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Retention Order of All 209 Chlorobiphenyl Compounds on Capillary Column SGE HT8

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Abstract

Retention order of all 209 chlorobiphenyl compounds on capillary column SGE HT8 were assigned using HRMS. Peak separation for each compound has improved in comparison with the 5% phenyl or equivalent phase, especially for non-*ortho* and mono-*ortho* CBs. Analysis for each congener can be finished within 30 minutes. Results shows HRGC(equipped HT8)/HRMS analysis is one of the best method for CBs' analysis.

Introduction

Chlorobiphenyls (CBs) are organohalogen compounds that comprise two benzene rings are jointed by one bridge. Figure 1 depicts the basic structure of CBs, together with the numbering system at the positions on the benzene rings where chlorine atoms can be substituted. There are 209 compounds by differing in the number (mono, di, tri, tetra, penta, hexa, hepta, octa, nona and deca) and position (2, 2', 3, 3', 4, 4', 5, 5', 6 and 6') of the chlorine atoms. The congener groups and number of compounds are shown in Table 1.

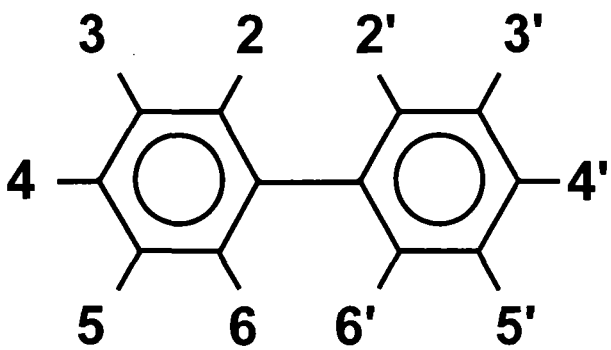

Figure 1. Basic structure of CB.

Table 1. Number of compounds for CBs.

congener	number of compounds
mono	3
di	12
tri	24
tetra	42
penta	46
hexa	42
hepta	24
octa	12
nona	3
deca	1

It is indispensable to obtain accurate data for each CBs compound in order to clear the behavior and fate of CBs in environment. Although no works, which reported the CBs analysis using all CBs standard combined with HRGC/HRMS, are available for us. There are a few reports in regard to the column coated with 5% phenyl phase combined with GC-ECD analysis. However the separation by 5% phenyl phases' is not so good for each compounds and it is widely accepted that only GC/MS analysis is enough to separate CBs' compounds. This report represents the retention order of the all 209 CBs compounds using a column that has a new liquid phase, all 209 CBs standards and HRGC/HRMS analysis.

Experimental Methods

For this study, all 209 CB standards were prepared from AccuStandard (USA). Strict measures were taken to minimize contamination and error during the standard preparation. Each standard was diluted by decane purified with multiple distillation. All processes were carried out in a clean room and room temperature was controlled in 20±0.5℃. Finally, 40 pg/ μ L solutions were prepared for HRGC/HRMS analysis. HRGC/HRMS measurement performed by the condition shown in Table 2 and 3.

Table 2. Conditions of HRGC for determination of CBs.

HRGC (HP5890II)	Column : 8% phenyl equivalent, polysiloxane-carborane ; HT8(SGE)
	50m length, 0.22mm I.D., 0.25 μ m film thickness
	Column Head pressure :25psi(He), Injection Port Temp. : 280℃
	Ramp of Oven Temp. :
	130℃(1min.)---[20℃/min]-->220℃(0min.)---[5℃/min.]-->320℃(hold)

Table 3. Conditions of HRMS for determination of CBs.

HRMS (VG Analytical, AutoSpec)	Interface Temp.: 325℃, Ion Source Temp.:335℃, Trap Current : 500 μ A
	Electron Energy : 40eV, Accelerating Voltage : 8KV
	Lock Mass : PFK
	Scan Rate : 150-400msec./scan, Resolution : 12000
	Measured Mass : mono CBs : 180.0393(M)*,190.0363(M+2),192.9888(PFK)
	di CBs : 220.0003(M)*,223.9974(M+2),230.9856(PFK)
	tri CBs : 255.9613(M)*,257.9584(M+2),268.9824(PFK)
	tetra CBs : 289.9224(M),291.9194(M+4)*,292.9824(PFK)
	penta CBs : 325.8804(M+2)*,327.8775(M+4),330.9792(PFK)
	hexa CBs : 359.8415(M+2)*,361.8385(M+4),354.9792(PFK)
	hepta CBs : 393.8025(M+2)*,395.7995(M+4),392.9761(PFK)
	octa CBs : 427.7635(M+2),429.7606(M+4)*,430.9729(PFK)
	nona CBs : 461.7245(M+2),463.7216(M+4)*,480.9697(PFK)
	deca CBs : 497.6826(M+4)*,499.6797(M+6),492.9697(PFK)

*measurement mass shown in Figure 2,3,4 and 5.

Results and Discussion

Mass chromatograms for mono, di, tetra, penta, hepta, octa, nona and deca CBs are shown in Figure 2,3,4 and 5(chromatograms for tri and hexa CBs are omitted here) together with the BZ# numbering system proposed by Ballschmiter and Zell [1]. Peak separation for each congener has improved in comparison with the 5% phenyl or equivalent phase, especially for non-*ortho* and mono-*ortho* CBs. This phenomenon is caused by a unique characteristic of HT8. It seems that there might be some affinity between co-planer structure of CB and carborane phase. Chromatogram of deca CB shows all analysis can be finished within 30 minutes.

The authors will publish all retention order and response factor for each CB compounds for an appropriate journal in near future.

Literature Cited

(1) Ballschmiter, K.; Zell, M. *Fresenius Z.Anal.Chem.* **1980**, 20-31.

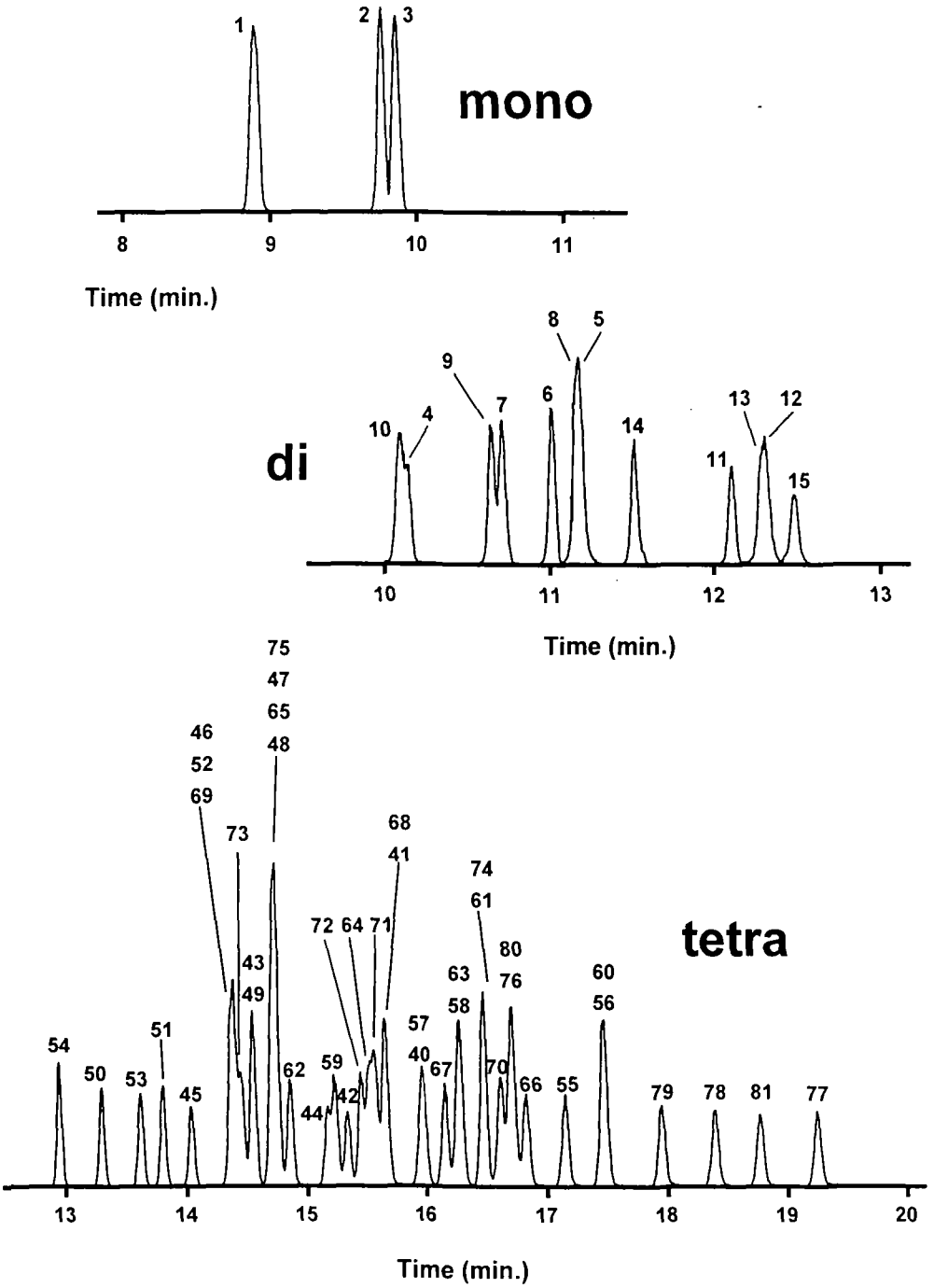

Figure 2. Chromatogram for mono-, di- and tetra-CBs.

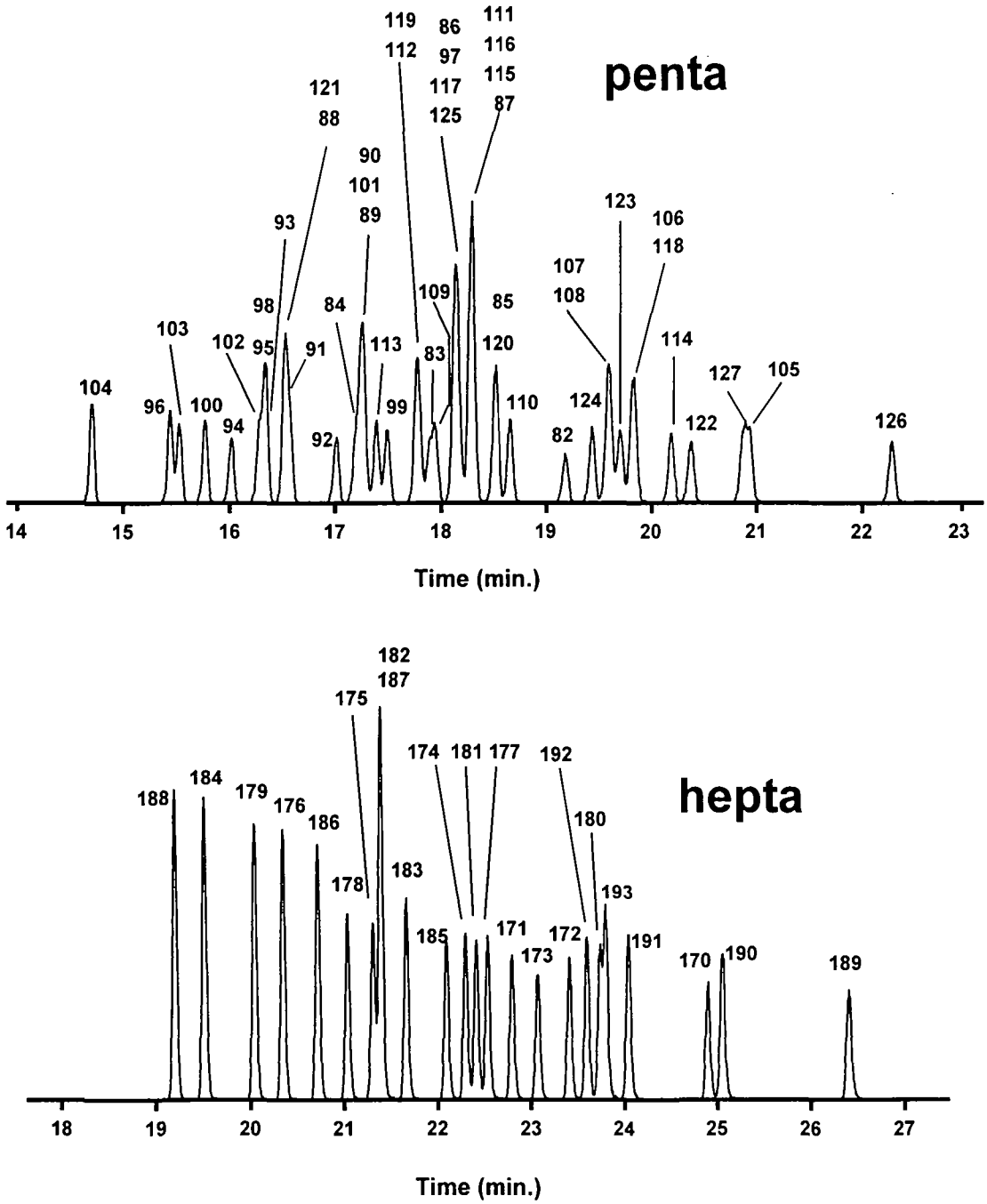

Figure 3. Chromatogram for penta- and hepta-CBs.

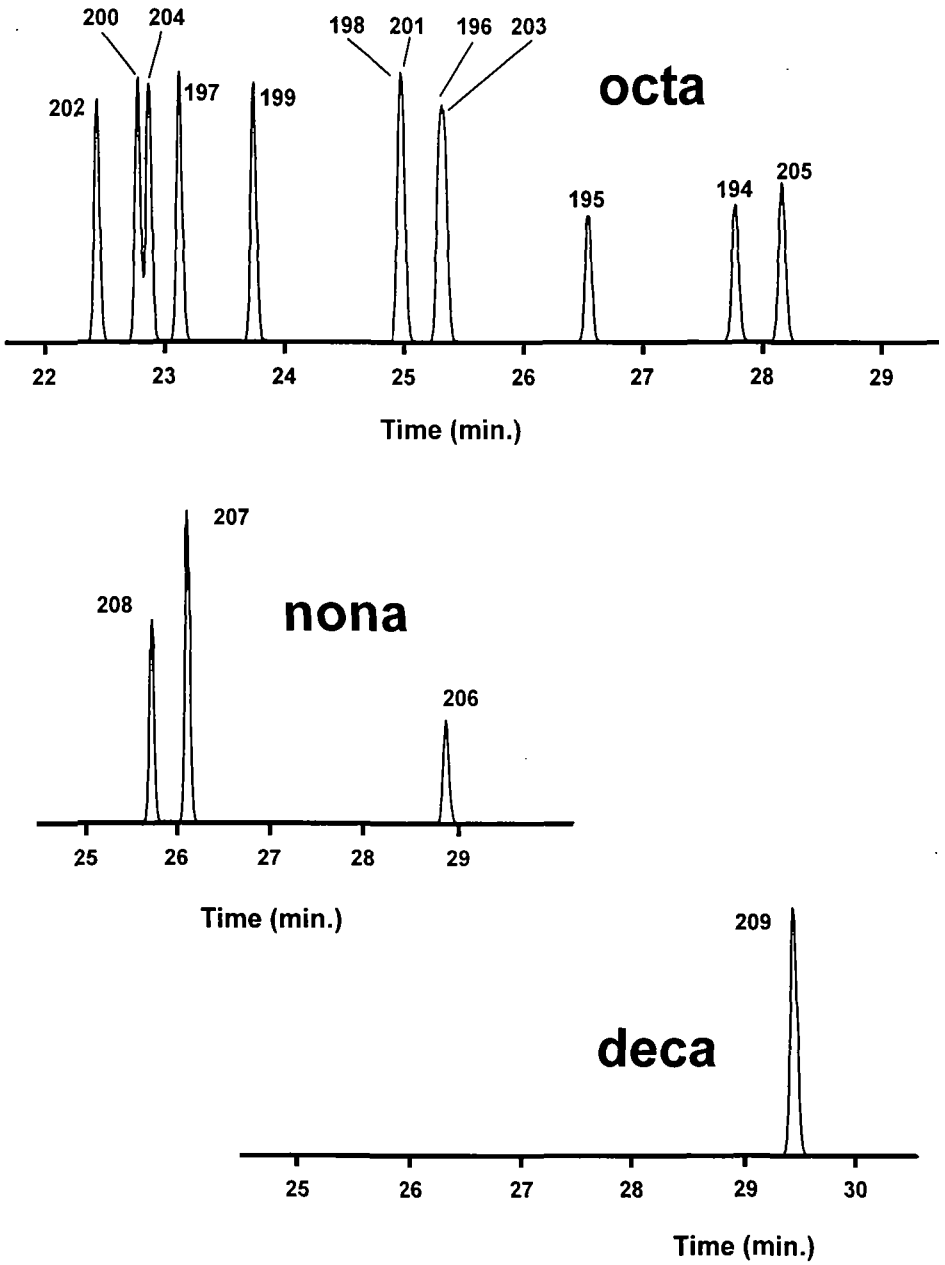

Figure 4. Chromatogram for octa-, nona- and deca-CB(s).