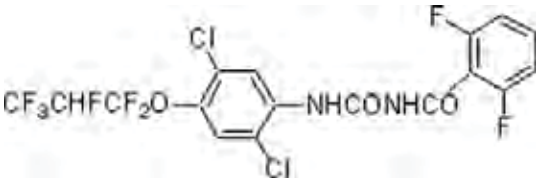
LUFENURON (286)

The first draft was prepared by Mr Christian Sieke, Federal Institute for Risk Assessment, Berlin, Germany

EXPLANATION

Lufenuron is an insect growth inhibitor that is active against larvae of Lepidoptera and Coleoptera. When ingested, lufenuron interferes with chitin synthesis, and prevents larvae from moulting. It was considered for the first time by the 2015 JMPR for toxicology and residues.

IDENTITY

ISO common name	Lufenuron
Chemical name	
IUPAC	(*RS*)-1-[2,5-dichloro-4-(1,1,2,3,3,3-hexafluoropropoxy)phenyl]-3-(2,6-difluorobenzoyl)urea
CA	N-[[[2,5-dichloro-4-(1,1,2,3,3,3-hexafluoropropoxy)phenyl]amino]carbonyl]-2,6-difluorobenzamide
CAS No.	103055-07-8
CIPAC No.	704
Structural formula	
Molecular formula	$C_{17}H_8Cl_2F_8N_2O_3$
Molecular mass	511.15 g/mol

Lufenuron consists of a pair of enantiomers. A chiral centre exists at the 2-position of the hexafluoropropoxy side-chain. Lufenuron technical active ingredient is manufactured under non-stereospecific conditions giving a racemate (R:S 50:50).

Specifications

Specifications for lufenuron were not yet developed by FAO.

PHYSICAL AND CHEMICAL PROPERTIES

Property	*Results*	*Method (test material)*	*Reference*
Melting point	168.7–169.4 °C	OECD 102 (Batch AMS 266/102, 99.7% purity)	Das, R, 1998 LUFEN_001
Boiling point & temperature of decomposition	Not measurable (decomposes) Decomposition starts to occur at about 242 °C	OECD 103 (Batch AMS 266/102, 99.7% purity)	Das, R, 2000 LUFEN_002
Appearance	Appearance—pure active substance: white fine powder (PAI)	Visual inspection (Batch AMS 266/102, 99.7% purity)	Das, R, 1998 LUFEN_003
Relative density	1.67 g cm^{-3} at 20 °C	OECD Guideline for Testing of Chemicals 109 (Batch AMS 266/102, 99.7% purity)	Fueldner, 1998, LUFEN_004

Property	*Results*	*Method (test material)*	*Reference*
Vapour pressure	$< 4 \times 10^{-6}$ Pa at 25 °C	OECD Guideline for Testing of Chemicals 104A (Batch AMS 266/101, 99.7% purity)	Geoffroy, 1992, LUFEN_005
Henry´s Law Coefficient	$< 4.4 \times 10^{-2}$ Pa m^3 mol^{-1}	Calculation	Born, 2008, LUFEN_006
Solubility in water including effect of pH	pH 5: 54 µg/L (25 °C) pH 7: 46 µg/L (25 °C) pH 9: 64 µg/L (25 °C)	OECD Guideline for Testing of Chemicals 105 (Batch AMS 266/102, 99.7% AI)	Das, R, 2002, LUFEN_007
Partition coefficient n-octanol / water	log P_{OW}=5.12 (25 °C, pure water)	OECD Guideline for Testing of Chemicals 117 (Batch AMS266/102, 99.7% AI)	Rodler, 1992, LUFEN_009
Dissociation constant	$pK_{a,1}$=10.18 at 20 °C in methanol:water mixtures	OECD Guideline for Testing Chemicals 112 (Batch AMS266/102, 99.7% AI)	Martin, 2002, LUFEN_010
UV/VIS absorption (max.) incl. ε	Wavelength [nm] / molar extinction coefficient [L/mol · cm] neutral solution 210 37293 255 16417 295 1648 acidic solution 210 30588 255 15165 295 2220 basic solution 230 20658 267 22440 295 4871 No absorption maximum between 350 nm and 750 nm was observed	OECD Guideline for Testing Chemicals 101 (Batch AMS266/102, 99.7% AI)	Oggenfuss, 2002, LUFEN_011
	Wavelength [nm] / molar extinction coefficient [L/mol · cm] methanol 290 5212 305 499 Absorption levels out above 300 nm	JMAFF Agchem Test Guidelines 12 (Batch ILA-178.3, 98.9% AI)	Mamouni, 2004, LUFEN_012
Photochemical degradation in water	pH 7, 25 °C (buffer) $t_{1/2}$ 11.2 ± 1.3 days (natural sunlight at 30–50 °N, 12:12 photocycle)	JMAFF Agchem Test Guidelines 12 (Batch ILA-178.3, 98.9% AI)	Mamouni, 2004, LUFEN_012
	Sterile buffer pH 7, 25 °C (Xenon arc light, λ≥ 290 nm) DT_{50}: 16 d continuous Xenon arc light equivalent to ca. 34 d clear summer sunlight at 30–40 °N)	EPA 540/9-82-021 ([^{14}C-dichlorophenyl]-label, AMS 266/101, 99.5% AI)	Ellgehausen, 1994, LUFEN_013
	Sterile buffer pH 7, 25 °C (Xenon arc light, λ≥ 290 nm) DT_{50}: 10.3 d continuous Xenon arc light equivalent to ca. 18 d clear summer sunlight at 30–40 °N)	EPA 540/9-82-021 ([^{14}C-dichlorophenyl]-label, AMS 266/101, 99.5% AI)	Ellgehausen, 1994, LUFEN_014
Quantum yield of direct photo-transformation	ϕ=0.0026 in 0.01 M phosphate buffer/ethanol mixture (1:1 v/v), λ=290 nm	UBA Draft Test Guideline "Phototrans-formation of Chemicals in Water, Part A, Direct Phototransformation", Berlin, FRG 1990 (Batch AMS 266/101, 99.5% AI)	Abildt, 1995, PYMET_015

Property	*Results*	*Method (test material)*	*Reference*
Solubility in organic solvents	The solubility in different organic solvents at 25 °C was determined to be : acetone 460 g/lL dichloromethane 84 g/lL ethyl acetate 330 g/lL hexane 0.10 g/lL methanol 52 g/lL octanol 8.2 g/lL toluene 66 g/lL	In-house method (Batch P.704809, 99.5% AI)	Kettner, 2000, LUFEN_008

Formulations

Lufenuron is primarily available as the following EC formulations:

Formulations registered containing lufenuron as active ingredient.

Formulation	*Content of active ingredients*	*Trade names*
EC	50 g/L	Match EC, Match 5 EC, Curyom 550 EC

METABOLISM AND ENVIRONMENTAL FATE

Metabolism studies were conducted using [dichlorophenyl-^{14}C]-lufenuron (dichlorophenyl-label) and [difluorophenyl-^{14}C]-lufenuron (difluorophenyl-label). The position of the label for both substances is presented in the following figures:

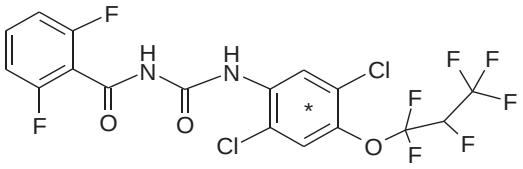


Figure 1 [dichlorophenyl -^{14}C]-lufenuron

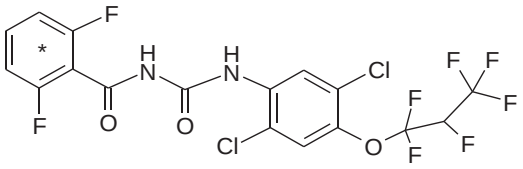
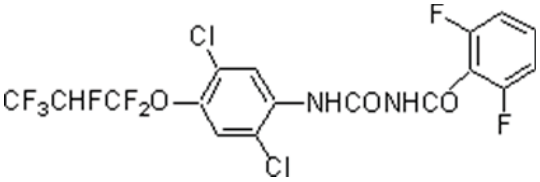
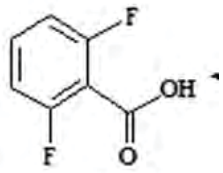
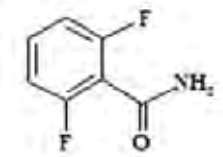
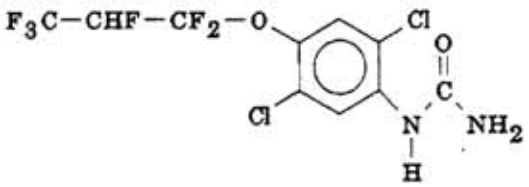
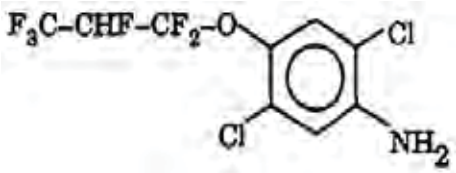
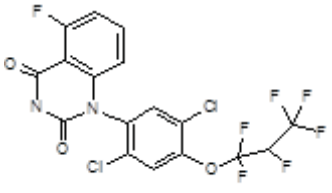


Figure 2 [difluorophenyl-^{14}C]-lufenuron

Chemical names, structures and code names of metabolites and degradation products of lufenuron are shown below.

Code Names	*Chemical Abstracts Name (IUPAC Name), molecular formula, molar mass*	*Structure*	*Where found*
Parent lufenuron, CGA 184699	(RS)-1-[2,5-dichloro-4-(1,1,2,3,3,3-hexafluoropropoxy) phenyl] -3-(2,6-difluorobenzoyl)urea		Cabbage leaves tomato fruit Goat—kidney, urine and

Code Names	*Chemical Abstracts Name (IUPAC Name), molecular formula, molar mass*	*Structure*	*Where found*
			faeces Hen—kidney, egg white, excreta
CGA149776	2,6-Difluoro-benzoic acid		Goat—faeces Hen—excreta Soil
CGA149772	2,6-Difluoro-benzamide		Goat—faeces Hen—egg white Soil
CGA238277	2,5-Dichloro-4-(1,1,2,3,3,3-hexafluoropropoxy) -phenyl-urea		Goat—faeces, Hens—kidney
CGA224443	N-[2,5-dichloro-4-(1,1,2,3,3,3-hexafluoropropoxy) -benzenamine		Soil
CGA301018			Water

Environmental fate in soil

For the investigation of the environmental fate of lufenuron the Meeting received studies on soil photolysis, hydrolysis, aerobic soil metabolism and the behaviour in confined rotational crops.

Soil photolysis

The soil surface photolytic behaviour of lufenuron on moist and dry soil was investigated by Ellgehausen (1994, LUFEN_026) using [^{14}C]dichlorophenyl ring-labelled lufenuron.

Moist and dry soil was dosed with radio-labelled lufenuron at 5 µg/cm^2 (equivalent to 500 g ai/ha). The samples were irradiated continuously at 25 °C for up to 17 days. Samples were taken at 0, 5, 9, 14, 19 and 26 days. One dark control sample was prepared in parallel.

For analysis, the soil layer was extracted by shaking with acetone (twice) followed by a mixture of acetone:water (80:20 v/v). Following each extraction step, the samples were centrifuged and the supernatants combined. The supernatants were concentrated, partitioned with dichloromethane and the radioactivity in each phase quantified by LSC. Characterisation and quantification of the photo-degradation products was conducted by HPLC.

The percentage recovery of the applied radioactivity is presented in Tables 1 and 2 and ranged from 99.4–103.8%. The recovery from the dark control plates was > 99% at the end of the study.

Table 1 Distribution of Applied Recovery in dry soil after Continuous Irradiation and results of the dark control sample

Degradate	Incubation period (hours)						Dark control
	0	168	240	288	336	408	
Lufenuron	99.55	91.21	87.97	88.71	85.23	85.46	99.3
CO_2	0	3.17	4.24	5.34	6.47	7.79	0.0
Unidentified degradates [a]	0.86	2.38	5.68	3.49	6.03	6.28	0.72
Unextracted	0.06	4.91	4.62	4.11	5.15	4.2	0.93
Organic volatiles	0	0.02	0.03	0.04	0.05	0.07	0.0
Total	100.5	101.7	102.5	101.7	102.9	103.8	100.9

[a] At least three components, none of which exceeded 3.8% AR

Table 2 Distribution of Applied Recovery in moist soil after Continuous Irradiation

Degradate	Incubation period (hours)					
	0	120	216	336	456	624
Lufenuron	96.95	93.99	93.25	91.76	90.9	90.05
CO_2	0	0.27	0.55	0.85	1.19	1.76
Unidentified degradates [a]	2.39	4.15	3.85	4.63	4.51	4.29
Unextracted	0.05	2.56	3.18	4.04	4.11	5.02
Organic volatiles	0	0	0	0	0	0
Total	99.4	101.0	100.8	101.3	100.7	101.1

[a] At least five components, none of which exceeded 1.9% AR

In a second experiment conducted by Ellgehausen (1994, LUFEN_027) [^{14}C]difluorophenyl-labelled lufenuron was used to investigate its behaviour under soil photolysis. The experimental conditions and analytical methods were identical to the ones used in the previous study for the [^{14}C]dichlorophenyl-label, however only dry soil was investigated.

The percentage recovery of applied radioactivity is presented in the following table and ranged from 99.7 to 102.1%. The recovery from the dark control plates was 101% at the end of the study.

Table 3 Distribution of Applied Recovery in Dry soil after Continuous Irradiation

Degradate	Incubation period (hours)						Dark control
	0	120	292	309	381	453	
Lufenuron	94.2	90.7	89.6	88.6	81.7	84.0	97.2
CGA149772 [a]	2.23	6.07	6.50	7.08	11.2	7.14	1.4
CO_2	0	1.32	2.06	3.85	4.85	6.34	0.0

Unidentified degradates [b]	3.2	1.7	1.4	0.51	1.9	1.8	1.5
Unextracted	0.04	2.25	1.77	1.94	2.37	2.14	0.96
Organic volatiles	0	0.01	0.02	0.04	0.05	0.07	0.0
Total	99.7	102.0	101.4	102.0	102.1	101.5	101.0

[a] The values in this row have not been adjusted for the 1.1% present in the starting material

[b] At least five components, none of which exceeded 1.6% AR

The amounts of lufenuron recovered decreased very slowly from 94.2% AR to 84.0% AR after 18.9 days continuous irradiation. CGA149772, the difluorobenzamide metabolite, reached a maximum of 11.2% AR after 15.8 days then decreased to 7.1% AR at the end of the study. A maximum 6.3% of carbon dioxide was evolved.

Hydrolysis

The stability of lufenuron in sterile buffer solutions was investigated using [dichlororphenyl-^{14}C] and [difluorophenyl-^{14}C]-lufenuron (Ellgehausen, 1992, LUFEN_025).

The test compounds were incubated under sterile conditions in buffer solutions contained in brown glass test tubes. A range of pH (5, 7 and 9) and temperature (25 ºC) conditions were applied to both difluorophenyl-labelled and dichlorophenyl-labelled lufenuron. In addition, a few experiments were conducted under more extreme conditions (pH 1 and 13) and temperature (50 and 70 °C) although not every combination was tested. Lufenuron and its degradation products were partitioned with dichloromethane and the amounts in each phase quantified by LSC and HPLC. Degradates were characterized, after derivatisation where necessary, by MS or GC-MS.

For the samples incubated at 25 °C, both labels showed virtually no degradation at pH 5, 7 and 9. Over 93% of the initial radioactivity was recovered as unchanged lufenuron. Only at pH 9, minor amounts of CGA238277 (3.9% AR) and CGA224443 (1.8% AR) for the dichlorophenyl-label and CGA149776 (3.8% AR) for the difluorophenyl-label were found.

Under more extreme conditions the parent substance was stable at pH 1 and 70 °C, representing more than 90% of the radioactivity after up to 168 hours. At pH 9 an accelerated degradation was observed. An overview of the degradation for the dichlorophenyl-label is presented in Tables 4 and 5, while the difluorophenyl-label results are presented in Tables 6 and 7.

Table 4 Hydrolysis of [^{14}C]dichlorophenyl-lufenuron at pH 9 and 50 °C (%AR)

Time (hours)	Lufenuron	CGA224443	CGA301018	CGA238277	Total
0	101.04	0	0	0	101.04
4	97.07	0	0	3.42	100.49
6	96.58	3.07	0	2.77	102.42
8	96.96	1.42	0	3.04	101.42
24	87.59	5.15	2.02	6.16	100.92
32	85.74	5.47	2.33	7.80	101.34
48	81.94	6.16	3.63	9.18	100.91
72	71.02	9.53	4.01	15.75	100.31
78	68.86	9.28	5.95	16.74	100.83
102	60.83	13.83	5.59	17.86	98.11
150	57.85	15.05	5.96	18.86	97.72
174	53.47	15.99	7.36	21.29	98.11

Table 5 Hydrolysis of [^{14}C]dichlorophenyl-lufenuron at pH 9 and 70 °C (%AR)

Time (hours)	Lufenuron	CGA224443	CGA301018	CGA238277	Unresolved	Total
0	99.12	0	0	0	0.88	100
2	73.16	11.27	4.35	10.08	1.12	99.98
4	46.99	24.14	9.16	18.22	1.22	99.73
7	30.3	33.56	10.76	24.08	1.38	100.08

24	10.5	46.74	14.97	24.98	1.77	98.96
48	7.29	53.11	14.67	13.68	3.51	92.26
72	8.75	49.03	16.09	19.16	1.8	94..83
96	10.95	51.62	15.75	10.68	2.09	91.09
120	1.77	60.94	15.35	8.96	4.44	91.46

Table 6 Hydrolysis of [^{14}C]difluorophenyl-lufenuron at pH 9 and 50 °C (%AR)

Time (hours)	Lufenuron	CGA301018	CGA149776	CGA149772	Total
0	98.46	0	0	0	98.46
24	70.40	3.43	13.63	10.59	98.05
48	56.98	4.67	21.54	16.08	99.27
72	33.42	7.47	32.63	24.77	98.29
96	18.63	8.82	41.33	30.33	99.11
120	24.05	7.89	39.13	27.5	98.57
144	18.86	9.38	40.29	30.22	98.75
168	7.33	10.89	45.95	34.86	99.03
192	14.65	11.01	41.5	31.17	98.33
216	14.5	11.15	39.55	33.44	98.64

Table 7 Hydrolysis of [^{14}C]difluorophenyl-lufenuron at pH 9 and 70 °C (%AR)

Time (hours)	Lufenuron	CGA301018	CGA301020	CGA149776	CGA149772	Total
0	99.97	0	0	0	0	99.97
2	21.81	11.25	0	24.72	42.71	100.49
4	17.33	11.99	0	25.69	45.74	100.75
7	6.44	11.27	0	30.4	52.54	100.65
24	0	15.08	0	29.57	55.24	99.89
48	0	14.4	0	32.29	53.29	99.98
72	0	14.57	0	30.76	55.18	100.51
96	0	12.97	1.77	31.84	51.68	98.27
120	0	12.43	1.29	31.22	54.49	99.43

In the experiments conducted at a pH of 13 with up to 70 °C incubation temperature, lufenuron was completely degraded within the first 24 hours. The primary hydrolysis products formed were CGA239786 (up to 51% AR after 96 h) and CGA301020 (up to 19% AR after 32 h) for the [^{14}C]dichlorophenyl-label and CGA149776 (up to 49% AR after 2.5 h) for the [^{14}C]difluorophenyl-label.

Aerobic soil metabolism

In a first set of studies the aerobic soil metabolism of lufenuron was investigated in two microbial active soil types and in their sterilised form.

Ref.: Ellgehausen (1991, LUFEN_028)

Test material: [^{14}C]dichlorophenyl-lufenuron Dose rate: 1 mg/kg

Duration: 361 days Temp: 20 °C

Moisture: 44.8% Soil: Collombey (sandy loam, micro. active)

pH 7.2 Organic carbon: 3.0%

Half-live (parent): 24 days two 1st order compartment model) ^{14}C accountability: 99–107%

% lufenuron remaining: 8.2% after 361 days

% mineralisation: up to 9.9% after 361 days

% unextracted: up to 70.7% after 240 days

Metabolites	Max (% TRR)	Day
CGA238277	24.3	14
CGA224443	26.9	59

Ref.: Ellgehausen (1991, LUFEN_028)

Test material: [^{14}C]dichlorophenyl-lufenuron — Dose rate: 1 mg/kg

Duration: 361 days — Temp: 20 °C

Moisture: 83.6% — Soil: Les Evouettes (loam, microbial active)

pH 6.8 — Organic carbon: 3.8%

Half-live (parent): 16 days two 1st order compartment model) — ^{14}C accountability: 100–110%

% lufenuron remaining: 4.2% after 361 days

% mineralisation: up to 15.1% after 361 days

% unextracted: up to 78.6% after 240 days

Metabolites	Max (% TRR)	Day
CGA238277	23.1%	14
CGA224443	21.6%	59

Ref.: Ellgehausen (1991, LUFEN_028)

Test material: ^{14}C-difluorophenyl-lufenuron — Dose rate: 1.2 mg/kg

Duration: 361 days — Temp: 20 °C

Moisture: 83.6% — Soil: Les Evouettes (loam, microbial active)

pH 6.8 — Organic carbon: 3.8%

Half-live (parent): 24 days two 1st order compartment model) — ^{14}C accountability: 80–103%

% lufenuron remaining: 1.8% after 361 days

% mineralisation: up to 58.6% after 361 days

% unextracted: up to 36.1% after 60 days

Metabolites	Max (% TRR)	Day
None		

The aerobic soil metabolism was also investigated in the same soil types as above without microbial activity (sterile soil). After up to 90 days only unchanged lufenuron was recovered for both radiolabels without significant mineralisation or an increase of unextracted residues.

In a second study Gonzalez-Valero (1991, LUFEN_030) investigated the degradation of [^{14}C]dichlorophenyl-lufenuron in two soil types.

Ref.: Gonzalez-Valero (1991, LUFEN_030)

Test material: [^{14}C]dichlorophenyl-lufenuron — Dose rate: 0.1 mg/kg dry soil

Duration: 149 days
Moisture: 40% MWC
pH 5.0
Half-live (parent): 83 days
% lufenuron remaining: 32.4% after 149 days
% mineralisation: 2.0% after 149 days
% unextracted: 24.6% after 149 days

Temp: 20 °C
Soil: Neuhofen (sand, sterilised)
Organic carbon: 1.78
^{14}C accountability: 95.4–101.5%

Metabolites	Max (% TRR)	Day
CGA238277	10.1%	82
CGA224443	32.8%	149

Ref.: Gonzalez-Valero (1991, LUFEN_030)

Test material: [^{14}C]dichlorophenyl-lufenuron Dose rate: 0.1 mg/kg dry soil

Duration: 100 days Temp: 20 °C

Moisture: 40% MWC Soil: Mosimann (sandy loam, sterilised)

pH: 7.3 Organic carbon: 1.08

Half-live (parent): 17 days ^{14}C accountability: 89.9–101.8%

% lufenuron remaining: 8.1% after 82 days

% mineralisation: 5.0% after 100 days

% unextracted: 56.8% after 100 days

Metabolites	Max (% TRR)	Day
CGA238277	31.8%	30
CGA224443	28.0%	61

The nature of the unextracted radioactivity was further investigated by van der Gaauw (2004, LUFEN_029). After 90 day incubation of two soil types (silt loam “Les Evouettes”; loamy sand “Collombey”) the samples were extracted with three different extractants: acetonitrile: water (4:1 v/v, “solvent”), 40 mM aqueous solution of hydroxypropyl-ß-cyclodextrin (“HPCD”) or 0.02 M aqueous calcium chloride solution ($CaCl_2$). The radioactive residues, CO_2 and biomass were investigated during the experiment. In the following table the mass balance for each of the soils and its extraction efficiencies are summarized:

Table 8 Mass balance of radioactivity in soil

		Recovered Radioactivity (% applied)				
Soil Type		Days after treatment / extraction system				
		0	90/solvent	90/HPCD	90/$CaCl_2$	121/solvent
Collombey	Extractable	96.4	19.9	23.3	1.0	16.0
	Soxhlet	–	5.9	10.5	–	4.0
	Reflux	–	5.7	–	–	6.0
	CO_2	–	13.7	12.7	12.8	13.8
	Unextracted	3.1	49.1	51.4	89.6	52.6
	TOTAL	99.6	94.2	97.9	103.4	92.5
Les Evouettes	Extractable	97.1	19.7	10.9	0.35	16.9
	Soxhlet	–	6.7	15.2	–	4.0
	Reflux	–	3.2	–	–	4.2
	CO_2	–	20.0	19.0	18.8	20.3
	Unextracted	4.6	44.5	56.9	75	49.6
	TOTAL	101.7	94.1	102.0	94.2	95.0

In the “solvent” and “HPCD” extracts the composition of the radioactivity was analysed. In addition the radioactivity associated to the biomass was characterized.

Table 9 Distribution of radioactivity

Soil	Degradate	Days after treatment/extraction system			
	(% of applied)	0	90/solvent	90/HPCD	121/solvent
Collombey	Lufenuron	96.4	10.8	6.7	10.6
	CGA238277	< 0.1	4.7	6.2	7.7
	CGA224443		1.2	11.6	
	Unknown M3		2.5	5.8	
	Unknown M4		6.5		1.6
	Unknowns (2)		0.1	3.6	
	TOTAL	96.4	25.8	33.9	19.9
Les Evouettes	Lufenuron	97.0	9.3	3.4	7.6
	CGA238277		9.3	5.2	6.8
	CGA224443		0.8	7.3	0.1
	Unknown M3		1.9	7.5	
	Unknown M4		4.0		4.9
	Unknowns (4)		1.1	2.7	1.3
	TOTAL	97.4	26.4	26.1	20.7

Table 9 Organic matter fractionation of the residue remaining from solvent extraction

	Recovered Radioactivity (% applied)	
Soil fraction	Collombey	Les Evouettes
Fulvic	5.9	5.0
Humic	15.6	10.9
Humin	27.7	28.7
Total	49.1	44.6

In addition the influence of the application technique was investigated by Ellgehausen (1994, LUFEN_033). In this study [^{14}C]difluorophenyl ring-labelled lufenuron was applied at 0.1 mg/kg to a silt loam soil (60% MHC) under three test conditions involving surface treatment, incorporation and surface treatment following incorporation after 14 days. For each of the three conditions the remaining residues of the parent substance were measured.

In the following tables the mass balance and the recovered parent substance at various sampling intervals are summarized.

Table 11 Mass balance for the applied radioactivity following three different treatment conditions

	% AR						
	0 d	7 d	14 d	21 d	34/35 d	71/72 d	91/92 d
Incorporated							
Extractable	96.3	58.6	36.9	26.0	16.9	9.8	8.8
CO2	–	14.5	27.5	35.0	42.4	50.2	52.0
Unextracted	4.4	27.2	35.3	37.9	37.6	35.8	37.2
Total	100.7	100.3	99.7	98.9	96.9	95.8	98.1
No incorporation							
Extractable	94.3	81.1	73.5	59.4	44.4	24.9	36.8
CO2	–	4.4	9.1	12.6	16.7	20.3	20.7
Unextracted	3.6	13.7	17.6	25.2	33.2	47.5	36.4
Total	98.0	99.2	100.2	97.3	94.3	92.6	93.8
Surface then mixing after 14 d							
Extractable	96.0	80.3	71.5	52.9	31.0	17.4	12.8
CO2	–	4.7	9.8	15.2	21.2	25.6	26.2
Unextracted	3.0	14.1	18.9	28.8	44.1	55.5	53.3
Total	99.0	99.1	100.2	96.9	96.3	98.5	92.2

Table 12 Parent lufenuron remaining and calculated DT_{50} values

	% AR							DT_{50}
Test Conditions	0 d	7 d	14 d	21 d	34/35 d	71/72 d	91/92 d	
Incorporated	93.3	57.4	36.9	26.0	15.0	8.9	7.9	9.4 d
No incorporation	94.3	81.1	73.5	59.4	44.4	30.1	35.2	32.5 d
No Incorporation (14 days) then mixing	96.0	80.3	71.5	52.9	31.0	16.2	11.8	32.3 d (0–14 d) 13.8 d (14–92 d)

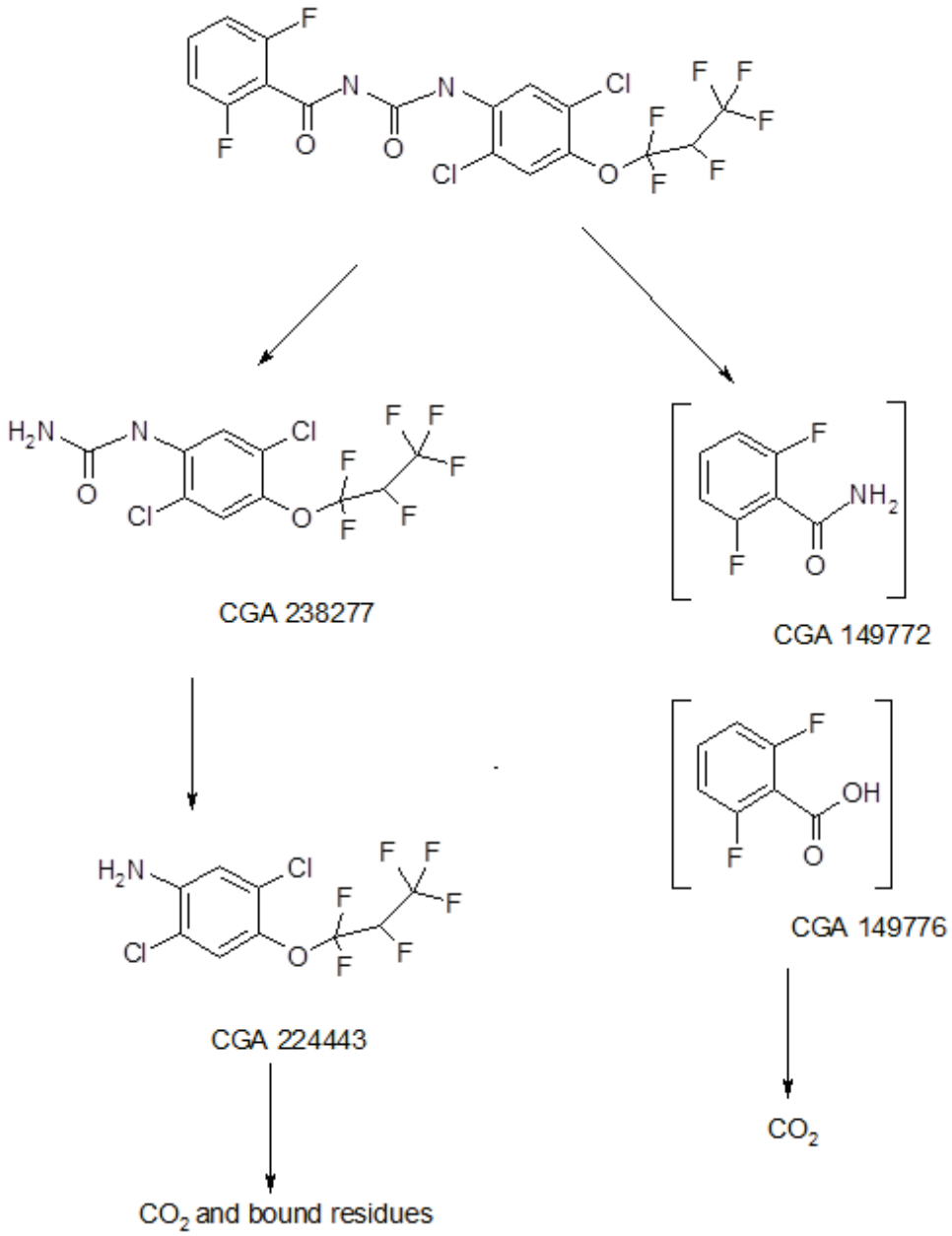


Figure 3 Proposed metabolic pathway of lufenuron in soil (aerobic)

Besides the parent substance the behaviour of the soil metabolite CGA149772 (2,6-difluorobenzamide) under aerobic conditions was investigated by Slangen (2003, LUFEN_034) in three different soil types using ^{14}C-phenyl ring-labelled CGA149772.

Soil was extracted by shaking with acetonitrile: acetic acid 98:2 following two more extractions with a mixture of acetonitrile: water 80:20 (v/v). Finally, the soil was extracted with water. The remaining soil debris was extracted with acetonitrile in a Soxhlet for six hours. All the supernatants were evaporated to aqueous and analysed by LSC followed by two different normal phase TLC methods and HPLC where possible. The sample of each soil type with the highest bound residue remaining after extraction was subjected to organic matter fractionation.

Ref.: Slangen (2003, LUFEN_034)

Test material: ^{14}C-phenyl-2,6-difluorobenzamide — Dose rate: 0.4 mg/kg

Duration: 120 days — Temp: 20 °C

Moisture: 45% MHC — Soil: Borstel

pH 5.14 — Organic carbon: 1.0

Half-live (CGA149772): 4.8 days — ^{14}C accountability: 87.6–104.5%

% CGA149772 remaining: < 0.1% after 120 days

% mineralisation: max. 59.5% after 56 days

% unextracted: max. 37.9% after 56 days

Metabolites	Max (% TRR)	Day
CGA149776	50.9	14

Ref.: Slangen (2003, LUFEN_034)

Test material: ^{14}C-phenyl-2,6-difluorobenzamide — Dose rate: 0.4 mg/kg

Duration: 120 days — Temp: 20 °C

Moisture: 45% MHC — Soil: Gartenacker

pH 7.23 — Organic carbon: 2.35

Half-live (CGA149772): 2.7 days — ^{14}C accountability: 90.5–102.8%

% CGA149772 remaining: < 0.1% after 120 days

% mineralisation: max. 62.8% after 120 days

% unextracted: max. 39.1% after 28 days

Metabolites	Max (% TRR)	Day
CGA149776	29.3	7

Ref.: Slangen (2003, LUFEN_034)

Test material: ^{14}C-phenyl-2,6-difluorobenzamide — Dose rate: 0.4 mg/kg

Duration: 120 days — Temp: 20 °C

Moisture: 45% MHC — Soil: Weide

pH 7.58 — Organic carbon: 1.94

Half-live (CGA149772): 4.0 days ^{14}C accountability: 93.6–103.1%

% CGA149772 remaining: < 0.1% after 120 days

% mineralisation: max. 64.6% after 120 days

% unextracted: max. 41.4% after 28 days

Metabolites	Max (% TRR)	Day
CGA149776	24.7	14

Soil degradation

The soil degradation of [^{14}C]dichlorophenyl-lufenuron and its primary metabolites CGA224443 and CGA238277 under varying moisture and temperature was investigated by Gonzalez-Valero (1991, LUFEN_031). A silt loam soil type (Les Evouettes) was incubated under different conditions described in the following table. For each condition, an amount of 0.1 mg ai/kg or 1 mg ai/kg soil was applied.

Table 13 Incubation conditions

Moisture content	Temperature (°C)	Concentration
30% field capacity	20	0.1 and 1.0 mg/kg
60% field capacity	20	0.1 and 1.0 mg/kg
60% field capacity	10	0.1 and 1.0 mg/kg

Based on these conditions, the following amounts of lufenuron, CGA224443 and CGA238277 were recovered.

Table 14 Radioactivity recovered as lufenuron in% AR (mean of both application rates)

	Sampling Interval (days)									
Test Conditions	0	7	14	21	28	42	60	90	120	180
60% FC, 20 °C	98.1	73.3	49.0	35.9	29.4	15.0	13.5	13.6	10.7	–
60% FC, 10 °C	98.8	89	76.9	–	52.8	42.1	30.2	23.3	18.3	13.3
30% FC, 20 °C	96.5	84.5	73.2	58.8	53.4	37.4	31.8	22.2	17.9	13.4

Table 15 Radioactivity recovered as CGA238277 in% AR (mean of both application rates)

	Sampling Interval (days)									
Test Conditions	0	7	14	21	28	42	60	90	120	180
60% FC, 20 °C	0	18.9	29.0	30.2	24.3	12.6	8.0	7.3	3.9	–
60% FC, 10 °C	0	7.25	14.4	–	24.7	26.5	27.5	21.2	15.8	10.6
30% FC, 20 °C	0	9.4	11.9	12.2	12.2	12.6	8.25	6.4	5	2.8

Table 16 Radioactivity recovered as CGA224443 in% AR (mean of both application rates)

	Sampling Interval (days)									
Test Conditions	0	7	14	21	28	42	60	90	120	180
60% FC, 20 °C	0	6.3	13.5	17.0	19.5	26.3	23.3	17.7	17.4	–
60% FC, 10 °C	0	2.8	5.8	–	9.1	16.8	23.0	26.0	28.3	24.7
30% FC, 20 °C	0	4.1	7.75	14.1	12.8	15.5	17.1	16.8	12.8	11.6

The modelling of DT_{50}- and DT_{90}-values based on this study was conducted by Sapiets (2003, LUFEN_032). By using first-order compartment models (FOMC) the following values were estimated:

Table 17 Calculated DT_{50}- and DT_{90}-values for lufenuron, CGA224443 and CGA238277

Compound	Model	DT_{50} (days)	DT_{90} (days)
Lufenuron	FOMC	13.7	81.1
CGA238277	FOMC	12.8	42.5
CGA224443	FOMC	35.8	118.8

Plant metabolism

The fate of lufenuron in plants was investigated following foliar spray application of [dichlorophenyl-14C]- and/or [difluorophenyl-14C]-radiolabelled active substance to tomato, cabbage and cotton.

In all samples unchanged lufenuron was the only residue compound detected, mainly present on the surface of the treated plant parts. No significant translocation was observed after treatment or direct stem injection. After several weeks, an uptake of the residue in treated leaves was observed, however the extracts contained lufenuron solely. In very minor amounts CGA238277 was detected at levels of 3.3% TRR or less. A proposed metabolic pathway scheme is presented in Figure 4.

Tomato

The metabolism of lufenuron was investigated in tomatoes after three spray applications with [dichlorophenyl-^{14}C]-lufenuron by Stingelin (1992, LUFEN_019). Fruit bearing plants were treated with rates equivalent to 0.03 kg ai/ha per application with one week intervals. The plants were kept in protected environments. Samples were collected from the same four plants 1 h after the first treatment, and 1 h, 12 d and 28 d after the final application (dissipation experiment). Foliage and mature fruits of four additional plants were collected 28 days after the final treatment to investigate the distribution and degradation of lufenuron.

In a second experiment four single fruits were treated by injection of 34 µg lufenuron. The fruits were sampled after 18 and 33 days.

The tomato fruits were washed three times (1 minute) in acetone (250 mL) to solubilise surface radioactivity; the levels of radioactivity in the washing were determined by liquid scintillation counting (LSC). The washed tomato fruits were frozen and homogenised under liquid nitrogen and the total radioactive residues (TRR) determined by combustion and LSC.

Extraction of the radioactive residues in the homogenised plant material was carried out using methanol-water (80:20, v/v) for two hours. This procedure was repeated until the radioactivity of the last extract was less than 5% of the first extract (maximum five extraction steps). Any remaining residues were subjected to Soxhlet extraction and finally unextracted residues were determined by combustion.

Extracts and washings were analysed by thin layer chromatography. Reference markers were visualized under UV light and areas of radioactivity detected using a radiochromatogram camera.

In all fruit and leaves samples from the foliar spray experiments most of the radioactivity was recovered in the surface wash, presenting 74–100% of the TRR. Minor amounts were also recovered primarily by methanol/water extraction, adding to total recoveries of radioactivity of 96–118% TRR. Lufenuron was the major residue identified in the combined surface wash and extracts, representing 93–99% of the TRR. In the extracts of fruits sampled 28 DALT, traces of CGA238277 were identified at 0.2% of the TRR (see Tables 18 Table and 19).

In mature fruits receiving a direct injection of lufenuron the results were comparable, with 90–95% of the radioactivity identified as unchanged lufenuron. Again CGA238277 was identified in minor amounts up to 2% of the TRR, and 5% of the total radioactivity remained unextracted.

Table 18 Summary of the distribution of radioactivity and residual [dichlorophenyl-^{14}C]-lufenuron in tomato fruits (dissipation experiment)

	1 hour after Application 1	1 hour after Application 3	12 days after Application 3	28 days after Application 3
TRR	0.58 mg eq/kg	1.216 mg eq/kg	0.84 mg eq/kg	0.694 mg eq/kg
Surface wash (surf.)	99.6% TRR	98.6% TRR	95.9% TRR	93.6% TRR
Methanol/water extraction (extr.)	Not analysed	3.5% TRR	10.0% TRR	1.7% TRR
Soxhlet extraction (extr.)	Not analysed	< 0.1% TRR	0.1% TRR	0.1% TRR
Lufenuron in combined extracts (surf.+extr.)	Not analysed	1.209 mg eq/kg (99.4% TRR)	0.822 mg eq/kg (97.9% TRR)	0.644 mg eq/kg (92.8% TRR)
CGA238277 (extr. only)	Not detected	Not detected	Not detected	0.2% TRR
Unextracted	Not analysed	0.1% TRR	0.1% TRR	0.2% TRR
Total (surf. + extr. + unextr.)	100% TRR	102.2% TRR	106.1% TRR	95.9% TRR

Table 19 Summary of the distribution of radioactivity and residual [dichlorophenyl-^{14}C]-lufenuron in tomato foliage and fruits (distribution and degradation experiment)

	Foliage (28 d DALT)	Green fruits (28 d DALT)	Red fruits (28 d DALT)	Combined fruits (28 d DALT)
TRR	0.467 mg eq/kg	0.03 mg eq/kg	0.44 mg eq/kg	0.199 mg eq/kg
Surface wash	Not determined	73.7% TRR	89.9% TRR	88.5% TRR
Methanol/water extraction	116.9% TRR	Not analysed	12.2% TRR	Not analysed
Soxhlet extraction	0.7% TRR	Not analysed	0.5% TRR	Not analysed
Lufenuron in combined extracts	0.444 mg eq/kg (95.1% TRR)	0.028 mg eq/kg (93.3% TRR)	0.43 mg eq/kg (97.7% TRR)	0.194 mg eq/kg (97.5% TRR)
Unextracted	0.6% TRR	Not analysed	0.2% TRR	Not analysed
Total (surf. + extr. + unextr.)	118.2% TRR	100% TRR	102.8% TRR	Not analysed

Cabbage

Cabbage plants (white cabbage) in a greenhouse were treated by Krauss (1994, LUFEN_020) with three spray applications of 0.02 kg ai/ha each (0.06 kg ai/ha total) in two week intervals using [dichlorophenyl-^{14}C]-lufenuron. Samples were taken one hour after the first and last application, and at crop maturity, 28 days after the last application. At each sampling the heads were separated into old/wrapper leaves and remaining heads.

Homogenised plant material was extracted five times with methanol-water (80:20, v/v) or until the radioactivity of the last extract was less than 5% of first extraction. Further extraction of the plant material was carried out using Soxhlet extraction with methanol. The amount of radioactivity in extracts was determined using liquid scintillation counting (LSC) and by combustion LSC of solid materials.

The nature of the residues in cabbage extracts was elucidated using normal and reverse phase thin layer chromatography. Reference markers were visualised under UV light and areas of radioactivity detected using a radiochromatogram camera.

In cabbage samples most of the radioactivity was present in part of the heads directly affected by the spray solution. Whole cabbage and older leaves gave TRR levels between 0.5–1.8 mg eq/kg, while the inner head contained lower radioactive residues of 0.2–0.3 mg eq/kg, and 89–101% of the TRR were extracted by methanol/water. In the extracts, unchanged parent lufenuron was the only major residue representing 88–98% of the TRR. The only other metabolite identified was CGA238277, representing up to 3.3% of the TRR (see Table 20).

Table 20 Summary of the distribution of radioactivity and residual [dichlorophenyl-^{14}C]-lufenuron in cabbage

	1 hour after Appl. 1	1 hour after application 3 (last application)		28 days after application 3 (last application)	
	Whole cabbage	Head cabbage	Old leaves	Head cabbage	Old leaves

	1 hour after Appl. 1	1 hour after application 3 (last application)		28 days after application 3 (last application)	
	Whole cabbage	Head cabbage	Old leaves	Head cabbage	Old leaves
TRR	0.501 mg eq/kg	0.301 mg eq/kg	1.659 mg eq/kg	0.195 mg eq/kg	1.790 mg eq/kg
Methanol/water extraction	90.1% TRR	100.7% TRR	89.3% TRR	96.9% TRR	96.3% TRR
Soxhlet extraction	0.9% TRR	2.3% TRR	1.7% TRR	4.7% TRR	3.0% TRR
Total extracts					
Start	1.0% TRR [a]	3.0% TRR [a]	–	–	1.3% TRR [a]
CGA238277	–	–	–	0.6% TRR [a]	3.3% TRR [a]
Lufenuron	0.446 mg eq/kg (89.0% TRR)	0.296 mg eq/kg (97.9% TRR)	1.46 mg eq/kg (88.0% TRR)	0.19 mg eq/kg (97.5% TRR)	1.702 mg eq/kg (95.1% TRR)
Unresolved	0.5% TRR [a]	1.1% TRR [a]	1.5% TRR [a]	1.6% TRR [a]	1.3% TRR [a]
Unextracted	0.1% TRR	0.2% TRR	0.1% TRR	0.5% TRR	0.4% TRR
Total (surf. + extr. + unextr.)	91.1% TRR	103.2% TRR	91.1% TRR	102.7% TRR	103.1% TRR

[a] Concentration not quantified in TLC system

Cotton

The investigation on the metabolism of lufenuron in cotton under glasshouse conditions was reported in two studies. In the first study by Stingelin (1991, LUFEN_021) [dichlorophenyl-^{14}C]-lufenuron formulated as EC50 product was applied with three spray applications at a rate equivalent to 0.03 kg ai/ha (total seasonal application rate 0.09 kg ai/ha). The first application was made at the beginning of flowering and further applications made at 14-day intervals. Sampling of leaves took place 1 hour, 1 day, 3 and 7 days after the first application and 14 days, 28 and 84 days (maturity) after the last application. At maturity, plants were also separated into stalks, leaves (old and new), green bolls, hulls, fibre and seeds

In addition, four cotton plants were injected (into the stalks) with radiolabelled lufenuron (100 µg) dissolved in acetone (2 µL). Two further injections were made at 14-day intervals. Harvested cotton plants from the injection experiment were separated into similar components, i.e. stalks, (region of the injection and remainder) leaves (old and new), green bolls, hulls, fibre and seeds.

All plants were kept in plastic containers in greenhouse.

At each interval, from the foliar application, the leaves were washed three times with a mixture of acetone-water (50:50; v/v). The washed leaves were then homogenized in the presence of methanol water (80:20, v/v).

The components from the mature cotton plants were homogenized in the presence of “dry ice” or after freezing with liquid nitrogen; in the case of dry hulls the samples were homogenized in a mill. For extraction of the radioactive residues, the homogenised plant material was suspended in a mixture of methanol-water (80:20; v/v). This procedure was repeated until the radioactivity of the last extract was equal or less than 5% of the radioactivity contained in the first extract.

The amount of radioactivity in extracts and post-extraction solids was determined using liquid scintillation counting (LSC) and by combustion LSC. The nature of the residues in extracts was elucidated using silica gel 60 F thin layer chromatography. Reference markers were visualised under UV light (254 nm) and areas of radioactivity detected using a radiochromatogram camera.

In cotton leaves most of the residue was recovered in the surface wash, however at the end of the experiment (84 DALT) approximated half of the radioactivity was present in the washed leaf extracts. In total, the extraction rates of leaves and other plant parts was high, leaving less than 3% unextracted. In the combined extracts, unchanged lufenuron was the only

residue identified in leaves, stalks and hulls, representing 89–100% of the TRR. Fibre, seeds and bolls did not contain sufficient radioactivity for identification (TRR ≤ 0.001 mg eq/kg).

Table 21 Summary of the distribution of radioactivity and residual [dichlorophenyl-^{14}C]-lufenuron in cotton foliage

	Leaves (1 hour after Appl. 1)	Leaves (1 day after Appl. 1)	Leaves (3 days after Appl. 1)	Leaves (7 days after Appl. 1)	Leaves (14 DALT)	Leaves (28 DALT)	Leaves (84 DALT)
TRR	2.453 mg eq/kg	2.374 mg eq/kg	1.79 mg eq/kg	0.64 mg eq/kg	3.334 mg eq/kg	2.74 mg eq/kg	4.912 mg eq/kg
Surface wash (surf.)	98.0% TRR	86.5% TRR	71.5% TRR	76.9% TRR	62.9% TRR	45.2% TRR	42.5% TRR
Methanol/water extraction (extr.)	1.9% TRR	13.2% TRR	28.1% TRR	22.8% TRR	35.6% TRR	52.6% TRR	54.3% TRR
Lufenuron in combined extracts (surf.+extr.)	2.406 mg eq/kg (98.1% TRR)	2.251 mg eq/kg (94.8% TRR)	1.646 mg eq/kg (91.9% TRR)	0.593 mg eq/kg (92.7% TRR)	3.102 mg eq/kg (93.0% TRR)	2.491 mg eq/kg (90.9% TRR)	4.364 mg eq/kg (88.8% TRR)
Unextracted	0.1% TRR	0.3% TRR	0.4% TRR	0.4% TRR	1.4% TRR	2.2% TRR	3.2% TRR
Total (surf. + extr. + unextr.)	100.0% TRR	100.0% TRR	100.0% TRR	100.1% TRR	99.9% TRR	100.0% TRR	100.0% TRR

Table 22 Summary of the distribution of radioactivity and residual [dichlorophenyl-^{14}C]-lufenuron in various cotton plant parts at maturity (84 DALT)

	Old Leaves	New Leaves	Stalks	Hulls	Fibre	Seeds	Green Bolls
TRR	1.487 mg eq/kg	0.014 mg eq/kg	0.026 mg eq/kg	0.092 mg eq/kg	< 0.001 mg eq/kg	< 0.001 mg eq/kg	0.001 mg eq/kg
Surface wash (surf.)	43.6% TRR	–	–	–	–	–	–
Methanol/water extraction (extr.)	58.7% TRR	109.4% TRR	116.2% TRR	103.9% TRR	n.a.	n.a.	n.a.
Soxhlet (extr.)	0.9% TRR	4.0% TRR	1.9% TRR	1.2% TRR	n.a.	n.a.	n.a.
Lufenuron in combined extracts (surf.+extr.)	1.415 mg eq/kg (95.2% TRR)	0.014 mg eq/kg (100% TRR)	0.026 mg eq/kg (100% TRR)	0.091 mg eq/kg (98.9% TRR)	n.a.	n.a.	n.a.
Unextracted	1.6% TRR	2.7% TRR	2.1% TRR	1.6% TRR	n.a.	n.a.	n.a.
Total (surf. + extr. + unextr.)	104.8% TRR	116.1% TRR	120.2% TRR	106.7% TRR	–	–	–

n.a.=Not analysed

The translocation experiment following stem injection showed that most of the applied radioactivity remained at the injection site (81.2% AR). Into close stalks (13.3% AR) and leaves (1.6-3.9% AR) a minor translocation was observed. In all samples the unchanged parent was the only residue identified (approximately 95–98% TRR).

In a second study conducted by Gentile (1991, LUFEN_022) cotton grown in greenhouse was treated with [^{14}C]difluorophenyl-lufenuron formulated as an EC50 product. Eight cotton plants were separately treated with three spray applications at a rate equivalent to 0.03 g ai/ha each (total seasonal application rate 0.09 g ai/ha). The first application was made at two months after sowing (no growth stage reported) and further applications made two and four weeks after the first application.

Sampling (three leaves from four plants) took place 2 hours after each application. At maturity, 52 days after the last application, plants were separated into stems, leaves (old and new), hulls, fibre and seeds.

At each interval from the foliar application, the leaves were washed twice with acetonitrile (surface wash). The washed leaves were then homogenized in the presence of acetonitrile-water (80:20, v/v). The unextracted radioactive residues were determined by combustion and liquid scintillation counting (LSC).

The components from the mature cotton plants were homogenized in the presence of liquid nitrogen. Radioactive residues in the homogenised plant material were extracted with acetonitrile-water (80:20, v/v). The procedure was repeated until the radioactivity of the last extract was equal or less than 5% of the radioactivity contained in the first extract. Residues remaining in the plant material were solubilised using Soxhlet extraction with acetonitrile. The amount of radioactivity in extracts was determined using liquid scintillation counting (LSC) and by combustion LSC in solid materials. The nature of the residues in extracts was elucidated using silica gel 60 F thin layer chromatography. Reference markers were visualised under UV light (254 nm) and areas of radioactivity detected using a TLC scanner.

In the leaves sampled at each interval at least 49% of the radioactivity was found in the surface wash. The total recovery of radioactivity was high, leaving less than 2% of the TRR unextracted. In the combined extracts unchanged lufenuron was the only residue identified, representing at least 92% of the TRR.

In other matrices (old leaves, stems, hulls and fibre) the methanol/water extract released the major part of the residue. Again, only unchanged lufenuron was present in the extracts at levels of 78.7–83.1% TRR. In seeds and new grown leaves the TRR was too low for further identification (0.003–0.005 mg eq/kg).

For a summary of the results please refer to Table 23.

Table 23 Summary of the distribution of radioactivity and residual [difluorophenyl-^{14}C]-lufenuron in various cotton plant parts at maturity (52 DALT)

Interval	Matrix	Total Residues [mg eq/kg]	Parent		Surface wash [% TRR]	Extracts			Total Rad. [% TRR]
			[mg eq/kg]	[% TRR]		Met./Water extract [% TRR]	Soxhlet extract [% TRR]	PES [% TRR]	
2 hours after Appl. 1	Leaves Plant 1	1.907	–	96.8	91.4	8.2	0.0	0.3	100
	Leaves Plant 2	2.379	–	96.6	92.2	7.5	0.1	0.2	100
	Leaves Plant 3	4.068	–	97.0	89.6	10.0	0.1	0.3	100
	Leaves Plant 4	4.592	–	96.1	92.4	7.1	0.1	0.3	100
	Leaves Mean	3.237	–	–	–	–	–	–	–
2 hours after Appl. 2	Leaves Plant 1	2.434	–	95.8	63.0	35.9	0.3	0.8	100
	Leaves Plant 2	5.103	–	97.0	83.2	16.0	0.1	0.6	100
	Leaves Plant 3	4.715	–	95.6	77.6	21.5	0.2	0.6	100
	Leaves Plant 4	6.233	–	95.3	79.1	20.1	0.2	0.6	100
	Leaves Mean	4.621	–	–	–	–	–	–	–
2 hours after Appl. 3	Leaves Plant 1	3.153	–	97.3	88.0	11.3	0.1	0.6	100
	Leaves Plant 2	3.777	–	97.3	76.1	22.5	0.2	1.2	100
	Leaves Plant 3	2.663	–	96.6	90.0	8.9	0.2	0.9	100
	Leaves Plant 4	2.342	–	96.2	81.4	17.6	0.1	0.8	100
	Leaves Mean	2.984	–	–	–	–	–	–	–
Maturity 52 DALT	Leaves Plant 1	1.85	–	92.1	49.2	48.8	0.8	1.2	100
	Leaves Plant 3	5.95	–	93.0	57.7	39.8	1.0	1.5	100
	Old leaves	2.089	1.95	93.3	n.p.	98.8	1.3	1.6	101.7
	New leaves	0.005	n.a.	n.a.	n.p.	n.a.	n.a.	n.a.	n.a.
	Stems	0.124	0.103	83.1	n.p.	91.7	1.5	1.2	94.4
	Hulls	0.687	0.541	78.7	n.p.	84.0	1.4	1.3	86.7
	Fibre	0.028	0.023	82.1	n.p.	91.7	1.7	5.5	98.9
	Seeds	0.003	n.a.	n.a.	n.p.	n.a.	n.a.	n.a.	n.a.

PES=Post-extraction solids

n.a. = Not analysed
n.p. = Not performed

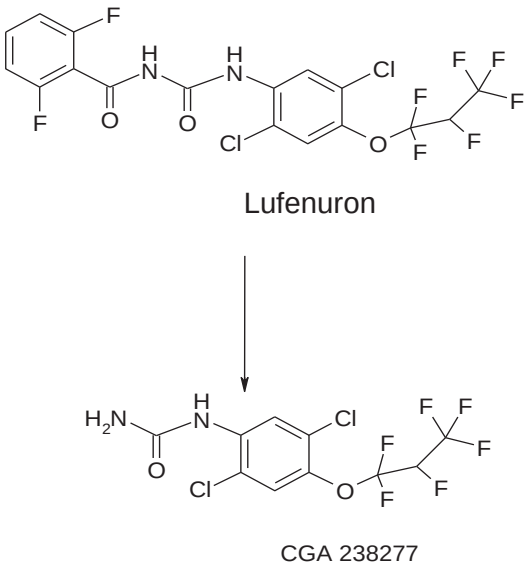


Figure 4 Proposed metabolic pathway of lufenuron in plants

Confined rotational crop studies

For the investigation of lufenuron in rotational crops two studies were conducted involving application of either [^{14}C]difluorophenyl- or [^{14}C]dichlorophenyl-lufenuron. The experiments using [^{14}C]difluorophenyl-lufenuron was conducted by Gentile (1992, LUFEN_023). Plant containers kept in a glasshouse received application to bare soil equivalent to 0.15 kg ai/ha. Lettuce, spring wheat, maize and carrots were planted in the treated soil 63 days after test substance application. Immature and mature samples of the crops were taken throughout the study and soil samples were taken at each sampling.

Fresh samples were homogenised in the presence of liquid nitrogen and dry plant parts, e.g. grain, were homogenised in a mill. For extraction of the radioactive residues the homogenised plant material was suspended in a mixture of acetonitrile-water (80:20; v/v). This procedure was repeated until the radioactivity of the last extract was equal or less than 5% of the radioactivity contained in the first extract. Non-extracted residues were solubilised using Soxhlet extraction with acetonitrile. The amount of radioactivity in extracts was determined using liquid scintillation counting (LSC) and by combustion LSC in solid materials.

The nature of the residues in extracts was elucidated using silica gel 60 F thin-layer chromatography. Reference markers were visualised under UV light (254 nm) and areas of radioactivity detected using a radiochromatogram camera.

The transfer of radioactivity into lettuce, wheat, maize and carrots grown as succeeding crops was very limited. In mature lettuce (126 d after treatment) the highest TRR levels of 0.047 mg eq/kg were found. 53% of the TRR was identified as unchanged parent (0.025 mg/kg). In other matrices only wheat straw (0.023 mg eq/kg, 0.007 mg lufenuron/kg) and immature carrots roots (0.023 mg eq/kg, no identification conducted) showed total radioactive residues above 0.01 mg eq/kg. No further identification was conducted for these matrices. In soil, nearly the entire extracted radioactivity was attributed to lufenuron. No further metabolites could be identified against the reference compounds CGA149772 or CGA149776.

Table 24 Distribution of total radioactivity and residues of lufenuron in succeeding lettuce grown in soil treated at a rate equivalent to 0.15 kg [^{14}C]difluorophenyl-lufenuron per ha

Days after treatment	Soil layer	Total residues		Parent	Extracted radioactivity		Unextracted	Total
					Cold	Soxhlet		
(PBI: 63 d)		[mg eq/kg]	[% TRR]	[mg eq/kg (% TRR)]	[% TRR]	[% TRR]	[% TRR]	[% TRR]
63	SOIL							
	0–5 cm	0.206	93.9	0.146	76.3	0.9	25.1	102.3
	5–10 cm	0.009	3.9	(70.8)	n.a.	n.a.	n.a.	n.a.
	10–20 cm	0.003	2.1	n.a.	n.a.	n.a.	n.a.	n.a.
	Total	0.066	100	n.a.	–	–	–	–
				–				
99	SOIL							
	0–5 cm	0.239	99.1	0.151	70.0	0.9	26.8	97.7
	5–10 cm	0.002	0.6	(63.2)	n.a.	n.a.	n.a.	n.a.
	10–20 cm	< 0.001	0.3	n.a.	n.a.	n.a.	n.a.	n.a.
	Total	0.087	100	n.a.	–	–	–	–
				n.a.				
	HEADS	0.004	100	n.a.	n.a.	n.a.	n.a.	n.a.
126	SOIL							
	0–5 cm	0.269	89.0	0.176	69.5	1.0	27.6	98.1
	5–10 cm	0.044	10.0	(65.4)	65.9	1.1	31.8	98.8
	10–20 cm	0.005	1.0	0.027	n.a.	n.a.	n.a.	n.a.
	Total	0.134	100	(61.4)	–	–	–	–
				n.a.				
				n.a.				
	HEADS	0.047	100	0.025	75.0	1.7	43.4	120.1
				(53.2)				

n.a.=Not analysed

Table 25 Distribution of total radioactivity and residues of lufenuron in succeeding wheat grown in soil treated at a rate equivalent to 0.15 kg [^{14}C]difluorophenyl-lufenuron per ha

Days after treatment	Soil layer	Total residues		Parent	Extracted radioactivity		Unextracted	Total
					Cold	Soxhlet		
(PBI: 63 d)		[mg eq/kg]	[% TRR]	[mg eq/kg (% TRR)]	[% TRR]	[% TRR]	[% TRR]	[% TRR]
63	SOIL							
	0–5 cm	0.221	94.4	0.155	75.0	1.0	26.1	102.1
	5–10 cm	0.012	4.3	(70.1)	75.9	1.4	29.2	106.5
	10–20 cm	0.002	1.4	0.009 (75)	n.a.	n.a.	n.a.	n.a.
	Total	0.071	100	n.a.	–	–	–	–
				–				
99	SOIL							
	0–5 cm	0.128	95.4	0.087 (68)	72.2	1.1	23.0	96.3
	5–10 cm	0.006	4.2	n.a.	n.a.	n.a.	n.a.	n.a.
	10–20 cm	< 0.001	0.4	n.a.	n.a.	n.a.	n.a.	n.a.
	Total	0.046	100	–	–	–	–	–
	WHOLE TOPS	0.005	100	n.a.	n.a.	n.a.	n.a.	n.a.
126	SOIL							
	0–5 cm	0.212	94.7	0.127	63.8	0.9	26.7	91.4
	5–10 cm	0.01	4.4	(59.9)	57.4	1.7	42.3	101.4
	10–20 cm	0.001	1.0	0.005 (50)	n.a.	n.a.	n.a.	n.a.
	Total	0.063	100	n.a.	–	–	–	–
				–				
	WHOLE TOPS	0.002	100	n.a.	n.a.	n.a.	n.a.	n.a.

Days	Soil layer	Total residues		Parent	Extracted radioactivity		Unextracte	Total
161	SOIL							
	0–5 cm	0.167	99.6	0.114	73.4	0.7	26.0	100.1
	5–10 cm	< 0.001	0.3	(68.3)	n.a.	n.a.	n.a.	n.a.
	10–20 cm	< 0.001	0.1	n.a.	n.a.	n.a.	n.a.	n.a.
	Total	0.063	100	n.a.	–	–	–	–
				–				
	STALKS	0.023	100	0.007	65.8	0.5	32.3	98.6
				(30.4)				
	HUSKS	0.002	100	n.a.	n.a.	n.a.	n.a.	n.a.
	GRAIN	0.007	100	n.a.	n.a.	n.a.	n.a.	n.a.

n.a.=Not analysed

Table 26 Distribution of total radioactivity and residues of lufenuron in succeeding maize grown in soil treated at a rate equivalent to 0.15 kg [^{14}C]difluorophenyl-lufenuron per ha

Days after treatment	Soil layer	Total residues		Parent	Extracted radioactivity		Unextracte d	Total
					Cold	Soxhlet		
(PBI: 63 d)		[mg eq/kg]	[% TRR]	[mg eq/kg (% TRR)]	[% TRR]	[% TRR]	[% TRR]	[% TRR]
63	SOIL							
	0–5 cm	0.405	96.4	0.311	81.6	0.6	18.6	100.8
	5–10 cm	0.019	3.4	(76.8)	79.3	1.1	23.0	103.4
	10–20 cm	< 0.001	0.2	0.014	n.a.	n.a.	n.a.	n.a.
	Total	0.14	100	(73.7)	–	–	–	–
				n.a.				
				–				
99	SOIL							
	0–5 cm	0.186	98.0	0.148	83.7	1.1	23.0	107.8
	5–10 cm	0.003	1.3	(79.6)	n.a.	n.a.	n.a.	n.a.
	10–20 cm	0.001	0.7	n.a.	n.a.	n.a.	n.a.	n.a.
	Total	0.066	100	n.a.	–	–	–	–
				–				
	TOPS	< 0.001	100	n.a.	n.a.	n.a.	n.a.	n.a.
126	SOIL							
	0–5 cm	0.23	97.8	0.138	63.9	1.1	31.3	96.3
	5–10 cm	0.004	1.6	(60.0)	n.a.	n.a.	n.a.	n.a.
	10–20 cm	< 0.001	0.6	n.a.	n.a.	n.a.	n.a.	n.a.
	Total	0.069	100	n.a.	–	–	–	–
				–				
	TOPS	0.002	100	n.a.	n.a.	n.a.	n.a.	n.a.
197	SOIL							
	0–5 cm	0.107	97.7	0.063	63.6	1.2	36.5	101.3
	5–10 cm	< 0.001	0.6	(58.9)	n.a.	n.a.	n.a.	n.a.
	10–20 cm	0.003	1.8	n.a.	n.a.	n.a.	n.a.	n.a.
	Total	0.047	100	n.a.	–	–	–	–
				–				
	STALKS	0.008	100	n.a.	n.a.	n.a.	n.a.	n.a.
	COBS	0.003	100	n.a.	n.a.	n.a.	n.a.	n.a.
	GRAIN	0.004	100	n.a.	n.a.	n.a.	n.a.	n.a.

n.a.=Not analysed

Table 27 Distribution of total radioactivity and residues of lufenuron in succeeding carrots grown in soil treated at a rate equivalent to 0.15 kg [^{14}C]difluorophenyl-lufenuron per ha

Days after treatment	Soil layer	Total residues		Parent	Extracted radioactivity		Unextracte d	Total
					Cold	Soxhlet		
(PBI:		[mg	[% TRR]	[mg eq/kg	[% TRR]	[% TRR]	[% TRR]	[% TRR]

Days	Soil layer	Total residues		Parent	Extracted radioactivity		Unextracte	Total
63 d)		eq/kg]		(% TRR)]				
63	SOIL							
	0–5 cm	0.169	92.1	0.128	79.9	1.0	24.5	105.4
	5–10 cm	0.015	7.2	(75.7)	71.6	1.0	30.0	102.6
	10–20 cm	0.001	0.6	0.009	n.a.	n.a.	n.a.	n.a.
	Total	0.006	100	(60.0)	–	–	–	–
				n.a.				
				–				
99	SOIL							
	0–5 cm	0.111	99.0	0.068	64.7	1.3	33.5	99.5
	5–10 cm	< 0.001	0.5	(61.3)	n.a.	n.a.	n.a.	n.a.
	10–20 cm	< 0.001	0.5	n.a.	n.a.	n.a.	n.a.	n.a.
	Total	0.041	100	n.a.	–	–	–	–
				–				
	WHOLE TOPS	0.008	100	n.a.	n.a.	n.a.	n.a.	n.a.
126	SOIL							
	0–5 cm	0.136	97.7	0.077	62.3	1.2	36.0	99.5
	5–10 cm	0.001	0.9	(56.6)	n.a.	n.a.	n.a.	n.a.
	10–20 cm	0.002	1.4	n.a.	n.a.	n.a.	n.a.	n.a.
	Total	0.047	100	n.a.	–	–	–	–
				–				
	WHOLE TOPS	0.008	100	n.a.	n.a.	n.a.	n.a.	n.a.
	ROOTS	0.023	100	n.a.	n.a.	n.a.	n.a.	n.a.
197	SOIL							
	0–5 cm	0.184	97.8	0.085	50.4	0.9	45.3	96.6
	5–10 cm	0.002	1.4	(46.2)	n.a.	n.a.	n.a.	n.a.
	10–20 cm	0.001	0.8	n.a.	n.a.	n.a.	n.a.	n.a.
	Total	0.06	100	n.a.	–	–	–	–
				–				
	WHOLE TOPS	0.005	100	n.a.	n.a.	n.a.	n.a.	n.a.
	ROOTS	0.005	100	n.a.	n.a.	n.a.	n.a.	n.a.

n.a.=Not analysed

In a second confined study in the field conducted by Stingelin (1992, LUFEN_024) [^{14}C]dichlorophenyl-lufenuron was applied to bare soil one at a rate equivalent to 0.13 kg ai/ha. After different plant-back intervals (PBI) lettuce (PBI 76 d), winter wheat (PBI 126 d), sugar beets (PBI 306 d) and maize (PBI 331 d) were planted/sown and grown to maturity. In addition soil samples from layers up to 30 cm depth were collected and analysed for residues.

Fresh samples were homogenised in the presence of liquid nitrogen and dry plant parts, e.g. grain, were homogenised in a mill. After homogenisation samples were combusted and the levels of radioactivity were measured by liquid scintillation counting (LSC).

None of the plant samples were extracted since the radioactive residues were < 0.01 mg/kg.

In soil samples most of the radioactivity was recovered in the first 5 cm soil layer (55–96% AR). At the end of the study (519 days after treatment) up to 27.9% AR moved into the 5–10 cm layer and up to 24.7% to the 10–20 cm layer. The transfer into even lower layers was minimal (< 7% AR). The analysis of the upper layers revealed lufenuron as the major residue. The only metabolites identified were CGA238277 and CGA224443, both not exceeding 0.014 mg eq/kg.

Table 28 Distribution of total radioactivity of lufenuron in succeeding crops grown under field conditions in soil treated at a rate equivalent to 0.13 kg [^{14}C]dichlorophenyl-lufenuron per ha

Crop/Plant-back interval	Matrix	Days after soil treatment	Days after planting/sowing	TRR in mg eq/kg

Lettuce (PBI 76 d)	Heads, immature	30	106	0.004
	Heads, mature	62	138	0.001
Wheat (PBI 126 d)	Whole tops	182	56	0.003
	Whole tops	307	181	< 0.001
	Whole tops	363	237	< 0.001
	Stalks	418	292	0.004
	Husks	418	292	0.001
	Grain	418	292	< 0.001
Sugar beets	Immature roots	363	57	0.002
(PBI 306 d)	Immature tops	363	57	0.002
	Immature roots	418	112	0.001
	Immature tops	418	112	< 0.001
	Roots	519	213	< 0.001
	Tops	519	213	< 0.001
Maize (PBI 331 d)	Whole tops	363	32	0.002
	Whole tops	418	87	< 0.001
	Stalks	495	164	0.003
	Cobs	495	164	< 0.001
	Grain	495	164	< 0.001

Animal metabolism

The Meeting received metabolism studies on laboratory animals, poultry and lactating goats using the difluorophenyl- and the dichlorophenyl-label of lufenuron.

The metabolism of lufenuron in livestock animals was minimal, showing only unchanged parent substance in all goat matrices. In poultry minor amounts of CGA149772 and CGA238277 were found in edible commodities, however at levels below 10% TRR or 0.01 mg eq/kg. Most of the radioactive residue was present in fat tissue, egg yolk and milk.

Laboratory animals

Lactating goats

The metabolic fate of lufenuron in lactating goats was investigated using [^{14}C]difluorophenyl- or [^{14}C]dichlorophenyl-lufenuron (Cameron, 1992, LUFEN_018 & Schulze-Aurich, 1992, LUFEN_017). The compound was administered to one lactating goat for each label in gelatine capsules at 5.4 ppm for the difluorophenyl-label (0.135 mg/kg body weight) and 6.0 ppm for the dichlorophenyl-label (0.15 mg/kg body weight) for ten consecutive days. Excreta and milk were collected daily. The animals were slaughtered approximately 24 hours after the last dose. Muscle, omental fat, peritoneal fat, liver, kidney, blood, bile and content of gastrointestinal tract/rumen were collected.

Radioactivity was measured by combustion and liquid scintillation counting. The composition of samples was investigated two months after sampling. Thin-layer chromatography was used to identify and characterize radioactive components in sample extracts.

The total recovery of the administered radioactivity was 95% for both labels. The majority of the radioactivity (73–74%) was found in the faeces. Radioactive residues in the edible tissues were 0.8–1.6% AR in muscle, 4.2–5.4% AR in fat, 0.28–0.3% AR in liver, 0.01–0.02% AR in kidney and 5.8–6.8% AR in milk. A summary of the recovered radioactivity is presented in Table 29.

Table 29 Radioactive residues in milk and tissues after oral administration of [^{14}C]difluorophenyl- (5.4 ppm) or [^{14}C]dichlorophenyl-lufenuron (6.0 ppm) for 10 consecutive days

	[^{14}C]\difluorophenyl-label (5.4 ppm)		[^{14}C]dichlorophenyl-label (6.0 ppm)	
Tissue	Mean radioactivity (mg/kg or mg/L lufenuron eq.)	Mean radioactivity (% of total dose)	Mean radioactivity (mg/kg or mg/L lufenuron eq.)	Mean radioactivity (% of total dose)

	[^{14}C]\difluorophenyl-label (5.4 ppm)		[^{14}C]dichlorophenyl-label (6.0 ppm)	
Tissue	Mean radioactivity (mg/kg or mg/L lufenuron eq.)	Mean radioactivity (% of total dose)	Mean radioactivity (mg/kg or mg/L lufenuron eq.)	Mean radioactivity (% of total dose)
Total milk	–	6.76	–	5.76
Muscle, hindquarter	0.066		0.039	
Muscle, forequarter	0.08	1.6	0.038	0.77
Muscle, Tenderloin	0.071		0.04	
Fat, omental	2.288		2.411	
Fat, subcutaneous	0.883	5.4	0.821	4.22
Fat, renal	2.434		1.64	
Liver	0.417	0.297	0.367	0.28
Kidney	0.114	0.017	0.118	0.014
Rumen and intestinal contents	0.35	5.04	0.75	10.1
Faeces	–	73.8	–	72.8
Cage wash	–	0.25	–	0.27
Total recovery	–	94.6	–	94.8

In milk radioactive residues approximated a plateau after one week of dosing. In the following table the total radioactivity recovered from milk is summarized.

Table 30 Mean radioactive residues in goat milk following 10 consecutive doses of [^{14}C]dichlorophenyl or [^{14}C]difluorophenyl lufenuron to lactating goats

	TRR mg eq/kg			
	[^{14}C]difluorophenyl-label (5.4 ppm)		[^{14}C]dichlorophenyl-label (6.0 ppm)	
Days of dosing	am milk	pm milk	am milk	pm milk
1	0.000	0.043	0.000	0.030
2	0.303	0.381	0.315	1.270
3	0.560	0.622	1.042	0.850
4	0.848	0.594	0.752	0.823
5	0.646	0.601	0.719	0.792
6	0.766	0.802	0.875	1.186
7	0.878	0.892	0.850	1.049
8	0.998	0.940	1.037	0.798
9	1.001	0.979	0.711	0.790
10	0.997	0.706	0.786	0.791
11	0.690	–	0.674	–

For both labels unchanged lufenuron was the only residue in tissues and milk, representing 73–94% of the TRR. Highest concentrations were present in fat tissue and milk. No separation between milk fat and skim milk was conducted.

In goat faeces and urine the majority of the residue also comprised of lufenuron. Varying levels, depending on the sampling period, of CGA238277, CGA149772 and CGA149776 were also found.

For the composition of radioactive residues in milk and tissues please see Tables 31 and 32.

Table 31 Extraction and analysis of radioactive residues in goats tissues and milk treated with [^{14}C]difluorophenyl labelled lufenuron (5.4 ppm)

	Metabolite Fractions in mg eq/kg (% TRR)				
	Fat	Muscle	Liver	Kidney	Milk
TRR	1.67	0.07	0.417	0.114	0.993
Identified					
Lufenuron (parent)	1.502 (89.9)	0.061 (87.0)	0.305 (73.1)	0.095 (83.3)	0.922 (92.8)
Unknown [a]	0.099 (6.0)	0.007 (9.5)	0.078 (18.8) [b]	0.014 (12.5)	0.066 (6.6)

	Metabolite Fractions in mg eq/kg (% TRR)				
	Fat	Muscle	Liver	Kidney	Milk
Unextracted	0.068 (4.1)	0.002 (3.5)	0.034 (8.1)	0.005 (4.2)	0.006 (0.6)

[a] Unresolved radioactivity in TLC system

[b] Two unresolved fractions

Table 32 Extraction and analysis of radioactive residues in goat tissues and milk treated with [^{14}C]difluorophenyl labelled lufenuron (6.0 ppm)

	Metabolite Fractions in mg eq/kg (% TRR)				
	Fat	Muscle	Liver	Kidney	Milk
TRR	2.02	0.039	0.367	0.118	0.737
Identified					
Lufenuron (parent)	1.817 (90)	0.035 (89.5)	0.291 (79.4)	0.105 (88.6)	0.689 (93.5)
Unknown [a]	0.14 (6.9)	0.003 (7.8)	0.043 (11.7) [b]	0.011 (9.1)	0.041 (5.6)
Unextracted	0.063 (3.1)	0.001 (2.7)	0.033 (8.9)	0.003 (2.3)	0.007 (0.9)

[a] Unresolved radioactivity in TLC system

[b:] Two unresolved fractions

Laying hens

The metabolic fate of lufenuron was investigated using [^{14}C]difluorophenyl- or [^{14}C]dichlorophenyl-lufenuron (Cameron, 1992, LUFEN_016 & Schulze-Aurich, 1992, LUFEN_017). For each label the compound was administered in gelatine capsules to three laying hens at doses of 3.4 ppm for the difluorophenyl-label (representing 2.6 mg/kg body weight) and of 5.2 ppm for the dichlorophenyl-label (representing 3.5 mg/kg body weight) for fourteen consecutive days. Excreta and eggs were collected daily. The animals were slaughtered approximately 24 hours after the last dose. Muscle, skin with attached fat, peritoneal fat, liver, kidney and content of gastrointestinal tract were collected.

Radioactivity was measured by combustion and liquid scintillation counting. The composition of samples was investigated two months after sampling. Thin-layer chromatography was used to identify and characterize radioactive components in sample extracts.

The total recovery of the administered radioactivity was 75–79%. The majority of the radioactivity (54–62%) was found in the excreta. Radioactive residues in the edible tissues were 0.55–1.15% AR in lean meat, 5.1–9.9% AR in fat, 0.4–0.58% AR in liver, 0.07% AR in kidney and 8.7–9.6% AR in eggs. A summary of the recovered radioactivity is presented in Table 33.

Table 33 Radioactive residues in eggs and tissues after oral administration of [^{14}C]difluorophenyl- (3.4 ppm) or [^{14}C]dichlorophenyl-lufenuron (5.2 ppm) for 14 consecutive days

	[^{14}C]difluorophenyl-label (3.4 ppm)		[^{14}C]dichlorophenyl-label (5.2 ppm)	
Tissue	Mean radioactivity (mg/kg or mg/L lufenuron eq.)	Mean radioactivity (% of total dose)	Mean radioactivity (mg/kg or mg/L lufenuron eq.)	Mean radioactivity (% of total dose)
Total egg	–	8.69	–	9.64
Lean meat	0.237	1.15	0.104	0.55
Skin + fat	2.56	Not calculated	1.296	Not calculated
Peritoneal fat	13.04	8.83	7.189	5.09
Liver	1.45	0.64	0.828	0.4
Kidney	0.737	0.09	0.524	0.07
Blood	0.292	0.14	0.189	0.1
Intestinal contents	–	0.15	–	0.21
Excreta	–	62.18	–	53.5
Cage wash	–	1.45	–	1.27

	[^{14}C]difluorophenyl-label (3.4 ppm)		[^{14}C]dichlorophenyl-label (5.2 ppm)	
Tissue	Mean radioactivity (mg/kg or mg/L lufenuron eq.)	Mean radioactivity (% of total dose)	Mean radioactivity (mg/kg or mg/L lufenuron eq.)	Mean radioactivity (% of total dose)
Total recovery	–	78.95	–	75.49

In eggs radioactive residues were mainly present in the egg yolk for both labels. A plateau was approximated at the end of the 14 days dosing period in the yolk while residues in egg white remained stable after more than 4 days. In the following table the total radioactivity recovered from egg white and egg yolk is summarized:

Table 34 Mean radioactive residues in hen egg following 14 consecutive doses of [^{14}C]dichlorophenyl or [^{14}C]difluorophenyl lufenuron to laying hens

	TRR mg eq/kg			
	[^{14}C]difluorophenyl-label (3.4 ppm)		[^{14}C]dichlorophenyl-label (5.2 ppm)	
Days of dosing	Egg white	Egg yolk	Egg white	Egg yolk
1	0.000	0.000	0.001	0.000
2	0.000	0.158	–	–
3	0.001	0.566	0.003	0.474
4	0.003	1.635	0.005	1.065
5	0.003	2.301	–	–
6	0.003	3.802	0.005	2.419
7	0.005	6.334	0.011	3.966
8	0.002	5.258	0.009	3.973
9	0.005	5.507	0.016	6.133
10	–	–	0.007	4.766
11	0.002	7.441	0.015	6.565
12	0.008	7.110	0.008	7.585
13	0.003	6.555	0.008	8.048
14	0.002	6.470	0.008	8.479

For both labels unchanged lufenuron was the major residue in all tissues and eggs, representing at least 79.3% of the TRR. Highest concentrations were present in poultry fat and egg yolk.

The only other metabolites identified were CGA149772 for the difluorophenyl-label (egg white, 0.001 mg eq/kg, 17.3% TRR) and CGA238277 for the dichlorophenyl-label (kidney and egg white, < 0.001–0.028 mg eq/kg, 5.3–7% TRR).

In hen excreta > 90% TRR was extracted. Lufenuron was the major component of the residue, i.e. > 82%. No other component accounted for > 5% TRR, CGA238277 represented 3% TRR and CGA149776 for < 4.3% TRR.

For the composition of radioactive residues in eggs and tissues please refer to Tables 35 and 36.

Table 35 Extraction and analysis of radioactive residues in hen tissues and eggs treated with [^{14}C]difluorophenyl labelled lufenuron (3.4 ppm)

	Metabolite Fractions in mg eq/kg (% TRR)					
	Fat	Liver	Kidney	Lean meat	Egg yolk	Egg white
TRR	9.763	1.451	0.737	0.237	8.048	0.008
Identified						
Lufenuron (parent)	9.148 (93.7	1.337 (92.1)	0.588 (79.8)	0.196 (82.6)	7.179 (89.2)	0.003 (37.6)
CGA149772	–	–	–	–	–	0.001 (17.3)
Unknown [a]	0.469 ((4.8)	0.087 (6.0)	0.128 (17.4)	0.029 (12.4)	0.249 (3.1)	0.003 (42.1)
Unextracted	0.146 (1.5)	0.028 (1.9)	0.021 (2.8)	0.012 (5.0)	0.62 (7.7)	< 0.001 (3.0)

[a] Unresolved radioactivity in TLC system

Table 36 Extraction and analysis of radioactive residues in hen tissues and eggs treated with [^{14}C]dichlorophenyl labelled lufenuron (5.2 ppm)

	Metabolite Fractions in mg eq/kg (% TRR)					
	Fat	Liver	Kidney	Lean meat	Egg yolk	Egg white
TRR	4.148	0.828	0.524	0.104	6.555	0.003
Identified						
Lufenuron (parent)	3.795 (91.5)	0.705 (85.1)	0.415 (79.3)	0.089 (85.7)	6.135 (93.6)	0.001 (44.1)
CGA238277	–	–	0.028 (5.3)	–	–	< 0.001 (7.0)
Unknown [a]	0.262 (6.3)	0.069 (8.3)	0.055 (10.6)	0.011 (10.7)	0.197 (3.0)	0.001 (37.4)
Unextracted	0.091 (2.2)	0.055 (6.6)	0.026 (4.9)	0.004 (3.6)	0.223 (3.4)	< 0.001 (11.4)

[a] Unresolved radioactivity in TLC system

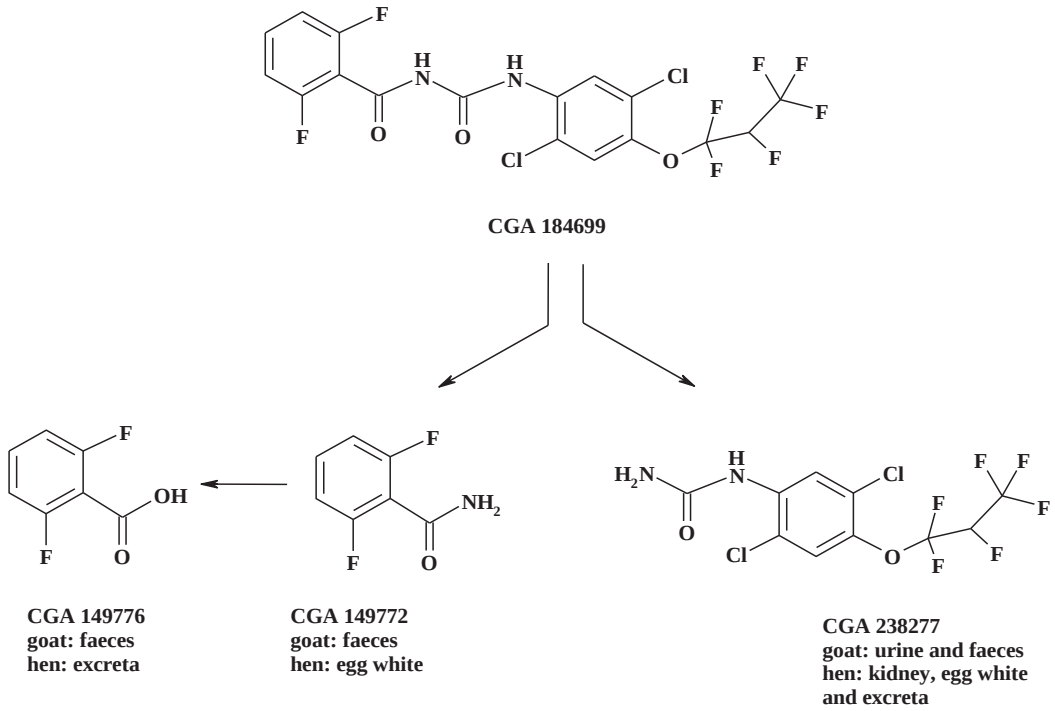


Figure 5 Metabolic pathway of lufenuron (CGA184699) in animals

RESIDUE ANALYSIS

Analytical methods

For lufenuron analytical methods were provided for plant and animal matrices. All plant matrices were validated with an LOQ of 0.01 mg/kg. For animal commodities a general LOQ of 0.02 mg/kg was validated.

The applicability of multi residue methods was confirmed on basis of DFG S19 for plant and animal matrices (LOQ 0.02 mg/kg for all commodities).

Table 37 Overview of analytical methods for lufenuron

Method	Matrix	Extraction	Clean-Up	Detection, LOQ

Method	Matrix	Extraction	Clean-Up	Detection, LOQ
REM 118.01 & modification REM 118.07	high water acidic	Methanol, partitioning against hexane/diethyl ether (9:1,v:v)	cyano SPE	REM 118.01: HPLC-UV (255 nm), LOQ: 0.02 mg/kg REM 118.07: HPLC-MS/MS m/z: 509.1 → 326.0 LOQ: 0.01 mg/kg
POPIT MET.015	High oil Difficult (coffee)	water, saturated sodium chloride solution, and hexane: ethyl ether (9:1,v:v)	C_{18} SPE	HPLC-UV, 255 nm LOQ: 0.02 mg/kg
POPIT MET.077.Rev05	High water High oil Difficult (coffee)	water, saturated sodium chloride solution, and hexane: ethyl ether (9:1,v:v)	C_{18} SPE	HPLC-MS/MS m/z 511.09 → 158.2 LOQ: 0.01 mg/kg
POP PAT 004 V01/V04	Dry High oil	water, saturated sodium chloride solution, and hexane: ethyl ether (9:1,v:v)	none	HPLC-MS/MS m/z 511.09 → 158.2 LOQ: 0.01 mg/kg
MRM DFG S19	High water Acidic Dry High oil	See DFG S19	See DFG S19	HPLC-MS/MS m/z 509 → 326 & 509 → 175 LOQ: 0.02 mg/kg
REM 118.04	Animal tissues Milk Blood	Fat/milk: acetonitrile Others: methanol	Silica gel SPE	HPLC-UV, 255 nm LOQs: Milk: 0.001 mg/kg Blood: 0.002 mg/kg Liver, kidney: 0.01 mg/kg Meat: 0.02 mg/kg Fat: 0.1 mg/kg
MRM DFG S19	Milk Eggs Animal tissues	See DFG S19	See DFG S19	HPLC-MS/MS m/z 509 → 326 & 509 → 175 LOQ: 0.02 mg/kg

Plant materials

Method REM 118.01 (Altenburger, 1988, LUFEN_035; Clarke, 2004, LUFEN_036) and Method REM 118.07 (Clarke, 2005, LUFEN_037)

Lufenuron residues were extracted from plant material by maceration in the presence of methanol. The extract filtered and diluted with water and sodium chloride solution. Lufenuron is partitioned into hexane/diethyl ether (9/1; v/v); the organic phase is reduced in volume, redissolved in hexane, and "cleaned up" using solid phase extraction on a cyano SPE cartridge (REM 118.01 only). The concentration of lufenuron is determined using HPLC-UV detection at 255 nm (REM 118.01) and in the current version HPLC-MS/MS (REM 118.07).

Table 38 Recovery data for method REM 118.01 (HPLC-UV: 255 nm) and its modification REM 118.01 (LC-MS/MS: m/z: 509.1 → 326.0) measuring lufenuron in plant matrices

Matrix	Fortification level (mg/kg)	n	Recovery range (%)	Recovery, mean (%)	RSD (%)	Reference, MRM transition
Tomato	0.01	5	92–108	98	10	Clarke (2004, LUFEN_035; 2005,
	0.1	5	87–109	96	11	LUFEN_037)
Oranges	0.01	5	71–85	78	8	m/z: 509.1 → 326.0
	0.1	5	69–84	77	9	
Grapes	0.01	5	82–94	88	6	
	0.1	5	83–89	85	3	
Tomato	0.02	5	77–95	86	9	Altenburger (1988, LUFEN_035)
	0.2	5	77–110	90	13	UV: 255 nm
Grapes	0.02	5	74–102	89	14	
	0.2	5	82–111	102	12	

Method POPIT MET.015 (Anonymous, 2002, LUFEN_038)

Method POPIT MET.015 provides for the determination of lufenuron in coffee beans and soybeans. The frozen raw sample is prepared by milling the whole sample with dry ice until the complete homogenization. 10 g of the sample is homogenized with 80 mL of methanol by milling. An 8 mL aliquot of the sample is mixed with 8 mL of water, 4 mL of saturated sodium chloride solution, and 4 mL of hexane: ethyl ether (9:1) solution, for a final volume of 24 mL. The upper layer is transferred to another vessel, evaporated at 40 °C, and re-dissolved in 2.5 mL hexane. The sample is cleaned up by silica solid phase extraction (SPE). The sample solution collected is evaporated at 40 °C, and dissolved in 2 mL of hexane: isopropanol: methanol (90:5:5) solution. The final sample solution is analysed by LC UV (255 nm).

Table 39 Recovery data for method POPIT MET.015 in coffee and soybeans

Matrix	Fortification level (mg/kg)	n	Recovery range (%)	Recovery, mean (%)	RSD (%)	Reference
Coffee beans	0.02	8	93–107	101	6	Anonymous (2002, LUFEN_038)
	0.2	4	86–92	90	3	HPLC-UV: 255 nm
Soybeans	0.02	7	82–87	84	3	
	0.2	5	71–76	75	3	

Method POPIT MET.077.Rev05 (Anonymous, 2008, LUFEN_039)

Method POPIT MET.077 provides for the determination of lufenuron in cotton, coffee, sunflowers, peaches sugarcane and sugar cane litter. The frozen sample is prepared by milling the whole sample with dry ice until the complete homogenization. 5 g of the sample is homogenized with 40 mL of methanol by milling. An 8 mL aliquot of the sample is mixed with 8 mL of water, 4 mL of saturated sodium chloride solution, and 4 mL of hexane: ethyl ether (9:1) solution, for a final volume of 24 mL. The upper layer is transferred to another vessel. The clean-up is repeated and a 4 mL aliquot of the hexane: ethyl ether (9:1) solution is added to the remaining layer. The upper layer is combined with the initial extract. This extract is evaporated at 40 °C, and re-dissolved in 2.5 mL hexane. The sample is then cleaned up by silica solid phase extraction (SPE) and analysed by LC-MS/MS.

Table 40 Recovery data for method POPIT MET.077.Rev05 in plant matrices

Matrix	Fortification level (mg/kg)	n	Recovery range (%)	Recovery, mean (%)	RSD (%)	Reference, MRM transition
Cotton seed	0.01	7	75–82	80	3	Anonymous (2008, LUFEN_039)
	0.1	6	75–82	79	3	m/z 511.09 → 158.2
Coffee beans	0.01	8	91–109	100	7	
	0.1	6	96–106	101	4	
Sunflower seed	0.01	8	87–102	95	5	
	0.1	6	82–93	89	6	
Peach	0.01	7	83–91	86	3	
	0.1	5	101–106	104	2	
	2.5	5	82–84	83	1	
Sugar cane	0.01	7	83–104	93	8	
	0.1	5	103–108	106	2	
Sugar cane litter	0.01	8	70–83	76	6	
	0.1	6	86–92	89	2	

Method POP PAT 004 V01/V04 (Anonymous, 2010, LUFEN_040)

Method POP PAT 004 provides for the determination of lufenuron in maize and soy. The frozen raw sample is prepared by milling the whole sample with dry ice until homogenous. The sample is homogenized with 20 mL of methanol by milling. A 4 mL aliquot of the sample is mixed with 4 mL of water, 4 mL of saturated sodium chloride solution, and 4 mL of hexane: ethyl ether (9:1) solution, to a final volume of 16 mL. The upper layer is transferred to another vessel. The clean-up is repeated, and a 4 mL aliquot of the hexane:ethyl ether (9:1) solution is added to the remaining layer. The upper

layer is combined with the initial extract. The combined sample is evaporated at 40 °C, re-dissolved in 1 mL methanol and analysed by LC-MS/MS.

Table 41 Recovery data for method POP PAT 004 V01/V04

Matrix	Fortification level (mg/kg)	n	Recovery range (%)	Recovery, mean (%)	RSD (%)	Reference, MRM transition
Maize grain	0.01	5	74–97	81	11	Anonymous (2010, LUFEN_040)
	0.1	5	71–73	72	1	m/z: 511 → 158 & m/z: 511 → 141
Soybean seeds	0.01	5	77–95	86	9	
	0.1	5	72–104	84	15	

Multi-residue method DFG S19 (extended revision) (Anspach, 2002, LUFEN_042 & Schulz, 2003, LUFEN_043)

A method, based on the DFG S19 (extended revision) multi-method, for routine monitoring of lufenuron in samples of plant material has been validated.

Lufenuron residues are extracted using module E1 for orange and tomato, E2 for wheat grain followed by clean up procedures according to module GPC (gel permeation chromatography). All samples are analysed by high performance liquid chromatography with tandem mass spectrometric detection, HPLC-MS/MS (m/z: 509 → 326 & m/z: 509 → 175).

Table 42 Recovery data for the multi-residue method DFG S19 in plant commodities

Matrix	Fortification level (mg/kg)	n	Recovery range (%)	Recovery, mean (%)	RSD (%)	Reference, MRM transition
Tomato	0.02	5	75–82	79	3	Anspach (2002, LUFEN_042)
	0.2	5	78–86	84	4	m/z: 509 → 326
Orange	0.02	5	69–85	79	8	
	0.2	5	74–88	80	7	
Maize grain	0.02	5	65–79	75	8	
	0.2	5	81–93	86	6	
Tomato	0.02	5	92–106	100	6	Schulz (2003, LUFEN_043)
	0.2	5	81–106	98	6	m/z: 509 → 326
Oilseed rape seeds	0.02	5	91–110	100	7	
	0.2	5	94–110	104	6	
Orange	0.02	5	77–93	86	6	
	0.2	5	81–90	86	3	
Maize grain	0.02	5	79–86	83	4	
	0.2	5	82–90	86	4	

Animal materials

Method REM 118.04 (Tribolet, 1995, LUFEN_041)

REM 118.04 provides for the determination of lufenuron in tissues, fat and milk. Tissue samples are extracted by maceration (liver and kidney) or shaking (meat) with methanol. In the case of fat, the sample is melted and lufenuron residues are extracted by shaking with acetonitrile. The extract is filtered and the acetonitrile reduced in volume. The residue is re-dissolved in methanol.

Milk samples are diluted with acetonitrile to precipitate proteins, filtered and the acetonitrile reduced in volume. The residue is re-dissolved in methanol.

The extracts, for all commodities, are diluted with water and sodium chloride solution. Lufenuron is partitioned into hexane/diethyl ether (9/1; v/v) and the organic phase is reduced in volume, re-dissolved in hexane, and “cleaned up” using solid phase extraction on a silica gel cartridge.

The concentration of lufenuron is determined by HPLC-UV at 255 nm.

Table 43 Recovery data for method REM 118.04 in animal matrices

Matrix	Fortification level (mg/kg)	n	Recovery range (%)	Recovery, mean (%)	RSD (%)	Reference
Milk	0.001	12	74–112	96	11	Tribolet (1995, LUFEN_041)
	0.01	9	86–116	94	11	HPLC-UV: 255 nm
Meat	0.01	7	61–112	82	25	
	0.02	2	86–89	88	–	
	0.1	4	81–84	83	2	
Liver	0.01	4	87–100	92	7	
	0.02	2	63–86	75	–	
	0.1	4	87–111	94	12	
Kidney	0.01	4	105–144	119	15	
	0.02	2	108–113	111	–	
	0.1	4	103–106	104	1	
Fat	0.01	11	53–109	76	23	
	0.1	4	68–80	72	8	
Blood	0.002	12	68–97	85	11	
	0.02	9	83–105	89	10	

Multi-residue method DFG S19 (extended revision) (Anspach, 2003, LUFEN_044 & Schulz, 2003, LUFEN_045)

A method, based on the DFG S19 (extended revision) multi-method, for samples of tissues, milk and eggs has been validated. For samples of milk, meat and eggs, samples are extracted with acetone. Water is added prior to extraction to maintain a ratio of 2/1 (v/v), taking into account the natural water content of the matrices. Ethyl acetate/cyclohexane (1/1; v/v) and sodium chloride are added and the mixture homogenised. An aliquot of the organic phase is applied and cleaned up using gel permeation chromatography. In the case of fat, samples are mixed with synthetic calcium silicate after addition of acetone and acetonitrile. An aliquot of the organic phase was cleaned up on gel permeation chromatography.

All samples were analysed for residues of lufenuron by high performance liquid chromatography with tandem mass spectrometric detection, HPLC-MS/MS (m/z: 509 → 326 & m/z: 509 → 175).

Table 44 Recovery data for the multi-residue method DFG S19 in animal commodities

Matrix	Fortification level (mg/kg)	n	Recovery range (%)	Recovery, mean (%)	RSD (%)	Reference, MRM transition
Milk	0.02	5	79–101	87	10	Anspach (2002, LUFEN_042)
	0.2	5	69–94	83	13	m/z: 509 → 326
Meat	0.02	5	69–84	78	7	
	0.2	5	71–93	79	11	
Eggs	0.02	5	78–129	104	17	
	0.2	5	79–101	88	11	
Fat	0.02	5	67–78	72	6	
	0.2	5	80–91	86	6	
Milk	0.02	5	76–102	87	14	Schulz (2003, LUFEN_043)
	0.2	5	105–117	109	5	m/z: 509 → 326
Meat	0.02	5	62–84	76	13	
	0.2	5	76–91	85	7	

Stability of pesticides in stored analytical samples

Plant matrices

Tribolet (1993, LUFEN_046)

Samples of cotton seed, cabbage and orange were fortified with lufenuron at a concentration of 0.5 mg/kg and stored under –18 °C. The samples were stored in plastic and glass vessels, however no difference between both materials was observed. Samples were taken for analysis at intervals up to 24 months in parallel to freshly fortified samples to estimate the procedural recovery. Analysis of the samples was performed according to the method REM 118.01.

In the study report the results for the stored samples are only reported as percentage of the fortified level corrected by the procedural recoveries. No measured concentrations were described.

Table 45 Recovered lufenuron residues in stored plant commodities after storage up to 24 months (Tribolet, 1993, LUFEN_046)

Matrix	Fortification level (mg/kg)	Storage period (months)	Residue level in stored samples corrected by procedural recoveries		Procedural recovery	
			Individual corrected values (% fortified)	Mean (%)	Individual values (%)	Mean (%)
Cottonseed	0.5	0	–	–	94, 92	93
		0.5	96, 97, 99, 100, 102, 105	100	94, 92	93
		1	94, 98, 98, 106	99	94, 92	93
		3	100, 103, 103, 104	103	89, 88	89
		6	102, 103, 108, 110	106	78, 89	84
		12	93, 95, 97, 98	96	91, 92	92
		24	98, 101, 103, 106	102	90, 90	90
Cabbage	0.5	0	–	–	83, 93	88
		0.5	89, 97, 98, 98, 100, 102	97	87, 87	87
		1	104, 107, 109, 110	108	85, 84	85
		3	101, 101, 102, 102	102	92, 92	92
		6	99, 100, 101, 102	101	87, 89	88
		12	95, 97, 98, 106	99	94, 94	94
		24	99, 99, 114, 115	107	79, 83	81
Orange	0.5	0	–	–	91, 92	92
		0.5	95, 97, 97, 98, 98, 106	99	90, 94	92
		1	97, 97, 100, 102	99	91, 92	92
		3	101, 103, 106, 112	106	91, 94	93
		6	88, 93, 95, 95	93	88, 89	89
		12	99, 99, 108, 108	104	89, 91	90
		24	99, 101, 103, 116	105	88, 89	89

Animal matrices

Tribolet (1995, LUFEN_047)

Storage stability of residues of lufenuron in bovine tissues and milk were conducted to support the data from the livestock feeding study. Samples of bovine muscle, liver, kidney, fat, milk and blood were fortified with lufenuron at a concentration of 0.2 mg/kg in tissues, 0.02 mg/kg in milk and 0.04 mg/kg in blood. Samples were stored at –18 °C for a period of 9 months, which covered the sample storage time in the study. Analysis of the samples (in triplicate) was performed according to the method REM 118.04.

Table 46 Residues of lufenuron in animal commodities after storage at –18 ºC (Tribolet, 1995, LUFEN_047)

Matrix	Forti	Storage	Lufenuron

	fication level (mg/kg)	period (months)	Residue level in stored samples		Procedural recovery
			Individual values in mg/kg (mean)	% nominal	%
Muscle	0.2	9	0.13, 0.14, 0.14 (0.14)	70	75
Liver	0.2	9	0.14, 0.14, 0.16 (0.15)	75	84
Kidney	0.2	9	0.14, 0.15, 0.16 (0.15)	75	90
Fat	0.2	9	0.14, 0.16, 0.17 (0.16)	80	77
Milk	0.02	9	0.015, 0.016, 0.016 (0.016)	80	79
Blood	0.04	9	0.032, 0.037, 0.041 (0.037)	93	90

USE PATTERN

Lufenuron is an insect growth inhibitor that is active against larvae of Lepidoptera and Coleoptera. It is used in a vegetable crops, oilseeds, root crops maize, sugarcane and coffee close to harvest.

Table 47 List of uses of lufenuron

Crop	Country	Application detail					
		Indoor/ Outdoor	Type	kg ai/ha	Growth stage at last treatment	No	PHI
Citrus fruit							
Citrus fruit	BR	Outdoor	Foliar spray	0.004 kg ai/hL	At infestation	1	28
Citrus fruit	CN	Outdoor	Foliar spray	0.033	At infestation	2	28
Pome fruit							
Apple	BR	Outdoor	Foliar spray	0.005 kg ai/hL	At infestation	4	14
Apple	CN	Outdoor	Foliar spray	0.05	At infestation	3	14
Stone fruit							
Peaches	BR	Outdoor	Foliar spray	0.005 kg ai/hL	At infestation	3	10
Brassica vegetables							
Cabbage	BR	Outdoor	Foliar spray	0.005 kg ai/hL	At infestation	2	7
Cabbage	CN	Outdoor	Foliar spray	0.03	At infestation	2	14
Fruiting vegetables—cucurbits							
Cucumber	BR	Outdoor	Foliar spray	0.0025 kg ai/hL	At infestation	4	7
Cucumber	ES	Indoor	Foliar spray	0.1	At infestation	2	7
Melon	ES	Indoor	Foliar spray	0.1	At infestation	3	7
Watermelon	ES	Indoor	Foliar spray	0.1	At infestation	3	7
Fruiting vegetables—other than cucurbits							
Pepper	ES	Indoor	Foliar spray	0.1	At infestation	3	7
Tomato	BR	Outdoor	Foliar spray	0.004 kg ai/hL	At infestation	4	10
Tomato	CN	Outdoor	Foliar spray	0.045	At infestation	2	7
Tomato	ES	Indoor	Foliar spray	0.1	At infestation	3	7
Leafy vegetables							
Lettuce	ES	Indoor	Foliar spray	0.03	At infestation	3	7

Crop	Country	Application detail					
		Indoor/ Outdoor	Type	kg ai/ha	Growth stage at last treatment	No	PHI
Pulses							
Beans	CN	Outdoor	Foliar spray	0.038	At infestation	3	7
Soybean	BR	Outdoor	Foliar spray	0.02	At infestation	2	35
Root and tuber crops							
Cassava	BR	Outdoor	Foliar spray	0.015	At infestation	3	7
Potato	BR	Outdoor	Foliar spray	0.04	At infestation	4	14
Potato	BR	Outdoor	Foliar spray	0.002	At infestation	3	14
Cereal grains							
Maize	BR	Outdoor	Foliar spray	0.015	At infestation	1	35
Wheat	BR	Outdoor	Foliar spray	0.005	At infestation	2	14
Grasses for sugar or syrup productions							
Sugar cane	BR	Outdoor	Foliar spray	0.02	At infestation	2	14
Tree nuts							
Coconut	BR	Outdoor	Foliar spray	0.0025 kg ai/h L	At infestation	1	14
Oilseeds							
Cotton	BR	Outdoor	Foliar spray	0.05	At infestation	1	28
Cotton	CN	Outdoor	Foliar spray	0.045	At infestation	2	28
Sunflower	BR	Outdoor	Foliar spray	0.015	At infestation	3	14
Seed for beverages and sweets							
Coffee	BR	Outdoor	Foliar spray	0.04	At infestation	2	7

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

Residue levels were reported as measured. Application rates were always reported as lufenuron equivalents. When residues were not detected they are shown as below the LOQ, e.g., < 0.01 mg/kg. Application rates, spray concentrations and mean residue results have generally been rounded to the even with two significant figures. HR and STMR values from the trials conducted according to maximum GAP have been used for the estimation of maximum residue levels. These results are underlined.

Laboratory reports included method validation including batch recoveries with spiking at residue levels similar to those occurring in samples from the supervised trials. Dates of analyses or duration of residue sample storage were also provided. Field reports provided data on the sprayers used and their calibration, plot size, residue sample size and sampling date. Although trials included control plots, no control data are recorded in the tables except where residues in control samples exceeded the LOQ. Residue data are recorded unadjusted for% recovery.

Lufenuron—supervised residue trials

Commodity	Indoor/Outdoor	Treatment	Countries	Table
Cucumber	Indoor	Foliar	France, Greece, Spain	48
Melons	Indoor	Foliar	Spain	49

Commodity	Indoor/Outdoor	Treatment	Countries	Table
Sweet Pepper	Indoor	Foliar	Greece, Italy, Spain	50
Tomato	Indoor	Foliar	Greece, Spain, Switzerland	51
Sweet corn	Outdoor	Foliar	Brazil	52
Soybeans	Outdoor	Foliar	Brazil	53
Potatoes	Outdoor	Foliar	Brazil	54
Maize	Outdoor	Foliar	Brazil	55
Sugarcane	Outdoor	Foliar	Brazil	56
Cotton	Outdoor	Foliar	China	57
Coffee	Outdoor	Foliar	Brazil	58

Table 48 Residues of lufenuron following foliar application to protected cucumbers

Location, Year (variety)	Application Form.	no	kg ai/ha	kg ai/hL	BBCH	Residues, mg/kg Sample	DAT	lufenuron	Report/Trial No., Reference, analytical method, validation data, storage period
France, Montfavet 2003 (Defens)	EC	2	0.12	0.01	89	Fruits	0 1 3 7 10	0.05 0.03 0.02 <u>0.01</u> < 0.01	03-5064, Osborne (2005, LUFEN_051) REM. 118.07, LOQ : 0.01 mg/kg, 75–79% Recovery (n=2), Storage: 13 months
France, Saint Andiol 2003 (Tyria)	EC	2	0.12	0.01	72	Fruits	0 7	0.10 <u>0.06</u>	03-5065, Osborne (2005, LUFEN_052) REM. 118.07, LOQ : 0.01 mg/kg, 74–92% Recovery (n=2), Storage: 12 months
Greece, Kenourgio 2000 (Hana)	EC	2	0.15	0.01	88	Fruits	0 7	0.02 <u>0.03</u> (< 0.02, 0.03)	Report 1048/00, Salvi (2001, LUFEN_053) REM. 118.01, LOQ : 0.02 mg/kg, 95–96% Recovery (n=2), Storage: 4 months
Greece, Kenourgio 2001 (Aris)	EC	2	0.15	0.01	89	Fruits	0 1 3 7 14	0.17 0.19 0.12 <u>0.04</u> (0.06, 0.03) < 0.02	Report 1063/01, Gasser (2001, LUFEN_054) REM. 118.01, LOQ : 0.02 mg/kg, 97–113% Recovery (n=2), Storage: 3 months
Greece, Kenourgio 1999 (Aris)	EC	2	0.15	0.01	89	Fruits	0 1 3 7	0.04 0.06 0.02 <u>0.02</u> (< 0.02, 0.02)	Report 1096/99, Tribolet (2000, LUFEN_055) REM. 118.01, LOQ : 0.02 mg/kg, 96–105% Recovery (n=2), Storage: 4 months
Spain, Motril 2004 (Baya)	EC	2	0.15	0.01	74	Fruits	0 1 3	0.06 0.06 0.04	04-5005, ES-IR-04-003, Gardinal (2006, LUFEN_050)

Location, Year (variety)	Application Form.	no	kg ai/ha	kg ai/hL	BBCH	Residues, mg/kg Sample	DAT	lufenuron	Report/Trial No., Reference, analytical method, validation data, storage period
							7 10	0.03 0.02	REM. 118.07, LOQ : 0.01 mg/kg, 90% Recovery (n=2), Storage: 12 months
Spain, Los Palacios 2000 (Torres)	EC	2	0.1	0.01	87	Fruits	0 3 7 10	0.15 0.09 0.06 (0.05, 0.06) < 0.02	Report 1042/00, Salvi (2001, LUFEN_048) REM. 118.01, LOQ : 0.02 mg/kg, 100–107% Recovery (n=2), Storage: 9 months
Spain, Los Palacios 2000 (Edona)	EC	2	0.11	0.01	87	Fruits	0 3 7 10	0.12 0.07 0.02 < 0.02	Report 1043/00, Salvi (2001, LUFEN_049) REM. 118.01, LOQ : 0.02 mg/kg, 97–110% Recovery (n=2), Storage: 8 months
Spain, Los Palacios 2001 (Darina)	EC	2	0.15	0.01	81	Fruits	0 1 3 7	0.09 0.07 0.06 0.02 (0.02, 0.02)	Report 1094/01, Gasser (2003, LUFEN_056) REM. 118.01, LOQ : 0.02 mg/kg, 85–99% Recovery (n=2), Storage: 9 months
Spain, Carchuna 2001 (Marumba)	EC	2	0.15	0.01	86	Fruits	0 7	0.06 0.03 (0.03, 0.03)	Report 1095/01, Gasser (2003, LUFEN_057) REM. 118.01, LOQ : 0.02 mg/kg, 92–129% Recovery (n=2), Storage: 7 months
Spain, Calahonda 2001 (Marumba)	EC	2	0.15	0.01	86	Fruits	0 7	0.08 0.02 (< 0.02, 0.02)	Report 1096/01, Gasser (2003, LUFEN_058) REM. 118.01, LOQ : 0.02 mg/kg, 75–79% Recovery (n=2), Storage: 7 months

DAT = Days after last treatment

BBCH 71–79 = 1st–9th fruit has reached typical size

BBCH 81–88=10–80% of fruits show typical fully ripe colour

BBCH 89=Fully ripe: fruits have typical fully ripe colour

Table 49 Residues of lufenuron following foliar application to protected melons

Location, Year (variety)	Application Form.	no	kg ai/ha	kg ai/hL	BBCH	Residues, mg/kg Sample	DAT	lufenuron	Report/Trial No., Reference, analytical method, validation data, storage period
Spain, Sanlúcar de Barrameda 2000 (Prima)	EC	3	0.1	0.01	89	Whole fruit [a]	0 3 7 10	0.09 0.06 0.07 0.04	1017/00, Salvi (2001, LUFEN_059) REM. 118.01, LOQ : 0.02 mg/kg, 70–108% Recovery (n=2), Storage: 8 months Sample segmented before storage
	EC	3	0.1	0.01	89	Whole fruit [a]	0 3	0.14 0.09 (0.09, 0.09)	1019/00, Salvi (2001, LUFEN_061)

Location, Year (variety)	Application Form.	no	kg ai/ha	kg ai/hL	BBCH	Residues, mg/kg Sample	DAT	lufenuron	Report/Trial No., Reference, analytical method, validation data, storage period
							7	0.06 (0.05, 0.06)	REM. 118.01, LOQ : 0.02 mg/kg, 109–110% Recovery (n=2), Storage: 8 months Sample segmented before storage
Spain, Sanlúcar de Barrameda 2000 (Melisa)	EC	3	0.1	0.01	89	Whole fruit [a]	0 3 7 10	0.16 0.12 (0.09, 0.15) <u>0.19</u> 0.1	1018/00, Salvi (2001, LUFEN_060) REM. 118.01, LOQ : 0.02 mg/kg, 75–91% Recovery (n=4), Storage: 7 months Sample segmented before storage
	EC	3	0.1	0.01	89	Whole fruit [a]	0 3 7	0.14 0.14 (0.105, 0.175) 0.15 (0.14, 0.16)	1020/00, Salvi (2001, LUFEN_062) REM. 118.01, LOQ : 0.02 mg/kg, 75–96% Recovery (n=4), Storage: 7 months Sample segmented before storage
Spain, Vistabella 2001 (Solarquin)	EC	3	0.1	0.01	78	Whole fruit [a] Peel Pulp	0 3 3 3	0.14 <u>0.06</u> [b] (0.06, 0.06) 0.09 (0.09, 0.09) < <u>0.02</u> (< 0.02, < 0.02)	1049/01, Gasser (2003, LUFEN_063) REM. 118.01, LOQ : 0.02 mg/kg, 95–102% Recovery (n=6), Storage: 3 months Whole fruit sample segmented before storage, peel/pulp samples separated in the field
Spain, Sanlúcar de Barrameda 2001 (Galia)	EC	3	0.1 0.11 0.1	0.01	87	Whole fruit [a] Peel Pulp	0 3 3 3	0.07 <u>0.02</u> [b] (0.02, 0.03) 0.04 (0.03, 0.05) < <u>0.02</u> (< 0.02, < 0.02)	1050/01, Gasser (2003, LUFEN_064) REM. 118.01, LOQ : 0.02 mg/kg, 95–102% Recovery (n=6), Storage: 3 months Whole fruit sample segmented before storage, peel/pulp samples separated in the field
Spain, Chipiona 2001 (Galia-F1)	EC	3	0.1	0.01	79	Whole fruit [a] Peel Pulp	0 3 7 10 3 3	0.03 0.02 [b] (0.02, 0.03) <u>0.03</u> 0.02 0.05 (0.04, 0.06) < <u>0.02</u> (< 0.02, < 0.02)	1051/01, Gasser (2003, LUFEN_065) REM. 118.01, LOQ : 0.02 mg/kg, 96–118% Recovery (n=6), Storage: 4 months Whole fruit sample segmented before storage, peel/pulp samples separated in the field
Spain, El Ejido 2001 (Siglo)	EC	3	0.1 0.09 0.1	0.01	85	Whole fruit [a]	0 3 7 10	0.13 0.12 [b] (0.1, 0.14) 0.07 <u>0.13</u>	1052/01, Gasser (2003, LUFEN_066) REM. 118.01, LOQ : 0.02 mg/kg, 96–118%

Location,	Application					Residues, mg/kg			Report/Trial No., Reference,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	lufenuron	analytical method, validation data, storage period
						Peel Pulp	3 3	0.18 (0.15, 0.21) < 0.02 (< 0.02, < 0.02)	Recovery (n=6), Storage: 4 months Whole fruit sample segmented before storage, peel/pulp samples separated in the field

[a] Calculated based on segment weight or peel/pulp ratio

DAT=Days after last treatment

BBCH 71–79 = 1st–9th fruit has reached typical size

BBCH 81–88=10–80% of fruits show typical fully ripe colour

BBCH 89 = Fully ripe: fruits have typical fully ripe colour

Table 50 Residues of lufenuron following foliar application to protected sweet peppers

Location,	Application					Residues, mg/kg			Report/Trial No., Reference,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	lufenuron	analytical method, validation data, storage period
Greece, Tyrnavos 2000 (Sammy RZ)	EC	3	0.15	0.1	89	Fruits	0 3	0.64 0.52 (0.37, 0.66)	1050/00, Salvi (2001, LUFEN_073) REM. 118.01, LOQ : 0.02 mg/kg, 94–105% Recovery (n=2), Storage: 7 months
(35-70 RZ)	EC	3	0.15	0.1	89	Fruits	0 3	0.98 0.74 (0.59, 0.88)	1051/00, Salvi (2001, LUFEN_074) REM. 118.01, LOQ : 0.02 mg/kg, 94–105% Recovery (n=2), Storage: 7 months
Greece, Tyrnavos 2001 (Sammy RZ)	EC	3	0.15	0.1	89	Fruits	0 1 3 7 14	0.21 0.34 0.25 0.18 (0.16, 0.21) 0.06	1064/01, Gasser (2003, LUFEN_075) REM. 118.01, LOQ : 0.02 mg/kg, 98–109% Recovery (n=2), Storage: 3 months
Greece, Tyrnavos 2001 (Sammy RZ)	EC	3	0.15	0.1	89	Fruits	0 7	0.67 0.42 (0.36, 0.49)	1065/01, Gasser (2003, LUFEN_076) REM. 118.01, LOQ : 0.02 mg/kg, 98–105% Recovery (n=2), Storage: 2 months
Italy, Bagnarola of Budrio 2001 (Sienor)	EC	3	0.15	0.1	82	Fruits	0 3 7 14	0.37 0.2 0.36 (0.29, 0.44) 0.23	1045/01, Gasser (2003, LUFEN_072) REM. 118.01, LOQ : 0.02 mg/kg, 93–106% Recovery (n=4), Storage: 4 months
Spain, El Mirador 1996 (Sonar)	EC	3	0.1	0.01	83	Fruits	–0 0 3 7 14 21	0.11 0.17 0.18 0.13 0.1 0.05	1013/97, Tribolet (1998, LUFEN_067) REM. 118.01, LOQ : 0.02 mg/kg, 84–108% Recovery (n=2), Storage: 3 months

Location,	Application					Residues, mg/kg			Report/Trial No., Reference,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	lufenuron	analytical method, validation data, storage period
Spain, El Ejido 1997 (Dulce Italiano)	EC	3	0.1	0.16 0.12 0.12	89	Fruits	–0 0 3 7 14 21	0.04 0.1 0.1 <u>0.08</u> 0.04 0.03	1015/97, Tribolet (1998, LUFEN_068) REM. 118.01, LOQ : 0.02 mg/kg, 67–95% Recovery (n=3), Storage: 9 months
Spain, El Ejido 1997 (Taranto)	EC	3	0.1	0.009	89	Fruits	–0 0 3 7 14 21	0.02 0.08 0.09 0.05 0.11 <u>0.17</u>	1016/97, Tribolet (1998, LUFEN_069) REM. 118.01, LOQ : 0.02 mg/kg, 81–92% Recovery (n=2), Storage: 4 months
(Mazurca)	EC	3	0.1	0.009	89	Fruits	–0 0 3 7 14 21	0.02 0.05 0.09 0.06 0.03 0.07	1017/97, Tribolet (1998, LUFEN_070) REM. 118.01, LOQ : 0.02 mg/kg, 65–94% Recovery (n=2), Storage: 4 months
Spain, El Ejido 1997 (Cadia)	EC	3	0.1	0.009 0.008 0.008	89	Fruits	–0 0 3 7 14 21	0.09 0.15 0.1 <u>0.13</u> 0.12 0.13	1018/97, Tribolet (1998, LUFEN_070) REM. 118.01, LOQ : 0.02 mg/kg, 83–94% Recovery (n=2), Storage: 8 months
Spain, Adra 1998 (Genil)	EC	3	0.1	0.01	89	Fruits	3 7	0.18 (0.17, 0.18) <u>0.18</u> (0.16, 0.19)	1139/98, Tribolet (1999, LUFEN_077) REM. 118.01, LOQ : 0.02 mg/kg, 85–86% Recovery (n=2), Storage: 3 months
Spain, Motril 1998 (Ciclon)	EC	3	0.1	0.01	89	Fruits	3 7	<u>0.54</u> (0.51, 0.56) 0.47 (0.47, 0.47)	1140/98, Tribolet (1999, LUFEN_078) REM. 118.01, LOQ : 0.02 mg/kg, 85–86% Recovery (n=2), Storage: 3 months

–0=Sampling before last application

DAT=Days after last treatment

BBCH 81–88 = 10–80% of fruits show typical fully ripe colour

BBCH 89=Fully ripe: fruits have typical fully ripe colour

Table 51 Residues of lufenuron following foliar application to protected tomatoes

Location,	Application					Residues, mg/kg			Report/Trial No., Reference,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	lufenuron	analytical method, validation data, storage period
Greece, Kenurgio 1999 (Noa)	EC	3	0.15	0.01	81	Fruits	0 7	0.04 (0.03, 0.05) 0.04 (0.03, 0.04)	1097/99, Tribolet (2000, LUFEN_090) REM. 118.01, LOQ : 0.02 mg/kg, 83–87% Recovery (n=2), Storage: 5 months
Greece, Kenourio	EC	3	0.1	0.01	89	Fruits	0 7	0.03 <u>0.02</u>	1049/00, Salvi (2001, LUFEN_085)

Location,	Application					Residues, mg/kg			Report/Trial No., Reference,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	lufenuron	analytical method, validation data, storage period
2000 (Noa)								(0.02, 0.02)	REM. 118.01, LOQ : 0.02 mg/kg, 79–98% Recovery (n=2), Storage: 3 months
Greece, Kenourigio 2001 (Noa)	EC	3	0.1	0.01	89	Fruits	0 7	0.23 <u>0.24</u> (0.2, 0.29)	1066/01, Gasser (2003, LUFEN_087) REM. 118.01, LOQ : 0.02 mg/kg, 100–111% Recovery (n=2), Storage: 2 months
Spain, Perellonet 1998 (Marmanda)	EC	3	0.1	0.02 0.015 0.012	72	Fruits	0 3 7 15 22	0.06 0.06 (0.05, 0.07) 0.05 <u>0.06</u> 0.06	1013/99, Tribolet (1999, LUFEN_079) REM. 118.01, LOQ : 0.02 mg/kg, 76–84% Recovery (n=2), Storage: 6 months
Spain, Cullera 1998 (Welkor)	EC	3	0.1	0.02 0.015 0.012	72	Fruits	0 3 7 15 22	0.03 0.05 (0.04, 0.06) 0.07 <u>0.08</u> 0.03	1014/99, Tribolet (1999, LUFEN_081) REM. 118.01, LOQ : 0.02 mg/kg, 83–91% Recovery (n=2), Storage: 7 months
Spain, Los Palacios 1998 (Genaro)	EC	3	0.1	0.01	81	Fruits	0 3 7 14 21	0.12 0.06 0.07 <u>0.09</u> 0.05	1051/98, Tribolet (1999, LUFEN_086) REM. 118.01, LOQ : 0.02 mg/kg, 85–95% Recovery (n=2), Storage: 5 months
Spain, Los Palacios 1999 (Bond)	EC	3	0.1	0.01	89	Fruits	0 7	0.09 (0.07, 0.11) <u>0.04</u> (0.02, 0.05)	1126/99, Tribolet (1999, LUFEN_091) REM. 118.01, LOQ : 0.02 mg/kg, 88–116% Recovery (n=2), Storage: 5 months
Spain, Los Palacios 1999 (Genaro)	EC	3	0.1	0.01	89	Fruits	0 7	0.08 (0.06, 0.09) <u>0.08</u> (0.06, 0.09)	1127/99, Tribolet (1999, LUFEN_092) REM. 118.01, LOQ : 0.02 mg/kg, 88–116% Recovery (n=2), Storage: 5 months
Spain, Perellonet 2000 (Marmanda)	EC	3	0.11	0.011	72	Fruits	0 3 7 10	0.05 0.09 <u>0.1</u> (0.07, 0.13) 0.1	1014/00, Salvi (2001, LUFEN_080) REM. 118.01, LOQ : 0.02 mg/kg, 70–110% Recovery (n=2), Storage: 9 months
Spain, Los Palacios 2000 (Bond)	EC	3	0.11 0.11 0.1	0.01	75	Fruits	0 3 7 10	0.05 0.04 <u>0.05</u> (0.04, 0.06) 0.03	1015/00, Salvi (2001, LUFEN_082) REM. 118.01, LOQ : 0.02 mg/kg, 100–108% Recovery (n=2), Storage: 9 months
Spain, Los Palacios	EC	3	0.11 0.1	0.01	75	Fruits	0 7	0.04 <u>0.04</u>	1016/00, Salvi (2001, LUFEN_083)

Location,	Application					Residues, mg/kg			Report/Trial No., Reference,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	lufenuron	analytical method, validation data, storage period
2000 (Bond)			0.1					(0.03, 0.04)	REM. 118.01, LOQ : 0.02 mg/kg, 76–83% Recovery (n=2), Storage: 9 months
Spain, Los Palacios 2001 (Genaro)	EC	3	0.1	0.01	83	Fruits	0 7	0.1 <u>0.08</u> (0.07, 0.1)	1092/01, Gasser (2003, LUFEN_088) REM. 118.01, LOQ : 0.02 mg/kg, 98–100% Recovery (n=2), Storage: 3 months
Spain, Penaflor 2001 (Bond)	EC	3	0.09 0.1 0.1	0.01	74	Fruits	0 1 3 7	0.08 0.07 0.05 <u>0.08</u> (0.07, 0.08)	1093/01, Gasser (2003, LUFEN_089) REM. 118.01, LOQ : 0.02 mg/kg, 98–102% Recovery (n=2), Storage: 3 months
Switzerland, Chessel 1998 (Paola)	EC	3	0.1	0.005	72	Fruits	0 3 7 14	0.13 0.15 <u>0.11</u> 0.07	1024/98, Tribolet (1998, LUFEN_084) REM. 118.01, LOQ : 0.02 mg/kg, 87–91% Recovery (n=2), Storage: 3 months

DAT=Days after last treatment

BBCH 71–79=1st–9th fruit has reached typical size

BBCH 81–88=10–80% of fruits show typical fully ripe colour

BBCH 89=Fully ripe: fruits have typical fully ripe colour

Table 52 Residues of lufenuron following foliar application to sweet corn (Method POP-PAT-004 v.03, LOQ: 0.01 mg/kg, 94–119% Recovery (n=6), Storage: 7 months)

Location,	Application					Residues, mg/kg			Report/Trial No., Reference
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	lufenuron	
Brazil, Bairro Lagoa Bonita 2009 (AL Bandeirante)	EC	2	0.015	0.005	53	Kernels	35	< 0.01	M09089-LZF, Matarazzo (2012, LUFEN_094)
Brazil, Colónia Benifica 2009 (30 R 50)	EC	2	0.015	0.005	51	Kernels	35	< 0.01	M09089-DMO, Matarazzo (2012, LUFEN_094)
Brazil, Rodovia Nova Veneza 2009 (Impacto)	EC	2	0.015	0.005	51	Kernels	35	< 0.01	M09089-MFG, Matarazzo (2012, LUFEN_094)
Brazil, Rodavia 2009 (Master)	EC	2	0.015	0.005	69	Kernels	35	< 0.01	M09089-JJB, Matarazzo (2012, LUFEN_094)

DAT=Days after last treatment

BBCH 51=Beginning of tassel emergence: tassel detectable at top of stem

BBCH 53=Tip of tassel visible

BBCH 69=End of flowering: stigmata completely dry

Table 53 Residues of lufenuron following foliar application to soybeans

Location,	Application					Residues, mg/kg			Report/Trial No., Reference
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	lufenuron	
Brazil, Rodovia	EC	4	0.038	0.025	76	Seeds, dry	35	< 0.01	T06014-JJB1, Ribeiro (2008,

Location,	Application					Residues, mg/kg			Report/Trial No., Reference
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	lufenuron	
2007 (Conquista)	EC	4	0.075	0.05	76	Seeds, dry	35	< 0.01	LUFEN_097)
Brazil, Rodovia	EC	4	0.038	0.025	88	Seeds, dry	35	< 0.01	T06014-JJB2, Ribeiro (2008,
2007 (BRS Valiosa)	EC	4	0.075	0.05	88	Seeds, dry	35	< 0.01	LUFEN_097)
Brazil, Ponta Grossa	EC	4	0.038	0.025	75	Seeds, dry	35	< 0.01	T06014-DMO, Ribeiro (2008,
2007 (CD 206)	EC	4	0.075	0.05	75	Seeds, dry	35	< 0.01	LUFEN_097)
Brazil, Rodovia 2009 (NK 9074 RR)	EC	2	0.008	0.004	80	Seeds, dry	35	< 0.01	M09092-JJB, Roncato (2011, LUFEN_098)
Brazil, Rodovia Nova Venecia 2009 (NK 9074)	EC	2	0.008	0.004	79	Seeds, dry	35	< 0.01	M09092-MFG, Roncato (2011, LUFEN_098)
Brazil, Carambei 2009 (BRS 230)	EC	2	0.008	0.004	81	Seeds, dry	35	< 0.01	M09092-DMO1, Roncato (2011, LUFEN_098) months
Brazil, Itaberá 2009 (M 5942)	EC	2	0.008	0.004	81	Seeds, dry	35	< 0.01	M09092-DMO2, Roncato (2011, LUFEN_098)

Ribeiro (2008, LUFEN_097)=Method POPIT MET.015 Rev 01, LOQ: 0.01 mg/kg, 85–106% Recovery (n=10), Storage: 5 months

Roncato (2011, LUFEN_098)=Method POP PAT 004 V00, LOQ: 0.01 mg/kg, 72–104% Recovery (n=10), Storage: 5 months

DAT=Days after last treatment

BBCH 75–79=About 50–100% of pods have reached final length (15–20 mm).

BBCH 81–88=About 10–80% of pods are ripe; beans final colour, dry and hard

Table 54 Residues of lufenuron following foliar application to potatoes (Method POP-PAT-004 v.04, LOQ: 0.01 mg/kg, 71–80% Recovery (n=10), Storage: 3 months)

Location,	Application					Residues, mg/kg			Report/Trial No., Reference
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	lufenuron	
Brazil, Pouso Alegre 2009 (Cupido)	EC	4	0.04	0.005	47	Tubers	7 14 21	< 0.01 < 0.01 < 0.01	M09086-JJB, Matarazzo (2012, LUFEN_093)
Brazil, Piedade 2009 (Agata)	EC	4	0.04	0.005	46	Tubers	7 14 21	< 0.01 < 0.01 < 0.01	M09086-LZF, Matarazzo (2012, LUFEN_093)
Brazil, Curitibanos 2009 (Atlantic)	EC	4	0.04	0.005	44	Tubers	7 14 21	< 0.01 < 0.01 < 0.01	M09086-DMO1, Matarazzo (2012, LUFEN_093)
Brazil, Carambei 2009 (Atlantic)	EC	4	0.04	0.005	44	Tubers	7 14 21	< 0.01 < 0.01 < 0.01	M09086-DMO2, Matarazzo (2012, LUFEN_093)

DAT=Days after last treatment

BBCH 41–47=10–70% of total final tuber mass reached

Table 55 Residues of lufenuron following foliar application to maize (Method POP-PAT-004 v.03, LOQ: 0.01 mg/kg, 94–119% Recovery (n=6), Storage: 7 months)

Location,	Application					Residues, mg/kg			Report/Trial No., Reference
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	lufenuron	
Brazil, Bairro Lagoa Bonita 2009 (AL Bandeirante)	EC	2	0.015	0.005	53	Grain	82	< 0.01	M09089-LZF, Matarazzo (2012, LUFEN_094)
Brazil, Colónia Benifica 2009 (30 R 50)	EC	2	0.015	0.005	51	Grain	66	< 0.01	M09089-DMO, Matarazzo (2012, LUFEN_094)
Brazil, Rodovia Nova	EC	2	0.015	0.005	51	Grain	78	< 0.01	M09089-MFG, Matarazzo

Location,	Application					Residues, mg/kg			Report/Trial No., Reference
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	lufenuron	
Veneza 2009 (Impacto)									(2012, LUFEN_094)
Brazil, Rodavia 2009 (Master)	EC	2	0.015	0.005	69	Grain	56	< 0.01	M09089-JJB, Matarazzo (2012, LUFEN_094)

DAT=Days after last treatment

BBCH 51=Beginning of tassel emergence: tassel detectable at top of stem

BBCH 53=Tip of tassel visible

BBCH 69=End of flowering: stigmata completely dry

Table 56 Residues of lufenuron following foliar application to sugarcane (Method POPIT MET.077, LOQ: 0.01 mg/kg, 83–108% Recovery (n=12), Storage: 3 months)

Location,	Application					Residues, mg/kg			Report/Trial No., Reference
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	lufenuron	
Brazil, Rodovia 2007 (SP 803280)	EC	2	0.025	0.013	45	Sugarcane	7 14 21 28	< 0.01 < 0.01 < 0.01 < 0.01	M08083-LZF1, Marconi (2008, LUFEN_095)
Brazil, Rio das Pedras 2007 (RB 3280)	EC	2	0.025	0.013	49	Sugarcane	7 14 21 28	< 0.01 0.02 < 0.01 < 0.01	M08083-LZF2, Marconi (2008, LUFEN_095)
Brazil, Baneirantes 2007 (RB 415)	EC	2	0.025	0.013	45	Sugarcane	7 14 21 28	< 0.01 0.02 < 0.01 0.02	M08083-LZF3, Marconi (2008, LUFEN_095)
Brazil, Tupaciguara 2007 (SP 832847)	EC	2	0.025	0.013	39	Sugarcane	7 14 21 28	0.01 0.01 0.02 0.02	M08083-JJB, Marconi (2008, LUFEN_095)

DAT=Days after last treatment

BBCH 39 = Maximum stem length or rosette diameter reached

BBCH 45–49=50–100% of harvestable vegetative plant parts or vegetatively propagated, organs have reached final size

Table 57 Residues of lufenuron following foliar application to cotton (unnamed HPLC-UV method, LOQ : 0.05 mg/kg, 84–94% Recovery)

Location,	Application					Residues, mg/kg			Report/Trial No., Reference
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	lufenuron	
China, Changsha 2007 (Xiangmian 15)	EC	1	0.023	n/s	n/s	Seeds	10 20 30	< 0.05 < 0.05 < 0.05	A7814A, Renbin (2008, LUFEN_096)
		2	0.023	n/s	n/s	Seeds	10 20 30	< 0.05 < 0.05 < 0.05	
		1	0.034	n/s	n/s	Seeds	10 20 30	< 0.05 < 0.05 < 0.05	
		2	0.034	n/s	n/s	Seeds	10 20 30	< 0.05 < 0.05 < 0.05	
China, Zhengzhou 2007	EC	1	0.023	n/s	n/s	Seeds	10 20 30	< 0.05 < 0.05 < 0.05	A7814A, Renbin (2008, LUFEN_096)

Location,	Application					Residues, mg/kg			Report/Trial No., Reference
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	lufenuron	
(Zhongmian 41)		2	0.023	n/s	n/s	Seeds	10 20 30	< 0.05 < 0.05 < 0.05	
		1	0.034	n/s	n/s	Seeds	10 20 30	< 0.05 < 0.05 < 0.05	
		2	0.034	n/s	n/s	Seeds	10 20 30	< 0.05 < 0.05 < 0.05	
China, Changsha 2008 (Xiangmian 15)	EC	1	0.023	n/s	n/s	Seeds	10 20 30	< 0.05 < 0.05 < 0.05	A7814A, Renbin (2008, LUFEN_096)
		2	0.023	n/s	n/s	Seeds	10 20 30	< 0.05 < 0.05 < 0.05	
		1	0.034	n/s	n/s	Seeds	10 20 30	< 0.05 < 0.05 < 0.05	
		2	0.034	n/s	n/s	Seeds	10 20 30	< 0.05 < 0.05 < 0.05	
China, Zhengzhou 2008	EC	1	0.023	n/s	n/s	Seeds	10 20 30	< 0.05 < 0.05 < 0.05	A7814A, Renbin (2008, LUFEN_096)
(Zhongmian 41)		2	0.023	n/s	n/s	Seeds	10 20 30	< 0.05 < 0.05 < 0.05	
		1	0.034	n/s	n/s	Seeds	10 20 30	< 0.05 < 0.05 < 0.05	
		2	0.034	n/s	n/s	Seeds	10 20 30	< 0.05 < 0.05 < 0.05	

n/s=Not stated

DAT = Days after last treatment

Table 58 Residues of lufenuron following foliar application to coffee (Method POPIT MET.077, LOQ: 0.01 mg/kg, 91–106% Recovery (n=12), Storage: 6 months)

Location,	Application					Residues, mg/kg			Report/Trial No., Reference
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	lufenuron	
Brazil, Holambra 2006 (Mundo Novo)	EC	2	0.04	0.01	87	Green beans (dry processed)	3 7 10	0.01 0.01 < 0.01	M05035-LZF1, Gois Fatima (2007, LUFEN_099)
Brazil, Santa Amélia 2006 (Obatá)	EC	2	0.04	0.01	87	Green beans (dry processed)	3 7 10	< 0.01 < 0.01 < 0.01	M05035-LZF2, Gois Fatima (2007, LUFEN_099)
Brazil, Monte Carmelo 2006 (Mundo novo)	EC	2	0.04	0.01	89	Green beans (dry processed)	3 7 10	< 0.01 < 0.01 < 0.01	M05035-JJB, Gois Fatima (2007, LUFEN_099)

DAT = Days after last treatment

BBCH 88 = Fruit is fully-ripe color and ready for picking

Fate of residues in storage and processing

Nature of residue during processing

The hydrolysis of lufenuron under processing conditions was investigated by Grout (2003, LUFEN_100). [^{14}C]diflurophenyl and [^{14}C]dichorophenyl labelled lufenuron was incubated in aqueous buffer solutions at a nominal concentration of 5 mg/L under three sets of conditions, each designed to simulate an appropriate process: 90 °C (pH 4, 20 minutes) to simulate pasteurisation, 100 °C (pH 5, 60 minutes), to simulate boiling, baking and brewing, and 120 °C (pH 6, 20 minutes) to simulate sterilisation.

Total recovered radioactivity was measured for each test solution. Radioactive components were characterized by fractionation and co-chromatography with authenticated reference compounds using HPLC.

Table 59 Hydrolysis of [^{14}C]dichorophenyl labelled lufenuron under simulated processing conditions

		% applied radioactivity				
Process represented	Sample	Lufenuron	CGA224443	CGA238277	Unknowns	Recovery
PH 4 90 °C 20 mins	1	99.0	0.0	0.0	0.0	100.9
	2	101.5	0.0	0.0	0.0	104.2
PH 5 100 °C 60 mins	1	93.4	6.3	0.5	1.1	103.5
	2	97.3	6.9	0.4	1.1	108.0
PH 6 120 °C 20 mins	1	114.0	0.0	0.0	0.0	115.9
	2	100.4	0.0	0.0	0.0	102.6

Table 60 Hydrolysis of [^{14}C]diflurophenyl labelled lufenuron under simulated processing conditions

		% applied radioactivity				
Process represented	Sample	Lufenuron	CGA149772	CGA149766	Unknowns	Recovery
PH 4 90 °C 20 mins	1	97.2	0.5	0.0	0.0	99.2
	2	100.9	0.6	0.0	0.0	103.3
PH 5 100 °C 60 mins	1	99.7	6.9	0.7	0.4	110.0
	2	99.7	3.8	0.5	0.2	106.2
PH 6 120 °C 20 mins	1	100.3	0.0	0.0	0.0	101.6
	2	102.9	0.0	0.0	0.0	104.9

Residues after processing

The fate of lufenuron during processing of raw agricultural commodity (RAC) was investigated in tomatoes using important processing procedures. As a measure of the transfer of residues into processed products, a processing factor was used, which is defined as:

Processing factor=Residue in processed product (mg/kg) ÷ Residue in raw agricultural commodity (mg/kg)

If residues in the RAC were below the LOQ, no processing factor could be derived. In case of residues below the LOQ, but above the LOD in the processed product, the numeric value of the LOQ was used for the calculation. If residues in the processed product were below the LOD, the numeric value of the LOQ was used for the calculation but the PF was expressed as "less than" (e.g. < 0.5).

Tomato

A study on the behaviour of lufenuron during processing of tomatoes was conducted by Sole (2003, LUFEN_101). Tomatoes grown outdoor in Southern France were treated three times with 0.03 kg lufenuron/ha each at one week intervals. Samples were harvested 8 days after the last application. Tomatoes were used for the production of tomato juice, canned tomato and tomatoes puree. The field sample was split into subsamples processed multiple times for each commodity:

- The washed tomatoes were produced by washing for 2 minutes in cold running water.
- Tomato juice was produced by quartering and blanching the washed tomatoes followed by sieving to remove the peel and seeds (wet pomace). The raw juice was pasteurised (20 minutes at 99 °C).
- Tomato puree was produced by concentrating raw juice to approximately 30% dry matter and then pasteurising (20 minutes at 93–95 °C).
- Canned tomatoes were produced by blanching washed tomatoes to remove the peel. The peeled tomatoes and portion of tomato juice from the juicing process were then sterilised in tins.

Table 61 Summary of lufenuron residues in tomato and processed commodities from a trial conducted in Southern France (Sole 2003, LUFEN_101)

Commodity	Lufenuron in mg/kg	Processing factor	Median or best estimate processing factor
Fruits (RAC)	0.029	–	–
Raw juice	< 0.005, 0.005	< 0.17, 0.17	0.17
Pasteurized juice	< 0.005(4)	< 0.17(4)	0.17
Wet pomace	0.23, 0.23, 0.25, 0.28	7.9, 7.9, 8.6, 9.7	8.3
Canned tomato	< 0.005(4)	< 0.17(4)	0.17
Raw paste	0.024, 0.032	0.83, 1.1	0.97
Pasteurized puree	0.023, 0.024, 0.025, 0.026	0.79, 0.83, 0.86.0.9	0.85

RAC=Raw agricultural commodity

Residues in animal commodities

Farm animal feeding studies

For the estimation of residues of lufenuron in animal matrices one lactating cow feeding study and one steer feeding study were submitted to the Meeting.

Lactating cows

In the first study residues in lactating cows were investigated by Tribolet (1995, LUFEN_102). The dose rates were approximately 0, 39, 230 and 415 µg lufenuron/kg body weight/day (equivalent to nominal concentrations of 0, 0.82, 4.3 and 8.6 mg/kg in the daily feed).

The cows in the treatment groups were fed with the lufenuron twice daily with the active ingredient mixed with pelleted feed, for a period of 28–29 days. Milk samples were collected pre-treatment and throughout the dosing period. At Day 29–30 the cows were slaughtered and samples of muscle (tenderloin, round steak), liver, kidney and fat (omental and peri-renal) were taken for analysis.

Milk and tissues were extracted and analysed for lufenuron using method REM 118.04. The LOQ for milk, blood and tissues are 1 µg/L, 10 µg/L and 0.01 mg/kg respectively.

In the control group no detectable residues of lufenuron were found. The findings in milk and tissues are summarized in the following table.

Table 62 Residues of lufenuron in cow tissues and milk following administration of lufenuron at 0.82, 4.3 and 8.6 ppm in the diet

Commodity	Sampling Interval (days)	Maximum Lufenuron Residues (mg/kg)		
		Group 2 (0.82 ppm)	Group 3 (4.3 ppm)	Group 4 (8.6 ppm)
Milk	1	0.005, 0.013, 0.012 (0.01)	0.062, 0.104, 0.16 (0.11)	0.28, 0.13, 0.14 (0.18)
	4	0.062, 0.105, 0.05 (0.072)	0.38, 0.48, 0.84 (0.57)	1.2, 1.2, 0.68 (1.0)
	7	0.076, 0.098, 0.036 (0.07)	0.62, 0.505, 0.565 (0.58)	1.3, 0.82, 0.6 (0.9)
	10	0.095, 0.12, 0.13 (0.12)	0.56, 0.76, 0.96 (0.76)	2.0, 1.4, 1.8 (1.7)

Commodity	Sampling Interval (days)	Maximum Lufenuron Residues (mg/kg)		
		Group 2 (0.82 ppm)	Group 3 (4.3 ppm)	Group 4 (8.6 ppm)
	14	0.136, 0.16, 0.121 (0.14)	0.68, 0.84, 0.96 (0.83)	2.2, 2.2, 1.6 (2.0)
	17	0.118, 0.184, 0.125 (0.14)	0.61, 0.84, 0.96 (0.89)	2.2, 2.1, 1.7 (2.0)
	21	0.15, 0.188, 0.132 (0.16)	0.71, 0.94, 1.15 (0.93)	2.1,2.7, 1.8 (2.2)
	24	0.105, 0.197, 0.122 (0.14)	0.55, 1.23, 1.18 (0.99)	2.3, 2.3, 2.8 (2.5)
	28	0.12, 0.167, 0.168 (0.15)	0.85, 0.99, 0.77 (0.87)	1.6, 1.9, 1.4 (1.6)
Milk—skim milk	28	0.007, 0.004, 0.008 (0.006)	0.023, 0.059, 0.032 (0.038)	0.049, 0.058, 0.057 (0.054)
Milk—cream	28	4.3, 2.3, 2.6 (3.1)	25, 28, 19 (24)	27, 30, 39 (32)
Muscle—tenderloin	29	0.02, 0.04, 0.04 (0.03)	0.09, 0.15, 0.26 (0.17)	0.26, 0.49, 0.54 (0.43)
Muscle—round steak	29	0.01, 0.02, 0.02 (0.02)	0.04, 0.09, 0.12 (0.08)	0.09, 0.16, 0.34 (0.2)
Liver	29	0.05, 0.06, 0.07 (0.06)	0.32, 0.39, 0.39 (0.37)	0.64, 0.67, 0.99 (0.77)
Kidney	29	0.03, 0.03, 0.04 (0.03)	0.19, 0.23, 0.23 (0.22)	0.32, 0.35, 0.42 (0.36)
Fat—peri-renal	29	0.53, 0.56, 0.84 (0.64)	3.9, 4.2, 5.3 (4.5)	6.3, 7.7, 10.1 (8.0)
Fat—omental	29	0.42, 0.57, 1.2 (0.73)	3.5, 3.6, 4.1 (3.7)	6.2, 7.0, 8.9 (7.4)

Steer

Residues of lufenuron in steer were also investigated by Tribolet (2000, LUFEN_103). Three groups of steers, Angus X Hereford, were used in this study, two treated and one control group. One group of three steers was dosed with capsules containing 0.2 mg lufenuron and a further treatment group of 12 steers were dosed with 10 mg of lufenuron. Each treatment group was dosed for 28 consecutive days. The dose rates were equivalent to nominal concentrations of 0.02 and 1 mg/kg in the daily feed (0.0006 and 0.031 mg/kg bw/day).

The lower dose group and three steers from the higher group were sacrificed 20–24 hours after the final dose and a further three steers were sacrificed at two week intervals, i.e. days 42, 56 and 70 after the commencement of dosing. At sacrifice, samples of blood, muscle (tenderloin, round steak), liver, kidney and fat (omental and peri-renal) were taken for analysis.

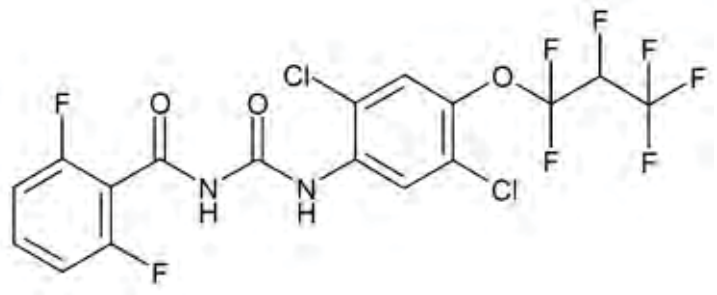
Tissues were extracted and analysed for lufenuron using method REM 118.04. The LOQ for blood and tissues are 2 µg/L, and 0.01 mg/kg respectively.

Table 63 Residues of lufenuron in steer tissues following administration of lufenuron at 0.02 and 1 ppm in the diet

Days	Lufenuron residues in mg/kg					
	Muscle—tenderloin	Muscle—round steak	Liver	Kidney	Fat—peri-renal	Fat—omental
Low dose group (0.02 ppm)						
28	< 0.01, < 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01, < 0.01 (< 0.01)	0.022, 0.038, 0.035 (0.032)	0.024, 0.038, 0.045 (0.036)
High dose group (1 ppm)						
28	< 0.01, < 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01, < 0.01 (< 0.01)	0.018, 0.027, 0.025 (0.023)	0.032, 0.022, 0.023 (0.026)	0.15, 0.26, 0.27 (0.23)	0.16, 0.24, 0.26 (0.22)
42	< 0.01, < 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01, < 0.01 (< 0.01)	0.01, 0.01, 0.01 (0.01)	0.011, 0.011, 0.014 (0.012)	0.066, 0.081, 0.1 (0.082)	0.071, 0.084, 0.12 (0.092)
56	< 0.01, < 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01, < 0.01 (< 0.01)	< 0.01, 0.01, 0.01 (0.01)	< 0.01, 0.01, 0.011 (0.01)	0.057, 0.072, 0.082 (0.07)	0.061, 0.077, 0.086 (0.075)
70	< 0.01, < 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01, 0.013 (0.011)	0.038, 0.038, 0.055 (0.044)	0.039, 0.041, 0.065 (0.048)

APPRAISAL

Lufenuron (ISO common name) is an insect growth inhibitor that is active against larvae of Lepidoptera and Coleoptera. When ingested, lufenuron interferes with chitin synthesis, and prevents larvae from moulting. It was considered for the first time by the 2015 JMPR for toxicology and residues.



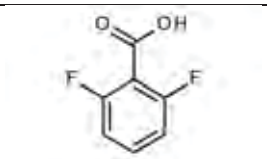
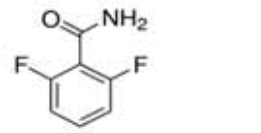
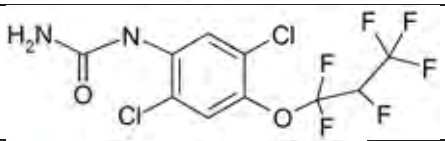
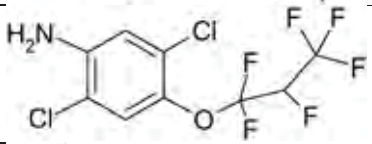
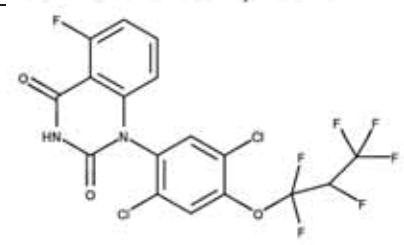
The IUPAC name of lufenuron is (RS)-1-[2,5-dichloro-4-(1,1,2,3,3,3-hexafluoropropoxy)phenyl]-3-(2,6-difluorobenzoyl)urea and the CA name is N-[[[2,5-dichloro-4-(1,1,2,3,3,3-hexafluoropropoxy)phenyl]amino]carbonyl]-2,6-difluorobenzamide.

Lufenuron consists of a pair of enantiomers. A chiral centre exists at the 2-position of the hexafluoropropoxy side-chain. Lufenuron technical active ingredient is manufactured under non-stereospecific conditions giving a racemate (R:S 50:50).

The physical-chemical properties of lufenuron indicate low volatility and no accelerated photochemical degradation in water. The octanol-water partition coefficient, log $P_{ow,}$ is 5.12.

Lufenuron radio-labelled either in the dichlorophenyl- or difluorophenyl-moiety was used in the metabolism and environmental fate studies.

The following abbreviations are used for the metabolites discussed below:

CGA149776	2,6-Difluoro-benzoic acid	
CGA149772	2,6-Difluoro-benzamide	
CGA238277	2,5-Dichloro-4-(1,1,2,3,3,3-hexafluoropropoxy)-phenyl-urea	
CGA224443	N-[2,5-dichloro-4-(1,1,2,3,3,3-hexafluoropropoxy)-benzenamine	
CGA301018	no chemical name submitted	

Environmental fate in soil

The Meeting received information for lufenuron on soil photolysis, aqueous hydrolysis, aerobic soil metabolism and soil degradation.

Soil photolysis using [dichlorophenyl-^{14}C]-lufenuron and [difluorophenyl-^{14}C]-lufenuron revealed no significant degradation (84–99% parent remaining after 17 days of continuous irradiation).

Hydrolysis in aqueous solutions representative of environmental conditions (25 °C) showed virtually no degradation at pH 5, 7 and 9 within 5 days. Under more extreme conditions the parent substance was stable at pH 1 and 70°C, representing more than 90% of the radioactivity. At pH 9 an accelerated degradation was observed at 50 °C and 70 °C with 0–53% of the parent remaining after 1–5 days. Depending on the label the cleavage products CGA224443 and CGA238277 and its counterparts CGA149776 and 2,6-difluorobenzamide (CGA149772) were observed. In addition both labelled compounds produced CGA301018 by loss of fluoride and ring closure.

In the aerobic soil metabolism studies lufenuron was degraded with half-lives of 9–24 days in microbial active soil and 17–83 days in sterilised soil. Cleavage of the parent molecule was the primary degradation step, leaving CGA238277 and CGA224443 for [dichlorophenyl-^{14}C]-lufenuron. For [difluorophenyl-^{14}C]-lufenuron no metabolites were identified. Unextracted residues in soil at the end of the studies were between 25–79% of the AR. Mineralisation ranged up to 59% AR.

2,6-difluorobenzamide (CGA149772), which is a common soil metabolite to other active substances, e.g., diflubenzuron, was investigated separately for its behaviour in soil. Within 120 days it was completely degraded, leaving CGA149776 as its main degradate within the first two weeks. Afterward the radioactivity was further degraded and remained unextracted (up to 41% AR) or was mineralized (up to 65% AR).

The soil degradation of lufenuron and its metabolites CGA238227 and CGA224443 was also investigated on three different soils under laboratory conditions. Following 1st-order kinetic, DT_{50} and DT_{90} values of 13.7 d and 81.1d for lufenuron, 12.8 d and 42.5 d for CGA238277 and of 35.8 d and 119 d for CGA224443 were calculated, respectively.

In summary the Meeting concluded that lufenuron is moderately quickly degraded in soil under laboratory conditions, presumably by microbial activity. To assess the degradation behaviour under field conditions, field dissipation studies would be required. The residue is stable against photolysis and hydrolysis under environmental conditions, however at high temperature and basic conditions cleavage of the parent molecule was observed.

Plant metabolism

The Meeting received plant metabolism studies for lufenuron following foliar application of either [dichlorophenyl-^{14}C]-lufenuron or [difluorophenyl-^{14}C]-lufenuron in cabbage, tomato and cotton.

For cabbage the metabolism of lufenuron was investigated with [dichlorophenyl-^{14}C]-lufenuron only. Greenhouse plants received three spray applications equivalent to 0.02 kg ai/ha each in two week intervals. Samples were taken one hour after the first and last application, and at crop maturity, 28 days after the last application.

In mature cabbage heads TRR levels were 0.195 mg eq/kg (up to 1.8 mg eq/kg in withered leaves). 97.5% of the TRR (0.19 mg eq/kg) was recovered as unchanged lufenuron. In the head cabbage as well as in withered leaves, CGA238277 was identified at estimated levels of 0.6% and 3.3% of the TRR, respectively. The actual amounts were not measured in the TLC system used. No further metabolites were found.

For tomatoes the metabolism of lufenuron was investigated with [dichlorophenyl-^{14}C]-lufenuron only. Fruit bearing plants kept in a protected environment were treated with three sprayings equivalent to 0.03 kg ai/ha per application with one week intervals. Samples were collected directly

after the first application and up to 28 DALA. In parallel 34µg lufenuron was directly injected into single fruits, which were sampled after 18 and 33 days.

Directly after the last foliar application, TRR levels in fruits were 1.2 mg eq/kg, degrading to 0.69 mg eq/kg after 28 days. TRR levels found in additional samples at 28 DAT were 0.47 mg eq/kg for leaves and 0.44 mg eq/kg in mature fruits. Newly developed green fruits had much lower total radioactive residues of 0.03 mg eq/kg. In all fruits receiving a foliar treatment > 89% of the residue was recovered in the surface wash. Unextracted residues were generally low (< 0.6% TRR).

The identification of the radioactivity (combined surface wash and extract) showed unchanged lufenuron as the major residue in fruits and leaves (93–98% TRR). Only in one fruit sample collected 28 DAT, minor amounts of CGA238277 (0.2% TRR, 0.0013 mg eq/kg) were found.

In mature fruits receiving a direct injection of lufenuron, the results from the extracts were comparable to foliar treated fruits. 90–95% of the radioactivity was identified as unchanged lufenuron. CGA238277 was identified in minor amounts up to 2% of the TRR. 5% of the TRR remained unextracted.

For cotton grown under glasshouse conditions the metabolism was investigated in two studies using [dichlorophenyl-^{14}C]-lufenuron or [difluorophenyl-^{14}C]-lufenuron.

For [dichlorophenyl-^{14}C]-lufenuron cotton plants received three foliar sprayings equivalent to 0.03 kg ai/ha each at 14 day interval, beginning at flowering. Sampling of leaves took place 1 hour, 1 day, 3 and 7 days after the first application and 14 days, 28 and 84 days (maturity) after the last application. In addition, four cotton plants received three stem injections (100 µg lufenuron each) made at 14-day intervals.

TRR levels found were up to 4.9 mg eq/kg in the leaves, < 0.001 mg eq/kg in seeds, 0.092 mg eq/kg in hulls and 0.001 mg eq/kg in green bolls. In leaves the amount of radioactivity in the surface wash decreased from 98% TRR after application 1 to 43% TRR at maturity (84 DALA).

The identification of the radioactivity (combined surface wash and extracts) showed 89–100% of the TRR as unchanged lufenuron. No metabolites were identified. In seeds and green bolls TRR levels were too low for further identification. Unextracted residues did not exceed 3.3% of the TRR.

The stem injection showed that most of the applied radioactivity remained at the injection site (81.2% AR). Minor translocation was observed into adjected stalks (13.3% AR) and leaves (1.6–3.9% AR). In all samples the unchanged parent was the only residue identified (~95–98% TRR).

For [difluorophenyl-^{14}C]-lufenuron the use pattern was comparable to the other label, but only the foliar treatment experiment was conducted. Samples of mature plant parts were collected 52 DALA.

TRR levels found were up to 5.95 mg eq/kg in leaves (52 DALA), 0.69 mg eq/kg in hulls and 0.003 mg eq/kg in seeds. In the leaves the surface wash contained most of the residue with 96% TRR directly after treatment and 49–58% TRR at maturity (52 DALA).

The identification again revealed unchanged lufenuron exclusively, representing >92% of the TRR in leaves and 79–83% TRR in other matrices. The TRR found in seeds was too low for identification. No further metabolites were detected.

Two confined rotational crop studies for lufenuron were submitted

In the first study [difluorophenyl-^{14}C]-lufenuron was applied under protected conditions to bare soil at a rate equivalent to 0.15 kg ai/ha. Lettuce, spring wheat, maize and carrots were planted in the treated soil 63 days after test substance application. The transfer of radioactivity into succeeding crops was very limited. In mature lettuce (126 d after treatment) the highest TRR level of 0.047 mg eq/kg was found. 53% of the TRR was identified as unchanged parent (0.025 mg/kg). In other matrices only wheat straw (0.023 mg eq/kg, 0.007 mg lufenuron/kg) and immature carrots roots (0.023 mg eq/kg, no identification conducted) showed total radioactive residues above 0.01 mg eq/kg. No further identification was conducted for these matrices. In soil samples, nearly the entire extracted

radioactivity was attributed to lufenuron. No residue of CGA149772 or CGA149776 could be identified in any sample.

In a second confined study conducted under field conditions [dichlorophenyl-^{14}C]-lufenuron was applied to bare soil once at a rate equivalent to 0.13 kg ai/ha. After different plant-back intervals (PBI) lettuce (PBI 76 d), winter wheat (PBI 126 d), sugar beets (PBI 306 days) and maize (PBI 331 d) were planted/sown and grown to maturity. TRR levels in all plant samples was between < 0.001 mg eq/kg and 0.004 mg eq/kg, which was too low for further identification.

In summary lufenuron is deposited on the plant surface and slowly adsorbed by leaves following direct treatment. On the surface and in plant tissue, the active substance is the only residue present in major amounts. Minor amounts of CGA238277 were identified in cabbage and tomato (up to 3.3% TRR). All plant metabolism studies for lufenuron were conducted under protected conditions. However, since lufenuron is not subject to photolysis the residue pattern in plants grown under field conditions is expected to be similar. Also, two of three studies were conducted with [dichlorophenyl-^{14}C]-lufenuron only. Since nearly the entire applied radioactivity was recovered as unchanged parent compound in these studies, no investigations with a second label are considered necessary.

For rotational crops the transfer of residues into succeeding crops from soil is very limited and mostly resulted in TRR levels too low for identification. In soil and in crop samples subject to identification parent lufenuron was the major residue. No further metabolites were identified.

Animal metabolism

Information was available on metabolism of lufenuron in laboratory animals, lactating goats and laying hens. Studies on rats, mice and dogs were evaluated by the WHO Core Assessment Group.

For lactating goats two studies were conducted involving daily administration of either ^{14}C-difluorophenyl-labelled lufenuron at 5.4 ppm (0.135 mg/kg bw) or ^{14}C-dichlorophenyl-lufenuron at 6.0 ppm (0.15 mg/kg bw) for ten consecutive days. The animals were slaughtered approximately 24h after the last dose.

The total recovery of the administered radioactivity was 95% for both labels. The majority of the radioactivity (73–74%) was found in the faeces. Radioactive residues in the edible tissues were 0.8–1.6% AR in muscle (0.038–0.08 mg eq/kg), 4.2–5.4% AR in fat (0.82–2.4 mg eq/kg), 0.28–0.3% AR in liver (0.37–0.42 mg eq/kg), 0.01–0.02% AR in kidney (0.11–0.12 mg eq/kg) and 5.8–6.8% AR in milk (up to 1.0 mg eq/kg). A plateau in milk was observed after approximately one week.

In tissues and milk unchanged parent was the only residue identified for both radiolabels, representing 73–94% of the TRR. The remaining radioactivity remained unresolved in the TLC-System used (6.6–19% TRR) or was not extracted from the sample (0.6–8.9% TRR).

Also for laying hens two studies were conducted involving daily administration of either ^{14}C-difluorophenyl-labelled lufenuron at 3.4 ppm (2.6 mg/kg bw) or ^{14}C-dichlorophenyl-lufenuron at 5.2 ppm (3.5 mg/kg bw) for fourteen consecutive days. The animals were slaughtered approximately 24h after the last dose.

The total recovery of the administered radioactivity was 75–79%. The majority of the radioactivity (54–62%) was found in the excreta. Radioactive residues in the edible tissues were 0.55–1.2% AR in lean meat (0.1–0.24 mg eq/kg), 5.1–9.9% AR in fat (7.2–13 mg eq/kg), 0.4–0.58% AR in liver (0.83–1.5 mg eq/kg), 0.07–0.09% AR in kidney (0.52–0.74 mg eq/kg) and 8.7–9.6% AR in eggs (up to 0.016 mg eq/kg in egg white and 8.5 mg eq/kg in egg yolk). In eggs a plateau was observed after one week for ^{14}C-difluorophenyl-lufenuron while residues for ^{14}C-dichlorophenyl-lufenuron showed a slight increase until the end of dosing.

In tissues and eggs unchanged parent lufenuron was the predominant residue, representing 79–94% TRR in all matrices except egg white. For the difluorophenyl-label the cleavage product CGA149772 was the only metabolite detected, being present in egg white at 0.001 mg eq/kg (17.3% TRR). For the dichlorophenyl-label its counterpart CGA238277 was found in minor amounts in kidney (0.028 mg eq/kg, 5.3% TRR) and egg white (< 0.001 mg eq/kg, 7.0% TRR). The remaining

radioactivity remained unresolved in the TLC-System used (3–42% TRR) or was not extracted from the sample (2–11% TRR).

In summary the metabolic degradation of lufenuron in livestock animals is very limited, showing parent as the predominant residue in all matrices. Minor amounts of the cleavage products CGA149772 and CGA238277 were found in poultry kidney and egg white.

Methods of residue analysis

The Meeting received analytical methods for the analysis of lufenuron in plant and animal matrices. The basic principle employs extraction by homogenisation with methanol or water and partitioning against hexane: ethyl ether (9:1,v:v). Clean-up is normally achieved by C18 solid-phase extraction. Residues are determined by liquid chromatography (LC) in combination with UV (255 nm) or tandem mass spectroscopy (MS/MS). Mass-transitions are m/z 509.1 → 326 for quantification and m/z 509→ 175 for confirmation. The methods submitted are suitable for measuring residues with a LOQ of 0.01 mg/kg in high water, high oil and high starch matrices while acidic matrices were validated with a LOQ of 0.02 mg/kg.

For animal matrices the analytical methods were comparable, however silica gel SPE was used for clean-up instead. Validated LOQs were 0.001 mg eq/kg for milk, 0.01 mg/kg for liver and kidney, 0.02 mg/kg for meat and 0.1 mg/kg for fat.

The application of multi-residue methods was tested with DFG S19 for both plant and animal matrices. The method was shown suitable with a general LOQ of 0.02 mg/kg for lufenuron.

Stability of residues in stored analytical samples

The Meeting received information on the storage stability of lufenuron in plant and animal matrices stored at -18°C.

In plant matrices with high water, high acid and high oil content parent lufenuron was stable for at least 24 months. High starch matrices were not tested.

In animal matrices (bovine tissues and milk) no significant degradation was observed within 9 months. No storage stability data were provided for poultry matrices and eggs.

Definition of the residue

The fate of lufenuron in plants was investigated after foliar application to tomatoes, cabbage and cotton. In all crop samples investigated unchanged lufenuron was the only major residue present, representing 79–100% TRR. The residue was mainly present as a surface residue. No significant transfer into untreated plant parts was observed.

In confined rotational crop studies the overall uptake of radioactivity was very limited. Only parent lufenuron could be detected in collected plant samples.

The Meeting concluded that lufenuron is the relevant residue in all plant matrices for compliance with MRLs and for dietary intake purposes. Analytical multi-residue methods are capable of measuring lufenuron in all plant matrices.

Livestock animal metabolism studies were conducted on lactating goats (5.4–6.0 ppm) and laying hens (3.4–5.2 ppm).

In both species unchanged parent lufenuron was the only residue identified in major amounts, representing 73–94% of the TRR in all matrices. In goat matrices and milk no other metabolites could be detected. In poultry matrices minor amounts of the cleavage products CGA149772 and CGA238277 were found, representing up to 17% TRR in egg white but at low levels (0.001 mg eq/kg) and 5.3% TRR in kidney (0.028 mg eq/kg). No further metabolites were found in poultry matrices or eggs.

The Meeting concluded that parent lufenuron is the relevant residue in all animal matrices for compliance with MRLs and for dietary intake purposes. Analytical multi-residue methods are capable of measuring lufenuron in all animal matrices.

In all species residue concentrations in fat tissues or egg yolk were at least one order of magnitude higher than in muscle tissues or egg white. The log P_{ow} of lufenuron is 5.12. The Meeting decided that residues of lufenuron are fat soluble.

Definition of the residue for compliance with MRL and for dietary intake for plant and animal commodities: *lufenuron*

The residue is fat-soluble.

Results of supervised residue trials on crops

The Meeting received supervised trial data for applications of lufenuron on various vegetables crops as well as for soya beans, maize, sugarcane, cotton and coffee conducted in Brazil, China and Europe.

Cucumber

Lufenuron is registered in Spain for cucumbers under protected conditions at rates of 2 × 0.1 kg ai/ha with a PHI of 7 days. Supervised field trials from France, Greece and Spain according to this GAP and at rates up to +50% higher were submitted.

In protected cucumbers residues of lufenuron following GAP treatment (±25%) were (n=4): 0.01, 0.02, 0.06, 0.06 mg/kg.

The Meeting concluded that four supervised trials on cucumber approximating GAP are insufficient for an evaluation and decided to explore the proportionality approach using trials at +50% GAP rate. Since some of the trials according to GAP were also conducted at slightly elevated rates, all data are proportionally adjusted to the Spanish GAP rate of 0.1 kg ai/ha:

In protected cucumbers treated with 0.1 kg ai/ha lufenuron residues were (no scaling factor): 0.06 mg/kg.

In protected cucumbers treated with 0.11 kg ai/ha lufenuron residues were (scaling factor 0.91): 0.018 mg/kg (0.91×0.02 mg/kg).

In protected cucumbers treated with 0.12 kg ai/ha lufenuron residues were (scaling factor 0.83): 0.0083 and 0.05 mg/kg (0.83×0.01 mg/kg and 0.83×0.06 mg/kg).

In protected cucumbers treated with 0.15 kg ai/ha lufenuron residues were (scaling factor 0.66): 0.013(3), 0.02(3), 0.026 mg/kg (0.66×0.02 mg/kg(3), 0.66×0.03 mg/kg(3) and 0.66×0.04. mg/kg)

The combined total dataset for lufenuron in protected cucumbers was (n=11): 0.0083, 0.013(3), 0.018, 0.02(3), 0.026, 0.05 and 0.06 mg/kg.

The Meeting estimated a maximum residues level of 0.09 mg/kg and a STMR of 0.02 mg/kg for lufenuron in cucumber.

Melons, except watermelons

Lufenuron is registered in Spain for melons under protected conditions at rates of 3 × 0.1 kg ai/ha with a PHI of 7 days. Supervised field trials from Spain according to this GAP were submitted.

All samples were segmented and in some trials already separated into pulp and peel in the field, which is against the current Codex sampling procedure. However, lufenuron was not metabolized in plant metabolism studies, even after direct injection into tomato fruits. In addition simulated hydrolysis indicated no degradation at pH 7 or lower, which is representive of fruits and vegetables. The Meeting therefore concluded that segmentation of samples in the field did not influence the magnitude of residues. The Meeting also noted that no contamination of melon pulp with peel residues during separation occurred and decided to use the data for its assessment.

Some trials submitted involved a last sampling at 3 DALA which is shorter than the PHI of the Spanish GAP of 7 days. In plant metabolism studies lufenuron was a surface residue not subject to degradation or metabolism. Also, melons near maturity have already finalized their growth and are only subject to ripening. Therefore the Meeting concluded that no different residue populations have to be expected for melons within the last week before harvest when sampled at 3 or 7 DALA and decided to take samples collected after three days also into account for the assessment. This conclusion is supported by several decline studies from 0 to 10 DALA, indicating no constant decrease of the residue concentration but the usual sampling variation within the results.

In protected melons (whole fruits) residues of lufenuron were (n=6): 0.02, 0.03, 0.06, 0.09, 0.13, 0.19 mg/kg.

In the corresponding pulp samples, if measured, residues of lufenuron were (n=4): < 0.02(4) mg/kg.

For melon, except watermelons, the Meeting estimated a maximum residues level of 0.4 mg/kg, based on whole melon fruits, except watermelons and an STMR of 0.02 mg/kg, based on pulp data.

Peppers, sweet

Lufenuron is registered in Spain for sweet peppers under protected conditions at rates of 3 × 0.1 kg ai/ha with a PHI of 7 days. Supervised field trials on sweet peppers from Greece, Italy and Spain according to this GAP were submitted.

In protected sweet peppers residues of lufenuron following GAP treatment (±25%) were (n=6): 0.08, 0.13, 0.13, 0.17, 0.18 and 0.54 mg/kg.

The Meeting estimated a maximum residues level of 0.8 mg/kg and an STMR of 0.15 mg/kg for lufenuron in sweet peppers.

Tomato

Lufenuron is registered in Spain for tomatoes under protected conditions at rates of 3 × 0.1 kg ai/ha with a PHI of 7 days. Supervised field trials on tomatoes from Greece, Spain and Switzerland according to this GAP were submitted.

In protected tomatoes residues of lufenuron following GAP treatment were (n=13): 0.02, 0.04, 0.04, 0.05, 0.06, 0.08(4), 0.09, 0.1, 0.11 and 0.24 mg/kg.

The Meeting estimated a maximum residues level of 0.4 mg/kg and an STMR of 0.08 mg/kg for lufenuron in tomatoes.

Sweet corn

The Meeting received supervised field trail information on sweet corn, however no corresponding GAP was made available to the Meeting and therefore no recommendation was made.

Soya beans

Lufenuron is registered in Brazil for soya beans at maximum rates of 2 × 0.02 kg ai/ha with a PHI of 35 days. Supervised field trials on soya beans from Brazil at exaggerated rates (3.8 times higher) and a higher number of treatments (four instead of two) were submitted.

In soya beans residues of lufenuron after exaggerated treatment were (n=3): < 0.01(3) mg/kg

The Meeting concluded that under consideration of the exaggerated treatment regime involved, the seeds being protected by the pod during applications and the non-systemic properties of the active substance observed in plant metabolism studies, no finite residue following treatment at GAP rate have to be expected. The Meeting estimated a maximum residues level of 0.01* mg/kg and an STMR of 0 mg/kg for lufenuron in soya beans (dry).

Potatoes

Lufenuron is registered in Brazil for potatoes at rates of 4 × 0.04 kg ai/ha with a PHI of 14 days. Supervised field trials from Brazil matching the GAP were submitted.

In potato tubers residues of lufenuron after treatment according to GAP were (n=4): < 0.01(4) mg/kg

Taking into account the non-systemic properties of the active substance, the Meeting concluded that residues in tuber above the LOQ are unlikely to occur and estimated a maximum residues level of 0.01* mg/kg and an STMR of 0.01 mg/kg for lufenuron in potatoes.

Maize

Lufenuron is registered in Brazil for maize at maximum rates of 2 × 0.01 kg ai/ha with a PHI of 35 days. All supervised field trials on maize submitted were sampled at significantly longer DAT intervals than the PHI.

The Meeting concluded that the data submitted for lufenuron in maize is insufficient for a recommendation.

Sugar cane

Lufenuron is registered in Brazil for sugar cane at rates of 2 × 0.02 kg ai/ha with a PHI of 14 days. Supervised field trials from Brazil matching the GAP were submitted.

In sugar cane residues of lufenuron after treatment according to GAP were (n=4): < 0.01 and 0.02(3) mg/kg

The Meeting concluded that the data submitted for lufenuron in sugar cane is insufficient for a recommendation.

Cotton

Lufenuron is registered in China for cotton at rates of 2 × 0.045 kg ai/ha with a PHI of 28 days. Supervised field trials from China according to this GAP were submitted, however the trial description did not included information on the stage of boll opening for cotton plants.

In cotton seeds residues of lufenuron after treatment according to GAP were (n=4): < 0.05(4) mg/kg

The Meeting concluded that the stage of boll opening is a sensitive parameter for residues following foliar application. Without this type of information, a set of four field trials in not considered sufficient for estimating maximum residue levels in cotton seed. Supportive information from plant metabolism studies cannot be taken into account as the active substance was applied before boll opening in these studies.

Coffee

Lufenuron is registered in Brazil for coffee at rates of 2 × 0.04 kg ai/ha with a PHI of 7 days. Supervised field trials from Brazil matching the GAP were submitted.

In coffee beans (dry processed) residues of lufenuron after treatment according to GAP were (n=4): < 0.01(3) and 0.01 mg/kg

The Meeting concluded that the data submitted for lufenuron in coffee is insufficient for a recommendation.

Fate of residues during processing

The Meeting received information on the hydrolysis of radio-labelled lufenuron as well as processing studies using unlabelled material in tomatoes.

In a hydrolysis study using [dichlorophenyl-^{14}C]-lufenuron or [difluorophenyl-^{14}C]-lufenuron, typical processing conditions were simulated (pH 4,5 and 6 with 90°C, 100°C and 120°C for 20, 60 and 20 minutes). No significant degradation of the parent was observed. For pH5 with 100°C for 60min a minor formation of CGA224443 and CGA149772 (up to 6.9% of the applied radioactivity) was observed.

The fate of lufenuron residues has been examined simulating household and commercial processing of tomatoes. Estimated processing factors for the commodities considered at this Meeting are summarized below.

Raw commodity	Processed commodity	Lufenuron		
		Individual processing factors	Mean or best estimate processing factor	STMR-P in mg/kg
Tomato (STMR: 0.08 mg/kg)	Juice, raw	<0.17, 0.17	0.17	0.014
	Puree	0.79, 0.83, 0.86.0.9	0.85	0.068
	Paste	0.83, 1.1	0.97	0.078
	Canned/preserve	<0.17(4)	0.17	0.014
	pomace, wet	7.9, 7.9, 8.6, 9.7	8.3	0.66

Residues in animal commodities

Farm animal feeding studies

The Meeting received feeding studies involving lufenuron on lactating cows and steers.

Three groups of lactating cows were dosed daily at levels of 0.82, 4.3 and 8.6 ppm in the diet for 28 consecutive days. Milk was collected throughout the whole study and tissues were collected on day 29 within 24 hrs after the last dose.

In milk residues of lufenuron were 0.16 mg/kg, 0.99 mg/kg and 2.5 mg/kg for the low, middle and high dose group, respectively. Skim milk and cream were analysed individually, showing residues of 0.006, 0.038 and 0.054 mg/kg for skim milk and 3.1, 24 and 32 mg/kg for cream.

In tissues mean concentrations of lufenuron with increasing dose rate were 0.03, 0.17 and 0.43 mg/kg in muscle, 0.06, 0.37 and 0.77 mg/kg in liver, 0.03, 0.22 and 0.36 mg/kg in kidney and 0.73, 4.5 and 8.0 mg/kg in fat.

In the steer study three groups of Angus steers were dosed 0.02 or 1 ppm in the diet for 28 consecutive days. Animals were sacrificed 24h after the last administrations (day 28).

Mean lufenuron residues in the low and high-dose animals were < 0.01 and < 0.01 mg/kg in muscle, < 0.01 and 0.023 mg/kg in liver, < 0.01 and 0.026 mg/kg in kidney and 0.036 and 0.23 mg/kg in fat, respectively.

Estimated maximum and mean dietary burdens of livestock and animal commodities maximum residue levels

Dietary burden calculations for beef cattle, dairy cattle, broilers and laying poultry are presented in Annex 6. The calculations were made according to the livestock diets from US-Canada, EU, Australia and Japan in the OECD Table (Annex 6 of the 2006 JMPR Report).

	Livestock dietary burden, lufenuron, ppm of dry matter diet							
	US-Canada		EU		Australia		Japan	
	max.	mean	max.	mean	max.	mean	max.	mean
Beef cattle	0.02	0.02	0.34	0.34	0.02	0.02	none	none
Dairy cattle	0.02	0.02	0.34 [a]	0.34 [b]	0.02	0.02	none	none
Poultry - broiler	none	none	0.01	0.01	none	none	none	none
Poultry - layer	none	none	0.01 [c]	0.01 [d]	none	none	none	none

[a] Highest maximum beef or dairy cattle burden suitable for MRL estimates for mammalian meat and milk

[b] Highest mean beef or dairy cattle burden suitable for STMR estimates for mammalian meat and milk
[c] Highest maximum broiler or laying hen burden suitable for MRL estimates for poultry products and eggs
[d] Highest mean broiler or laying hen burden suitable for STMR estimates for poultry products and eggs
none - no relevant feed items

Animal commodities maximum residue levels

For beef and dairy cattle a maximum and mean dietary burden of 0.34 ppm was estimated. Two feeding studies on lactating cows and steers were submitted. Since no accumulation of residues in steers compared to dairy cows was observed, the Meeting decided to base its recommendations for mammalian products on the lactating cow feeding study, generally showing higher residues at identical intake levels.

Lufenuron feeding study	Feed level	Total residue				
	(ppm)	(mg/kg) in milk	(mg/kg) in muscle	(mg/kg) in kidney	(mg/kg) in liver	(mg/kg) in fat
Maximum residue level: dairy cattle						
Feeding study (HR for each dose group, except for milk)	0.82	0.16 (cream: 3.1)	0.04	0.04	0.07	1.2
Dietary burden and residue estimate	0.34	0.066 (cream: 1.2)	0.017	0.017	0.029	0.5
STMR dairy cattle						
Feeding study (Mean for each dose group)	0.82	0.16 (cream: 3.1)	0.03	0.03	0.06	0.73
Dietary burden and residue estimate	0.34	0.066 (cream: 1.2)	0.012	0.012	0.025	0.3

The Meeting estimated STMR values of 0.012 mg/kg for muscle, 0.025 mg/kg for edible offal (based on liver) and 0.3 for fat. Corresponding maximum residue levels were estimated at 0.04 mg/kg for edible offal, mammalian (based on liver) and 0.7 mg/kg for meat (based on the fat) and mammalian fat.

For milk, an STMR and a MRL of 0.066 mg/kg and 0.1 mg/kg were estimated, respectively. Based on the data for cream, the Meeting also estimated an STMR and MRL of 1.2 mg/kg and 2 mg/kg for lufenuron in milk fat, respectively.

For poultry a maximum and mean dietary burden of 0.01 ppm was estimated. No farm animal feeding studies were provided for poultry. Therefore the Meeting decided to make its recommendations based on the ^{14}C-difluorophenyl-labelled poultry metabolism study which showed higher residues than the corresponding ^{14}C-dichlorophenyl-labelled experiment.

Lufenuron feeding study	Feed level	Total residue				
	(ppm)	(mg/kg) in eggs	(mg/kg) in muscle	(mg/kg) in kidney	(mg/kg) in liver	(mg/kg) in fat
Mean and maximum residue level: poultry						
^{14}C-difluorophenyl-labelled metabolism study	3.4	2.5 [a]	0.196	0.588	1.34	9.15
Dietary burden and residue estimate	0.01	0.01	0.0006	0.0017	0.004	0.027

[a] In the metabolism study egg white and egg yolk were analysed separately. To estimate residues in whole eggs, an average ratio of 65% egg white and 35% egg yolk was taken into account: 0.65 × 0.003 mg eq/kg in egg white + 0.35 × 7.18 mg eq/kg in egg yolk=2.5 mg eq/kg in whole eggs

The Meeting estimated STMR values of 0.01 mg/kg for eggs, 0.0006 mg/kg for poultry meat, 0.004 mg/kg for poultry edible offal of (based on liver) and 0.027 mg/kg for poultry fat. Corresponding maximum residue levels for lufenuron were estimated at 0.02 mg/kg for eggs, poultry meat and edible offal of and at 0.04 mg/kg for poultry fat.

RECOMMENDATIONS

On the basis of the data from supervised trials, the Meeting concluded that the residue levels listed in Annex 1 were suitable for estimating maximum residue limits and for IEDI assessment.

Definition of the residue for compliance with MRL and for dietary intake purposes for plant and animal commodities: *Lufenuron*

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
VC 0424	Cucumbers	0.09		0.02	
MO 0105	Edible offal (Mammalian)	0.04		0.025	
PE 0112	Eggs	0.02		0.01	
MF 0100	Mammalian fats	0.7		0.3	
MM 0095	Meat (from mammals other than marine mammals)	0.7 (F)		Muscle: 0.012 Fat: 0.3	
VC 0046	Melon, except watermelons	0.4		0.02 (pulp)	
ML 0106	Milks	0.1		0.066	
FM 0183	Milk fats	2		1.2	
VO 0445	Pepper, sweet	0.8		0.15	
VR 0589	Potato	0.01*		0.01	
PF 0111	Poultry fats	0.04		0.027	
PM 0110	Poultry meat	0.02		0.0006	
PO 0111	Poultry, edible offal of	0.02		0.004	
VD 0541	Soya beans (dry)	0.01*		0	
VO 0448	Tomato	0.4		0.08	
JF 0048	Tomato juice			0.014	
MW 0448	Tomato puree			0.068	
VW 0448	Tomato paste			0.078	
	Tomato preserve			0.014	
	Tomato wet pomace			0.66	

FURTHER WORK OR INFORMATION

- Poultry feeding study

DIETARY RISK ASSESSMENT

Long-term intake

The evaluation of lufenuron has resulted in recommendations for MRLs and STMRs for raw and processed commodities. The International Estimated Daily Intakes for the 17 GEMS/Food cluster diets, based on this years estimated STMRs, were in the range 0–4% of the maximum ADI of 0.02 mg/kg bw. The results are shown in Annex 3 to the 2015 Report.

The Meeting concluded that the long-term intake of residues of lufenuron from uses that have been considered by the JMPR is unlikely to present a public health concern.

Short-term intake

For short-term intake, an ARfD was considered unnecessary. The Meeting concluded that the short-term intake of lufenuron residues from uses considered by the Meeting is unlikely to present a public health concern.

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