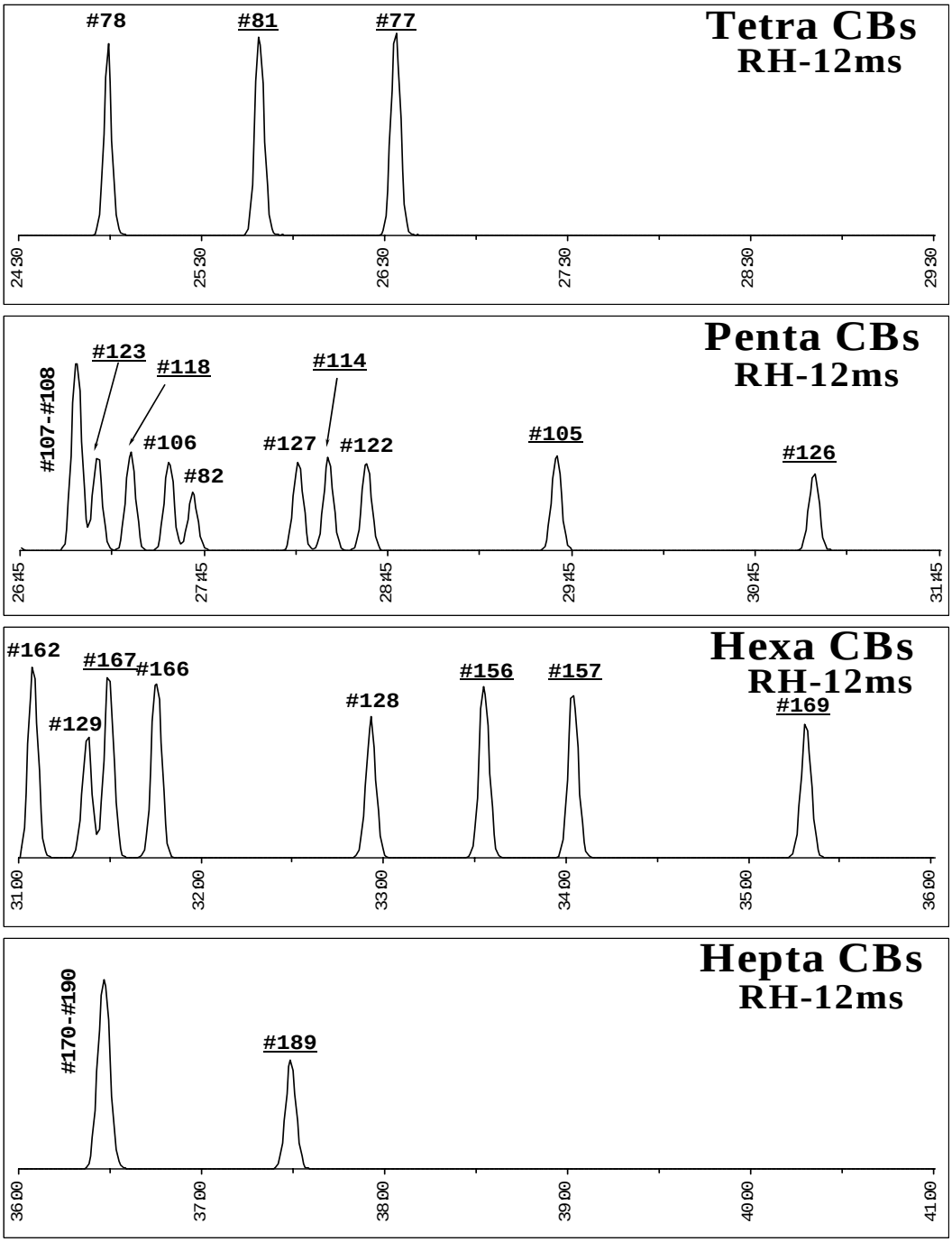

Figure 3. Elution order of tetra-hepta PCBs for RH-12ms.
Please find full description at “http://www.metocean.co.jp/new/gijutu_panfu5.htm”.