Analysis method for PCBs in reclaimed oil using a fast-GC triple stage quadrupole mass spectrometer with the 13-component quantitation method

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Abstract It is necessary for companies supplying reclaimed oil to analyze polychlorinated biphenyls (PCBs), because there is a possibility of the presence of contaminants due to trace-level PCBs in the reclaimed oil. However, common analysis methods of PCBs are time-consuming and complicated. Fast-GC triple stage quadrupole mass spectrometer with the 13-component quantitation method is an official method for analyzing PCBs in insulating oil in Japan. This method is extremely fast and simplified. The purpose of this study involves an investigation of the aforementioned fast and simple method for potential use in the analysis of reclaimed oil. Furthermore, it was attempted to combine the method with sample preparation involving only hexane dilution. The effect of sample dilutions corresponding to 100, 300, and 500 times was evaluated for reducing the matrix effect. The matrix effect was suppressed at a dilution ratio equal to or exceeding 300 times. Calibration curves of four points, namely 0.01, 0.05, 0.1, and 0.5 ng/mL, (ignored origin) by using an internal standard method were prepared for the 13 components. The square of regression coefficient ($R^2$) values of all calibration curves exceeded 0.997. This method was adopted for the analysis of reclaimed oil containing 0.5 μg/mL PCBs, which corresponds to the judgment criteria, and accurate quantitation (accuracy value, 94.0–102%) and good repeatability (%RSD, 3.6%) were obtained. Furthermore, the required sensitivity was maintained even when 800 samples were analyzed without a cleaning ion source and an exchanging analysis column.

Keywords Polychlorinated biphenyls · Reclaimed oil · Fast-GC · Triple stage quadrupole mass spectrometer · Sample preparation

Introduction

In the manufacture and supply of reclaimed oil, safety and good quality of the end products is important. Suppliers of reclaimed oils collect waste lubricating oils such as engine oil, gear oil, hydraulic oil, heavy oil, and insulating oil as raw materials, and recycle these oils at their plants. The lubricating waste oil could potentially be contaminated with trace-level polychlorinated biphenyls (PCBs). Therefore, it is necessary for companies supplying reclaimed oil to analyze PCBs in reclaimed oil for quality control. Furthermore, an important issue involves speeding up PCB analysis to increase the production efficiency of reclaimed oil. Many studies have developed methods for the analysis of PCBs in waste oil. However, the aforementioned methods are extremely time-consuming in terms of performing a series involving sample preparation, GC measurement, and data analysis. Specifically, methods including sulfuric acid treatment, partition with dimethyl sulf oxide (DMSO)/hexane, partition with...
dimethylformamide (DMF)/hexane, florasil column cleanup, and silica gel column cleanup are used in sample preparation, and the operations require skilled technologies in terms of analysis and operation time (Copland and Gohmann 1982; Orazio et al. 1989; Larsen et al. 1991; Lawn and Toffel 1987; Gordon et al. 1982; Sandra et al. 1988; Suzuki et al. 1991; Koizumi and Yoshimura 1984). Conversely, with respect to the analysis of insulation oil analysis in Japan, a few pretreatment techniques were developed to aid in performing the analysis in a more quick and easy manner (Takahashi and Honda 2010; Shimizu 2010; Hamada 2010). Furthermore, analytical methods by sample preparation with only hexane dilution were developed in gas chromatography combined with negative chemical ionization (GC/NCI) and gas chromatography combined with high resolution mass spectrometry (GC/HRMS) (Takasuga et al. 2006; Machii et al. 2003). In this study, we attempted to perform an analysis by using a gas chromatography combined with triple stage quadrupole mass spectrometer (GC/MS/MS) with high selectivity involving only hexane dilution. The GC/MS/MS was selected because of its simple operability and maintainability when compared with those of GC/NCI and GC/HRMS. Additionally, the detection limit of recent GC/MS/MS is equivalent to that of GC/HRMS due to technological innovations, and this is also adopted in the official method for the analysis of dioxins in food by the European Union (EC 589/2014 and EC 709/2014). The selection of the analytical column is an important factor that can provide a high speed in GC analysis. Extant studies examined several columns as columns for PCB analysis (Frame 1997), and this typically corresponds to DB-5MS and HT8-PCB in Japan (Matsumura et al. 2002). However, the analysis time approximately equals or exceeds 40 min when these columns are used. In contrast, studies reported on high-speed analytical methods involving the use of a VF-Rapid MS PCB screen column that elute all PCB congeners of three Arochlors within 4 min (Cochran 2002). The column corresponds to a wide bore column fitted with a resistance column with a length of 6 m and an inner diameter of

| Table 1 List of concentrations of EC-5488 (unit: ng/mL) |
|-----------------------------------------------|
| Native                                       |
| IUPAC# | CS1 | CS2 | CS3 | CS4 |
| 18     | 1   | 5   | 10  | 50  |
| 28     | 1   | 5   | 10  | 50  |
| 44     | 1   | 5   | 10  | 50  |
| 52     | 1   | 5   | 10  | 50  |
| 70     | 1   | 5   | 10  | 50  |
| 101    | 1   | 5   | 10  | 50  |
| 110    | 1   | 5   | 10  | 50  |
| 118    | 1   | 5   | 10  | 50  |
| 138    | 1   | 5   | 10  | 50  |
| 149    | 1   | 5   | 10  | 50  |
| 153    | 1   | 5   | 10  | 50  |
| 180    | 1   | 5   | 10  | 50  |
| 187    | 1   | 5   | 10  | 50  |
| Labeled                                     |
| 28     | 10  | 10  | 10  | 10  |
| 52     | 10  | 10  | 10  | 10  |
| 70     | 10  | 10  | 10  | 10  |
| 101    | 10  | 10  | 10  | 10  |
| 118    | 10  | 10  | 10  | 10  |
| 138    | 10  | 10  | 10  | 10  |
| 141    | 10  | 10  | 10  | 10  |
| 153    | 10  | 10  | 10  | 10  |
| 180    | 10  | 10  | 10  | 10  |

| Table 2 SRM conditions: precursor ion and product ion (unit: m/z) and collision energy (unit: eV) |
|---------------------------------------------------------------|
| Compounds | Precursor ion | Product ion | Collision energy |
| TrCB      | 257.8         | 186          | 34              |
| TrCB      | 255.8         | 151          | 50              |
| TrCB      | 255.8         | 186          | 32              |
| TrCB-13C12| 269.8         | 198          | 34              |
| TrCB-13C12| 267.8         | 163          | 50              |
| TrCB-13C12| 267.8         | 198          | 32              |
| TeCB      | 291.8         | 222          | 34              |
| TeCB      | 291.8         | 220          | 34              |
| TeCB      | 291.8         | 257          | 12              |
| TeCB-13C12| 303.8         | 234          | 34              |
| TeCB-13C12| 303.8         | 232          | 34              |
| TeCB-13C12| 303.8         | 269          | 12              |
| PeCB      | 325.9         | 256          | 34              |
| PeCB      | 325.9         | 254          | 34              |
| PeCB      | 325.9         | 291          | 12              |
| PeCB-13C12| 337.9         | 268          | 34              |
| PeCB-13C12| 337.9         | 266          | 34              |
| PeCB-13C12| 337.9         | 303          | 12              |
| HxCB      | 361.7         | 290          | 30              |
| HxCB      | 359.8         | 290          | 35              |
| HxCB      | 359.8         | 325          | 15              |
| HxCB-13C12| 373.7         | 302          | 30              |
| HxCB-13C12| 371.8         | 302          | 35              |
| HxCB-13C12| 371.8         | 337          | 15              |
| HpCB      | 395.8         | 325.9        | 30              |
| HpCB      | 393.8         | 323.9        | 35              |
| HpCB      | 391.8         | 321.9        | 30              |
| HpCB-13C12| 407.8         | 337.9        | 30              |
| HpCB-13C12| 405.8         | 335.9        | 35              |
| HpCB-13C12| 403.8         | 333.9        | 30              |
0.1 mm at the tip. This column was adopted in the present study. The analysis cycle time using this column approximately corresponds to 13.5 min including the oven cooling time. In the ordinary data analysis of PCBs, it is necessary to perform data processing on 209 congeners, and this constitutes a time-consuming process similar to that of pretreatment. Japan’s insulating oil manual presents a method for quantifying the total PCB using statistical analysis based on the quantitative values of 13 components and is available online at http://www.env.go.jp/press/files/jp/17471.pdf (Ministry of Environment May 11, 2011). In this method, since only 13 components are the target components, it is overwhelmingly convenient when compared with the ordinary method. This method is applicable to GC/MS, GC/HRMS, and GC/MS/MS, but cannot be used with NCI. In this study, we investigate the applicability of the fast-GC triple stage quadrupole mass spectrometer with 13-component quantitation method to the analysis of reclaimed oil.

### Materials and methods

#### Regents, chemicals, and sample

Calibration solution of PCBs: EC-5488 and internal standard solutions; EC-5379, and EC-5450 were purchased from Cambridge Isotope Laboratories Inc. (MA, USA). The EC-5488 contains reference solutions of four concentrations, namely CS 1, CS 2, CS 3, and CS 4. The concentrations of these solutions are given in Table 1. The solutions were diluted with hexane to prepare 0.01, 0.05, 0.1, and 0.5 ng/mL unlabeled PCB congeners with 0.1 ng/mL 13C-labeled PCB congeners. The four standard solutions were used to prepare 4-point internal standard calibration curves with “ignore origin.” The reclaimed oil sample in which PCBs were not detected was prepared by Kiraku Kogyo Co., Ltd. (Shiga, Japan). Kanechlor (KC) (300, 400, 500, and 600) and KC-Mix (KC-300: KC-400: KC-500: KC-600 = 1:1:1:1) for preparing reclaimed oil containing PCBs were purchased from GL Sciences Inc. (Tokyo, Japan).

#### Sample preparation

Dilution of reclaimed oil with hexane was performed for the sample preparation. An evaluation was performed as to whether sample dilutions by 100, 300, and 500 times reduce the matrix effect. The diluted solutions were prepared by adding 1, 3, and 5 mL of 0.1 ng/mL internal solution to 0.010 g reclaimed oil. The evaluation was performed by comparing the diluted solutions to internal standard solutions for peak intensities as obtained by GC/MS/MS analysis. Specifically, 13C-labeled PCB congeners (#28, #52, #70, #101, #118, #138, #153, and #180) were used as the internal standards.

### Table 3 Thirteen components used for calculation and PCB congeners contained in each component

| Type of homologue | Component number | IUPAC number of major PCB congener |
|-------------------|------------------|-------------------------------------|
| TrCB              | 1                | 17 18                               |
|                   | 2                | 28 31                               |
| TeCB              | 3                | 49 52                               |
|                   | 4                | 44                                  |
|                   | 5                | 58 61 63 66 70 74 76                |
| PeCB              | 6                | 89 90 101 113                       |
|                   | 7                | 85 110 120                          |
|                   | 8                | 107 118 123                         |
| HxCB              | 9                | 139 140 147 149                     |
|                   | 10               | 132 153 168                         |
|                   | 11               | 130 138 158 160 163 164             |
| HpCB              | 12               | 175 182 183 187                     |
|                   | 13               | 172 180 191 193                     |

### Table 4 Table of correspondence between 13 target components and 13C-labeled PCB congeners as internal standards

| Component1 | Component2 | Component3 | Component4 | Component5 | Component6 | Component7 |
|------------|------------|------------|------------|------------|------------|------------|
| #28        | #28        | #52        | #52        | #70        | #101       | #118       |
| Component8 | Component9 | Component10| Component11| Component12| Component13|
| #118       | #153       | #153       | #138       | #180       | #180       |

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standard (IS) to correct the sensitivity of the peaks of the 13 components. The concentration of IS in the sample was adjusted to 0.1 ng/mL.

GC/MS/MS analysis and quantification

Additionally, PCB analysis was performed by GC/MS/MS (7890B GC/7010B triple quadrupole MS Agilent Technologies, USA). The samples were analyzed with a VF-Rapid MS PCB screen column (Agilent Technologies, USA), which corresponds to a wide bore analysis column (10 m, 0.53 mm, 0.25 μm) combined with pre-restrictor (0.6 m, 0.1 mm) with the following temperature programs: 85 °C for 1 min—40 °C/min—305 °C for 3 min. The analysis time only corresponds to 13.5 min per sample including cooling oven time. The GC/MS/MS was operated in selected reaction monitoring (SRM). The SRM conditions are given in Table 2. Quantitation of total PCB concentration by using the 13-component quantitation method was conducted by using the PCBs calculation software provided by EIS Japan Co., Ltd. (Shizuoka, Japan). Table 3 shows the 13 components used for calculation and PCB congeners contained in each component. Table 4 shows the correspondence between the 13 target components and the 13C-labeled PCB congeners as internal standards.

Results and discussion

Investigation of dilution rate

Figure 1 and Figure 2 show the SRM chromatograms of 0.1 ng/mL internal standard substances in 100, 300, and 500 times diluted reclaimed oil relative to the chromatograph of...
Fig. 2  SRM chromatograms of 0.1 ng/mL internal standard substances in hexane (reference) and 100, 300, and 500 times diluted reclaimed oil (#118, 153, 138, 180)

Table 5  Relative peak area values with respect to the peak area value of the hexane solution (reference) corresponding to 100

| Component   | #28 | #52 | #70 | #101 | #118 | #153 | #138 | #180 |
|-------------|-----|-----|-----|------|------|------|------|------|
| 100 diluted reclaimed oil | 93.0 | 72.5 | 56.7 | 60.7 | 32.9 | 33.7 | 17.1 | 4.76 |

Table 6  Signal to noise ratio of the 13 components at 0.01 ng/mL and noise calculation range

| Component   | Component1 | Component2 | Component3 | Component4 | Component5 | Component6 | Component7 |
|-------------|------------|------------|------------|------------|------------|------------|------------|
| Signal to noise | 22.1       | 16.0       | 6.5        | 6.0        | 6.0        | 4.1        | 5.0        |
| Noise calculation range | 2.90–3.00 min | 2.90–3.00 min | 3.45–3.55 min | 3.45–3.55 min | 3.45–3.55 min | 3.50–3.60 min | 3.50–3.60 min |
| Signal to noise | 6.9         | 11.8       | 8.3        | 10.3       | 9.5        | 15.5       |            |
| Noise calculation range | 3.50–3.60 min | 3.95–4.05 min | 3.95–4.05 min | 3.95–4.05 min | 4.00–4.10 min | 4.00–4.10 min |
0.1 ng/mL internal standard substances in the hexane solution (reference). Table 5 shows each relative peak area value of the chromatograms when the peak area value of reference is 100. With respect to the 100 times diluted solution, the area values of the four congeners with fast retention time (#28, #52, #70, and #101) exceeded half of the reference while the area values of three congeners with slow retention time (#118, #153, and #138) corresponded to 17.1–33.7% of the reference. Furthermore, the area value of #180 was 4.76% of the reference, and this resulted in a large decrease in strength. Conversely, with respect to the conditions involving 300 and 500 times, the area values of all congeners excluding congener #180 exceeded half of the reference, namely 54.4–147%. Furthermore, the area values of #180 corresponded to 38 and 43.8%. Hence, the influence of the matrix was suppressed at a dilution ratio equal to or exceeding 300 times. Therefore, the dilution ratio was determined as 300 times. It was thought the fact that the values of #28, #52, #70, #101, and #118 exceeded 100% is attributed to a positive matrix effect. As widely-known, the matrix effects in GC/MS analysis are negative as well as positive due to the occurrence of ion enhancement (Coelho and Franco 2016). However, the 13C-labeled PCB congeners were used as congeners in the internal standard substance, and thus it is assumed that the ion enhancement can be corrected.

**Calibration curve**

The 4-point (0.01, 0.05, 0.1, and 0.5 ng/mL) calibration curves with ignore origin by internal standard method were prepared for the 13 components. The 13 components at 0.01 ng/mL were detectable. The signal to noise (S/N) (peak to peak) values of each component corresponded to 4.1–22.1

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**Table 7** Analytical curve equation and the square of regression coefficient (R²)

| Component  | Analytical curve equation | R²     |
|------------|---------------------------|--------|
| Component1 (TrCB) | $y = 0.011258 x + 0.062471$ | 0.999913 |
| Component2 (TrCB) | $y = 0.010924 x + 0.083801$ | 0.999942 |
| Component3 (TeCB) | $y = 0.009558 x + 0.065022$ | 0.999710 |
| Component4 (TeCB) | $y = 0.009907 x - 0.077633$ | 0.997655 |
| Component5 (TeCB) | $y = 0.009053 x + 0.092591$ | 0.999340 |
| Component6 (PeCB) | $y = 0.009509 x - 0.057573$ | 0.999702 |
| Component7 (PeCB) | $y = 0.006831 x + 0.077679$ | 0.999794 |
| Component8 (PeCB) | $y = 0.006565 x + 0.054137$ | 0.999410 |
| Component9 (HxCB) | $y = 0.011756 x - 0.048442$ | 0.998965 |
| Component10 (HxCB) | $y = 0.011713 x - 0.009537$ | 0.999773 |
| Component11 (HxCB) | $y = 0.007932 x + 0.030521$ | 0.999812 |
| Component12 (HpCB) | $y = 0.008407 x - 0.019222$ | 0.999759 |
| Component13 (HpCB) | $y = 0.007983 x + 0.020156$ | 0.999865 |

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TeCB
m/z 225.8 → 186.0
TeCB
m/z 291.8 → 222.0
PeCB
m/z 325.9 → 256.0
HxCB
m/z 359.8 → 290.0
HpCB
m/z 393.8 → 323.9

Fig. 3 SRM chromatograms of the reclaimed oil containing 0.5 μg/mL KC-mix
The analytical curve equation and square of regression coefficient ($R^2$) of the calibration curves are listed in Table 7. The $R^2$ values of all calibration curves exceeded 0.997.

Japanese regulations specify that the maximum residue limit is 0.5 $\mu$g/mL and that the limit of detection (LOD) in analysis

### Table 8

|        | 1  | 2  | 3  | 4  | 5  | Average | SD   | %RSD |
|--------|----|----|----|----|----|---------|------|------|
| 0.5 $\mu$g/mL | 0.533 | 0.500 | 0.520 | 0.490 | 0.495 | 0.508 | 0.0181 | 3.6 |
| 0.05 $\mu$g/mL | 0.0741 | 0.0730 | 0.0818 | 0.0779 | 0.0720 | 0.0758 | 0.00408 | 5.4 |

**Trueness of total PCBs concentration**

Fig. 5 Comparison of SRM chromatograms of IS on June 23, 2016 and August 30, 2016 (#28, 52, 70, 101)
method is less than 0.15 μg/mL. Therefore, it is necessary to correctly quantify 0.5 μg/mL. Reclaimed oil containing 0.15 and 0.5 μg/mL Kanechlor standard was analyzed, and the total PCB concentration was calculated. Additionally, reclaimed oil containing lower and higher concentrations (0.05 and 2.0 μg/mL) were also analyzed. The samples of reclaimed oil containing 0.05, 0.5, and 2.0 μg/mL of the KC-mix were repeatedly analyzed five times. Accuracy is derived as follows: [The measured value]/[The true value] × 100 (%). The targeted 13 components were detected in all the reclaimed oil samples by adding the KC standard. Figure 3 shows the SRM chromatograms of the reclaimed oil containing 0.5 μg/mL KC-mix. Figure 4 shows the bar chart with error bars indicating the accuracy of the total PCB concentration. The accuracy values of 0.5 μg/mL were 94.0–102%. Additionally, the accuracy values of 0.15 and 2.0 μg/mL corresponded to 91.5–116% and 99.9%, respectively. The results indicate that the quantification of the concentrations in the range of 0.15 to 2.0 μg/mL was accurate. Conversely, the accuracy values of 0.05 μg/mL were 144–164%, and this exceeded the actual concentration. However, this method can be applied sufficiently to PCB analysis in reclaimed oil given that 0.5 μg/mL denotes the judgment criteria.

### Repeatability and stability

Table 8 shows the relative standard deviation (%RSD) of total PCB concentration (μg/mL) in reclaimed oil containing 0.5 and 0.05 μg/mL KC-mix, and the %RSD values were 3.6 and 5.4%, respectively. This indicates good repeatability even at low concentrations. Figures 5 and 6 show the SRM chromatograms of IS obtained by analysis of the reclaimed oil on June 23, 2016 and on August 30, 2016, respectively. We analyzed 800 or more samples of reclaimed oils between the two analyses. During these 800 or more analyses, a cleaning ion source and an exchanging analysis column were not performed. On the other hand, an exchanging of GC liner and a tuning of quadrupole mass were performed several times. Table 9 shows the signal to noise (S/N) (peak to peak) on June 23, 2016 and August 30, 2016, and the reduction rate.

|       | #28 | #52 | #70 | #101 | #118 | #138 | #153 | #180 |
|-------|-----|-----|-----|------|------|------|------|------|
| June 23, 2016 | 67.1 | 25.7 | 29.1 | 45.2 | 40.1 | 32.5 | 37.4 | 28.0 |
| August 30, 2016 | 48.8 | 23.8 | 22.6 | 9.3  | 7.9  | 29.3 | 31.7 | 29.9 |
| Reduction rate(%) | 73  | 93  | 78  | 21   | 20   | 90   | 85   | 107  |
sensitivity of components 6, 7, and 8 that corresponds to 5 chlorinated. However, the data shown in “Calibration curve” and “Trueness of total PCBs concentration” were acquired after August 30, 2016 without cleaning ion source and exchanging analysis column. Therefore, this reveals that the proposed method was stable and maintains the required sensitivity even when 800 samples were analyzed.

Conclusion

In this study, a method to analyze PCBs in reclaimed oil was proposed by using fast-GC triple stage quadrupole MS/MS with 13-component quantitation method. The method consists of pretreating 300-fold dilution with hexane, GC/MS/MS analysis of 13.5 min per sample using VF-Rapid MS PCB screen column, and quantitative calculation with 13 components. The method was adopted for the analysis of reclaimed oil containing 0.5 μg/mL PCBs, and this denotes that judgment criteria including accurate quantitation (accuracy value, 94.0–102%) and good repeatability (%RSD, 3.6%) were obtained. Additionally, with respect to the stability of GC/MS/MS system, the required sensitivity was maintained even when 800 samples were analyzed without a cleaning ion source and an exchanging analysis column. These results indicate that the proposed fast and simple analysis method satisfies sensitivity, repeatability, and stability requirements.

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