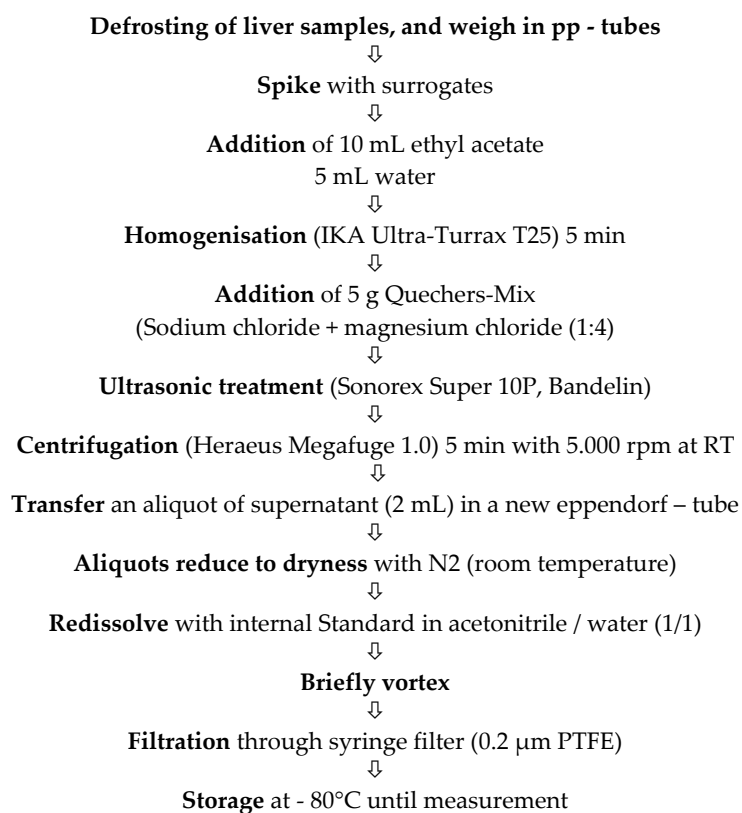


**Table S1.** TP – analysis: Workflow of sample preparation.



**Table S2.** TP – analysis: Configuration of LC-MS/MS

<b>Liquid chromatography</b>		<b>Agilent 1290 Infinity II</b>	
Autosampler temperature		10 °C	
Injection volume		5 µL	
Syringe rinse		100 µL	
Analytical column		Agilent Zorbax Eclipse C18 (2.6 µm, 50 mm, 1.8 mm i.d.)	
Column temperature		40 °C	
Mobile phase A		Water + 1mmol NH <sub>4</sub> F	
Mobile phase B		Methanol / ACN (65/35)	
Gradient program	Time (min)	A (%)	B (%)
	0.0	98	2
	0.5	98	2
	3.0	2	98
	4.0	2	98
	4.1	98	2
	4.0	98	2
Flow rate		500 µL/min	
<b>Mass spectrometer</b>		<b>QTRAP 6500+ (SCIEX)</b>	
Mode		positive ESI	
Ion spray potential		5500 V	
Source temperature		400 °C	
Scan type		Multiple Reaction Monitoring	
Dwell time		50 ms	
Software		Analyst 1.7.1	
Quantification		relative peak area	

**Table S3.** TP analysis - LC-MS/MS - MRM-conditions of the analyte, surrogate and internal standard (precursor (Q1) and product ions (Q3) in m/z and declustering potential (DP), entrance potential (EP), collision energy (CE) and cell exit potential (CXP) in V).

Q1 Mass	Q3 Mass	Analyte	DP	EP	CE	CXP
Positive mode						
221	179	Atrazine-d5 (IS)	21	10	25	2.5
381	364	Triptolide-d3 (SURR)	26	10	17	18
361	115	Triptolide 1	136	10	101	14
378	361	Triptolide 2	26	10	17	18

**Table S4.** TP – analysis: Confirmation by Enhanced Product Ion spectra.

<b>EPI (enhanced product ion spectra)</b>			
Mass range	DP	EP	CE
(m/z)		(V)	
50 – 450 m/z	-50	-10	-30 ( ±15)

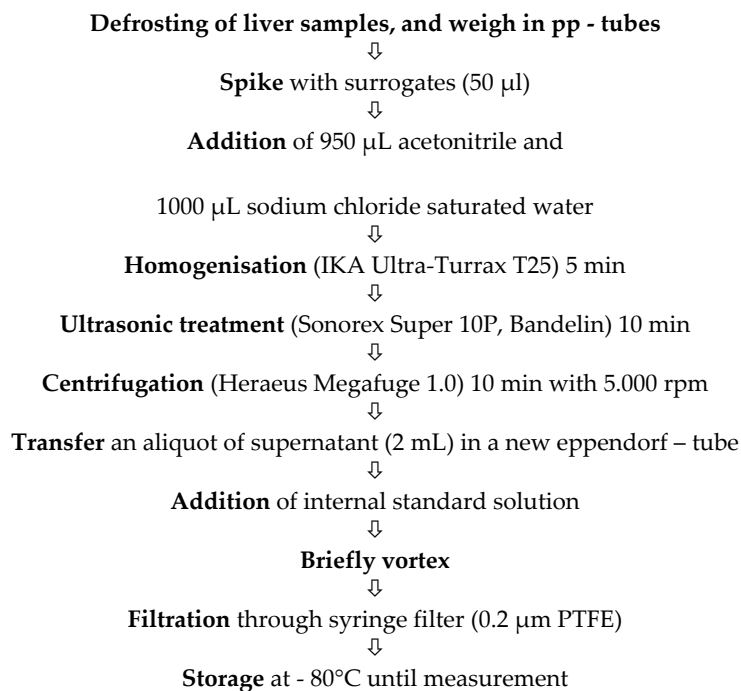
**Table S5.** TP – analysis: Validation - Recovery (REC) and relative standard deviation (RSD) with pig liver (n = 5); in control samples (n = 2) all not detected; n. d. (not detected) = < Reporting Limit (RL).

Analyte	10 ng/g		100 ng/g	
	REC	RSD	REC	RSD
Triptolide	78	7	87	6
Triptolide-d3	75	5	80	8

**Table S6.** TP – analysis: Validation - Recovery (REC) of the surrogate triptolide-d3, over all trial liver and testicle samples + relative standard deviation (RSD) with 5 ng/sample wet weight.

Triptolide-d3 in trial samples	n	REC	RSD %
Liver	60	81	11
Testicle	45	70	24

**Table S7.** VCD – analysis: Workflow of sample preparation.



**Table S8.** VCD – analysis: Configuration of GC-MS.

Gas chromatography	Trace GC Ultra (Thermo Scientific)
Autosampler temperature	10 °C
Injector type	Split/Splitless
Injector temperature	210 °C
Injection technique	Splitless (0-3 min) wSurge (200 kPa, 1.5 min)
Injection volume	1 µL
Analytical column	ZB-624-plus (1.4 µm, 30 m, 0.25 mm i.d.)
Carrier gas	He 5.0 / 1.2 ml/min (const flow)
Column temperature	70°C (2') > 10°C/min > 280°C (3')
Mass spectrometer	TSQ Quantum GC XLS(Thermo Scientific)
Mode	EI (electron impact ionisation)
Source temperature	240°C
Transfer line temperature	275°C
Scan type	Full scan (30-650 m/z)
Software	Xcalibur 4.3.73.11
Quantification	Relative peak area

**Table S9.** VCD – analysis: GC-MS – retention time and detected masses of analytes used for identification and quantification of the analyte, surrogate and internal standard on quadrupole 1.

Analyte	Retention time (min)	Q1 Mass (m/z)
4-Vinylcyclohexene dioxide	13.5 /13.6	Σ 67, 79, 81
Piperitone (SURR)	13.9	Σ 82, 95, 110
cis-Cyclodecene (SURR)	11.3	Σ 67, 81, 54
2-Cyclohexen-1-one (IS)	8.2	Σ 68, 96

**Table S10.** VCD – analysis: Validation – Recovery (REC) and relative standard deviation (RSD) of 4-vinylcyclohexene dioxide and the surrogates cis-cyclodecene and piperitone in calf liver (control samples (n = 2) all not detected) for method development. The recovery of the surrogates, over all trial liver samples + relative standard deviation (RSD) were done with 1000 ng/g wet weight.

Analyte	n	1000 ng/g	
		REC	RSD
		%	
4-Vinylcyclohexene dioxide	5	110	15
cis-Cyclodecene in recovery	5	106	7
Piperitone in recovery samples	5	141	8
cis-Cyclodecene in trial samples	60	84	15
Piperitone in trial samples	60	113	21