TEXTE
133/2022

Global approach (Part A): Are substances more persistent than test systems lead to believe? Non-extractable residues: experimental examination of suitable extraction methods in view of a long-term risk for the environment

Annex

For our Environment

Umwelt Bundesamt

TEXTE 133/2022

Environmental Research of the
Federal Ministry for the
Environment, Nature Conservation, Nuclear Safety
and Consumer Protection

Project No. (FKZ) 3713 63 413 1

Report No. FB000707/ENG

Global approach (Part A): Are substances more persistent than test systems lead to believe? Non-extractable residues: experimental examination of suitable extraction methods in view of a long-term risk for the environment

Annex

by

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On behalf of the German Environment Agency

Imprint

Publisher:
Umweltbundesamt
Wörlitzer Platz 1
06844 Dessau-Roßlau
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Internet: www.umweltbundesamt.de

/umweltbundesamt.de
/umweltbundesamt

Study performed by:
Bundesanstalt für Gewässerkunde (Federal Institute of Hydrology)
Am Mainzer Tor 1
56068 Koblenz

Study completed in:
2021

Edited by:
Section Fachgebiet IV 2.2 - Umweltbewertung von Arzneimitteln
Astrid Wiemann

Publication as pdf:
http://www.umweltbundesamt.de/publikationen

ISSN 1862-4804

Dessau-Roßlau, Dezember 2022

A1 Annex

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A2 Soil and Substance selection

A2.1 Selection of substances of interest

Pre-selection of compounds (see Table A1) for further testing at BFG was based on three pillars:

(i) Data on formation of non-extractable residues (NER) from internal databases provided by the UBA

(ii) Data on NER formation from scientific literature

(iii) Results and experiences of BFG from incubation experiments and extraction of compounds from solid matrices

Table A1: List of potential target substances that were used for the soil incubation experiments

Substance	CAS number	Main application
Acetaminophen	103-90-2	Analgesic
Amprolium	137-88-2	Coccidiostat
Benzyldimethyldodecylammonium*	139-07-1	Disinfectant
Carbendazim	10605-21-7	Fungicide
Climbazole	38083-17-9	Fungicide
Dimethomorph	110488-70-5	Fungicide
Ethinylestradiol	57-63-6	Estrogen
Fenoxycarb	72490-01-8	Insecticide
Fenpropimorph	67564-91-4	Fungicide
Florfenicole	76639-94-6	Antibiotic
Flumequine	42835-25-6	Antibiotic
Isoproturon	34123-59-6	Herbicide
Ketoconazole	65277-42-1	Fungicide
Mebendazole	31431-39-7	Antihelmintic
Mesosulfuron methyl	208465-21-8	Herbicide
Propiconazole	60207-90-1	Fungicide
Triclosan	3380-34-5	Disinfectant

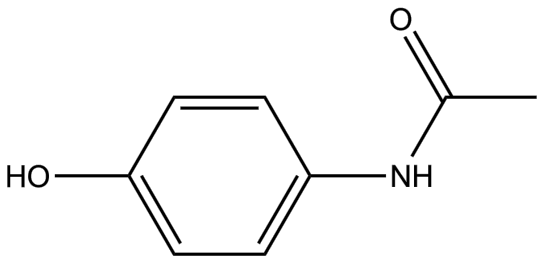
* either bromide of chloride, CAS number is given for the chloride salt

In the following chapters the most relevant environmental data (field of application, physico-chemical properties, occurrence, and fate) of the 17 pre-selected substances as well as their reported potentials to form NER are briefly discussed.

A2.1.1 Acetaminophen (Paracetamol)

The analgesic and antipyretic agent acetaminophen (also named paracetamol, Figure A1) might be one of the most well-known drugs worldwide. Acetaminophen is a very polar substance (log K_{OW} = 0.5; log K_{OC} = 1.3-1.5; (US EPA 2012)) with a pK_a of 9.4 (Wan et al. 2003). Acetaminophen is known to be readily biodegradable during wastewater treatment and in soils (Li et al. 2014, Radjenovic et al. 2007) and therefore despite the high concentrations of acetaminophen entering wastewater treatment plants (WWTPs), concentration in WWTP effluents and receiving waters are relatively low (Radjenovic et al. 2007). However, a rapid and intense formation of NER of acetaminophen in soils and sediments has been observed. In sediments, after 100 days 60% NER formation was reported, while after a 120 day incubation with different soils 64-78% of BERS were determined (both data provided by UBA (2013)). During the incubations with soil a rapid transformation of acetaminophen was observed (DT_{50} ~ 1 d) also showing the biodegradability of acetaminophen. In a recent study, the biologically controlled formation of NER of acetaminophen was observed (Li et al. 2014).

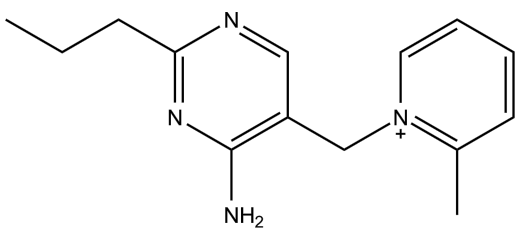
Figure A1: Chemical structure of the analgesic drug acetaminophen



A2.1.2 Amprolium

The coccidiostat amprolium is mainly applied in poultry. Amprolium is a quaternary ammonium compound and therefore permanently positively charged. Amprolium is a rather polar substance with a log K_{OW} of -2.5 and restive log K_{OC} values of 0-4 (both obtained from US EPA (2012)). Scientific literature data on occurrence and fate of amprolium in the environment is scarce. However, it was determined in effluents of a poultry farm in concentrations of up to 290 ng L^{-1} (Song et al. 2010). In soils, after a 120 day incubation of amprolium 60-70% of NER were observed while the respective DT_{50} were 44-70 days (UBA 2013).

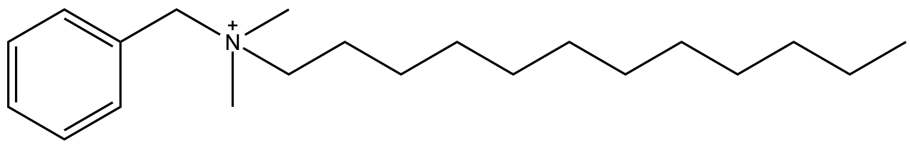
Figure A2: Chemical structure of the coccidiostat amprolium



A2.1.3 Benzyldimethyldodecylammonium salt (BDDA)

Benzyldimethyldodecylammonium chloride (or bromide) is a cationic surface-acting agent that is widely applied for three different purposes: i) as biocide and disinfectant agent, ii) as cationic surfactant and iii) as phase transfer catalyst in the chemical industry. Due to its high usage rates, BDDA is frequently detected in various environmental compartments. In hospital effluents, concentrations of up to 100 mg L^{-1} were determined (Kreuzinger et al. 2007). Due to its permanently positive charge and the long apolar alkyl chain benzyldimethyldodecylammonium is highly sorptive with respective log K_{OW} and log K_{OC} values of 2.9 and 2.5-5.5 (US EPA 2012) and therefore extremely high concentrations of BDDA in sludge, soil and sediments were reported (Li and Brownawell 2009, Martinez-Carballo et al. 2007). While UBA was not able to provide data on NER formation of BDDA in soils, a study currently performed by BFG in collaboration with Ed Topp from AAFC (Agriculture and Agri-Food Canada) revealed a potential for NER formation and therefore BDDA was also selected as target substance for the pre-experiments.

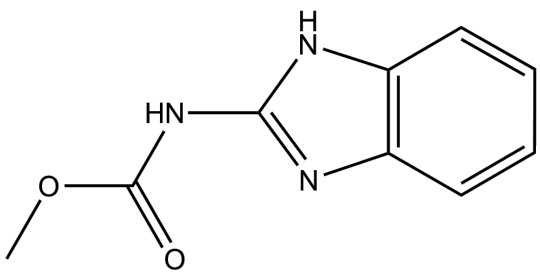
Figure A3: Chemical structure of benzyldimethylammomium salt (chloride or bromide)



A2.1.4 Carbendazim

The fungicide carbendazim is widely used for the protection of fruits, vegetables and crops. It is also applied as additive in sealants to prevent from fungal infestation. The pK_a of carbendazim is 4.5 (Mazellier et al. 2003). Due to its relatively high polarity ($logK_{OW}$ = 1.5 (US EPA 2012), $logK_{OC}$ = 1.9 (Wick et al. 2011)), carbendazim is predominantly present in the dissolved phase. Carbendazim is not removed during conventional wastewater treatment and concentrations in WWTP effluents are usually in the mid ng L^{-1} range (BFG 2014b). Carbendazim was not detected in leachates from agricultural soils where frequently a mixtures of sewage sludge and treated wastewater is irrigated and only very low concentrations (~ ng g^{-1}) in the solid phase were determined (BFG 2014a). Scientific literature reports a high potential of NER formation for carbendazim in soils as e.g. Lewandowska and Walorczyk (2010) determined 50 % NER in 1000 days after the application to the soil. In addition, data provided by the UBA give high values for NER formation ranging from 43 to 81 % after 120 days using five different soils (UBA 2013). Mineralization rates were usually less than 15 % and 2-aminobenzimidazole was always identified as major transformation product (TP) resulting from the cleavage of the amide structure.

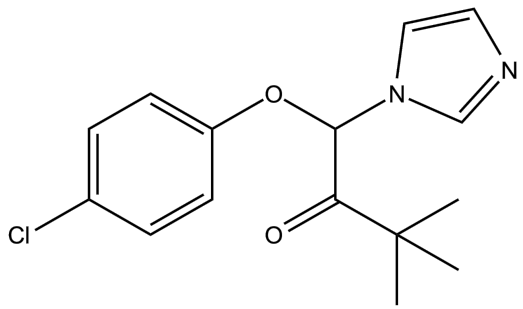
Figure A4: Chemical structure of the fungicide carbendazim



A2.1.5 Climbazole

The fungicide climbazole is an anti-dandruff agent that is a common ingredient of shampoos and cosmetics. Climbazole has a pK_a of 7.5 (Wick et al. 2010) and a log K_{OW} of 1.7 (US EPA 2012). The respective log K_{OC} is depended on the pH value of the matrix and varies between 3-4 in a pH from 6 to 8.5 (Wick et al. 2014). It is commonly detected in WWTP influents, WWTP effluents, wastewater impacted rivers as well as sewage sludge, sediment and soils (BFG 2014a, b, Wick et al. 2014). During wastewater treatment and in soils, climbazole is transformed into one major and persistent TP (Wick et al. 2014). While neither scientific literature nor the UBA was able to provide profound data for formation of NER in soil, BFG observed unclosed mass balances when incubating climbazole in soils and therefore climbazole was added to the list of potential target substances.

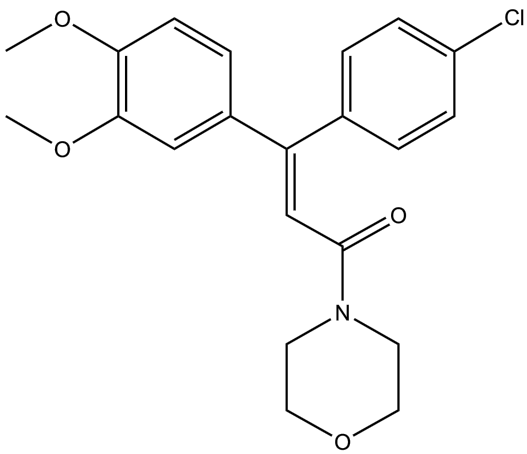
Figure A5: Chemical structure of the fungicide climabzole



A2.1.6 Dimethomorph

Dimethomorph in an antifungal agent that is used for the protection of crops and fruits. It is moderately sorbing to solid matrices and possesses of log K_{OW} of 2.7 (US EPA 2012) and a log K_{OC} of 2.2 was determined in sewage sludge (Wick et al. 2011). In the aquatic environment dimethomorph can be transformed by photochemical processes (Calza et al. 2008). In soil, a DT_{50} of 12-19 days was determined (Liang et al. 2011) while only scarce information on biologically formed TPs are available. In data provided by the UBA, a strong NER formation of dimethomorph in soils of 18-57 % was reported. Mineralization was between five and 30 % and the DT_{50} for dimethomorph were 41-96 days (UBA 2013).

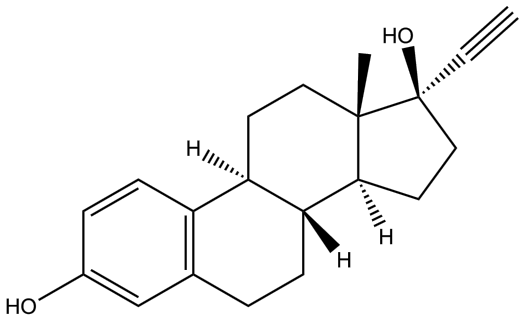
Figure A6: Chemical structure of the fungicide dimethomorph



A2.1.7 Ethinylestradiol (EE2)

The estrogen ethinylestradiol (EE2), is one of the most discussed substances for a potential inclusion on the list of priority substances defined in the Water Framework Directive. The proposed Environmental Quality Standard is as low as 35 pg L^{-1} due to the low no-effect concentrations. The log K_{OW} of EE2 is 3.7 and the modelled log K_{OC} are between 2.7 and 4.6 (US EPA 2012). EE2 is only partially eliminated during biological wastewater treatment (Ternes et al. 1999) and the predominant transformation reactions are hydroxylations and dehydrations (Kresinova et al. 2012). In sediments, 50-63 % NER formation was observed after an incubation of 99 days (UBA 2013).

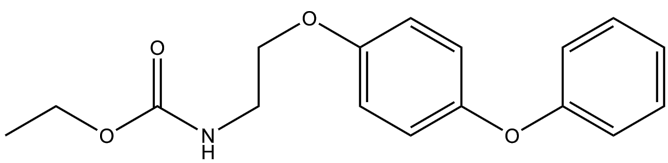
Figure A7: Chemical structure of the estrogen ethinylestradiol



A2.1.8 Fenoxycarb

The insecticide fenoxycarb is widely applied in fruit growing and vinery. It is also used as ingredient in wood protection agents. The pK_a of fenoxycarb is 12.1 and the log K_{OW} and log K_{OC} provided in the EPI Suite are 4.3 and 3.3-3.7 (US EPA 2012). Scientific literature data on the occurrence and fate of fenoxycarb in the aquatic and terrestric environment is very scarce and not reported here. However, fenoxycarb possesses a high potential to generate NER in soils: data provided by UBA listed 41-68 % formation after incubation in different soils over about 90 days (UBA 2013). The DT_{50} of fenoxycarb in these experiments ranged from two to 21 days.

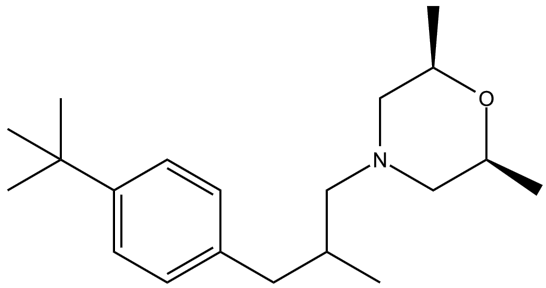
Figure A8: Chemical structure of the insecticide fenoxycarb



A2.1.9 Fenpropimorph

Fenpropimorph is a morpholine-derived fungicide that is widely used in agriculture for the protection of cereal crops. It is also used as ingredients in timber preservatives against blight. The pK_a of fenpropimorph is 7.5 (Taton et al. 1987). The log K_{OW} is given at 4.9 in the EPI Suite (US EPA 2012) and a log K_d of 2.2 was determined in soil (Spliid 2001). In soils, fenpropimorph is mainly transformed to one TP – fenpropimorph acid – which could still be detected several months after application of fenpropimorph to soil (Spliid 2001). In data provided by UBA, NER formations of 33-56 % after incubations of 91 or 119 days are listed (UBA 2013). In these tests, mineralization rates were relatively high (33 – 49 %) and the respective DT_{50} values ranged from 10 to 124 days.

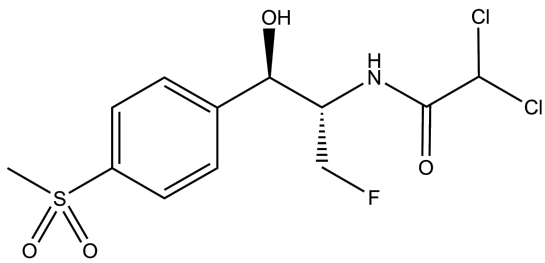
Figure A9: Chemical structure of the fungicide fenpropimorph



A2.1.10 Florfenicol

Florfenicol is a veterinary antibiotic drug. It has a pK_a of 9.0 (Mitchell et al. 2013) and a log K_{OW} of -0.1 (Mitchell et al. 2013). Florfenicol has been detected in animal farm-effluent, river, and pond water in concentrations of up to 2.8 µg L^{-1} (Wei et al. 2012). Sun et al. (2012) determined a good photochemical elimination in surface waters (DT_{50} ~ 2 days) for florfenicol while it was more stable in experiments with river sediments. During anaerobic digestion, florfenicol was rapidly transformed but the resulting TPs were almost stable for 40 days (Mitchell et al. 2013). UBA provided a large dataset on the formation of florfenicol NER in soils and NER formation of up to 69 % after incubation of 92 days was reported. In accordance with the studies by Mitchell et al. (2013) a rapid transformation was observed and respective DT_{50} values ranged from one to eleven days (UBA 2013).

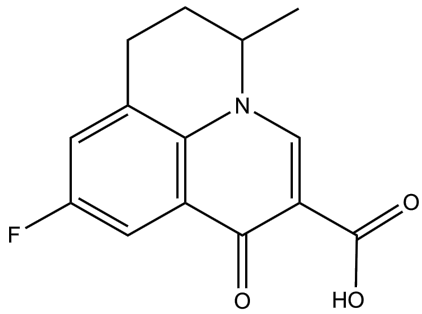
Figure A10: Chemical structure of the antibiotic drug florfenicol



A2.1.11 Flumequine

Flumequine is an antibiotic drug belonging to the group of the fluoroquinolones. It is a rather polar substance with a log K_{OW} of 1.6 and a modelled log K_{OC} of 1.2-1.6 (US EPA 2012). The pK_a of flumequine is 6.3 (Babić et al. 2007). Cvancarova et al. (2013) observed a biotransformation of flumequine by ligninolytic fungi and a subsequent a formation of persistent TPs. In surface waters, flumequine can be eliminated by photochemical processes and the main photo TPs were identified (Sirtori et al. 2012). Nevertheless, flumequine was detected in large rivers in concentrations of the mid ng L^{-1} range (Tamtam et al. 2008). In data by UBA, flumequine exhibited an NER formation between 32 and 96 % after 120 days of incubation and the respective DT_{50} was > 120 days (UBA 2013).

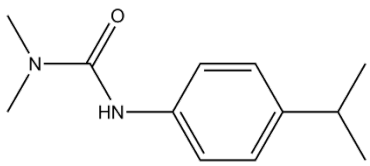
Figure A11: Chemical structure of the antibiotic flumequine



A2.1.12 Isoproturon

Isoproturon is an herbicide that is mainly used for the protection of cereal crops. The log K_{OW} of isoproturon is 2.5 and the log K_{OC} is 1.5 (both Wick et al. (2011)). While mainly applied in agriculture, isoproturon is also frequently detected in raw and treated wastewater (BFG 2014b). The biological transformation of isoproturon is well investigated and transformation reactions mainly consist of demethylations and hydroxylations (Badawi et al. 2009, Penning et al. 2010). Isoproturon is known for intense NER formation: Lehr et al. (1996) reported of 56-61 % NER formation soil after an incubation of 32 days while Barriuso et al. (2008) listed 56-68 % formation of NER and simultaneous mineralization rates between ten and 22 %. In accordance, studies provided by UBA also reported high NER formation rates of 42-59 % after 100 to 181 days and mineralization rates ranged from 17 to 32 %. The main TP was identified as N-desmethyl-isoproturon (UBA 2013).

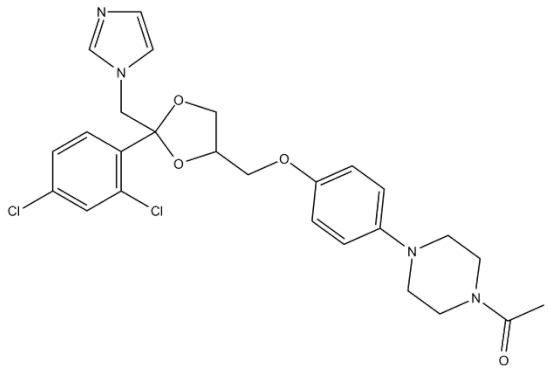
Figure A12: Chemical structure of the herbicide isoproturon



A2.1.13 Ketoconazole

The fungicide ketoconazole is mainly used in human medicine against acromycosis of the skin and as ingredient of anti-dandruff shampoos. The pK_a values of ketoconazole are 3.3 und 6.5 (Wan et al. 2003). Due to its high apolarity (log K_{OW} = 4.4) ketoconazole is strongly sorbing to solid matrices and the log K_{OC} is 3.5-4.3 (US EPA 2012). Ketoconazole was detected in raw wastewater in concentrations of up to 100 ng L^{-1} and 230 µg kg^{-1} in secondary sludge (Huang et al. 2012). Moreover, ketoconazole is extremely persistent against hydrolysis (Skiba et al. 2000). Although neither UBA nor scientific literature was able to provide information on NER formation of ketoconazole in soils or sediment, ketoconazole was added to the list of potential target substances due to BFG's experiences with low extraction efficiencies of ketoconazole from solids (sludge, soil, sediment).

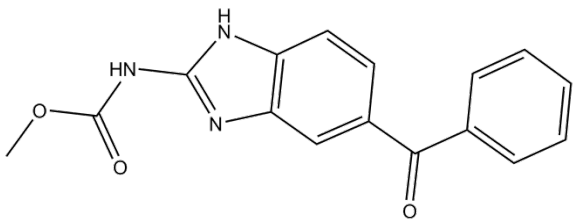
Figure A13: Chemical structure of the fungicide ketoconazole



A2.1.14 Mebendazole

Mebendazole is a benzimidazole drug that is applied to treat infestations by worms (antihelmintic drug). It is used both in human and veterinary medicine, sometimes in combination with the fellow antihelmintic drug closantel. The pK_a of mebendazole is 3.4 (Wan et al. 2003). The respective log K_{OW} and log K_{OC} values provided by the EPI Suite are 2.8 and 3.0-3.5 (US EPA 2012) indicating a moderate sorption affinity of mebendazole. During human and veterinary metabolism, mebendazole is transformed by hydroxylation and amination reactions (Liu et al. 2010). Literature data on the occurrence and fate of mebendazole in the environment is scarce. However, an intense formation of NER in soils has been reported. Data provided by the UBA (2013) are e.g. 26-65% NER after 180 days of incubation or 27-64% NER formation after 118 days with a respective DT_{50} of 22-138 days.

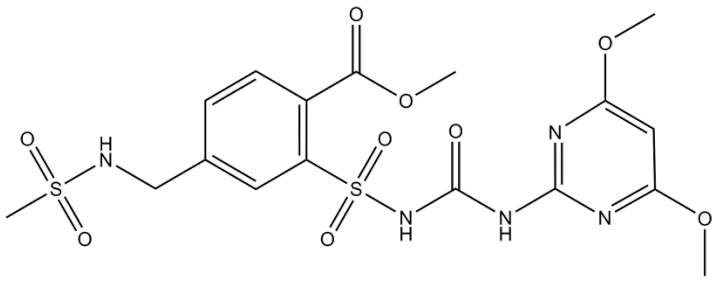
Figure A14: Chemical structure of the antihelmintic drug mebendazole



A2.1.15 Mesosulfuron methyl

The mesosulfuron methyl is the pro drug of the herbicide mesosulfuron which is widely used for the projection of crops and vegetables. The pK_a of mesosulfuron methyl is 4.4 (European Commission 2004) and the log K_{OW} and log K_{OC} values are -2 to 1 and 1.6 to 2.8 (European Commission 2004). At neutral pH, mesosulfuron is persistent against both hydrolysis and photochemical transformation (European Commission 2004) and Lazartigues et al. (2011) were able to quantify mesosulfuron methyl in river sediments. In soils, 28-67 % NER are formed after a 90 day incubation of mesosulfuron methyl and mineralization rates during these tests ranged from six to 47 % (UBA 2013).

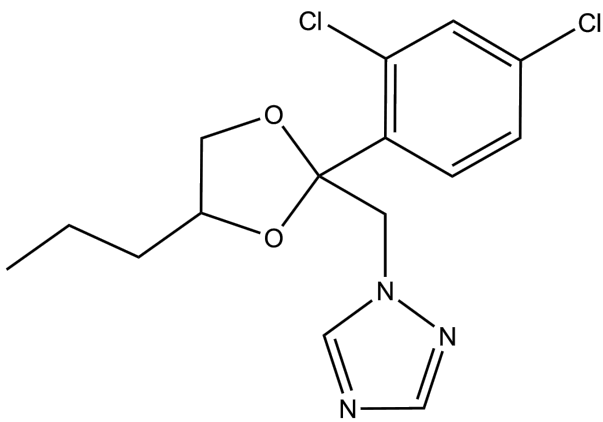
Figure A15: Chemical structure of the herbicide mesosulfuron methyl



A2.1.16 Propiconazole

Propiconazole is a triazole fungicide that is widely used for the protection of crops and fruits in agriculture. Propiconazole is also a common ingredient of timber preservatives and is applied a mixture of its four stereoisomers. The pK_a of propiconazole is 1.1 (Wick et al. 2011), the log K_{OW} 3.7 (US EPA 2012) and a log K_{OC} of 3.1 was determined (Wick et al. 2011). Therefore, it is moderately sorbing to solid matrices. In sediments, a slow DT_{50} for propiconazole of approx. 50 days was determined and the loss was attributed to aerobic transformation (Garrison et al. 2011). In surface waters, propiconazole can be transformed by direct and indirect phototransformation processes with respective half-life of some days (Vialaton et al. 2001). In soils, propiconazole is pretty persistent and almost no mineralization is determined. Barriuso et al. (2008) reported of 47% NER formation after 120 d with only 2 % mineralization. Additional data provided by the UBA (2013) indicate a slightly lower potential for NER formation and values of up to 27 % were provided.

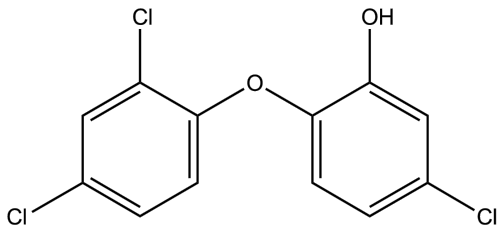
Figure A16: Chemical structure of the fungicide propiconazole



A2.1.17 Triclosan

Triclosan is a widely used biocide that is common ingredient of personal care products such as soaps or toothpastes. Due to its high usage rates, triclosan is present in high concentrations in sludge amended soil and wastewater effluent. Concentrations of up to 10 µg L^{-1} were detected in effluents from WWTPs (Chen et al. 2011). During biological wastewater treatment and in soil is mainly transformed to one TP (methyl-triclosan) which is pretty stable in the environment (Butler et al. 2012, Chen et al. 2011). Triclosan is a rather apolar substance that possesses a log K_{OW} of 4.8 and log K_{OC} of 4.6 (Wick et al. 2011) and therefore strongly sorbs to solid matrices like soil, sludge or sediments. In ^{14}C-labelled experiments in soils with, up to 80% after 40 days were observed while the mineralization rate was less than 15 % (Al-Rajab et al. 2009). Additionally, in data provided by the UBA, maximum NER formation of triclosan ranged from 28 to 76% while the mineralization rates were also always less than 20% (UBA 2013). In accordance to scientific literature, methyl-triclosan was always identified as min TP (12-24% of spiked triclosan).

Figure A17: Chemical structure of the micro biocide triclosan



A2.2 Selection of potential soils for the experiments

Table A2: Overview of the physicochemical properties of the six soils used for the soil incubation experiments

	Lufa 2.1	Lufa 2.2	Lufa 2.3	Lufa 2.4	BS[1]	Eurosoil 5
C_{org} (%)	0.62	1.87	0.94	2.42	1.4	5.96
N (%)	0.05	0.17	0.08	0.2	0.13	0.23
pH (-)	5.1 ± 0.4	5.5 ± 0.2	6.8 ± 0.2	7.1 ± 0.2	6.9 ± 0.2	4.1 ± 0.2
CEC (meq/100g)	4 ± 0.7	10 ± 0.5	10 ± 1.3	30 ± 5.1	30 ± 10	n.a.
Sand (%)	90	80	60	26	90	82
Silt (%)	8	13	31	46	8	13
Clay	2	7	9	28	2	5
Texture[2]	Ss	Sl2	Sl3	Lt2	Ss	Su2
	pure sand	weakly loamy sand	medium loamy sand	weakly clayey loam	pure sand	weakly silty sand

[1] Soil from agricultural field site where mixtures of sewage sludge and wastewater have been irrigated for more than 60 years, site is located near Braunschweig, [2] Classification after AG Boden (1994), n.a.: not available

A2.3 Experimental approach and analytics

A2.3.1 Batch incubations

For each setup, 50 g of soil (calculations based on dry weight) were incubated in slender beakers and target analytes were spiked at a concentration of 500 ng g^{-1}. To get a homogeneous spiking of the soil with the target substances the following procedure was applied. A 5 g subsample of each soil was put in a small flask and 5 mL of a mixture (set up in methanol) that contained each substance in a concentration of 5 mg L^{-1} were added. The soil was well mixed with the spiked methanolic solution and methanol was let evaporate over night. Then, this spiked subsample of soil was well mixed with the non-spiked soil using overhead shaker for 2 hours. For each soil the gravimetric water content was adjusted to 20% and beakers with the soils were put into a climate cabinet. Incubations were performed at 20 °C at 100% humidity to prevent from evaporation and changes in water content during experiments. Additionally, a perforated parafoil was put on each beaker. Samples from each soil were taken after putting the soil into the climate cabinet (0 days) and then again after 12, 22 and 33 days. For sampling, approx. 10 g of soil (fresh weight) were taken and frozen. A subsample was used for the determination of water content (constant during incubation for all soils). Samples were then freeze-dried and stored dry until further sample processing.

A2.3.2 Ultrasonic Extraction (USE)

The freeze-dried soil samples were extracted by ultrasonic extraction to check for weaker sorption of substances. Thereto, 0.5 – 2.0 g of dry soil were weighted into class centrifuge tubes. The amount of soil was chosen depending on the C_{org} content of the respective soil. This was applied to obtain a similar matrix in all extracts. For Eurosoil 5, approx. 0.5 g of soil was extracted, for Lufa 2.2, Lufa 2.4 and BS approx. 1.0 g was extracted and for Lufa 2.1 and Lufa 2.3 approx 2.0 g of soil was extracted. Before extraction, 25 ng of a

surrogate standard containing isotopic labelled standards were added (25 µL of a mix of 1 mg L^{-1}). Then, 10 mL of ultra pure water/methanol/acetone (1:1:1, v/v/v) were added. Extraction was performed for 10 min at a temperature of 40 °C. Afterwards, the extract was centrifuged and the supernatant was decanted. Extraction steps were repeated four times. The extracts (~ 40 mL) were then diluted with ultra pure water to a total volume of 50 mL was filtrated through a glass fibre filter. Approx. 1 mL of the filtrate was transferred into an HPLC vial and stored at 4°C until further analysis.

A2.3.3 Accelerated Solvent Extraction (ASE)

The freeze-dried soil samples were extracted by accelerated solvent extraction to check for stronger bonding of substances. The used amounts of soil and addition of isotopic labels standards were equivalent to the ultrasonic extraction procedure. Extraction was achieved by pressurized liquid extraction (PLE) with an Accelerated Solvent Extraction system (ASE 200, Dionex, Idstein, Germany). The respective amounts of soil (0.5 – 2.0 g) were filled into extraction cells (22 mL) and isotopic labelled internal standards were added. Cells were pre-filled with sea sand and soil was mixed with the sea sand. PLE was accomplished by four extraction cycles with a mixture of ultra pure water/methanol/acetone (1:1:1, v/v/v) at a pressure of 100 bar and a temperature of 80 °C. Subsequently, the extract was diluted with ultra pure water to a volume of 50 mL. Approx. 1 mL of the filtrate was transferred into an HPLC vial and stored at 4°C until further analysis.

A2.3.4 HPLC-MS/MS

Determination of organic contaminants was done by a LC-(ESI)-MS/MS system consisting of a binary LC pump (Agilent 1260) and a tandem mass spectrometer (API 6500, AB Sciex, Darmstadt, Germany). Quantification was done using isotopic dilution methods. Separation was achieved by a binary gradient of ultra pure water (positive mode: 0.1% FA, negative mode: pure water) and ACN (pos. mode: 0.1% FA, neg. mode: pure ACN) on a Zorbax-Ca8 column (150 x 2.1 mm, 3.5 µm, Agilent). The flow rate was 300 µL min-1, column temperature was set to 35 °C and injection volume was 40 µL for positive mode (80 µL for negative mode). Calibration was linear in the range from 0 to 25,000 ng L-1 and the limits of quantification were 0.2 – 10 ng g-1 depending on substance and amount of extracted soil. For each substance, at least two transitions were monitored in multi reaction mode (MRM). Except for triclosan, all other substances were determined in the positive ionization mode. Table A3 gives more detailed on precursor and product ions as well as assigned internal standards for the substances where no authentic isotopic labelled standard were available. It was not possible to measure acetaminophen, amprolium and ethinylestradiol in the same chromatographic run than the other compounds due to different physicochemical properties of these substances. Due to an instrument breakdown and limited measuring time, so far results for these three compounds are incomplete and not reported here.

Table A3: Details on mass transitions and assigned isotopic labelled surrogate standards

Substance	MRM 1	MRM 2	Isotopic labelled standard
BDDA	304.3>91.1	304.3>212.2	BDDA-D5
Carbendazim	192.1>160.1	192.1>132.1	Carbendazim-D4
Climbazole	295.1>197.0	295.1>69.0	Climbazole-D4
Dimethomorph	388.1>301.1	388.1>165.1	Isoproturon-D6
Fenoxycarb	302.3>116.1	302.3>88.1	Climbazole-D4
Fenpropimorph	304.3>117.1	304.3>147.2	Isoproturon-D6
Florfenicol	377.2>243.1	377.2>243.1	Isoproturon-D6
Flumequine	262.2>244.2	262.2>201.1	Isoproturon-D6
Isoproturon	207.1>165.1	207.1>72.1	Isoproturon-D6
Ketoconazole	531.1>244.1	533.1>491.1	Ketoconazole-D8
Mebendazole	296.3>264.1	296.3>77.0	Mebendazole-D3
Mesosulfuron methyl	504.2>182.0	504.2>83.0	Climbazole-D4
Propiconazole	344.1>161.1	342.>159.1	Propiconazole-D5
Triclosan	287.0>35.0	289.0>37.0	13C12-Triclosan

A2.4 Results and discussion

In the following, the results for the seventeen individual substances (as far as currently available) are shortly reported and discussed.

A2.4.1 Acetaminophen

Due to an instrument breakdown, no data of acetaminophen from the incubation experiments are currently available.

A2.4.2 Amprolium

Due to an instrument breakdown, no data of amprolium from the incubation experiments are currently available.

A2.4.3 Benzyldimethyldodecylammonium salt

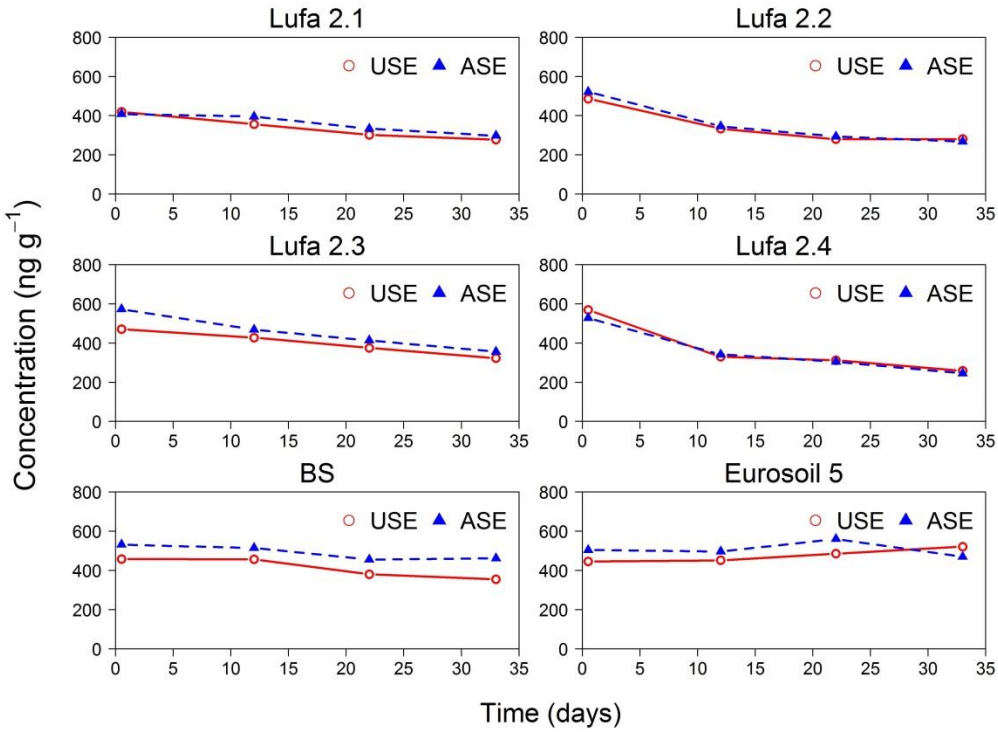
The time trends of BDDA during the soil incubation experiments are given in Figure A18. The main results are as follows:

In all soils, the target concentration of 500 ng g^{-1} of benzyldimethyldodecyl-ammonium salt was measured at the beginning of the incubation (within the precision of the applied method (± 20 %)).

For soils Lufa 2.3, BS and Eurosoil 5 a slightly higher concentration of BDDA was determined with ASE compared to USE.

Concentrations of BBDA decreased in incubations experiments with soil LuFa 2.2, 2.3 and 2.4 over time. No substantial change in extractable concentration of BBDA over time was observed in incubations with Lufa 2.1, BS and Eurosoil 5.

Figure A18: Time trend of extractable benzyldimethyldodecylammonium salt concentrations during the incubation experiments; USE: Ultrasonic Extraction; ASE: Accelerated Solvent Extraction



A2.4.4 Carbendazim

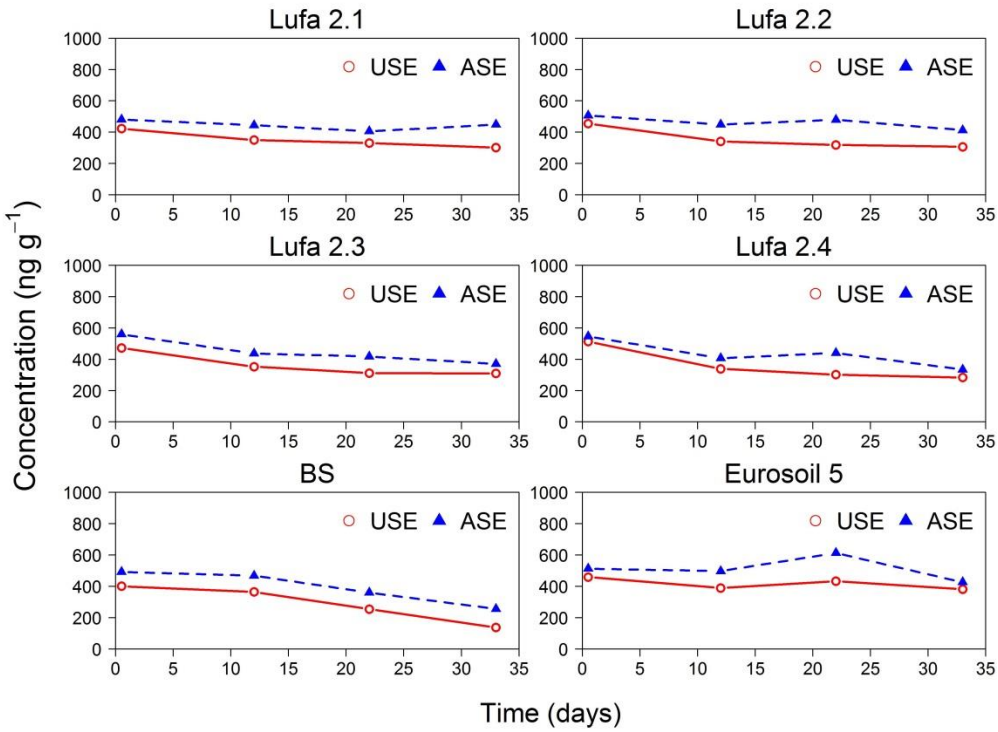
The time trends of carbendazim during the soil incubation experiments are given in Figure A19. The main results are as follows:

Extraction with ASE resulted in higher extraction efficiencies of carbendazim than USE in all soils.

In all soils, the target concentration of 500 ng g^{-1} was measured at the beginning of the incubation when using ASE (within the precision of the applied method (± 20 %)).

Concentrations of carbendazim decreased in incubations experiments with soils Lufa 2.3 and 2.4 as well as BS over time. No substantial change in extractable concentration of carbendazim over time was observed in incubations with Lufa 2.1, Lufa 2.2 and Eurosoil 5.

Figure A19: Time trend of extractable carbendazim concentrations during the incubation experiments; USE: Ultrasonic Extraction; ASE: Accelerated Solvent Extraction



A2.4.5 Climbazole

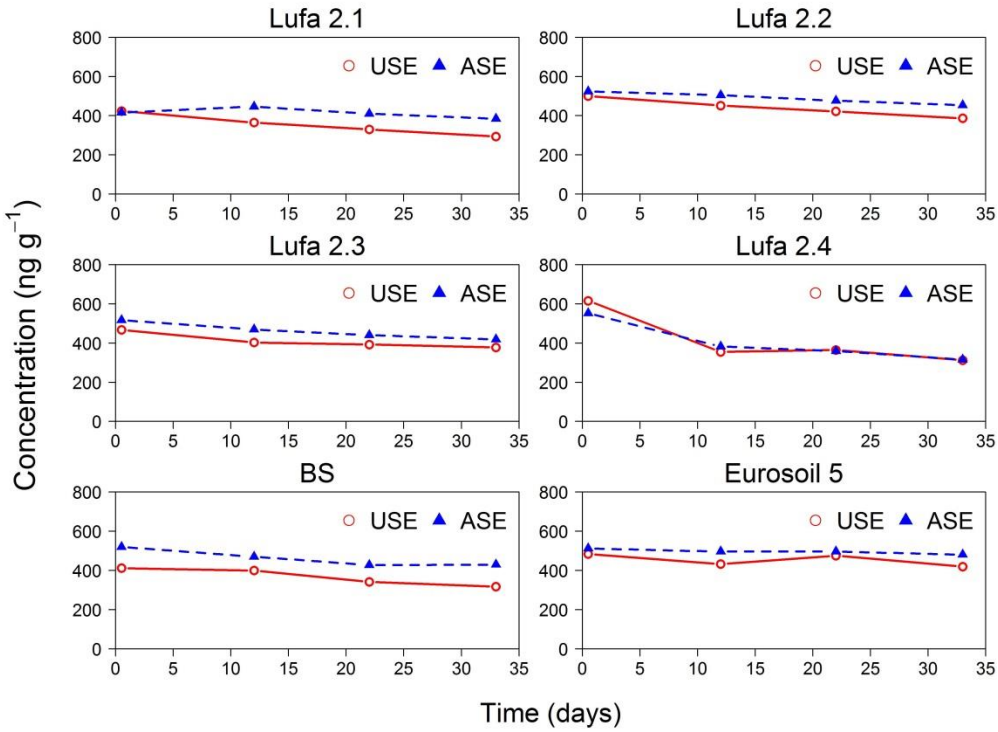
The time trends of climbazole during the soil incubation experiments are given in Figure A20.The main results are as follows:

There might be a slightly higher extraction efficiency of climbazole when using ASE compared to USE. However, except for soils Lufa 2.3 and BS differences are small and scatter over the course of the experiments.

Except for Lufa 2.1 and 2.4 the target concentration of 500 ng g^{-1} was measured at the beginning of the incubation (within the precision of the applied method (± 20 %)).

Concentrations of climbazole decreased in incubations experiments with soil Lufa 2.4. In all other soils, no substantial decrease of climbazole concentration over time was observed.

Figure A20: Time trend of extractable climbazole concentrations during the incubation experiments; USE: Ultrasonic Extraction; ASE: Accelerated Solvent Extraction



In addition to climbazole, we also determined a transformation product of climbazole (called climbazole TP which is formed by a reduction of the keto group) in the soil extracts on a semi-quantitative basis. The time trends of normalized peaks areas of climbazole TP during the soil incubation experiments are given in Figure A21. The main results are as follows:

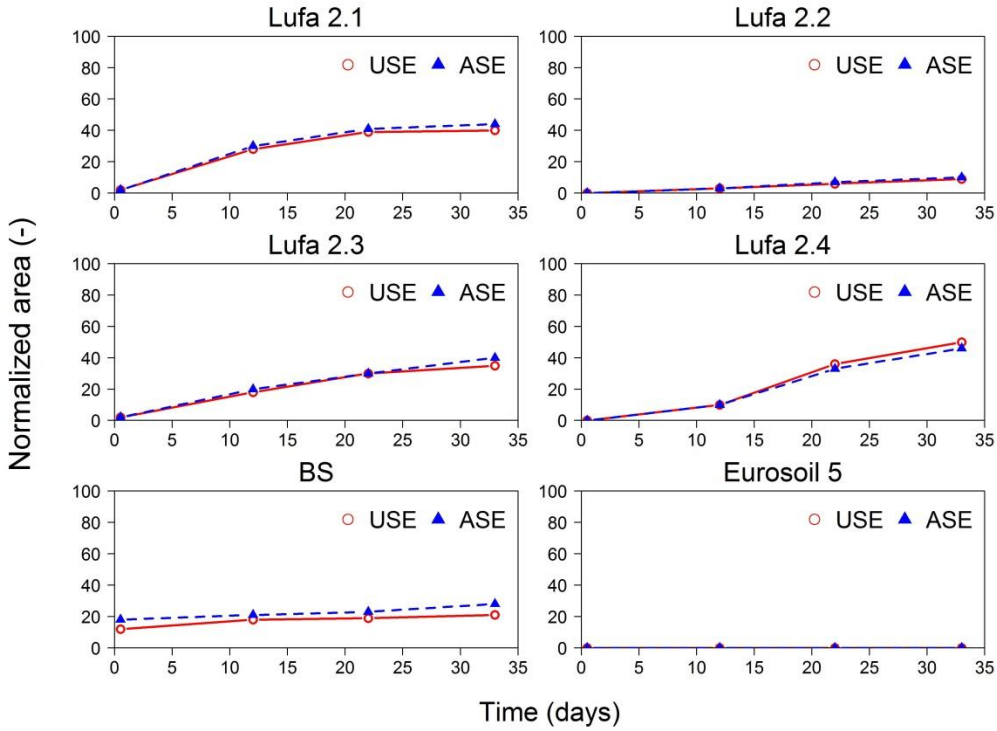
The amount of extractable climbazole TP was equal when using USE and ASE.

The formed amount of climbazole TP was only small compared to the spiked amount of climbazole. Estimated concentrations of climbazole based on peaks areas are < 5 % of initial climbazole concentrations.

Except for soil Eurosoil 5, climbazole TP was formed in all five other soils. Formation rates were highest soil Lufa 2.4 where the strongest reduction of the initial climbazole concentration was observed.

In Soil BS, climbazole TP was already present at the beginning of the incubations since this soil frequently receives input of climbazole via treated wastewater and sewage sludge.

Figure A21: Time trend of extractable climbazole TP (TP of climbazole, displayed as normalized peak area) during the incubation experiments; USE: Ultrasonic Extraction; ASE: Accelerated Solvent Extraction



A2.4.6 Dimethomorph

The time trends of dimethomorph during the soil incubation experiments are given in Figure A22. The main results are as follows:

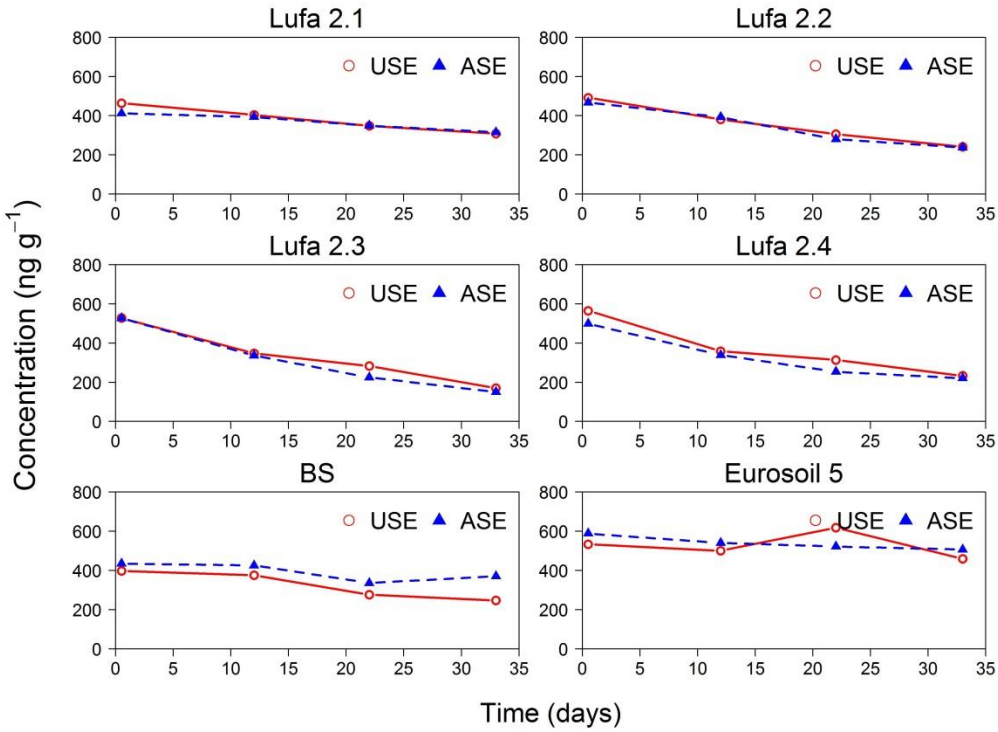
In all soils, the target concentration of 500 ng g^{-1} of dimethomorph was measured at the beginning of the incubation (within the precision of the applied method (± 20 %)). This was achieved without having an authentic internal standard.

Only for soil BS, there was a slightly higher amount of extractable dimethomorph when using ASE instead of USE. For all other soils, differences between the two extraction techniques were insignificant.

In Eurosoil, the amount of extractable dimethomorph was constant over the time course of the experiment. In all other soils, the amounts decreased over time potentially due to combined effect of transformation of dimethomorph and formation of NER.

The extractable concentrations of dimethomorph decreased strongest in soils Lufa 2.3 and 2.4.

Figure A22: Time trend of extractable dimethomorph concentrations during the incubation experiments; USE: Ultrasonic Extraction; ASE: Accelerated Solvent Extraction



A2.4.7 Ethinylestradiol

Due to an instrument breakdown, no data of ethinylestradiol from the incubation experiments are currently available.

A2.4.8 Fenoxycarb

The time trends of fenoxycarb during the soil incubation experiments are given in Figure A23. The main results are as follows:

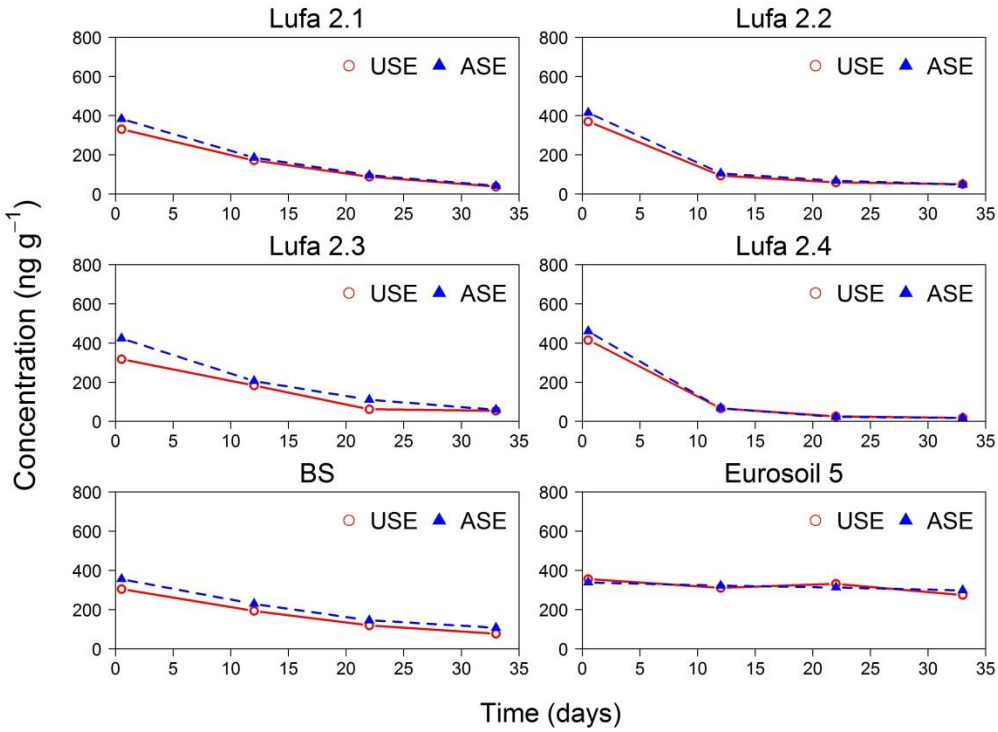
In all soils, the target concentration of 500 ng g^{-1} of fenoxycarb was slightly underestimated. This might be either caused by (i) an immediate transformation of fenoxycarb, (ii) an instantaneous formation of NER or (iii) a systematic error in the quantification of fenoxycarb (quantification via climbazole-D4).

There was no substantial difference in the extraction efficacy for fenoxycarb using ASE or USE.

Except for Eurosoil 5, extractable concentrations of fenoxycarb decreased over time due to transformation reactions and/or formation of NER. Concentration reductions were strongest for soils Lufa 2.2 and Lufa 2.4.

Since concentrations of fenoxycarb in Eurosoil 5 were constant over time, the systematic underestimation of the initial fenoxycarb concentrations is most likely caused by the non-availability of an authentic isotopic-labelled standard and not by formation of NER and/or transformation.

Figure A23: Time trend of extractable fenoxycarb concentrations during the incubation experiments; USE: Ultrasonic Extraction; ASE: Accelerated Solvent Extraction



A2.4.9 Fenpropimorph

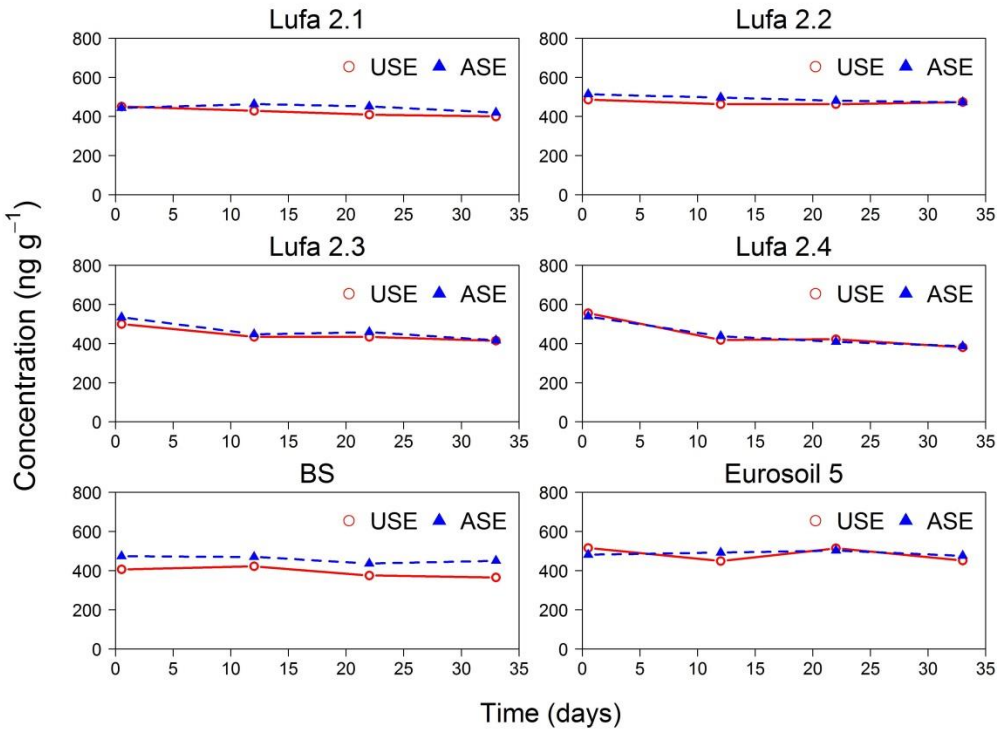
The time trends of fenpropimorph during the soil incubation experiments are given in Figure A24. The main results are as follows:

In all soils, the target concentration of 500 ng g^{-1} of fenpropimorph was measured at the beginning of the incubation (within the precision of the applied method (± 20 %)). This was achieved without having an authentic internal standard.

Only in soil BS, ASE resulted in a slightly higher extraction efficacy than USE.

Only for soils Lufa 2.3 and Lufa 2.4, a slight reduction of extractable concentration of fenpropimorph over time was observed.

Figure A24: Time trend of extractable fenpropimorph concentrations during the incubation experiments; USE: Ultrasonic Extraction; ASE: Accelerated Solvent Extraction



A2.4.10 Florfenicol

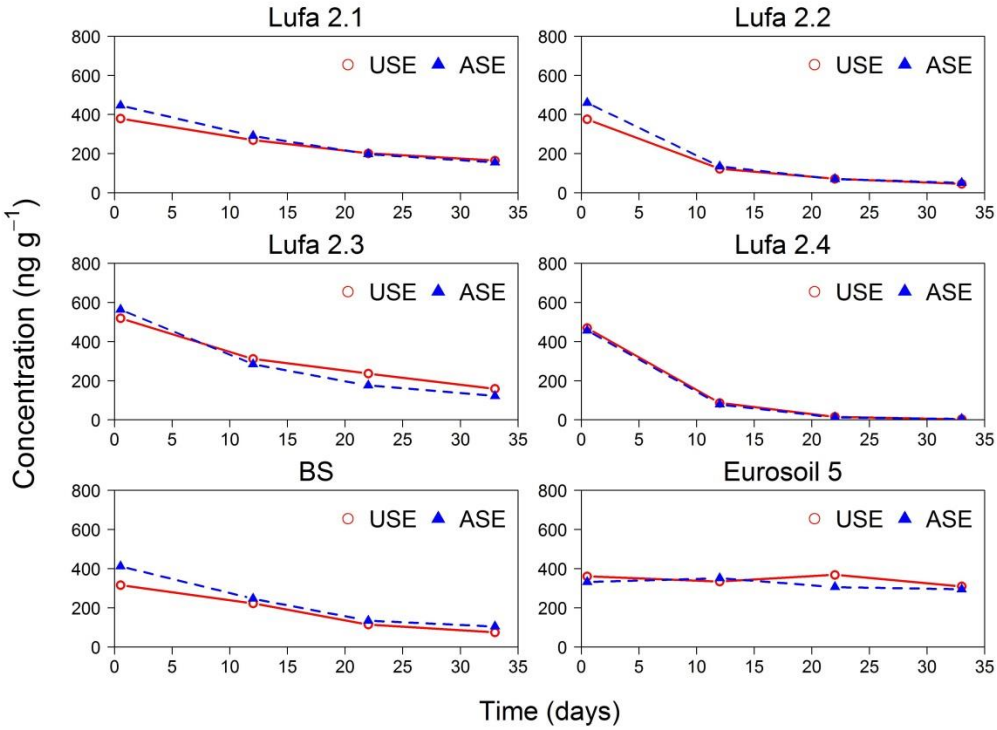
The time trends of florfenicol during the soil incubation experiments are given in Figure A25. The main results are as follows:

In some soils (BS and Eurosoil 5), the target concentration of 500 ng g^{-1} of florfenicol was slightly underestimated. This might be either caused by an instantaneous formation of NER or a systematic error in the quantification of florfenicol (quantification via isoproturon-D6).

Except for Eurosoil 5, extractable concentrations of florfenicol decreased over time due to transformation reactions and/or formation of NER. Concentration reductions were strongest for soils Lufa 2.2 and Lufa 2.4.

There was no significant difference in the extraction efficacy when using USE or ASE.

Figure A25: Time trend of extractable florfenicol concentrations during the incubation experiments; USE: Ultrasonic Extraction; ASE: Accelerated Solvent Extraction



A2.4.11 Flumequine

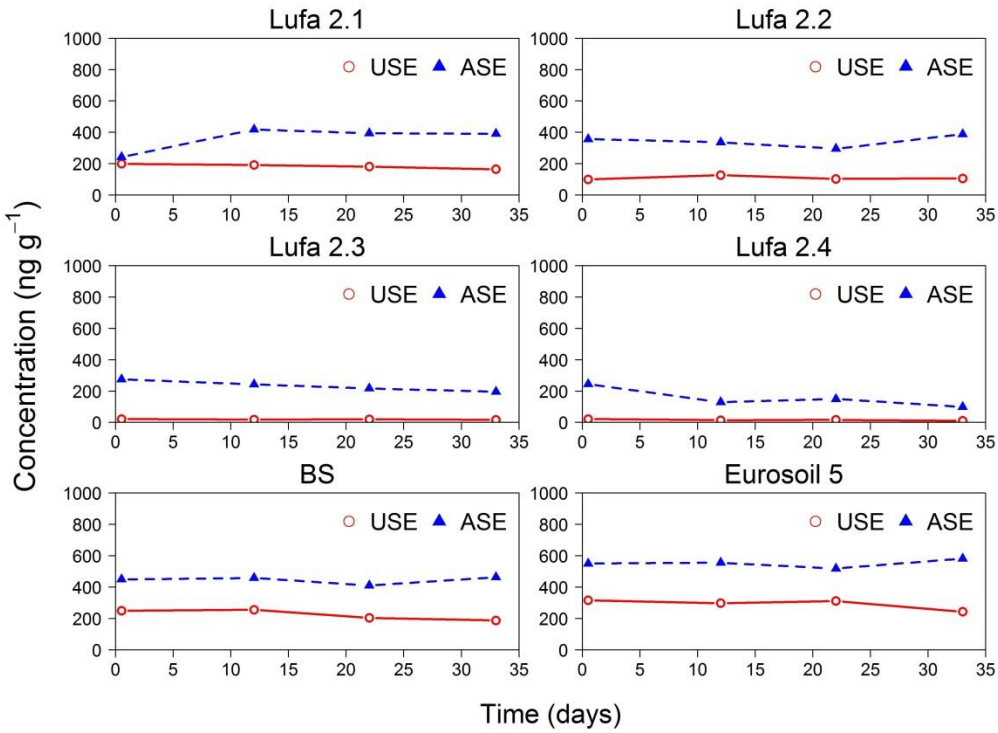
The time trends of flumequine during the soil incubation experiments are given in Figure A26. The main results are as follows:

In all soils, ASE resulted in significant higher extraction efficacies of flumequine than ASE. The low initial value for ASE in soil Lufa 2.1 may potentially be caused by an incomplete mixing at the beginning of the experiment.

Both ASE and USE did not result in measuring the target concentration of 500 ng g^{-1} flumequine. Since the (extractable) concentrations of flumequine were (almost) constant over the time course of the experiment, this may either be caused by an instantaneous formation of NER during the preparations steps of the experiment or by a systematic error since no authentic isotropic labelled standard was available (flumequine was quantified using isoproturon-D6). However, this systematic error then has to be matrix depended (extracts made by ASE or USE) and therefore, a rapid initial formation of NER seems more likely.

Only in soil Lufa 2.4, a slightly reduction of extractable flumequine over time was observed when ASE was used. This decreasing trend is concordant with the lowest extraction efficacies of flumequine using USE in all six soils. Hence, the formation of NER for flumequine might be proportional to the clay content of the soil (Lufa 2.4 has the highest clay content).

Figure A26: Time trend of extractable flumequine concentrations during the incubation experiments; USE: Ultrasonic Extraction; ASE: Accelerated Solvent Extraction



A2.4.12 Isoproturon

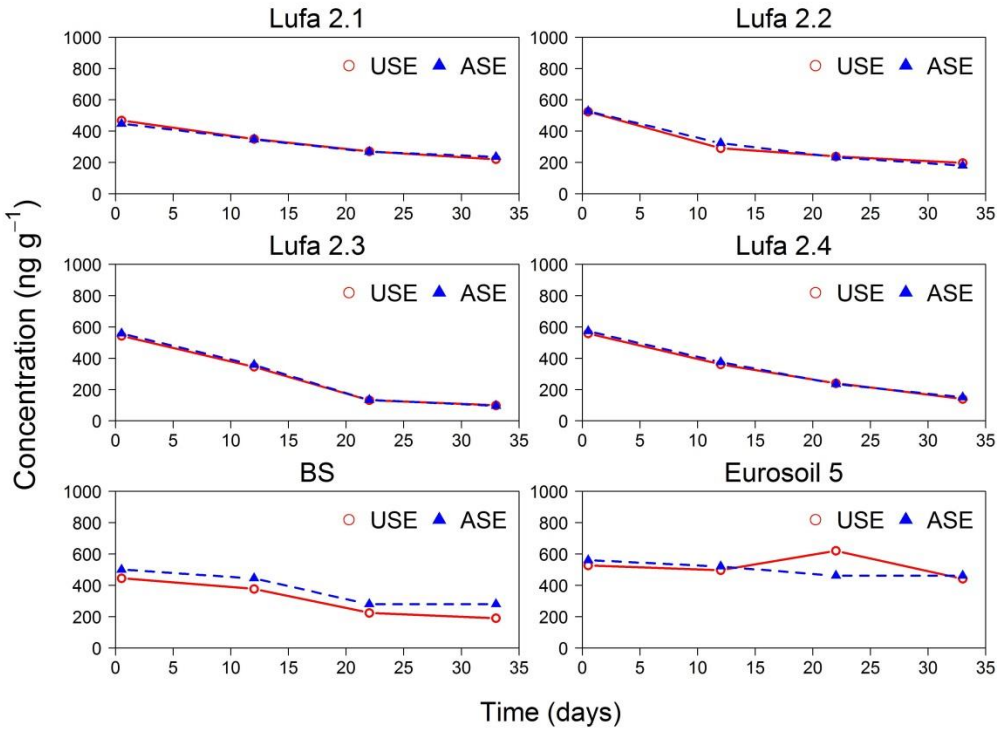
The time trends of isoproturon during the soil incubation experiments are given in Figure A27. The main results are as follows:

In all soils, the target concentration of 500 ng g^{-1} of isoproturon was measured at the beginning of the incubation (within the precision of the applied method (± 20 %)).

Except for soil, BS, concentrations determined in extracts derived from USE or ASE were equal at all sampling points. In extracts from soil BS, ASE achieved slightly higher extraction efficacies than USE at all sampling dates.

Except for Eurosoil 5, extractable amounts of isoproturon decreased over time and concentration decrease was highest in soils Lufa 2.3 and Lufa 2.4. This decrease is potentially governed by transformation processes of isoproturon or by a biologically induced NER isoproturon formation.

Figure A27: Time trend of extractable isoproturon concentrations during the incubation experiments; USE: Ultrasonic Extraction; ASE: Accelerated Solvent Extraction



A2.4.13 Ketoconazole

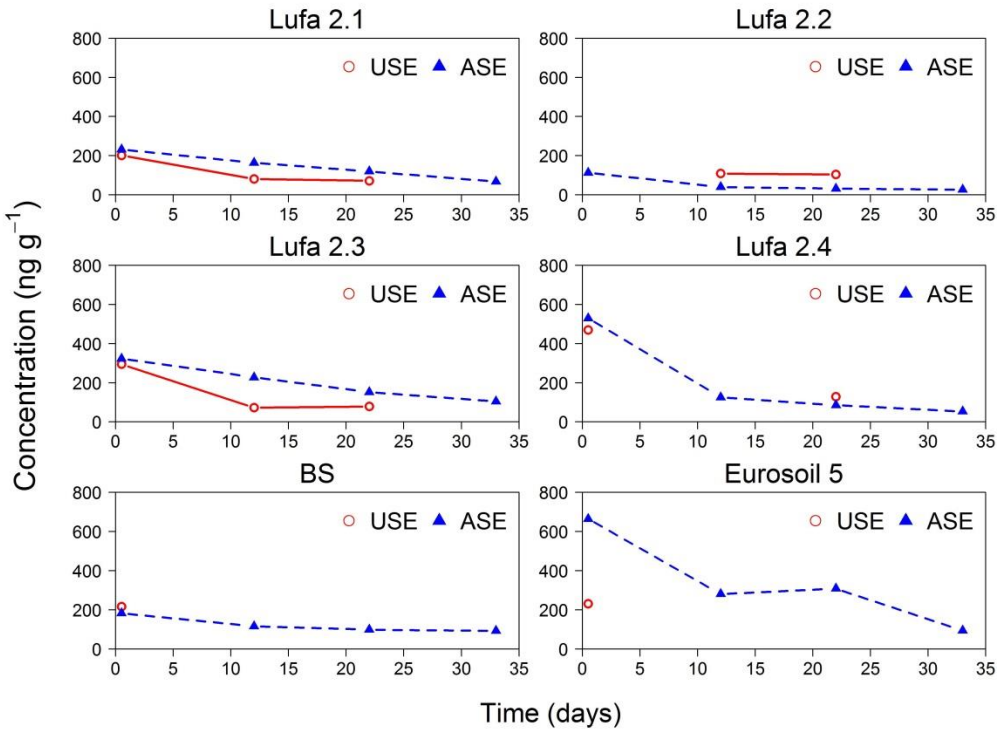
The time trends of ketoconazole during the soil incubation experiments are given in Figure A28. The main results are as follows:

The initial concentration of 500 ng g^{-1} of ketoconazole was never determined in soils using both ASE and USE. While concentrations (ASE) were underestimated in soils Lufa 2.1, Lufa 2.2, Lufa 2.3 and BS, too high values were determined in soils Lufa 2.4 and Eurosoil 5.

For several samples of all soils, calculation of concentrations after the extraction via USE was not possible (missing red lines/circles Figure A28). This was caused by a too low recovery of the internal standard ketoconazole-D8. Although this standard was added to the soil directly before the extraction, no peak was found in many samples. If ketoconazole-D8 was not recovered also no peak of the spiked ketoconazole was visible. There are two major explanations for this: (i) ketoconazole-D8 immediately formed a very strong binding to the soils, USE was too weak the break this binding and therefore ketocanzole-D8 and ketoconazole itself were not recovered. (ii) The samples derived by USE were filtered through a glass fibre filter after extraction and centrifugation. This procedure was not applied to samples after ASE since these extracts were less turbid and filtration was not necessary. Potentially, ketoconazole and ketoconazole-D8 were removed by filtering the USE samples. Also note that for the few USE samples where ketoconazole was quantified, the absolute recoveries and peak intensities were really low and therefore the analytical uncertainty of these values are much bigger than for the ASE extraction samples.

In incubations with all six soils, the extracted amounts of ketoconazole after ASE decreased over the time course of the experiments. This decrease was most pronounced for soil Lufa 2.4 and Eurosoil 5 – the soils with the height C_{org} content.

Figure A28: Time trend of extractable ketoconazole concentrations during the incubation experiments; USE: Ultrasonic Extraction; ASE: Accelerated Solvent Extraction



A2.4.14 Mebendazole

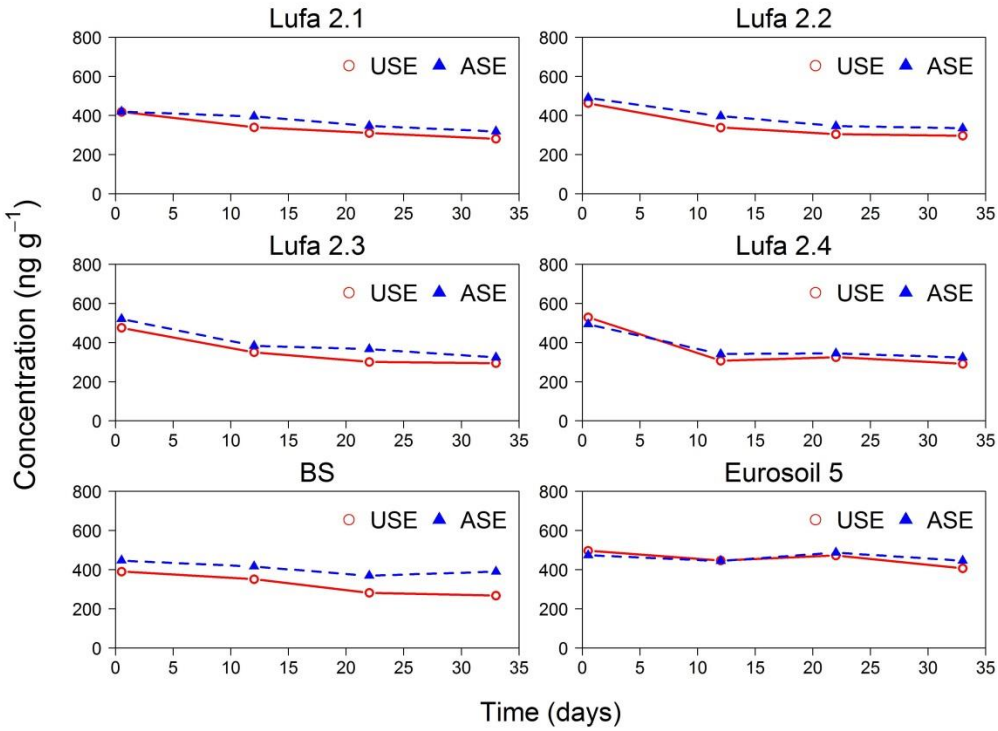
The time trends of mebendazole during the soil incubation experiments are given in Figure A29. The main results are as follows:

In most soils, the target concentration of 500 ng g^{-1} of mebendazole was measured at the beginning of the incubation (within the precision of the applied method (± 20 %)). In Lufa 2.1 and BS, the initial determined concentration was only about 400 ng g^{-1} due to unknown reasons.

Except for Eurosoil 5, ASE resulted in a slightly higher extraction efficacy than USE. However, this increased extractability of mebendazole when using ASE was only of minor importance (<10%).

For all four Lufa soils, a slight decrease in the extraction efficacies of mebendazole over the time courses of the incubation texts was observed. In contrast, the extraction efficiency in BS and Eurosoil 5 was constant over time.

Figure A29: Time trend of extractable mebendazole concentrations during the incubation experiments; USE: Ultrasonic Extraction; ASE: Accelerated Solvent Extraction



A2.4.15 Mesosulfuron methyl

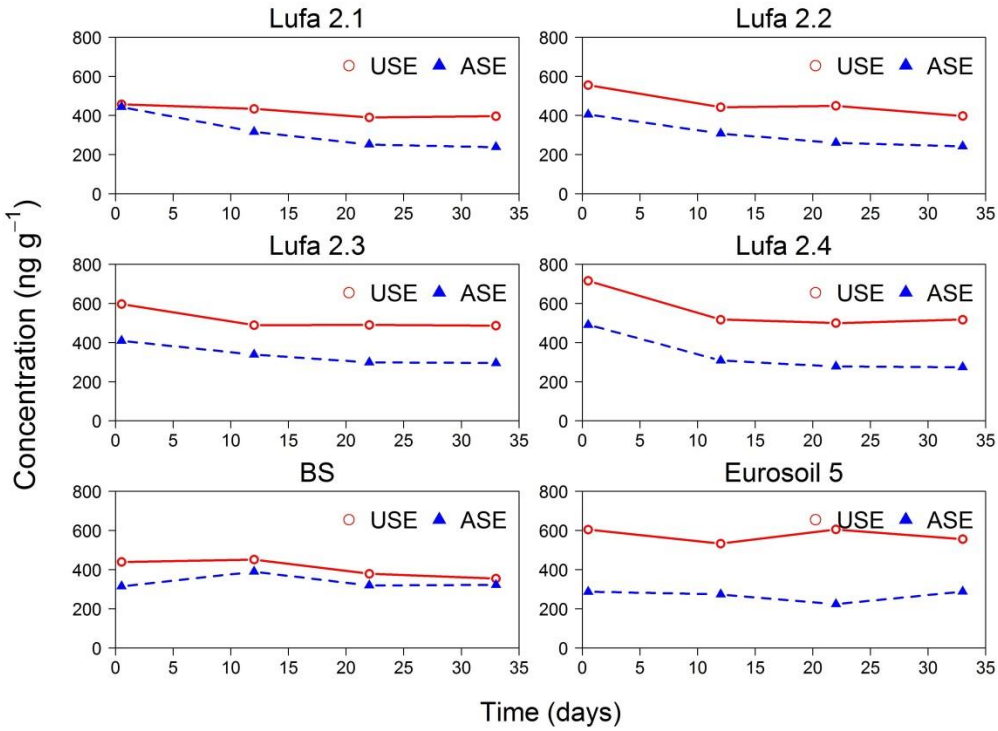
The time trends of mesosulfuron methyl during the soil incubation experiments are given in Figure A30. The main results are as follows:

The initial concentration of 500 ng g^{-1} of mesosulfuron methyl was only sporadically determined in soils using both ASE and USE. While concentrations were underestimated after ASE, USE tended to overdetermine the initial concentration of mesosulfuron methyl. This is most presumably explained by the missing authentic isotopic labelled surrogate standard (mesosulfuron methyl was quantified using climbazole-D4)

In samples from all time points and all soils, USE resulted in higher extracted amounts of mesosulfuron methyl than ASE. This is the only substances where a higher extraction results by USE was observed. Additionally, the ratio of the concentration determined by USE and ASE seems to be matrix depended and this overestimation by USE was highest in soils with the highest C_{org} contents (Lufa 2.2, Lufa 2.4 and Eurosoil 5).

Except for soil Lufa 2.4, the extractability of mesosulfuron methyl (compared to the concentrations determined in the initial samples) did not decrease over the time course of the experiment.

Figure A30: Time trend of extractable mesosulfuron methyl concentrations during the incubation experiments; USE: Ultrasonic Extraction; ASE: Accelerated Solvent Extraction



A2.4.16 Propiconazole

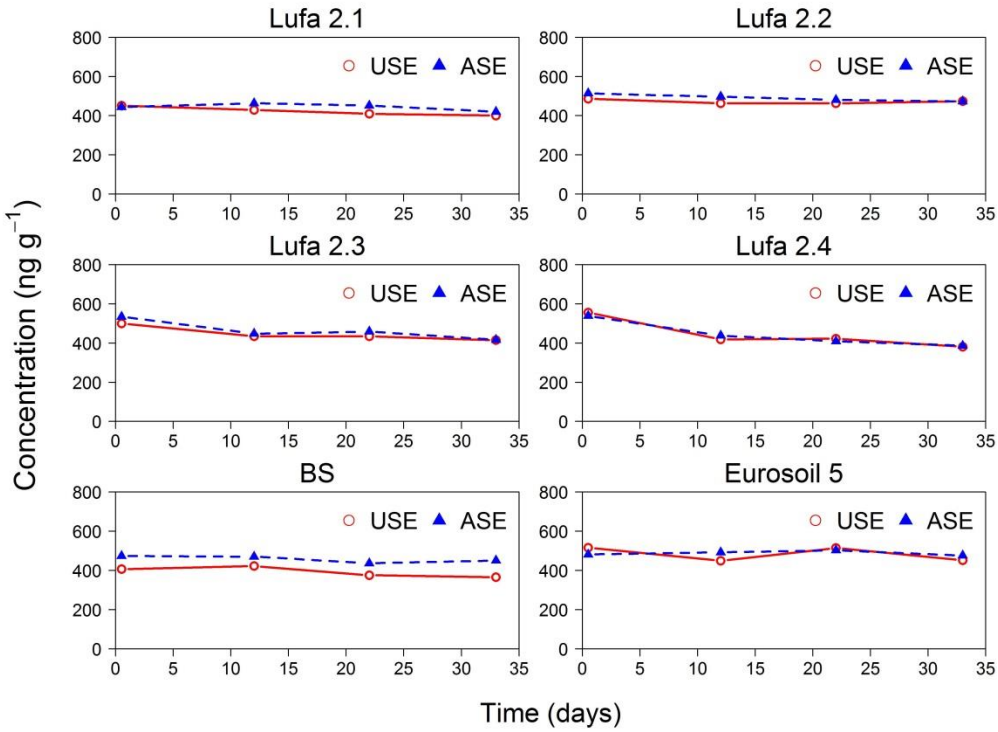
The time trends of propiconazole during the soil incubation experiments are given in Figure A31.The main results are as follows:

In all soils, the target concentration of 500 ng g^{-1} of propiconazole was measured at the beginning of the incubation (within the precision of the applied method (± 20 %)).

Except for soil BS, the extractability of propiconazole using ASE was equal to the extraction efficacy of USE. In soil BS, a slightly higher concentration was determined using ASE.

Concentrations of propiconazole decreased in incubations experiments with soil Luda 2.3 and Lufa 2.4 over time. No substantial change in extractable concentration of propiconazole over time was observed in incubations with Lufa 2.1, Lufa 2.2, BS and Eurosoil 5.

Figure A31: Time trend of extractable propiconazole concentrations during the incubation experiments; USE: Ultrasonic Extraction; ASE: Accelerated Solvent Extraction



A2.4.17 Triclosan

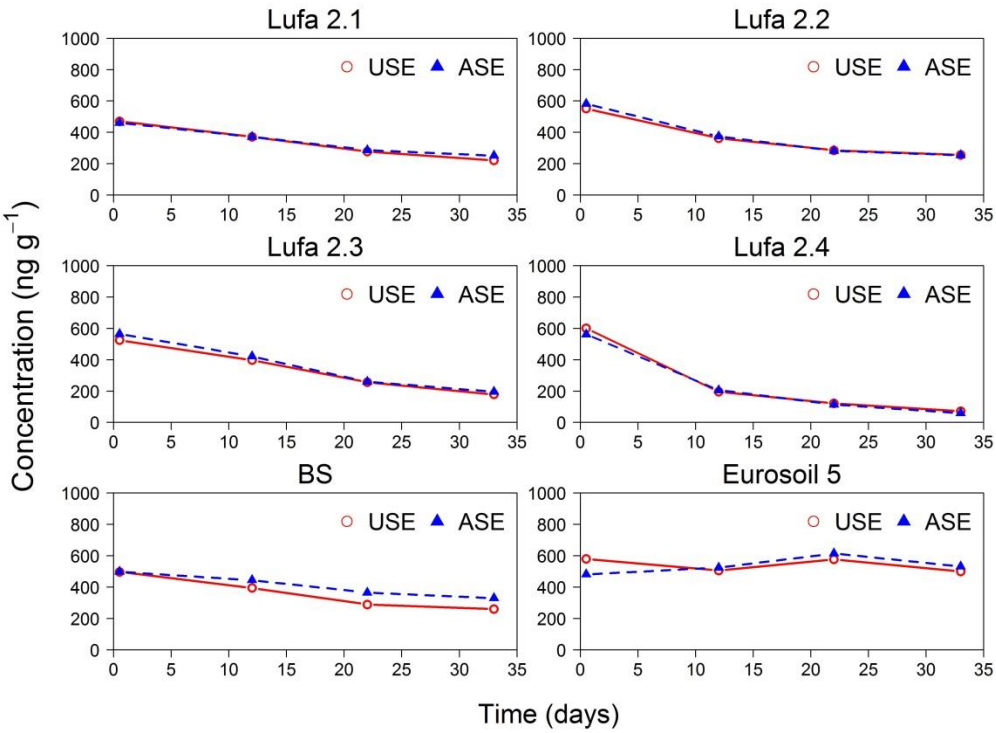
The time trends of triclosan during the soil incubation experiments are given in Figure A32. The main results are as follows:

In all soils, the target concentration of 500 ng g^{-1} of triclosan was met at the beginning of the incubation (within the precision of the applied method (± 20 %)).

Except for soil BS, the extractability of triclosan using ASE was equal to the extraction efficacy of USE. In soil BS, a slightly higher concentration was determined using ASE and the difference increased over the time course of the experiment.

Concentrations of triclosan decreased in incubations experiments with all soils except for Eurosoil 5. The reduction of the extraction success decreased with the clay content of the soils as it was highest for soil Lufa 2.4. This extractability loss over time was either caused by the formation of NER, a transformation of triclosan (to methyl-triclosan) or a combination of both processes.

Figure A32: Time trend of extractable triclosan concentrations during the incubation experiments; USE: Ultrasonic Extraction; ASE: Accelerated Solvent Extraction



A2.5 Final Selection

In this chapter, the main results of the incubation experiments are summarized and the decisions made on the basis of previous knowledge and the results from the tests are briefly listed.

Table A4: Extractable proportion (% of initial concentration) of each substance after 33 days of incubation in the six different soils using ASE.

	Lufa 2.1	Lufa 2.2	Lufa 2.3	Lufa 2.4	BS	Euro-soil 5	Mean[2]	SD[2]
BDDA	73	51	62	47	87	93	69	19
Carbendazim	93	82	66	61	52	83	73	16
Climbazole	93	87	81	57	83	94	82	13
Dimethomorph	77	51	29	44	85	86	62	24
Fenoxycarb	11	12	14	4	30	88	27	31
Fenpropimorph	80	77	51	12	86		65	29
Florfenicol	35	11	22	1	26	89	31	31
Flumequine	161	109	71	41	103	106	98	40
Isoproturon	53	34	17	26	56	82	45	24
Ketoconazole	29	24	33	10	51	14	27	15
Mebenbazole	76	68	62	66	87	94	76	13
Mesosulfuron methyl	54	60	72	56	103	100	74	22
Propiconazole	94	92	78	72	95	99	88	11
Triclosan	55	44	35	11	66	110	53	34
Mean[1]	70	57	50	36	72	87		
SD[1]	37	30	24	25	25	23		

[1]Mean values and standard deviation (SD) of residual extractable fraction of all 14 substances in the respective soil, [2]Mean values and standard deviation (SD) of residual extractable fraction of a substance in the six different soils

A3 Experimental comparison study of soil extraction methods

A3.1 Substances and supplier HPLC-MS/MS-method

Table A5: Substances used for the experimental comparison study of soil extraction methods

	Substance	CAS	Formula	Supplier	ESI	RT	DW	Q1	Q3a/Q3b	DP	CEa/CEb
Nervous System	Diazepam	439-14-5	C16H13ClN2O	Promochem	pos	12.1	40	285	193/154	55	37/10
	Diazepam d5	65854-76-4	C16H13ClN2O	Promochem	pos	12.1	40	290	154	55	37
	Primidone	125-33-7	C12H14N2O2	Sigma Aldrich	pos	7.0	40	219.0	162.0/91.0	40	16/39
	Primidone d5	73738-06-4	C12H9D5N2O2	TRC	pos	7.0	40	224	167.1	40	17
	Carbamazepine (CBZ)	298-46-4	C15H12N2O	Sigma Aldrich	pos	9.5	40	237.1	194.0/179.1	71	27/49
	Carbamazepine (CBZ) 15N13C	1173022-00-8	13CC14H1215N2O	Campro Scientific	pos	9.5	40	239	192	61	29
	Amisulpride	71675-85-9	C17H27N3O4S	TRC	pos	5.8	40	370.2	424.0/196.0	106	39/59
	Amisulpride d5	1216626-17-3	C17H22D5N3O4S	TRC	pos	5.8	40	375.2	242	106	39
	Oxazepam	604-75-1	C15H11ClN2O2	Sigma Aldrich	pos	10.0	40	287.1	241.0/104.0	61	47/81
	Oxazepam d5	65854-78-6	C15H6D5ClN2O2	Sigma Aldrich	pos	10.1	40	292.1	246	81	47
	Citalopram	59729-32-7	C20H21FN2O	Labmix24	pos	8.0	40	325.2	109.1/262.1	85	37/27
	Citalopram d4	1219908-84-5	$C_{20}H_{17}D_4FN_2O$	TRC	pos	8.0	40	331.2	109.1	85	37
Cardiovascular System	Sotalol	959-24-0	C12H20N2O3S	Dr. Ehrenstorfer	pos	5.1	40	273	213.0/134.0	46	26/37
	Sotalol d6	59729-32-7	C12H14D6N2O3S	LabMix24	pos	5.1	40	279	214	46	25
	Metoprolol	56392-17-7	C15H25NO3	Sigma Aldrich	pos	6.3	40	268	74.0/116.0	75	35/27
	Metoprolol d7	1219798-61-4	C15H18D7NO3	Campro Scientific	pos	6.3	40	275	123	80	27
	Aliksiren	173334-57-1	C30H53N3O6	TRC	pos	8.4	40	552.4	436.3/534.4	70	28/28
	Aliksiren d6	1246815-96-2	C30H47D6N3O6	TRC	pos	8.4	40	558.4	436.3	60	31
Antiinfectives for systemic use	Climbazol	38083-17-9	C15H17ClN2O2	Dr. Ehrenstorfer	pos	10.1	40	293	197.0/69.0	50	23/37
	Climbazol d4	1185117-79-6	C15H13D4ClN2O2	TRC	pos	10.1	40	297	201	50	23
	Sulfamethoxazole	723-46-6	C10H11N3O3S	Sigma Aldrich	pos	7.9	40	254.1	156.0/188.0	66	23/21
	Sulfamethoxazole d4	1020719-86-1	C10H7D4N3O3S	LGC	pos	7.9	40	258	160	66	23
	Clarithromycin	81103-11-9	C38H69NO13	Abbott	pos	8.9	40	748.5	590.4/158.1	86	27/39
	Clarithromycin-N-methyl d3	NA	C38H66D3NO13	TRC	pos	8.9	40	751.5	161.2	70	40
	Azithromycin	83905-01-5	C38H72N2O12	Sigma Aldrich	pos	8.9	40	749.5	291.4/158.1	100	40/55
	Azithromycin d3	163921-65-1	C38H69D3N2O12	LGC	pos	8.9	40	752.5	594.4	100	41

	Substance	CAS	Formula	Supplier	ESI	RT	DW	Q1	Q3a/Q3b	DP	CEa/CEb
	Fluconazole	86386-73-4	C13H12F2N6O	TRC	pos	7.1	40	307.1	238.1/220.1	70	20/25
	Fluconazole d4	1124197-58-5	C13H8D4F2N6O	TRC	pos	7.1	40	311.1	223.1	70	25
	Mebendazole	31431-39-7	C16H13N3O3	Riedel-de Haen	pos	9.7	40	296.3	264.1/77	100	70/30
	Mebendazole d3	1173021-87-8	C16H10D3N3O3	Fluka	pos	9.7	40	299.3	264.1	100	30
Alimentary Tract and Metabolism / Blood and Blood forming organs / Antineoplastic / Respiratory system	Sitagliptin	654671-78-0	C16H15F6N5O	TRC	pos	6.8	40	408.1	235.1/174.0	51	29/33
	Sitagliptin d4	1432063-88-1	C16H17F6N5O5P	TRC	pos	6.8	40	412.1	239.1	26	27
	Clopidogrel	120202-66-6	C16H16ClNO2S	TRC	pos	14.9	40	322.1	212.0/184.0	31	23/31
	Clopidogrel d4	1219274-96-0	C16H12D4ClNO2S	TRC	pos	14.9	40	326.1	216.1	31	23
	Diphenhydramine	147-24-0	C17H21NO	TRC	pos	7.9	40	256.2	167.0/152.0	30	20/50
	Diphenhydramine d4	1219795-16-0	C17H17D4NO	TRC	pos	7.9	40	262.2	152	30	55
Musculo-Skeletal System	Diclofenac (DCF)	15307-79-6	C14H10Cl2NO2	Sigma Aldrich	pos	12.9	40	296	215.0/250.0	46	27/19
	Diclofenac d4	153466-65-0	C14H7D4Cl2NO2	Dr. Ehrenstorfer	pos	12.9	40	300	219	46	27
	Naproxen	22204-53-1	C14H14O3	Sigma Aldrich	neg	11.5	40	229.1	170.0/185.0	-50	-22/-11
	Naproxen d3	958293-77-1	C14H11D3O3	Dr. Ehrenstorfer	neg	11.5	40	232	173	-30	-20
	Fenoxycarb	72490-01-8	C17H19NO4	Fluka	pos	13.8	40	302.3	116.1/88.1	85	30/16
	Fenoxycarb 13C6	NA	C1113C6H19NO4	AlsaChim	pos	13.8	40	308.3	122.1	85	16
	Epoxiconazole	133855-98-8	C17H13ClFN3O	Sigma Aldrich	pos	13.0	40	330.1	121.0/75.0	70	35/95
	Epoxiconazol d4	NA	$C_{17}H_9D_4ClFN_3O$	TRC	pos	13.0	40	334.1	125.3	60	35
Fungizides/Herbicides/Insectizides	Propiconazole	60207-90-1	C15H17Cl2N3O2	Dr. Ehrenstorfer	pos	14.1	40	342.1/344.1*	159.0/161.0*	76	45/37
	Propiconazole d5	1246818-14-3	C152H5H12Cl2N3O2	LGC	pos	14.1	40	347.2	159.1	80	34
	Tebuconazole	107534-96-3	C16H22ClN3O	Dr. Ehrenstorfer	pos	13.2	40	308.1	70.0/125.0	81	49/45
	Tebuconazole d6	NA	C162H6H16ClN3O	Dr. Ehrenstorfer	pos	13.2	40	314.3	72.1	84	59
	DEET	134-62-3	C12H17NO	Sigma Aldrich	pos	10.9	40	192.1	119.1/91.1	51	25/43
	DEET d7	1219799-37-7	C12H10D7NO	Sigma Aldrich	pos	10.9	40	199.1	126.1	86	24
	Fenpropimorph	67564-91-4	C20H33NO	Sigma Aldrich	pos	9.7	40	304.3	147.2/117.1	81	77/41
	Diuron	330-54-1	C9H10Cl2N2O	Dr. Ehrenstorfer	neg	10.9	40	231.0/233.0*	186.0/186.0	-60	-25/-25
	Diuron d6	1007536-67-5	C9H4D6Cl2N2O	Dr. Ehrenstorfer	neg	10.9	40	237	186	-70	-25
	Imidacloprid	138261-41-3	C9H10ClN5O2	Sigma Aldrich	pos	7.7	40	256.1	209.0/175.1	60	25/30
	Imidacloprid d4	1015855-75-0	C9D4H6ClN5O2	Dr. Ehrenstorfer	pos	7.7	40	260.1	179.1	80	25
	Isoproturon	34123-59-6	C12H18N2O	Dr. Ehrenstorfer	pos	10.9	40	207.0	72.0/165.1	65	35/22
	Isoproturon d6	217487-17-7	C12H12D6N2O	Sigma Aldrich	pos	10.9	40	213.2	78	65	30

	Substance	CAS	Formula	Supplier	ESI	RT	DW	Q1	Q3a/Q3b	DP	CEa/CEb
	Mecoprop	7085-19-0	C10H11ClO3	Dr. Ehrenstorfer	neg	11.7	40	215.0/213.0*	143.0/141.0*	-35	-20/-20
	Mecoprop d3	352431-15-3	C10H8D3ClO3	TRC	neg	11.7	40	216	144	-40	-25
	Metamitron	41394-05-2	C10H10N4O	Sigma Aldrich	pos	7.2	40	203.1	104.0/175.1	65	33/23
	Metamitron d5	NA	C10H5D5N4O	HPC	pos	7.2	40	208.1	180.1	60	23
	Metazachlor	67129-08-2	C14H16ClN3O	Sigma Aldrich	pos	11.5	40	278.1	210.0/134.1	35	15/30
	Metazachlor d6	1246816-51-2	C14H10D6ClN3O		pos	11.5	40	284.1	140.1	45	30
	Metolachlor	51218-45-2	C15H22ClNO2	Chem Service	pos	13.7	40	284.1	252.0/286.1	45	20/35
	Metolachlor d6	1219803-97-0	$C_{15}{}^{2}H_6H_{16}ClNO_2$	LGC	pos	13.7	40	290.1	258.1	45	20
	Terbutryn	886-50-0	C10H19N5S	Dr. Ehrenstorfer	pos	12.5	40	242.0	186.0/91.0	50	25/38
	Terbutryn d5	NA	C10D5H14N5S	Sigma Aldrich	pos	12.5	40	247.0	191.0	50	25
	Terbuthylazine	5915-41-3	C9H16ClN5	Dr. Ehrenstorfer	pos	12.3	40	230.1	174.1/104.0	61	25/45
	Terbuthylazine d5	222986-60-9	C9H11ClD5N5	Dr. Ehrenstorfer	pos	12.3	40	235.2	104.0	61	45
	Irgarol	28159-98-0	C11H19N5S	Riedel de Haen	pos	12.7	40	254.0	198.0/83.0	70	26/41
	Irgarol d9	1189926-01-9	C11H10D9N5S	Dr. Ehrensdorfer	pos	12.7	40	263.0	199.0	40	29
Industrial chemicals/Lifestyle compounds/Disinfectants/Sweetener/Bitterant	Carbanilide	102-07-8	C13H12N2O	Sigma Aldrich	neg	11.4	40	211	92	-15	-80/-5
	Methyltriphenylphosphonium bromide	1779-49-3	C19H18P	Sigma Aldrich	pos	8.1	40	277.1	183.1/108.1	100	59/51
	Methyl-d3-triphenylphosphonium bromide	1787-44-6	CD3P(C6H5)3Br	Sigma Aldrich	pos	8.1	40	280.1	183.1	100	68
	(Methoxymethyl)triphenylphosphonium chloride	4009-98-7	C20H20OP	Sigma Aldrich	pos	8.6	40	307.1	183.1/185.1	85	54/31
	Tetrabutylammonium bromide	1643-19-2	C16H36N	Sigma Aldrich	pos	9.8	40	242.3	142.0/100.0	100	34/45
	Tetra-d28-propylammonium bromide	1941-30-6	C12H28N	Sigma Aldrich	pos	6.5	40	186.2	114.1/142.1	50	34/28
	Tetra-d28-propylammonium bromide	284474-84-6	CD3CD2CD2)4N+Br -	Sigma Aldrich	pos	6.5	40	214.4	166.4	80	31
	Triclocarban	101-20-2	C13H9Cl3N2O	Sigma Aldrich	neg	15.0	40	313.0/315.0*	160.0/162.0*	-60	-20/-18
	Triclocarban d4	1219799-29-7	C13H5D4Cl3N2O	CDN isotopes	neg	15.0	40	317	160	-80	-18
	Denatonium	3734-33-6	C21H29N2O	Sigma Aldrich	pos	8.2	40	325.2	86.3/91.2	60	28/50
	Flecainide d3	127413-31-4	C17H17D3F6N2O3	SCBT	pos	8.1	40	418.2	401.2	70	35

A3.2 Results of the extraction experiments

A3.2.1 Calculated Quartiles for the generation of the box plot graphic

Table A6: After verification of the data min a Grubbs-outlier test, then data with an outlier coefficient of > 1.5 were excluded from further data evaluation

	Iso-hexane	Ehtyl acetate	Acetone	Methanol	Methanol/ Acetone (50/50)	Methanol/ Acetone (50/50) + 1% formic acid	Methanol/ Acetone/Water (50/25/25)
Minium	0%	0%	0%	27%	18%	16%	67%
First Quartile	0%	0%	9%	64%	62%	61%	85%
Median	1%	21%	39%	79%	82%	80%	94%
Third Quartile	27%	58%	76%	93%	96%	93%	98%
Maximum	67%	94%	96%	99%	100%	99%	100%

A3.2.2 Relative extraction efficiencies for each compound and extraction method

Table A7: Relative extraction efficiencies for each compound and extraction method. The spike concentration was 20ng/g per substances.

Substance		ASE_M_A_W			ASE_MeOH			ASE_Aceton			ASE_M_A			ASE_M_A_FA			ASE_Ethylacetat			ASE_Isohexan			MASE_M_A_W			Sch_NW_M_A_W			USE_M_A_W		
		Lufa 2.2	Lufa 2.3	Lufa 2.4	Lufa 2.2	Lufa 2.3	Lufa 2.4	Lufa 2.2	Lufa 2.3	Lufa 2.4	Lufa 2.2	Lufa 2.3	Lufa 2.4	Lufa 2.2	Lufa 2.3	Lufa 2.4	Lufa 2.2	Lufa 2.3	Lufa 2.4	Lufa 2.2	Lufa 2.3	Lufa 2.4	Lufa 2.2	Lufa 2.3	Lufa 2.4	Lufa 2.2	Lufa 2.3	Lufa 2.4	Lufa 2.2	Lufa 2.3	Lufa 2.4
Aliskiren	Concentration [ng/g]	13.36	11.02	9.54	3.16	1.71	0.66	0.00	0.00	0.00	2.08	2.09	2.00	2.91	1.68	1.34	0.00	0.00	0.00	0.00	0.00	0.00	9.88	10.76	11.02	9.39	8.56	4.15	10.37	8.92	5.28
	Rel. recovery	67%	55%	48%	16%	9%	3%	0%	0%	0%	10%	10%	10%	15%	8%	7%	0%	0%	0%	0%	0%	0%	49%	54%	55%	47%	43%	21%	52%	45%	26%
Amisulpride	Concentration [ng/g]	18.67	11.69	11.25	7.39	3.40	2.82	0.04	0.04	0.00	9.47	4.97	3.83	10.70	4.29	2.30	0.03	0.00	0.00	0.00	0.00	0.00	13.95	11.63	9.61	9.62	8.59	2.62	10.02	6.66	3.25
	Rel. recovery	93%	58%	56%	37%	17%	14%	0%	0%	0%	47%	25%	19%	53%	21%	11%	0%	0%	0%	0%	0%	0%	70%	58%	48%	48%	43%	13%	50%	33%	16%
Azithromycin	Concentration [ng/g]	7.54	4.82	4.63	6.02	5.08	4.68	0.38	0.00	0.63	4.99	4.10	4.23	4.46	3.37	3.05	0.00	0.00	0.00	0.00	0.00	0.00	1.73	1.45	2.49	7.04	5.53	2.51	6.57	5.53	3.03
	Rel. recovery	38%	24%	23%	30%	25%	23%	2%	0%	3%	25%	20%	21%	22%	17%	15%	0%	0%	0%	0%	0%	0%	9%	7%	12%	35%	28%	13%	33%	28%	15%
Carbanilide	Concentration [ng/g]	10.79	11.80	13.29	12.63	12.85	14.04	11.71	12.07	10.55	13.63	13.14	13.81	13.11	13.53	14.66	13.21	14.40	12.28	2.59	2.15	0.05	3.50	6.24	7.17	8.65	9.86	8.91	8.70	8.90	9.17
	Rel. recovery	54%	59%	66%	63%	64%	70%	59%	60%	53%	68%	66%	69%	66%	68%	73%	66%	72%	61%	13%	11%	0%	18%	31%	36%	43%	49%	45%	43%	45%	46%
Carbamazepine	Concentration [ng/g]	17.57	15.98	17.58	16.85	14.76	16.47	11.92	10.63	6.81	16.27	14.38	15.00	16.77	14.01	15.61	7.51	5.85	3.19	0.23	0.07	0.00	27.68	15.03	26.79	15.48	15.40	14.84	15.13	13.31	13.56
	Rel. recovery	88%	80%	88%	84%	74%	82%	60%	53%	34%	81%	72%	75%	84%	70%	78%	38%	29%	16%	1%	0%	0%	138%	75%	134%	77%	77%	74%	76%	67%	68%
Citalopram	Concentration [ng/g]	15.93	8.68	7.78	5.81	3.81	2.16	0.25	0.16	0.15	8.75	6.14	5.34	10.46	4.40	2.15	0.00	0.00	0.00	0.00	0.00	0.00	9.68	11.12	8.07	7.13	5.77	1.87	9.78	4.85	2.69
	Rel. recovery	80%	43%	39%	29%	19%	11%	1%	1%	1%	44%	31%	27%	52%	22%	11%	0%	0%	0%	0%	0%	0%	48%	56%	40%	36%	29%	9%	49%	24%	13%
Clarithromycin	Concentration [ng/g]	7.40	4.65	2.88	5.84	4.71	3.55	0.60	0.09	0.19	5.41	3.96	3.31	4.59	2.82	2.42	0.00	0.00	0.10	0.00	0.00	0.11	1.48	1.37	1.92	6.98	5.23	1.85	6.25	4.36	2.25
	Rel. recovery	37%	23%	14%	29%	24%	18%	3%	0%	1%	27%	20%	17%	23%	14%	12%	0%	0%	1%	0%	0%	1%	7%	7%	10%	35%	26%	9%	31%	22%	11%
Climbazole	Concentration [ng/g]	16.99	15.63	14.32	7.51	11.23	11.72	5.54	4.41	9.35	10.49	12.53	13.55	9.86	10.49	11.64	1.72	3.19	5.81	0.20	0.00	0.00	15.38	16.80	16.72	4.95	6.65	6.19	9.56	10.68	10.05
	Rel. recovery	85%	78%	72%	38%	56%	59%	28%	22%	47%	52%	63%	68%	49%	52%	58%	9%	16%	29%	1%	0%	0%	77%	84%	84%	25%	33%	31%	48%	53%	50%
Clopidogrel	Concentration [ng/g]	2.09	0.85	4.98	2.12	0.86	4.87	1.93	0.74	3.74	2.08	0.78	5.05	3.09	2.09	4.96	1.80	0.55	2.81	1.46	0.42	0.85	0.97	0.36	2.45	1.41	0.58	3.27	1.84	0.68	4.18
	Rel. recovery	10%	4%	25%	11%	4%	24%	10%	4%	19%	10%	4%	25%	15%	10%	25%	9%	3%	14%	7%	2%	4%	5%	2%	12%	7%	3%	16%	9%	3%	21%
DEET	Concentration [ng/g]	17.56	14.45	4.13	17.85	15.02	4.58	17.93	14.87	3.91	18.42	15.10	4.62	18.34	13.49	4.63	15.06	11.55	3.34	13.81	10.43	0.86	18.20	18.07	6.23	16.08	14.05	3.80	16.18	13.51	3.35
	Rel. recovery	88%	72%	21%	89%	75%	23%	90%	74%	20%	92%	76%	23%	92%	67%	23%	75%	58%	17%	69%	52%	4%	91%	90%	31%	80%	70%	19%	81%	68%	17%
Denatonium	Concentration [ng/g]	17.27	14.56	17.23	13.93	11.92	11.38	4.38	0.61	0.40	13.84	12.89	13.87	14.53	12.60	13.32	0.00	0.00	0.00	0.00	0.00	0.00	3.11	2.51	3.74	12.96	12.76	7.86	12.23	10.99	9.64
	Rel. recovery	86%	73%	86%	70%	60%	57%	22%	3%	2%	69%	64%	69%	73%	63%	67%	0%	0%	0%	0%	0%	0%	16%	13%	19%	65%	64%	39%	61%	55%	48%
Diazepam	Concentration [ng/g]	16.46	15.90	17.50	17.09	16.03	17.18	13.64	12.88	10.31	17.01	15.87	17.12	16.70	15.49	17.03	10.12	8.12	5.41	7.93	4.51	0.29	15.01	15.74	16.44	14.24	14.87	13.20	15.43	14.60	14.25
	Rel. recovery	82%	80%	87%	85%	80%	86%	68%	64%	52%	85%	79%	86%	83%	77%	85%	51%	41%	27%	40%	23%	1%	75%	79%	82%	71%	74%	66%	77%	73%	71%
Diclofenac	Concentration [ng/g]	1.51	0.07	0.29	1.36	0.23	0.02	0.89	0.00	0.00	1.39	0.17	0.00	1.41	0.08	0.37	0.00	0.00	0.00	0.00	0.00	0.00	1.41	0.00	0.00	1.04	0.08	0.26	1.22	0.00	0.21
	Rel. recovery	8%	0%	1%	7%	1%	0%	4%	0%	0%	7%	1%	0%	7%	0%	2%	0%	0%	0%	0%	0%	0%	7%	0%	0%	5%	0%	1%	6%	0%	1%
Diphenhydramine	Concentration [ng/g]	7.14	7.27	5.38	3.36	3.63	2.59	0.64	0.24	0.24	3.83	5.68	5.37	3.90	5.50	2.62	0.00	0.00	0.00	0.00	0.00	0.00	4.91	9.88	7.03	1.94	2.17	0.27	3.21	2.28	0.91
	Rel. recovery	36%	36%	27%	17%	18%	13%	3%	1%	1%	19%	28%	27%	19%	28%	13%	0%	0%	0%	0%	0%	0%	25%	49%	35%	10%	11%	1%	16%	11%	5%
Diuron	Concentration [ng/g]	15.38	14.04	14.44	13.45	12.31	12.62	12.04	10.37	8.34	11.87	11.00	10.61	12.70	11.47	11.09	8.83	7.10	5.68	5.65	4.23	0.34	0.00	0.00	0.01	13.35	13.69	11.73	12.61	11.29	10.87
	Rel. recovery	77%	70%	72%	67%	62%	63%	60%	52%	42%	59%	55%	53%	63%	57%	55%	44%	35%	28%	28%	21%	2%	0%	0%	0%	67%	68%	59%	63%	56%	54%
Emitricitabine	Concentration [ng/g]	0.00	4.95	0.00	0.00	3.68	0.00	0.00	0.00	0.00	0.00	1.64	0.00	0.00	0.51	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	1.90	0.00	0.00	5.52	1.58	0.00	1.08	0.00
	Rel. recovery	0%	25%	0%	0%	18%	0%	0%	0%	0%	0%	8%	0%	0%	3%	0%	0%	0%	0%	0%	0%	0%	0%	10%	0%	0%	28%	8%	0%	5%	0%
Epoxyconazole	Concentration [ng/g]	17.63	16.30	17.53	18.15	16.77	17.18	16.14	14.28	10.69	18.02	16.68	16.51	17.87	16.24	16.41	14.66	10.42	5.75	12.45	7.43	0.51	14.99	15.31	16.42	13.31	14.20	12.76	15.83	14.93	13.74
	Rel. recovery	88%	82%	88%	91%	84%	86%	81%	71%	53%	90%	83%	83%	89%	81%	82%	73%	52%	29%	62%	37%	3%	75%	77%	82%	67%	71%	64%	79%	75%	69%
Fenoxycarb	Concentration [ng/g]	2.43	2.09	1.35	2.44	2.23	1.77	2.54	2.42	1.77	2.55	2.37	1.99	2.42	2.04	1.81	2.36	2.13	1.68	2.04	1.46	0.72	1.71	1.86	1.51	1.81	1.62	1.21	2.09	1.86	1.43
	Rel. recovery	12%	10%	7%	12%	11%	9%	13%	12%	9%	13%	12%	10%	12%	10%	9%	12%	11%	8%	10%	7%	4%	9%	9%	8%	9%	8%	6%	10%	9%	7%
Fenpropimorph	Concentration [ng/g]	19.92	15.52	6.87	16.15	13.50	6.19	13.35	13.08	5.14	16.61	14.48	6.24	18.80	15.36	6.83	6.55	9.57	3.91	1.70	0.44	0.22	19.46	18.38	7.70	12.14	10.12	2.96	15.09	12.61	4.58
	Rel. recovery	100%	78%	34%	81%	68%	31%	67%	65%	26%	83%	72%	31%	94%	77%	34%	33%	48%	20%	8%	2%	1%	97%	92%	39%	61%	51%	15%	75%	63%	23%
Fluconazole	Concentration [ng/g]	17.30	14.99	17.13	12.48	11.93	13.81	2.67	0.64	3.07	5.74	3.77	9.63	7.35	6.32	11.51	0.46	0.35	0.36	0.00	0.02	0.00	10.37	15.88	13.67	15.15	15.38	15.20	4.14	3.48	3.85
	Rel. recovery	86%	75%	86%	62%	60%	69%	13%	3%	15%	29%	19%	48%	37%	32%	58%	2%	2%	2%	0%	0%	0%	52%	79%	68%	76%	77%	76%	21%	17%	19%
Imidacloprid	Concentration [ng/g]	16.78	17.02	13.68	14.95	9.07	9.20	10.95	11.02	6.63	16.58	14.77	13.13	20.26	13.80	14.37	6.69	6.23	4.79	0.00	0.00	0.00	0.00	0.00	0.00	14.75	15.99	12.60	11.02	10.27	10.86
	Rel. recovery	84%	85%	68%	75%	45%	46%	55%	55%	33%	83%	74%	66%	101%	69%	72%	33%	31%	24%	0%	0%	0%	0%	0%	0%	74%	80%	63%	55%	51%	54%
Irgarol	Concentration [ng/g]	15.92	13.02	15.55	16.63	13.96	15.64	15.73	13.73	12.80	17.24	14.44	16.64	17.30	12.54	17.09	13.36	10.54	9.98	6.83	2.45	1.00	15.38	13.52	15.37	12.98	11.75	12.53	14.87	12.66	13.39
	Rel. recovery	80%	65%	78%	83%	70%	78%	79%	69%	64%	86%	72%	83%	86%	63%	85%	67%	53%	50%	34%	12%	5%	77%	68%	77%	65%	59%	63%	74%	63%	67%
Isoproturon	Concentration [ng/g]	10.15	6.39	13.27	8.72	5.51	10.14	8.10	4.64	6.81	6.94	4.39	7.99	7.68	4.45	8.34	7.04	4.10	5.29	5.39	2.30	0.08	0.00	0.00	0.00	10.27	6.70	11.47	9.62	5.93	10.34
	Rel. recovery	51%	32%	66%	44%	28%	51%	40%	23%	34%	35%	22%	40%	38%	22%	42%	35%	20%	26%	27%	11%	0%	0%	0%	0%	51%	33%	57%	48%	30%	52%

Substance		ASE_M_A_W			ASE_MeOH			ASE_Aceton			ASE_M_A			ASE_M_A_FA			ASE_Ethylacetat			ASE_Isohexan			MASE_M_A_W			Sch_NW_M_A_W			USE_M_A_W		
		Lufa 2.2	Lufa 2.3	Lufa 2.4	Lufa 2.2	Lufa 2.3	Lufa 2.4	Lufa 2.2	Lufa 2.3	Lufa 2.4	Lufa 2.2	Lufa 2.3	Lufa 2.4	Lufa 2.2	Lufa 2.3	Lufa 2.4	Lufa 2.2	Lufa 2.3	Lufa 2.4	Lufa 2.2	Lufa 2.3	Lufa 2.4	Lufa 2.2	Lufa 2.3	Lufa 2.4	Lufa 2.2	Lufa 2.3	Lufa 2.4	Lufa 2.2	Lufa 2.3	Lufa 2.4
Mebendazole	Concentration [ng/g]	16.40	13.05	16.30	14.64	12.00	14.63	7.17	4.98	4.31	14.04	11.17	13.07	14.12	11.38	14.04	3.54	1.97	1.76	0.21	0.14	1.28	1.85	2.17	1.83	10.74	9.56	8.36	11.16	9.54	10.53
	Rel. recovery	82%	65%	81%	73%	60%	73%	36%	25%	22%	70%	56%	65%	71%	57%	70%	18%	10%	9%	1%	1%	6%	9%	11%	9%	54%	48%	42%	56%	48%	53%
Metamitron	Concentration [ng/g]	10.73	10.50	7.84	11.98	9.66	11.28	4.74	5.06	0.50	9.56	8.57	13.60	14.92	8.84	10.17	6.54	4.20	4.13	0.34	0.00	0.00	15.03	14.57	12.24	7.64	9.05	8.22	7.63	6.94	6.89
	Rel. recovery	54%	52%	39%	60%	48%	56%	24%	25%	2%	48%	43%	68%	75%	44%	51%	33%	21%	21%	2%	0%	0%	75%	73%	61%	38%	45%	41%	38%	35%	34%
Metazachlor	Concentration [ng/g]	11.89	10.32	8.30	11.44	10.73	8.39	9.72	8.25	4.93	11.46	10.07	7.74	11.32	9.77	8.00	8.19	5.73	2.77	7.63	5.36	0.46	7.82	10.22	6.93	10.46	10.57	7.58	10.74	9.43	6.94
	Rel. recovery	59%	52%	42%	57%	54%	42%	49%	41%	25%	57%	50%	39%	57%	49%	40%	41%	29%	14%	38%	27%	2%	39%	51%	35%	52%	53%	38%	54%	47%	35%
Methyltriphenyl-Phosphonium bromide	Concentration [ng/g]	10.85	12.04	10.13	14.60	9.27	2.76	2.23	0.20	0.08	14.06	12.11	6.49	12.44	8.84	4.84	0.00	0.00	0.00	8.23	7.68	8.89	0.00	0.00	0.00	10.51	9.03	2.50	9.32	7.26	2.62
	Rel. recovery	54%	60%	51%	73%	46%	14%	11%	1%	0%	70%	61%	32%	62%	44%	24%	0%	0%	0%	41%	38%	44%	0%	0%	0%	53%	45%	12%	47%	36%	13%
Methoxymethyltriphenyl-Phosphonium bromide	Concentration [ng/g]	18.81	13.56	15.15	19.50	18.34	15.41	4.34	0.49	0.21	18.54	17.28	17.56	12.82	14.65	16.74	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	11.46	9.87	1.78	12.62	10.51	4.49
	Rel. recovery	94%	68%	76%	98%	92%	77%	22%	2%	1%	93%	86%	88%	64%	73%	84%	0%	0%	0%	0%	0%	0%	0%	0%	0%	57%	49%	9%	63%	53%	22%
Metoprolol	Concentration [ng/g]	14.92	15.84	14.93	13.25	12.17	11.48	0.76	0.30	0.37	13.02	11.65	11.44	12.78	11.85	11.53	0.00	0.00	0.00	0.00	0.00	0.00	13.50	16.26	11.91	11.94	13.75	7.43	10.79	10.60	7.77
	Rel. recovery	75%	79%	75%	66%	61%	57%	4%	1%	2%	65%	58%	57%	64%	59%	58%	0%	0%	0%	0%	0%	0%	68%	81%	60%	60%	69%	37%	54%	53%	39%
Metolachlor	Concentration [ng/g]	15.90	14.33	12.82	15.82	14.50	13.36	14.24	12.81	8.93	16.10	14.60	13.90	16.17	12.29	14.13	12.08	9.11	5.83	10.69	8.01	3.03	14.66	14.38	14.14	13.90	13.35	12.31	14.48	13.22	11.76
	Rel. recovery	79%	72%	64%	79%	73%	67%	71%	64%	45%	81%	73%	69%	81%	61%	71%	60%	46%	29%	53%	40%	15%	73%	72%	71%	69%	67%	62%	72%	66%	59%
Naproxen	Concentration [ng/g]	2.65	2.07	0.31	2.17	1.25	0.00	1.29	0.60	0.00	2.23	1.30	0.00	2.60	1.41	0.31	1.53	0.81	0.00	0.17	0.00	0.00	4.05	3.09	1.00	1.89	1.74	0.00	1.95	1.41	0.00
	Rel. recovery	13%	10%	2%	11%	6%	0%	6%	3%	0%	11%	7%	0%	13%	7%	2%	8%	4%	0%	1%	0%	0%	20%	15%	5%	9%	9%	0%	10%	7%	0%
Oxazepam	Concentration [ng/g]	7.84	10.48	12.32	5.54	5.04	1.08	2.60	1.08	0.25	5.99	5.16	1.36	6.50	7.11	7.30	1.29	0.63	0.17	0.00	0.00	0.00	0.00	0.00	0.00	7.57	9.96	5.71	7.41	8.71	8.06
	Rel. recovery	39%	52%	62%	28%	25%	5%	13%	5%	1%	30%	26%	7%	32%	36%	36%	6%	3%	1%	0%	0%	0%	0%	0%	0%	38%	50%	29%	37%	44%	40%
Primidone	Concentration [ng/g]	17.07	15.22	16.99	15.80	14.41	16.26	10.72	11.51	5.52	18.97	19.92	14.53	22.92	16.05	13.88	5.71	5.66	1.81	0.24	0.00	0.00	16.74	17.60	19.29	16.45	16.45	16.54	16.02	14.53	14.50
	Rel. recovery	85%	76%	85%	79%	72%	81%	54%	58%	28%	95%	100%	73%	115%	80%	69%	29%	28%	9%	1%	0%	0%	84%	88%	96%	82%	82%	83%	80%	73%	73%
Propioconazole	Concentration [ng/g]	17.47	16.44	16.96	17.34	16.87	15.29	17.13	15.54	10.63	17.45	17.19	16.69	17.54	16.24	16.74	15.41	12.01	6.63	13.33	9.10	0.79	16.19	16.61	17.06	12.02	14.12	12.74	16.03	15.40	13.94
	Rel. recovery	87%	82%	85%	87%	84%	76%	86%	78%	53%	87%	86%	83%	88%	81%	84%	77%	60%	33%	67%	45%	4%	81%	83%	85%	60%	71%	64%	80%	77%	70%
Sitagliptin	Concentration [ng/g]	13.37	8.41	8.93	2.44	1.97	1.66	0.10	0.07	0.11	3.66	3.56	4.55	4.31	2.46	1.00	0.00	0.00	0.00	0.00	0.00	0.00	5.51	5.95	4.96	3.21	1.96	0.36	5.81	4.25	2.18
	Rel. recovery	67%	42%	45%	12%	10%	8%	1%	0%	1%	18%	18%	23%	22%	12%	5%	0%	0%	0%	0%	0%	0%	28%	30%	25%	16%	10%	2%	29%	21%	11%
Sulfamethoxazole	Concentration [ng/g]	4.96	3.21	2.57	3.08	2.08	1.00	2.78	2.53	0.67	4.57	2.93	0.87	4.39	1.96	2.45	0.00	0.00	0.00	0.00	0.00	0.00	6.27	4.76	3.52	1.90	1.15	0.60	2.49	1.01	0.52
	Rel. recovery	25%	16%	13%	15%	10%	5%	14%	13%	3%	23%	15%	4%	22%	10%	12%	0%	0%	0%	0%	0%	0%	31%	24%	18%	10%	6%	3%	12%	5%	3%
Sotalol	Concentration [ng/g]	1.96	1.71	1.67	2.77	3.33	1.98	0.07	0.00	0.00	1.84	2.08	2.00	3.87	2.24	1.68	0.00	0.00	0.00	0.00	0.00	0.00	3.50	3.62	2.35	0.00	0.00	0.00	2.50	1.50	1.00
	Rel. recovery	10%	9%	8%	14%	17%	10%	0%	0%	0%	9%	10%	10%	19%	11%	8%	0%	0%	0%	0%	0%	0%	18%	18%	12%	0%	0%	0%	13%	8%	5%
Tebuconazole	Concentration [ng/g]	15.72	15.43	12.02	16.31	14.64	12.96	13.66	13.30	9.60	15.25	14.47	14.98	17.18	12.93	13.70	13.10	10.17	6.38	8.67	5.14	0.00	14.32	14.85	14.59	12.07	14.04	11.99	13.33	13.26	12.87
	Rel. recovery	79%	77%	60%	82%	73%	65%	68%	66%	48%	76%	72%	75%	86%	65%	68%	65%	51%	32%	43%	26%	0%	72%	74%	73%	60%	70%	60%	67%	66%	64%
Terbutylazine	Concentration [ng/g]	14.82	14.49	15.00	14.46	14.11	14.09	14.55	13.45	11.43	15.23	13.99	14.75	13.82	11.75	13.34	13.76	10.78	8.66	11.21	8.60	5.52	9.16	9.35	10.77	13.09	12.98	13.04	13.41	12.47	12.27
	Rel. recovery	74%	72%	75%	72%	71%	70%	73%	67%	57%	76%	70%	74%	69%	59%	67%	69%	54%	43%	56%	43%	28%	46%	47%	54%	65%	65%	65%	67%	62%	61%
Terbutryn	Concentration [ng/g]	17.17	14.63	14.88	17.10	15.50	15.40	15.71	14.94	13.00	17.33	15.85	15.77	17.20	14.87	15.78	13.54	11.44	9.66	4.26	1.73	0.79	16.17	15.49	15.20	14.12	13.15	12.10	15.29	14.10	12.90
	Rel. recovery	86%	73%	74%	86%	77%	77%	79%	75%	65%	87%	79%	79%	86%	74%	79%	68%	57%	48%	21%	9%	4%	81%	77%	76%	71%	66%	61%	76%	71%	64%
Tetrabutylammonium	Concentration [ng/g]	20.25	16.62	17.71	15.71	14.23	9.03	12.27	2.43	3.71	15.78	15.72	14.83	17.23	14.63	7.16	0.00	0.11	0.00	0.00	0.00	0.00	12.60	12.19	10.23	16.73	15.73	10.15	9.76	11.68	9.72
	Rel. recovery	101%	83%	89%	79%	71%	45%	61%	12%	19%	79%	79%	74%	86%	73%	36%	0%	1%	0%	0%	0%	0%	63%	61%	51%	84%	79%	51%	49%	58%	49%
Tetrapropylammonium	Concentration [ng/g]	20.22	17.11	20.89	19.04	17.37	15.95	12.32	1.31	1.48	21.32	18.12	18.66	18.88	15.76	18.89	0.00	0.00	0.00	0.00	0.00	0.00	16.66	15.47	13.30	15.36	15.28	11.53	16.33	15.75	14.26
	Rel. recovery	101%	86%	104%	95%	87%	80%	62%	7%	7%	107%	91%	93%	94%	79%	94%	0%	0%	0%	0%	0%	0%	83%	77%	67%	77%	76%	58%	82%	79%	71%
Triclocarban	Concentration [ng/g]	15.80	17.97	16.82	15.62	19.41	16.61	15.20	18.63	14.67	15.94	19.54	17.48	15.92	18.48	17.44	13.92	15.83	12.28	0.41	0.56	0.37	5.97	9.25	9.50	9.01	11.85	7.96	13.61	16.25	13.28
	Rel. recovery	79%	90%	84%	78%	97%	83%	76%	93%	73%	80%	98%	87%	80%	92%	87%	70%	79%	61%	2%	3%	2%	30%	46%	48%	45%	59%	40%	68%	81%	66%

A4 OECD 307 transformation experiments

A4.1 Material and methods

A4.1.1 Instruments

The following instruments were used for the OECD 307 study.

A4.1.1.1 Transformation test

Table A8: Used instruments for the transformation test.

Instrument	Producer	Type/model
Vacuum pump 1	KNF	N920AP.29.18
Vacuum pump 2	KNF	N920AP.29.19
Vacuum pump 3	KNF	N920KT.29.18G
Overhead shaker	GFL	3040
Hand mixer	Bosch	CNHR22

A4.1.1.2 Radio HPLC

Table A9: Used parts of the HPLC-system.

Instrument	Producer	Type/model
HPLC solvent bottle distributor	Shimazu	Reservoir Tray
HPLC controller	Shimazu	CBM-20A
HPLC pump A	Shimazu	LC-10ADVP
HPLC pump B	Shimazu	LC-10ADVP
HPLC UV-detector	Shimazu	SPD-10AVP
HPLC injector	Shimazu	SIL-10ADVP
HPLC degaser	Shimazu	DGV-14A
HPLC columnoven	Shimazu	CT0-10ASVP
Radio-detector	Perkin Elmer	Radiomatic 610 TR

A4.1.1.3 HCl hydrolysis

Table A10: Components used for the HCl-hydrolysis.

Instrument	Producer	Type/model
HCl-cooler	Huber	Minichiller
Heating mantle 1	Schwabe	EMP200P
Heating mantle 2	Winkler	Fi-L

A4.1.1.4 Other instruments

Table A11: Additional used instruments for the OECD 307.

Instrument	Producer	Type/model
Oxidizer	Perkin Elmer	Sample Oxidizer Model 307
Lyophilization	Christ	Alpha 2-4 LSC
PLE	Büchi Switzerland	Speed Extractor E-914
LSC	Perkin Elmer	Tri-Carb 2800 TR
Analytical balance 1	Mettler Toledo	AT200
Analytical balance 2	Kern	770
micro scales	Sartorius	Quintix 2102-1S
Horizontal shaker	Edmund Bühler	KL-2
Drying cabinet	Memmert	UL 40
Ultrasonic bath	Bandelin	RK 103 H

A4.1.2 Chemicals

All used chemicals for the OECD 307 experiment.

A4.1.2.1 Transformation test

Table A12: Used chemicals for the transformation test.

Chemical	Producer	Other	Labeling	Specific activity	Total activity
14C labelled TCS	Hartmann analytic		(U)-Ring	61.0 mCi/mmol	9,25 MBq
14C labelled ACT	Hartmann analytic		(U)-Ring	50 - 60 mCi/mmol	9,25 MBq
14C labelled FEC	Hartmann analytic		(U)-Ring	30 - 60 mCi/mmol	10,00 MBq
TCS	Sigma Aldrich		-	-	-
ACT	Merck		-	-	-
FEC	Fluka Analytical		-	-	-
Sodium hydroxide	Carl Roth	Ph.Eur., USP, BP	-	-	-
Paraffin	Carl Roth		-	-	-
Thiazole yellow G	Fluka Analytical	Indicator	-	-	-

A4.1.2.2 Oxidizer

Table A13: Chemicals used for the combustion with the oxidizer.

Chemical	Producer	Other
Combust Aid	Perkin Elmer	Combustion reagent
Carbo-Sorb® E	Perkin Elmer	Sample oxidizer cocktail
Permafluor® E+	Perkin Elmer	pseudocumene-based cocktail

A4.1.2.3 PLE

Table A14: Used chemicals for the PLE extraction.

Chemical	Producer	Other
Methanol	Merck	EMPLURA®
Deionized and purified H_20		
Acetone	Merck	EMPLURA®

A4.1.2.4 HCl hydrolysis

Table A15: Chemicals used for the HCl-hydrolysis.

Chemical	Producer	Other
Hydrochloric acid		
ULTIMA GOLD LLT	Perkin Elmer	

A4.1.2.5 Other chemicals

Table A16: Additional used chemicals for the OECD 307 experiment.

Chemical	Producer	Other
Ethanol	Merck	absolute EMPLURA®
Heptane	Roth	
Calcium chloride	Merck	Salt for 3SBE
Ethylenediaminetetraacetic acid	Roth	
Ultima Gold	Perkin Elmer	liquid scintillation cocktail
Ultima Gold XR	Perkin Elmer	liquid scintillation cocktail
Hionic-Fluor	Perkin Elmer	cocktail for samples of high ionic strength
FLO-SCINT III	Perkin Elmer	liquid scintillation cocktail
Trimethylchlorosilane	VWR	for gaschromatography, Silylation reagent

A4.1.3 Material

The following materials were used for the OECD 307 study.

A4.1.3.1 Transformation test

Table A17: Materials used for the OECD 307 experiment.

Material	Producer	Other
Bosch hand mixer	Bosch	MFQ4835DE, 575 W
Brown glass bottle	Carl Roth	Duran-Protect, 250ml
Bottle, with lateral approach	MAPHY – Naturwissenschaftliche Lehrmittel	250ml, GL45, GL18
Bottle-multi-port distributor	Carl Roth	GLS 80, aus PTFE
Clear glass bottle	Carl Roth	DURAN®-Protect, 250mL, 1L, 2L
Neopoint needle 0,50 x 16 mm	Ehrhardt Medizinprodukte GmbH	
Polyamide-hose	Festo	50m, diameter 4mm, PAN-4X0,75-GE
throttle check valve	Festo	
Blind stop	JK Pneumatik GmbH & Co.KG	6mm-5mm hose nozzle, IQS-standard
Reducing plug-In connector	JK Pneumatik GmbH & Co.KG	D: 6 mm, D1: 4 mm
SUPRA Special needle, 0,9 x 55 mm	Ehrhardt Medizinprodukte GmbH	
SUPRA Special needle, 1,1 x 120 mm	Ehrhardt Medizinprodukte GmbH	
SUPRA Special needle, 1,5 x 100 mm	Ehrhardt Medizinprodukte GmbH	
SUPRA Special needle, 1,5 x 40 mm	Ehrhardt Medizinprodukte GmbH	
SUPRA Special needle, 2,00 x 120 mm	Ehrhardt Medizinprodukte GmbH	
SUPRA Special needle, 2,00 x 120 mm	Ehrhardt Medizinprodukte GmbH	
SUPRA Special needle, 2,00 x 40 mm	Ehrhardt Medizinprodukte GmbH	
Woulff bottle	Carl Roth	500mL

A4.1.3.2 Oxidizer

Table A18: Materials used for combustion with the Oxidizer.

Material	Producer	Other
Combusto-cone	Perkin Elmer	rigid
Combusto-Pad	Perkin Elmer	

A4.1.3.3 PLE

Table A19: Materials used for PLE extraction.

Material	Producer	Other
Glass fiber filter bottom	Büchi	Glass fiber
Filter,top for PLE	Büchi	Cellulose
Threads bottle ND24 (EPA)	A-Z Analytik-Zubehör GmbH	Clear glass, 60 mL, 140x27,5
Metal frit	Büchi	
Sea-sand, cleaned with acid and annealed f. a.	TH. Geyer	CHEMSOLUTE®
UltraBondTM caps ND24	A-Z Analytik-Zubehör GmbH	Silicone / PTFE, cored cap (EPA-quality)
Expansion element	Büchi	40 mL E-914
Extraction cell	Büchi	40 mL E-914
Glass beads	VWR	diameter 2mm

A4.1.3.4 Radio chemical analysis

Table A20: Used materials for the LSC measurement.

Material	Producer	Other
Super Polyethylene Vial	Perkin Elmer	20 mL, with quick closure

A4.2 Results of the transformation test of triclosan

A4.2.1 Results of the transformation test of triclosan after incubation period

Table A21: Distribution of radioactivity after incubation with triclosan in Lufa 2.2.

	Time (d)	Volatile	SD	CO_2	SD	Soil	SD	Sum	SD
TCS Lufa 2.2	0	0%	0%	0%	0%	100%	4%	100%	4%
	1	0%	0%	0%	0%	100%	5%	101%	5%
	4	0%	0%	1%	0%	102%	5%	102%	5%
	7	0%	0%	1%	0%	100%	5%	101%	5%
	14	0%	0%	4%	0%	99%	5%	103%	5%
	20	0%	0%	3%	0%	103%	5%	106%	6%
	34	0%	0%	6%	0%	98%	4%	104%	5%
	60	0%	0%	9%	0%	90%	7%	99%	7%
	100	0%	0%	11%	0%	96%	5%	107%	5%

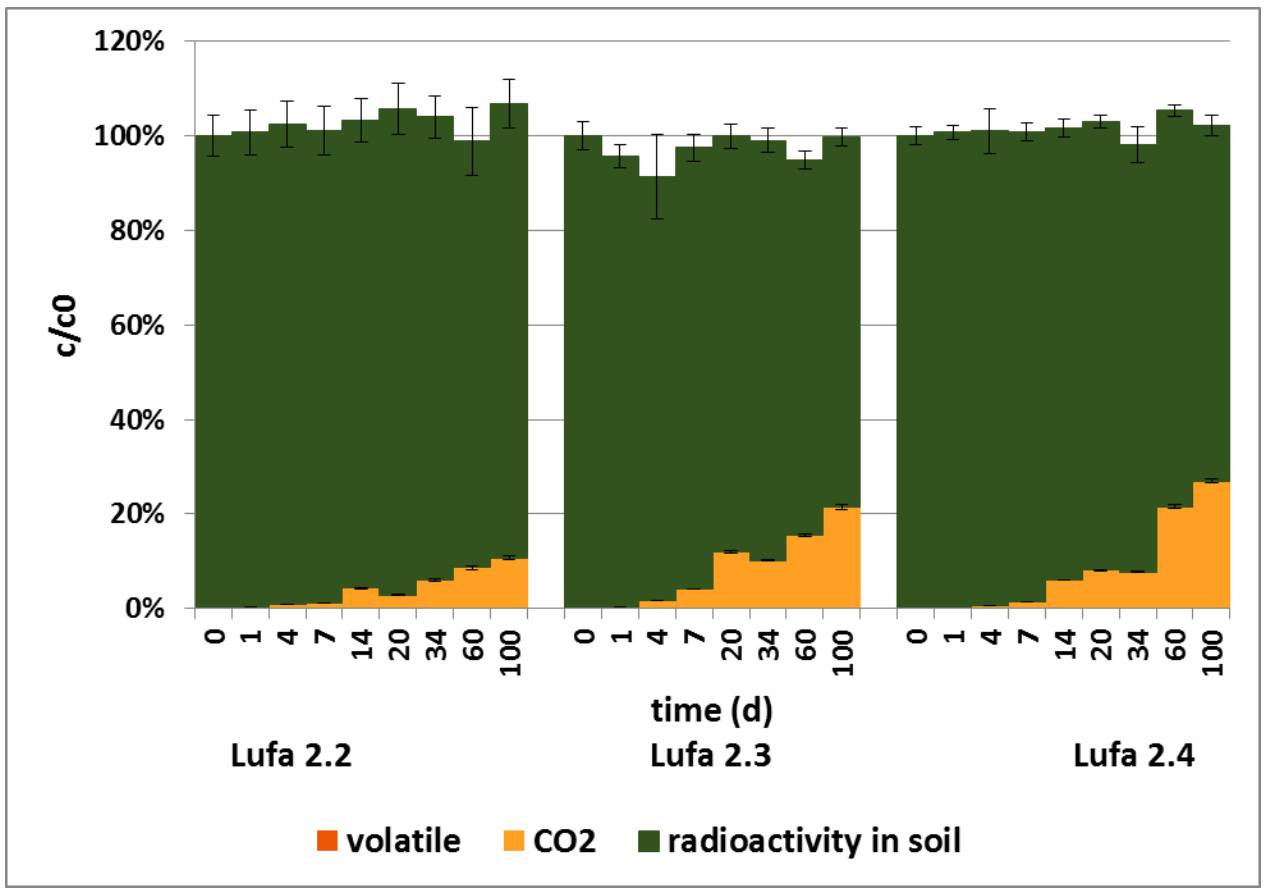
Table A22: Distribution of radioactivity after incubation with triclosan in Lufa 2.3.

	Time (d)	Volatile	SD	CO_2	SD	Soil	SD	Sum	SD
TCS Lufa 2.3	0	0%	0%	0%	0%	100%	3%	100%	3%
	1	0%	0%	0%	0%	95%	2%	96%	2%
	4	0%	0%	2%	0%	90%	9%	91%	9%
	7	0%	0%	4%	0%	93%	3%	97%	3%
	20	0%	0%	12%	0%	88%	2%	100%	3%
	34	0%	0%	10%	0%	89%	3%	99%	3%
	60	0%	0%	16%	0%	79%	2%	95%	2%
	100	0%	0%	21%	1%	78%	2%	100%	2%

Table A23: Distribution of radioactivity after incubation with triclosan in Lufa 2.4.

	Time (d)	Volatile	SD	CO_2	SD	Soil	SD	Sum	SD
TCS Lufa 2.4	0	0%	0%	0%	0%	100%	2%	100%	2%
	1	0%	0%	0%	0%	101%	1%	101%	1%
	4	0%	0%	1%	0%	100%	5%	101%	5%
	7	0%	0%	1%	0%	99%	2%	101%	2%
	14	0%	0%	6%	0%	96%	2%	102%	2%
	20	0%	0%	8%	0%	95%	1%	103%	1%
	34	0%	0%	8%	0%	90%	4%	98%	4%
	60	0%	0%	21%	0%	84%	1%	105%	1%
	100	0%	0%	27%	0%	75%	2%	102%	2%

Figure A33 Distribution of radioactivity after incubation with triclosan in Lufa 2.2, Lufa 2.3 and Lufa 2.4.



A4.2.2 Results of the transformation test of triclosan including summarised batch extraction

Table A24: Distribution of radioactivity after incubation with triclosan in Lufa 2.2 and subsequent three step batch extraction.

	Time (d)	CO_2	SD	EF	SD	NER	SD	Sum	SD
TCS Lufa 2.2	0	0%	0%	82%	4%	18%	1%	100%	5%
	1	0%	0%	82%	5%	19%	3%	101%	7%
	4	1%	0%	88%	5%	13%	1%	102%	6%
	7	1%	0%	84%	7%	16%	3%	101%	8%
	14	4%	0%	75%	4%	24%	2%	103%	5%
	20	3%	0%	81%	5%	22%	3%	106%	7%
	34	6%	0%	71%	5%	27%	5%	104%	7%
	60	9%	0%	62%	5%	28%	3%	99%	6%
	100	11%	0%	62%	3%	34%	3%	107%	6%

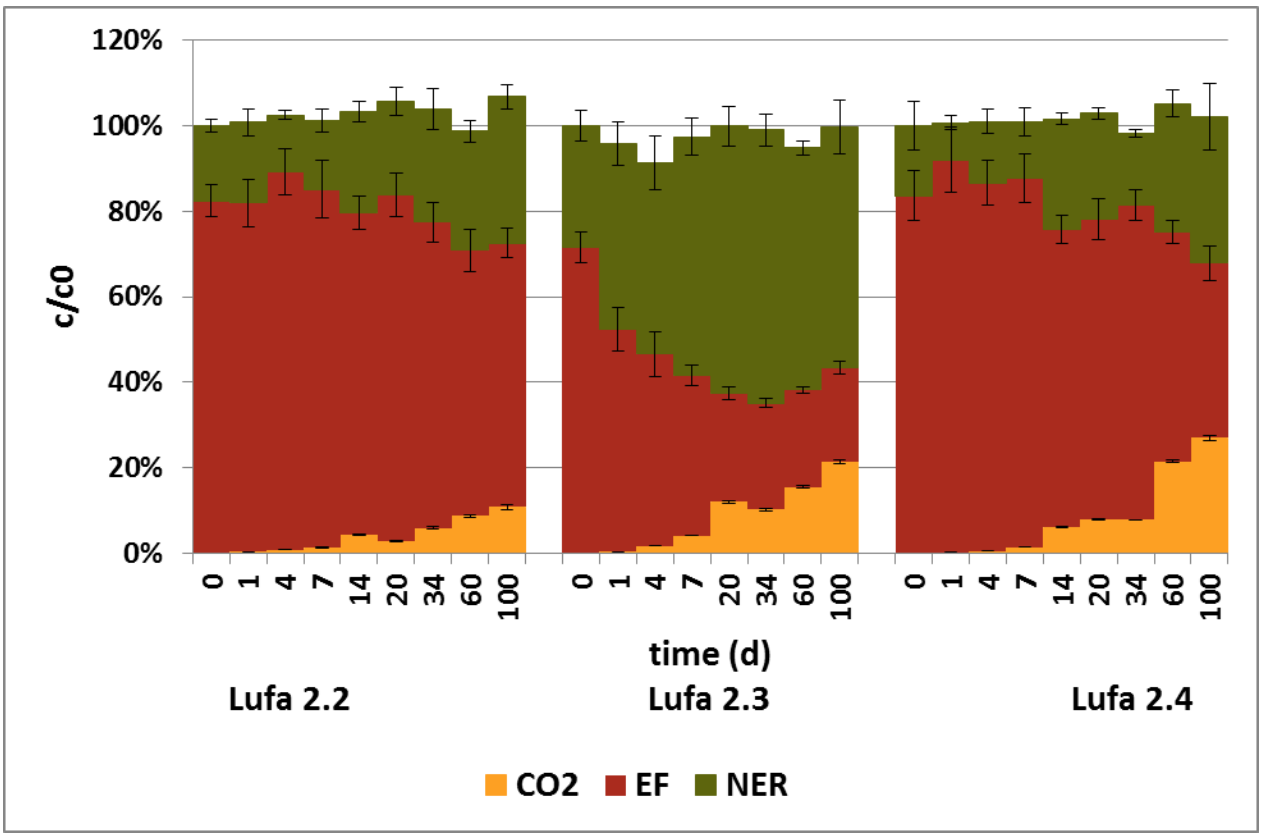
Table A25: Distribution of radioactivity after incubation with triclosan in Lufa 2.3 and subsequent three step batch extraction.

	Time (d)	CO_2	SD	EF	SD	NER	SD	Sum	SD
TCS Lufa 2.3	0	0%	0%	72%	4%	28%	4%	100%	5%
	1	0%	0%	52%	5%	43%	5%	96%	7%
	4	2%	0%	45%	5%	45%	6%	91%	8%
	7	4%	0%	37%	2%	56%	4%	97%	5%
	20	12%	0%	25%	2%	62%	5%	100%	5%
	34	10%	0%	25%	1%	64%	4%	99%	4%
	60	16%	0%	23%	1%	57%	2%	95%	2%
	100	21%	1%	22%	2%	56%	6%	100%	7%

Table A26: Distribution of radioactivity after incubation with triclosan in Lufa 2.4 and subsequent three step batch extraction.

	Time (d)	CO_2	SD	EF	SD	NER	SD	Sum	SD
TCS Lufa 2.4	0	0%	0%	84%	6%	16%	6%	100%	8%
	1	0%	0%	92%	8%	9%	2%	101%	8%
	4	1%	0%	86%	5%	14%	3%	101%	6%
	7	1%	0%	86%	6%	13%	3%	101%	7%
	14	6%	0%	70%	3%	26%	1%	102%	4%
	20	8%	0%	70%	5%	25%	1%	103%	5%
	34	8%	0%	74%	4%	17%	1%	98%	4%
	60	21%	0%	54%	3%	30%	3%	105%	4%
	100	27%	0%	41%	4%	34%	8%	102%	9%

Figure A34 Distribution of radioactivity after incubation with triclosan in Lufa 2.2, Lufa 2.3 and Lufa 2.4 and subsequent three step batch extraction.



A4.2.3 Results of the transformation test of triclosan including separated batch extraction

Table A27: Distribution of radioactivity after incubation with triclosan in Lufa 2.2 and subsequent three step batch extraction with separated fractions of the extractable fraction.

	Time (d)	CO_2	SD	EF1	SD	EF2	SD	EF3	SD	NER	SD	Sum	SD
TCS Lufa 2.2	0	0%	0%	1%	0%	9%	0%	73%	3%	18%	1%	100%	5%
	1	0%	0%	1%	0%	22%	1%	59%	4%	19%	3%	101%	7%
	4	1%	0%	1%	0%	49%	3%	38%	2%	13%	1%	102%	6%
	7	1%	0%	1%	0%	51%	4%	33%	3%	16%	3%	101%	8%
	14	4%	0%	1%	0%	19%	1%	56%	3%	24%	2%	103%	5%
	20	3%	0%	1%	0%	32%	2%	49%	3%	22%	3%	106%	7%
	34	6%	0%	0%	0%	18%	1%	53%	3%	27%	5%	104%	7%
	60	9%	0%	0%	0%	16%	1%	46%	4%	28%	3%	99%	6%
	100	11%	0%	0%	0%	7%	0%	54%	3%	34%	3%	107%	6%

Figure A35: Distribution of radioactivity after incubation with triclosan in Lufa 2.2 and subsequent three step batch extraction with separated fractions of the extractable fraction.

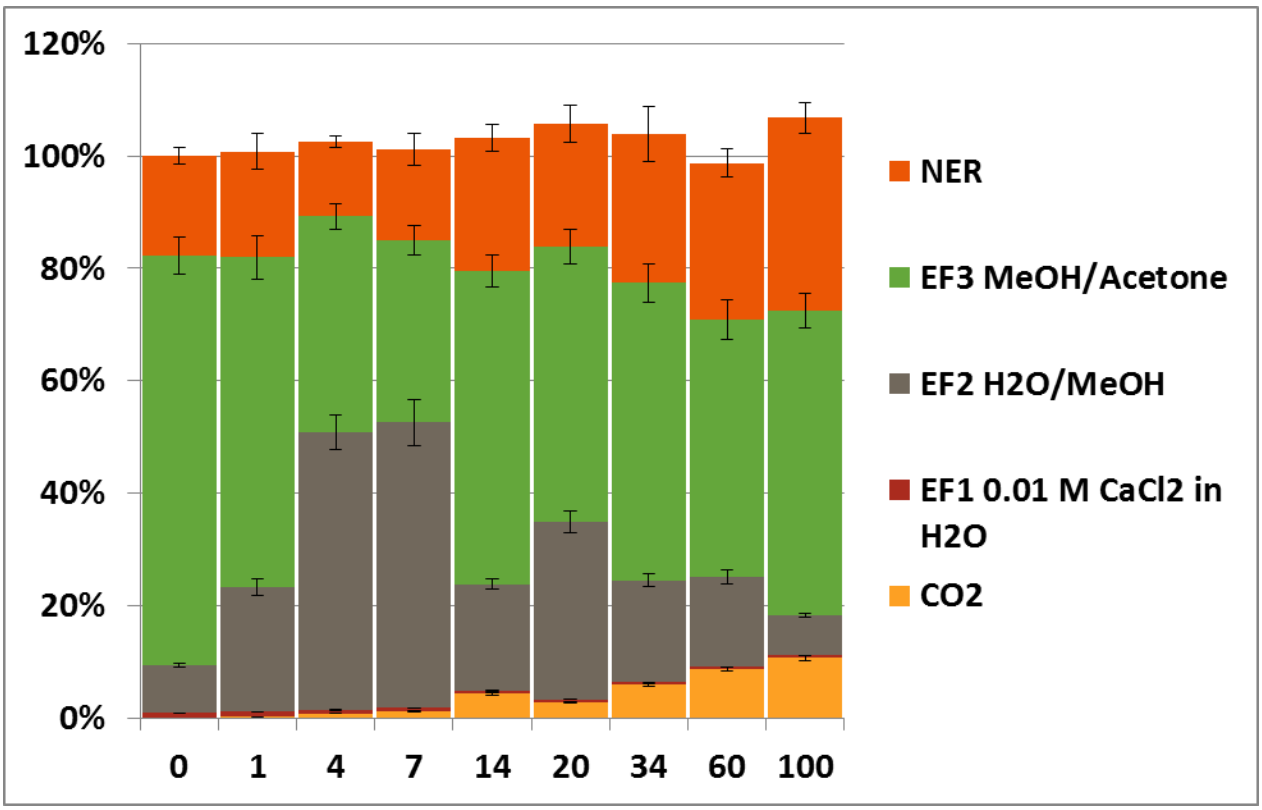


Table A28: Distribution of radioactivity after incubation with triclosan in Lufa 2.3 and subsequent three step batch extraction with separated fractions of the extractable fraction.

	Time (d)	CO_2	SD	EF1	SD	EF2	SD	EF3	SD	NER	SD	Sum	SD
TCS Lufa 2.3	0	0%	0%	2%	0%	21%	1%	48%	2%	28%	4%	100%	5%
	1	0%	0%	1%	0%	31%	3%	20%	2%	43%	5%	96%	7%
	4	2%	0%	1%	0%	32%	4%	12%	1%	45%	6%	91%	8%
	7	4%	0%	1%	0%	11%	1%	25%	2%	56%	4%	97%	5%
	20	12%	0%	1%	0%	12%	1%	13%	1%	62%	5%	100%	5%
	34	10%	0%	1%	0%	7%	0%	18%	1%	64%	4%	99%	4%
	60	16%	0%	1%	0%	7%	0%	16%	1%	57%	2%	95%	2%
	100	21%	1%	0%	0%	5%	0%	17%	1%	56%	6%	100%	7%

Figure A36: Distribution of radioactivity after incubation with triclosan in Lufa 2.3 and subsequent three step batch extraction with separated fractions of the extractable fraction.

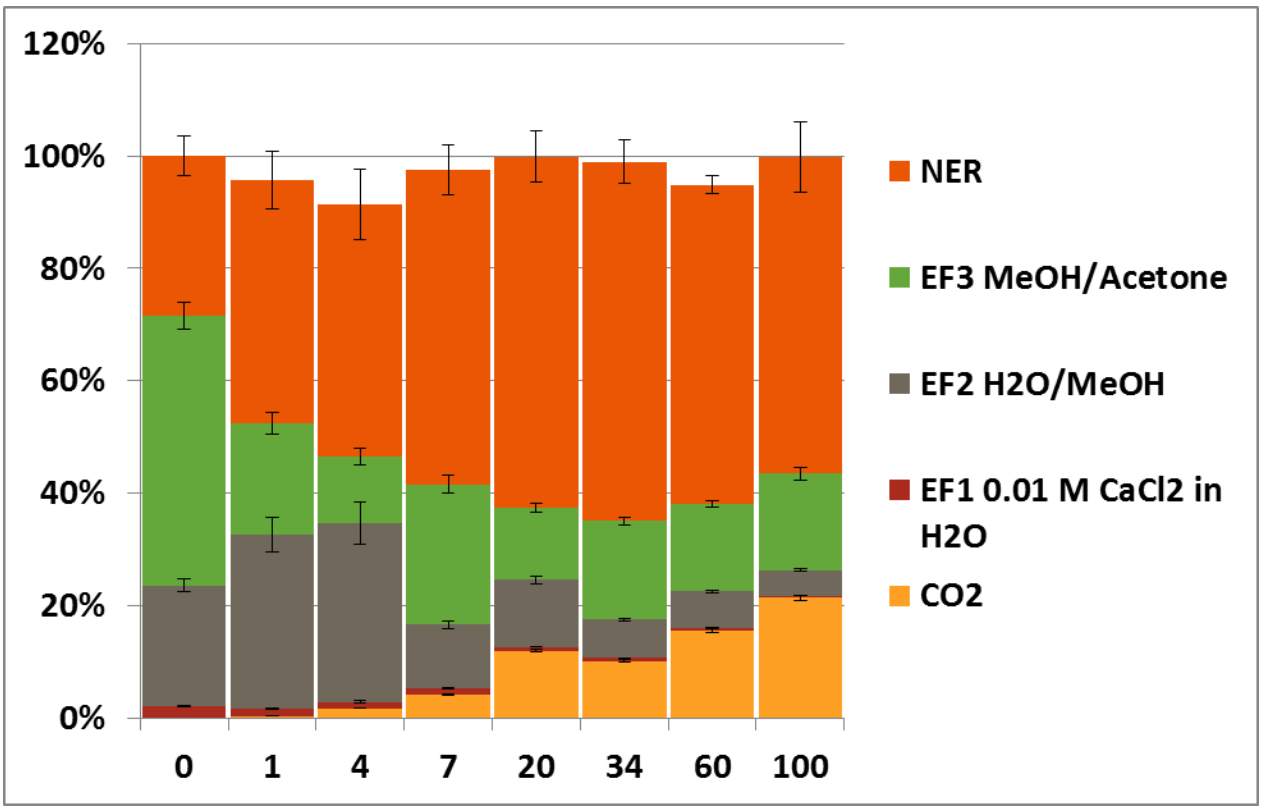
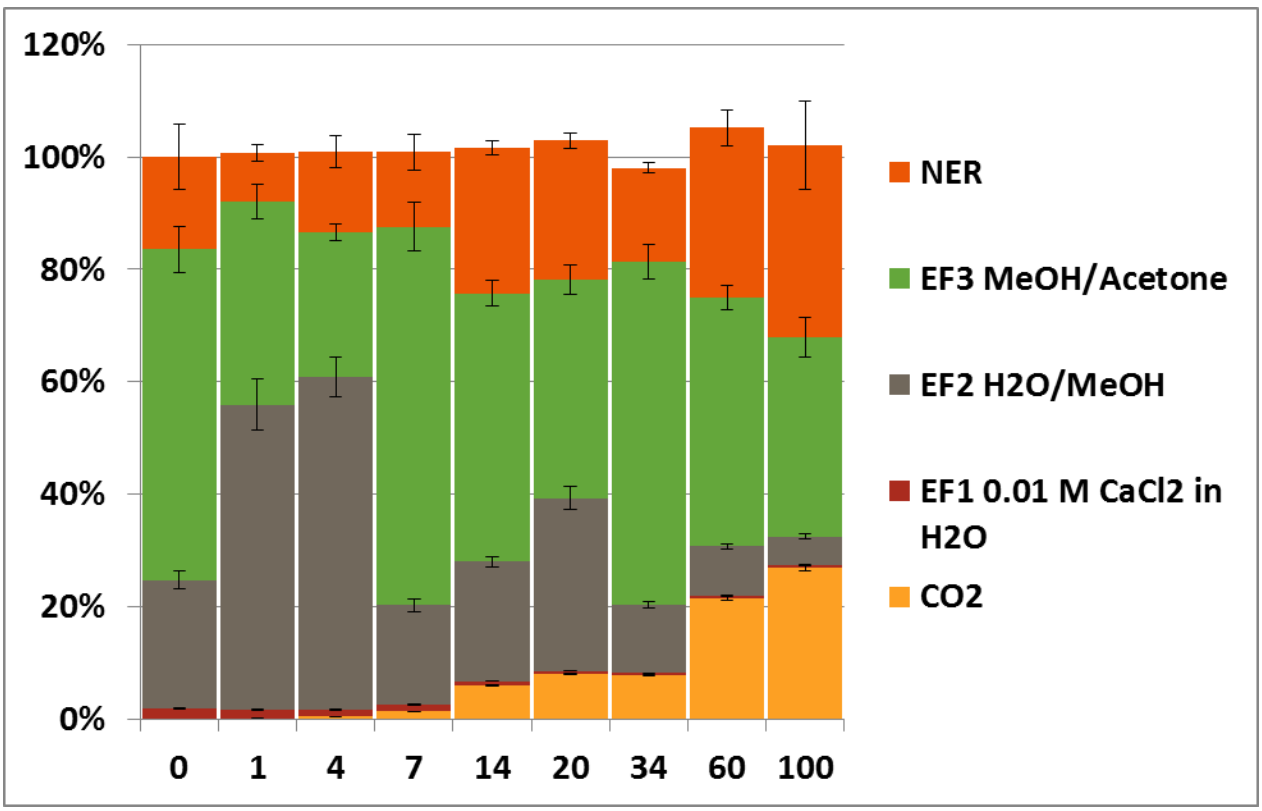


Table A29: Distribution of radioactivity after incubation with triclosan in Lufa 2.4 and subsequent three step batch extraction with separated fractions of the extractable fraction.

	Time (d)	CO_2	SD	EF1	SD	EF2	SD	EF3	SD	NER	SD	Sum	SD
TCS Lufa 2.4	0	0%	0%	2%	0%	23%	2%	59%	4%	16%	6%	100%	8%
	1	0%	0%	1%	0%	54%	5%	36%	3%	9%	2%	101%	8%
	4	1%	0%	1%	0%	59%	4%	26%	2%	14%	3%	101%	6%
	7	1%	0%	1%	0%	18%	1%	67%	4%	13%	3%	101%	7%
	14	6%	0%	1%	0%	21%	1%	48%	2%	26%	1%	102%	4%
	20	8%	0%	1%	0%	31%	2%	39%	3%	25%	1%	103%	5%
	34	8%	0%	0%	0%	12%	1%	61%	3%	17%	1%	98%	4%
	60	21%	0%	1%	0%	9%	0%	44%	2%	30%	3%	105%	4%
	100	27%	0%	0%	0%	5%	1%	35%	4%	34%	8%	102%	9%

Figure A37: Distribution of radioactivity after incubation with triclosan in Lufa 2.4 and subsequent three step batch extraction with separated fractions of the extractable fraction.



A4.2.4 Results of the transformation test of triclosan including batch extraction and subsequent Radio-HPLC analysis

Table A30: Distribution of radioactivity after the transformation test with triclosan in Lufa 2.2 following three step batch extraction and subsequent Radio-HPLC analysis.

	Time (d)	CO_2	SD	TCS	SD	MeTCS	SD	Unknown ES	SD	NER	SD	Sum	SD
TCS Lufa 2.2	0	0%	0%	82%	4%	0%	0%	0%	0%	18%	1%	100%	5%
	7	1%	0%	74%	6%	4%	0%	6%	2%	16%	3%	101%	8%
	34	6%	0%	55%	4%	12%	1%	5%	1%	27%	5%	104%	7%
	60	9%	0%	43%	5%	13%	2%	7%	1%	28%	3%	99%	6%
	100	11%	0%	28%	3%	22%	3%	12%	2%	34%	3%	107%	6%

Figure A38: Distribution of radioactivity after the transformation test with triclosan in Lufa 2.2 following three step batch extraction and subsequent Radio-HPLC analysis.

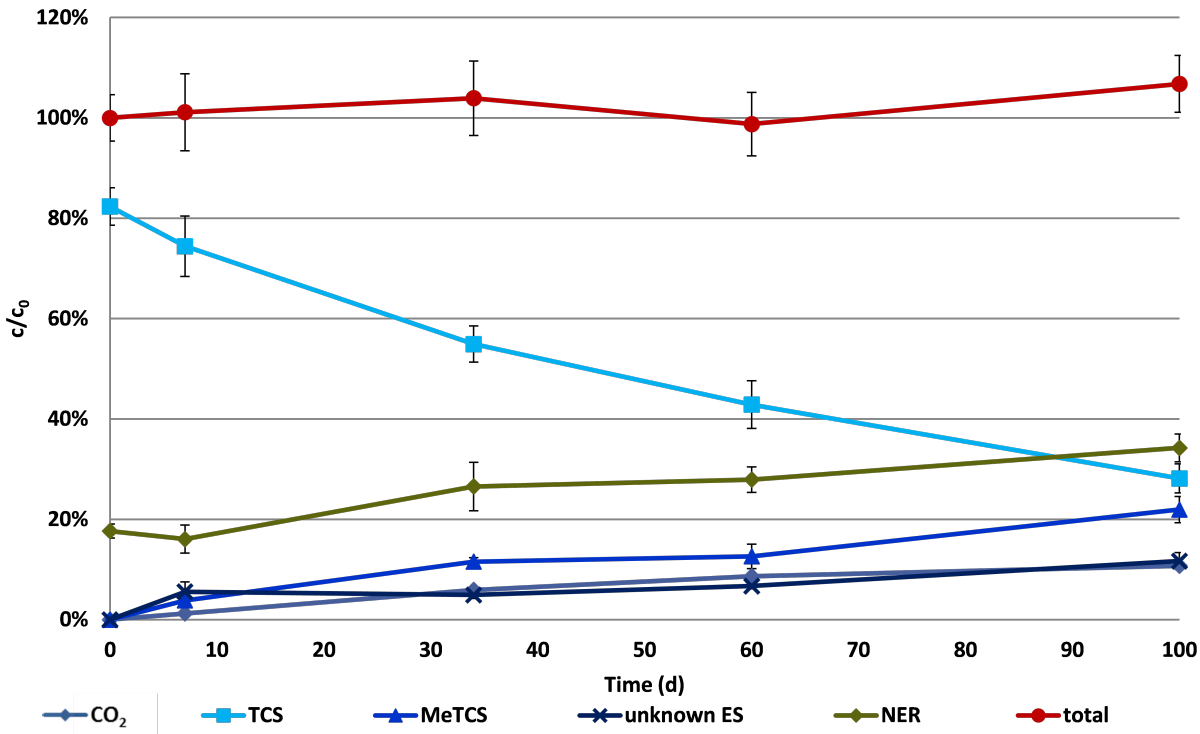


Figure A39: Radioactive decay graph after transformation test with triclosan in Lufa 2.2 following three step batch extraction and subsequent Radio-HPLC analysis.

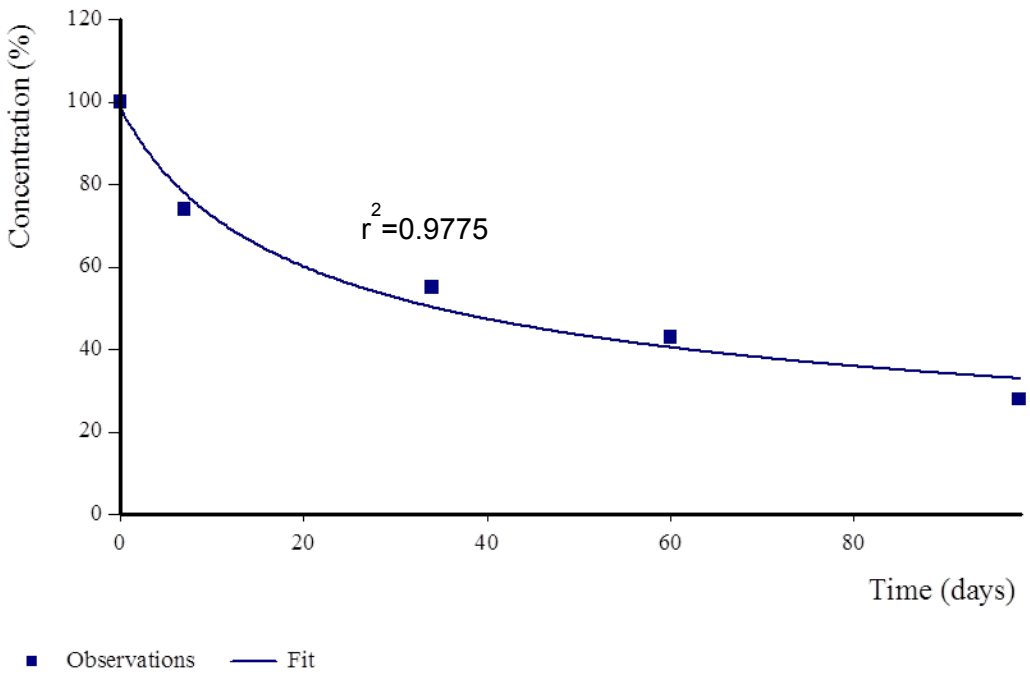


Table A31: Radioactive decay for the transformation test with triclosan in Lufa 2.2 following three step batch extraction and subsequent Radio-HPLC analysis.

	Time (days)	Value (%)	Predicted Value	Residual
TCS Lufa 2.2	0	100	98.7	1.3
	7	74	77.91	-3.91
	34	55	50.28	4.722
	60	43	40.45	2.548
	98	28	32.98	-4.98

Table A32: Distribution of radioactivity after the transformation test with triclosan in Lufa 2.3 following three step batch extraction and subsequent Radio-HPLC analysis.

	Time (d)	CO_2	SD	TCS	SD	MeTCS	SD	Unknown ES	SD	NER	SD	Sum	SD
TCS Lufa 2.3	0	0%	0%	72%	4%	0%	0%	0%	0%	28%	4%	100%	5%
	7	4%	0%	22%	5%	9%	1%	6%	5%	56%	4%	97%	5%
	34	10%	0%	7%	1%	13%	2%	5%	3%	64%	4%	99%	4%
	60	16%	0%	4%	1%	13%	2%	6%	3%	57%	2%	95%	2%
	100	21%	1%	3%	0%	16%	2%	2%	1%	56%	6%	100%	7%

Figure A40: Distribution of radioactivity after the transformation test with triclosan in Lufa 2.3 following three step batch extraction and subsequent Radio-HPLC analysis.

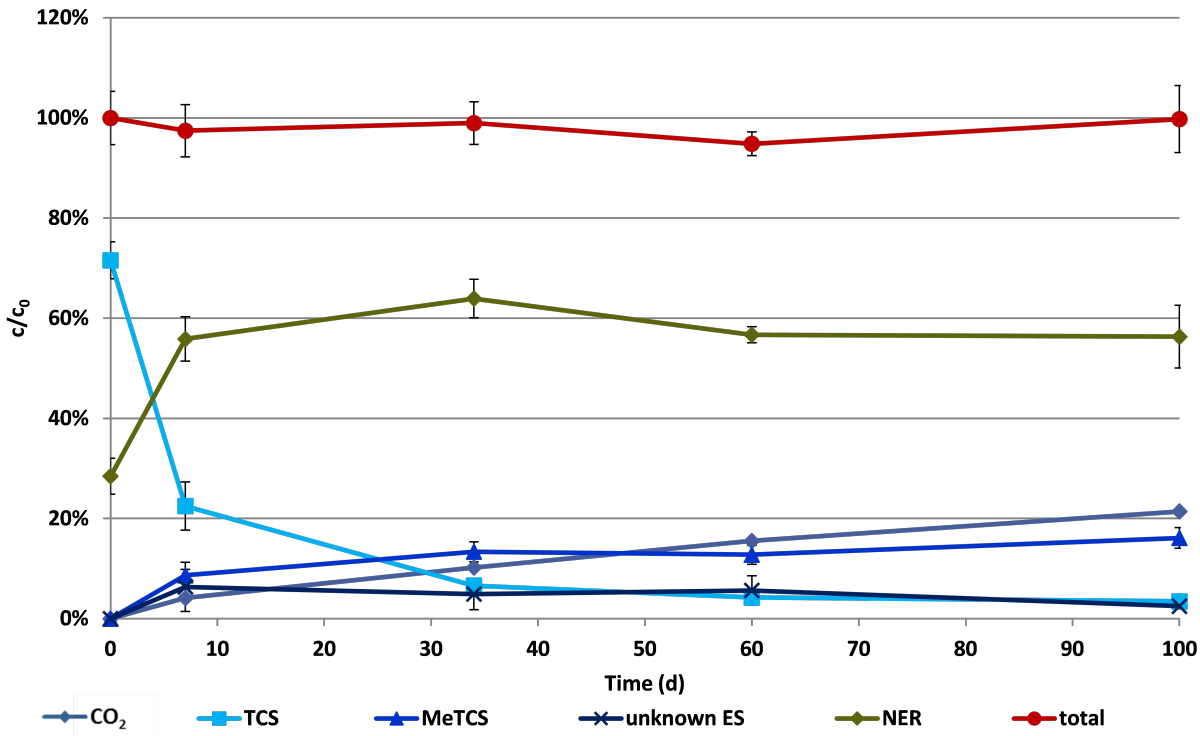


Figure A41: Radioactive decay graph after transformation test with triclosan in Lufa 2.3 following three step batch extraction and subsequent Radio-HPLC analysis.

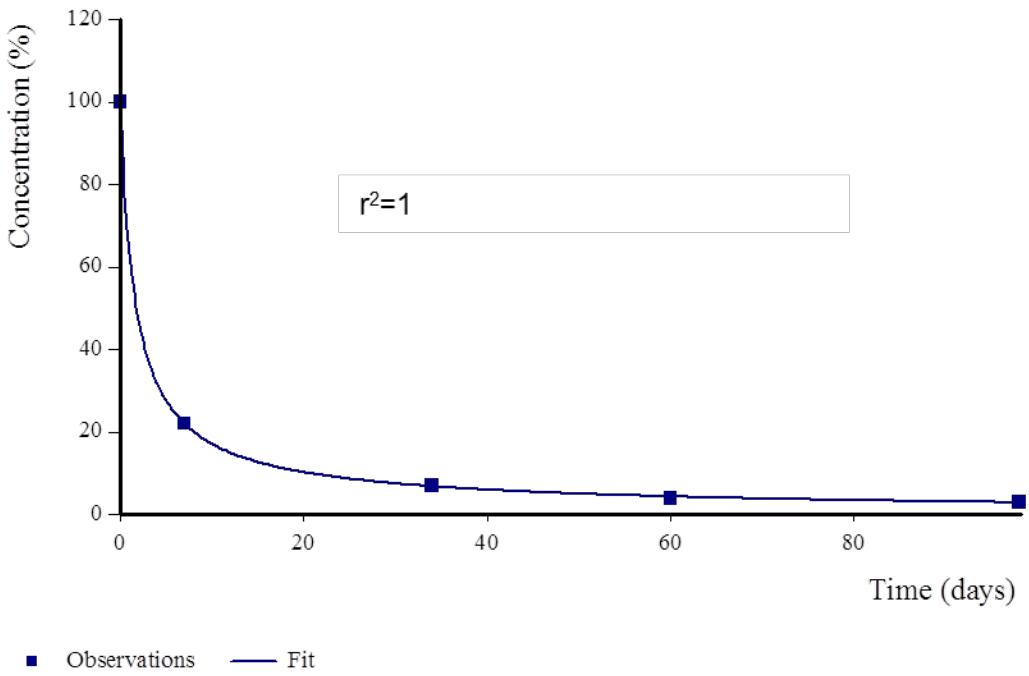


Table A33: Radioactive decay for the transformation test with triclosan in Lufa 2.3 following three step batch extraction and subsequent Radio-HPLC analysis.

	Time (days)	Value (%)	Predicted Value	Residual
TCS Lufa 2.3	0	100	100	0.0003833
	7	22	22.02	-0.02008
	34	7	6.772	0.2283
	60	4	4.32	-0.3203
	98	3	2.917	0.083

Table A34: Distribution of radioactivity after the transformation test with triclosan in Lufa 2.4 following three step batch extraction and subsequent Radio-HPLC analysis.

	Time (d)	CO_2	SD	TCS	SD	MeTCS	SD	Unknown ES	SD	NER	SD	Sum	SD
TCS Lufa 2.4	0	0%	0%	84%	6%	0%	0%	0%	0%	16%	6%	100%	8%
	7	1%	0%	32%	9%	42%	3%	12%	2%	13%	3%	101%	7%
	34	8%	0%	6%	0%	62%	9%	6%	2%	17%	1%	98%	4%
	60	21%	0%	5%	1%	43%	10%	5%	11%	30%	3%	105%	4%
	100	27%	0%	3%	1%	34%	5%	4%	0%	34%	8%	102%	9%

Figure A42: Distribution of radioactivity after the transformation test with triclosan in Lufa 2.4 following three step batch extraction and subsequent Radio-HPLC analysis.

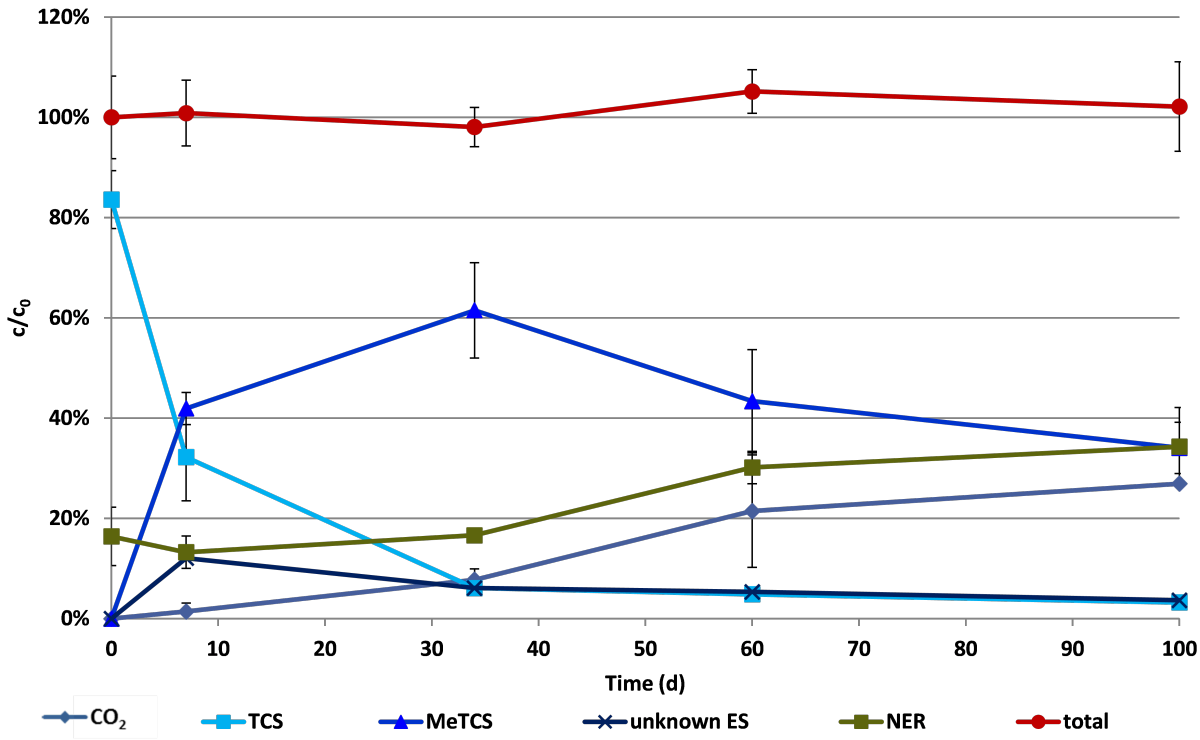


Figure A43: Radioactive decay graph after transformation test with triclosan in Lufa 2.4 following three step batch extraction and subsequent Radio-HPLC analysis.

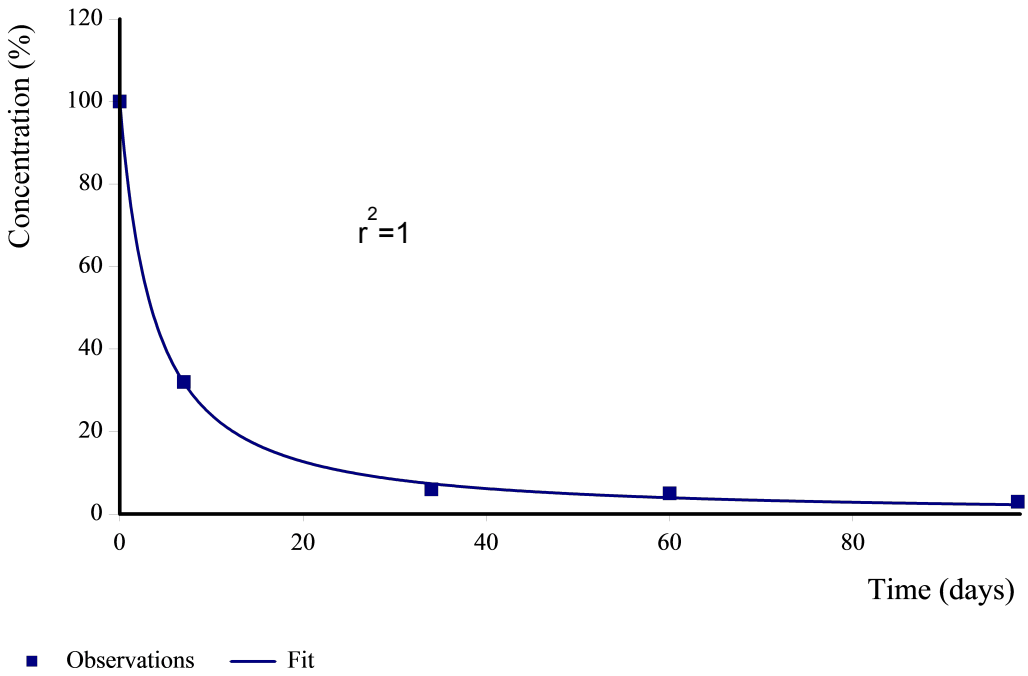


Table A35: Radioactive decay for the transformation test with triclosan in Lufa 2.4 following three step batch extraction and subsequent Radio-HPLC analysis.

	Time (days)	Value (%)	Predicted Value	Residual
TCS Lufa 2.4	0	100	100	-0.01345
	7	32	31.83	0.1737
	34	6	7.362	-1.362
	60	5	3.945	1.055
	98	3	2.255	0.7454

A4.2.5 Results of the transformation test of triclosan including separated batch extraction and PLE

Table A36: Distribution of radioactivity after the transformation test with triclosan in Lufa 2.2 following three step batch extraction with separated fractions of the extractable fraction and subsequent PLE.

	Time (d)	5	SD	EF1	SD	EF2	SD	EF3	SD	PLE-EF	SD	PLE-NER	SD	Sum	SD
TCS Lufa 2.2	7	1%	0%	1%	0%	51%	4%	33%	3%	2%	0%	14%	3%	101%	6%
	34	6%	0%	0%	0%	18%	1%	53%	3%	8%	1%	19%	4%	104%	6%
	60	9%	0%	0%	0%	16%	1%	46%	4%	7%	1%	20%	2%	99%	5%
	100	11%	0%	0%	0%	7%	0%	54%	3%	6%	1%	28%	3%	107%	6%

Figure A44: Distribution of radioactivity after the transformation test with triclosan in Lufa 2.2 following three step batch extraction with separated fractions of the extractable fraction and subsequent PLE.

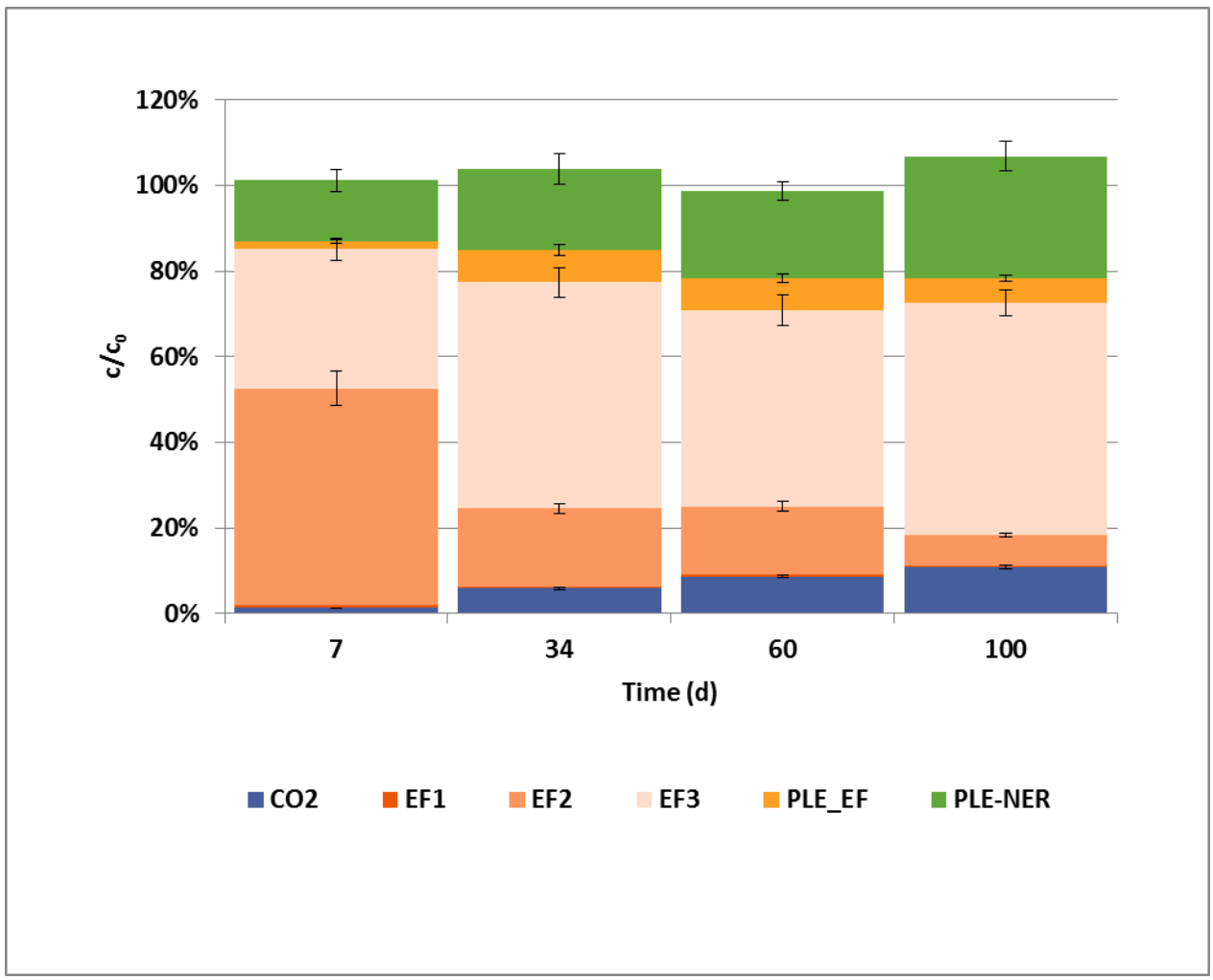


Table A37: Distribution of radioactivity after the transformation test with triclosan in Lufa 2.3 following three step batch extraction with separated fractions of the extractable fraction and subsequent PLE.

	Time (d)	CO_2	SD	EF1	SD	EF2	SD	EF3	SD	PLE-EF	SD	PLE-NER	SD	Sum	SD
TCS Lufa 2.3	7	4%	0%	1%	0%	11%	1%	25%	2%	8%	1%	47%	6%	97%	7%
	34	10%	0%	1%	0%	7%	0%	18%	1%	9%	1%	55%	5%	99%	6%
	60	16%	0%	1%	0%	7%	0%	16%	1%	7%	0%	50%	4%	95%	4%
	100	21%	1%	0%	0%	5%	0%	17%	1%	8%	1%	48%	6%	100%	6%

Figure A45: Distribution of radioactivity after the transformation test with triclosan in Lufa 2.3 following three step batch extraction with separated fractions of the extractable fraction and subsequent PLE.

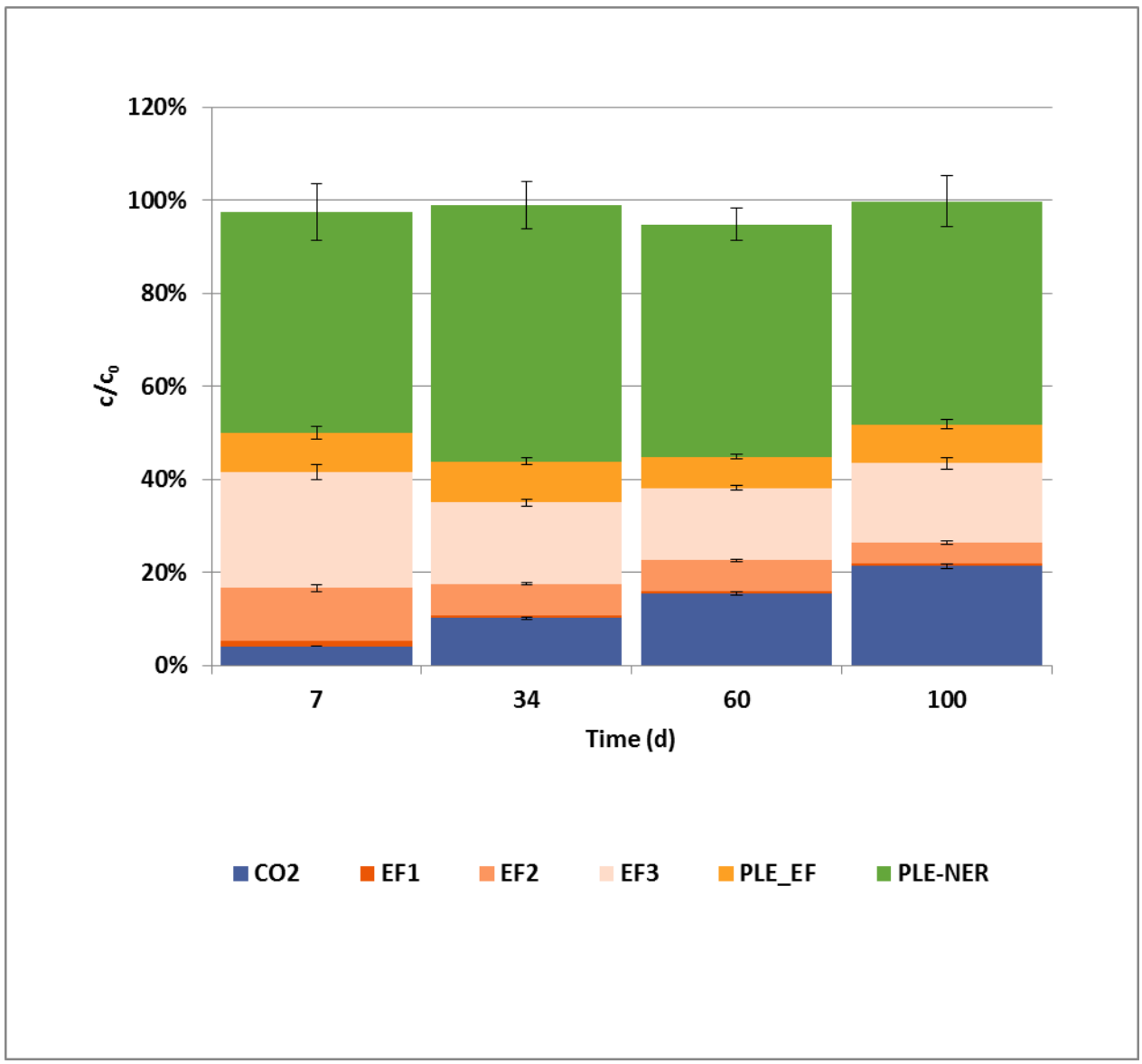
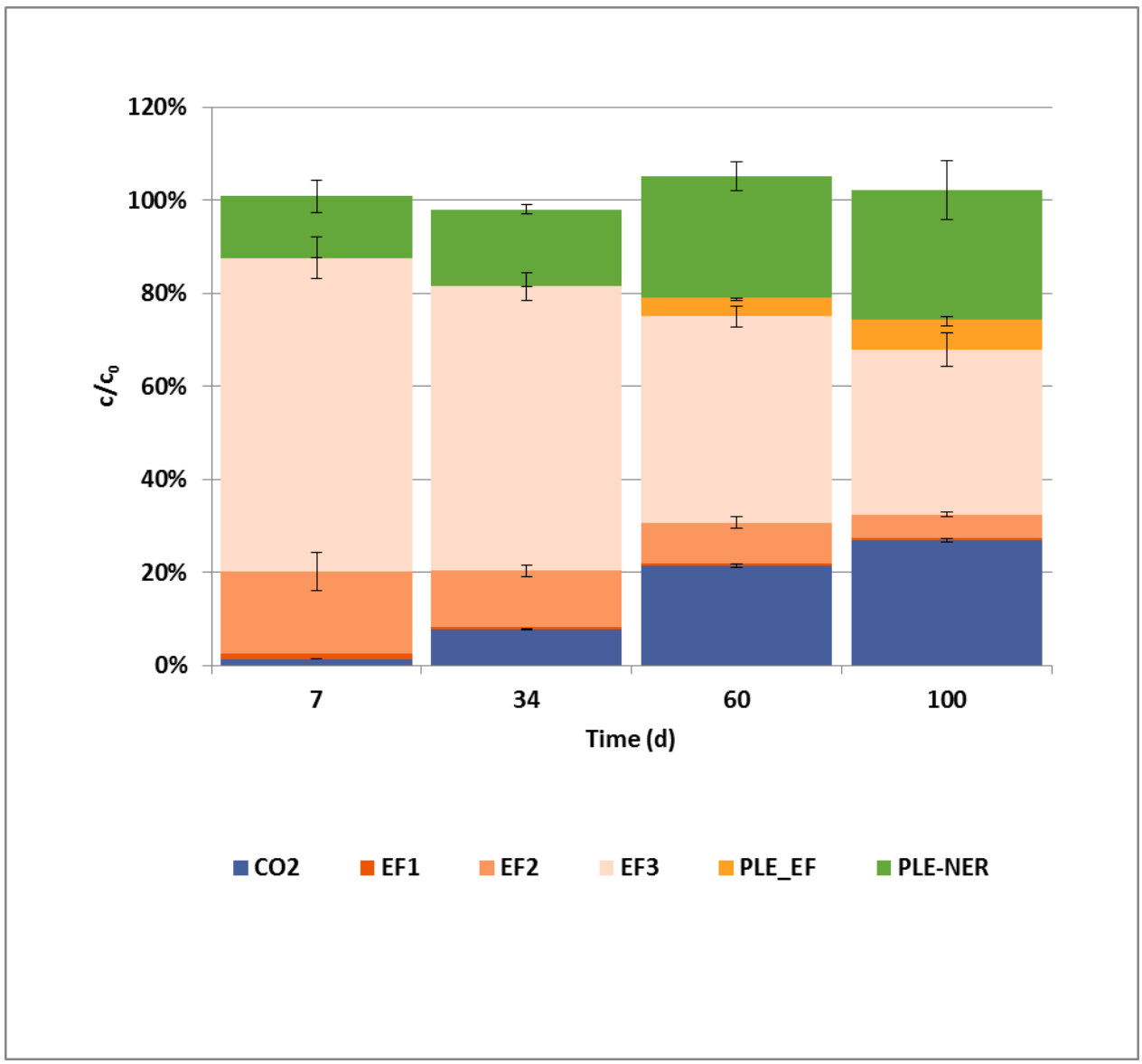


Table A38: Distribution of radioactivity after the transformation test with triclosan in Lufa 2.4 following three step batch extraction with separated fractions of the extractable fraction and subsequent PLE.

	Time (d)	CO_2	SD	EF1	SD	EF2	SD	EF3	SD	PLE-EF	SD	PLE-NER	SD	Sum	SD
TCS Lufa 2.4	7	1%	0%	1%	0%	18%	1%	67%	4%	0%	0%	13%	4%	101%	5%
	34	8%	0%	0%	0%	12%	1%	61%	3%	0%	0%	17%	1%	98%	2%
	60	21%	0%	1%	0%	9%	0%	44%	2%	4%	1%	26%	3%	105%	4%
	100	27%	0%	0%	0%	5%	1%	35%	4%	7%	2%	28%	6%	102%	7%

Figure A46: Distribution of radioactivity after the transformation test with triclosan in Lufa 2.4 following three step batch extraction with separated fractions of the extractable fraction and subsequent PLE.



A4.2.6 Results of the transformation test of triclosan including batch extraction and PLE

Table A39: Distribution of radioactivity after the transformation test with triclosan in Lufa 2.2 following three step batch extraction and subsequent PLE.

	Time (d)	CO_2	SD	EF	SD	PLE-EF	SD	PLE-NER	SD	Sum	SD
TCS Lufa 2.2	7	1%	0%	84%	7%	2%	0%	14%	3%	101%	6%
	34	6%	0%	71%	5%	8%	1%	19%	4%	104%	6%
	60	9%	0%	62%	5%	7%	1%	20%	2%	99%	5%
	100	11%	0%	62%	3%	6%	1%	28%	3%	107%	6%

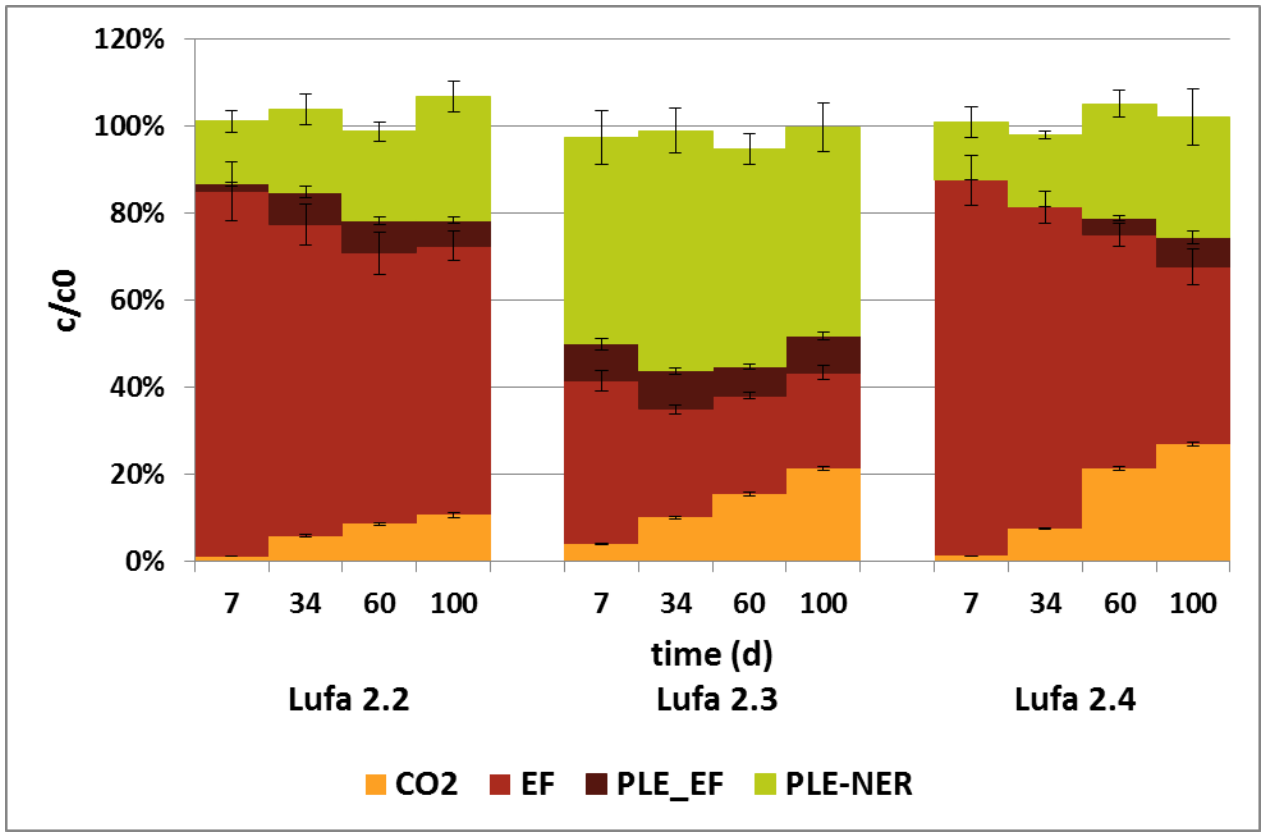
Table A40: Distribution of radioactivity after the transformation test with triclosan in Lufa 2.3 following three step batch extraction and subsequent PLE.

	Time (d)	CO_2	SD	EF	SD	PLE-EF	SD	PLE-NER	SD	Sum	SD
TCS Lufa 2.3	7	4%	0%	37%	2%	8%	1%	47%	6%	97%	7%
	34	10%	0%	25%	1%	9%	1%	55%	5%	99%	6%
	60	16%	0%	23%	1%	7%	0%	50%	4%	95%	4%
	100	21%	1%	22%	2%	8%	1%	48%	6%	100%	6%

Table A41: Distribution of radioactivity after the transformation test with triclosan in Lufa 2.4 following three step batch extraction and subsequent PLE.

	Time (d)	CO_2	SD	EF	SD	PLE-EF	SD	PLE-NER	SD	Sum	SD
TCS Lufa 2.4	7	1%	0%	86%	6%	0%	0%	13%	4%	101%	5%
	34	8%	0%	74%	4%	0%	0%	17%	1%	98%	2%
	60	21%	0%	54%	3%	4%	1%	26%	3%	105%	4%
	100	27%	0%	41%	4%	7%	2%	28%	6%	102%	7%

Figure A47: Distribution of radioactivity after the transformation test with triclosan in Lufa 2.2, Lufa 2.3 and Lufa 2.4 following three step batch extraction and subsequent PLE.

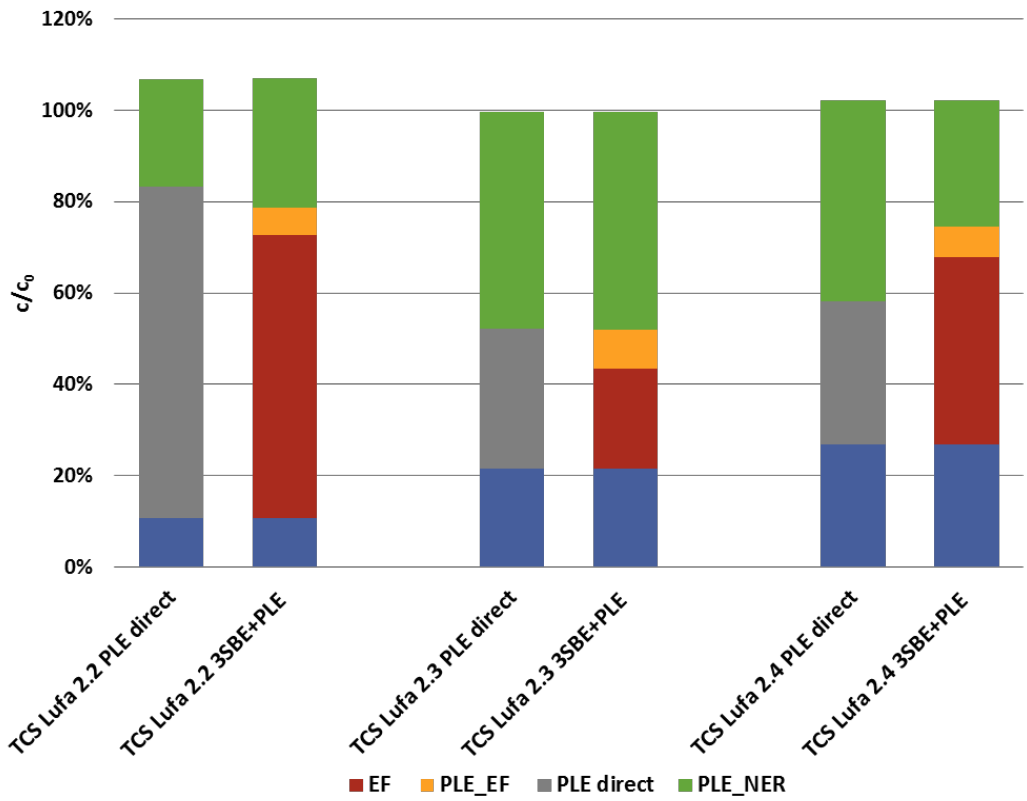


A4.2.7 Results of the transformation test of triclosan after direct PLE

Table A42: Comparison of the extraction efficiency between the three step batch extraction and the direct PLE with triclosan.

	Time (d)	CO_2	SD	3SBE-EF	SD	PLE-EF	SD	PLE-NER	SD	Sum	SD
TCS Lufa 2.2 PLE direct	100	11%	0%	-	-	73%-	4%-	23%	1%	107%	5%
TCS Lufa 2.2 3SBE+PLE	100	11%	0%	62%	3%	6%	1%	28%	3%	107%	6%
TCS Lufa 2.3 PLE direct	100	21%	1%	-	-	31%	1%	48%	2%	100%	3%
TCS Lufa 2.3 3SBE+PLE	100	21%	1%	22%	2%	8%	1%	48%	6%	100%	6%
TCS Lufa 2.4 PLE direct	100	27%	1%	-	-	31%	3%	44%	4%	102%	5%
TCS Lufa 2.4 3SBE+PLE	100	27%	1%	41%	4%	7%	2%	28%	6%	102%	7%

Figure A48: Comparison of the extraction efficiency between the three step batch extraction and the direct PLE with triclosan.



A4.3 Results of the transformation test of fenoxycarb

A4.3.1 Results of the transformation test of fenoxycarb after incubation period

Table A43: Distribution of radioactivity after incubation with fenoxycarb in Lufa 2.2.

	Time (d)	Volatile	SD	CO2	SD	Soil	SD	Sum	SD
FEC Lufa 2.2	0	0%	0%	0%	0%	100%	5%	100%	6%
	1	0%	0%	10%	3%	90%	4%	100%	5%
	4	0%	0%	24%	2%	76%	3%	100%	5%
	11	0%	0%	33%	2%	67%	3%	100%	4%
	15	0%	0%	34%	2%	66%	3%	100%	4%
	21	0%	0%	37%	1%	63%	2%	100%	4%
	35	0%	0%	44%	2%	56%	2%	100%	4%
	60	0%	0%	40%	2%	60%	3%	100%	5%
	100	0%	0%	48%	3%	52%	3%	100%	5%

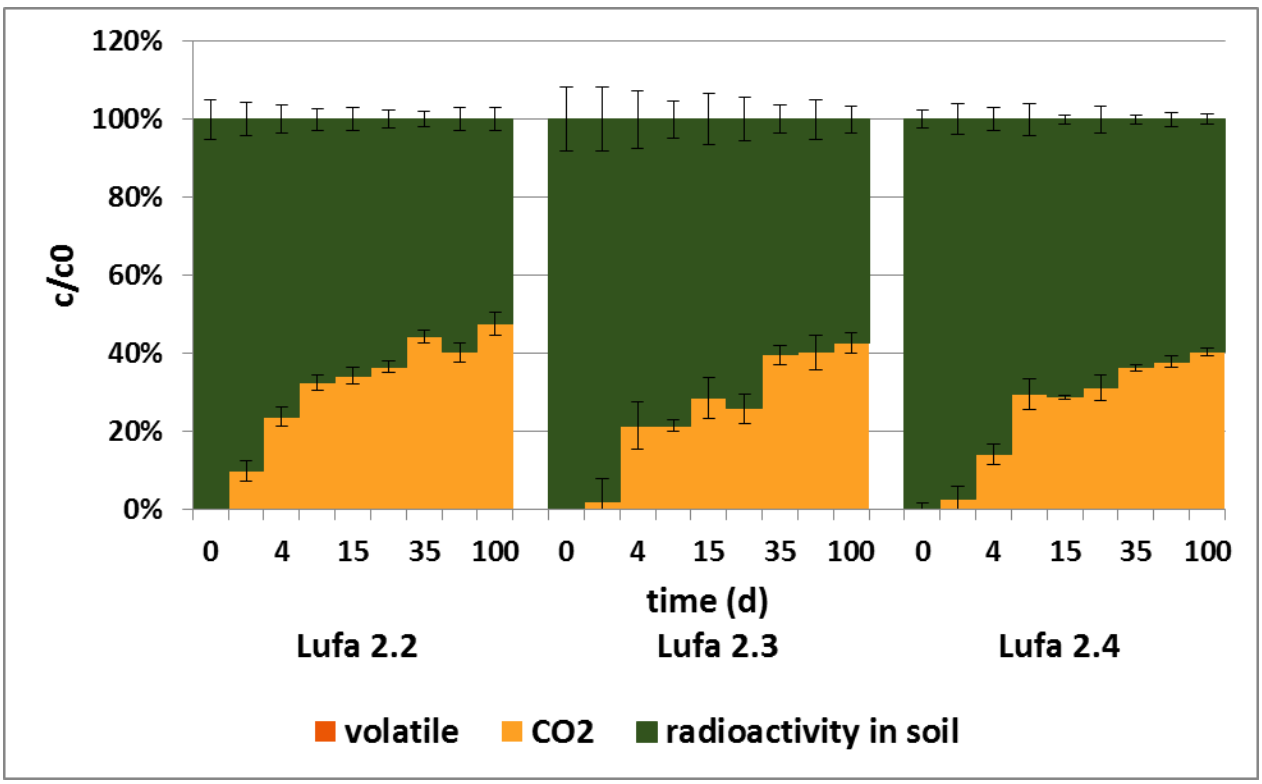
Table A44: Distribution of radioactivity after incubation with fenoxycarb in Lufa 2.3.

	Time (d)	Volatile	SD	CO_2	SD	Soil	SD	Sum	SD
FEC Lufa 2.3	0	0%	0%	0%	0%	100%	8%	100%	10%
	1	0%	0%	2%	6%	98%	8%	100%	10%
	4	0%	0%	22%	6%	78%	7%	100%	10%
	11	0%	0%	22%	2%	78%	5%	100%	6%
	15	0%	0%	29%	5%	71%	7%	100%	9%
	21	0%	0%	26%	4%	74%	5%	100%	8%
	35	0%	0%	40%	2%	60%	4%	100%	6%
	60	0%	0%	40%	4%	60%	5%	100%	8%
	100	0%	0%	43%	3%	57%	3%	100%	6%

Table A45: Distribution of radioactivity after incubation with fenoxycarb in Lufa 2.4.

	Time (d)	Volatile	SD	CO_2	SD	Soil	SD	Sum	SD
FEC Lufa 2.4	0	0%	0%	0%	2%	100%	2%	100%	3%
	1	0%	0%	3%	4%	97%	4%	100%	5%
	4	0%	0%	14%	3%	86%	3%	100%	4%
	11	0%	0%	30%	4%	70%	4%	100%	6%
	15	0%	0%	29%	1%	71%	1%	100%	2%
	21	0%	0%	31%	3%	69%	3%	100%	5%
	35	0%	0%	36%	1%	64%	1%	100%	2%
	60	0%	0%	38%	2%	62%	2%	100%	3%
	100	0%	0%	40%	1%	60%	1%	100%	2%

Figure A49: Distribution of radioactivity after incubation with fenoxycarb in Lufa 2.2, Lufa 2.3 an Lufa 2.4.



A4.3.2 Results of the transformation test of fenoxycarb including summarised batch extraction

Table A46: Distribution of radioactivity after incubation with fenoxycarb in Lufa 2.2 and subsequent three step batch extraction.

	Time (d)	CO_2	SD	EF	SD	NER	SD	Sum	SD
FEC Lufa 2.2	0	0%	0%	73%	6%	27%	6%	100%	10%
	1	10%	3%	46%	4%	44%	6%	100%	8%
	4	24%	2%	23%	2%	54%	8%	100%	9%
	11	33%	2%	16%	2%	51%	8%	100%	9%
	15	34%	2%	13%	1%	53%	8%	100%	8%
	21	37%	1%	13%	1%	50%	2%	100%	4%
	35	44%	2%	12%	1%	44%	7%	100%	8%
	60	40%	2%	11%	2%	49%	12%	100%	13%
	100	48%	3%	8%	0%	45%	3%	100%	5%

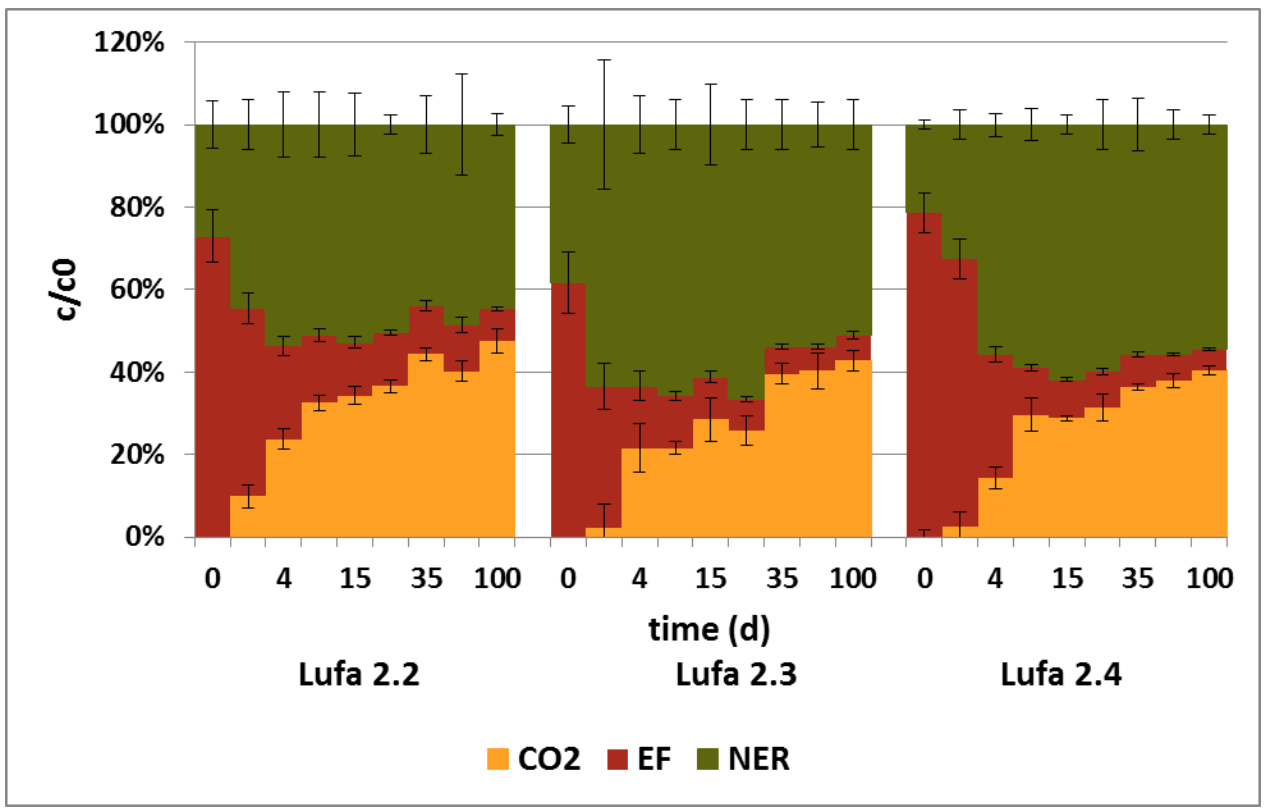
Table A47: Distribution of radioactivity after incubation with fenoxycarb in Lufa 2.3 and subsequent three step batch extraction.

	Time (d)	CO_2	SD	EF	SD	NER	SD	Sum	SD
FEC Lufa 2.3	0	0%	0%	62%	7%	38%	4%	100%	11%
	1	2%	6%	34%	6%	64%	16%	100%	18%
	4	22%	6%	15%	4%	63%	7%	100%	11%
	11	22%	2%	13%	1%	66%	6%	100%	8%
	15	29%	5%	10%	1%	61%	10%	100%	12%
	21	26%	4%	8%	1%	67%	6%	100%	8%
	35	40%	2%	7%	1%	54%	6%	100%	8%
	60	40%	4%	6%	1%	54%	6%	100%	8%

Table A48: Distribution of radioactivity after incubation with fenoxycarb in Lufa 2.4 and subsequent three step batch extraction.

	Time (d)	CO_2	SD	EF	SD	NER	SD	Sum	SD
FEC Lufa 2.4	0	0%	2%	79%	5%	21%	1%	100%	5%
	1	3%	4%	65%	5%	33%	3%	100%	7%
	4	14%	3%	30%	2%	56%	3%	100%	4%
	11	30%	4%	11%	1%	59%	4%	100%	6%
	15	29%	1%	9%	0%	62%	2%	100%	3%
	21	31%	3%	9%	1%	60%	6%	100%	7%
	35	36%	1%	8%	1%	56%	6%	100%	7%
	60	38%	2%	6%	0%	56%	3%	100%	4%
	100	40%	1%	5%	0%	55%	2%	100%	3%

Figure A50: Distribution of radioactivity after incubation with fenoxycarb in Lufa 2.2, Lufa 2.3 and Lufa 2.4 and subsequent three step batch extraction.



A4.3.3 Results of the transformation test of fenoxycarb including separated batch extraction

Table A49: Distribution of radioactivity after incubation with fenoxycarb in Lufa 2.2 and subsequent three step batch extraction with separated fractions of the extractable fraction.

	Time (d)	CO_2	SD	EF1	SD	EF2	SD	EF3	SD	NER	SD	Sum	SD
FEC Lufa 2.2	0	0%	0%	6%	1%	42%	4%	25%	2%	27%	6%	100%	10%
	1	10%	3%	3%	0%	26%	2%	16%	1%	44%	6%	100%	8%
	4	24%	2%	1%	0%	12%	1%	9%	1%	54%	8%	100%	9%
	11	33%	2%	1%	0%	9%	1%	7%	1%	51%	8%	100%	9%
	15	34%	2%	0%	0%	8%	1%	5%	1%	53%	8%	100%	8%
	21	37%	1%	1%	0%	7%	0%	6%	0%	50%	2%	100%	4%
	35	44%	2%	0%	0%	6%	1%	5%	1%	44%	7%	100%	8%
	60	40%	2%	0%	0%	5%	1%	6%	1%	49%	12%	100%	13%
	100	48%	3%	0%	0%	3%	0%	4%	0%	45%	3%	100%	5%

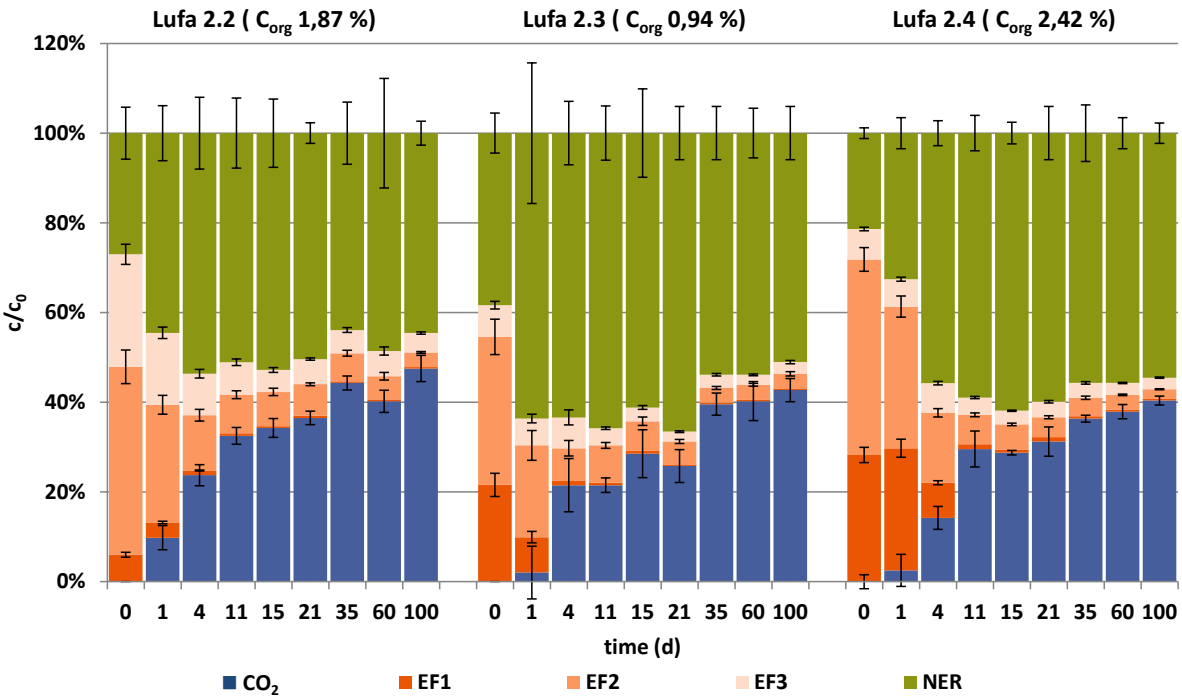
Table A50: Distribution of radioactivity after incubation with fenoxycarb in Lufa 2.3 and subsequent three step batch extraction with separated fractions of the extractable fraction.

	Time (d)	CO_2	SD	EF1	SD	EF2	SD	EF3	SD	NER	SD	Sum	SD
FEC Lufa 2.3	0	0%	0%	22%	3%	33%	4%	7%	1%	38%	4%	100%	11%
	1	2%	6%	8%	1%	20%	3%	6%	1%	64%	16%	100%	18%
	4	22%	6%	1%	0%	7%	2%	7%	2%	63%	7%	100%	11%
	11	22%	2%	1%	0%	8%	1%	4%	0%	66%	6%	100%	8%
	15	29%	5%	1%	0%	7%	1%	3%	0%	61%	10%	100%	12%
	21	26%	4%	0%	0%	5%	0%	2%	0%	67%	6%	100%	8%
	35	40%	2%	0%	0%	3%	0%	3%	0%	54%	6%	100%	8%
	60	40%	4%	0%	0%	3%	0%	2%	0%	54%	6%	100%	8%
	100	43%	3%	0%	0%	3%	0%	3%	0%	51%	6%	100%	8%

Table A51: Distribution of radioactivity after incubation with fenoxycarb in Lufa 2.4 and subsequent three step batch extraction with separated fractions of the extractable fraction.

	Time (d)	CO_2	SD	EF1	SD	EF2	SD	EF3	SD	NER	SD	Sum	SD
FEC Lufa 2.4	0	0%	2%	28%	2%	44%	3%	7%	0%	21%	1%	100%	5%
	1	3%	4%	27%	2%	32%	2%	6%	0%	33%	3%	100%	7%
	4	14%	3%	8%	0%	16%	1%	7%	0%	56%	3%	100%	4%
	11	30%	4%	1%	0%	7%	0%	4%	0%	59%	4%	100%	6%
	15	29%	1%	1%	0%	6%	0%	3%	0%	62%	2%	100%	3%
	21	31%	3%	1%	0%	4%	0%	3%	0%	60%	6%	100%	7%
	35	36%	1%	1%	0%	4%	0%	3%	0%	56%	6%	100%	7%
	60	38%	2%	0%	0%	3%	0%	3%	0%	56%	3%	100%	4%
	100	40%	1%	0%	0%	2%	0%	3%	0%	55%	2%	100%	3%

Figure A51: Distribution of radioactivity after incubation with fenoxycarb in Lufa 2.2, Lufa 2.3 and Lufa 2.4 and subsequent three step batch extraction with separated fractions of the extractable fraction.



A4.3.4 Results of the transformation test of fenoxycarb including batch extraction and subsequent Radio-HPLC analysis

Table A52: Distribution of radioactivity after the transformation test with fenoxycarb in Lufa 2.2 following three step batch extraction and subsequent Radio-HPLC analysis.

	Time (d)	CO_2	SD	FEC	SD	FEC-OH	SD	Unknown ES	SD	NER	SD	Sum	SD
FEC Lufa 2.2	0	0%	0%	73%	6%	0%	0%	0%	0%	27%	6%	100%	10%
	11	33%	2%	7%	1%	6%	1%	4%	1%	51%	8%	100%	9%
	35	44%	2%	6%	1%	3%	0%	4%	1%	44%	7%	100%	8%
	60	40%	2%	4%	1%	3%	1%	4%	1%	49%	12%	100%	13%
	100	48%	3%	3%	0%	2%	0%	3%	0%	45%	3%	100%	5%

Figure A52: Distribution of radioactivity after the transformation test with fenoxycarb in Lufa 2.2 following three step batch extraction and subsequent Radio-HPLC analysis.

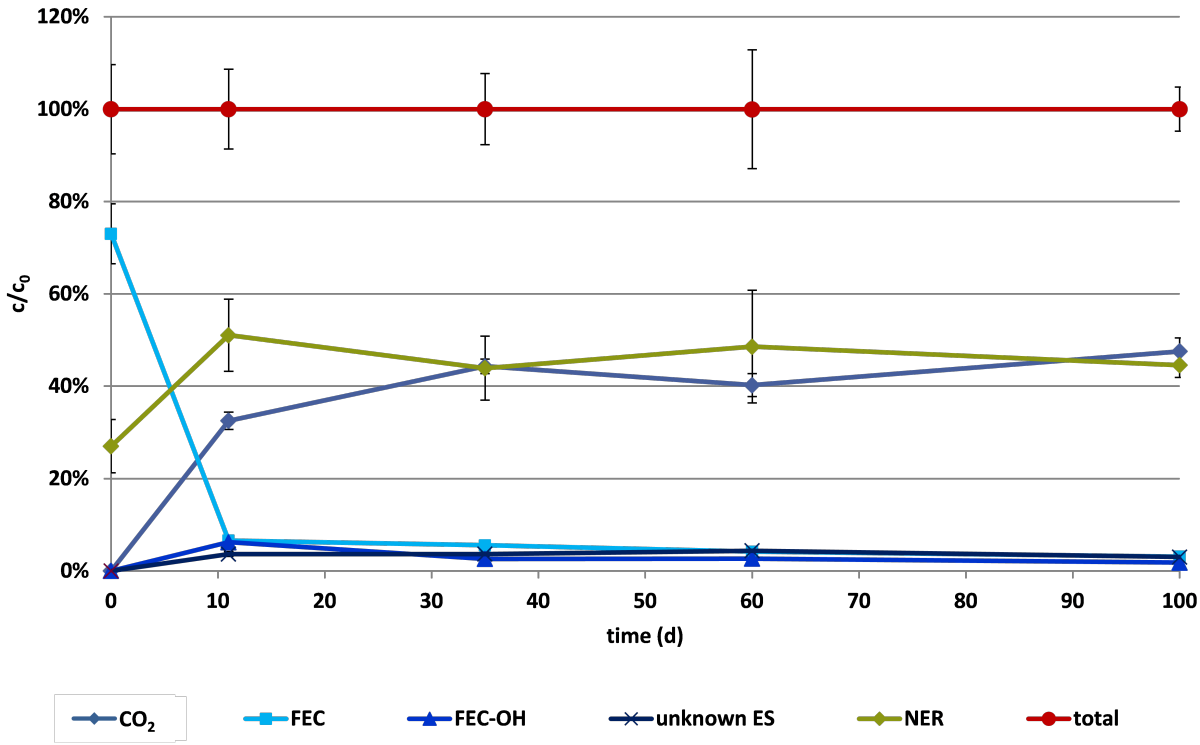


Figure A53: Radioactive decay graph after transformation test with fenoxycarb in Lufa 2.2 following three step batch extraction and subsequent Radio-HPLC analysis.

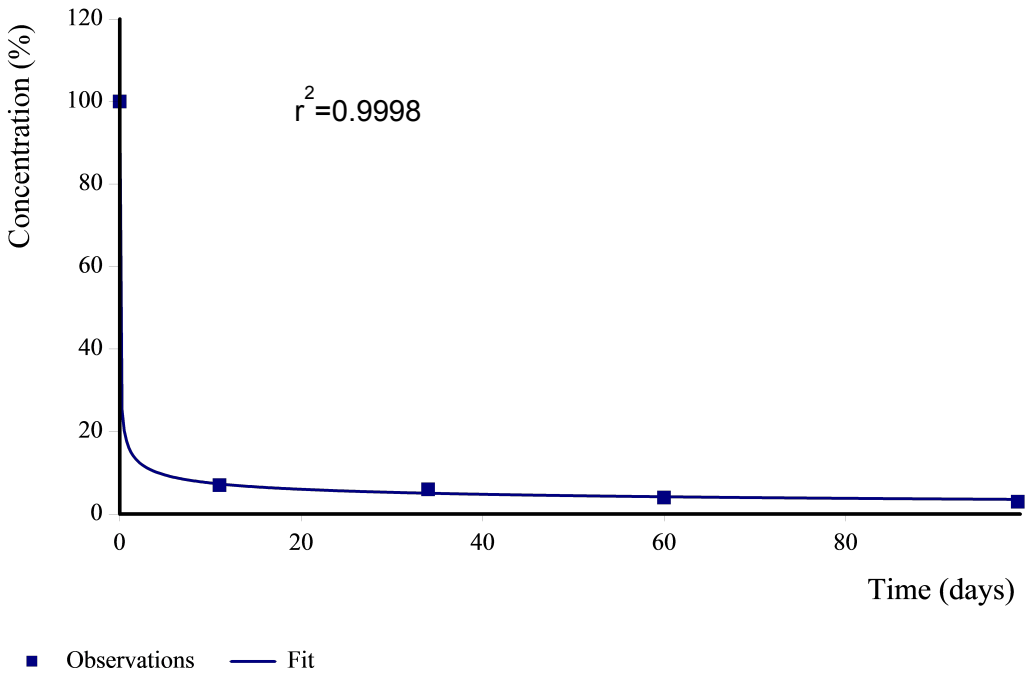


Table A53: Radioactive decay for the transformation test with fenoxycarb in Lufa 2.2 following three step batch extraction and subsequent Radio-HPLC analysis.

	Time (days)	Value (%)	Predicted Value	Residual
FEC Lufa 2.2	0	100	100	1.557E-06
	7	7	7.301	-0.3005
	34	6	5.036	0.9642
	60	4	4.177	-0.1772
	98	3	3.542	-0.5424

Table A54: Distribution of radioactivity after the transformation test with fenoxycarb in Lufa 2.3 following three step batch extraction and subsequent Radio-HPLC analysis.

	Time (d)	CO_2	SD	FEC	SD	FEC-OH	SD	Unknown ES	SD	NER	SD	Sum	SD
FEC Lufa 2.3	0	0%	0%	62%	7%	0%	0%	0%	0%	38%	4%	100%	11%
	11	22%	2%	8%	1%	2%	0%	3%	0%	66%	6%	100%	8%
	35	40%	2%	4%	1%	1%	0%	1%	0%	54%	6%	100%	8%
	60	40%	4%	4%	0%	1%	0%	1%	0%	54%	6%	100%	8%
	100	43%	3%	4%	1%	1%	0%	1%	0%	51%	6%	100%	8%

Figure A54: Distribution of radioactivity after the transformation test with fenoxycarb in Lufa 2.3 following three step batch extraction and subsequent Radio-HPLC analysis.

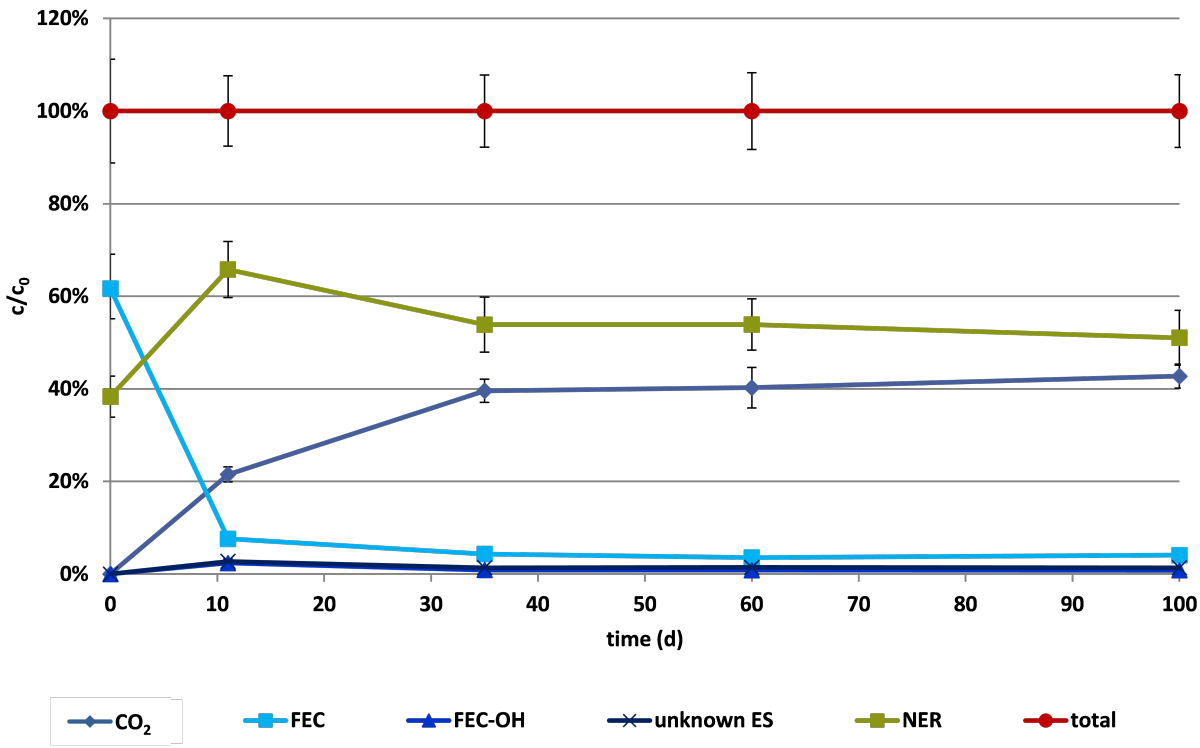


Figure A55: Radioactive decay graph after transformation test with fenoxycarb in Lufa 2.3 following three step batch extraction and subsequent Radio-HPLC analysis.

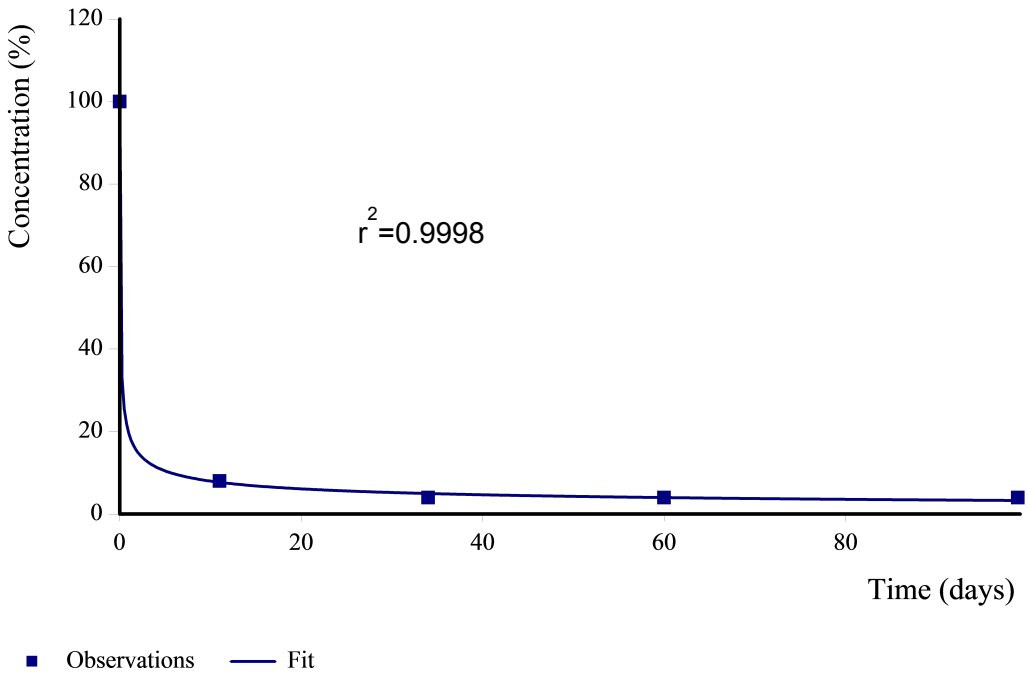


Table A55: Radioactive decay for the transformation test with fenoxycarb in Lufa 2.3 following three step batch extraction and subsequent Radio-HPLC analysis.

	Time (days)	Value (%)	Predicted Value	Residual
FEC Lufa 2.3	0	100	100	-1.358E-05
	7	8	7.7	0.2999
	34	4	4.963	-0.9626
	60	4	3.978	0.02229
	98	4	3.273	0.7272

Table A56: Distribution of radioactivity after the transformation test with fenoxycarb in Lufa 2.4 following three step batch extraction and subsequent Radio-HPLC analysis.

	Time (d)	CO_2	SD	FEC	SD	FEC-OH	SD	Unknown ES	SD	NER	SD	Sum	SD
FEC Lufa 2.4	0	0%	0%	79%	5%	0%	0%	0%	0%	21%	1%	100%	5%
	11	30%	4%	6%	1%	3%	0%	2%	0%	59%	4%	100%	6%
	35	36%	1%	5%	1%	1%	0%	2%	0%	56%	6%	100%	7%
	60	38%	2%	4%	0%	1%	0%	2%	0%	56%	3%	100%	4%
	100	40%	1%	3%	0%	1%	0%	2%	0%	55%	2%	100%	3%

Figure A56: Distribution of radioactivity after the transformation test with fenoxycarb in Lufa 2.4 following three step batch extraction and subsequent Radio-HPLC analysis.

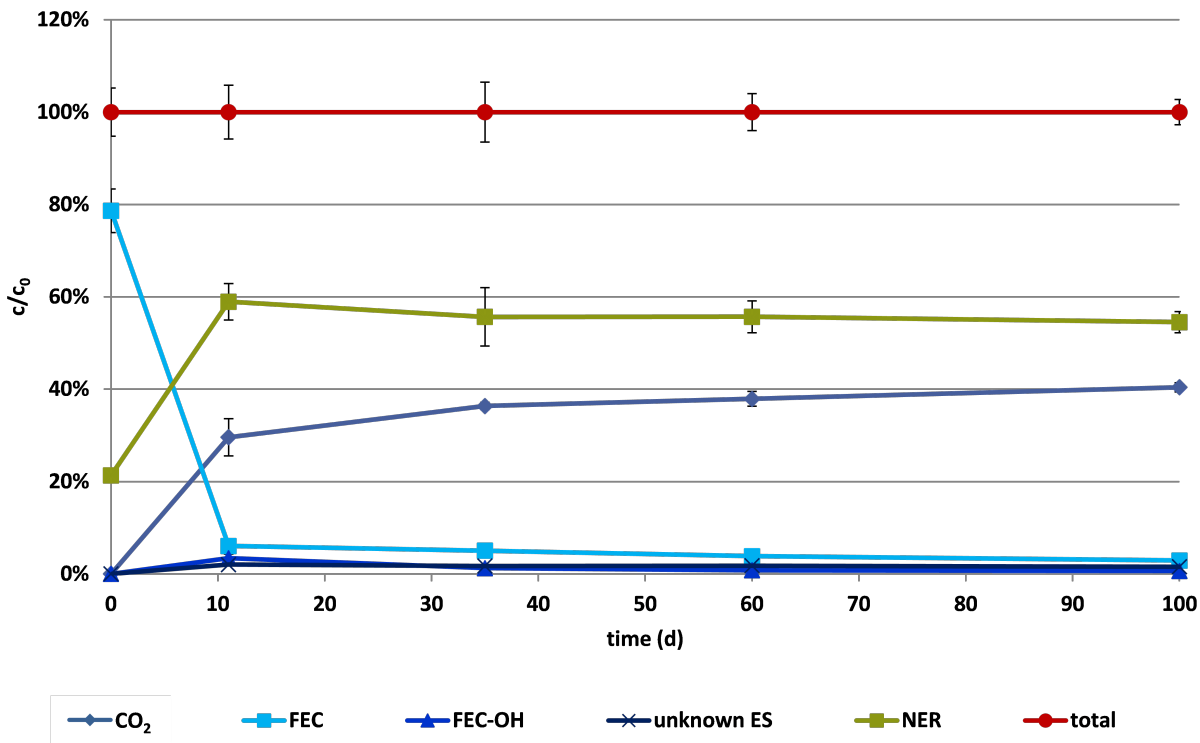


Figure A57: Radioactive decay graph after transformation test with fenoxycarb in Lufa 2.4 following three step batch extraction and subsequent Radio-HPLC analysis.

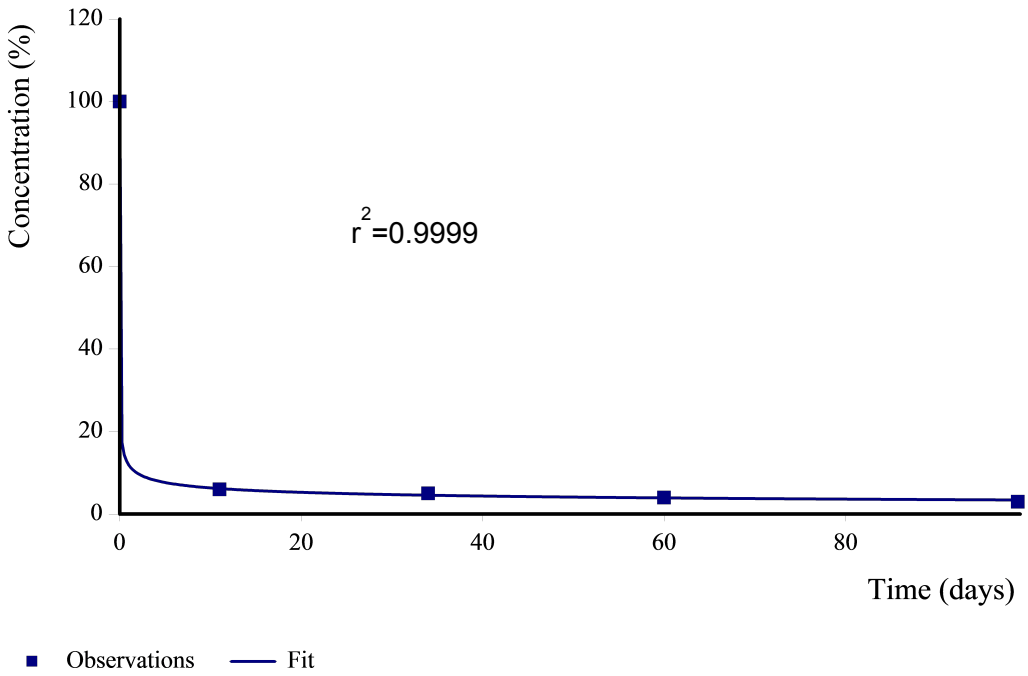


Table A57: Radioactive decay for the transformation test with fenoxycarb in Lufa 2.4 following three step batch extraction and subsequent Radio-HPLC analysis.

	Time (days)	Value (%)	Predicted Value	Residual
FEC Lufa 2.4	0	100	100	2.123E-07
	7	6	6.177	-0.177
	34	5	4.547	0.453
	60	4	3.897	0.1028
	98	3	3.402	-0.4018

A4.3.5 Results of the transformation test of fenoxycarb including batch extraction and PLE

Table A58: Distribution of radioactivity after the transformation test with fenoxycarb in Lufa 2.2 following three step batch extraction and subsequent PLE.

	Time (d)	CO_2	SD	EF	SD	PLE-EF	SD	PLE-NER	SD	Sum	SD
FEC Lufa 2.2	11	33%	2%	16%	2%	8%	1%	43%	9%	100%	10%
	35	44%	2%	12%	1%	6%	1%	38%	6%	100%	7%
	60	40%	2%	11%	2%	5%	2%	43%	11%	100%	12%
	100	48%	3%	8%	0%	3%	0%	41%	4%	100%	5%

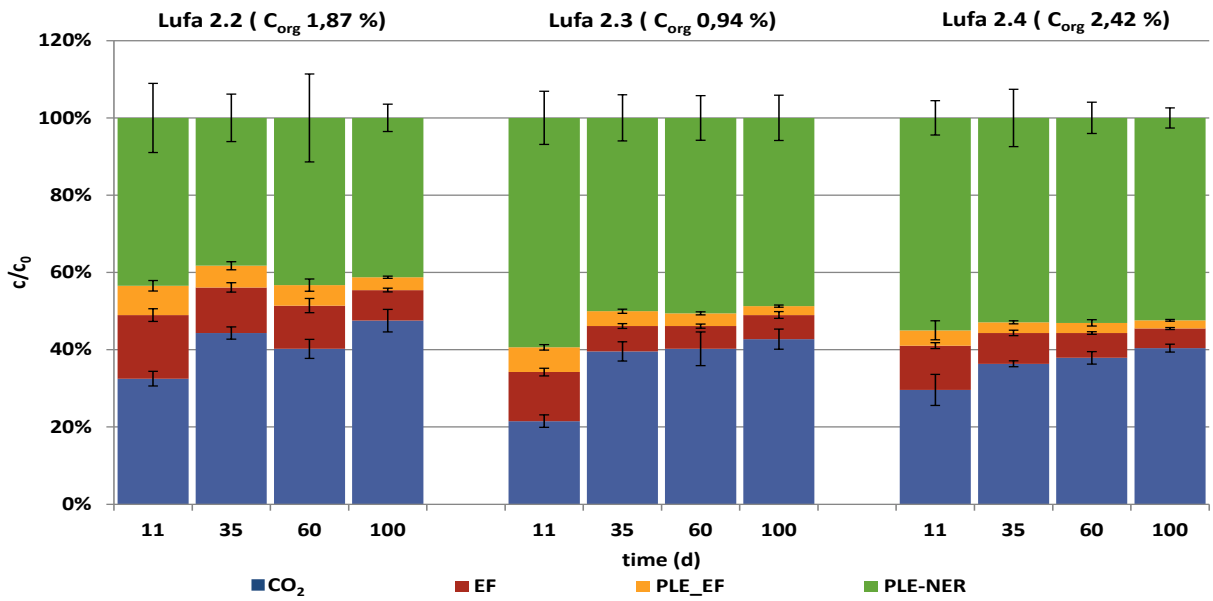
Table A59: Distribution of radioactivity after the transformation test with fenoxycarb in Lufa 2.3 following three step batch extraction and subsequent PLE.

	Time (d)	CO_2	SD	EF	SD	PLE-EF	SD	PLE-NER	SD	Sum	SD
FEC Lufa 2.3	11	22%	2%	13%	1%	6%	1%	59%	7%	100%	8%
	35	40%	2%	7%	1%	4%	1%	50%	6%	100%	8%
	60	40%	4%	6%	1%	3%	0%	51%	6%	100%	9%
	100	43%	3%	6%	1%	2%	0%	49%	6%	100%	8%

Table A60: Distribution of radioactivity after the transformation test with fenoxycarb in Lufa 2.4 following three step batch extraction and subsequent PLE.

	Time (d)	CO_2	SD	EF	SD	PLE-EF	SD	PLE-NER	SD	Sum	SD
FEC Lufa 2.4	11	30%	4%	11%	1%	4%	2%	55%	4%	100%	7%
	35	36%	1%	8%	1%	3%	0%	53%	7%	100%	8%
	60	38%	2%	6%	0%	3%	1%	53%	4%	100%	5%
	100	40%	1%	5%	0%	2%	0%	52%	3%	100%	3%

Figure A58: Distribution of radioactivity after the transformation test with fenoxycarb in Lufa 2.2, Lufa 2.3 and Lufa 2.4 following three step batch extraction and subsequent PLE.



A4.3.6 Results of the transformation test of fenoxycarb including separated batch extraction and PLE

Table A61: Distribution of radioactivity after the transformation test with fenoxycarb in Lufa 2.2 following three step batch extraction with separated fractions of the extractable fraction and subsequent PLE.

	Time (d)	CO_2	SD	EF1	SD	EF2	SD	EF3	SD	PLE-EF	SD	PLE-NER	SD	Sum	SD
FEC Lufa 2.2	11	33%	2%	1%	0%	9%	1%	7%	1%	8%	1%	43%	9%	100%	10%
	35	44%	2%	0%	0%	6%	1%	5%	1%	6%	1%	38%	6%	100%	7%
	60	40%	2%	0%	0%	5%	1%	6%	1%	5%	2%	43%	11%	100%	12%
	100	48%	3%	0%	0%	3%	0%	4%	0%	3%	0%	41%	4%	100%	5%

Figure A59: Distribution of radioactivity after the transformation test with fenoxycarb in Lufa 2.2 following three step batch extraction with separated fractions of the extractable fraction and subsequent PLE.

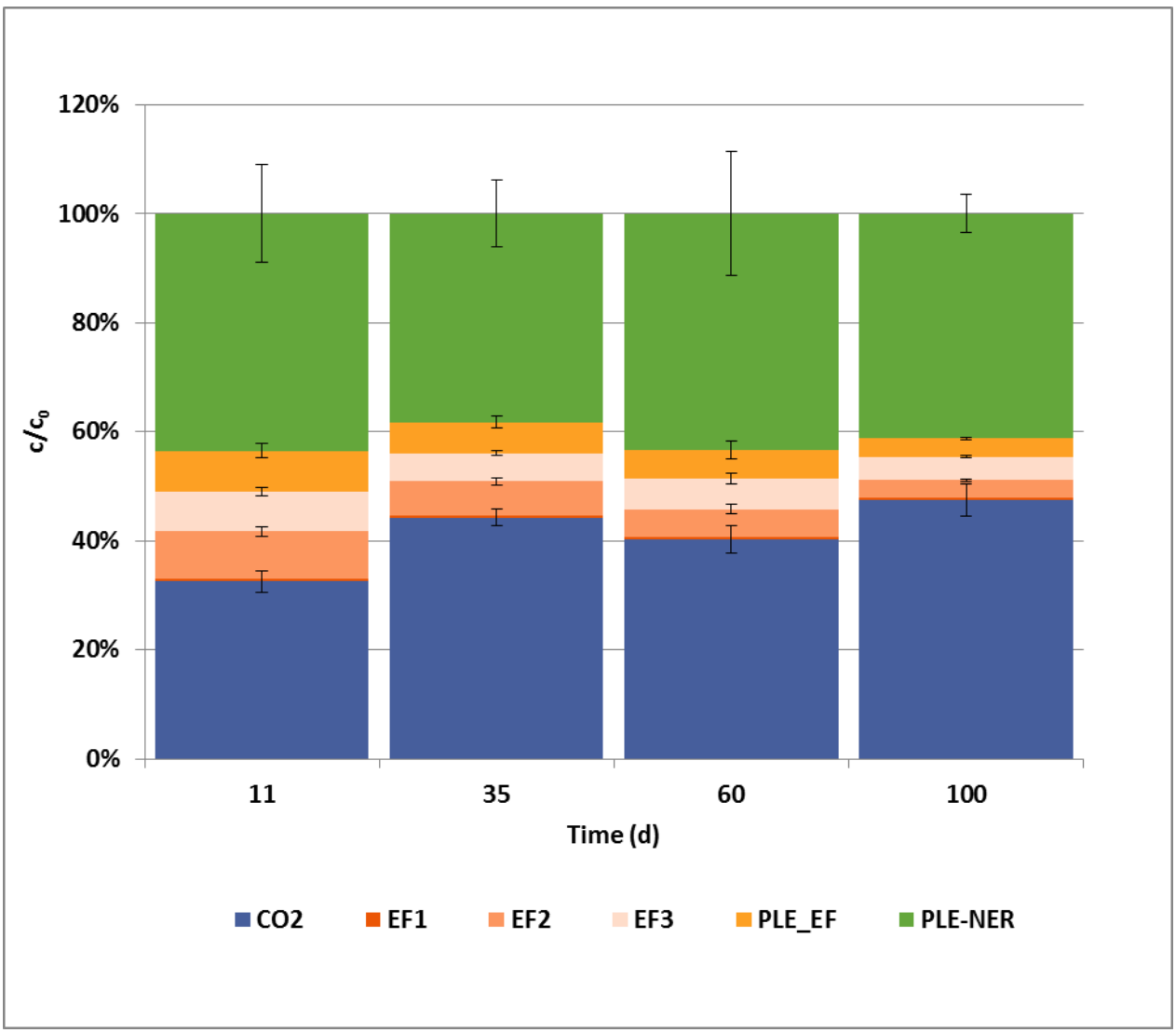


Table A62: Distribution of radioactivity after the transformation test with fenoxycarb in Lufa 2.3 following three step batch extraction with separated fractions of the extractable fraction and subsequent PLE.

	Time (d)	CO_2	SD	EF1	SD	EF2	SD	EF3	SD	PLE-EF	SD	PLE-NER	SD	Sum	SD
FEC Lufa 2.3	11	22%	2%	1%	0%	8%	1%	4%	0%	6%	1%	59%	7%	100%	8%
	35	40%	2%	0%	0%	3%	0%	3%	0%	4%	1%	50%	6%	100%	8%
	60	40%	4%	0%	0%	3%	0%	2%	0%	3%	0%	51%	6%	100%	9%
	100	43%	3%	0%	0%	3%	0%	3%	0%	2%	0%	49%	6%	100%	8%

Figure A60: Distribution of radioactivity after the transformation test with fenoxycarb in Lufa 2.3 following three step batch extraction with separated fractions of the extractable fraction and subsequent PLE.

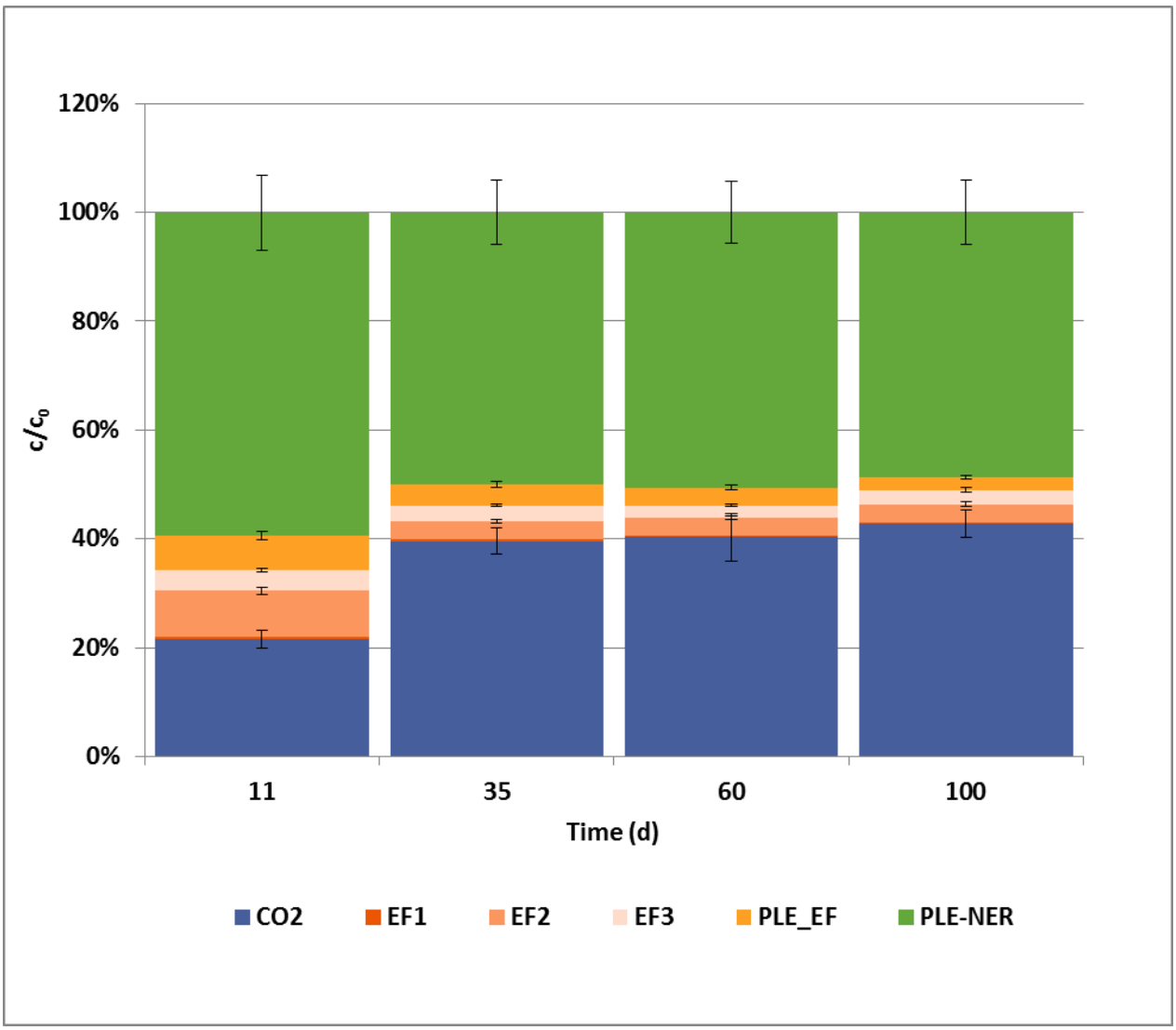
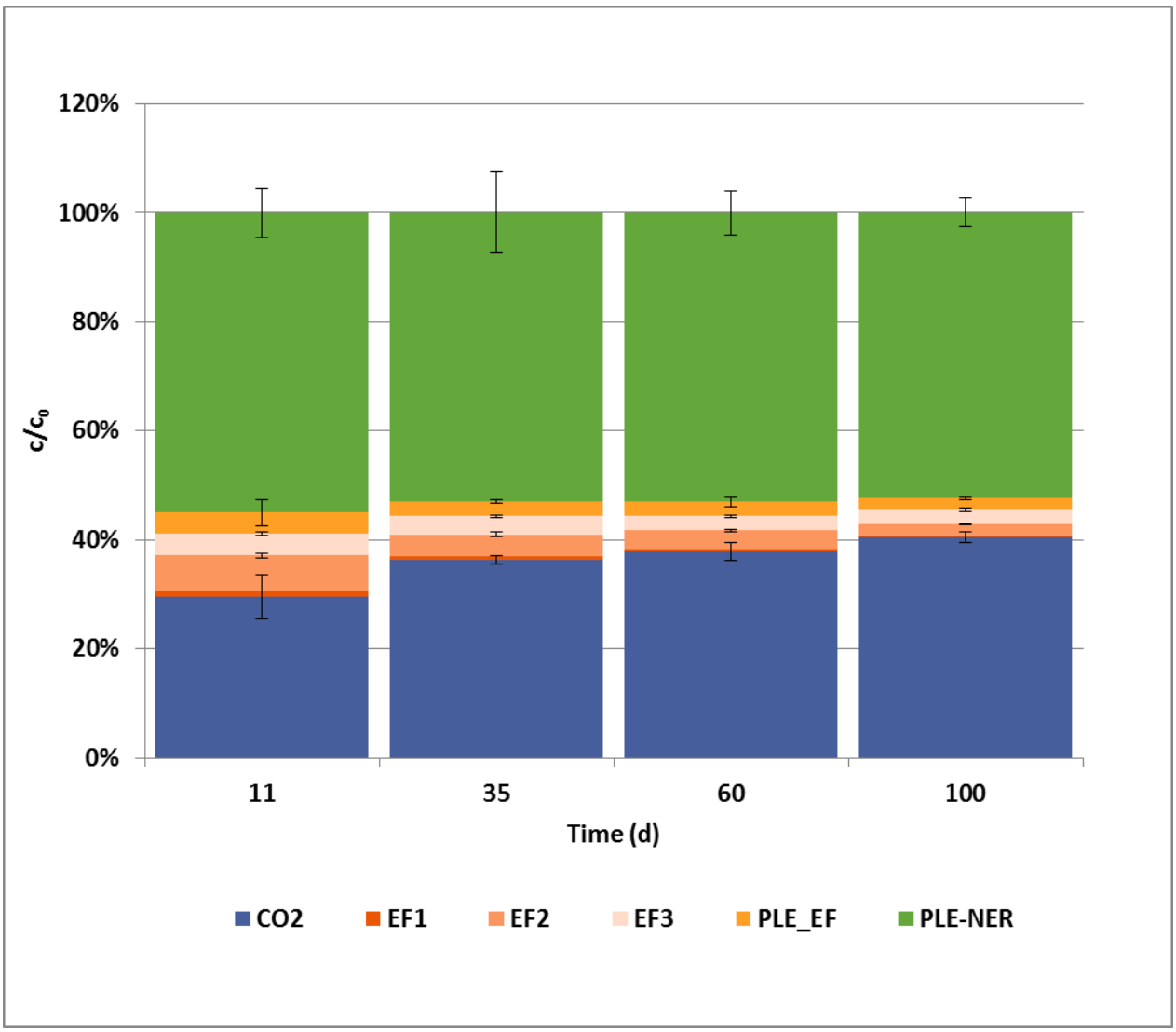


Table A63: Distribution of radioactivity after the transformation test with fenoxycarb in Lufa 2.4 following three step batch extraction with separated fractions of the extractable fraction and subsequent PLE.

	Time (d)	CO_2	SD	EF1	SD	EF2	SD	EF3	SD	PLE-EF	SD	PLE-NER	SD	Sum	SD
FEC Lufa 2.4	11	30%	4%	1%	0%	7%	0%	4%	0%	4%	2%	55%	4%	100%	7%
	35	36%	1%	1%	0%	4%	0%	3%	0%	3%	0%	53%	7%	100%	8%
	60	38%	2%	0%	0%	3%	0%	3%	0%	3%	1%	53%	4%	100%	5%
	100	40%	1%	0%	0%	2%	0%	3%	0%	2%	0%	52%	3%	100%	3%

Figure A61: Distribution of radioactivity after the transformation test with fenoxycarb in Lufa 2.4 following three step batch extraction with separated fractions of the extractable fraction and subsequent PLE.



A4.3.7 Results of the transformation test of fenoxycarb including batch extraction and PLE

Table A64: Distribution of radioactivity after the transformation test with fenoxycarb in Lufa 2.2 following three step batch extraction and subsequent PLE.

	Time (d)	CO_2	SD	EF	SD	PLE-EF	SD	PLE-NER	SD	Sum	SD
FEC Lufa 2.2	11	33%	2%	16%	2%	8%	1%	43%	9%	100%	10%
	35	44%	2%	12%	1%	6%	1%	38%	6%	100%	7%
	60	40%	2%	11%	2%	5%	2%	43%	11%	100%	12%
	100	48%	3%	8%	0%	3%	0%	41%	4%	100%	5%

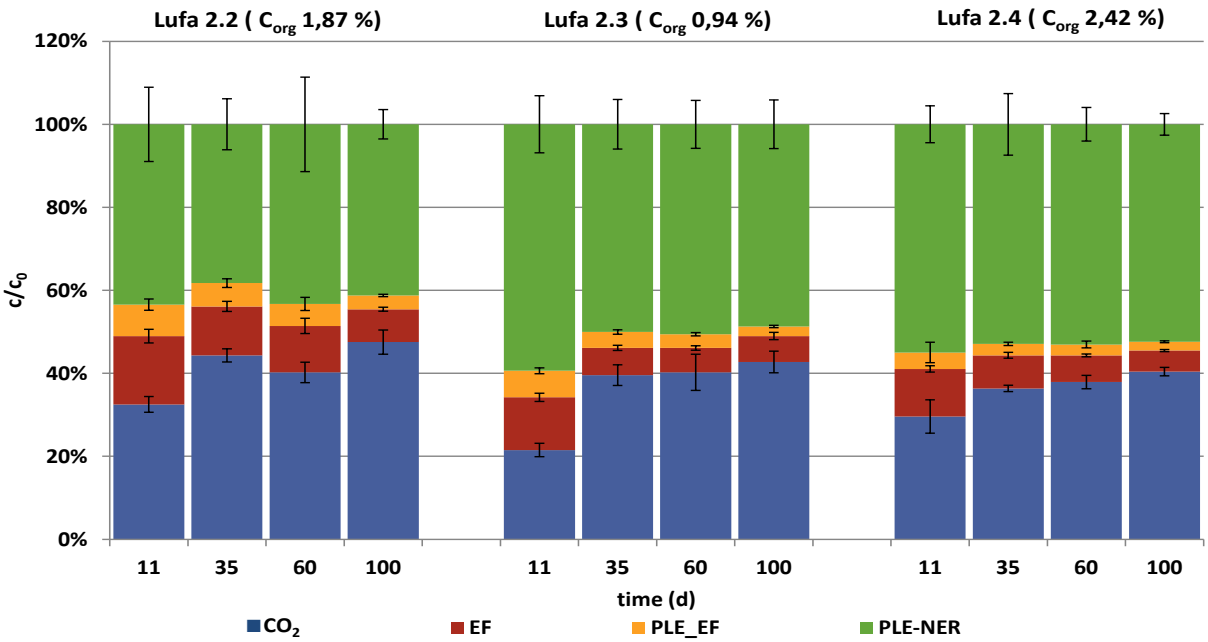
Table A65: Distribution of radioactivity after the transformation test with fenoxycarb in Lufa 2.3 follwing three step batch extraction and subsequent PLE.

	Time (d)	CO_2	SD	EF	SD	PLE-EF	SD	PLE-NER	SD	Sum	SD
FEC Lufa 2.3	11	22%	2%	13%	1%	6%	1%	59%	7%	100%	8%
	35	40%	2%	7%	1%	4%	1%	50%	6%	100%	8%
	60	40%	4%	6%	1%	3%	0%	51%	6%	100%	9%
	100	43%	3%	6%	1%	2%	0%	49%	6%	100%	8%

Table A66: Distribution of radioactivity after the transformation test with fenoxycarb in Lufa 2.4 following three step batch extraction and subsequent PLE.

	Time (d)	CO_2	SD	EF	SD	PLE-EF	SD	PLE-NER	SD	Sum	SD
FEC Lufa 2.4	11	30%	4%	11%	1%	4%	2%	55%	4%	100%	7%
	35	36%	1%	8%	1%	3%	0%	53%	7%	100%	8%
	60	38%	2%	6%	0%	3%	1%	53%	4%	100%	5%
	100	40%	1%	5%	0%	2%	0%	52%	3%	100%	3%

Figure A62: Distribution of radioactivity after the transformation test with fenoxycarb in Lufa 2.2, Lufa 2.3 and Lufa 2.4 following three step batch extraction and subsequent PLE.

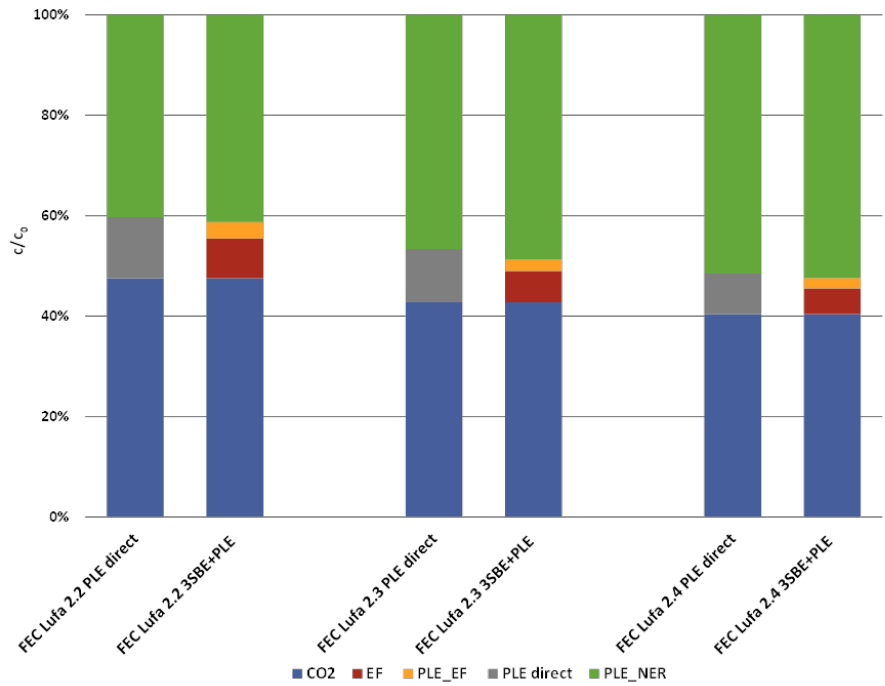


A4.3.8 Results of the transformation test of fenoxycarb after direct PLE

Table A67: Comparison of the extraction efficiency between the three step batch extraction and the direct PLE with fenoxycarb.

	Time (d)	CO_2	SD	3SBE-EF	SD	PLE-EF	SD	PLE-NER	SD	Sum	SD
FEC Lufa 2.2 PLE direct	100	48%	3%	-	-	12%	1%	40%	3%	100%	5%
FEC Lufa 2.2 3SBE+PLE	100	48%	3%	8%	0%	3%	0%	41%	4%	100%	5%
FEC Lufa 2.3 PLE direct	100	43%	3%	-	-	11%	1%	47%	4%	100%	7%
FEC Lufa 2.3 3SBE+PLE	100	43%	3%	6%	1%	2%	0%	49%	6%	100%	8%
FEC Lufa 2.4 PLE direct	100	40%	1%	-	-	8%	0%	52%	1%	100%	2%
FEC Lufa 2.4 3SBE+PLE	100	40%	1%	5%	0%	2%	0%	52%	3%	100%	3%

Figure A63: Comparison of the extraction efficiency between the three step batch extraction and the direct PLE with fenoxycarb.



A4.4 Results of the transformation test of acetaminophen

A4.4.1 Results of the transformation test of acetaminophen after incubation period

Table A68: Distribution of radioactivity after incubation with acetaminophen in Lufa 2.2.

	Time (d)	Volatile	SD	CO2	SD	Soil	SD	Sum	SD
ACT Lufa 2.2	0	0%	0%	0%	0%	100%	1%	100%	1%
	1	0%	0%	1%	0%	100%	2%	101%	2%
	2	0%	0%	3%	0%	95%	1%	98%	1%
	5	0%	0%	6%	0%	98%	2%	104%	2%
	8	0%	0%	7%	0%	94%	1%	101%	1%
	12	0%	0%	8%	0%	92%	3%	101%	3%
	16	0%	0%	10%	0%	90%	3%	99%	3%
	21	0%	0%	10%	0%	94%	2%	105%	2%
	35	0%	0%	14%	0%	92%	4%	106%	5%

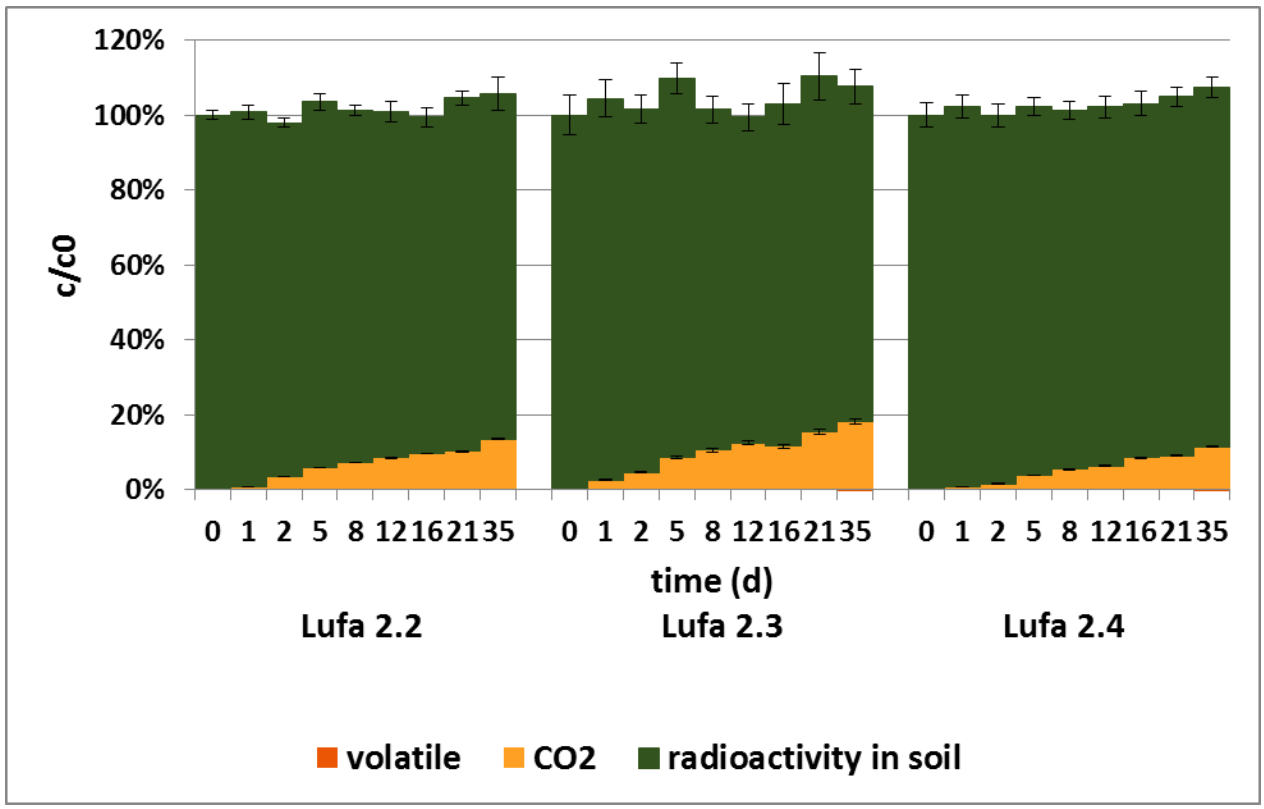
Table A69: Distribution of radioactivity after incubation with acetaminophen in Lufa 2.3.

	Time (d)	Volatile	SD	CO_2	SD	Soil	SD	Sum	SD
ACT Lufa 2.3	0	0%	0%	0%	0%	100%	5%	100%	5%
	1	0%	0%	3%	0%	102%	5%	104%	5%
	2	0%	0%	5%	0%	97%	4%	102%	4%
	5	0%	0%	9%	0%	101%	4%	110%	4%
	8	0%	0%	11%	0%	91%	4%	101%	4%
	12	0%	0%	12%	0%	87%	3%	99%	4%
	16	0%	0%	12%	0%	91%	5%	103%	6%
	21	0%	0%	15%	1%	95%	6%	110%	7%
	35	0%	0%	18%	1%	89%	5%	108%	5%

Table A70: Distribution of radioactivity after incubation with acetaminophen in Lufa 2.4.

	Time (d)	Volatile	SD	CO_2	SD	Soil	SD	Sum	SD
ACT Lufa 2.4	0	0%	0%	0%	0%	100%	3%	100%	3%
	1	0%	0%	1%	0%	102%	3%	102%	3%
	2	0%	0%	2%	0%	98%	3%	100%	3%
	5	0%	0%	4%	0%	99%	2%	102%	2%
	8	0%	0%	5%	0%	96%	2%	101%	3%
	12	0%	0%	6%	0%	96%	3%	102%	3%
	16	0%	0%	8%	0%	95%	3%	103%	3%
	21	0%	0%	9%	0%	96%	2%	105%	3%
	35	0%	0%	11%	0%	96%	3%	107%	3%

Figure A64: Distribution of radioactivity after incubation with acetaminophen in Lufa 2.2, Lufa 2.3 and Lufa 2.4.



A4.4.2 Results of the transformation test of acetaminophen including summarised batch extraction

Table A71: Distribution of radioactivity after incubation with acetaminophen in Lufa 2.2 and subsequent three step batch extraction.

	Time (d)	CO_2	SD	EF	SD	NER	SD	Sum	SD
ACT Lufa 2.2	0	0%	0%	5%	0%	95%	12%	100%	12%
	1	1%	0%	5%	0%	95%	2%	101%	2%
	2	3%	0%	3%	0%	91%	9%	98%	9%
	5	6%	0%	4%	0%	94%	7%	104%	7%
	8	7%	0%	4%	0%	90%	13%	101%	13%
	12	8%	0%	5%	0%	88%	3%	101%	3%
	16	10%	0%	2%	0%	88%	9%	99%	9%
	21	10%	0%	2%	0%	92%	2%	105%	2%
	35	14%	0%	2%	0%	90%	7%	106%	7%

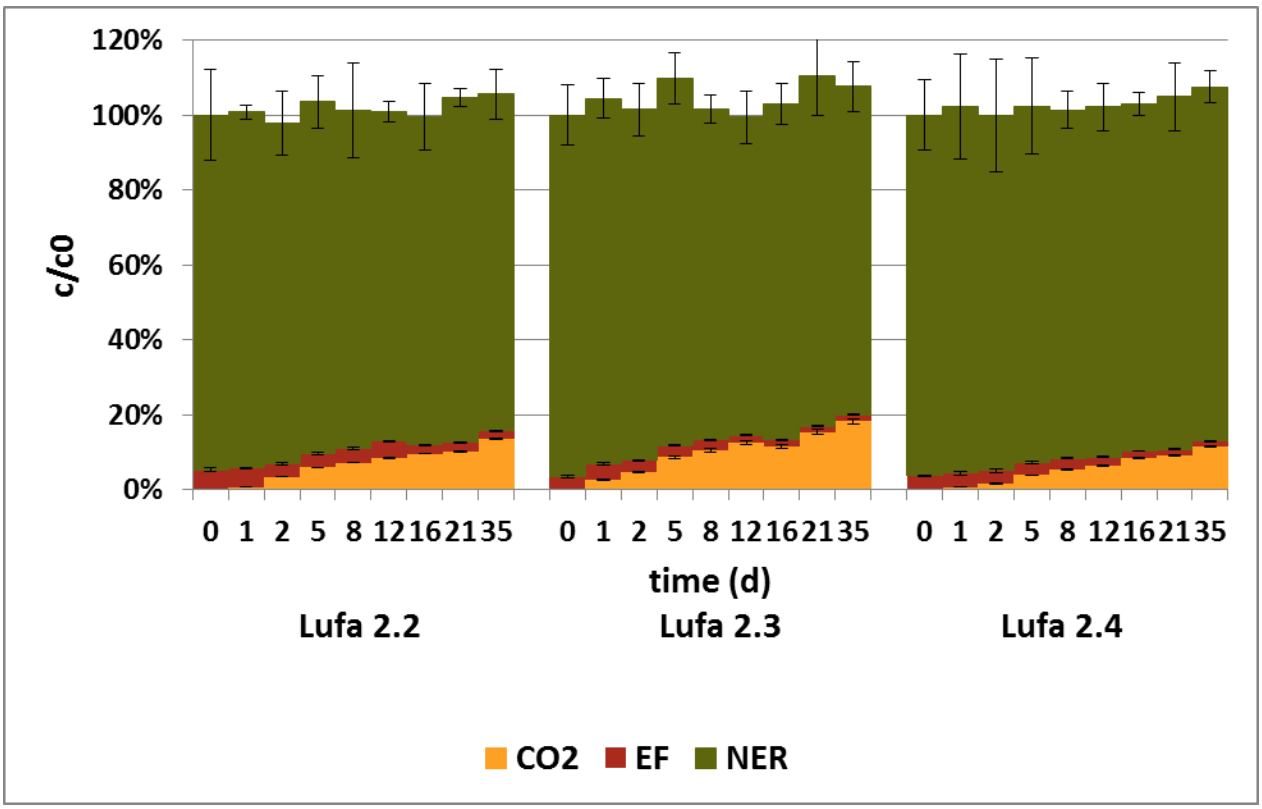
Table A72: Distribution of radioactivity after incubation with acetaminophen in Lufa 2.3 and subsequent three step batch extraction.

	Time (d)	CO_2	SD	EF	SD	NER	SD	Sum	SD
ACT Lufa 2.3	0	0%	0%	3%	0%	97%	8%	100%	8%
	1	3%	0%	4%	0%	98%	5%	104%	5%
	2	5%	0%	3%	0%	94%	7%	102%	7%
	5	9%	0%	3%	0%	98%	7%	110%	7%
	8	11%	0%	3%	0%	88%	4%	101%	4%
	12	12%	0%	2%	0%	85%	7%	99%	7%
	16	12%	0%	2%	0%	90%	5%	103%	6%
	21	15%	1%	2%	0%	93%	10%	110%	11%
	35	18%	1%	2%	0%	88%	7%	108%	7%

Table A73: Distribution of radioactivity after incubation with acetaminophen in Lufa 2.4 and subsequent three step batch extraction.

	Time (d)	CO_2	SD	EF	SD	NER	SD	Sum	SD
ACT Lufa 2.4	0	0%	0%	4%	0%	96%	9%	100%	10%
	1	1%	0%	4%	0%	98%	14%	102%	14%
	2	2%	0%	3%	0%	95%	15%	100%	15%
	5	4%	0%	3%	0%	95%	13%	102%	13%
	8	5%	0%	3%	0%	93%	5%	101%	5%
	12	6%	0%	2%	0%	93%	6%	102%	6%
	16	8%	0%	2%	0%	93%	3%	103%	3%
	21	9%	0%	2%	0%	94%	9%	105%	9%
	35	11%	0%	2%	0%	95%	4%	107%	4%

Figure A65: Distribution of radioactivity after incubation with acetaminophen in Lufa 2.2, Lufa 2.3 and Lufa 2.4 and subsequent three step batch extraction.



A4.4.3 Results of the transformation test of acetaminophen including separated batch extraction

Table A74: Distribution of radioactivity after incubation with acetaminophen in Lufa 2.2 and subsequent three step batch extraction with separated fractions of the extractable fraction.

	Time (d)	CO_2	SD	EF1	SD	EF2	SD	EF3	SD	NER	SD	Sum	SD
ACT Lufa 2.2	0	0%	0%	2%	0%	2%	0%	1%	0%	95%	12%	100%	12%
	1	1%	0%	2%	0%	2%	0%	1%	0%	95%	2%	101%	2%
	2	3%	0%	1%	0%	1%	0%	1%	0%	91%	9%	98%	9%
	5	6%	0%	1%	0%	2%	0%	1%	0%	94%	7%	104%	7%
	8	7%	0%	1%	0%	2%	0%	1%	0%	90%	13%	101%	13%
	12	8%	0%	1%	0%	2%	0%	1%	0%	88%	3%	101%	3%
	16	10%	0%	1%	0%	1%	0%	1%	0%	88%	9%	99%	9%
	21	10%	0%	1%	0%	1%	0%	1%	0%	92%	2%	105%	2%
	35	14%	0%	1%	0%	1%	0%	1%	0%	90%	7%	106%	7%

Table A75: Distribution of radioactivity after incubation with acetaminophen in Lufa 2.3 and subsequent three step batch extraction with separated fractions of the extractable fraction.

	Time (d)	CO_2	SD	EF1	SD	EF2	SD	EF3	SD	NER	SD	Sum	SD
ACT Lufa 2.3	0	0%	0%	1%	0%	1%	0%	1%	0%	97%	8%	100%	8%
	1	3%	0%	2%	0%	2%	0%	1%	0%	98%	5%	104%	5%
	2	5%	0%	1%	0%	1%	0%	1%	0%	94%	7%	102%	7%
	5	9%	0%	1%	0%	1%	0%	1%	0%	98%	7%	110%	7%
	8	11%	0%	1%	0%	1%	0%	1%	0%	88%	4%	101%	4%
	12	12%	0%	1%	0%	1%	0%	1%	0%	85%	7%	99%	7%
	16	12%	0%	0%	0%	1%	0%	1%	0%	90%	5%	103%	6%
	21	15%	1%	0%	0%	1%	0%	1%	0%	93%	10%	110%	11%
	35	18%	1%	1%	0%	0%	0%	1%	0%	88%	7%	108%	7%

Table A76: Distribution of radioactivity after incubation with acetaminophen in Lufa 2.4 and subsequent three step batch extraction with separated fractions of the extractable fraction.

	Time (d)	CO_2	SD	EF1	SD	EF2	SD	EF3	SD	NER	SD	Sum	SD
ACT Lufa 2.4	0	0%	0%	1%	0%	1%	0%	1%	0%	96%	9%	100%	10%
	1	1%	0%	1%	0%	1%	0%	1%	0%	98%	14%	102%	14%
	2	2%	0%	1%	0%	1%	0%	1%	0%	95%	15%	100%	15%
	5	4%	0%	1%	0%	1%	0%	1%	0%	95%	13%	102%	13%
	8	5%	0%	1%	0%	1%	0%	1%	0%	93%	5%	101%	5%
	12	6%	0%	1%	0%	1%	0%	1%	0%	93%	6%	102%	6%
	16	8%	0%	1%	0%	1%	0%	1%	0%	93%	3%	103%	3%
	21	9%	0%	1%	0%	1%	0%	1%	0%	94%	9%	105%	9%
	35	11%	0%	0%	0%	1%	0%	0%	0%	95%	4%	107%	4%

A4.4.4 Results of the transformation test of acetaminophen including batch extraction and PLE

Table A77: Distribution of radioactivity after the transformation test with acetaminophen in Lufa 2.2 following three step batch extraction and subsequent PLE.

	Time (d)	CO_2	SD	EF	SD	PLE-EF	SD	PLE-NER	SD	Sum	SD
ACT Lufa 2.2	5	6%	0%	4%	0%	6%	1%	88%	8%	104%	8%
	16	10%	0%	2%	0%	5%	1%	83%	20%	99%	20%
	21	10%	0%	2%	0%	6%	1%	86%	33%	105%	33%
	35	14%	0%	2%	0%	3%	0%	87%	11%	106%	11%

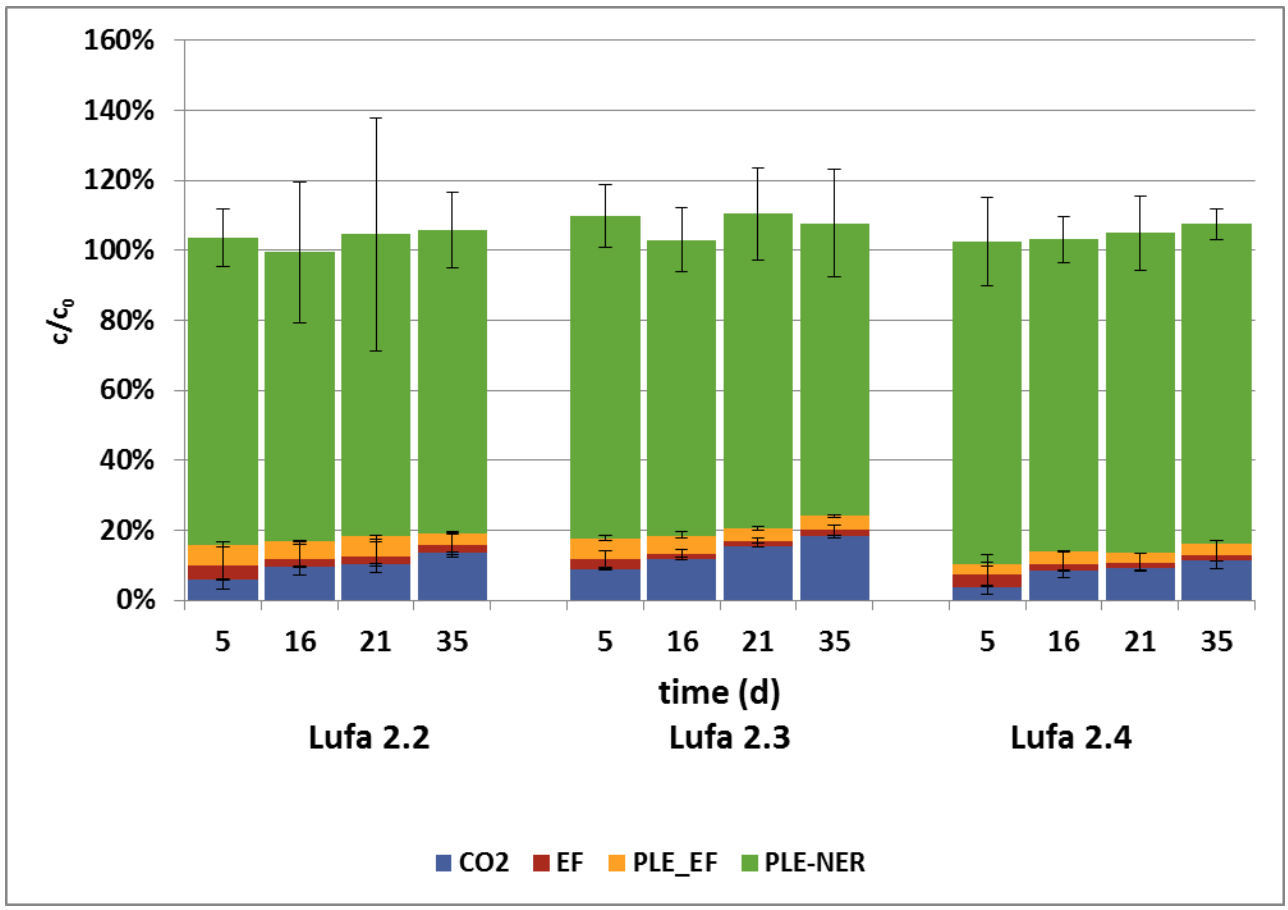
Table A78: Distribution of radioactivity after the transformation test with acetaminophen in Lufa 2.3 following three step batch extraction and subsequent PLE.

	Time (d)	CO_2	SD	EF	SD	PLE-EF	SD	PLE-NER	SD	Sum	SD
ACT Lufa 2.3	5	9%	0%	3%	0%	6%	1%	92%	9%	110%	9%
	16	12%	0%	2%	0%	5%	0%	85%	9%	103%	9%
	21	15%	1%	2%	0%	4%	1%	90%	13%	110%	13%
	35	18%	1%	2%	0%	4%	1%	84%	15%	108%	16%

Table A79: Distribution of radioactivity after the transformation test with acetaminophen in Lufa 2.4 following three step batch extraction and subsequent PLE.

	Time (d)	CO_2	SD	EF	SD	PLE-EF	SD	PLE-NER	SD	Sum	SD
ACT Lufa 2.4	5	4%	0%	3%	0%	3%	0%	92%	12%	102%	13%
	16	8%	0%	2%	0%	4%	0%	89%	6%	103%	7%
	21	9%	0%	2%	0%	3%	1%	91%	11%	105%	11%
	35	11%	0%	2%	0%	3%	0%	91%	4%	107%	5%

Figure A66: Distribution of radioactivity after the transformation test with acetaminophen in Lufa 2.2, Lufa 2.3 and Lufa 2.4 following three step batch extraction and subsequent PLE.



A4.4.5 Results of the transformation test of acetaminophen including separated batch extraction and PLE

Table A80: Distribution of radioactivity after the transformation test with acetaminophen in Lufa 2.2 following three step batch extraction with separated fractions of the extractable fraction and subsequent PLE.

	Time (d)	CO_2	SD	EF1	SD	EF2	SD	EF3	SD	PLE-EF	SD	PLE-NER	SD	Sum	SD
ACT Lufa 2.2	5	6%	0%	2%	0%	2%	0%	1%	0%	6%	1%	88%	8%	104%	8%
	16	10%	0%	1%	0%	1%	0%	1%	0%	5%	1%	83%	20%	99%	20%
	21	10%	0%	1%	0%	1%	0%	1%	0%	6%	1%	86%	33%	105%	33%
	35	14%	0%	1%	0%	1%	0%	1%	0%	3%	0%	87%	11%	106%	11%

Figure A67: Distribution of radioactivity after the transformation test with acetaminophen in Lufa 2.2 following three step batch extraction with separated fractions of the extractable fraction and subsequent PLE.

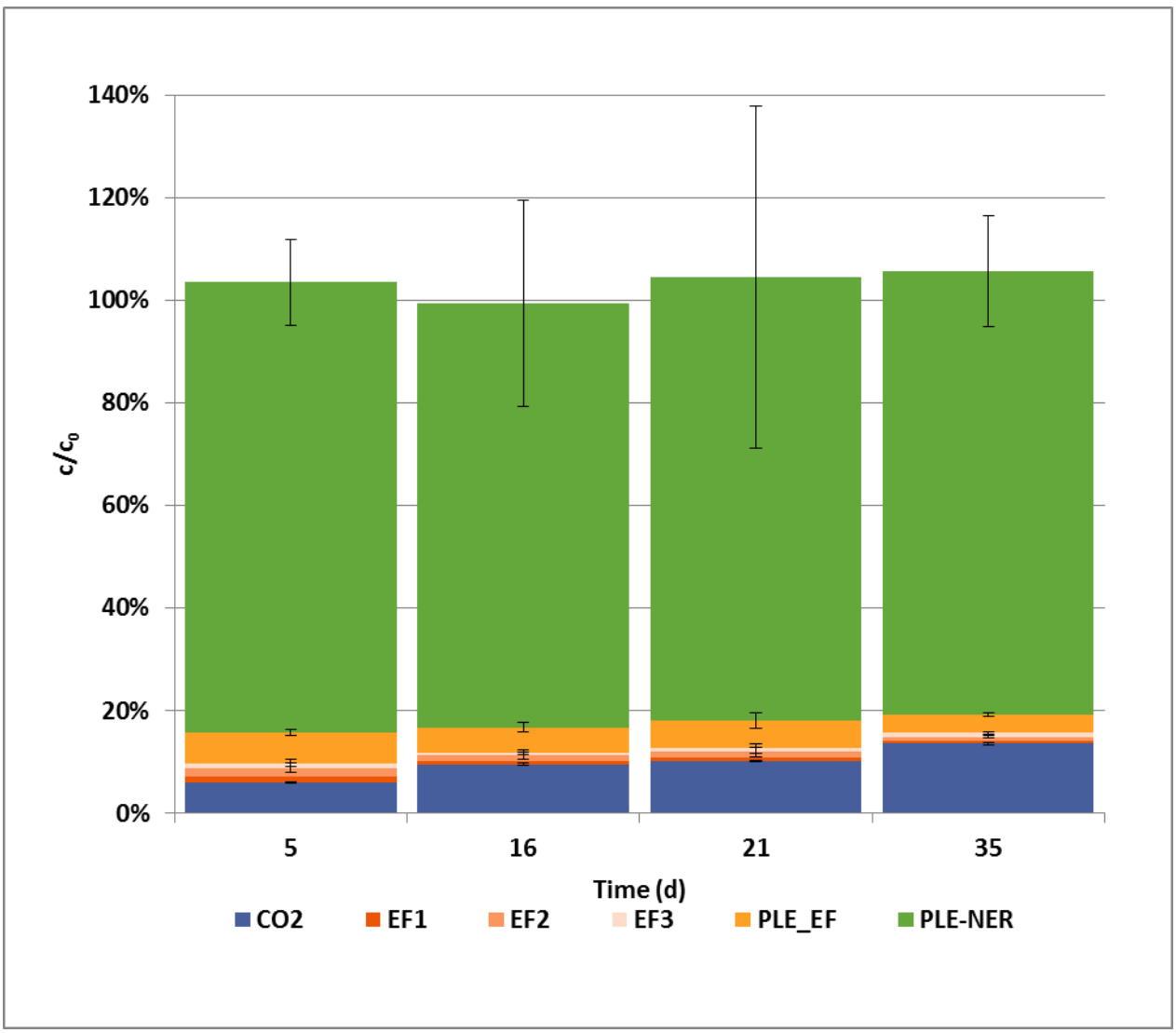


Table A81: Distribution of radioactivity after the transformation test with acetaminophen in Lufa 2.3 following three step batch extraction with separated fractions of the extractable fraction and subsequent PLE.

	Time (d)	CO_2	SD	EF1	SD	EF2	SD	EF3	SD	PLE-EF	SD	PLE-NER	SD	Sum	SD
ACT Lufa 2.3	5	9%	0%	1%	0%	1%	0%	1%	0%	6%	1%	92%	9%	110%	9%
	16	12%	0%	0%	0%	1%	0%	1%	0%	5%	0%	85%	9%	103%	9%
	21	15%	1%	0%	0%	1%	0%	1%	0%	4%	1%	90%	13%	110%	13%
	35	18%	1%	1%	0%	0%	0%	1%	0%	4%	1%	84%	15%	108%	16%

Figure A68: Distribution of radioactivity after the transformation test with acetaminophen in Lufa 2.3 following three step batch extraction with separated fractions of the extractable fraction and subsequent PLE.

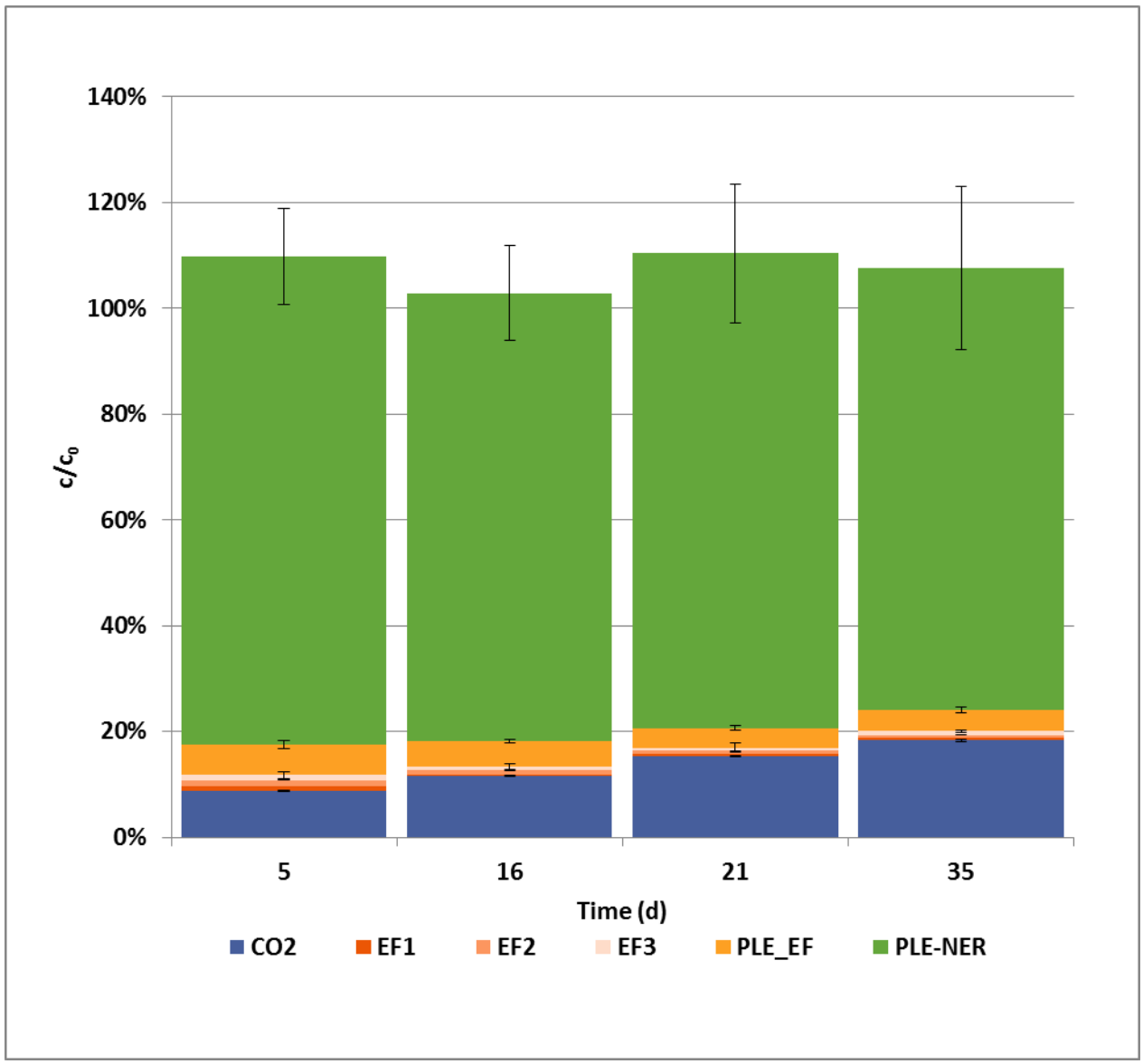


Table A82: Distribution of radioactivity after the transformation test with acetaminophen in Lufa 2.4 following three step batch extraction with separated fractions of the extractable fraction and subsequent PLE.

	Time (d)	CO_2	SD	EF1	SD	EF2	SD	EF3	SD	PLE-EF	SD	PLE-NER	SD	Sum	SD
ACT Lufa 2.4	5	4%	0%	1%	0%	1%	0%	1%	0%	3%	0%	92%	12%	102%	13%
	16	8%	0%	1%	0%	1%	0%	1%	0%	4%	0%	89%	6%	103%	7%
	21	9%	0%	1%	0%	1%	0%	1%	0%	3%	1%	91%	11%	105%	11%
	35	11%	0%	0%	0%	1%	0%	0%	0%	3%	0%	91%	4%	107%	5%

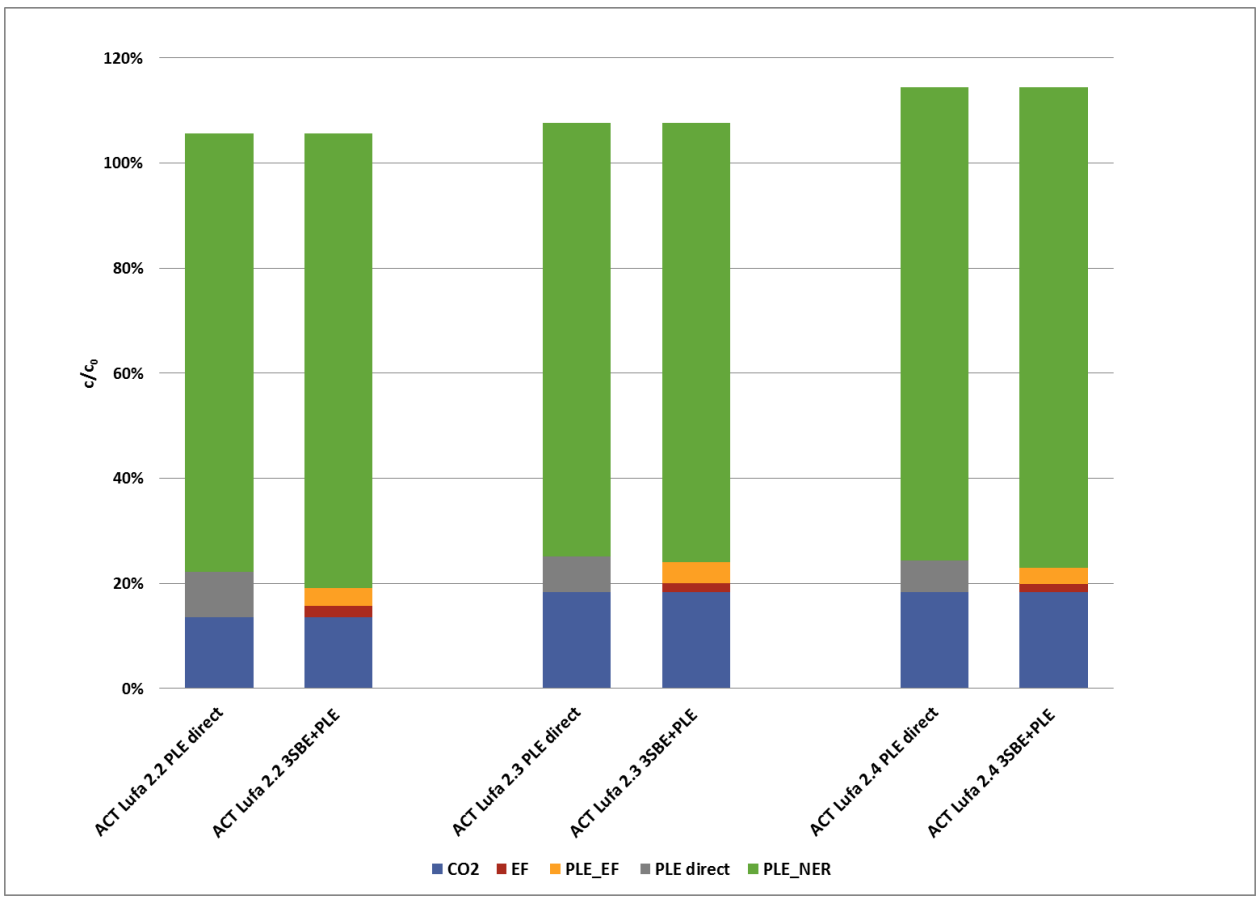
Figure A69: Distribution of radioactivity after the transformation test with acetaminophen in Lufa 2.4 following three step batch extraction with separated fractions of the extractable fraction and subsequent PLE.

A4.4.6 Results of the transformation test of triclosan after direct PLE

Table A83: Comparison of the extraction efficiency between the three step batch extraction and the direct PLE with acetaminophen.

	Time (d)	CO_2	SD	3SBE-EF	SD	PLE-EF	SD	PLE-NER	SD	Sum	SD
ACT Lufa 2.2 PLE direct	35	14%	0%	-	-	9%	1%	83%	5%	106%	5%
ACT Lufa 2.2 3SBE+PLE	35	14%	0%	2%	0%	3%	0%	87%	11%	106%	11%
ACT Lufa 2.3 PLE direct	35	18%	1%	-	-	7%	1%	82%	6%	108%	6%
ACT Lufa 2.3 3SBE+PLE	35	18%	1%	2%	0%	4%	1%	84%	15%	108%	16%
ACT Lufa 2.4 PLE direct	35	18%	1%	-	-	6%	0%	90%	4%	107%	5%
ACT Lufa 2.4 3SBE+PLE	35	18%	1%	2%	0%	3%	0%	91%	4%	107%	5%

Figure A70: Comparison of the extraction efficiency between the three step batch extraction and the direct PLE with acetaminophen.



A4.5 Silylation

A4.5.1 Mobilised radioactivity by silylation for triclosan

Table A84: Distribution of radioactivity after the transformation test with triclosan in Lufa 2.2, following three step batch extraction, PLE and silylation.

	Time (d)	Mobilise radioactivity	SD	Remaining radioactivity	SD
TCS Lufa 2.2	7	1%	0%	13%	3%
	34	2%	0%	17%	4%
	60	2%	0%	19%	2%
	100	3%	1%	25%	3%

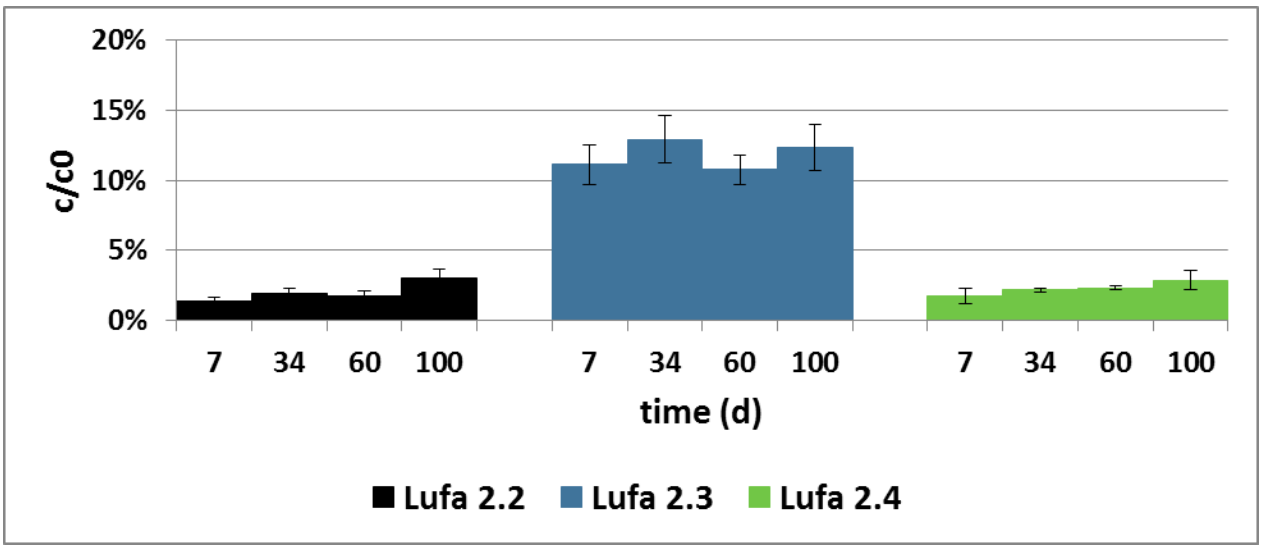
Table A85: Distribution of radioactivity after the transformation test with triclosan in Lufa 2.3, following three step batch extraction, PLE and silylation.

	Time (d)	Mobilise radioactivity	SD	Remaining radioactivity	SD
TCS Lufa 2.3	7	11%	1%	36%	6%
	34	13%	2%	42%	5%
	60	11%	1%	39%	4%
	100	12%	2%	36%	6%

Table A86: Distribution of radioactivity after the transformation test with triclosan in Lufa 2.4, following three step batch extraction, PLE and silylation.

	Time (d)	Mobilise radioactivity	SD	Remaining radioactivity	SD
TCS Lufa 2.4	7	2%	1%	12%	4%
	34	2%	0%	14%	1%
	60	2%	0%	24%	3%
	100	3%	1%	25%	6%

Figure A71: Mobilisation of radioactivity after the transformation test with triclosan in Lufa 2.2, Lufa 2.3 and Lufa 2.4, following three step batch extraction, PLE and silylation.



A4.5.2 Mobilised radioactivity by silylation for fenoxycarb

Table A87: Distribution of radioactivity after the transformation test with fenoxycarb in Lufa 2.2, following three step batch extraction, PLE and silylation.

	Time (d)	Mobilise radioactivity	SD	Remaining radioactivity	SD
FEC Lufa 2.2	11	4%	1%	40%	9%
	35	4%	1%	34%	6%
	60	3%	1%	40%	11%
	100	3%	0%	38%	4%

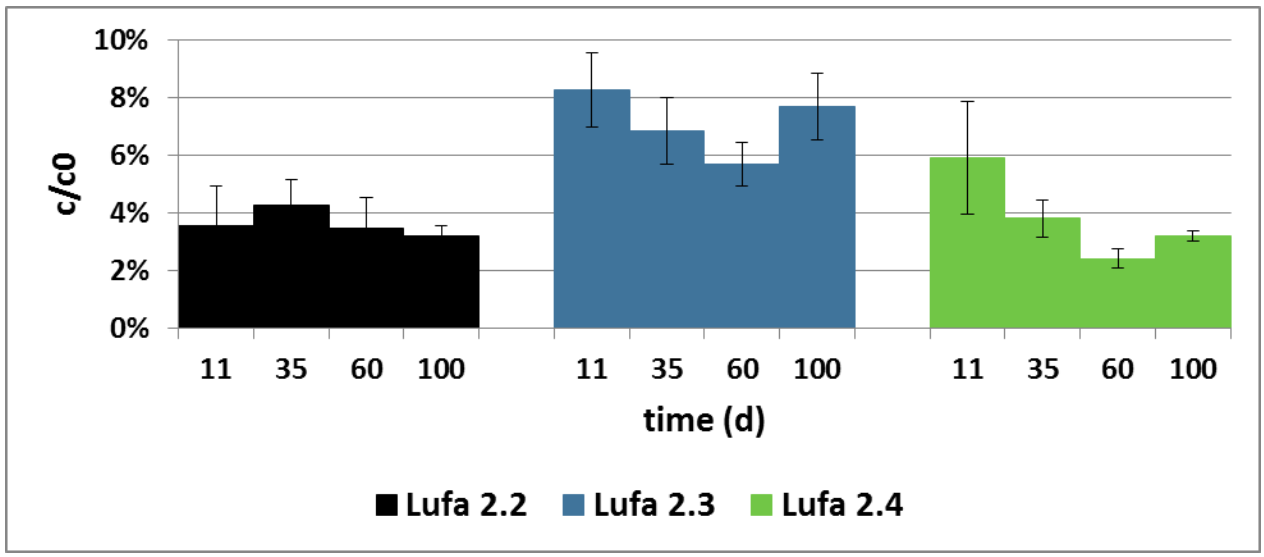
Table A88: Distribution of radioactivity after the transformation test with fenoxycarb in Lufa 2.3, following three step batch extraction, PLE and silylation.

	Time (d)	Mobilise radioactivity	SD	Remaining radioactivity	SD
FEC Lufa 2.3	11	8%	1%	51%	7%
	35	7%	1%	43%	6%
	60	6%	1%	45%	6%
	100	8%	1%	41%	6%

Table A89: Distribution of radioactivity after the transformation test with fenoxycarb in Lufa 2.4, following three step batch extraction, PLE and silylation.

	Time (d)	Mobilise radioactivity	SD	Remaining radioactivity	SD
FEC Lufa 2.4	11	6%	2%	49%	5%
	35	4%	1%	49%	7%
	60	2%	0%	51%	4%
	100	3%	0%	49%	3%

Figure A72: Mobilisation of radioactivity after the transformation test with fenoxycarb in Lufa 2.2, Lufa 2.3 and Lufa 2.4, following three step batch extraction, PLE and silylation.



A4.5.3 Mobilised radioactivity by silylation for acetaminophen

Table A90: Distribution of radioactivity after the transformation test with acetaminophen in Lufa 2.2, following three step batch extraction, PLE and silylation.

	Time (d)	Mobilise radioactivity	SD	Remaining radioactivity	SD
ACT Lufa 2.2	5	4%	0%	84%	8%
	16	4%	1%	79%	20%
	21	4%	1%	83%	33%
	35	5%	1%	81%	11%

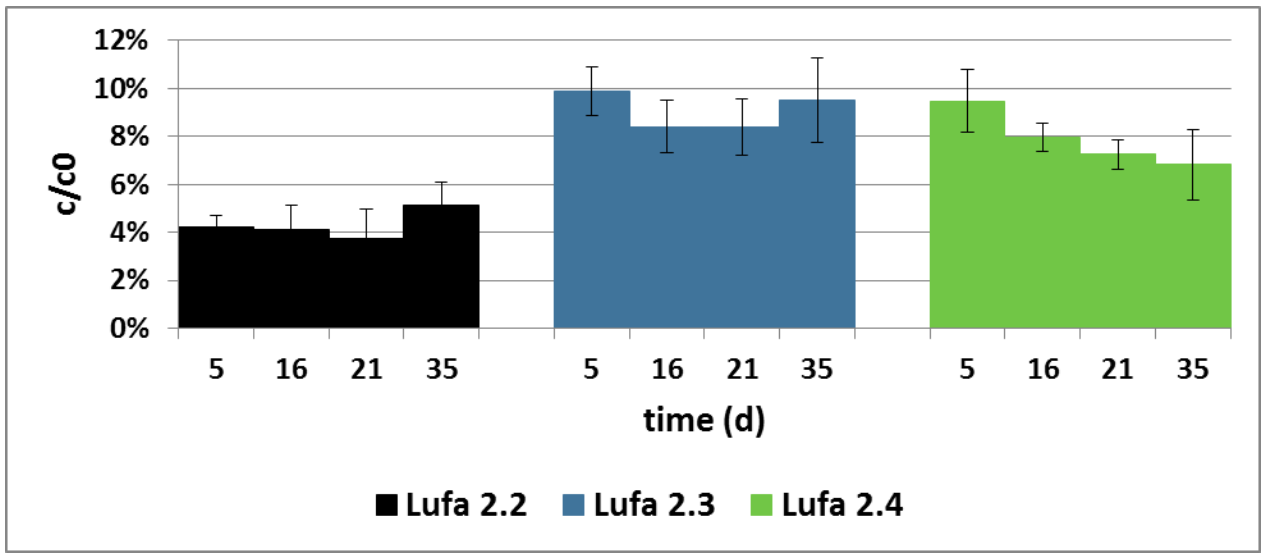
Table A91: Distribution of radioactivity after the transformation test with acetaminophen in Lufa 2.3, following three step batch extraction, PLE and silylation.

	Time (d)	Mobilise radioactivity	SD	Remaining radioactivity	SD
ACTLufa 2.3	5	10%	1%	83%	9%
	16	8%	1%	76%	9%
	21	8%	1%	81%	13%
	35	10%	2%	74%	15%

Table A92: Distribution of radioactivity after the transformation test with acetaminophen in Lufa 2.4, following three step batch extraction, PLE and silylation.

	Time (d)	Mobilise radioactivity	SD	Remaining radioactivity	SD
ACT Lufa 2.4	5	9%	1%	83%	13%
	16	8%	1%	81%	6%
	21	7%	1%	84%	11%
	35	7%	1%	85%	5%

Figure A73: Mobilisation of radioactivity after the transformation test with acetaminophen in Lufa 2.2, Lufa 2.3 and Lufa 2.4, following three step batch extraction, PLE and silylation.



A4.6 EDTA-extraction

A4.6.1 Mobilised radioactivity by EDTA-extraction for triclosan

Table A93: Distribution of radioactivity after the transformation test with triclosan in Lufa 2.2, Lufa 2.3 and 2.4, following three step batch extraction, PLE and EDTA-extraction.

	Time (d)	Mobilise radioactivity	SD	Remaining radioactivity	SD
TCS Lufa 2.2	100	4%	1%	24%	3%
TCS Lufa 2.3	100	14%	4%	34%	7%
TCS Lufa 2.4	100	5%	1%	23%	6%

Figure A74: Distribution of radioactivity after the transformation test with triclosan in Lufa 2.2, Lufa 2.3 and 2.4, following three step batch extraction, PLE and EDTA-extraction.

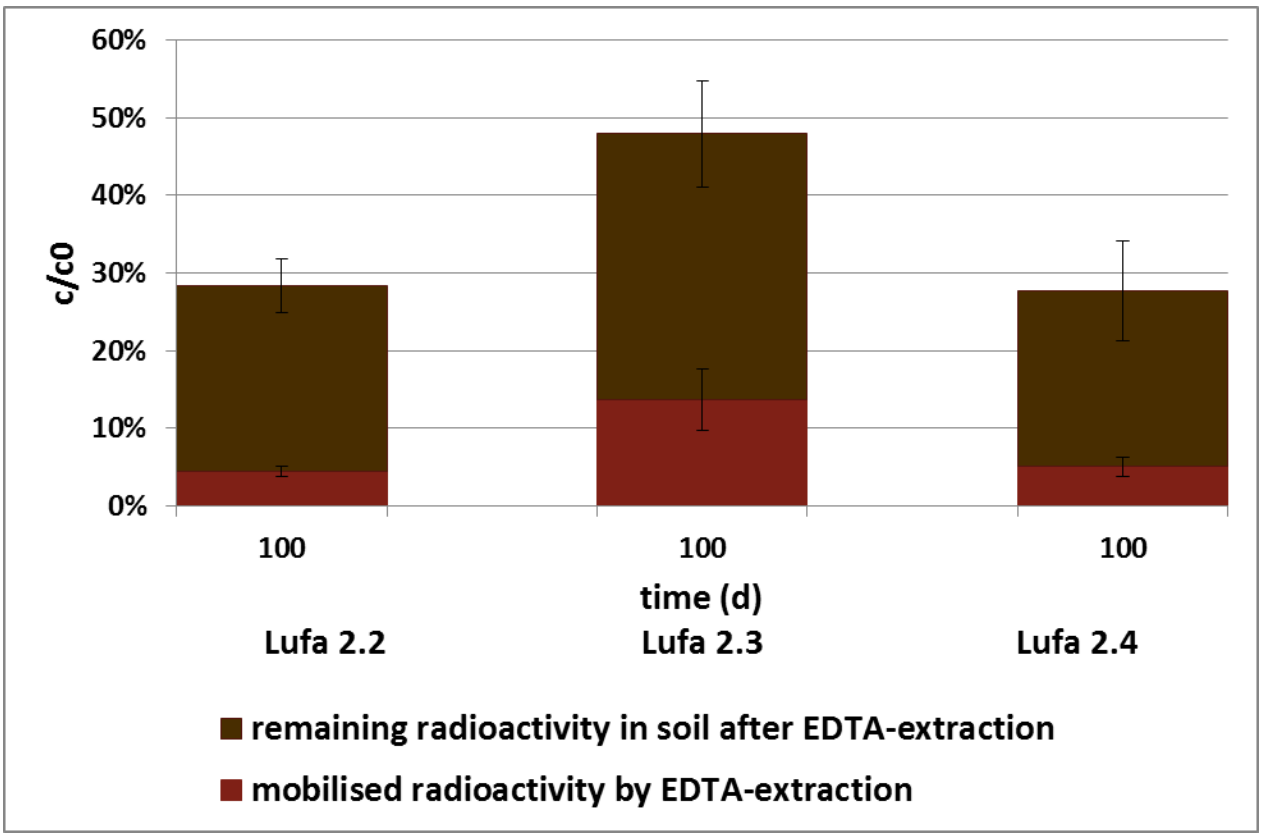


Table A94: Distribution of radioactivity after the transformation test with fenoxycarb in Lufa 2.2, Lufa 2.3 and Lufa 2.4, following three step batch extraction, PLE and EDTA-extraction.

	Time (d)	Mobilise radioactivity	SD	Remaining radioactivity	SD
FEC Lufa 2.2	100	6%	1%	35%	4%
FEC Lufa 2.3	100	10%	1%	39%	6%
FEC Lufa 2.4	100	8%	1%	44%	3%

Figure A75: Distribution of radioactivity after the transformation test with fenoxycarb in Lufa 2.2, Lufa 2.3 and Lufa 2.4, following three step batch extraction, PLE and EDTA-extraction.

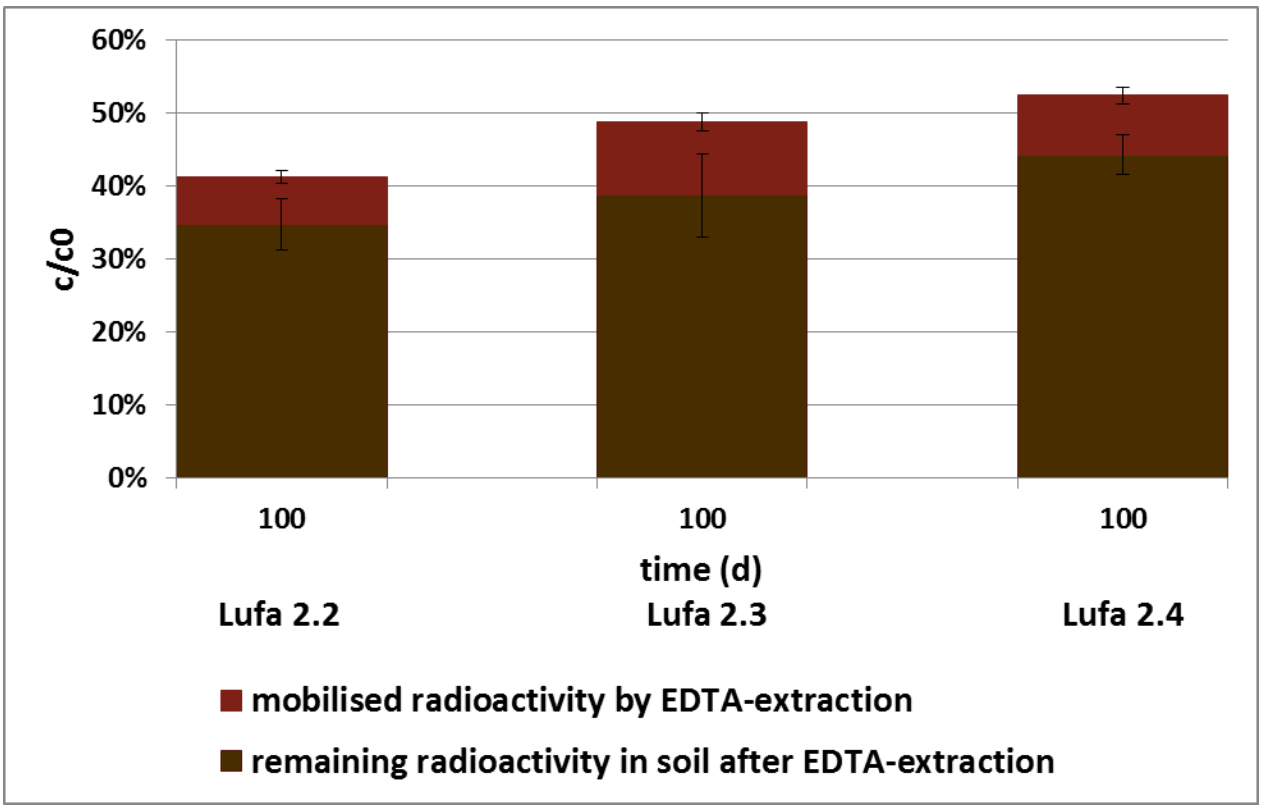
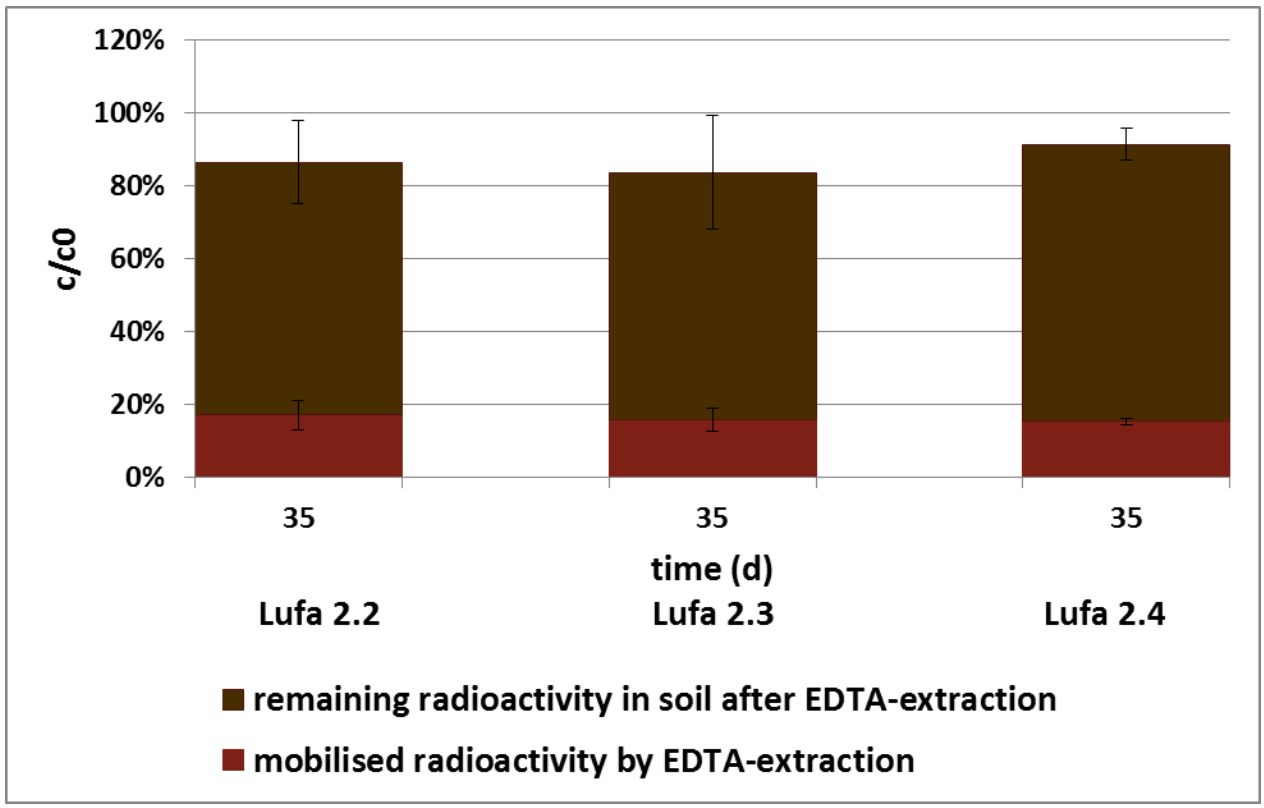


Table A95: Distribution of radioactivity after the transformation test with acetaminophenin Lufa 2.2, Lufa 2.3 and Lufa 2.4, following three step batch extraction, PLE and EDTA-extraction.

	Time (d)	Mobilise radioactivity	SD	Remaining radioactivity	SD
ACT Lufa 2.2	35	17%	4%	69%	12%
ACT Lufa 2.3	35	16%	3%	68%	16%
ACT Lufa 2.4	35	15%	1%	76%	4%

Figure A76: Distribution of radioactivity after the transformation test with acetaminophen in Lufa 2.2, Lufa 2.3 and lufa 2.4, following three step batch extraction, PLE and EDTA-extraction.



A4.7 HCl-hydrolysis

A4.7.1 Mobilised radioactivity by HCl-hydrolysis for triclosan

Table A96: Distribution of radioactivity after the transformation test with triclosan in Lufa 2.2, following three step batch extraction, PLE and HCl-hydrolysis.

	Time (d)	Mobilise radioactivity	SD	Remaining radioactivity	SD
TCS Lufa 2.2	7	4%	1%	10%	3%
	34	8%	2%	11%	4%
	60	6%	1%	15%	2%
	100	8%	1%	20%	3%

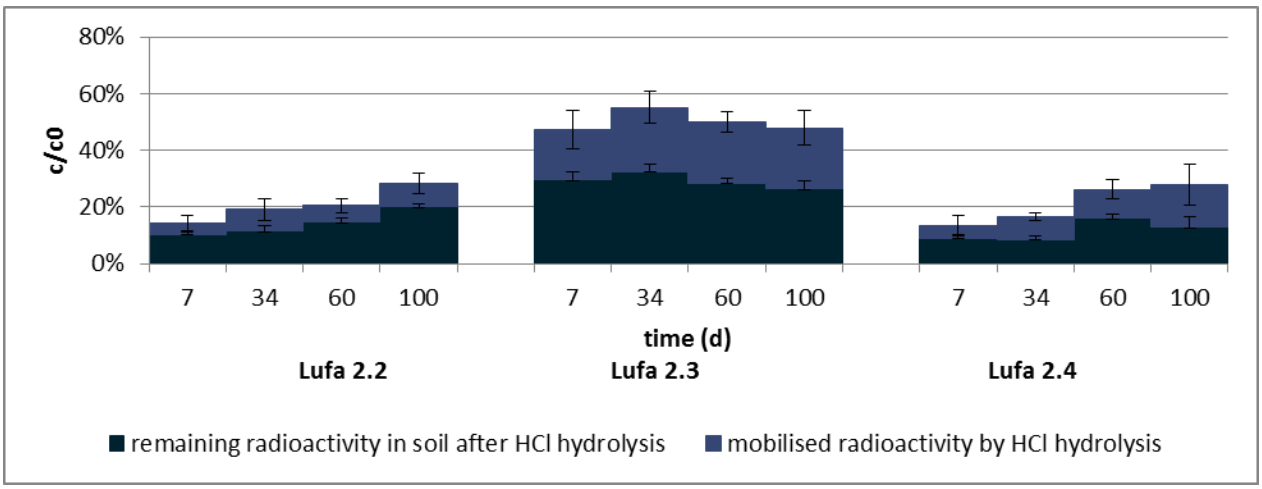
Table A97: Distribution of radioactivity after the transformation test with triclosan in Lufa 2.3, following three step batch extraction, PLE and HCl-hydrolysis.

	Time (d)	Mobilise radioactivity	SD	Remaining radioactivity	SD
TCS Lufa 2.3	7	18%	3%	29%	7%
	34	23%	3%	33%	6%
	60	21%	2%	28%	4%
	100	21%	3%	26%	6%

Table A98: Distribution of radioactivity after the transformation test with triclosan in Lufa 2.4, following three step batch extraction, PLE and HCl-hydrolysis.

	Time (d)	Mobilise radioactivity	SD	Remaining radioactivity	SD
TCS Lufa 2.4	7	4%	1%	9%	4%
	34	8%	1%	9%	1%
	60	10%	1%	16%	3%
	100	15%	4%	13%	7%

Figure A77: Distribution of radioactivity after the transformation test with triclosan in Lufa 2.2, Lufa 2.3 and Lufa 2.4, following three step batch extraction, PLE and HCl-hydrolysis.



A4.7.2 Mobilised radioactivity HCl-hydrolysis for fenoxycarb

Table A99: Distribution of radioactivity after the transformation test with fenoxycarb in Lufa 2.2, following three step batch extraction, PLE and HCl-hydrolysis.

	Time (d)	Mobilise radioactivity	SD	Remaining radioactivity	SD
FEC Lufa 2.2	11	19%	4%	25%	10%
	35	20%	4%	18%	7%
	60	17%	6%	27%	13%
	100	18%	2%	23%	4%

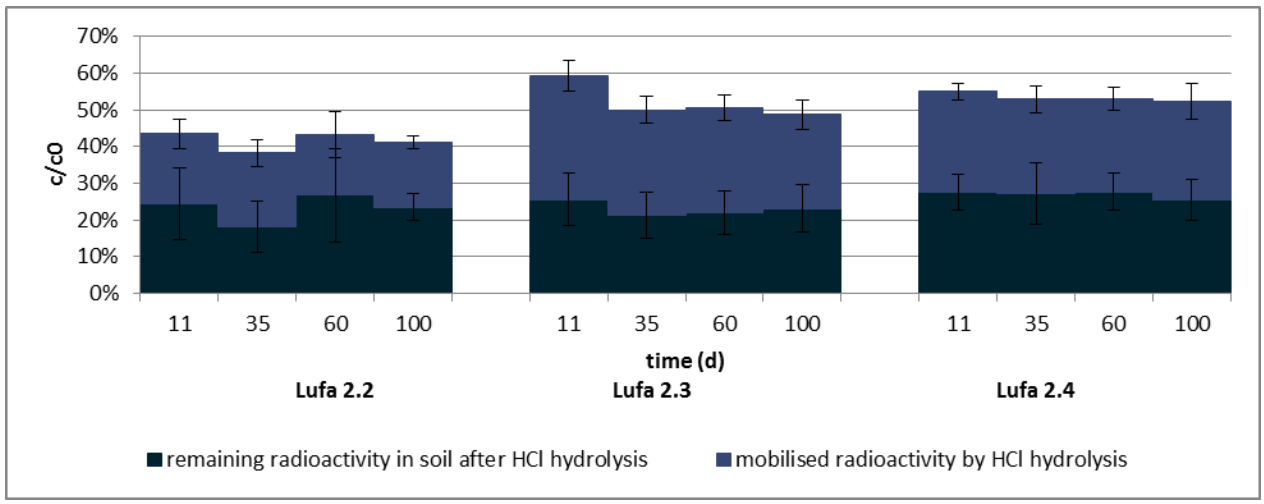
Table A100: Distribution of radioactivity after the transformation test with fenoxycarb in Lufa 2.3, following three step batch extraction, PLE and HCl-hydrolysis.

	Time (d)	Mobilise radioactivity	SD	Remaining radioactivity	SD
FEC Lufa 2.3	11	34%	4%	26%	7%
	35	29%	4%	21%	6%
	60	29%	3%	22%	6%
	100	26%	4%	23%	6%

Table A101: Distribution of radioactivity after the transformation test with fenoxycarb in Lufa 2.4, following three step batch extraction, PLE and HCl-hydrolysis.

	Time (d)	Mobilise radioactivity	SD	Remaining radioactivity	SD
FEC Lufa 2.4	11	28%	2%	27%	5%
	35	26%	4%	27%	8%
	60	25%	3%	28%	5%
	100	27%	5%	25%	6%

Figure A78: Distribution of radioactivity after the transformation test with fenoxycarb in Lufa 2.2, Lufa 2.3 and Lufa 2.4, following three step batch extraction, PLE and HCl-hydrolysis.



A4.7.3 Mobilised radioactivity by HCl-hydrolysis for acetaminophen

Table A102: Distribution of radioactivity after the transformation test with acetaminophen in Lufa 2.2, following three step batch extraction, PLE and HCl-hydrolysis.

	Time (d)	Mobilise radioactivity	SD	Remaining radioactivity	SD
ACT Lufa 2.2	5	23%	3%	65%	9%
	16	26%	7%	56%	21%
	21	24%	10%	63%	35%
	35	22%	3%	65%	11%

Table A103: Distribution of radioactivity after the transformation test with acetaminophen in Lufa 2.3, following three step batch extraction, PLE and HCl-hydrolysis.

	Time (d)	Mobilise radioactivity	SD	Remaining radioactivity	SD
ACTLufa 2.3	5	35%	7%	58%	11%
	16	36%	4%	49%	9%
	21	34%	6%	56%	14%
	35	36%	10%	48%	18%

Table A104: Distribution of radioactivity after the transformation test with acetaminophen in Lufa 2.4, following three step batch extraction, PLE and HCl-hydrolysis.

	Time (d)	Mobilise radioactivity	SD	Remaining radioactivity	SD
ACT Lufa 2.4	5	31%	5%	61%	13%
	16	29%	4%	60%	7%
	21	28%	5%	63%	12%
	35	27%	3%	64%	5%

Figure A79: Distribution of radioactivity after the transformation test with acetaminophen in Lufa 2.2, Lufa 2.3 and Lufa 2.4, following three step batch extraction, PLE and HCl-hydrolysis.