DETERMINATION OF DIOXIN-LIKE POLYCHLORINATED BIPHENYL IN PIG FEED BY GAS CHROMATOGRAPHY TRIPLE QUADRUPOLE MASS SPECTROMETRY IN COMPARISON WITH HIGH-RESOLUTION GAS CHROMATOGRAPHY/HIGH-RESOLUTION MASS SPECTROMETRY

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ABSTRACT

This study evaluates and compares the performance of gas chromatography triple quadrupole mass spectrometry and high-resolution gas chromatography/high-resolution mass spectrometry for determining dioxin-like PCBs (dl-PCBs) in pig feed. The gas chromatography triple quadrupole mass spectrometry method exhibited strong analytical performance, with limits of detection ranging from 0.03 to 0.05 pg/g and recovery efficiencies between 89% and 103%. It demonstrated good repeatability, reproducibility, and accuracy. Most toxic dioxin-like polychlorinated biphenyl congeners were found in all feed samples, except PCB#81 in P1 and PCB#157 in P2 and P3. The total toxic equivalent values ranged from 0.019 to 0.039 pg TEQ/g. The toxic equivalent results obtained by gas chromatography triple quadrupole mass spectrometry were in close agreement with those from high-resolution gas chromatography/high-resolution mass spectrometry, with relative percent differences generally below 10%, indicating high method concordance. These findings confirm that of gas chromatography triple quadrupole mass spectrometry is a reliable and efficient alternative to high-resolution gas chromatography/high-resolution mass spectrometry for routine monitoring of dioxin-like polychlorinated biphenyls in complex feed samples. Its precision, sensitivity, and cost-effectiveness make it well-suited for regulatory compliance and ensuring animal feed safety.

XÁC ĐỊNH POLYCHLORINATED BIPHENYL TƯƠNG TỰ ĐIOXIN TRONG THỨC ĂN CHO LỢN BẰNG PHƯƠNG PHÁP SẮC KÝ KHÍ KHỐI PHỔ BA TỨ CỰC SO SÁNH VỚI PHƯƠNG PHÁP SẮC KÝ KHÍ PHÂN GIẢI CAO/PHỔ KHỐI PHÂN GIẢI CAO

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TỪ KHÓA

dl-PBCs
An toàn thức ăn chăn nuôi
Thức ăn cho lợn
GC-MS/MS
HRGC/HRMS

TÓM TẮT

Nghiên cứu này đánh giá và so sánh hiệu suất của phương pháp sắc ký khí khối phổ ba tứ cực và phương pháp sắc ký khí phân giải cao/phổ khối phân giải cao để xác định PCB tương tự dioxin trong thức ăn cho lợn. Phương pháp sắc ký khí khối phổ ba tứ cực cho thấy hiệu suất phân tích mạnh mẽ, với giới hạn phát hiện từ 0,03 đến 0,05 pg/g và hiệu suất thu hồi từ 89% đến 103%. Phương pháp này cho thấy khả năng lặp lại, khả năng tái lặp và độ chính xác tốt. Hầu hết các đồng loại dl-PCB độc hại đều được tìm thấy trong tất cả các mẫu thức ăn, ngoại trừ PCB#81 ở P1 và PCB#157 ở P2 và P3. Giá trị tổng độc tính tương đương (TEQ) dao động từ 0,019 đến 0,039 pg TEQ/g. Kết quả TEQ thu được bằng phương pháp sắc ký khí khối phổ ba tứ cực gần giống với kết quả từ phương pháp sắc ký khí phân giải cao/phổ khối phân giải cao, với chênh lệch phần trăm tương đối thường dưới 10%, cho thấy sự nhất quán cao giữa các phương pháp. Những phát hiện này khẳng định rằng sắc ký khí khối phổ ba tứ cực là một phương pháp thay thế đáng tin cậy và hiệu quả cho sắc ký khí phân giải cao/phổ khối phân giải cao để theo dõi thường quy PCB tương tự dioxin trong các mẫu thức ăn phức tạp. Độ chính xác, độ nhạy và hiệu quả về chi phí của phương pháp này hoàn toàn phù hợp với quy định và hỗ trợ đảm bảo an toàn thức ăn chăn nuôi.

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1. Introduction

Polychlorinated biphenyls (PCBs), a group of 209 synthetic chlorinated compounds, have been widely used in industrial applications such as dielectric fluids, flame retardants, and hydraulic systems due to their chemical stability and thermal resistance [1], [2]. Among these, twelve congeners exhibit toxicological behavior similar to dioxins and are classified as dioxin-like PCBs (dl-PCBs) [3]. These compounds are highly lipophilic, persistent in the environment, and bioaccumulate through the food chain, posing significant risks to human and animal health. The toxic effects of dl-PCBs include carcinogenicity, endocrine disruption, immunosuppression, and developmental toxicity, prompting global regulatory measures to monitor and control their presence in food and feed [4] - [6].

Animal feed, particularly pig feed, represents a major route of dl-PCB contamination in the food production system [7], [8]. Livestock consuming contaminated feed may accumulate these toxicants in adipose tissues, leading to their transfer to meat and other edible products consumed by humans. Given the potential for chronic exposure and associated health risks, monitoring dl-PCBs in feed products by maximum residue limits (MRLs) established by regulatory authorities such as the European Union (EU) is essential [9]. Accurate, sensitive, and cost-effective analytical techniques are therefore required for the routine surveillance of dl-PCBs in complex feed matrices. Traditionally, high-resolution gas chromatography coupled with high-resolution mass spectrometry (HRGC/HRMS) has been the benchmark method for determining dioxins and dl-PCBs in environmental and food samples [10], [11]. HRGC/HRMS provides excellent sensitivity, selectivity, and precision, enabling the detection of ultra-trace levels of these contaminants. Gianfranco Diletti et. al. [12] developed a semi-automated HRGC-HRMS method for determining 12 dl-PCBs in feed and animal-based foods, achieving reduced sample preparation time, good specificity, low detection limits (0.2–1.3 pg/g fat), high recoveries (> 80%), and successful application to 177 samples. However, the application of HRGC/HRMS in routine monitoring is often limited by high equipment and operational costs, long analysis time, and the need for highly trained personnel [13]. These constraints have led the search for cheaper, more efficient, and similar analytical alternatives.

In recent years, gas chromatography coupled with triple quadrupole mass spectrometry (GC-MS/MS) has gained increasing attention as a viable alternative for dl-PCBs determination [14], [15]. Modern GC-MS/MS instruments, operated in multiple reaction monitoring (MRM) mode, offer high selectivity and low detection limits, making them suitable for analyzing trace contaminants in complex matrices. In the study [16], a GC-MS/MS method was developed for trace analysis of PCDD/Fs and dl-PCBs in food and feed. Detection limits ranged from 0.018 – 0.17 pg/g for PCDD/Fs and 0.13 – 0.36 pg/g for dl-PCBs. The method showed good repeatability at 10 pg/mL for 2,3,7,8-TCDD/F. It offered wide linear ranges: 0.5–200 ng/mL for PeCDD/Fs and 0.2–2000 ng/mL for dl-PCBs. Analytical performance was comparable to HRGC/HRMS and met EU regulatory standards.. Franchina et al. [14] evaluated a novel GC-QQQMS/MS system with a PTV injector and advanced collision cell for analyzing regulated PCDD/Fs and PCBs in food and feed, demonstrating high precision (1.9–15% RSD), accuracy (>80%), and compliance with EU regulations, with results comparable to traditional GC-HRMS in both congener-specific and TEQ measurements. GC-MS/MS also presents advantages in terms of shorter run times, lower maintenance costs, and broader applicability in routine laboratories. Nonetheless, questions remain regarding the comparability of GC-MS/MS with HRGC/HRMS, particularly in terms of accuracy, toxic equivalency (TEQ) values, and overall reliability for regulatory purposes. This study aims to evaluate the feasibility and reliability of GC-MS/MS for the determination of dl-PCBs in pig feed, using HRGC/HRMS as a reference method. By comparing analytical performance, sensitivity, and toxic equivalency (TEQ) values between the two techniques, we assess the suitability of GC-MS/MS for routine monitoring and regulatory compliance in feed safety programs.

2. Experiments

2.1. Chemicals

All solvents and chemicals require analytical purity from Sigma-Aldrich. Concentrated sulfuric acid (H_2SO_4), granulated sodium sulfate (Na_2SO_4), granulated potassium hydroxide (KOH), sodium chloride (NaCl), neutral aluminum oxide (Al_2O_3), and AX Cambridge Isotope Laboratory (CIL-USA) provided the $^{12}C12$ standard solutions (for method accuracy and repeatability), $^{13}C12$ isotope-labeled standard solutions (companion standards for recovery efficiency), and dl-PCB calibration curve standard solutions.

2.2. Sample preparation and processing

The three pig feed samples (P1, P2, P3) differ in color (Figure 1), with P3 having a slightly darker tone. These visual differences suggest variations in formulation or processing methods. 20 g of pig feed, spiked with standard (EC-4937 and EC-5396), were Soxhlet extracted with toluene for 5 h, followed by a second extraction with a toluene:ethanol (9:1) mixture for 16 h Combined extracts were concentrated using a rotary evaporator and exchanged with 80 mL n-hexane. The extract was washed sequentially with 98% H_2SO_4, 5% NaCl, and 20% KOH, then dehydrated using Na_2SO_4. Clean-up was performed using a multilayer silica gel column and a custom activated carbon column, from which dl-PCBs were eluted with toluene at 118 ºC. After toluene evaporation, 30 mL of n-hexane was added and the solution was passed through an Al_2O_3 column. dl-PCBs were collected using a dichloromethane:n-hexane (95:5) eluent. Solvent was evaporated before GC-MS/MS analysis. Isotope-labeled standards were co-evaporated with N_2 to assess recovery, and the final 20 µL of sample was transferred into a vial for analysis.

Figure 1. *Image of 3 pig feed samples (P1, P2, P3)*

2.3. Examination of specimens utilizing GC-MS/MS and comparision with HRGC/HRMS

Gas chromatography utilized a TR-DIOXIN capillary column (60 m × 0.25 mm × 0.25 µm; ThermoFisher Scientific) with splitless injection (1 µL). The oven programme started at 150 °C (held 2 min), climbed to 220 °C at 20 °C/min (held 16 min), and reached 300 °C at 5 °C/min (held 1.5 min) for 39 minutes. Carriers were 1.2 mL/min helium. Interface and ion source temperatures were 290 and 280 °C. A 250 µA emission current was used for electron ionization (EI) at 70 eV. The system targeted 12 harmful dl-PCB congeners with precursor/product ion transitions. A Thermo TSQ 8000 Evo triple quadrupole GC-MS/MS instrument examined the extracted extract.

A 6-point calibration curve (CS1-CS6, EC-5396) was created to determine the linear range, needing $R^2 > 0.9995$ and RSD < 15%. Five blank and five spiked pig feed samples with 12C-dl-PCBs at CS1-equivalent levels were used to investigate detection and quantification limitations. The LOQ sample was created by adding 20 µL of EC-4935 (0.2 ng/mL) for 4 pg/sample dl-PCBs. The mean value C, standard deviation SD and relative standard deviation RSD are calculated according to the following formulas:

$$\bar{C} = \frac{\sum_{i=0}^{n} C_i}{n} \quad (1)$$

$$SD = \sqrt{\frac{\sum(C_i - \bar{C})^2}{n-1}} \quad (2)$$

$$RSD = \frac{SD}{\bar{C}} \times 100\% \quad (3)$$

$$LOD = 3.747 \times SD \quad (4)$$

$$LOQ = 10 \times SD \quad (5)$$

Where: n: Number of tests, C_i: Measured value at the i-th test, $\bar{C}$: Average value of the tests

Repeatability and reproducibility were assessed using five pig feed samples spiked with ^{12}C-dl-PCBs at CS3-equivalent levels. The Mid sample was prepared by adding 200 µL of EC-4935 (0.2 ng/mL), resulting in 40 pg/sample dl-PCBs. Recovery efficiency is determined by checking the content of the companion standard calculated according to the content of Recovery standard added last before analysis on the device according to the following formula:

$$H = \frac{m}{m_o} \times 100\% \quad (6)$$

Where: H: Recovery efficiency (%), m: Concentration of ^{13}C standard in the actual sample measured, m_o: Concentration of ^{13}C standard in the input standard.

Concentrations of dl-PCBs in three pig feed samples were determined by both GC-MS/MS and high-resolution HRGC/HRMS. Response Factors (RF), Relative percent difference (RPD) and result bias were used to compare the performance and consistency of the two analytical methods.

3. Results and discussion

3.1. Linearity range of the calibration curve, limits of detection and quantification

The linearity range of the dl-PCB standard curve was determined by constructing a 6-point standard curve of the EC-5396 standard curve from CS1 to CS6 by the GC-MS/MS method. The results of the analysis of the dl-PCB standard points are presented in Table 1.

Table 1. *Linear range of the standard curve*

No	Analytical compounds	C, pg/µL	R^2	RF	RSD, %
1	3,4,4',5-TeCB (81)	0.2-2000	0.999993	1.17	2.91
2	3,3',4,4'-TeCB (77)	0.2-2000	0.999987	1.14	2.72
3	2',3,4,4',5-PeCB (123)	0.2-2000	0.999907	1.10	4.58
4	2,3',4,4',5-PeCB (118)	0.2-2000	0.999857	1.12	3.55
5	2,3,4,4',5-PeCB (114)	0.2-2000	0.999903	1.07	6.25
6	2,3,3',4,4'-PeCB (105)	0.2-2000	0.999036	1.15	3.70
7	3,3',4,4',5-PeCB (126)	0.2-2000	0.999521	1.04	2.30
8	2,3',4,4',5,5'-HxCB (167)	0.2-2000	0.999879	1.12	4.97
9	2,3,3',4,4',5-HxCB (156)	0.2-2000	0.999905	1.13	4.06
10	2,3,3',4,4',5'-HxCB (157)	0.2-2000	0.999998	1.14	3.13
11	3,3',4,4',5,5'-HxCB (169)	0.2-2000	0.999986	1.14	2.61
12	2,3,3',4,4',5,5'-HpCB (189)	0.2-2000	0.999871	1.05	1.49

The data in Table 1 indicate that the correlation coefficient R^2 exceeds 0.999, and the relative standard deviation is below 15.0%. The results obtained align with the evaluation criteria (R^2 > 0.99 and RSD < 15%). The linear range of the dl-PCBs standard curve is 0.2 to 2000 pg/µL.

The detection limit and quantification limit for each dl-PCB congener in the pig feed sample matrix were established based on the standard deviation (SD) of the analytical data at the limit of quantification (LOQ) point. The analytical results indicated that the LOQ was established at the lowest point on the standard curve, as all requisite criteria were satisfied at this LOQ point. All analytes exhibited a signal-to-noise ratio exceeding 3; bias was less than 15%; recovery ranged from 80% to 102%; and relative standard deviation was below 10%. Calculate the Limit of

Detection (LOD) for each analyte in the sample matrix using the standard deviation (SD) at the LOQ survey point, following the formula: LOD = 3.747 x SD (p-value n = 5 with Degrees of freedom a = 0.01, hence Student's coefficient t = 3.747) [17]. Table 2 presents the findings of the determination of LOD and LOQ.

Table 2. *Limit of detection (LOD) of dl-PCB in pig feed matrix and limit of quantification (LOQ)*

No	Analytical compounds	LOD, pg/g	LOQ, pg/g
1	3,4,4',5-TeCB (81)	0.049	0.131
2	3,3',4,4'-TeCB (77)	0.028	0.075
3	2',3,4,4',5-PeCB (123)	0.045	0.120
4	2,3',4,4',5-PeCB (118)	0.041	0.109
5	2,3,4,4',5-PeCB (114)	0.036	0.096
6	2,3,3',4,4'-PeCB (105)	0.021	0.056
7	3,3',4,4',5-PeCB (126)	0.048	0.128
8	2,3',4,4',5,5'-HxCB (167)	0.034	0.091
9	2,3,3',4,4',5-HxCB (156)	0.049	0.131
10	2,3,3',4,4',5'-HxCB (157)	0.022	0.059
11	3,3',4,4',5,5'-HxCB (169)	0.050	0.133
12	2,3,3',4,4',5,5'-HpCB (189)	0.023	0.061
	Total TEQ dl-PCB, pg/g	**0.006**	**0.026**

The findings presented in Table 2 indicate that the LOD values of the hazardous congeners of dl-PCB fall within the range of 0.03 to 0.05 pg/g. All of the samples that were used for the determination of the LOD had a total TEQ that was lower than 0.065 pg WHO2005-TEQ/g. The total TEQ at the LOQ point is 0.026 pg WHO2005-TEQ/g, which is a significant amount lower than the maximum limit in animal feed, which is 0.75 pg WHO2005-TEQ/g. The findings indicate that the GC/MS-MS method is appropriate for the analytical criteria in accordance with the legislation that are in place within the jurisdiction of the European Union [2].

3.2. Repeatability, reproducibility and recovery efficiency

Determination of repeatability and reproducibility of each toxic congener of dl-PCB in pig feed samples was determined through the standard deviation SD of the Mid sample analysis results. Repeatability, reproducibility, and recovery are presented in Table 3.

Table 3. *Repeatability, reproducibility, and recovery of 13C-dl-PCB companion standard*

No	Analytical compounds	Repeatability			Reproducibility	Recovery (n = 15)	
		SD	RSD	B, %	H, %	Average, %	RSD
1	3,4,4',5-TeCB (81)	0.97	2.35	3.29	103.3	94.1 ± 5.39	5.7
2	3,3',4,4'-TeCB (77)	1.40	3.44	1.33	101.3	89.3 ± 4.96	5.6
3	2',3,4,4',5-PeCB (123)	0.79	1.99	-0.32	99.7	104.1 ± 4.03	3.9
4	2,3',4,4',5-PeCB (118)	1.73	4.48	-3.44	96.6	101.5 ± 4.69	4.6
5	2,3,4,4',5-PeCB (114)	2.39	5.58	7.14	107.1	97.9 ± 3.85	3.9
6	2,3,3',4,4'-PeCB (105)	1.68	4.42	-4.94	95.1	102.3 ± 5.23	5.1
7	3,3',4,4',5-PeCB (126)	2.32	5.70	1.94	101.9	93.4 ± 5.78	6.2
8	2,3',4,4',5,5'-HxCB (167)	1.56	3.90	0.10	100.1	99.4 ± 6.79	6.8
9	2,3,3',4,4',5-HxCB (156)	1.00	2.52	-0.69	99.3	99.6 ± 7.65	7.7
10	2,3,3',4,4',5'-HxCB (157)	1.66	4.32	-4.30	95.7	102.1 ± 5.68	5.6
11	3,3',4,4',5,5'-HxCB (169)	0.77	1.96	-1.88	98.1	97.4 ± 6.97	7.2
12	2,3,3',4,4',5,5'-HpCB (189)	1.10	2.71	1.51	101.5	102.7 ± 5.75	5.6

In accordance with the European Union's standards, the findings indicated that the amount of bias (b%) was less than 15%, the recovery (H%) of the analyte was within the range of 85.5-107%, the relative standard deviation (% RSD) was less than 10%, and the repeatability percentage (% RSD) was less than 10%. According to the recovery efficiencies (H%) of the

labeled companion standards for each sample matrix from all 15 samples (5 blank samples, 5 LOQ samples, and 5 mid samples) (Table 3), the recovery efficiencies of the companion standards ranged from 89 to 103%, which is in accordance with the requirements of US EPA Method 1668C. In summary, The GC-MS/MS method's repeatability, reproducibility, and recovery efficiency ensure accurate quantification of dl-PCB standards in pig feed matrice.

3.3. dl-PCBs analytical in pig feed and comparision with HRGC/HRMS

The levels of dl-PCBs in three pig feed samples were quantified using GC-MS/MS and high-resolution gas chromatography-mass spectrometry (HRGC/HRMS). The analytical findings indicated that certain congeners of dl-PCBs were identified using HRGC/HRMS, but not by GC-MS/MS. The analytical results (RPD, %) for the majority of harmful congeners of dl-PCBs from both devices did not surpass 20%, demonstrating that the GC-MS/MS system effectively analyzed dl-PCBs in the pig feed sample matrix. Most of these diet samples included toxic dl-PCB congeners except PCB#169. Almost all dangerous dl-PCB congeners were found in P1 pig feed, except PCB#81. Most harmful dl-PCB congeners were found in P2 and P3 pig feed samples, except PCB#157. The total toxicity equivalent (TEQ) of dl-PCB in all three pig feed samples ranged from 0.019 to 0.039 pg TEQ/g. The higher content of substances in sample P3 compared to P1 and P2 may be due to differences in raw material origin, manufacturing process or storage, leading to higher accumulation of dl-PCBs. In addition, P3 may contain components from industrial grease or contaminated additives.

Table 4. *dl-PCBs concentrations in pig feed samples (ND - not detected)*

No	Analytical compounds	P1			P2			P3		
		MSMS	HRMS	RPD, %	MSMS	HRMS	RPD, %	MSMS	HRMS	RPD, %
1	PCB#81	ND	ND	ND	5.793	5.67	2.1	0.613	0.634	3.4
2	PCB#77	1.093	1.261	14.2	2.400	2.218	7.9	3.645	3.08	16.8
3	PCB#123	0.084	0.083	0.6	0.345	0.313	9.8	0.174	0.144	18.6
4	PCB#118	3.862	3.308	15.4	10.490	9.010	15.2	15.863	16.932	6.5
5	PCB#114	0.104	0.109	4.7	0.454	0.410	10.2	0.638	0.656	2.8
6	PCB#105	1.685	1.809	7.1	3.924	3.949	0.6	5.019	5.279	5.1
7	PCB#126	0.180	0.181	0.7	0.162	0.185	13.0	0.367	0.393	6.8
8	PCB#167	0.128	0.152	17.0	0.222	0.253	13.2	0.935	0.988	5.5
9	PCB#156	0.361	0.342	5.5	0.277	0.288	4.0	0.595	0.542	9.4
10	PCB#157	0.090	0.096	6.2	ND	ND	ND	ND	ND	ND
11	PCB#169	ND	ND	ND	ND	ND	ND	ND	ND	ND
12	PCB#189	1.240	1.220	1.6	0.233	0.197	16.6	ND	ND	ND
	Total TEQ dl-PCB, pg/g	**0.019**	**0.018**	**5.4**	**0.019**	**0.021**	**10.0**	**0.039**	**0.041**	**5.0**

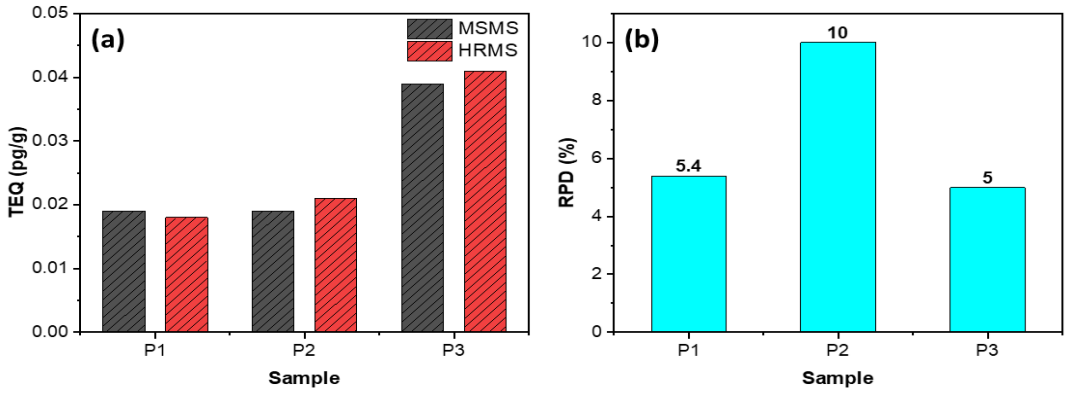

Figure 2. *Comparison of analytical results to determine TEQ dl-PCB concentration in pig feed samples by GC-MS/MS and HRGC-HRMS methods*

The comparison of analytical results from GC-MS/MS (MSMS) and HRGC-HRMS (HRMS)

methods for determining TEQ dl-PCB concentrations in pig feed samples (P1–P3) demonstrates strong agreement, as shown in both the bar graphs in Figure 2. TEQ values across all samples were closely matched between methods, with differences ranging from 0.001 to 0.002 pg/g. The relative percent differences (RPDs) were all ≤ 10%, with the highest in P2 (10.0%) and the lowest in P3 (5.0%), indicating acceptable analytical precision. These findings suggest that GC-MS/MS provides comparable accuracy to HRGC-HRMS in quantifying dl-PCBs and can serve as a reliable, cost-effective alternative for routine monitoring in feed safety assessments.

4. Conclusion

This study illustrates that GC-MS/MS is a dependable and effective analytical approach for quantifying dl-PCB contents in pig feed, yielding results comparable to the traditional HRGC-HRMS technique. The findings validate that the approach offers an extensive linear dynamic range with superior detection capabilities, rendering it robust and appropriate for regulatory monitoring of dl-PCBs in intricate feed matrices, such as pig feed. The GC-MS/MS method's repeatability, reproducibility, and recovery efficiency ensure the accurate quantification of dl-PCB standards in pig feed matrices. The TEQ values derived from both methods exhibited significant concordance across all evaluated samples, with relative percent deviations generally under 10%. These findings validate the feasibility of GC-MS/MS as an economical substitute for the routine surveillance of dioxin-like PCBs in animal feed, providing adequate accuracy and precision for regulatory adherence and food safety assurance.

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