Anal Bioanal Chem (2014) 406:1073–1080
DOI 10.1007/s00216-013-7160-2

RESEARCH PAPER

Validation of chlordecone analysis for native and remediated French West Indies soils with high organic matter content

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Received: 29 March 2013 / Revised: 13 June 2013 / Accepted: 19 June 2013 / Published online: 9 July 2013

Abstract An analytic method was developed and validated for the analysis of chlordecone in the three main types of French West Indies soils: Ferralsol, Andosol and Nitisol with and without the addition of Daramend® and compost amendment used in a remediation process. The method consists in analysis by gas chromatography coupled with triple quadrupole mass spectrometry after pressurised liquid extraction. The high natural content of organic matter in the soils coupled with the additional exogenous organic matter from the amendments tested lead to a complex extract. Transnonachlor was used as surrogate to correct the results for extraction efficiency, and ^{13}C chlordecone was added as internal standard to mimic as closely as possible the behaviour of chlordecone and suppress possible side effects during its analysis. The key parameters of the method (linearity, repeatability, interday precision, specificity, extraction efficiency and limit of quantification) were validated in accordance with the NF T 90-210 standard method. The limit of quantification is 0.03 mg/kg. Uncertainty (k=2) was 40 % for concentrations lower than or equal to 1 mg/kg, and 30 % for concentrations greater than 1 mg/kg.

Keywords Soil · Accuracy profile · Chlordecone · Pressurised liquid extraction · Gas chromatography · Mass spectrometry

Published in the special issue *Analytical Science in France* with guest editors Christian Rolando and Philippe Garrigues.

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Introduction

An organochlorine insecticide ($C_{10}Cl_{10}O$) of the bishomocubane family, chlordecone (CLD), was used in the French West Indies (FWI) between 1972 and 1993 to protect banana plantation against banana weevil. Over the last 10 years, environmental monitoring networks have highlighted high and frequent contamination of waters, sediments and fish fauna [1]. The intrinsic characteristics of CLD (very high persistence and sorption on solids), the quantity of CLD employed (300 tonnes), the areas concerned (hundreds of square kilometre), the socio-economic impacts (limitation of consumption of locally produced food, bans on fishing) and health concerns [2, 3] have resulted in chlordecone in the FWI becoming a major environmental, social and economic issue.

The environmental problem posed by CLD in the FWI is worsened by the fact that the contaminated soils are of volcanic origin and may contain significant amounts of amorphous clay (allophane) which has particular exchange characteristics and complexes the organic matter [4].

Two processes for remediating the chlordecone-contaminated soils have been tested in vitro [5]. The first process requires the addition of compost, up to 30 % by dry mass. The second process, *Daramend*®, requires the addition, up to 12 %, of a mixture of zero valent iron and plant organic matter.

The difficulty for the quantification of chlordecone in soils comes from the difficulty in obtaining efficient extraction and an analytical method sufficiently robust to not be unduly affected by the complex matrix.

Only two articles have been dedicated to the extraction and measurement of CLD in French West Indies soils. The first article describes a method based on extraction by pressurised liquid extraction (PLE) followed by GC/MS analysis; the method was validated according to ISO criteria for a mixture of poorly characterised FWI soils [6]. The limit of quantification, 1 mg/kg, is however insufficient when

compared to the threshold (0.25 mg/kg) considered as being likely to lead to the contamination of cultivated plants [7]. Studies that use infrared spectrometry have given some interesting results but the analysis is based on statistical interpretation of spectral lines that requires preliminary calibration, and the process revealed poor correlation for high concentrations of CLD (>12 mg/kg; [8]). The other papers dedicated to the analysis of CLD generally concern developments made on calibration solutions [9–11].

The aim of the present study was to develop and validate an analytical method for the analysis of CLD in the three main types of FWI soils: Ferralsol (FRL), Andosol (AND) and Nitisol (NIT) [4]. The method takes into consideration the input of exogenous organic matter from the remediation processes [5]. Gas chromatography coupled with mass spectrometry (GC-MS/MS triple quadrupole) was used, following PLE with the use of ^{13}C chlordecone (CLD13) as internal standard with the objective of reducing the quantification limit.

Materials and methods

Chemicals

The standard chlordecone hydrate (CAS 143-50-0) is commercialised by CIL Cluzeau (Sainte-Foy-La-Grande, France) in solid form. The internal standard CB53 (CAS 41464-41-9) and the surrogate trans-nonachlor (CAS 39765-80-5) are available from Sigma-Aldrich (St. Quentin Fallavier, France). CB53 is obtained at 10 mg/L in isooctane and trans-nonachlor in a solid form. CLD13 at 100 mg/L in nonane is commercialised by LGC Standards (Molsheim, France). All of these standards have purity greater than 98 %. Analytic-grade organic solvents (acetone, cyclohexane and hexane) were obtained from Fisher Scientific (Illkirch, France).

Preparation of standard solutions

The individual standards were prepared from solid standards dissolved in acetone (200 mg/L) and stored at −18 °C. A 1 mg/L mixture was then prepared by dilution with cyclohexane. The calibration solutions were then prepared by successive dilutions in cyclohexane in order to obtain a range of concentrations from 15 to 250 μg/L. At the end, the final solvent of calibration solutions contained (*v*/*v*) 0.5 % isooctane from the internal standard solution CB53, 0.2 % nonane from the internal standard CLD13 and 0.02 % (15 μg/L) to 0.25 % (250 μg/L) acetone from CLD and trans-nonachlor solutions.

Instrumentation

The extraction procedure (PLE) was carried out using an ASE 350 system from Dionex S.A. Corporation (Voisins-le-Bretonneux, France) with 22 mL stainless steel vessels. GC/MSMS analysis was performed with a Bruker system (Marne la Vallée, France) composed of a GC450 gas chromatography apparatus equipped with an 1177 injector, a Combi Pal (CTC) autosampler and a 300MS triple quadrupole mass spectrometer.

The injector was equipped with a 4×6.3×78.5 mm liner with fibreglass and Sky™ deactivation. The compounds were separated on an Rxi-1MS (30 m, 0.25 mm ID, 0.25 μm) column from Restek (Lisses, France).

Soil samples

The three main types of FWI soil—AND, NIT and FRL—were collected from Guadeloupe and Martinique. The soil AND is characterised by a very high organic matter content, approximately four times greater than the other two soils, a pronounced acidic pH and a CEC that is 1.4 to 2.2 times that of the other two soils. The granulometric clay content of Andosol is markedly lower than that of FRL and NIT (Table 1).

For the study of the remediation processes, the mesocosms were subjected to (1) in situ chemical reduction (ISCR) with input of the Daramend® soil amendment (60–80 % of plant organic matter and 20–40 % of zero valent microscopic iron) up to 12 % by dry weight of the soil to be treated [12] or (2) input of compost to 30 % by weight (equivalent to 50 % by volume).

Procedure

Sample preparation

Before analysis, the soil was dried at 38 °C (±1 °C) for a period of 72 h. After drying, the residual humidity of the soil varied between 3 and 8 %, depending on the soil type. The sample was reduced by crushing followed by grinding to a particle size of less than 80 μm.

Table 1 Physico-chemical properties of the three French West Indies soils contaminated by chlordecone

Soil	Clay %	Silt %	Sand %	$CaCO_3$ %	Organic matter %	CEC (mEq/100 g)	pH water	pH KCl
Andosol	23.6	46.7	16.2	0.2	13.4	37.5	5.3	4.8
Ferralsol	59.1	30.1	7.0	<0.1	3.9	17.2	5.6	4.9
Nitisol	37.8	42.3	16.2	0.2	3.7	27.0	6.1	5.2

Extraction

Extraction was performed using PLE. This system enables extraction from 5 g of soil (spiked with 0.2 mL of a 10 mg/L solution of trans-nonachlor used as surrogate) by a 50/50 *v*/*v* mixture of acetone and hexane. Soils were mixed with Hydromatrix (to remove moisture) and introduced into a 22-mL cell with a piece of cellulose paper in the bottom. The temperature was 100 °C, with 3 cycles per sample, a 60 % flush volume, a static time of 5 min and a purge time of 120 s.

In this study, a surrogate was added before the extraction in order to evaluate the extraction process. Trans-nonachlor ($C_{10}H_5C_9$) was selected as it includes, as CLD, a carbon cycle and a high number of chlorine atoms, and was not present in the soils being studied. The trans-nonachlor recovery rate was used to correct CLD result. Previous studies have shown that it perfectly mimicked the behaviour of CLD.

Two internals standards were added before analysis. CB53 was used for the trans-nonachlor calibration and CLD13 for CLD calibration.

The extract was reduced to a volume of approximately 10 mL by means of a nitrogen stream. An aliquot of 1 mL was taken (fractionated to 1/10). The extract fractionated was reduced by nitrogen stream and completed with cyclohexane three times. The final extract was finalised with 1 ml of cyclohexane. Before GC/MSMS analysis, an input of the internal standards was performed with 0.1 mL of a 2 mg/L solution of CLD13 and 0.5 mg/L CB53 in order to quantify CLD and trans-nonachlor, respectively.

The final extract was composed (*v*/*v*) of 0.5 % isooctane and 0.2 % nonane from individual standards CB53 and CLD13, respectively. The equivalent amount of soil sample in these final extract is 0.5 g soil sample per mL.

Gas chromatography–mass spectrometry

The injection volume in the GC system was 1 μL at 100 μL/s. The temperature of the injector was maintained at 280 °C. The injector was operated in splitless mode at 1 mL/min in constant.

The syringe was rinsed before (three times in 5 μL of cyclohexane) and after the injection of the extract and the standards (five times in 5 μL of acetone and two times in 5 μL of cyclohexane). The column was heated to 50 °C for 1.1 min then to 250 °C with a gradient of 30 °C/min and finally to 310 °C at 10 °C/min and isothermally for 1.5 min. The transfer line was at 310 °C. The ionisation source, using positive electron impact, was at 250 °C. The collision gas pressure (Argon in CID) was 1.5 mTorr. The resolutions of Q1 and Q3 were fixed at 1.5 and 1.2, respectively.

The most intense fragment for CLD and CLD13 corresponds to the cyclopentadiene $^{12}C_5Cl_6$ [13] and $^{13}C_5Cl_6$. CLD and CLD13 show up as ions with the same mass (*m*/*z* from 275 to 283). The most abundant mass of CLD13 with less than 0.1 % interference with the masses of CLD is the mass *m*/*z* 281. The transition for the quantification of the CLD13 is therefore 281>246.

The retention times and detection parameters are shown in Table 2.

Validation procedure

Validation was undertaken in accordance with the ISO 17025 [14] standard method and, since there is no equivalent standard for soils, by applying the requirements of the NF T 90-210 [15] standard relative to methods of water analysis. This validation includes the characterisation of the calibration curve, the efficiency of extraction, the study of the specificity, the characterisation of the limit of quantification, the determination of the repeatability and interday precision and the estimation of measurement uncertainties.

Results and discussion

Influence of the solvent in the preparation of standard stock solutions

Huckins et al. [16] have demonstrated the effects of solvent on CLD (reactions to form a hemiacetal, reduction or increase in

Table 2 Detection parameters in GC/MSMS (electron impact)

Compound	RT (min)	MRM transitions	
		Quantification transition, *m*/*z* (% abundance, collision energy V)	Qualification transitions (% abundance, collision energy V)
Chlordecone	9.6	272>237 (100, 15)	270>235 (60, 15); 272>235 (20, 15)
Chlordecone C13	9.6	281>246 (33, 15)	279>244 (100, 15)
Trans-nonachlor	8.9	409>300 (100, 25)	407>298 (64, 25); 411>302 (62, 25)
CB53	7.9	292>222 (100, 25)	292>257 (100, 10)

the compound's signal depending on the solvents). We therefore studied the influence of varying the solvent used in the preparation of the stock solution. Three solvents (methanol, isooctane and acetone) of differing polarity were tested for the preparation of the stock solution (200 mg/L). Three calibrations (50 to 250 μg/L) were prepared from these solutions by dilution in cyclohexane. The results showed that CLD response is 2.2 times and 1.8 times greater with methanol and acetone, respectively, relative to isooctane. Therefore, the higher the polarity of the solvent used for the preparation of the stock solution, the better the response in GC/MS of the CLD, with methanol giving thus the best response.

The influence of the quantity of polar solvent (methanol) in the injected standards was also tested. The addition of 0.2 % of methanol in a standard containing only isooctane increased the signal of the CLD by a factor of 2.

This signal increase can be explained by the affinity of the compound with polar solvents despite its characteristic of being poorly soluble in water [17] (1–2 mg/L at 20 °C for acidic and neutral pH). CLD is likely to evolve into a more soluble form to form the chlordecone hydrate ($C_{10}H_2Cl_{10}(OH)_2$), resulting from a reaction of water with the ketone function of the CLD to form a "gem-diol" [18].

These tests showed the influence of the composition of the final solvent on the response of CLD during gas chromatography analysis and hence the influence of the preparation of the calibration solutions and the importance of the choice and nature of the solvent used in the stock solution. It is critical that the final solvents used for the samples and for the calibration solutions are identical in order to have coherent results.

Consequently, solvents used for the extraction were a hexane/acetone mixture, while cyclohexane was used as final solvent before injection into the GC/MS. Accordingly, acetone was used as the solvent for the stock solution and cyclohexane for the preparation of the calibration solutions.

Additional checks of the extraction method and mass spectrometry

As Andosol soil contains allophones which have particular exchange characteristics [19, 20]; the closure of the nanoporosity of this soil type by drying might result in a less efficient extraction of CLD. Although the drying used in the study was undertaken at only 38 °C, when the influence of drying on nanoporosity is likely to be most pronounced at temperatures higher than 100 °C (T. Woignier, personal communication to C. Mouvet, April 2011), the effect of the drying was nevertheless verified. A sample of raw Andosol (with a humidity of 60 %) was divided into two subsamples for an extraction after drying/grinding (particle size less than 80 μm, see "Sample preparation") and an extraction on the original, wet, not ground sample. This operation was performed in triplicate. The mean values in the wet soil, 14.5 mg/kg with a standard deviation of 0.2 mg/kg, is not significantly different from that of the dried soil, 12.5 mg/kg with a standard deviation of 0.3 mg/kg, as measurement uncertainty is 30 % ("Repeatability, interday precision and uncertainty of the

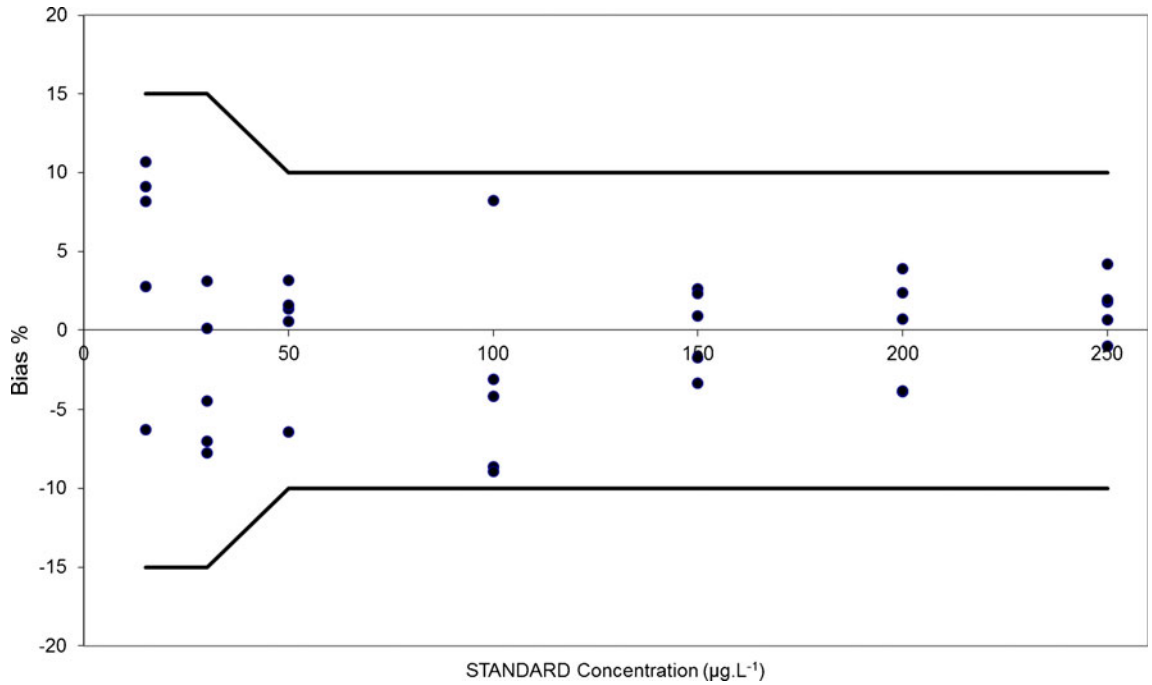

Fig 1 Observed bias (in percent) and maximum tolerated range allowed for the calibration range (15 to 250 μg/L)

Table 3 Method validation data: method efficiency, specificity, limit of quantification and repeatability/interday precision

Parameter tested	Soil type	Spiking of chlordecone (mg/kg)	Mean of concentrations measured (mg/kg) (nominal concentration subtracted)	Mean recovery (%)	Number of analyses	CV repeatability (%)	CV interday precision (%)
Efficiency of extraction	Nitisol	0.1	0.14	139	2	1	
		0.25	0.30	118	2	21	
		0.5	0.66	133	2	2	
		1	1.21	121	2	2	
		10	10.4	104	2	2	
	Ferralsol	0.1	0.11	109	2	2	
		0.25	0.24	96	2	1	
		0.5	0.68	135	2	1	
		1	1.1	113	2	13	
		10	11.1	111	2	1	
	Andosol	1	0.92	92	2	13	
		10	9.3	93	2	8	
Specificity (modified soil)	Nitisol[a]	0	1.3		5	16	
	Nitisol[b]	0	1.1		5	14	
	Ferralsol[a]	0	2.6		5	7	
	Ferralsol[b]	0	2.4		5	5	
	Andosol[a]	0	15.3		5	6	
	Andosol[b]	0	14.2		5	10	
Limit of quantification	Nitisol	0.03	0.022	73	4	4	13
	Ferralsol	0.03	0.023	75	4	9	11
	Andosol	0.03	0.024	81	4	8	8
Repeatability/interday precision	Nitisol	0	1.1		10	4	8
	Ferralsol	0	2.0		10	4	6
	Andosol	0	16.1		10	8	9

[a] Quantification by standard addition method

[b] Quantification by calibration curve

method" section). The extractions were therefore performed on soil dried at 38 °C for AND and the two other soils.

Validation of results

Calibration

Calibration was performed using seven concentrations from 15 to 250 μg/L (equivalent to 0.03 to 0.5 mg/kg in the soil, respectively). Each calibration was prepared five times from independent solutions adhering to conditions of intermediate precision (same person, different day).

Weighted linear regression in $1/x$ was chosen as the calibration model.

According to the NF EN T 90-210 requirement, bias (difference between the calculated concentration and theorical value) was calculated for each standard. The maximum observed bias was 11 % (observed for the lowest value) with a correlation coefficient greater than 0.996 (Fig. 1).

Efficiency of extraction

The efficiency of the extraction was evaluated by spiking non-contaminated NIT and AND soils (<0.03 mg/kg) and moderately contaminated FRL soil (0.23 mg/kg, no typical FRL free of CLD contamination was available). NIT and FRL were spiked at 0.1, 0.25, 0.5, 1 and 10 mg/kg in duplicates. AND was spiked only at 1 and 10 mg/kg in duplicates, as lower levels are not found in the soils studied. The results are shown in Table 3.

All of the biases were lower than 40 % for spiking levels between 0.1 and 0.5 mg/kg for the NIT and FRL soils, and within ±22 % for spiking levels between 1 and 10 mg/kg for most of the NIT, FRL and AND soils. These data will be taken

into account in the determination of the uncertainty of the method with 40 % for level between 0.03 and 1 mg/kg.

Specificity

The high natural content of organic matter in the soils, in particular Andosol (up to 13–15 %, Table 1), and the addition of exogenous organic matter result in a complex matrix that could induce matrix effects or affect the extraction efficiency.

In order to evaluate if the matrix effects linked to the modification of the soil by the ISCR remediation process are correctly taken into account with the use of CLD13, the concentrations of CLD obtained through quantification of the extract by calibration in the solvent were compared with those obtained using the standard additions method. This was done for three levels of concentration added to the extract for the three soils and four different additions of exogenous organic matter (6, 12 % of Daramend® and 5, 30 % of compost) and one control (total of experimental conditions, five for each soil). All of the soils were prepared at the same day. Dilution by the input of organic matter is taken into account in the results.

The results for the means of the five concentrations, measured in the soils by standard additions method and calibration curve with internal standard (Table 3), demonstrate that the use of CLD13 as internal standard is appropriate to take into account the matrix effects due to the addition of organic matter.

The results of CLD in four different additions of exogenous organic matter, obtained by calibration curve, were then compared to the control to verify the influence of the addition of organic matter (due to the remediation process) on the determination of CLD concentration in the three soils (Fig. 2). All of the concentrations in the three soils with added organic matter were equivalent to the concentration of the original soil (without addition) when the uncertainty of the method (cf. "Repeatability, interday precision and uncertainty of the method" section) was taken into account.

This specificity was also evaluated by comparing the results of the extraction efficiency of the three soils (cf. "Efficiency of extraction").

The method is thus applicable for all of the three soils with the input of organic matter coming from the Daramend® and compost.

Limit of quantification

The concentration of 0.03 mg/kg (equivalent to the first concentration level of the calibration) was tested as the limit of quantification by spiking the 3 natural soils (NIT, FRL and AND).

The limit of quantification (LOQ) of 0.03 mg/kg was evaluated within conditions of repeatability by doubling the extraction (two extractions from two samples of the same batch) for the three soils, and interday precision by performing this series of extractions on the three soils on six different dates.

The contents found after spiking at the limit of quantification are shown in Table 3.

The accuracy of the limit of quantification presupposed should be checked against the maximum tolerated range of 60 % of the limit of quantification (a value obtained by convention from the NF T 90-210).

The NF T 90-210 standard specifies also the execution of a test indicating if the observed deviation between the mean

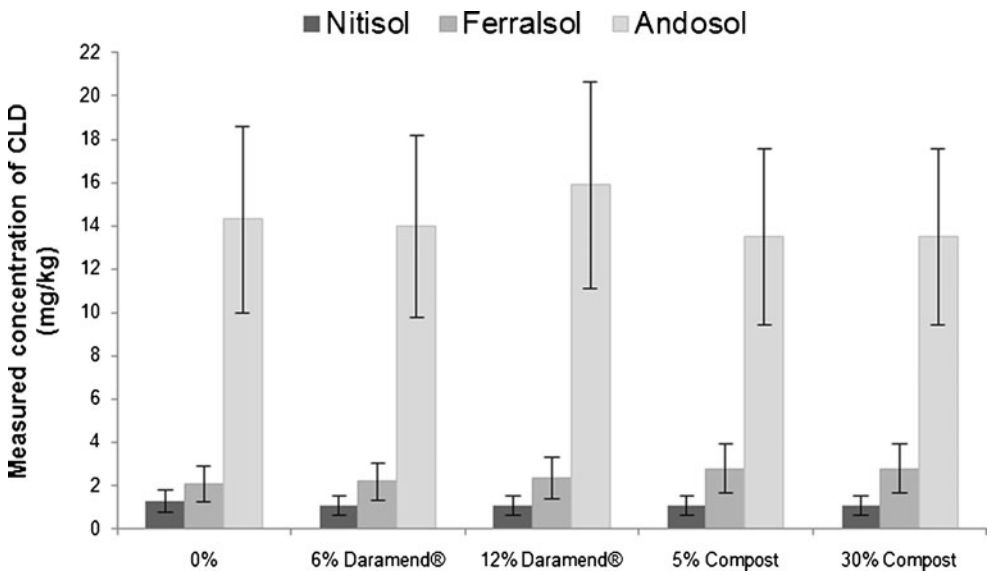

Fig 2 Concentration of CLD measured by calibration curve (with expanded uncertainly) in a soil with and without an input of exogenous organic matter in the soils (Daramend®:mixture of Iron(0) and organic matter; compost)

of the results and the spiking value is significant. It is therefore required to calculate a standardised difference (E_N) [14] in the following manner:

$$E_N = \frac{\mathrm{LOQ} - z\mathrm{LOQ}}{\sqrt{\left(\frac{U_{\mathrm{interday}}{}^2}{n} + U_{\mathrm{ref}}{}^2\right)}}$$

Where LOQ is the spiking value, zLOQ the mean of the measured values, U_{interday} the coefficient of interday variation, n the number of values used to calculate the mean and U_{ref} standard uncertainty associated with the reference value (estimated by calculation at 1 %).

The two criteria (maximum tolerated range of 60 % and $E_N<2$) are fulfilled: (1) the mean contents measured (±2 times the standard deviation of the intermediate precision), in this case 0.018 to 0.027 mg/kg, has to be located in the range of ±60 % of the spiking value (0.012 to 0.048 mg/kg), and (2) the value of E_N is less than 2. The limit of quantification of 0.03 mg/kg is validated.

Repeatability, interday precision and uncertainty of the method

An experimental design, of the accuracy profile type, was used to validate the repeatability and interday precision. It consists in analysing the variance and calculating the intra and inter-series variations. The repeatability and interday precision was achieved by doubling the extraction for the three soils on five different dates. The analyses were performed independently for each of the five series of analysis.

The coefficients of interday variation were 8, 6 and 9 % for the three levels of concentration of CLD (1, 2 and 16 mg/kg) of the NIT, FRL and AND soils (Table 3), respectively.

The uncertainty is calculated from the evaluation of the repeatability/interday precision of the three soils, from tests relative to the limit of quantification, and from study of the extraction efficiency.

By definition, the uncertainty (U) is a function of the intermediate precision (U_{interday}) and of the bias of the method (U_b) (ISO/DIS 11352 [21]).

$$U = \sqrt{U_{\mathrm{ineteday}}{}^2 + U_b{}^2}$$

The component U_b is calculated from the root mean square of the observed bias (RMS_b) when spiking was performed during the validation and the uncertainty on the concentration of spiking level (U_{add}).

$$U_b = \sqrt{\mathrm{RMS_b}{}^2 + U_{\mathrm{add}}{}^2}$$

$$\mathrm{RMS_b} = \sqrt{\frac{\sum b_i{}^2}{n}}$$

Where b_i is the observed bias compared with a total recovery of 100 % and n the number of tests (where $n \geq 6$).

The values of the coefficients of interday variation (U_{interday}) were 10, 8, 6 and 9 % for the soils at 0.03 mg/kg (NIT, FRL and AND), 1 mg/kg (NIT), 2 mg/kg (FRL) and 16 mg/kg (AND), respectively.

The uncertainty for the value of the spiking level at the LOQ (U_{add}) was 1 %. It was calculated during the weighing operations performed during the preparation of the different solutions.

The mean bias (RMS_b) calculated from observed biases during the study of the extraction efficiency is 13 %. The uncertainty U is increased by the factor $k=2$ (expanded uncertainty at a confidence level of approximately 95 %). The measuring uncertainty associated with the CLD result is 40 % for level between 0.03 and 1 mg/kg, and 30 % for a concentration greater than 1 mg/kg.

Conclusions

The method described is suitable for the analysis of chlordecone in the three main types of French West Indies soils taking account of the soils high content of organic carbon and supplementary inputs thereof due to remediation processes. The limit of quantification of the method is 0.03 mg/kg. The associated measuring uncertainty is 40 % for a concentration between 0.03 and 1 mg/kg and 30 % for a concentration above 1 mg/kg. This method ensures that the effects observed in remediation studies are only due to the remediation processes based on ISCR and addition of compost, not to analytical artefacts.

However, the accuracy could not be estimated since there is no reference material for CLD and inter-laboratory tests have yet to be conducted.

Acknowledgments The assistance given by Y.M. Cabidoche (INRA Guadeloupe) for the identification of the sites from where the contaminated soils were retrieved was much appreciated. Samples of non-contaminated soils were collected in Martinique with the logistical and scientific help of the IRD/CNRS, L. Rangon, T. Woignier and H. Macarie; we thank them. The INRA-11-AAP DEMICHLORD-02-01 convention enabled the finalisation of certain aspects of the work described herein. We gratefully thank the French Ministry of Environment for its financial support through contracts SU 000 6693 MEEDDM/DGPR and DGPR N 181 SU 0007446 Grenelle Environnement.

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