Development and Validation of a Gas Chromatography-Mass Spectrometry Method for the Determination of PCBs in Transformer Oil Samples-Application on Real Samples from Botswana

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Abstract

A procedure in which a retention time window for the determination of polychlorinated biphenyls (PCBs) congeners including those not presented in this study but of similar molar masses in transformer oil has been developed and validated. This procedure was successfully applied in the determination of transformer oil samples from Botswana for comparison with a preliminary inventory for PCB levels in transformer oil and other receptacles suspected to contain PCBs and developed during the formulation of the Botswana National Implementation Plan (NIP) for the sound management of persistent organic pollutants (POPs). Recoveries in the developed procedure initially meant for 39 PCB congeners ranged from 44.5±0.31 to 111.9±4.75% uncorrected for bias, while lineairties i.e. r² values ranged from 0.9624 to 0.9999 with method detection limits (MDLs) varying from 0.02 to 0.13 ppm (w/v). Application of this procedure on real samples from Botswana showed that the transformer oil contained no PCBs although the preliminary data using screening equipment and kits for indicative inventory data showed high PCB contents in some cases of as much as 456 ppm (w/v). It is proposed here that this method be adopted in the SADC region for confirmation of presence and determination of the levels of PCBs where they are detected in order to improve on the preliminary inventories that were developed during the NIP development exercise for the Stockholm Convention (SC) in these countries.

Keywords: PCBs; GC-MS; Botswana; Stockholm convention; SADC; National implementation plan

Introduction

Polychlorinated biphenyls (PCBs) are a class of persistent organic pollutants (POPs) used as dielectric fluids because of their chemical, physical stability and electrical insulating properties [1]. They are compounds synthesized from the substitution of hydrogen atoms by chlorine atoms in the biphenyl molecule as shown in Figure 1. There are 209 possible PCB congeners that have only been synthesized under laboratory conditions [2].

Investigations in many parts of the world have revealed widespread diffusion of PCBs in the environment [3]. Between 1950 and 1980, over 1.3 million tons of PCBs were produced worldwide [4]. Although the manufacture of PCBs since the 1970s was largely banned, their continued use in existing materials was not. The interest in PCBs as environmental pollutants began in 1966 when they were catalogued as hazardous waste [5].

Due to their physical and chemical properties, PCBs are highly persistent in the environment and tend to accumulate progressively in soils, plants and animals and proportionally to the trophic level in the food chain and are classified as probably carcinogenic [6,7]. Due to the increased awareness that persistent organic pollutants (POPs) pose to human health and the environment, the Governing Council of the United Nations Environment Programme (UNEP) in May 1995 requested in its decision 18/32 that an international assessment process be undertaken of an initial list of 12 POPs (aldrin, chlordane, 1,1,1-trichloro-2,2-di(4-chlorophenyl)ethane (DDT), dieldrin, dioxins, endrin, furans, hexachlorobenzene, heptachlor, mirex, PCBs and toxaphene). The Council also requested that the Intergovernmental Forum on Chemical Safety (IFCS) develop recommendations on international action for consideration by the UNEP Governing Council and the World Health Assembly no later than 1997 [8]. Negotiations led to the adoption of the Stockholm Convention (SC) on POPs, which was opened for signature at a Conference of Plenipotentiaries held from 22 to 23 May 2001 in Stockholm, Sweden (Stockholm Convention, 2004). The SC, which entered into force in 2004, provides for the protection of human health and the environment from POPs. The SC outlines measures for monitoring and elimination of PCBs in transformers and other receptacles by 2025.

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Botswana became party to the SC on the 28th of October 2002. As such Botswana is obliged to implement requirements of the SC. The Botswana National implementation plan (NIP) for POPs, showed that there were about 500 pieces of equipment containing PCBs, thus a total oil of about 16,520 cubic metres suspected to contain PCBs in equipment in use [9]. Indicative results of the preliminary screening exercise used for the preparation of the NIP in Botswana for the presence of PCBs in transformer oil and other receptacles, were done using analytical equipment and test kits such as the CLOR-N Oil 50 that would only provide general tests and therefore preliminary indicative data. This was the same in most other SADC state parties to the SC.

While several methods [10-20] have been used for the determination of PCBs in transformer oil and other matrices, suffice to mention that adaptability of these methods depends on a particular region or geographical location of the analysts. This is mainly due to the cost associated with acquisition of instruments, their service/maintenance and affordability of reagents and other consumables. Some examples of extraction methods that have been employed in the determination of PCBs in transformer oil are solvent extraction by shaking, solvent extraction in a soxhlet or Soxtec apparatus, focused microwave-assisted solvent extraction (FMASE), in a soxhlet apparatus, ultra sonication extraction (USE), microwave-accelerated extraction (MAE), accelerated solvent extraction (ASE; also known as PFE or PLE), supercritical fluid extraction (SFE), membrane extraction techniques and solid-based extraction techniques [21]. A recent study [22] compared the efficiencies of extraction of PCBs in food with different sorbents including florisil but did not include the sulfoxide sorbent. This study employed electron capture detector (ECD) for detection. The main detectors that have been used are the ECD and mass spectrometry employing various mass analysers such as the quadrupole ion trap (QIT), quadrupole and the time of flight in the electron ionization mode [23].

The study reported here focused on developing a simple, robust, selective and sensitive retention time window that can be used to determine the concentrations of PCBs in transformer oil and other receptacles suspected to contain PCBs in the Southern African Development Conference (SADC) region. Eventually, all SADC countries could compare their preliminary PCB inventories in their NIPs with the data adduced from scientific determination such as this one. Samples in this study were taken from some sites in Botswana and determined for the presence of PCBs using the method developed in this study.

Experimental
Reagents and chemicals

HPLC grade hexane, acetone and sulphuric acid were purchased from Merck South Africa (SA). The Solid phase extraction (SPE) cartridges, Florisil sep-pak vacuum 20 cc, 2 g, were purchased from Waters Ireland. Supelclean™ Sulfoxide sep-pak vacuum 12 cc, 3 g were purchased from Supelco Analytical USA. The PCB standards including the analytical standard containing 39 PCB congeners mix, C-CS-01, 10 µg/mL were all purchased from AccuStandard Inc. (New Haven, CT, USA).

Apparatus

The Agilent 5975 C inert XL EI/CI MSD and the Waters GCT Premier GC-MS system were used. Solid phase extraction (SPE) was carried out on an IST Vacc master with 10 sample processing manifolds acquired from Biotage®, Charlotte, NC, USA. This is a vacuum manifold (Vacc Master) with capabilities of adjusting the pressure to attain the desired flow rate.

Chromatographic conditions

The optimization of the Gas Chromatography (GC) separation parameters for the PCBs was done using an Agilent GC 6890 equipped to an Agilent 7683 B series injector. The detector used was a Time of Flight (TOF) GCT Premier Micro-Mass mass spectrometer. The system was operated using the Masslynx Version 4.1 acquisition software. The experimental protocol was designed in such a way that the optimized separation parameters would be transferred to another Agilent GC system equipped with a similar injector. The column used was a DB5-MS (methylpolysiloxane, 5% Phenyl groups), 30 m x 0.25 mm i.d., and 0.25 µm thickness. After optimization of the separation parameters, mass spectrometry parameters were optimized using an Agilent GC 7890A coupled to an Agilent 5975 C inert XL EI/CI MSD with a Triple axis detector manufactured by Agilent technologies, USA. The rest of the experiments including method detection limits were determined using this instrument. The instrument has the capability of being operated in both full and selected ion monitoring (SIM) scan modes. On this system, an HP5-MS column (methylpolysiloxane, 5% phenyl groups), 30m x 0.25mm i.d., 0.25µm thickness which is equivalent to the DB5-MS column used on the GCT Premier was used. Helium was used as a carrier gas at a flow rate of 1 mL/min. 1 µl of sample/standard solutions was injected. The GC optimized oven program was as follows: The column was held at 80°C for 2 min and then ramped at 10°C/min up to 270°C. The injector was held at an optimized temperature of 270°C.

Mass spectrometry

The mass spectrometer was operated in the electron ionization (EI) mode at electron energy of 70 electron volts (eV) and a scan range of 50-500 m/z. The emission current set by the instrument autotune function was 34 µA. The source and analyzer temperatures were optimized and held at 230 and 150°C, respectively, throughout. The SIM and full scan modes of operation were compared and the best mode used in the quantification of real samples. The acquisition software on this system was MSD Chemstation. Data analysis was accomplished by a combination of Productivity Chemstation and the Automated Mass Spectral Deconvolution and Identification System (AMDIS) by the National Institute for Standards and Technology (NIST).

Sample collection and storage

The sample used for method validation was one of the transformer oils collected during an inventory exercise conducted in collaboration with the Botswana Department of Waste Management and Pollution Control (DWMPC) in November 2010. Samples were collected in glass sampling bottles initially rinsed with n-hexane 95% UL (HPLC grade). 25 transformer oil samples were collected and stored in ice and transported to the laboratory for storage in the cold room maintained at 4°C before extraction and analysis. In this study, six out of twenty five samples were analyzed. Sample collection from the transformers entailed opening the drainage tap on the side of the transformer and draining the oil into the glass sample bottle. Some transformers did not have a drainage tap and these were sampled using a glass pipette previously rinsed in hexane. The coordinates for each sample were not recorded for future sampling and analysis except for the transformers, which were located in, protected areas. These transformers’ coordinates were not recorded. The coordinates are shown in Table 1.
Sample preparation

Samples: The samples were diluted in the ratio of 1:500 v/v transformer oil samples to n-hexane. 2 mL of the diluted transformer oil sample was cleaned up with 5 mL concentrated sulphuric acid. This was adopted from the USEPA Method 3665A and slightly modified by adjusting the volumes of sulfuric acid added. The method involved shaking the mixture with concentrated sulphuric acid for a minute, allowing the phases to separate, removing the acid layer and repeating the process until the acid layer was no longer coloured. The concentrated sulphuric acid charred most of the transformer oil resulting in a crude black mixture of acid and oil. After acid treatment, the treated sample was washed twice with 5 mL fractions of de-ionised water to remove any traces of acid. Each 5 mL wash was done in a minute. The extract was passed through anhydrous sodium sulphate analytical reagent (AR) quality to remove moisture. The sample was ready for SPE clean up.

SPE Clean up and extraction: A comparison of the PCB extraction efficiencies was done using two types of SPE cartridges. The Supelco supelclean sulfoxide 3 g, 6 mL cartridges and the Waters florisil, 2 g sorbent, 12 cc.

Supelclean Sulfoxide SPE cartridges: The SPE normal procedure of conditioning, loading, washing and elution was followed. The conditioning was accomplished by eluting 10 mL of acetone to remove residual moisture from the supelclean sulfoxide cartridges. This was followed by adding 20 mL of n-hexane to equilibrate the cartridges. The pre-treated sample was loaded onto the cartridge and washed with 5.5 mL of n-hexane. Elution was done with 13 mL of n-hexane. The eluate was concentrated to 1 mL by gentle nitrogen blow. The samples extracts were transferred into GC vials ready for analysis.

Florisil SPE cartridges: Conditioning for the florisil cartridges was done by passing 8 mL of hexane through the cartridge. The solvent was not available.

Table 1: Coordinates for some transformer samples collected during the inventory exercise.

| Sample Code | Coordinate                  | Sample Type        | Transformer Fabrication Date | Sample Collection Date |
|-------------|-----------------------------|--------------------|------------------------------|------------------------|
| SSKA 004    | E. 24°43'37.1" S. 25°55'22.6" | Transformer oil   | 1983                         | 04/11/2010             |
| SSKA 009    | E. 24°43'37.1" S. 25°55'22.6" | Transformer oil   | 1983                         | 04/11/2010             |
| SSKA 024    | E. 24°32'39.8" S. 25°55'58.8" | Transformer oil   | 1990                         | 04/11/2010             |
| SSKB 002    |                             | Transformer oil   | 1989                         | 05/11/2010             |
| Glen Valley 006 |                             | Transformer oil       | n/a*                         | 05/11/2010             |
| Mnaminasha 003 | E. 24°31'17.9" S. 23°59'18.5" | Transformer oil   | 1998                         | 05/11/2010             |

*n/a- not available
** Transformers in security protected areas

Table 2: Retention times, AMDIS match factors and SIM ions.

| Congener                  | Retention Time (min) | % Match Factor (simple) | SIM ion (m/z) | Congener                  | Retention Time (min) | SIM ion (m/z) | % Match Factor (simple) |
|--------------------------|----------------------|-------------------------|---------------|--------------------------|----------------------|---------------|-------------------------|
| 2-Chlorobiphenyl         | 10.30                | 97                      | 188           | 2,2',3,4,5-Pentachlorobiphenyl | 18.18               | 326           | 98                      |
| 3-Chlorobiphenyl         | 11.45                | 98                      | 188           | 2,2',3,4,5-Pentachlorobiphenyl | 18.64               | 326           | 97                      |
| 4-Chlorobiphenyl         | 11.57                | 96                      | 188           | 2,2',4,4',5-Pentachlorobiphenyl | 18.83               | 326           | 97                      |
| 2,2'-Dichlorobiphenyl    | 12.07                | 99                      | 222           | 2,3,3',5,5'-Hexachlorobiphenyl | 19.06               | 360           | 96                      |
| 2,3'-Dichlorobiphenyl    | 12.79                | 100                     | 222           | 2,2',3,4,5,5'-Hexachlorobiphenyl | 19.20               | 360           | 96                      |
| 2,4'-Dichlorobiphenyl    | 13.06                | 100                     | 222           | 2,2',3,4,5,5'-Hexachlorobiphenyl | 19.68               | 360           | 96                      |
| 2,5'-Dichlorobiphenyl    | 13.23                | 100                     | 222           | 2,2',3,4,5,5'-Hexachlorobiphenyl | 19.82               | 360           | 96                      |
| 2,2',3-Trichlorobiphenyl | 13.69                | 99                      | 256           | 2,2',3,3',4,5,5'-Heptachlorobiphenyl | 20.06               | 394           | 96                      |
| 2,2',5-Trichlorobiphenyl | 14.27                | 100                     | 256           | 2,2',3,4,5,6-Hexachlorobiphenyl | 20.36               | 360           | 96                      |
| 2,2',6-Trichlorobiphenyl | 14.72                | 99                      | 256           | 2,2',3,4,5,6-Hexachlorobiphenyl | 20.67               | 394           | 94                      |
| 2,3,4'-Trichlorobiphenyl | 15.26                | 99                      | 256           | 2,2',3,4,5,6-Hexachlorobiphenyl | 21.22               | 394           | 94                      |
| 2,3',4-Trichlorobiphenyl | 15.44                | 99                      | 256           | 2,2',3,4,5,6-Hexachlorobiphenyl | 21.34               | 394           | 94                      |
| 2,4,4'-Trichlorobiphenyl | 15.82                | 99                      | 256           | 2,2',3,4,5,6-Hexachlorobiphenyl | 21.60               | 394           | 94                      |
| 2,2',3,5'-Tetrachlorobiphenyl | 16.20              | 98                      | 292           | 2,2',3,4,5,6-Hexachlorobiphenyl | 21.93               | 394           | 97                      |
| 2,2',5,5'-Tetrachlorobiphenyl | 16.63              | 98                      | 292           | 2,2',3,4,5,6-Octachlorobiphenyl | 22.89               | 430           | 94                      |
| 2,3,3',4'-Tetrachlorobiphenyl | 16.86              | 98                      | 292           | 2,2',3,4,5,6-Octachlorobiphenyl | 23.08               | 430           | 96                      |
| 2,3',4,4'-Tetrachlorobiphenyl | 17.23              | 98                      | 292           | 2,2',3,4,5,5',6-Octachlorobiphenyl | 24.05               | 430           | 94                      |
| 2,3',4,5-Tetrachlorobiphenyl | 17.46              | 98                      | 292           | 2,2',3,4,5,5',6-Octachlorobiphenyl | 24.80               | 430           | 92                      |
| 2,3',4,6-Tetrachlorobiphenyl | 17.60              | 98                      | 292           | 2,2',3,4,5,5',6-Nonachlorobiphenyl | 26.25               | 464           | 89                      |
| 2,4,4',5'-Tetrachlorobiphenyl | 17.94              | 97                      |               |                          |                      |               |                         |

Table 2: Retention times, AMDIS match factors and SIM ions.
allowed to soak the sorbent bed for 5 minutes before it was allowed to pass through. The sample was loaded onto the cartridge, washed with 5 mL of hexane and eluted with 10 mL of hexane. The initial steps of sample pre-treatment were followed, as in the supelclean sulfoxide cartridges, i.e. spiking of oil sample and acid treatment.

Results and Discussion

A mixture of 39 PCB congeners i.e. mix C-CS-01, 10 µg/mL which was used through out this work was identified using AMDIS version 3.2 developed by NIST. Broadly and as a rule of the thumb, a simple match factor between 70 and 80% indicated a good match, between 80 and 90% is a very good match and between 90 and 100% is an excellent match and thus confirms the presence of the analyte of interest. Results of the deconvolution using a combination of parameters are shown in Table 2. Table 2 also shows the ions that were selected to be used in all subsequent SIM experiments. The minimum match factor was that of 2,2',3,4,5,6-hexachlorobiphenyl, at 87%. Almost all the other congeners had match factors above 90% with the highest being 100%. As seen from Table 2, there was an unusual elution pattern which was observed. The general elution pattern as has been observed previously [24] showed an increase in the retention time with an increase in the number of chlorine atoms. However, 2,2',3,4,5,6-heptachlorobiphenyl with 7 chlorine atoms eluted earlier than 2,2',4,4',5,5'-hexachlorobiphenyl with 6 chlorine atoms which was confirmed by AMDIS. We speculated that this was due to the distribution of the chlorine atoms around the two benzene rings. Even though the hexachlorobiphenyl has less chlorine atoms, the benzene ring for the heptachlorobiphenyl is hindered by the chlorine atoms thus preventing interaction with the non polar stationary phase material and thus results in the quicker elution of the heptachlorobiphenyl. In contrast, the 2,2',4,4',5,5'-hexachlorobiphenyl congener, the benzene rings are less esoterically hindered by the chlorine atoms thus increasing interactions with the non polar stationary phase resulting in longer retention of this congener.

The Optimized conditions for the Supelclean Sulfoxide and Florisil SPE cartridges were determined as shown in Table 3 below.

Recoveries using the two cartridges with optimum extraction conditions

Figure 1 shows the recoveries obtained. From the results, the florisil cartridge exhibited selectivity towards early eluting PCB congeners, while the Supelclean Sulfoxide cartridge showed on average reasonable recoveries for all PCB congeners. This effect confirms the non-selectivity of the Supelclean Sulfoxide cartridge whose adsorption properties are hinged on the interaction of the electrophilic sulfur atom and the π electron cloud arising from the aromatic rings of the PCB congeners, implying the chlorine atoms do not significantly affect this interaction.

Comparison of full and SIM scan modes

Since the Supelclean Sulfoxide cartridge showed the best extraction characteristics of the two cartridges, experiments were designed to compare full and SIM scan modes performance of the mass spectrometer. Validation and real sample analysis experiments were also done using the Supelclean Sulfoxide cartridge because of this observation. Initially, the peak areas obtained by running a sample extract which was spiked with 0.20 ppm of the 39 congener mix standard and initially screened and found to contain no PCBs, in full and SIM scan modes. The results of this comparison are shown in Figure 2 below.

Results from these experiments showed significant signal loss when SIM is employed as the scan mode. This phenomenon has been observed before [25] since isolation of one or a set of ions implies signal loss compared to full scan where all ions can be used for quantification purposes. However, when method detection limits (MDLs) were compared using full scan and SIM, the opposite was observed as seen in Figure 4. This was due to the enhanced selectivity that SIM affords and thus ultimately compensates the loss in sensitivity due to signal loss as seen in Figure 3 which is a contrast of peak areas obtained in the full and SIM scan modes.

Figure 4 shows a comparison of the MDLs, which were determined by extracting a spiked transformer oil sample, with 0.2 ppm of the 39-congener mix standard and which was initially screened and found to contain no PCBs. The spiked sample extracts were analyzed in full and SIM scan modes using the Supelclean™ Sulfoxide SPE cartridge.

The runs for these extracts were repeated 7 times in each scan mode and the run for the pure 0.2 ppm standard repeated 3 times. The peak areas and the standard deviation for seven injections were computed, and the corresponding peak areas for the extracts converted to their corresponding concentrations using the peak areas for the 0.2 ppm of the 39 congener mix standard. The MDLs were calculated by multiplying the standard deviation, s, and the student t-value at 6 degrees of freedom and 95% confidence limit as shown in Equation 1 below:

$$\text{MDL} = ts$$  \hspace{1cm} (1)

Figure 5a is a full scan total ion chromatogram (TIC) of the 39 PCB congeners while Figure 5b is a SIM ion chromatogram of the same. The two chromatograms show the selectivity that SIM affords in contrast to the earlier.

Method Validation

Results obtained for the instrument detection limits (IDLs), limits of quantitation (LOQs), MDLs and IDLs, are shown in Table 3. The LOQs were obtained in a similar manner as the MDLs except that pure standards were used instead of sample extracts for the seven injections that afforded the required standard deviations used in Equation 1. In contrast to MDLs, IDLs were lower followed by LOQs. This was expected because there is only the noise arising from the instrument when IDLs are evaluated as opposed to two noise components i.e. from the instrument and the matrix in the evaluation of MDLs. The IDLs ranged from 0.004 to 0.08 ppm. The MDLs ranged from 0.02 to 0.13 ppm. The LOQs ranged from 0.01 to 0.3 ppm.

The lineairities of the calibration curves for LOQ values were determined are shown in Table 3. The values ranged from 0.9624 to 0.9999. A selected congener peak was used as an internal standard to quantify all the other 38 congeners. At the end of this, another congener was selected and its peak used to quantify the first congener, which was used as an internal standard for the other 38. The percentage recoveries for the
congeners using the Spelclean™ Supelco cartridges and SIM are also shown in Table 4. From the results obtained, the percent recoveries for the congeners ranged from 44.53±0.31 to 111.93±4.75% uncorrected for bias. Three congeners were recovered at a higher than normal level of greater than 100%. Analysis of the samples, however, showed that no PCB congeners were present and as such the greater than 100% recovery could have been due to instrumental noise resulting in fluctuations in the peak areas of the analyte peaks in the spiked sample.
Application to real world samples

All six samples that were analyzed using the method described here were found to contain no PCBs as shown in Table 4. These results were compared with the ones obtained using the Dexsil L2000 DX, version 1.27 PCB/chloride analyzer. The Dexsil L2000 PCB/chloride analyzer extracts PCBs from transformer oil and dissociates them using a sodium reagent thus freeing chloride ions. The instrument then uses a chloride ion selective electrode (ISE), employing the 1242 oil method, which is embedded in the instrument [26]. Results that were originally obtained by this instrument are also shown in Table 4. The results showed that the transformer oil samples contained PCBs at the levels indicated. Perhaps the reason for this is that, the DX analyser responds to the presence of chloride ions in solution. This implies that any chloride in a sample regardless of its source could be detected to give a false positive. The method developed in this study gave 0.0189 ppm as the lowest MDL, an inference that no PCBs were present in the samples as stipulated by the SC can be made and thus the results given by the DX analyzer were false positives.

Conclusions

A rapid, selective and sensitive procedure for the determination of PCBs in transformer oil has been developed, validated and tested on real samples. The only disadvantage of the method would relate to the cost, service and maintenance of the instrumentation involved. The procedure proved to be robust and can be adopted for use in the SADC region where screening for POPs such as PCBs is mandatory due to the fact most of the countries that make up the region are party to the SC. The results obtained from real samples showed that the test kits for preliminary investigations to aid establishment of baseline inventories in the formulation of NIPs for SADC State Parties to the SC could have...
were established to aid in developing National Implementation Plans (NIPs). The one proposed here and compare them to baseline inventories that

been unreliable. This places the need to re-screen all suspected PCB containing equipment in the SADC region using reliable methods like

2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl 0.0098 0.0326 0.9995 \( y = 2.51x + 0.0705 \) 0.0420 54.08±3.43

2,2',3,3',4,4'5,6-Octachlorobiphenyl 0.0192 0.0641 0.9979 \( y = 2.64x + 1.44 \) 0.0349 57.41±0.80

2,2',3,3',4,5,5',6-Octachlorobiphenyl 0.0162 0.0533 0.9985 \( y = 1.93x + 0.231 \) 0.0530 72.47±0.80

2,2',3,3',4,5,5',6-Nonachlorobiphenyl 0.0098 0.0326 0.9995 \( y = 2.51x + 0.0705 \) 0.0420 54.08±3.43

Table 4: IDLs, LOQs, MDLs,\( r^2 \) values, regression equations and percent recoveries using the Supelclean™ Sulfoxide cartridge for the 39 PCB congeners.

Table 5: Results of six real transformer oil samples.

Table 5: Results of six real transformer oil samples.

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