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Determination of Chlorpropham (CIPC) residues, in the concrete flooring of potato stores, using quantitative (HPLC UV/VIS) and qualitative (GCMS) methods.

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Highlights

CIPC is lost to the concrete fabric of potato stores during application.

HPLC UV/VIS method developed and validated to detect CIPC in concrete.

CIPC presence in concrete confirmed by GCMS.

CIPC is persistent in the concrete flooring of potato stores at 4 cm depth.

Top 1 cm of flooring contains > 90 % CIPC.


Abstract

Isopropyl-N-(3-chlorophenyl) carbamate (CIPC, common name Chlorpropham) is commonly used for post-harvest sprout inhibition in stored potatoes. It is applied as a thermal fog which results in loss to the fabric of the store and the atmosphere. Recently, there have been concerns in the United Kingdom because of cross contamination of other crop commodities that were stored in buildings with a history of CIPC usage. This cross contamination may have occurred because of retained residues in the fabric of the stores. The retention of CIPC in concrete is poorly understood; therefore the requirement for a robust analytical method for the detection and quantification of CIPC in concrete is a critical first step in tackling this problem. A method using High-Performance Liquid Chromatography with ultraviolet detection (HPLC UV/VIS) was validated. CIPC recoveries at three concentration levels (0.4, 4.0 and 40.0 µg g\(^{-1}\)) were in the range of 90.7-97.0 % with relative standard deviations between 2.14-3.01 %. The limits of detection and quantification were 0.03 and 0.1 µg g\(^{-1}\), respectively. This study confirmed that CIPC was persistent in concrete to a depth of 4 cm, with > 90 % within the top 1 cm of the flooring.

Keywords

Chlorpropham, Concrete, Potato Stores, HPLC UV/VIS, GCMS.
1. Introduction

Potato (*Solanum tuberosum* L.), with an annual production of 375 million tons (Flis *et al.*, 2014), is globally the fourth largest staple crop after rice, wheat and maize (Wang *et al.*, 2011). The potato is an essential part of the diet for more than a billion people worldwide (Cicatelli *et al.*, 2014) and is a staple food in temperate regions of the world, while in other parts of the world it is generally used as a vegetable (Kibar, 2012).

China produces nearly 60 million tonnes, followed by India, Russia and the USA (Food Innovation Online Corp, 2017). The total production in the United Kingdom (UK) for the 2015 crop year was 5.49 million tonnes (AHDB-Potatoes). Potato tubers can remain suitable for consumption or processing through long periods of storage after harvest. They are considered one of the most important foods worldwide because of their long storage life which enables the potato processing industry to operate year-round in locations where potatoes can only be produced during a favourable growing season. After harvest, potato tubers are dormant for several weeks but continue to be metabolically active. However, as the tubers progress through the physiological aging process, from the dormant to the non-dormant phase, they become able to produce sprouts which have the potential to grow into a new plant (Daniels-Lake *et al.*, 2013).

Sprouting of stored potatoes results in weight loss and a decrease in nutritive value (Hajšslová and Davidek, 1986). Sprouting also increases glycoalkaloid production during storage (Friedman and Levin, 2016) which in turn causes a bitter taste (Maga and Fitzpatrick, 1980). Sprouting can be minimised, to maintain their long-term quality, by the use of chemical sprout inhibitors. The most common and effective in the potato industry is Isopropyl-N-(3-chlorophenyl) carbamate (CIPC, common name Chlorpropham, Figure 1) (Frazier and Olsen, 2014). CIPC is typically applied as a hot fog on harvested potatoes which causes deposition of solid residues on the potatoes (Gouseti *et al.*, 2015). Prior to the CIPC
Stewardship Group recommendations which implemented lower CIPC application rates (PICSG, 2016), the greatest total dose of CIPC that could be applied to potatoes in the UK was 63.75 g per tonne (McGowan et al., 2009). Recent research conducted using application rates of 12-14 g per tonne for potatoes for the fresh market (held at a storage temperature of 3-4°C), and 23-26 g per tonne for potatoes for processing (held at a storage temperature of 7-9°C), has shown that 23 - 25 % and 5 - 10% of input CIPC, respectively, was retained as a residue on tubers after storage (Briddon et al., 2014). Non-target fates of CIPC have been detailed by Smith et al. (2013) and include losses to the environment, including the atmosphere, soil, waterways and the fabric of the store.

The persistence of CIPC in soil and aquatic environments has been extensively studied. The half-lives of CIPC in soil at temperatures of 15 ºC and 29 ºC were 163 and 27 days, respectively, whereas in lake water, the half-life was 2208 days (Smith and Bucher, 2012). On the contrary, the persistence of CIPC in the fabric of stores is poorly understood and this has recently become of concern in the UK because of cross contamination of other crop commodities that were stored in buildings with a history of CIPC usage. This is particularly problematic as the presence of detectable amounts of CIPC in any commodity, except ware potatoes, renders it unfit for use in the EU. This also applies to manufactured food products, with the exception of potato products. For instance, cross contaminated wheat that was used in the production of baby rusks (Farley Brand Heinz) resulted in thousands of packets of the product being withdrawn from UK shops after they were found to be contaminated with CIPC (Curtis, 2006).

The problem of cross contamination of other crop commodities by CIPC can be attributed in part to the changing commercial pressures within agriculture and also advancement in instrumental analysis. Many of the medium to small size potato producers, in particular, are moving away from the potato sector and wish to utilise the vacated stores for other purposes.
This has always caused problems, even for those farmers remaining in the potato sector, who also grow cereals, due to the fact that seed crops of any type coming in contact with a CIPC contaminated store, even for a short period of time, may have impairment of germination and blanking. Chemical residues in crops are a serious trade barrier (Randhawa et al., 2014) and the utilisation of storage facilities previously treated with CIPC for any commodity is problematic in terms of cross contamination and may lead to an exceedance of the Maximum Residue Level (MRL). In the European Union, MRLs are established at the limit of quantification if a pesticide is not authorised for use on a specific crop (EFSA, 2011). In the UK, crops such as cereals, onions and oilseeds do not have approval for CIPC use and therefore they have an MRL at the limit of quantification (limit of determination) which is 0.01 mg kg\(^{-1}\) (EFSA, 2011; personal communication T Cowl, CRD-HSE 20 April 2017). As the detection limits for pesticides continue to improve due to advancements in instrumental analysis, crops can now be analysed and deemed to be contaminated at levels that in the past would have been below the limit of detection.

The residual quantities of CIPC lost to the fabric of the store are important, particularly if the store will be used for housing other crops. Over the years, various analytical methods have been employed to detect CIPC in soil (Clark and Wright 1970; Alsehli, 2014), water (Guzik, 1978; Park et al., 2009; Passananti et al., 2014; Alsehli, 2014), air (Boyd and Duncan, 1986a; Boyd and Duncan, 1986 b; Park, 2004), on stored potatoes (Camire et al., 1995; Khan et al., 2012; Mohammed 2012; Mohammed 2015) and in bacterial metabolism (Clark and Wright, 1970; Vega et al., 1984). However, studies on CIPC analysis in building materials are almost completely lacking. Boyd and Duncan (1986b) reported on the accumulation of CIPC residues in the concrete structures of a potato store and demonstrated concentrations in the concrete walls and flooring in the range of 130-290 mg kg\(^{-1}\) and 2050-9470 mg kg\(^{-1}\), respectively. However, this method involved time-consuming Soxhlet extraction followed by
a flame ionisation detection (GC-FID) method which is less sensitive for CIPC compared to other methods such as HPLC UV/VIS (Mohammed, 2014). Therefore, alternative, higher sensitivity analytical methods are required to detect CIPC in building materials, to allow informed recommendations to be made to farmers about the re-use of stores for other crop commodities.

The objectives of this present study were to develop a simple and robust method for detecting CIPC in concrete and to use this method to investigate the concentration of CIPC and the spatial and depth distributions in the concrete flooring of a contaminated store.

![Figure 1. The chemical structure of Isopropyl-N-(3-chlorophenyl) carbamate (CIPC, common name chlorpropham).](image)

**2. Materials and Methods**

**2.1 Material**

Isopropyl N-(3-chloro-phenyl) carbamate (CIPC, 98 % purity) was obtained from Sigma-Aldrich (Dorset, United Kingdom). HPLC-grade solvents (acetone and acetonitrile) were purchased from Fisher Scientific (Loughborough, United Kingdom). Ultrapure water was obtained using a Millipore Elix® 5 water purification system (Molsheim, France). HPLC grade vials with PTFE screw caps (Agilent technologies, USA), syringe filters (13 mm) with 0.2 µm PTFE membrane (VWR International, USA), luer lock syringes (3 ml) (HSW
NORM-JECT® Germany) and 20 ml glass vials (PerkinElmer, USA) were obtained from Crawford Scientific Ltd, UK.

A reconstituted stone slab of dimensions 100 × 200 × 50 mm (Bradstone UK), containing a mixture of stone and cement, was chosen to represent the concrete content of the store floor.

2.2 Preparation of CIPC stock solution

A stock solution of CIPC was prepared in acetonitrile at a concentration of 1000 µg mL⁻¹. Calibration standard solutions (0.01-1.0 µg mL⁻¹) and spiking solutions were prepared by diluting the 1000 µg mL⁻¹ stock with acetonitrile as required.

2.3 Sample preparation

2.3.1. Preparation of blank concrete matrix for method development.

Concrete blocks (Brandstone, UK) were chopped into approximately 25 g portions using a hammer and chisel. These were then crushed using a Retsch® Jaw Crusher (Haan, Germany), collected in a 125 µm Endecotts sieve (London, England) and shaken at 175 r.p.m for 30 minutes on a Retsch® Shaker (Haan, Germany). Sieved samples (≤125 µm), representing the blank concrete matrix, were stored in 20 ml screw cap glass vials at room temperature. Recovery experiments were performed using 5 g portions of blank concrete matrix, spiked with CIPC stock solutions of 100 µg mL⁻¹ and 1000 µg mL⁻¹ to give concentrations of 0.4, 4.0 and 40 µg g⁻¹ CIPC to concrete content. A 30 minute period was allowed for the CIPC to interact with the concrete matrix and for evaporation of solvent. Five replicates were prepared for each spiking level. Extracted concrete samples of 40 µg g⁻¹ CIPC to concrete content were diluted 1:10 with acetonitrile (100 %) prior to analysis.
2.3.2. Coring of concrete flooring in a potato store and preparation of industrial concrete cores for analysis.

The floor was cored using a Titan drill and a 52 mm diamond-tipped corer. A pilot drill bit (8 mm diameter) was used to enable the corer to grip the concrete surface. The pilot drill bit penetrated the concrete to a depth of 0.5 cm and was subsequently removed from the drill. This prevented contamination of the lower layers during the coring process. The coring was continued with the diamond tipped corer and extracted cores were wiped free of dust. A new pilot drill was used for each core and the corer was cleaned with methanol between samples. It was postulated that CIPC may penetrate the concrete to a depth of 3 cm, therefore, a 7 cm core was initially collected to assess the depth of penetration and the risk of contamination during the drilling and processing of samples. The intact 7 cm core was weighed prior to sectioning (using a Lapidary trim saw (Mukilteo, USA) into 1 cm layers, from the bottom to the top to prevent CIPC residues from transferring to the lower layers. The layers were crushed and prepared, as previously described, in ascending order from bottom to top. Two other cores were collected from the same store and the length of each core, and hence the number of layers from each, depended on the ease of penetration of the drill into the concrete. These cores were processed in the same manner as the 7 cm core. A section of clean concrete block, which was used in the method development, was routinely crushed and analysed in between contaminated industrial core samples to ensure the robustness of the processing method.

2.4. Extraction procedure.

After the 30 minute interaction period for recovery experiments, acetonitrile (20 mL) was added to the respective 5 g samples of spiked blank concrete matrix. The samples were
placed on an orbital shaker (IKA) set at 20°C for 30 minutes at 175 r.p.m and then left at room temperature overnight. The following morning, the samples were shaken for a further 15 minutes at 20°C and 175 r.p.m. A glass Pasteur pipette was used to transfer the supernatant from each container into 20 mL glass vials. For each sample, a 2 mL disposable syringe was used to remove approximately 1 mL of the supernatant which was filtered through a 0.2 µm PTFE membrane syringe filter into a 2 mL HPLC vial and the extract stored at 4 °C for analysis. Finally, 20 µL of the extract was analysed by high-performance liquid chromatography with UV detection (HPLC UV/VIS) and gas chromatography mass spectrometry (GCMS). The extraction procedure for the processed industrial concrete samples was the same as for the recovery experiments, with the exception of the weight of concrete extracted. One gram of crushed industrial concrete of particle size 125 µm, from each layer of a core, was separately extracted in 20 mL acetonitrile. Extracted industrial concrete samples were diluted to a concentration range of 0.01 to 1.0 µg mL$^{-1}$ prior to analysis.

2.5. Instrumentation and operation conditions.

2.5.1 HPLC UV/VIS

The analysis of the CIPC residues in concrete was carried out using a High-Performance Liquid Chromatography system (Shimadzu, Kyoto, Japan) with a Rheodyne® injector model 7725, an isocratic pump (LC-20 AD Prominence Liquid Chromatograph Shimadzu), a DGU-20 A$_3$ Prominence Degasser (Shimadzu) and a SPD-20 A Prominence UV/VIS Detector (Shimadzu). Data acquisition and processing were performed with LC Solution software release 3.40.

The chromatographic separation was performed at 25°C on a Genesis analytical column (250 mm × 10 mm i.d. 4 µm). The mobile phase was acetonitrile and ultrapure water in a ratio
60:40 (v/v) which was delivered at a flow rate of 1.5 mL min\(^{-1}\). The UV chromatographs were recorded at 210 nm. The identification of CIPC in concrete samples was achieved by comparing the retention times with those of standard CIPC solutions. Residual CIPC was flushed from the injector between analyses using 3 mL of acetonitrile. The column was washed with acetonitrile for 10 or 20 minutes between analyses, then an acetonitrile blank was analysed between analyses to ensure that there was no carryover.

2.5.2. GCMS

A Shimadzu GC MS-QP 2010 instrument was used for analysis. Separations were carried out using a ZB-5MS column (30 m x 0.25 mm i.d. 0.25 μm film thickness) with a stationary phase comprising 5% Phenyl-Arylene and 95% Dimethylpolysiloxane (Phenomenex®, UK). The temperatures of the injector and detector were set at 220 °C and 260 °C, respectively. The injection volume was 1 μL at a purge flow of 3 mL min\(^{-1}\) in splitless mode. Total run time for analysis was 19.20 minutes with an initial temperature of 80 °C and hold time of 0.5 minutes, followed by a four step temperature increase: i) + 30 °C min\(^{-1}\) to 125 °C for one minute, ii) + 25 °C min\(^{-1}\) to 180 °C for 3 minutes, iii) + 25 °C min\(^{-1}\) to 280 °C for 4 minutes, iv) + 20 °C min\(^{-1}\) to 300 °C for 2 minutes. The carrier gas was helium which was maintained at a constant pressure of 10.3 psi with a linear velocity of 38.1 cm sec\(^{-1}\) at 80.0 °C (oven temperature). Parameters for the MS were as follows: electron impact (EI) source temperature of 260 °C, interface temperature of 250 °C. Ion masses were scanned from 40 to 350 m/z at 3333 scans per second. Data acquisition and processing were performed with LabSolution software, GCMS Solution version 2.50 SU1. The identification of CIPC in concrete samples was achieved by comparing the mass spectral patterns with those of standard CIPC solutions and by using NIST/EPA/NIH mass spectral library (NIST 05) and NIST mass spectral search program version 2.0d.
3. Results and discussion

3.1. Method validation for HPLC UV/VIS

The only relevant HPLC UV/VIS method previously developed is for CIPC in potato extracts by Khan et al. (2008, unpublished) and discussed in detail in Mohammed (2012). The method developed here is for concrete and is validated for accuracy, precision and linearity. Prior to the development of the method for concrete, the instrument response for CIPC was assessed and the method was validated using CIPC standard solutions. The precision of the standard solutions was determined using 10 replicate injections of 1 µg mL$^{-1}$ CIPC solution with high precision obtained for the standard solutions (Relative Standard Deviation (RSD) % = 1.59). Linearity was assessed (in triplicate) using standard calibration curves that were constructed by plotting the signal intensity versus the concentration of CIPC in the standard solutions. Excellent linearity was obtained in the concentration range from 0.01 to 1.0 µg mL$^{-1}$, with correlation coefficients ($R^2$) greater than 0.99. Extracted industrial concrete samples were diluted to fit this concentration range prior to analysis (Figure 2). Sensitivity was evaluated by estimating the limit of quantification (LOQ) and limit of detection (LOD) using a repeat injection method (n=10) (Mohammed et al., 2014). The LOD and LOQ values, with respect to the instrument response to CIPC, were evaluated using 0.01 µg mL$^{-1}$ CIPC solutions. The LOD and LOQ values of 0.001 µg mL$^{-1}$ and 0.004 µg mL$^{-1}$ were equal to 3 and 10 times the standard deviation (SD) of the 0.01 µg mL$^{-1}$ CIPC solution, respectively. The LOD and LOQ values, with respect to the extraction procedure, were evaluated using spiked (nominal level 0.04 µg g$^{-1}$; n=10) and non-spiked (n=10) concrete samples. The SD was calculated using a repeat injection method for both spiked (n=10) and non-spiked (n=10) samples, as depicted in the following equation: $SD = \sqrt{[(SD_s)^2 + (SD_b)^2]}$ where spiked and non-spiked concrete are
designated SD$_s$ and SD$_b$, respectively. The LOD and LOQ values of 0.03 µg g$^{-1}$ and 0.1 µg g$^{-1}$ were equal to 3 and 10 times the SD of the spiked and non-spiked concrete matrix extracts respectively (Mohammed, 2012). The accuracy and precision of the method with respect to concrete was determined by recovery tests (n=5) conducted at three concentration levels, using the blank concrete matrix spiked at concentration levels of 0.4, 4.0 and 40 µg g$^{-1}$. The extraction procedure was highly efficient with recoveries greater than 90%. The precision was assessed by calculating the %RSD of the five determinants per concentration (Table 1).

Table 1. Quantitative determination of CIPC in spiked reconstituted concrete (n=5) using HPLC UV/VIS at 210 nm.

<table>
<thead>
<tr>
<th>Nominal concentration in extract (µg g$^{-1}$)</th>
<th>Mean concentration recovered from concrete (µg g$^{-1}$)</th>
<th>Recovery % (± % RSD)</th>
<th>LOD (µg g$^{-1}$)</th>
<th>LOQ (µg g$^{-1}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.4</td>
<td>0.388</td>
<td>97.0 ± 3.01</td>
<td>0.03</td>
<td>0.1</td>
</tr>
<tr>
<td>4.0</td>
<td>3.747</td>
<td>93.7 ± 2.43</td>
<td></td>
<td></td>
</tr>
<tr>
<td>40.0</td>
<td>36.275</td>
<td>90.7 ± 2.14</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

The HPLC UV/VIS method was developed using an isocratic system with manual injection. Although reproducible data were generated using this system, there were limitations in terms of the time required for manual washing of the injector and column which became more apparent when ‘real’ concrete samples were analysed. For the purpose of high throughput...
analysis, the following recommendations are made. A binary system with automated injection would allow a programme with a gradient step and an injector rinse step to be included. This would remove residual CIPC from the column and injector respectively, thus ensuring no carry over between analyses. The concrete contents of the blocks used in the method development can be considered as modern concrete. The method was initially used to assess the CIPC concentrations in a research potato store and was efficient in terms of CIPC quantification. The concrete content and structural integrity of concrete in other commercial stores may be different from the concrete contents of the blocks used in the method development therefore, further assessment and development may be required.
Figure 2. HPLC UV/VIS chromatograms at retention time 7.2 minutes for: (A) A CIPC standard solution of 1.0 µg mL$^{-1}$ and (B) an industrial concrete sample contaminated with CIPC.

3.2 Depth distribution of CIPC into the concrete flooring of a research potato store.

The depth of CIPC distribution into the concrete flooring of the research potato store was determined by sectioning concrete cores, obtained from designated positions in the floor of the store, into one centimetre depth increments. Each increment from the respective cores was processed, extracted and the resulting industrial concrete samples analysed by HPLC UV/VIS. The depth of CIPC distribution was expressed as µg of CIPC per gram of concrete in each layer. The majority of CIPC (between 90 and 100 %) was found in the top centimetre layer of each core, with a decrease in concentration in the subsequent layers. CIPC penetrated the concrete flooring of the store to a depth between 3 and 4 cm. The concentrations in layers 1-7 of the 7 cm core were 23, 1.5, 0.31, 0.22, 0.0, 0.0 and 0.0 µg g$^{-1}$, respectively. The absence of CIPC in the last three layers demonstrates that there was no contamination during the drilling process. The levels in the second core were 266 and 1.2 µg g$^{-1}$ for the top and second layer respectively; whereas for the third core, the values were 179, 0.53 and 0.26 µg g$^{-1}$ for the top, second and third layer, respectively.
3.3 Qualitative analysis of CIPC in spiked and industrial concrete using GCMS.

A GCMS method to confirm the presence of CIPC in industrial concrete samples and the spiked blank concrete matrix was also developed. Both spiked and industrial concrete samples gave mass spectral patterns which were consistent with the expected spectrum obtained from the NIST database:

(http://webbook.nist.gov/cgi/cbook.cgi?Name=chlorpropham&Units=SI&cMS=on#Mass-Spec)

(213/215 m/z: parent ion; 153/154 m/z: m-chlorophenyl isocyanate; 171/173 m/z: free acid formed from isopropyl residue; 127/129 m/z: chloraniline) (Figure 3).

3.4 The risks of cross contamination of crops stored in the vicinity of contaminated concrete flooring.

In this study, a research store with 23 years of CIPC applications was assessed for CIPC presence and levels in the concrete flooring. This store was routinely steam cleaned after the applications, however, the levels detected in the cores demonstrate that CIPC is persistent, even after extensive cleaning. This highlights the possible risks of cross contamination of crops in this research store and potentially, even higher risks in commercial stores which do not employ cleaning strategies. The issue of cross contamination is currently of concern in the UK, and crop assurance schemes such as Red Tractor recommend risk assessments to ensure that crops can be safely stored without becoming cross contaminated. A preliminary risk assessment method is proposed here, employing the data derived for 2 cores using the methods outlined above. These data are ideal for determining the concentrations and presence of CIPC in concrete and will provide useful input data for developing calculations involving risk assessments. For example, using the total CIPC concentration in the 7-cm deep core (25.03 µg g⁻¹), a risk assessment can be calculated using the density of concrete (2,400 kg m⁻³).
or 2.4 g cm$^{-3}$). The amount of CIPC present in 1 m$^2$ of the concrete flooring will be 601.4 mg m$^{-2}$ [0.07 x 2400 x average CIPC concentration (25.03 / 7) mg]. Assuming that wheat is stored to a depth of 5 m, that the average density of wheat is 720 kg m$^{-3}$ and that an estimated 20% of the CIPC migrates into the wheat in any one season, then the average concentration in the wheat would be 0.03 mg kg$^{-1}$. The permissible limit of CIPC in crops other than potatoes is 0.01 mg kg$^{-1}$, therefore we can conclude that the average concentration in the wheat is a factor of x3 higher than the limit, however, the 20% loss per season from concrete is likely to be a significant overestimation, given that we have detected CIPC up to a depth of 4 cm in a store where 25 years had elapsed since the last application (Douglas et al., unpublished).

Similarly, if we take the 2-cm depth core, which has a total of 267.2 µg g$^{-1}$, and using the same parameters as above, this would equate to 0.36 mg kg$^{-1}$ in the wheat, 36x the limit.

The ease of volatilization of a chemical is related to its vapour pressure and the rate of movement from the volatilizing surface (Smith and Bucher, 2012), therefore, it may be feasible for CIPC to volatilize from the concrete surface into the headspace of the store. Tomlin (2003) quotes a vapour pressure value for CIPC of 24 mP at 20 °C while Taylor and Spencer (1990) quote 1.3 mP at 25 °C. However, there is no information pertaining to the volatilization of CIPC from concrete. Current research is being conducted to investigate the rate of volatilization from a concrete surface and the route of cross contamination of crop commodities in stores with a history of CIPC usage. This will provide invaluable information for improved risk assessments and decontamination strategies, including the application of sealants to the concrete flooring to prevent CIPC volatilization.
Figure 3. GCMS chromatograms and mass spectra obtained at a retention time of 9.1 minutes for a CIPC standard solution of 10.0 µg mL⁻¹ (A & B), a spiked concrete sample at 40 µg g⁻¹ CIPC to concrete content (C & D) and an industrial concrete sample contaminated with CIPC (E & F).
4. Conclusion.

A simple, reproducible and more sensitive analytical method for determining residues of chlorpropham (CIPC) in concrete, using HPLC UV/VIS, was developed and validated. Acceptable recoveries of \(\geq 90\%\) were obtained with high precision (\% RSD 2.14-3.01). Triplicate analyses from a representative industrial concrete sample suggested that the accuracy, precision and selectivity of the proposed method were satisfactory for CIPC detection in the concrete of commercial potato stores. The limits of detection and quantification were 0.03 and 0.10 µg g\(^{-1}\), respectively, allowing application of the method for very low residue levels. The presence of CIPC in the concrete flooring was confirmed by GCMS. An assessment of CIPC distribution in the flooring showed that it can persist to a depth of 4 cm. This highlights the risk of possible cross contamination of other crop commodities stored in potato stores with a history of CIPC usage. A preliminary risk assessment calculation, using an actual CIPC level in the concrete flooring of a research store (266 µg g\(^{-1}\) in the top 1-cm), suggests the potential risk of cross contamination of grain (0.36 mg kg\(^{-1}\)), exceeding the permissible limit.

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