

Fachtagung

Fortschritte in der Suspected- und Non-target-Analytik

LfU Augsburg 27. - 28. März 2014

Triple TOF 5600 von AB Sciex und die Software MasterView

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T. Bader, R. Harsch und R. Winzenbacher

Zweckverband Landeswasserversorgung, Langenau



Zweckverband
Landeswasserversorgung



Handeln | Kommunizieren

RISK Identifizieren | Bewerten
IDENT

gefördert vom:

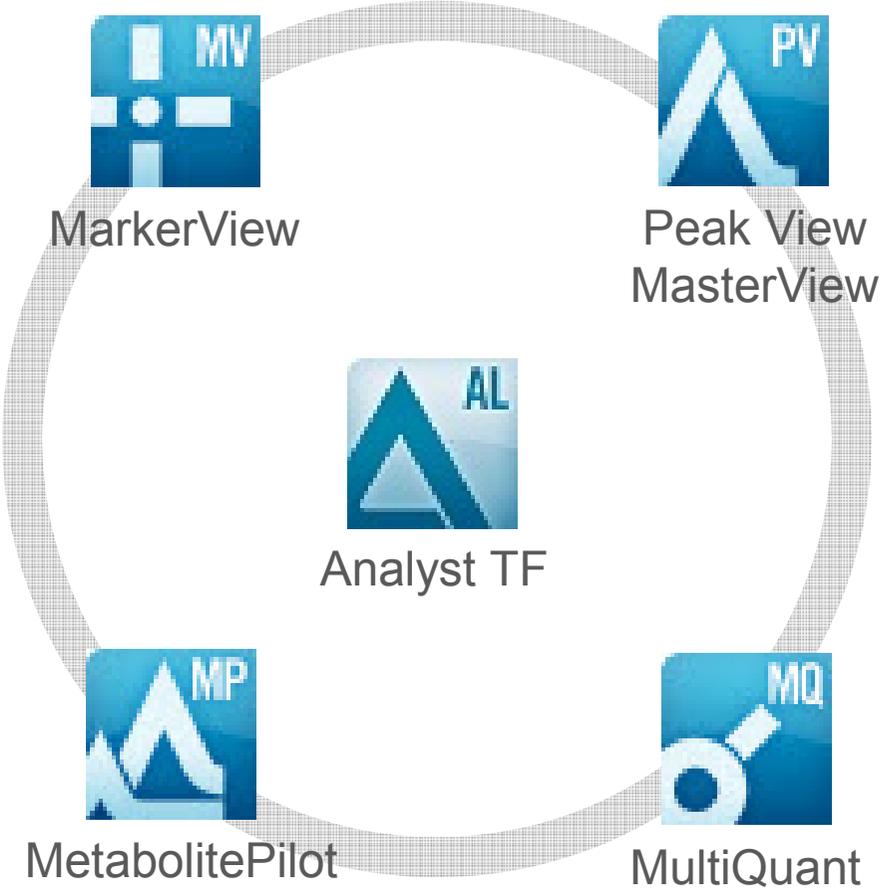


Bundesministerium
für Bildung
und Forschung

Komplettpaket von AB Sciex

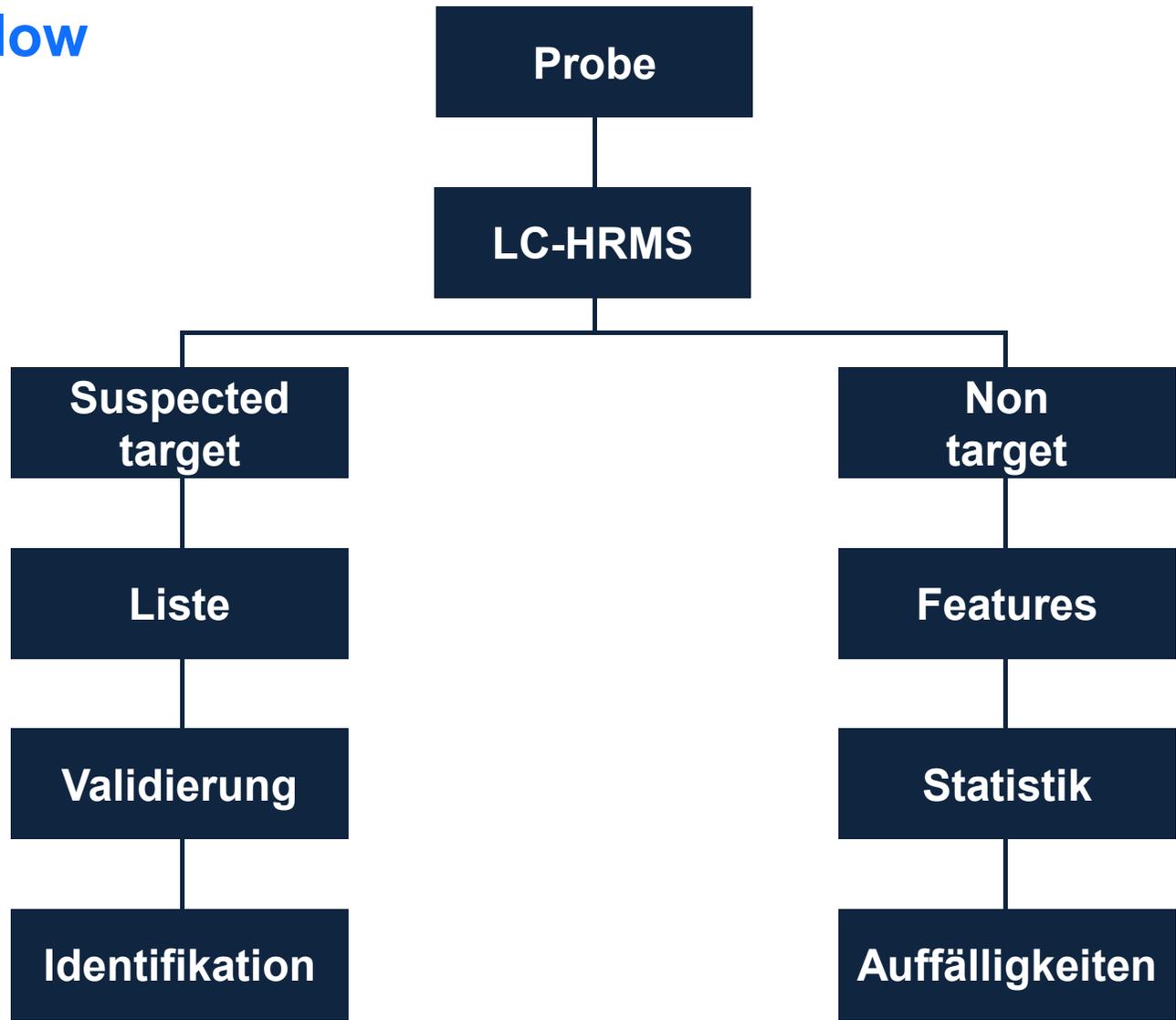


AB Sciex Triple TOF 5600

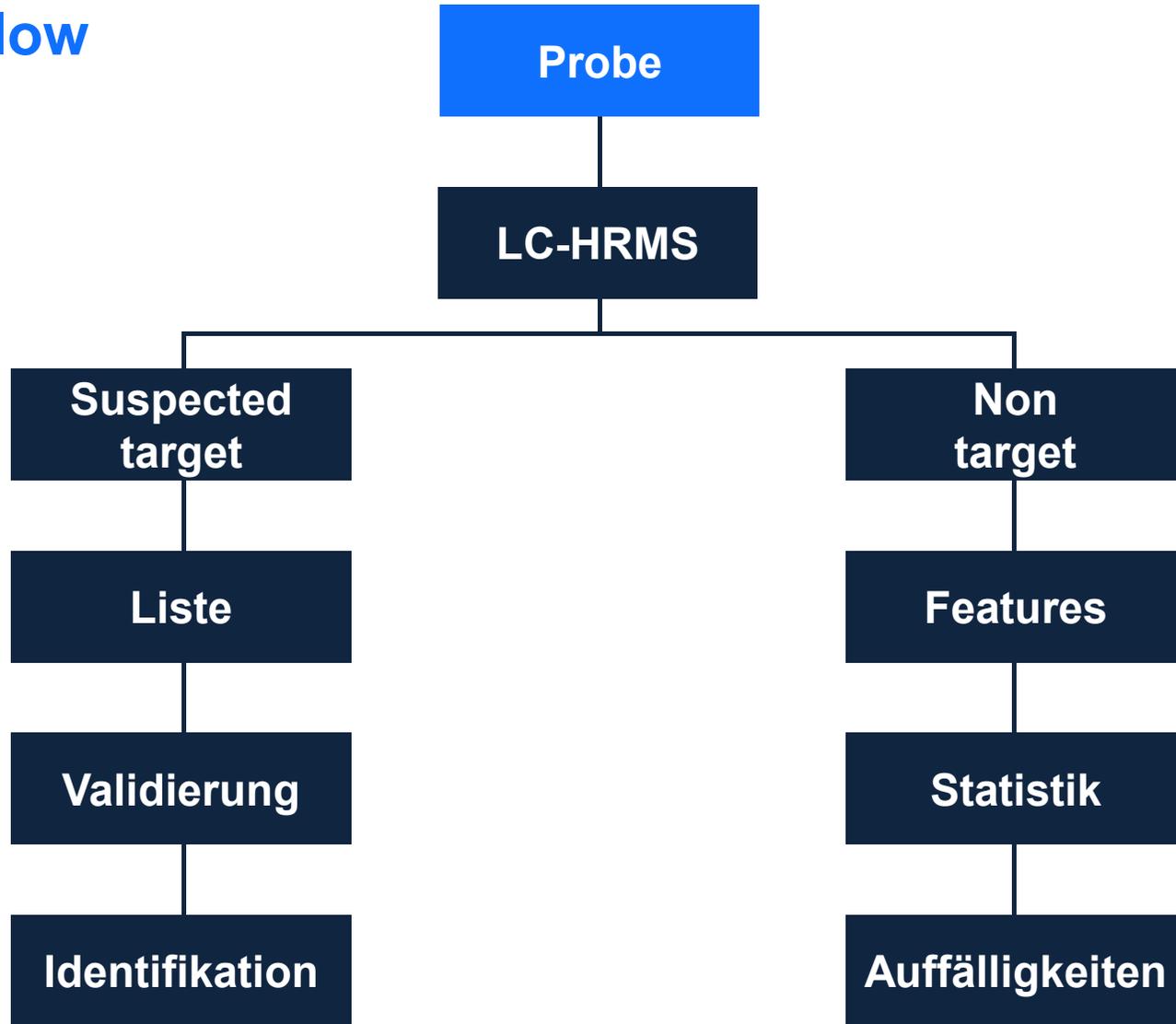


AB Sciex Softwaretools

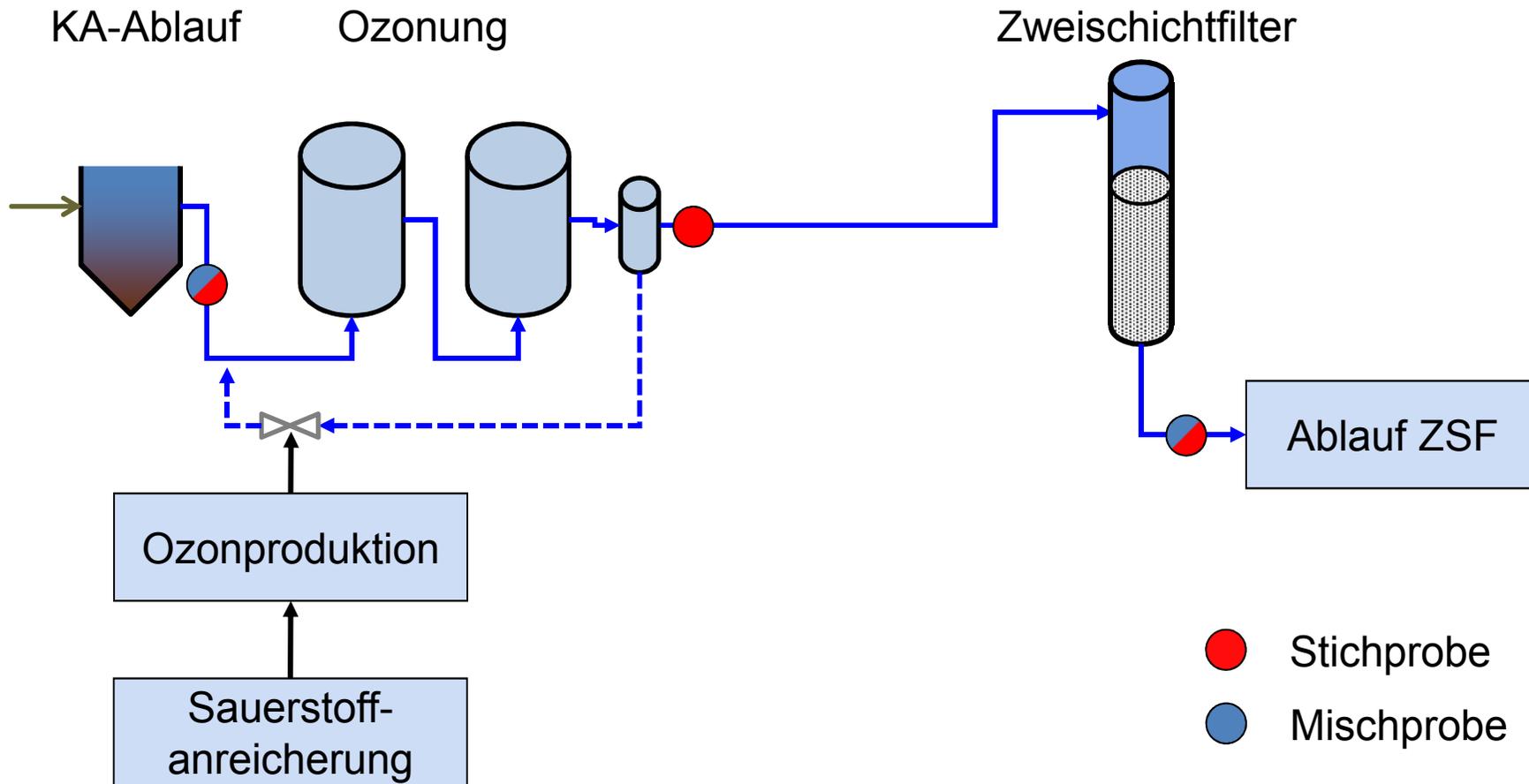
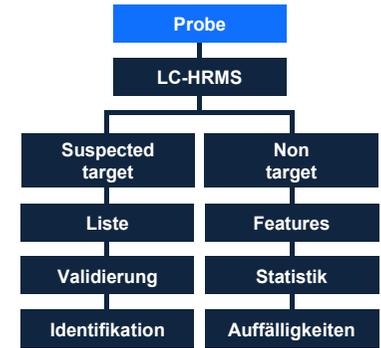
Workflow



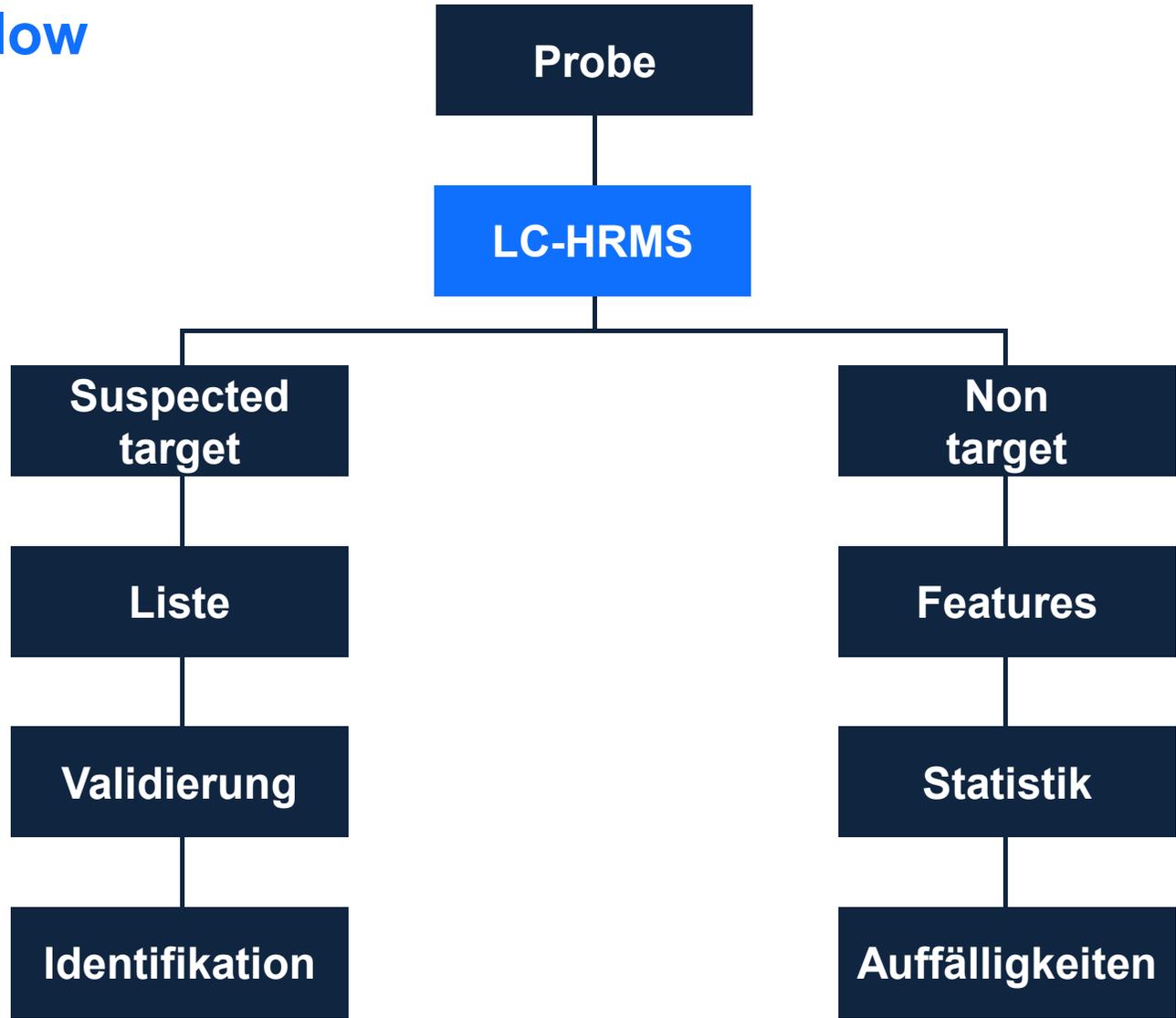
Workflow



Aufbau Pilotanlage

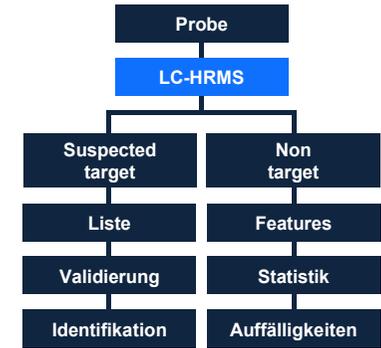


Workflow



Methodenparameter LC

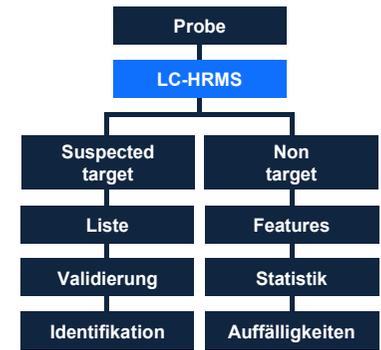
Shimadazu HPLC LC20/30-Serie



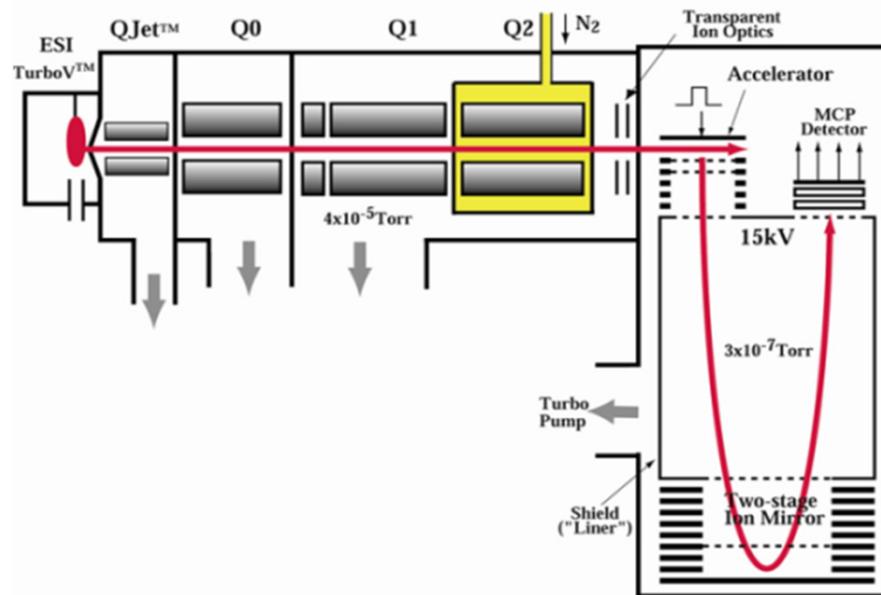
- Trennbedingungen
 - Vorsäule: Phenomenex SecuryGuard AQ C18
4.0 x 2.0 mm i.D.
 - Säule: Agilent Zorbax Eclipse Plus C18, 3.5 µm,
2.4 x 150 mm
 - Gradient: Eluent A: Ultra LC-MS-Wasser + 0.1 V% HCOOH
Eluent B: Acetonitril + 0.1 V% HCOOH
 - Flussrate: 0,3 mL/min
 - Temperatur: 40 °C
 - Injektionsvolumen: 100 µL (Direktinjektion)

Methodenparameter MS

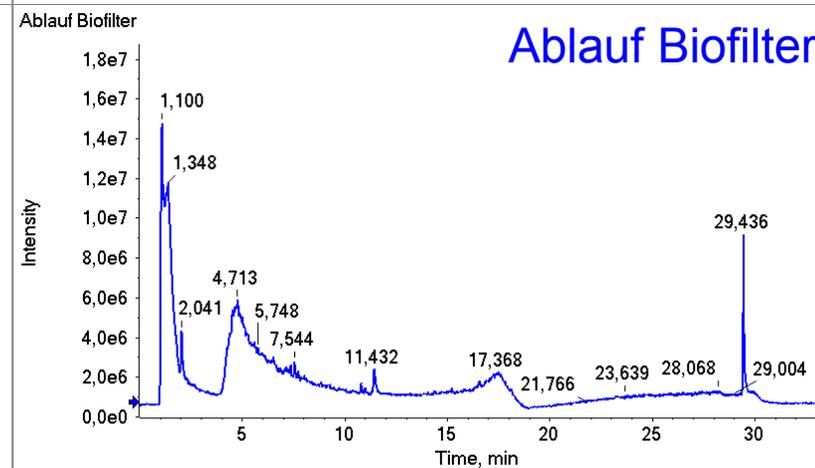
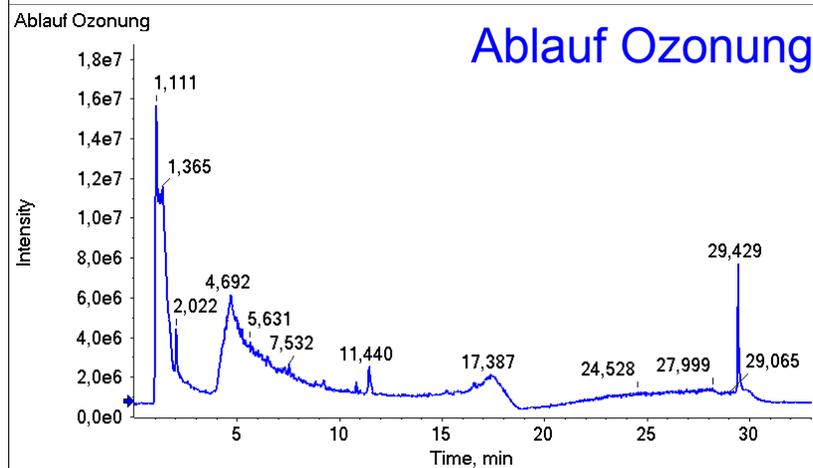
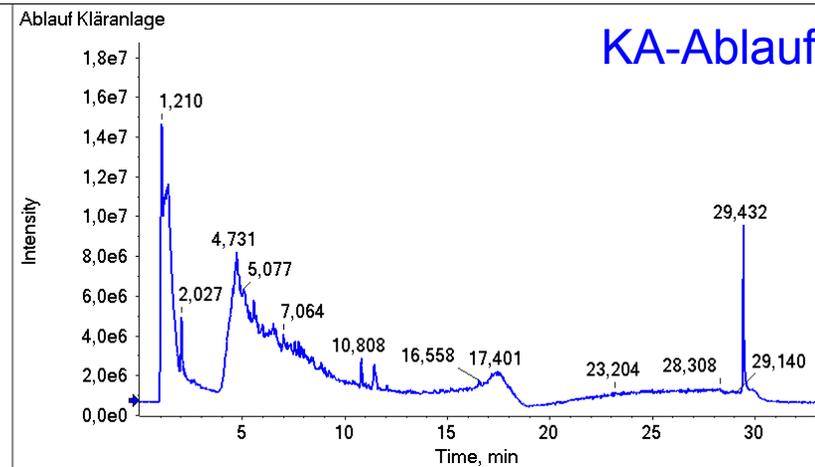
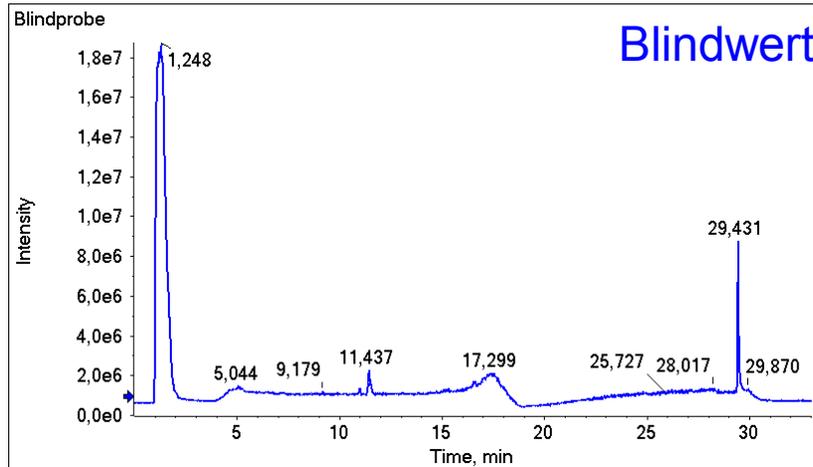
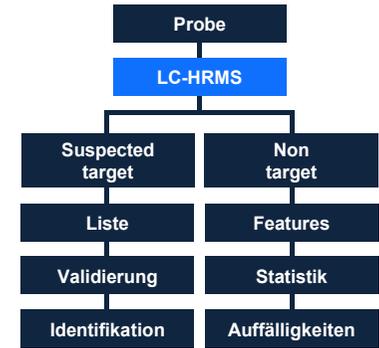
AB Sciex Triple TOF 5600



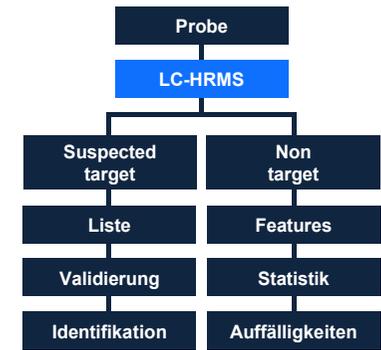
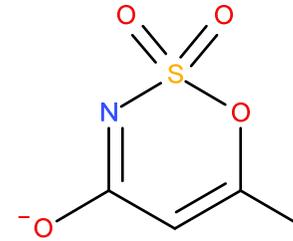
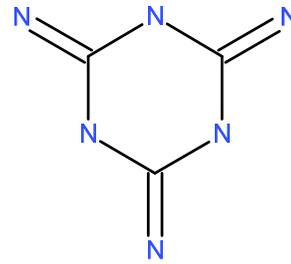
- Elektrospray-Ionisation
 - positiver und negativer Modus
 - Selber Gradient und identisches Ionisationshilfsmittel für beide Modi



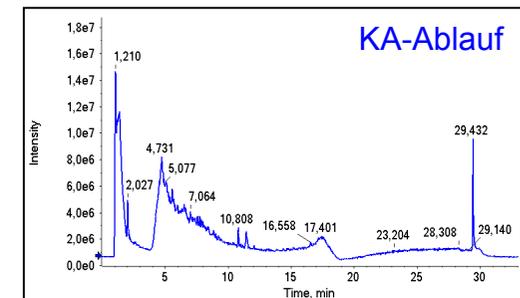
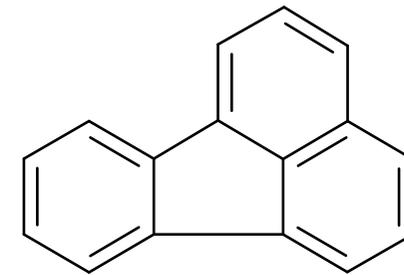
TICs (Rohdaten)



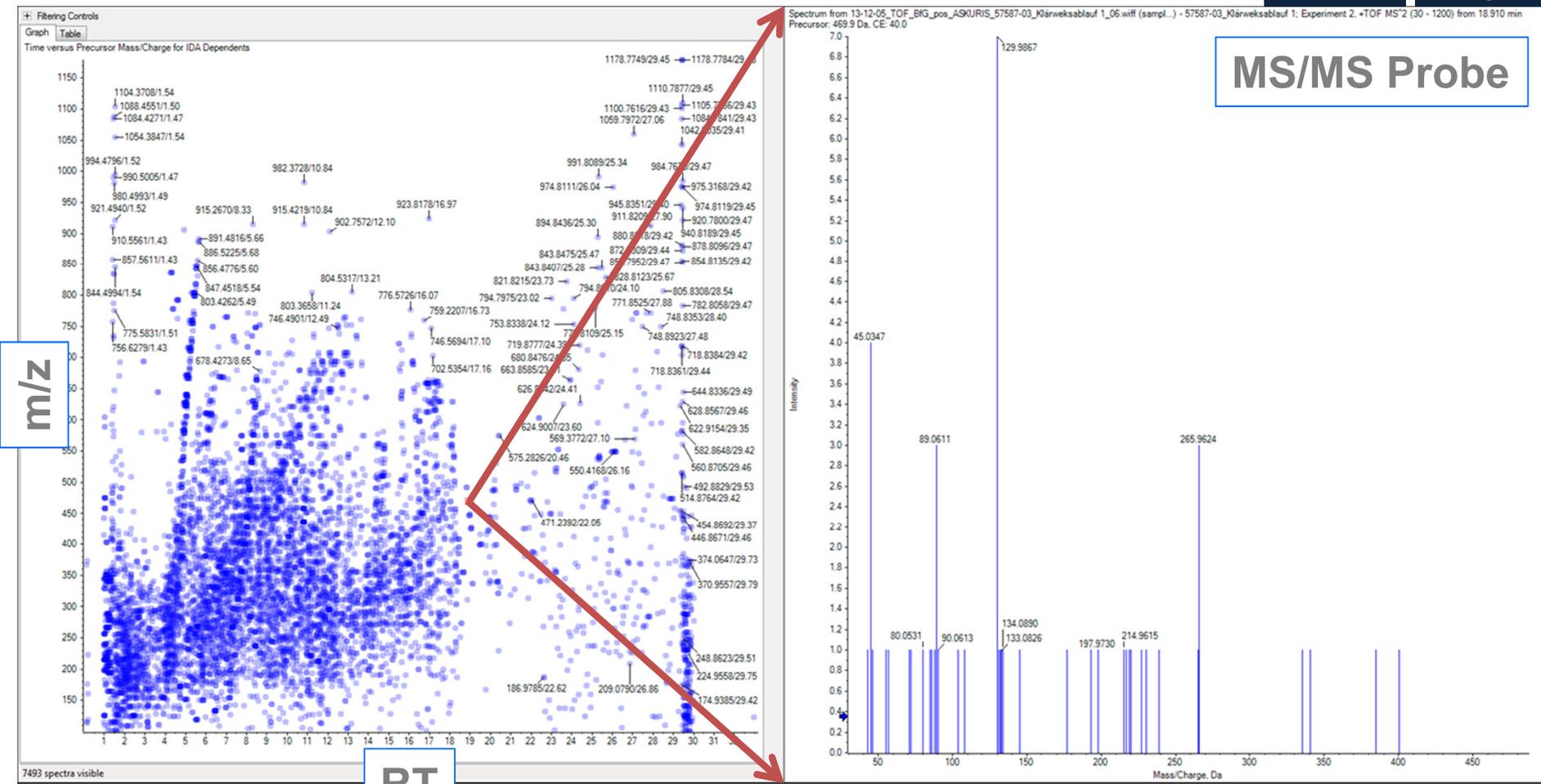
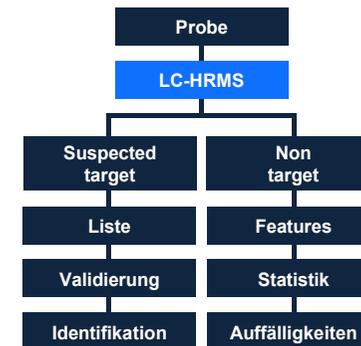
Grenzen der Methode



- RPLC Sehr polare, ionische Verbindungen
- ESI Keine Ionisierung mittels ESI (Kohlenwasserstoffe)
- MS Außerhalb von m/z-Bereich
Unterhalb LOD
- IDA Anzahl ist begrenzt
→ relativ viele Nachmessungen



Darstellung ALLER gemessenen MS/MS mit IDA-Explorer



m/z

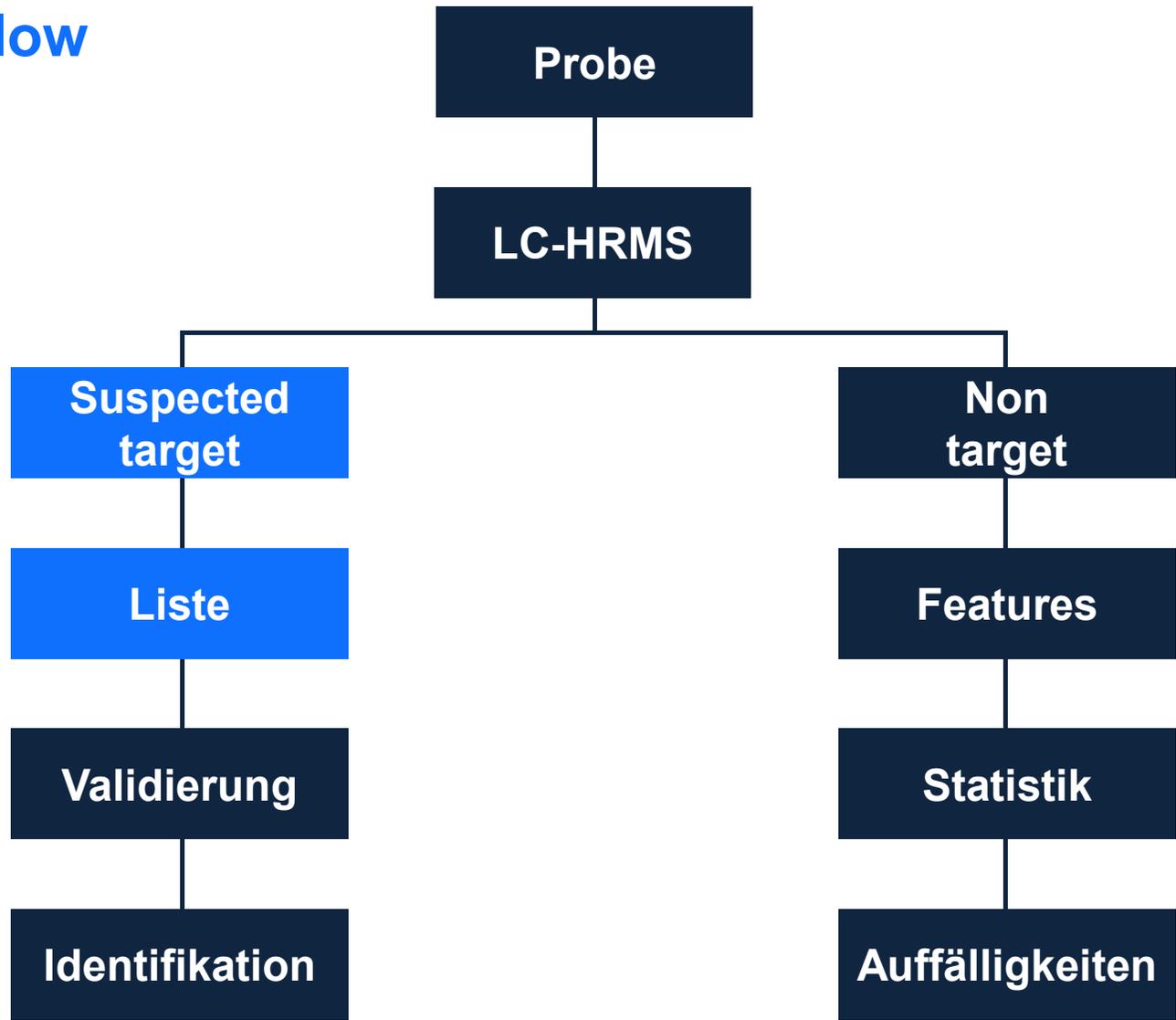
RT



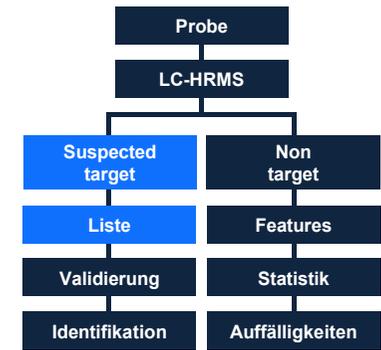
Zweckverband Landeswasserversorgung



Workflow



Suspected Target Screening

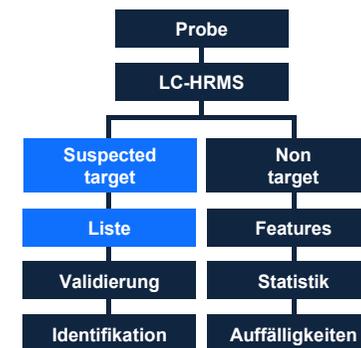


allgemeine Liste (z.B. aus Literatur, Substanzklassen, ...)

SuMS-List		Zweckverband Landeswasserversorgung		LW																																																																																																						
Substanzname	CAS-Nr.	Formel	SMILES	Substanzklasse	Substanz	Referenz	HS-Nr.	HS-Nr. 2	HS-Nr. 3	HS-Nr. 4	HS-Nr. 5	HS-Nr. 6	HS-Nr. 7	HS-Nr. 8	HS-Nr. 9	HS-Nr. 10	HS-Nr. 11	HS-Nr. 12	HS-Nr. 13	HS-Nr. 14	HS-Nr. 15	HS-Nr. 16	HS-Nr. 17	HS-Nr. 18	HS-Nr. 19	HS-Nr. 20	HS-Nr. 21	HS-Nr. 22	HS-Nr. 23	HS-Nr. 24	HS-Nr. 25	HS-Nr. 26	HS-Nr. 27	HS-Nr. 28	HS-Nr. 29	HS-Nr. 30	HS-Nr. 31	HS-Nr. 32	HS-Nr. 33	HS-Nr. 34	HS-Nr. 35	HS-Nr. 36	HS-Nr. 37	HS-Nr. 38	HS-Nr. 39	HS-Nr. 40	HS-Nr. 41	HS-Nr. 42	HS-Nr. 43	HS-Nr. 44	HS-Nr. 45	HS-Nr. 46	HS-Nr. 47	HS-Nr. 48	HS-Nr. 49	HS-Nr. 50	HS-Nr. 51	HS-Nr. 52	HS-Nr. 53	HS-Nr. 54	HS-Nr. 55	HS-Nr. 56	HS-Nr. 57	HS-Nr. 58	HS-Nr. 59	HS-Nr. 60	HS-Nr. 61	HS-Nr. 62	HS-Nr. 63	HS-Nr. 64	HS-Nr. 65	HS-Nr. 66	HS-Nr. 67	HS-Nr. 68	HS-Nr. 69	HS-Nr. 70	HS-Nr. 71	HS-Nr. 72	HS-Nr. 73	HS-Nr. 74	HS-Nr. 75	HS-Nr. 76	HS-Nr. 77	HS-Nr. 78	HS-Nr. 79	HS-Nr. 80	HS-Nr. 81	HS-Nr. 82	HS-Nr. 83	HS-Nr. 84	HS-Nr. 85	HS-Nr. 86	HS-Nr. 87	HS-Nr. 88	HS-Nr. 89	HS-Nr. 90	HS-Nr. 91	HS-Nr. 92	HS-Nr. 93	HS-Nr. 94	HS-Nr. 95	HS-Nr. 96	HS-Nr. 97	HS-Nr. 98	HS-Nr. 99	HS-Nr. 100
ca. 3200 Substanzen																																																																																																										



Liste 20 Substanzen



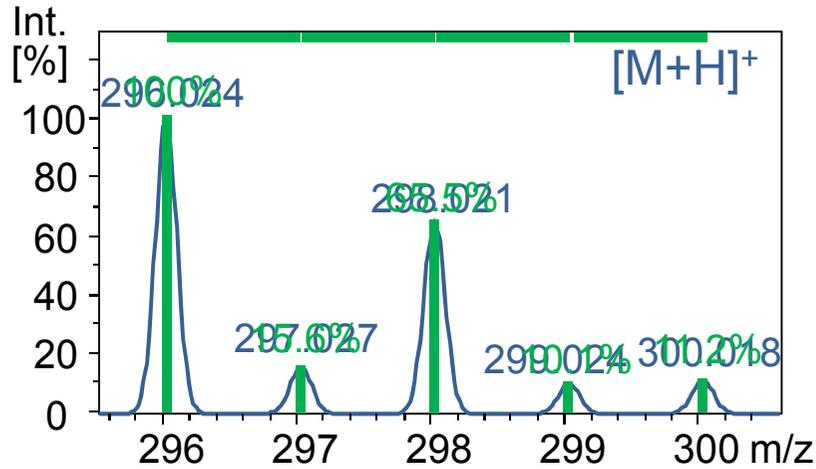
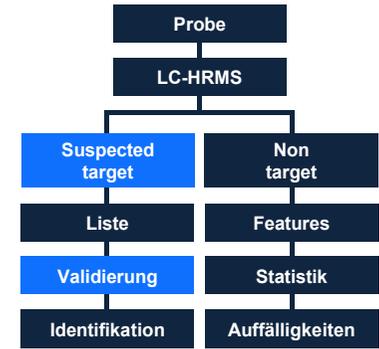
vorgegebene Liste (z.B. bestimmte Substanzen je nach Fragestellung)

	Stoffname	Summenformel	Neutrale Masse	ESI	RT	Fragmente aus DAIOS														
1	Acetylaminoantipyrin (AAA)	C13H15N3O2	245,11643	pos	5,06	104,050	94,064	83,061	43,020											
2	Aminoantipyrin (AA)	C11H13N3O	203,10586	pos	4,67															
3	Benzotriazol	C6H5N3	119,04835	pos	5,50	39,200	65,039	92,050												
4	Bezafibrat	C19H20Cl1N1O4	361,10809	pos	10,39	274,060	154,090													
5	Carbamazepin	C15H12N2O	236,09496	pos	8,44	220,076	194,096	179,073	165,070	44,014	91,055	116,049								
6	Diclofenac	C14H11Cl2N1O2	295,01668	pos	12,03	214,0	249,8	236,0	179,0	178,0	222,0	208,0								
7	4-Hydroxydiclofenac	C14H11Cl2N1O3	311,01160	pos	9,86															
8	Formylaminoantipyrin (FAA)	C12H13N3O2	231,10078	pos	5,10	56,051	77,038	83,061	94,066	104,049										
9	Gabapentin	C9H17NO2	171,12593	pos	4,70	154,0	137,0	67,0	91,0	95,0	119,0									
10	Irbesartan	C25H28N6O	428,23246	pos	8,80	207,0	195,0	180,0												
11	Metoprolol	C15H25N1O3	267,18344	pos	5,50	77,0	116,0	103,0	159,0	121,0	191,0	72,0	74,0	133,0	166,0					
12	Oxazepam	C15H11Cl1N2O2	286,05091	pos	8,80	241,0	269,0	104,0	163,0	77,0	231,0	128,0	214,0							
13	Phenazon	C11H12N2O	188,09496	pos	5,75	56,051	58,066	77,040	104,050	147,091										
14	Phenylethylmalonamid (PEMA)	C11H14N2O2	206,10553	pos	5,25															
15	Primidon	C12H14N2O2	218,10553	pos	5,80	162,0	91,0	115,0	119,0	117,0	65,0	106,0	134,0							
16	Sotalol	C12H20N2O3S1	272,11946	pos	4,47	213,0	255,0	133,0	134,0	176,0	106,0									
17	Sucralose	C12H19Cl3O8	396,01455	neg	5,30	358,8														
18	Toluolsulfonamid (TSA)	C7H9N1O2S	171,03540	neg																
19	Tolyltriazol	C7H7N3	133,06400	pos	6,46	77,0	79,0													
20	Valsartansäure	C14H10N4O2	266,08038	pos	7,29	206,0	178,0	151,0												

Suspected target

Diclofenac

Summenformel: $C_{14}H_{11}Cl_2NO_2$



Exakte Masse: 296.024 → EIC

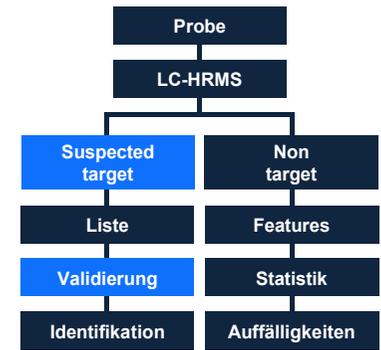
Isotopenmuster

- Intensität
- Abstand

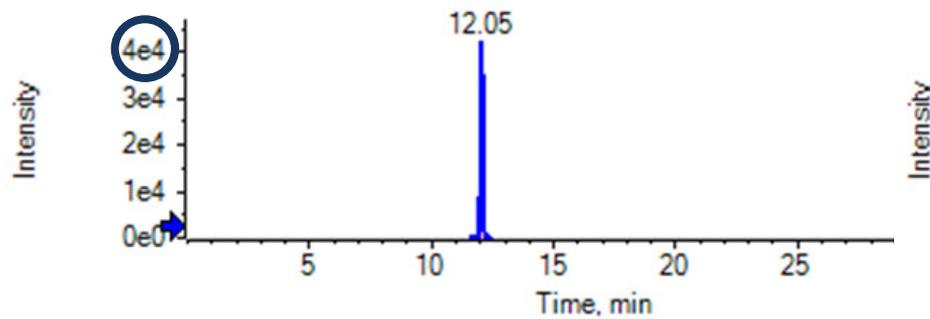


Mass Error Mass Error (ppm)	Retention Time % Error	Isotope Isotope Ratio % Difference
< 25.0	< 5.0	< 10.0
< 50.0	< 15.0	< 20.0
>= 50.0	>= 15.0	>= 20.0

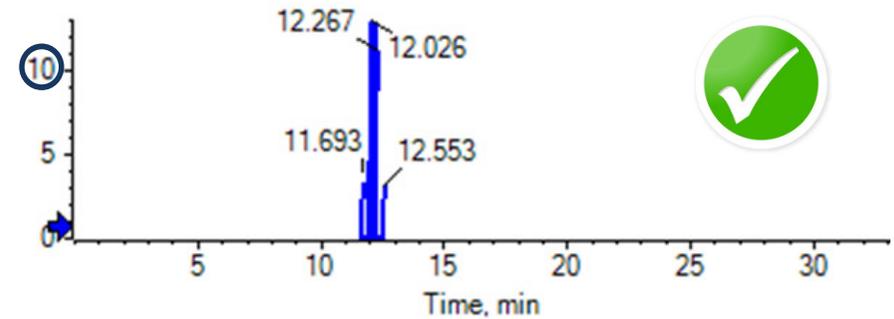
Betrachtung von XICs in verschiedenen Messungen



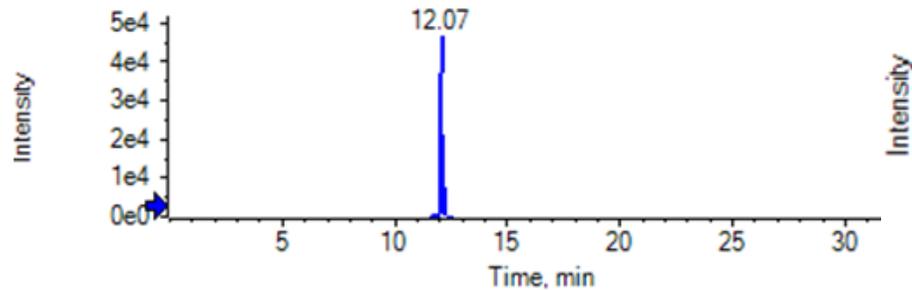
Probe



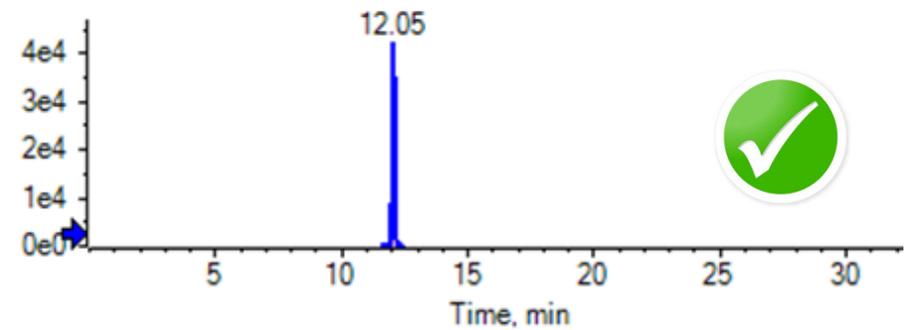
0-Injektion



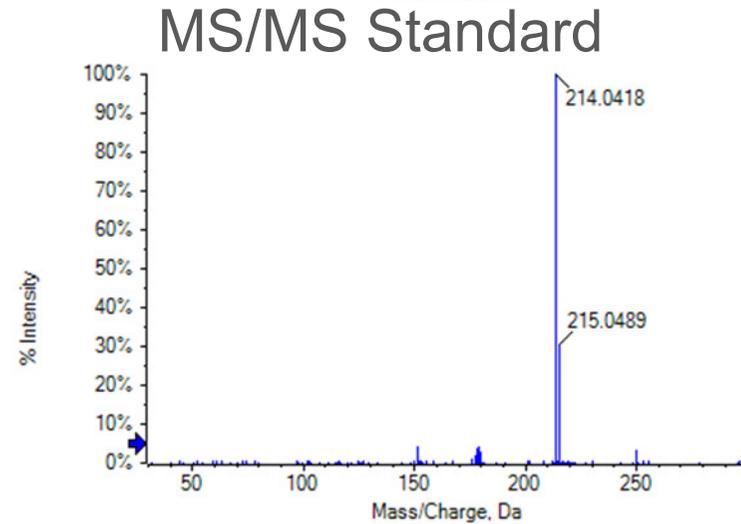
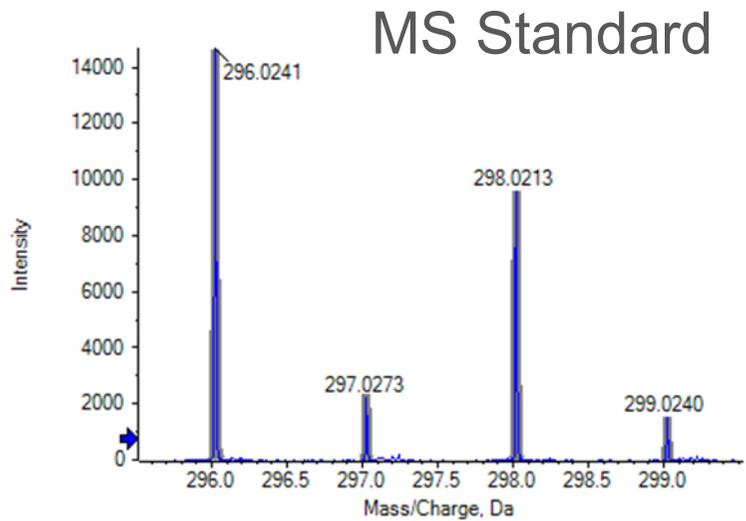
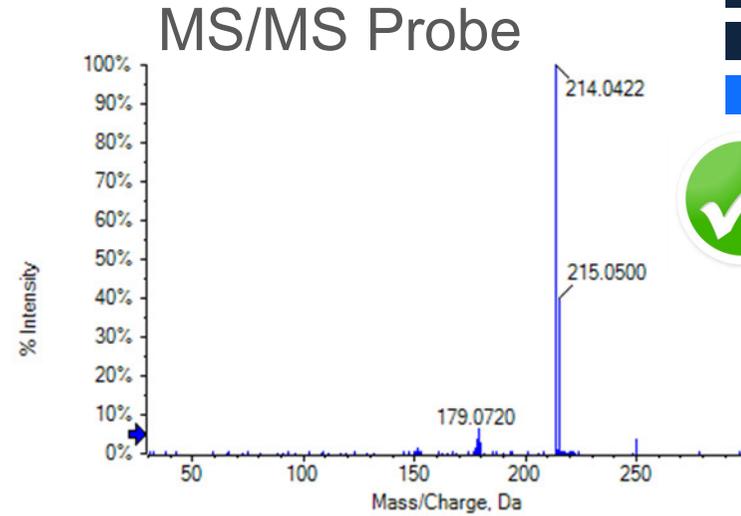
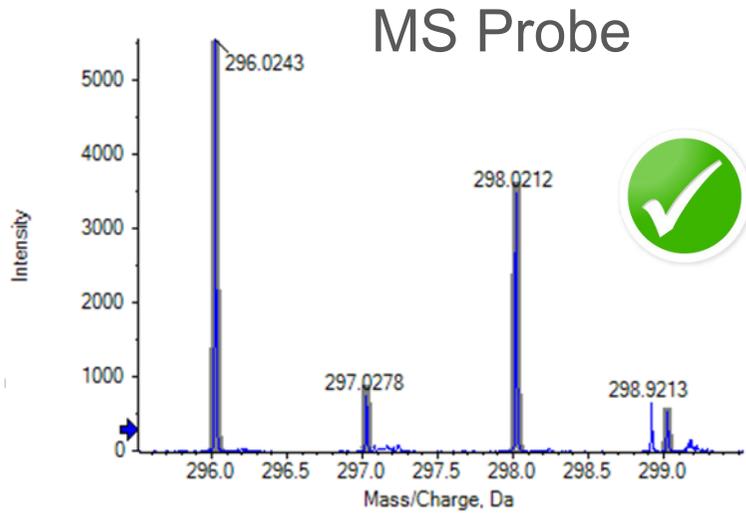
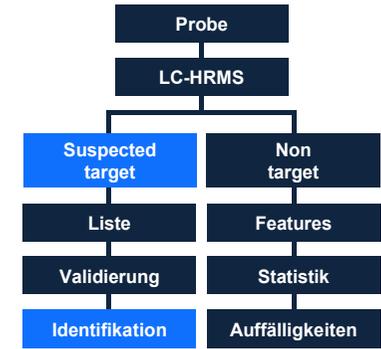
Probe



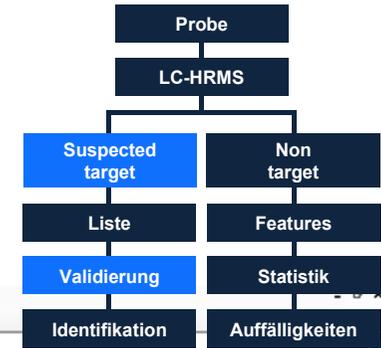
Standard



Identifikation



Manuelle Auswertung mit MasterView



XIC Probe

XIC Standard

MasterView 14-03-11_Askuris_57587-03_Klarwerksablauf_ausgewertet_reprocess.XIClist

C	T	R	L	Wiff file Name	Sample Name	Number of positive results
✓	✓	✓	✓	14-02-07_RT_SuMS_Substanzen_pos_Std 5ugl_03	Sample 1	81
✓	✓	✓	✓	13-12-05_TOF_BKG_pos_ASKURIS_57587-01_Blindprobe 1_05	Sample 1	37
✓	✓	✓	✓	13-12-05_TOF_BKG_pos_ASKURIS_57587-01_Blindprobe 1_05	Sample 2	35
✓	✓	✓	✓	13-12-05_TOF_BKG_pos_ASKURIS_57587-03_Klarwerksablauf 1_06	Sample 1	81
✓	✓	✓	✓	13-12-05_TOF_BKG_pos_ASKURIS_57587-03_Klarwerksablauf 1_06	Sample 2	76
✓	✓	✓	✓	13-12-05_TOF_BKG_pos_ASKURIS_57587-05_Ablauf Ozonanlage 1_07	Sample 1	59
✓	✓	✓	✓	13-12-05_TOF_BKG_pos_ASKURIS_57587-05_Ablauf Ozonanlage 1_07	Sample 2	65
✓	✓	✓	✓	13-12-05_TOF_BKG_pos_ASKURIS_57587-07_Ablauf Biofilter 1_08	Sample 1	58
✓	✓	✓	✓	13-12-05_TOF_BKG_pos_ASKURIS_57587-07_Ablauf Biofilter 1_08	Sample 2	60
✓	✓	✓	✓	13-12-05_TOF_BKG_pos_ASKURIS_57587_0-Injektion_03	Sample 1	26
✓	✓	✓	✓	13-12-05_TOF_BKG_pos_ASKURIS_57587_0-Injektion_03	Sample 2	24

Liste der prozessierten Proben

#	Mass	RT	Isotope	Name	Formula	Isotope	Mass (Da)	Adduc t	Int Std	Extraction Mass (Da)	Width (Da)	Width (ppm)	Expected RT (min)	RT (min)	Width (min)	Fragment Mass (Da)
99	✓	✓	✓	✓	✓	✓	295.01668	+	296.02396	0.005	16.891	12.06	12.06	1		
85	✓	✓	✓	✓	✓	✓	203.13101	+	204.13829	0.005	24.493	10.7	10.7	1		
86	✓	✓	✓	✓	✓	✓	219.12593	+	220.13321	0.005	22.714	0	1			
90	✓	✓	✓	✓	✓	✓	278.13789	+	279.14517	0.005	17.912	0	1			
100	✓	✓	✓	✓	✓	✓	266.09038	+	267.08765	0.005	18.72	0	1			
101	✓	✓	✓	✓	✓	✓	311.0116	+	312.01888	0.005	16.025	0	1			
119	✓	✓	✓	✓	✓	✓	230.14191	+	231.14919	0.005	21.631	0	1			
122	✓	✓	✓	✓	✓	✓	290.13789	+	291.14517	0.005	17.174	4.96	0.5			
130	✓	✓	✓	✓	✓	✓	266.16304	+	267.17032	0.005	18.715	4.4	1			
133	✓	✓	✓	✓	✓	✓	340.98578	+	341.99305	0.005	14.62	12.05	1			
137	✓	✓	✓	✓	✓	✓	286.05091	+	287.05818	0.005	17.418	0	1			
140	✓	✓	✓	✓	✓	✓	236.09496	+	237.10224	0.005	21.088	0	1			
141	✓	✓	✓	✓	✓	✓	252.08988	+	253.09715	0.005	19.755	0	1			
144	✓	✓	✓	✓	✓	✓	270.10044	+	271.10772	0.005	18.443	6	1			
146	✓	✓	✓	✓	✓	✓	432.04776	+	433.05504	0.005	11.546	4.7	1			
160	✓	✓	✓	✓	✓	✓	341.14093	+	342.14821	0.005	14.614	0	1			
164	✓	✓	✓	✓	✓	✓	267.18344	+	268.19072	0.005	18.643	5.5	1			
165	✓	✓	✓	✓	✓	✓	350.22268	+	351.22996	0.005	12.78	0	1			
171	✓	✓	✓	✓	✓	✓	254.09429	+	255.10157	0.005	19.6	0	1			
178	✓	✓	✓	✓	✓	✓	410.08962	+	411.0969	0.005	12.163	15.1	1			
180	✓	✓	✓	✓	✓	✓	259.15723	+	260.16451	0.005	19.219	0	1			
181	✓	✓	✓	✓	✓	✓	307.14514	+	308.15242	0.005	16.226	12.2	1			
183	✓	✓	✓	✓	✓	✓	341.12969	+	342.13697	0.005	14.614	4.6	1			
192	✓	✓	✓	✓	✓	✓	776.85412	+	777.8614	0.005						
193	✓	✓	✓	✓	✓	✓	776.85412	+	777.8614	0.005						
194	✓	✓	✓	✓	✓	✓	369.17223	+	370.1795	0.005						
203	✓	✓	✓	✓	✓	✓	299.15214	+	300.15942	0.005						
206	✓	✓	✓	✓	✓	✓	272.17763	+	273.18491	0.005	18.303	15.2	1			

XIC Liste



Zweckverband Landeswasserversorgung



- 18 -
Handeln | Kommunizieren

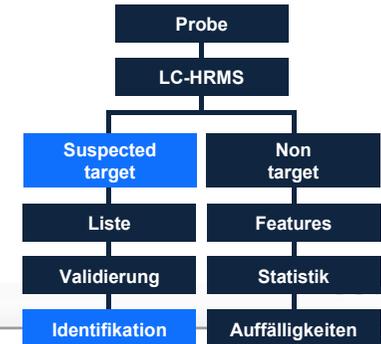
RISK IDENT Identifizieren | Bewerten



Bundesministerium für Bildung und Forschung

gefördert vom:

Manuelle Auswertung mit MasterView



MasterView 14-03-11_Askuris_57587-03_Klarwerksablauf_ausgewertet_reprocess.XIClist

CTRL	Wiff file Name	Sample Name	Number of positive results
✓	14-02-07_RT_SuMS_Substanzen_pos_Std_SugL_03	Sample 1	81
✓	13-12-05_TOF_BKG_pos_ASKURIS_57587-01_Blindprobe_1_05	Sample 1	37
✓	13-12-05_TOF_BKG_pos_ASKURIS_57587-01_Blindprobe_1_05	Sample 2	35
✓	13-12-05_TOF_BKG_pos_ASKURIS_57587-03_Klarwerksablauf_1_06	Sample 1	81
✓	13-12-05_TOF_BKG_pos_ASKURIS_57587-03_Klarwerksablauf_1_06	Sample 2	76
✓	13-12-05_TOF_BKG_pos_ASKURIS_57587-05_Ablauf Ozonanlage 1	Sample 1	59

#	Name	Formula	Isotope	Mass (Da)	Adduct	Int. Std.	Extraction Mass (Da)	Width (Da)	Width (ppm)	Expected RT (min)	RT Width (min)	Fragment Mass (Da)
85	Crotamiton	C13H17NO	0	203.13101	+H		204.13829	0.005	24.493	10.7	1	
86	Ritalinsäure	C13H17NO2	0	219.12593	+H		220.13321	0.005	22.714	0	1	
90	Pentoxifyllin	C13H18N4O3	0	278.13789	+H		279.14517	0.005	17.912	0	1	
99	Diclofenac	C14H11Cl2NO2	0	296.01668	+H		296.02396	0.005	16.891	12.06	1	
100	Valeriansäure	C14H10N4O2	0	266.08038	+H		267.08765	0.005	18.72	0	1	
101	4-Hydroxy-diclofenac	C14H11Cl2NO3	0	311.0116	+H		312.01888	0.005	16.02	0	1	
119	Propyphenazon	C14H18N2O	0	230.14191	+H		231.14919	0.005	21.63	0	1	
122	Trimethoprim	C14H18N4O3	0	290.13789	+H		291.14517	0.005	17.17	0	1	
130	Atenolol	C14H22N2O3	0	266.16304	+H		267.17032	0.005	18.72	0	1	

MS Probe: Spectrum from 13-12-05_TOF_BKG_pos_ASKURIS_57587-03_Klarwerksablauf_1_06.wiff (Klarwerksablauf 1; Experiment 1; +TOF MS (100 - 1200) from 12.081 to 12.122 min)

MS Standard: Spectrum from 14-02-07_RT_SuMS_Substanzen_pos_StdSugL_03.wiff (sample 1) - Std SugL; Experiment 1; +TOF MS (100 - 1200) from 12.051 to 12.090 min)

MS/MS Probe: Spectrum from 13-12-05_TOF_BKG_pos_ASKURIS_57587-03_Klarwerksablauf_1_06.wiff (sample 1) - 57587-03_Klarwerksablauf 1; Experiment 9; +TOF MS² (30 - 1200) from 12.051 min

MS/MS Standard: Spectrum from 14-02-07_RT_SuMS_Substanzen_pos_Std_SugL_03.wiff (sample 1) - Std SugL; Experiment 5; +TOF MS² (30 - 1200) from 12.077 min



Zweckverband
Landeswasserversorgung



- 19 -
Handeln | Kommunizieren

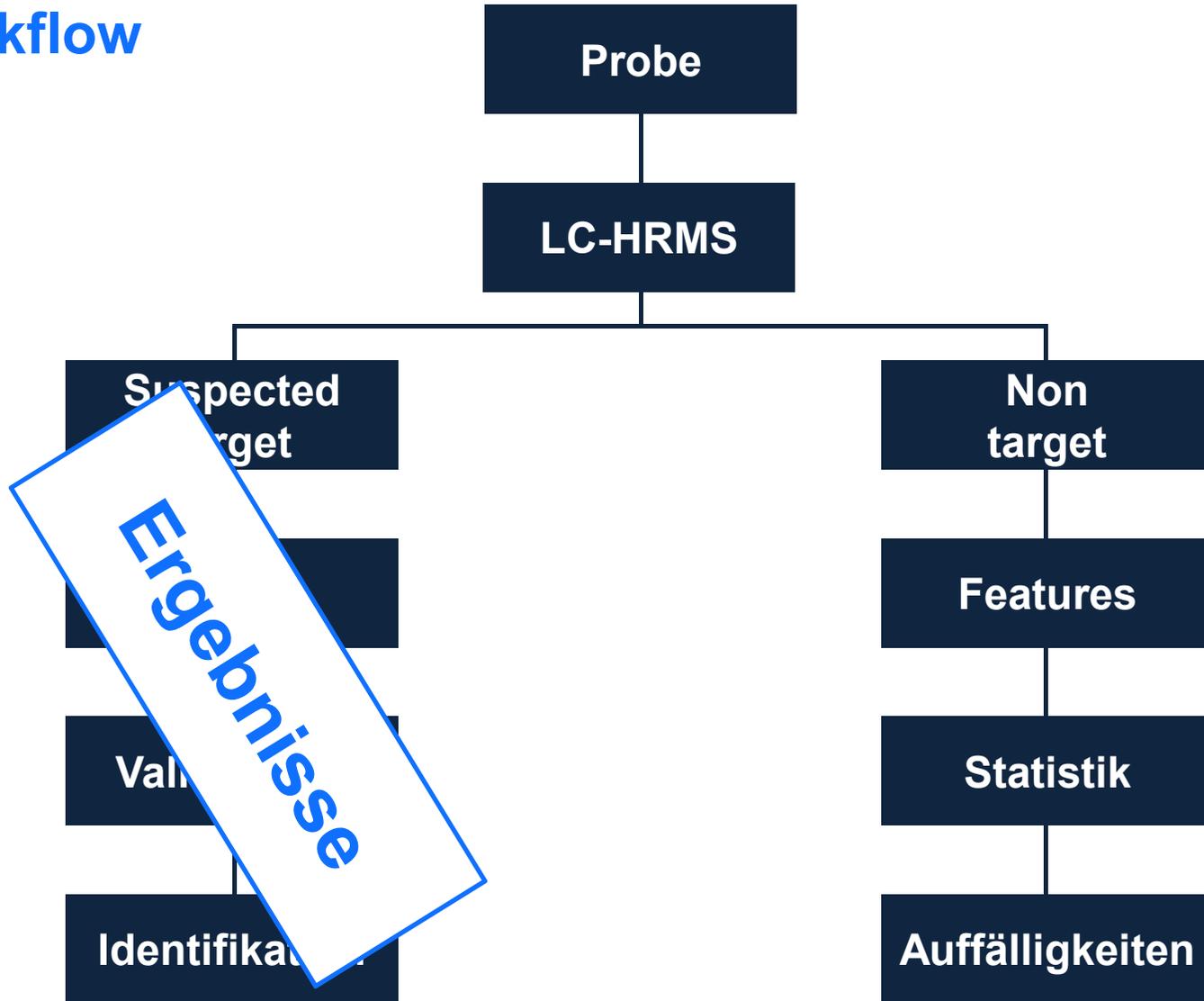
RISK Identifizieren | Bewerten
IDENT



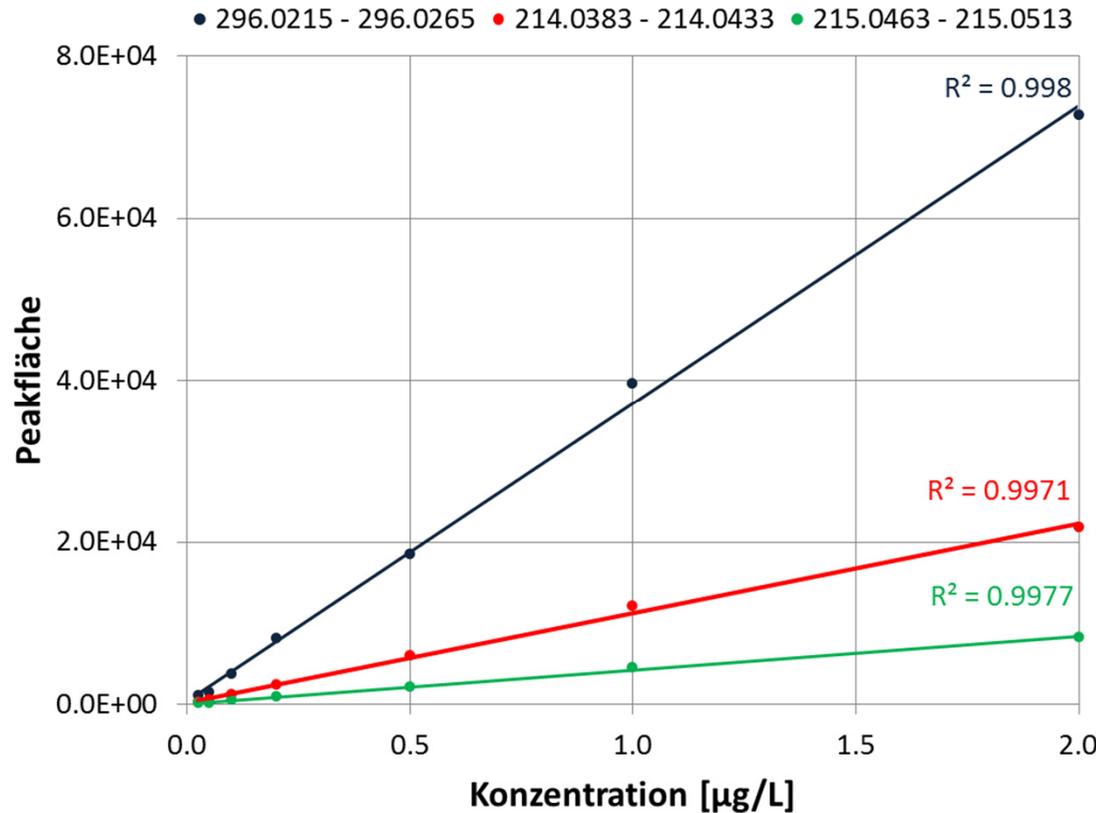
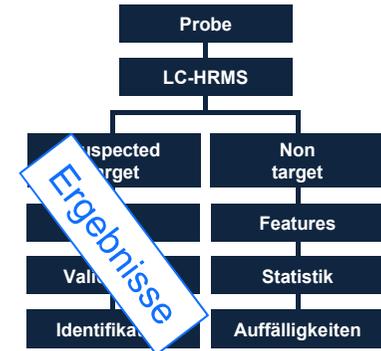
Bundesministerium
für Bildung
und Forschung

gefördert vom:

Workflow



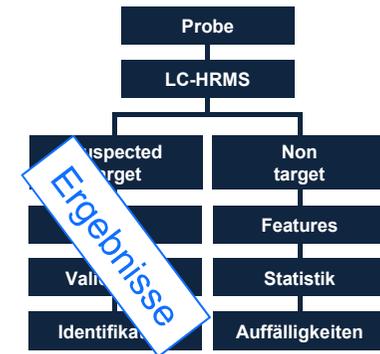
Kalibrierung am Beispiel von Diclofenac



Kalibrierung

- MS:
Exakte Masse
- MSMS:
Erster Massenübergang
Zweiter Massenübergang

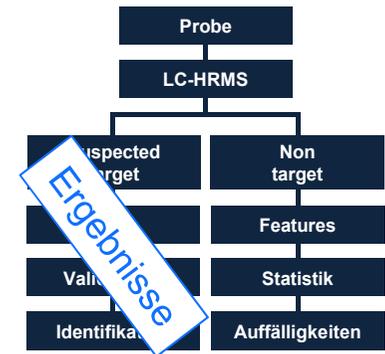
Quantifizierungsergebnisse 20 Substanzen



Stoffname	Konzentration [µg/L]					
	Blindprobe	Ablauf Kläranlage	Ablauf Ozonanlage	Trend	Ablauf Biofilter	Trend
Acetylaminoantipyrin (AAA)	< 0,03	2,2	< 0,03	↓	< 0,03	⇒
Aminoantipyrin (AA)	< 0,05	0,12	< 0,05	↓	< 0,05	⇒
Benzotriazol	< 0,05	5,9	2,7	↘	2,9	⇒
Bezafibrat	< 0,03	1,2	0,28	↘	0,24	⇒
Carbamazepin	< 0,03	1,7	< 0,03	↓	< 0,03	⇒
Diclofenac	< 0,03	6,0	0,03	↘	< 0,03	↓
4-Hydroxydiclofenac	< 0,03	0,06	0,14	↑	0,14	⇒
Formylaminoantipyrin (FAA)	< 0,03	8,5	< 0,03	↓	< 0,03	⇒
Gabapentin	< 0,03	17,0	10,0	↘	10,0	⇒
Irbesartan	< 0,03	0,23	0,13	↘	0,12	⇒
Metoprolol	< 0,05	3,7	1,1	↘	< 0,05	↓
Oxazepam	< 0,03	0,07	0,03	↘	0,03	⇒
Phenazon	< 0,03	0,37	< 0,03	↓	< 0,03	⇒
Phenylethylmalonamid (PEMA)	< 0,05	0,55	0,39	↘	0,39	⇒
Primidon	< 0,03	0,54	0,32	↘	0,31	⇒
Sotalol	< 0,05	0,19	< 0,05	↓	< 0,05	⇒
Sucralose	n.n.	2,5	2,5	⇒	2,5	⇒
Toluolsulfonamid (TSA)	n.b.	n.b.	n.b.	-	n.b.	-
Tolyltriazol	< 0,03	4,8	1,8	↘	1,7	⇒
Valsartansäure	< 0,05	8,2	4,4	↘	4,2	⇒

- ↓ Entfernung bis unter die Bestimmungsgrenze
- ↘ signifikante Abnahme
- ↑ Zunahme
- ⇒ keine Veränderung

Identifizierte Substanzen (mittels Referenzmaterial mit RT und MS/MS)



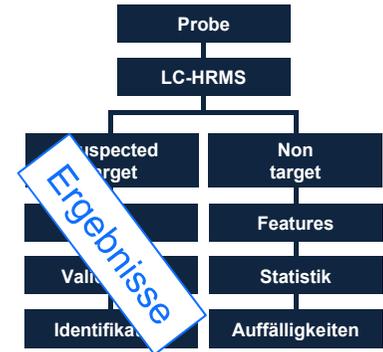
Ablauf Kläranlage UND Ablauf Ozonung

Name
4-Hydroxy-diclofenac
Amidotrizesäure
Lamotrigin
PEMA
Caffeine
Sulfathiazole
Iomeprol
Dimethoate
Diclofenac
1-Adamantylamin
Primidon
HMMM (2,4,6-Tris[bis(methoxymethylamino)-1,3,5-triazine
Benazolin
Valsartansäure
DEET (N,N-Diethyl-meta-toluamid)
Carbamazepin-Epoxid
10,11-Dihydroxy-10,11-dihydrocarbamazepin

Name
1-H-Benzotriazol
Oxazepam
Valsartan
Irbesartan
Tolyltriazol (Isomeregemisch)
Bezafibrat
Terbutryn
Metoprolol
Atenolol
Candesartan
2-(Methylthio)benzothiazol
Bisoprolol
Telmisartan
Sulfamethoxazol
Tramadol
Phenazon
4-Formylaminoantipyrine

34 Substanzen

Identifizierte Substanzen (mittels Referenzmaterial mit RT und MS/MS)



NUR Ablauf Kläranlage

Name
1-Hydroxy-benzotriazole
4-Acetamidoantipyrine
4-Hydroxy-1H-benzotriazole
Acetyl-Sulfamethoxazol
Amisulprid
Azithromycin
Carbamazepin
Carbendazim
Clarithromycin
Crotamiton
Diuron
Eprosartan
Erithromycin

Name
Indometacin
Isoproturon
Ketoprofen
Lamotrigin (oxidierte Form)
Losartan
Pethoxamid Met -42
Propranolol
Propyphenazon
Ritalinsäure
Roxithromycin
Sotalol
Sulpirid
Trimethoprim

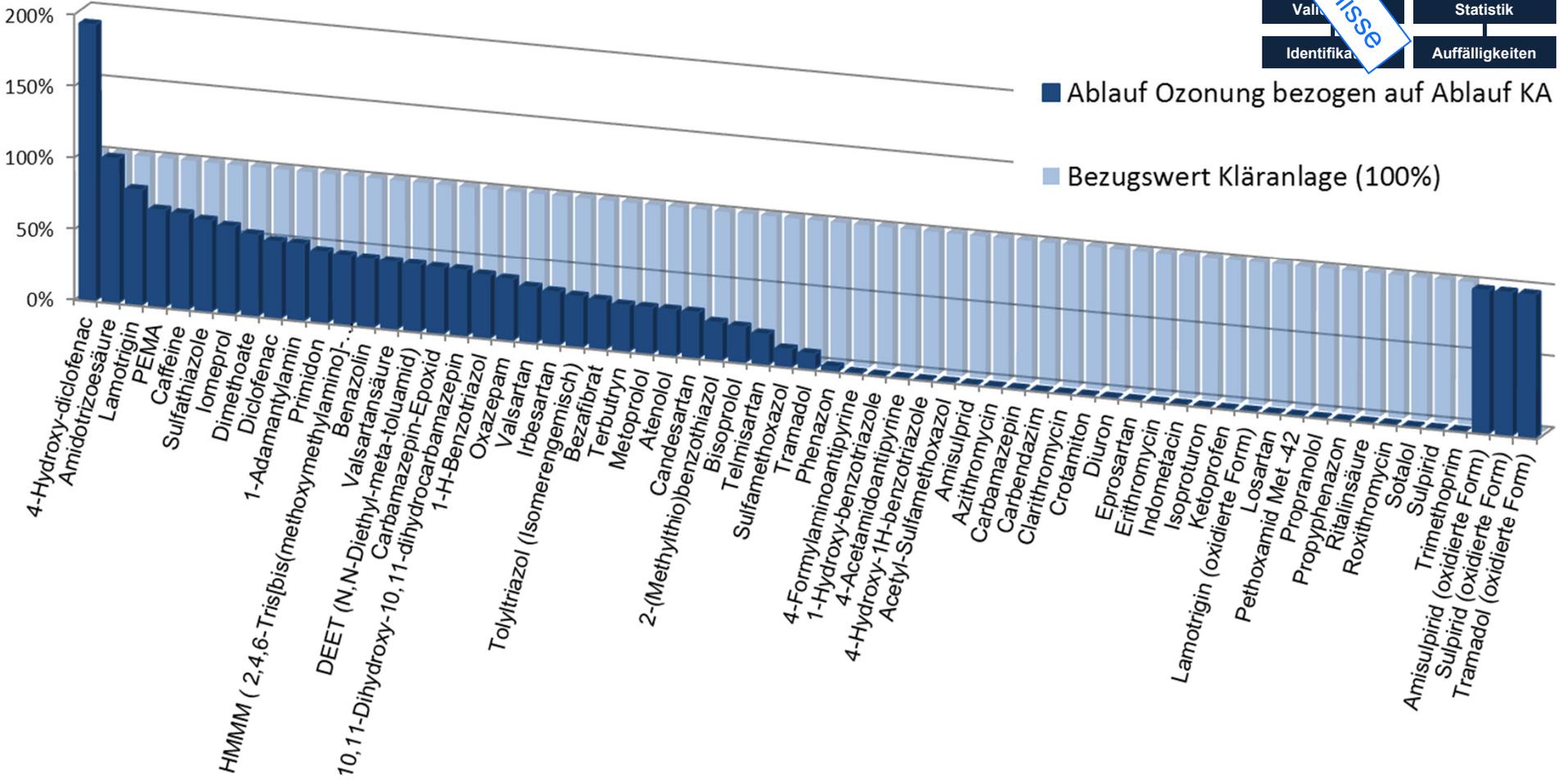
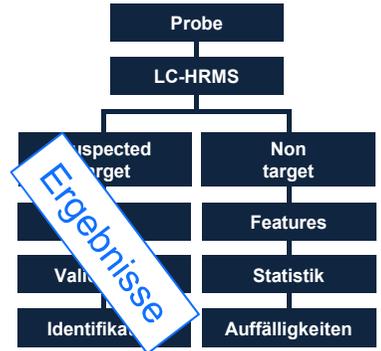
26 Substanzen

NUR Ablauf Ozonanlage

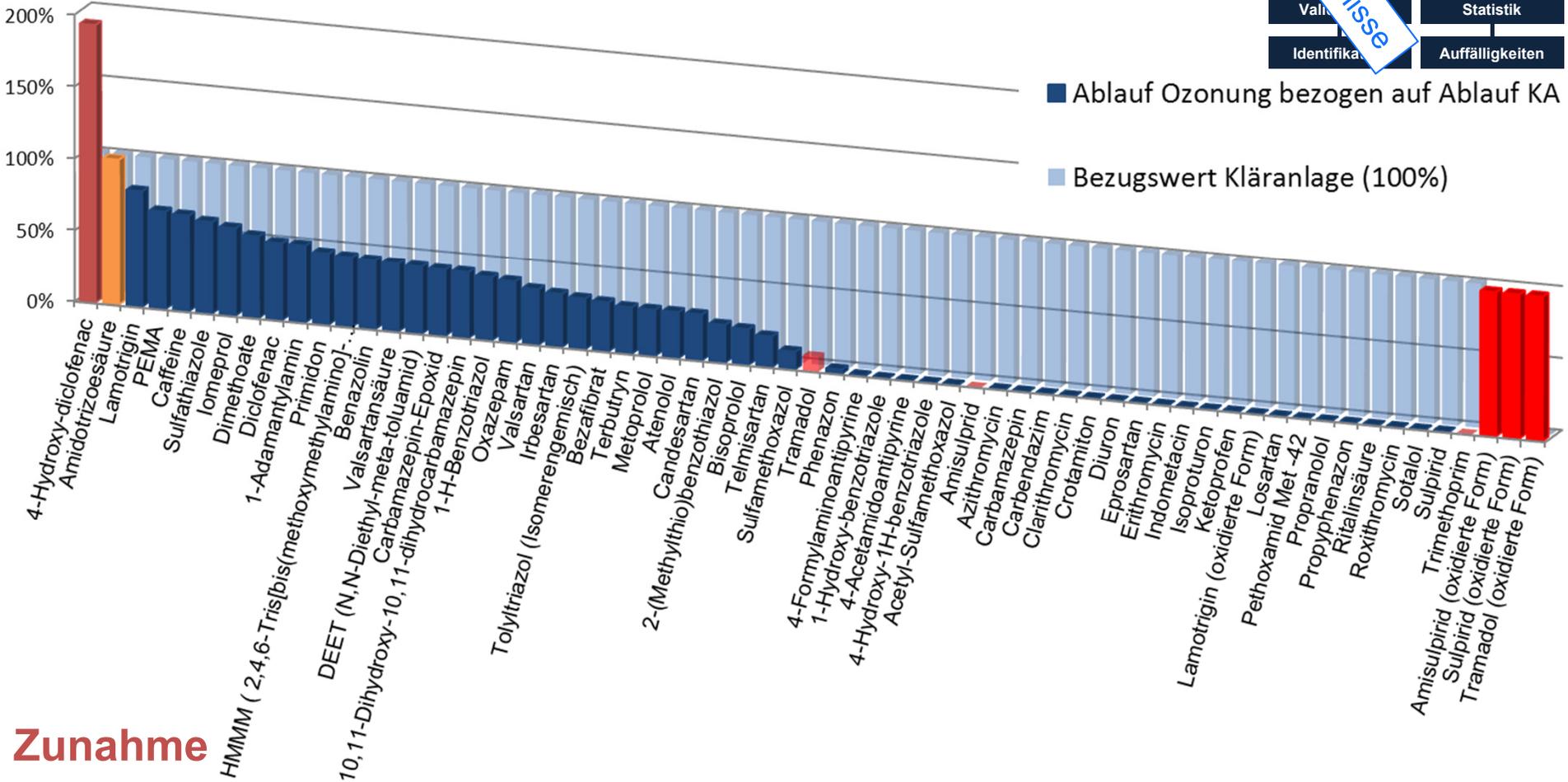
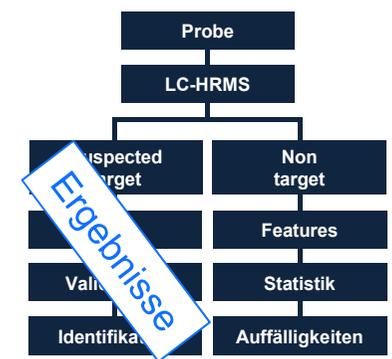
Name
Amisulpirid (oxidierte Form)
Sulpirid (oxidierte Form)
Tramadol (oxidierte Form)

3 Substanzen

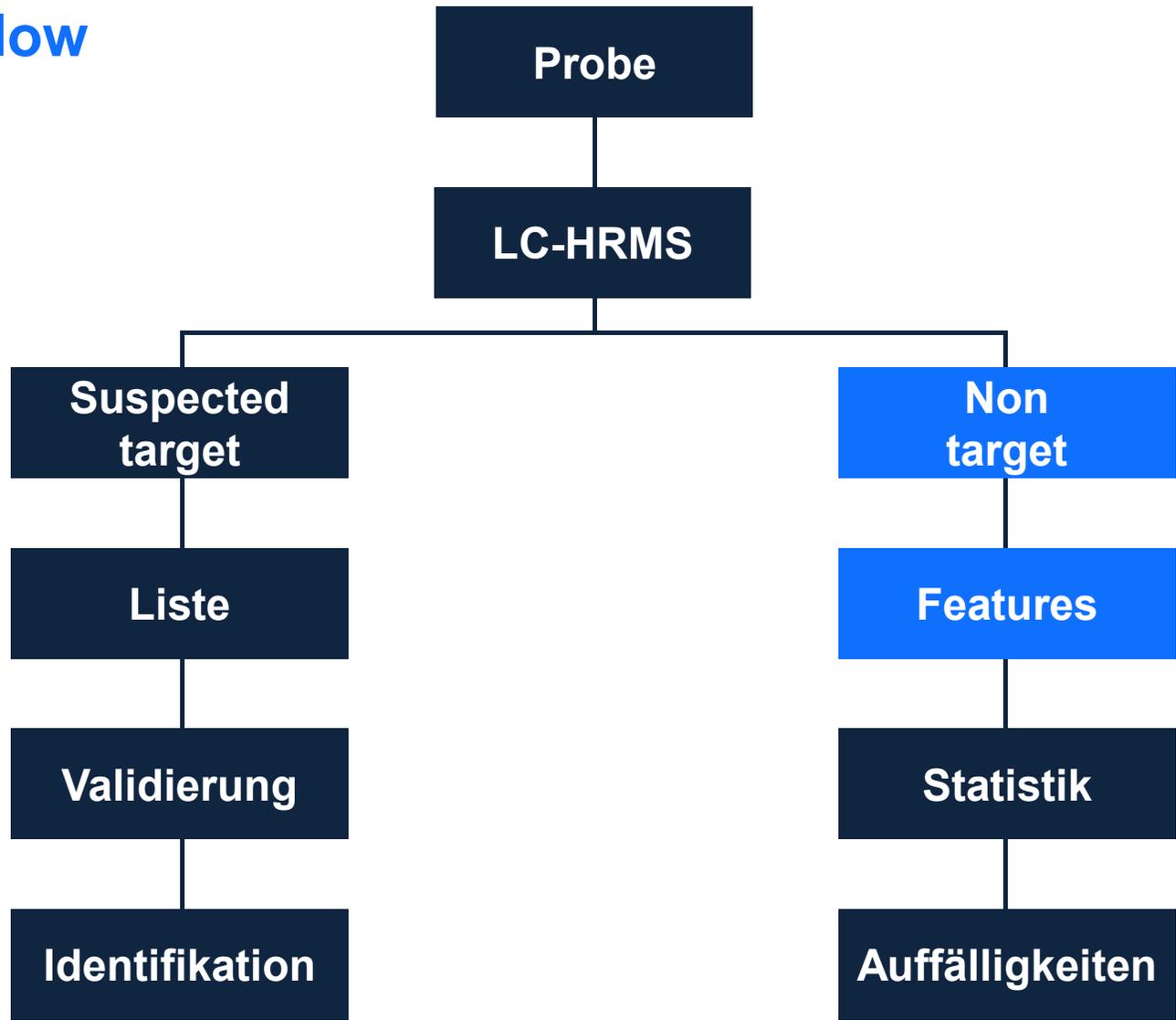
Verhalten der identifizierten Substanzen



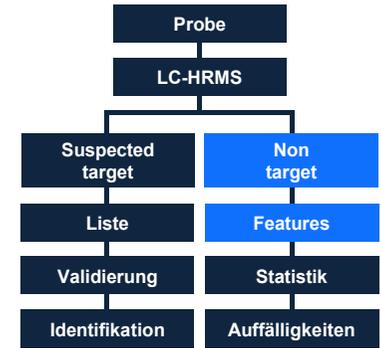
Verhalten der identifizierten Substanzen



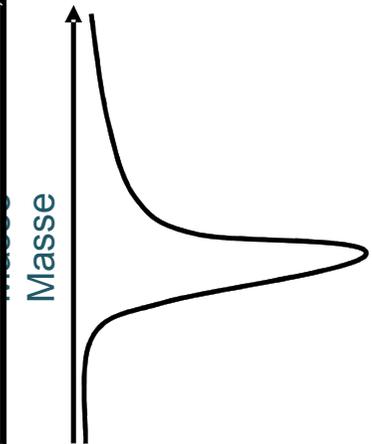
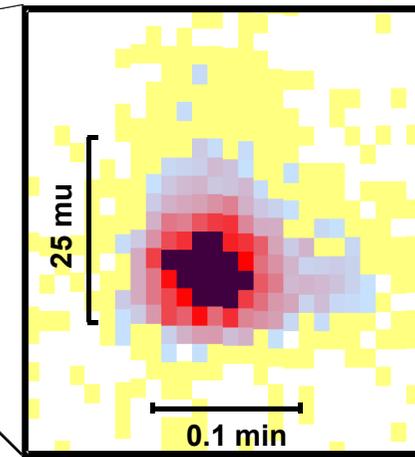
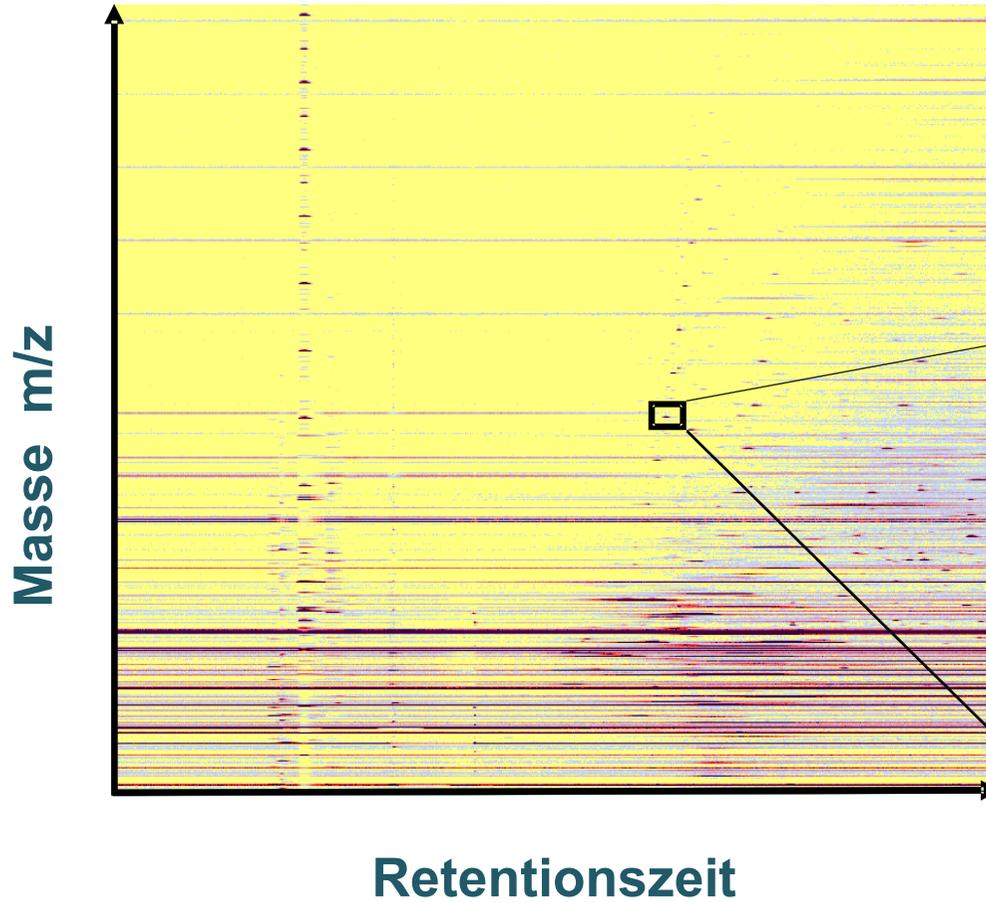
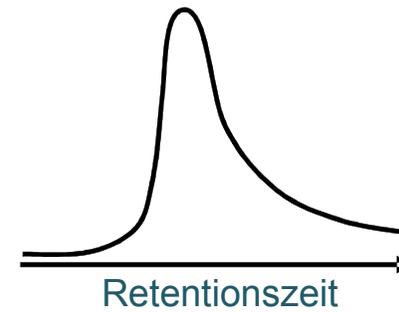
Workflow



Contour-Plot

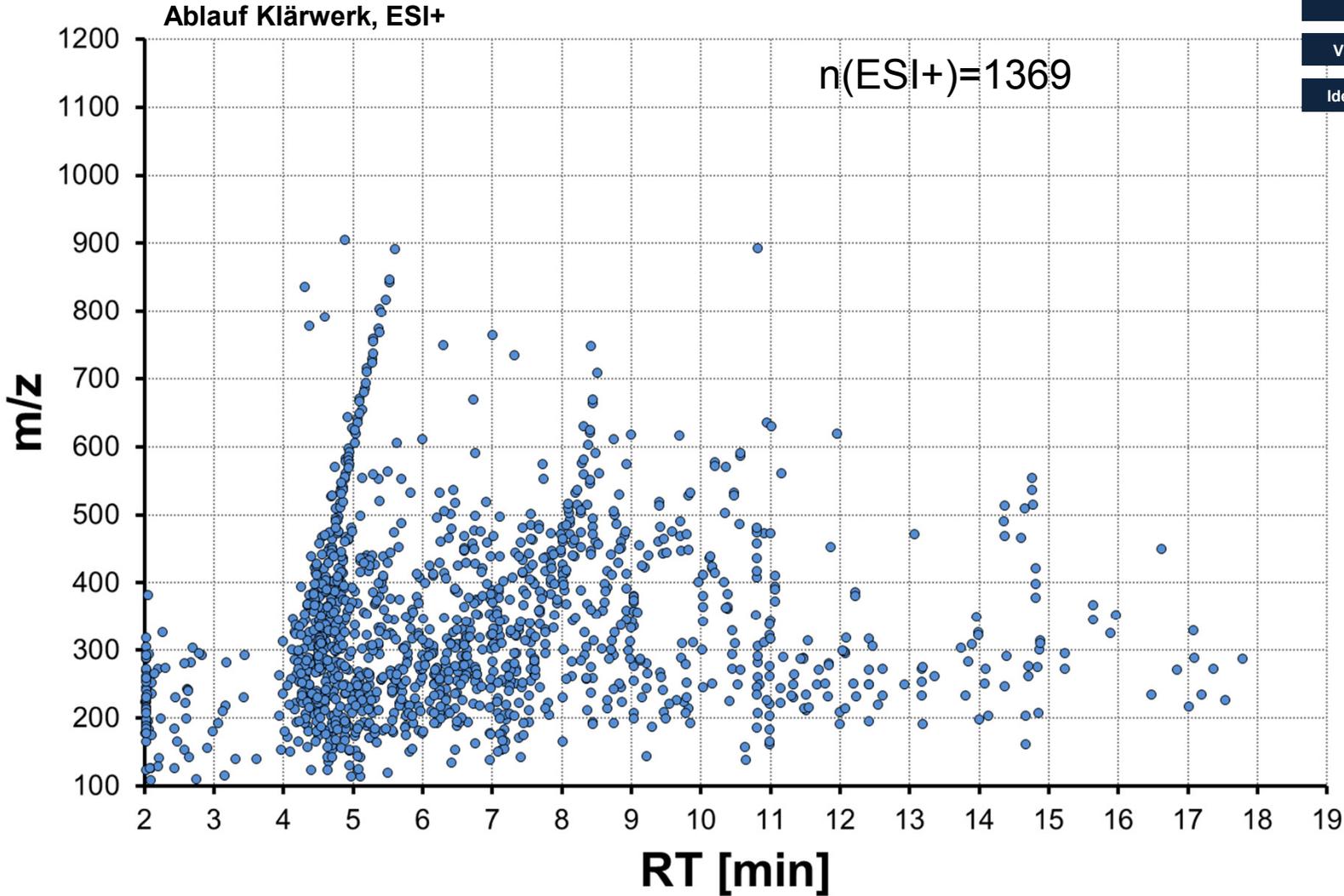
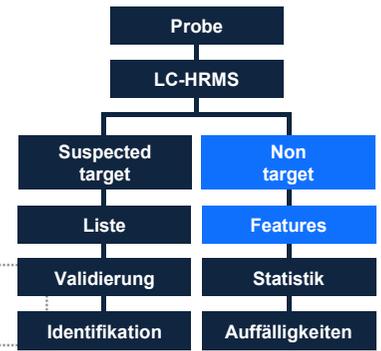


Elutions-Profil
Chromatographie

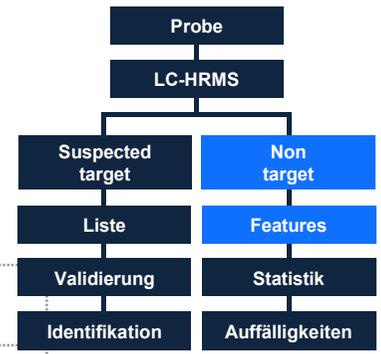


Massen-Profil
Massenspektrum

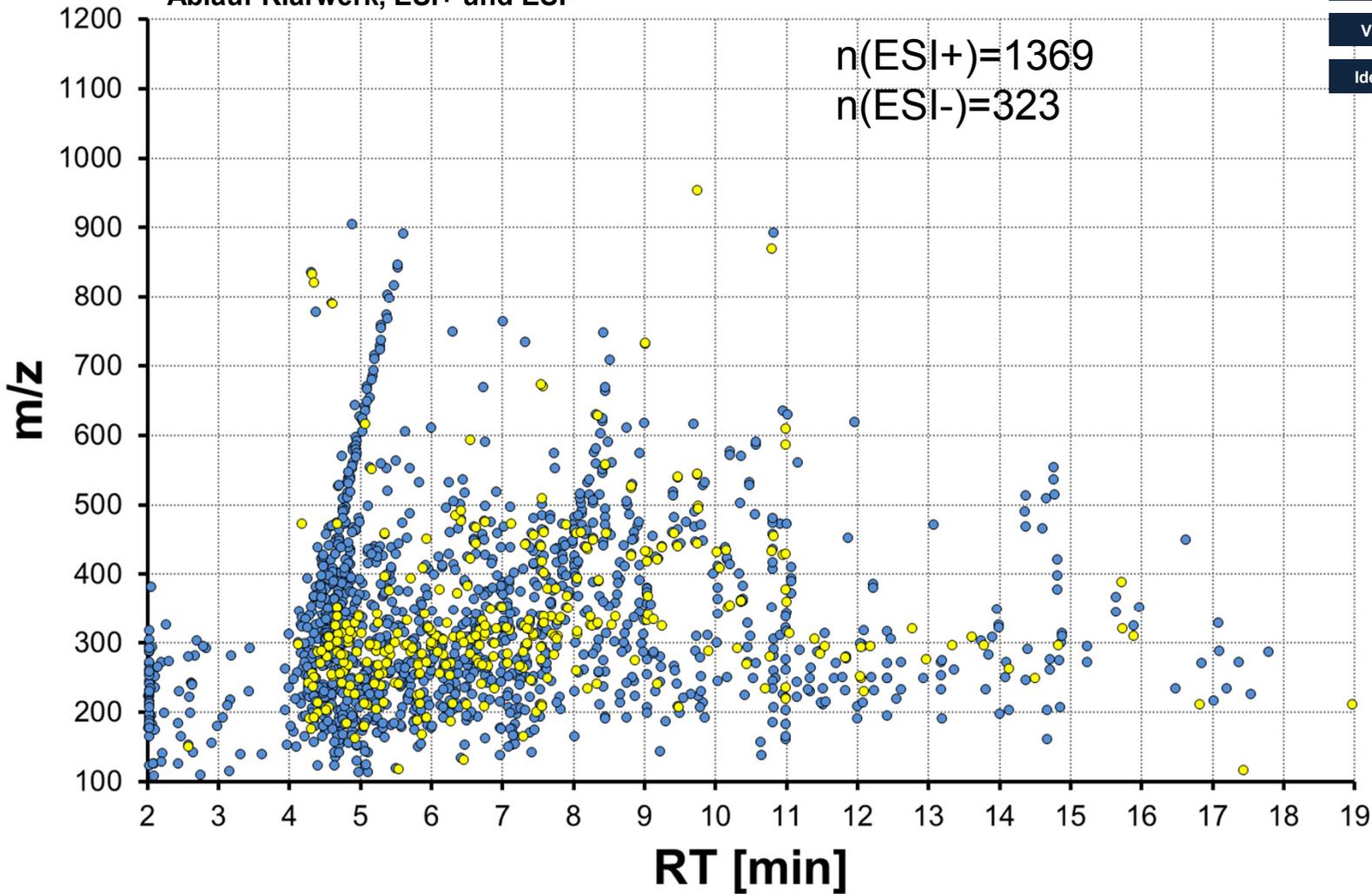
Punktwolke



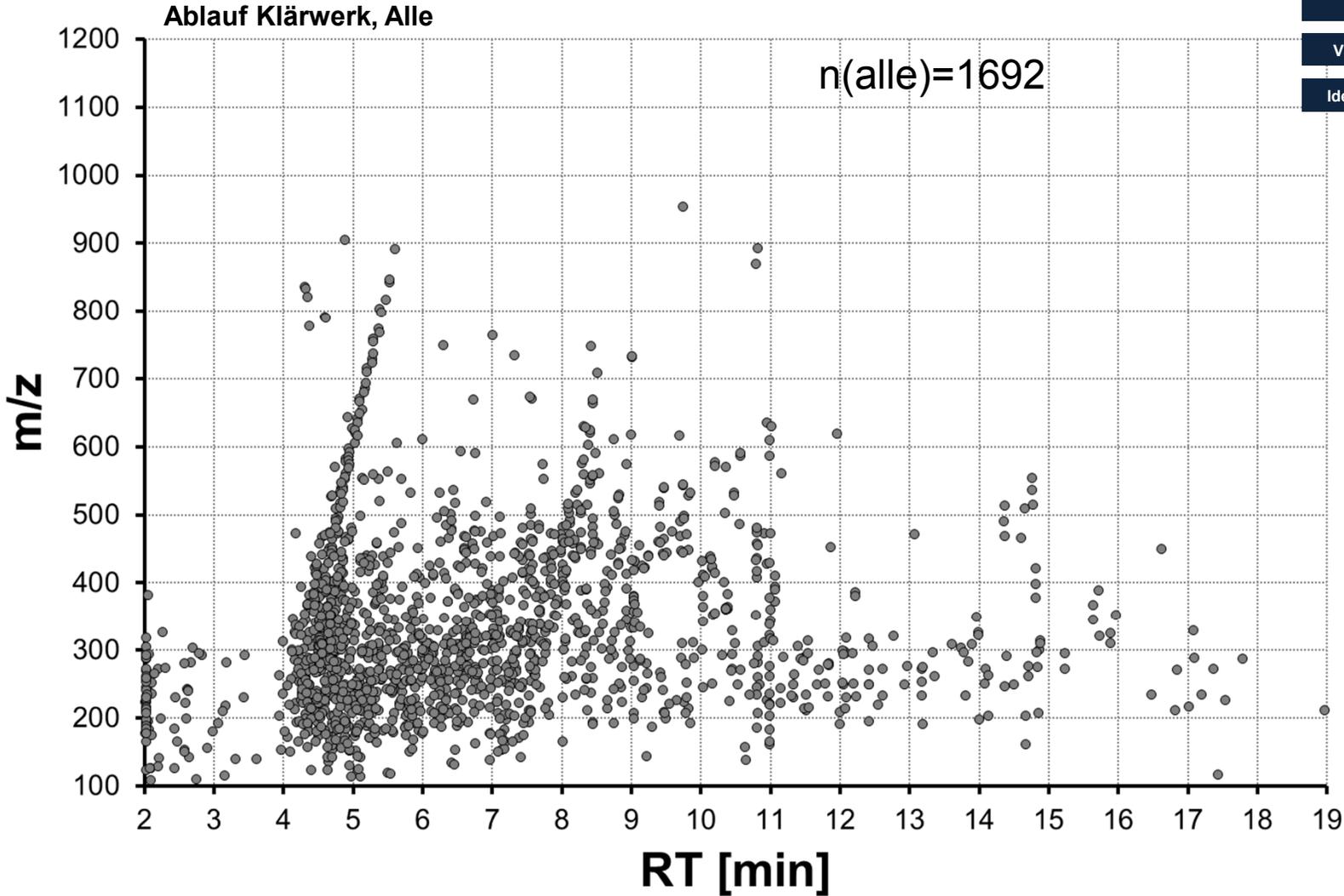
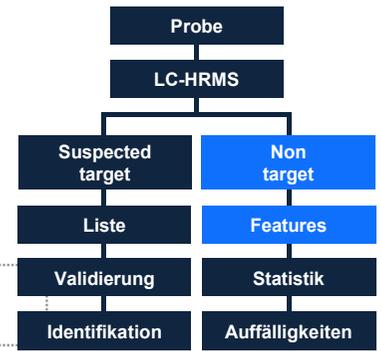
Punktvolke



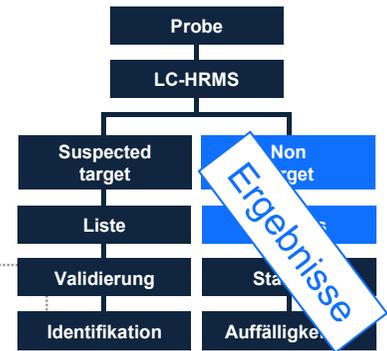
Ablauf Klärwerk, ESI+ und ESI-



Punktwolke

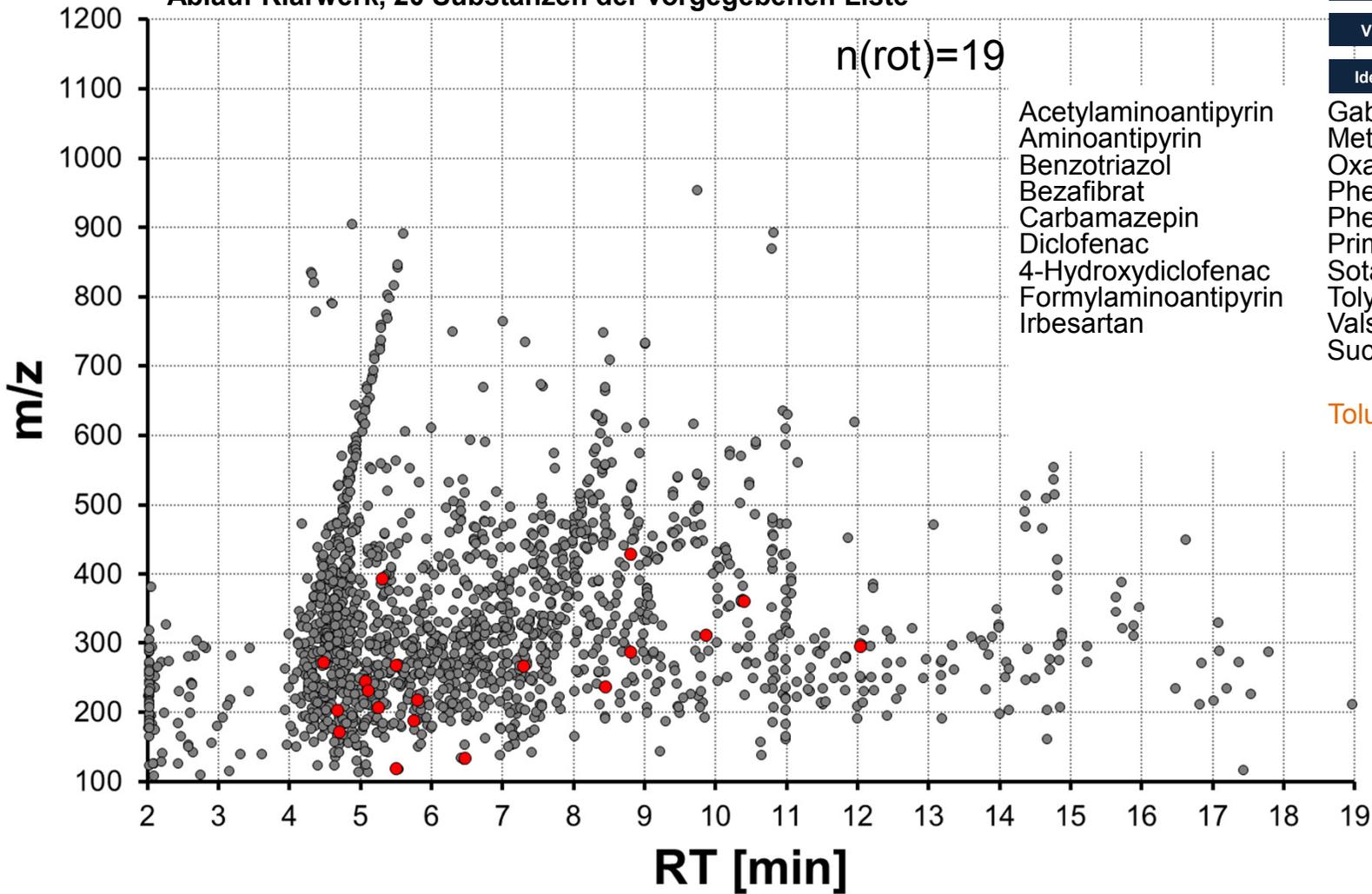


Punktvolke



Ablauf Klärwerk, 20 Substanzen der vorgegebenen Liste

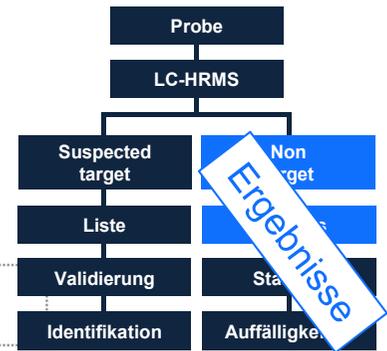
n(rot)=19



- Acetylaminoantipyrin
- Aminoantipyrin
- Benzotriazol
- Bezafibrat
- Carbamazepin
- Diclofenac
- 4-Hydroxydiclofenac
- Formylaminoantipyrin
- Irbesartan
- Gabapentin
- Metoprolol
- Oxazepam
- Phenazon
- Phenylethylmalonamid
- Primidon
- Sotalol
- Tolyltriazol
- Valsartansäure
- Sucralose

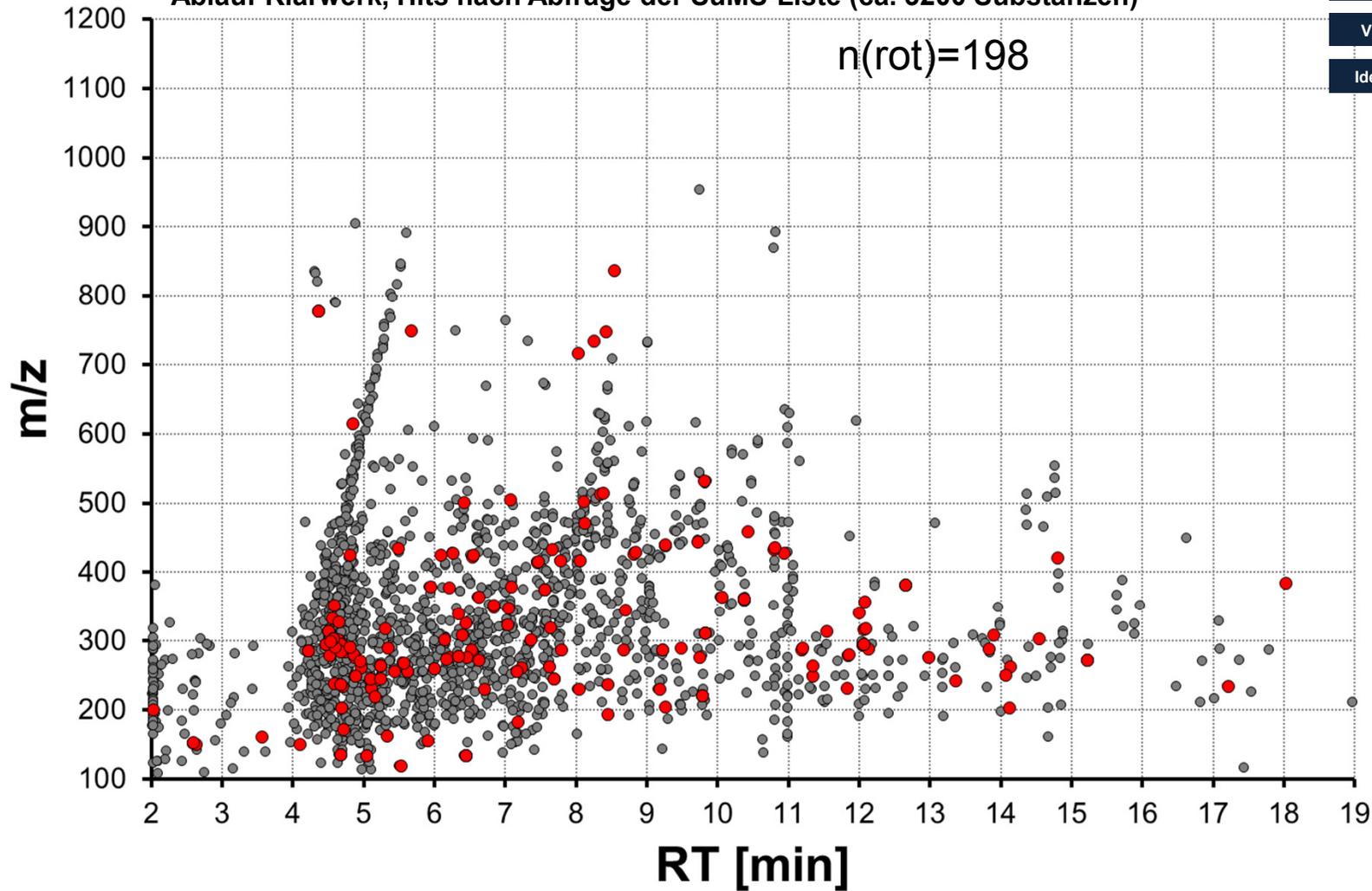
Toluolsulfonamid

Punktvolke

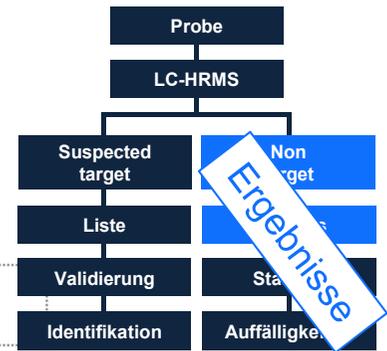


Ablauf Klärwerk, Hits nach Abfrage der SuMS-Liste (ca. 3200 Substanzen)

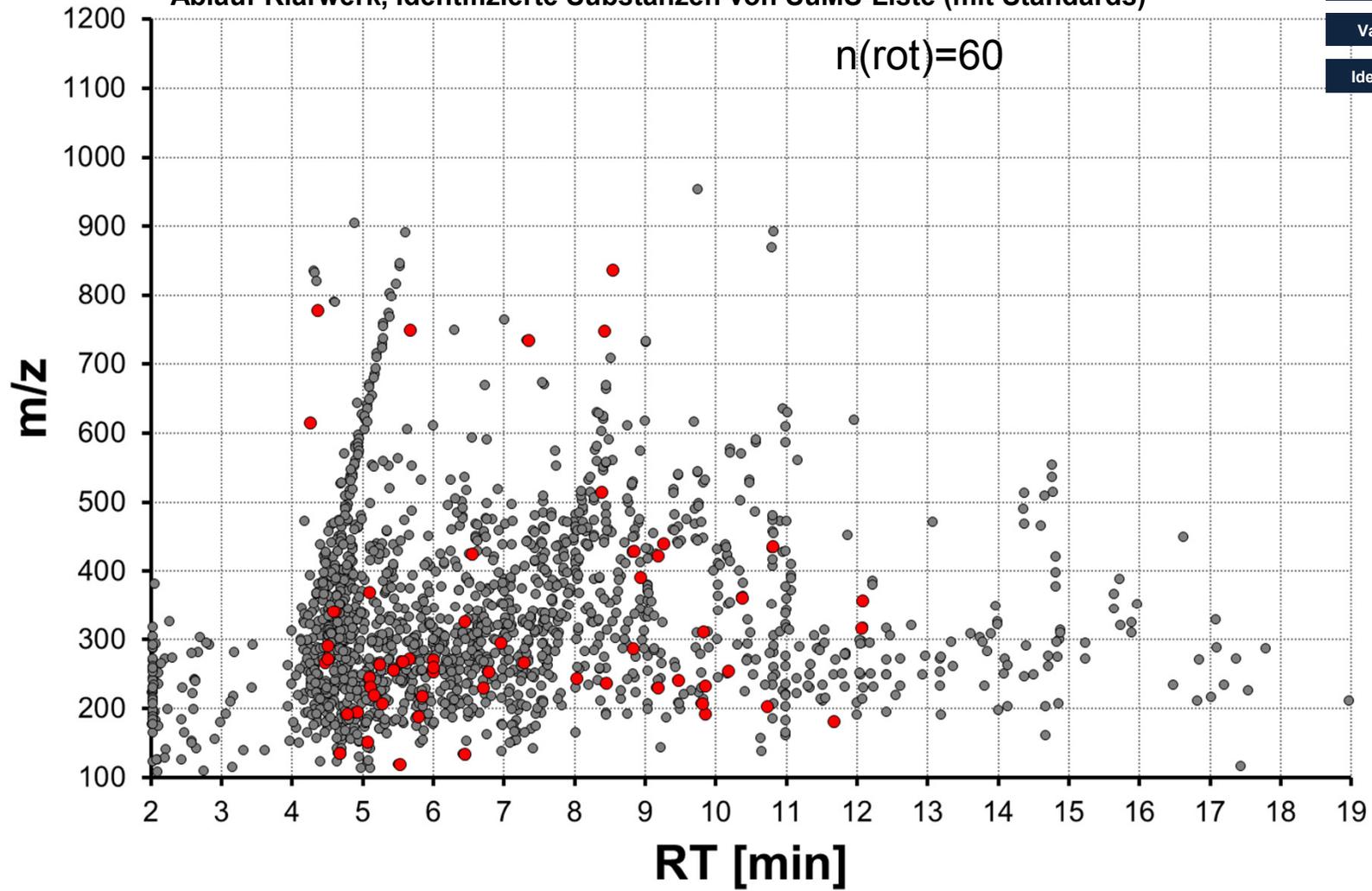
n(rot)=198



Punktvolke



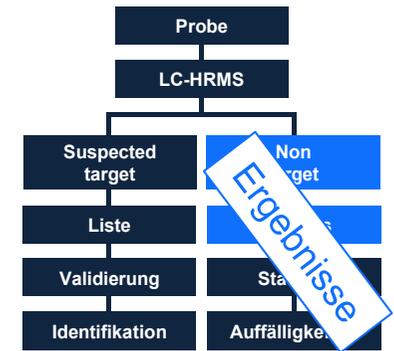
Ablauf Klärwerk, Identifizierte Substanzen von SuMS-Liste (mit Standards)



Abfrage NTS-Features (KA-Ablauf) in Stoff-Ident ESI positiv

aus 1369 Features

3 Treffer für m/z 263,1885
logP zur genaueren Zuordnung



Gefunden: 694 Einträge · 694 sichtbar

Filter Bereichsfilter (z.B. 1.45-2.54) Nur Beste Herunterladen

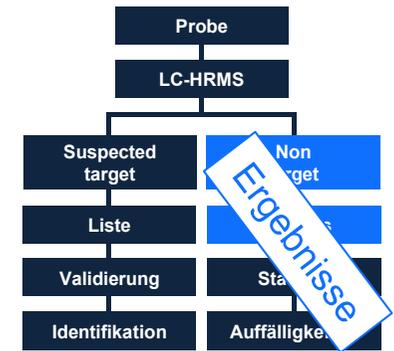
Target identifier	Best match	Monoisotopic m/z	Δ mass	logP	Δ logP	Name	CAS	EC Number	Elemental formula	SMILES	IUPAC
264,2/5,2 (697)		263.1885	0.0001	3.48	2.94	BUTETAMATE	14007-64-i		C16H25NO2	CCC(C(=O)OCCN(CC)CC)c1ccc	2-(
264,2/5,2 (697)		263.1885	0.0001	2.45	1.91	tramadol	27203-92-i		C16H25NO2	COc1cccc(c1)C1(O)CCCC1CN	2-[
264,2/5,2 (697)	X	263.1885	0.0001	2.29	1.75	O-Desmethylvenlafaxine	93413-62-i		C16H25NO2	CN(C)CC(c1ccc(O)cc1)C1(O)CC	4-[
237,1/8,4 (483)	X	236.0950	0.0009	2.77	0.97	Carbamazepine	298-46-4		C15H12N2O	NC(=O)N1c2cccc2C=Cc2cccc	2-a

O-Desmethylvenlafaxine Textform Herunterladen

Eigenschaft	Wert	Quelle	Zusatz	Editor	Letzte Änderung
name	O-Desmethylvenlafaxine	Zweckverband Landes		Anne Bayer	2014.02.25
elemental formula	C16H25NO2	chemicalize.org		Anne Bayer	2014.03.07
accurate mass	263.1885290495	calculated		Anne Bayer	2014.03.07
CAS	93413-62-8	Zweckverband Landes		Anne Bayer	2014.02.25
SMILES	CN(C)CC(c1ccc(O)cc1)C1(O)CC	chemicalize.org		Anne Bayer	2014.03.07
IUPAC	4-[2-(dimethylamino)-1-(1-hy	chemicalize.org		Anne Bayer	2014.03.07

Abfrage NTS-Features (KA-Ablauf) in Stoff-Ident ESI negativ

aus 323 Features



Gefunden: 173 Einträge 173 sichtbar Herunterladen

Filter Bereichsfilter (z.B. 1.45-2.54) Nur Beste

Target identifier	Best match	Monoisotopic mass	Δ mass	logP	Δ logP	Name	CAS	EC Number	Elemental formula	SMILES	IUPAC
427,2/8,8 (351)	X	428.2325	-0.0003	5.32	3.30	irbesartan	138402-11		C25H28N6O	CCCC1=NC2(CCCC2)C(=O)N	2-b
439,2/9,2 (363)	X	440.1597	-0.0002	4.93	2.84	candesartan	139481-59		C24H20N6O3	CCOc1nc2cccc(C(O)=O)c2n1C	2-e

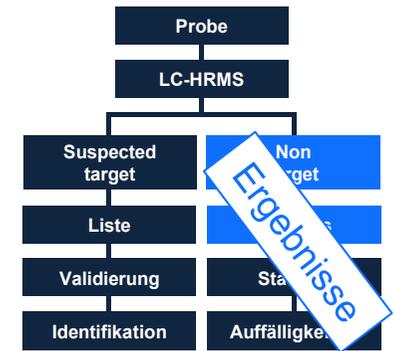
irbesartan Textform Herunterladen

Allgemein

Eigenschaft	Wert	Quelle	Zusatz	Editor	Letzte Änderung
name	irbesartan	Oberacher 2011		Anne Bayer	2014.02.25
elemental formula	C25H28N6O	Oberacher 2011		Anne Bayer	2014.02.25
accurate mass	428.23245955989995	calculated		Anne Bayer	2014.03.07
CAS	138402-11-6	Oberacher 2011		Anne Bayer	2014.02.25
SMILES	CCCC1=NC2(CCCC2)C(=O)N	chemicalize.org		Anne Bayer	2014.02.25
IUPAC	2-butyl-3-({4-[2-(1H-1,2,3,4-	chemicalize.org		Anne Bayer	2014.03.07

logP

Beispiel: Treffer in Stoff-Ident für Diclofenac



Gefunden: 694 Einträge - 694 sichtbar Herunterladen

Filter Bereichsfilter (z.B. 1.45-2.54) Nur Beste

Target identifier	Best match	Monoisotopic mass	Δ mass	logP	Δ logP	Name	CAS	EC Number	Elemental formula	SMILES	IUPAC
296,0/12,1 (950)		295.0167	0.0006	6.09	2.94	Meclofenamic acid	644-62-2		C ₁₄ H ₁₁ Cl ₂ N ₂ O ₂	Cc1ccc(Cl)c(Nc2ccccc2C(=O)=O	2-(2,6-Dichlorophenyl)amin
296,0/12,1 (950)	X	295.0167	0.0006	4.26	1.11	Diclofenac	15307-86-5		C ₁₄ H ₁₁ Cl ₂ N ₂ O ₂	OC(=O)Cc1ccccc1Nc1c(Cl)cccc	2-[2-(2,6-Dichlorophenyl)amin
447,3/9,7 (1801)	X	446.2430	0.0001	3.91	1.79	irbesartan-metabolite 446			C ₂₅ H ₃₀ N ₆ O ₂	CCCCC(=O)NC1(CCCC1)C(=O)	1-piperidinyl-4-oxo-1,2,3,4-tetrahydro-5H-benzodiazepin-5-one

Diclofenac Textform Herunterladen

Allgemein

Eigenschaft	Wert	Quelle	Zusatz	Editor	Letzte Änderung
name	Diclofenac	Drewes et al. 2008		Anne Bayer	2014.02.25
elemental formula	C ₁₄ H ₁₁ Cl ₂ N ₂ O ₂	Drewes et al. 2008		Anne Bayer	2014.02.25
accurate mass	295.0166840229	calculated		Anne Bayer	2014.03.07
CAS	15307-86-5	Drewes et al. 2008		Anne Bayer	2014.02.25
SMILES	OC(=O)Cc1ccccc1Nc1c(Cl)cccc	NORMAN 2011		Anne Bayer	2014.03.06
IUPAC	2-[2-(2,6-Dichlorophenyl)amin	NORMAN 2011		Anne Bayer	2014.03.06

Datenbanksuche mit Treffern aus Stoff-Ident in DAIOS

DAIOS-Suche: Diclofenac ergibt: MS/MS-Daten UND Transformation Tree

Diclofenac

Name	CAS	Exact mass	Molare mass
Diclofenac	15307-86-5	295.0167	296.15

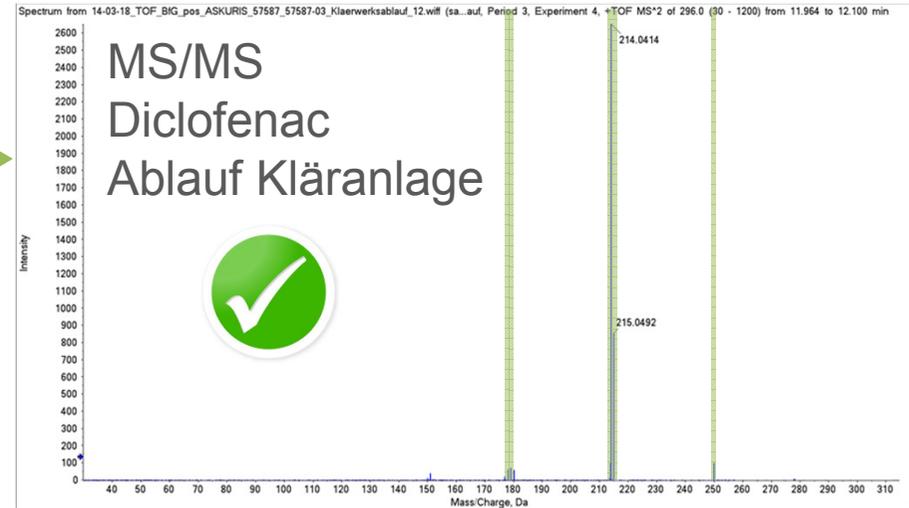
Fragmentmassen

Property	Value	Fragments
Precursor Mass	296.1000	214.0000
Ion	+H	249.8000
		236.0000
		179.0000
		178.0000
		222.0000
		208.0000

Transformation Tree

```

    graph LR
      A[Diclofenac] --> B[4-Hydroxydiclofenac]
  
```



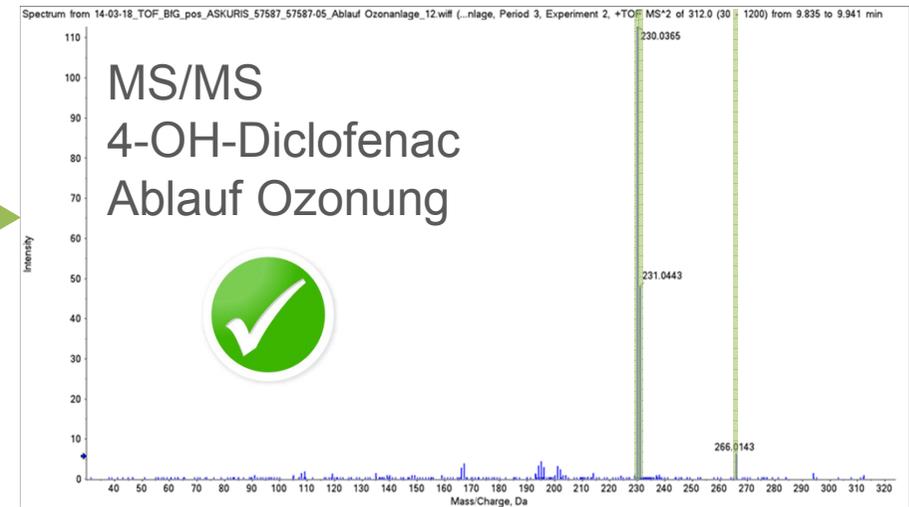
4- Hydroxydiclofenac

4-Hydroxydiclofenac

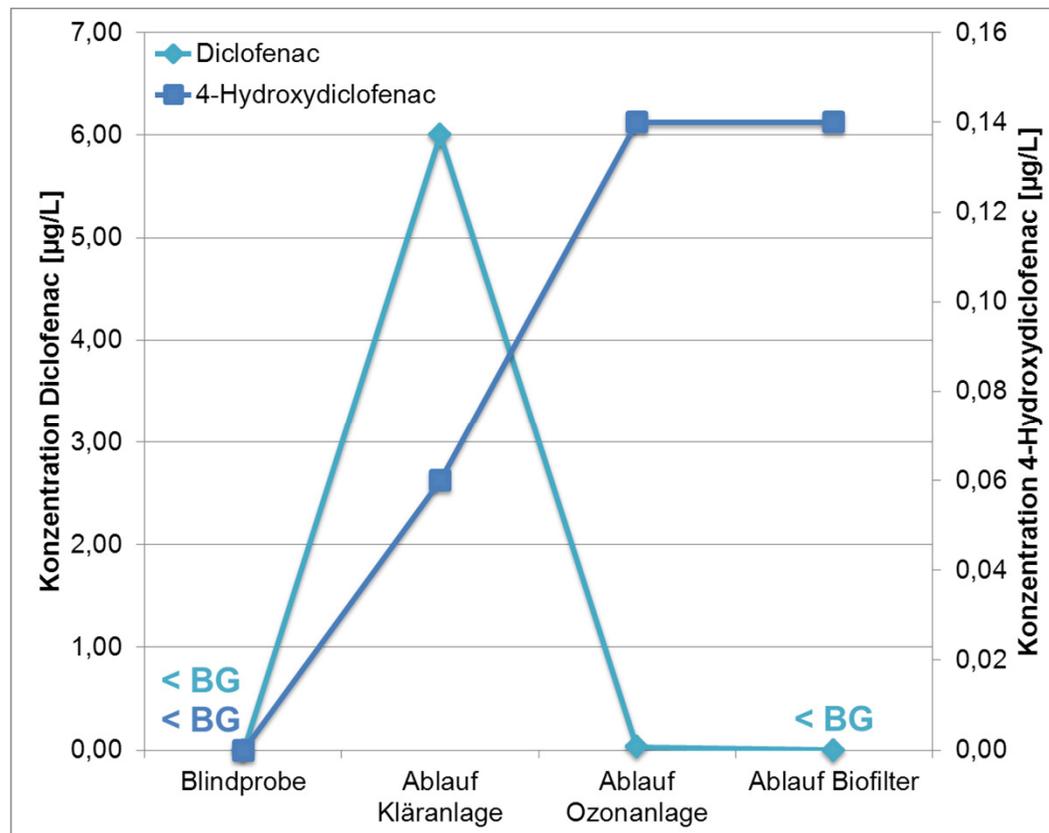
Property	Value
Name	4-Hydroxydiclofenac
IUPAC	2-((2,6-dichloro-4-hydroxyphenyl)amino)phenylacetic acid
Synonym	4'-Hydroxy Diclofenac, [2-(2,6-Dichloro-4-hydroxy-phenylamino)phenyl]acetic acid
CAS Registry Number	64118-84-9
Chemical formula	C14H11Cl2NO3
Status	Incomplete (ref)
Source	
Exact mass	311.0116
Molare mass	312.15
Nominal mass	311

Fragmentmassen

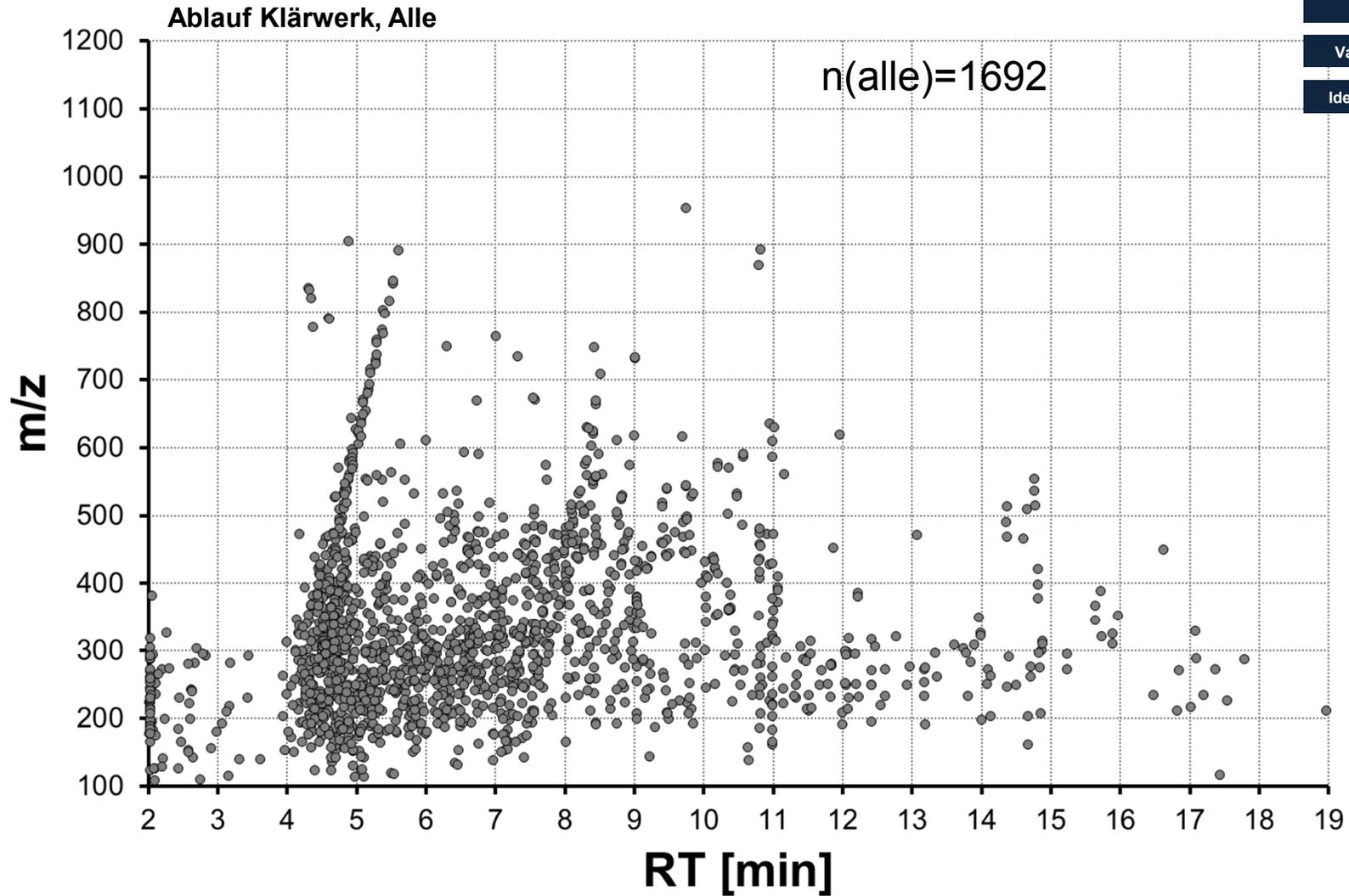
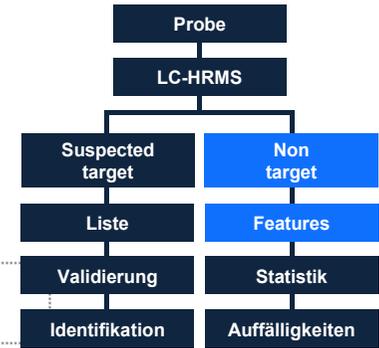
Property	Value	Fragments
Precursor Mass	311.0116	230.0367
Ion	+H	231.0444
		266.0132



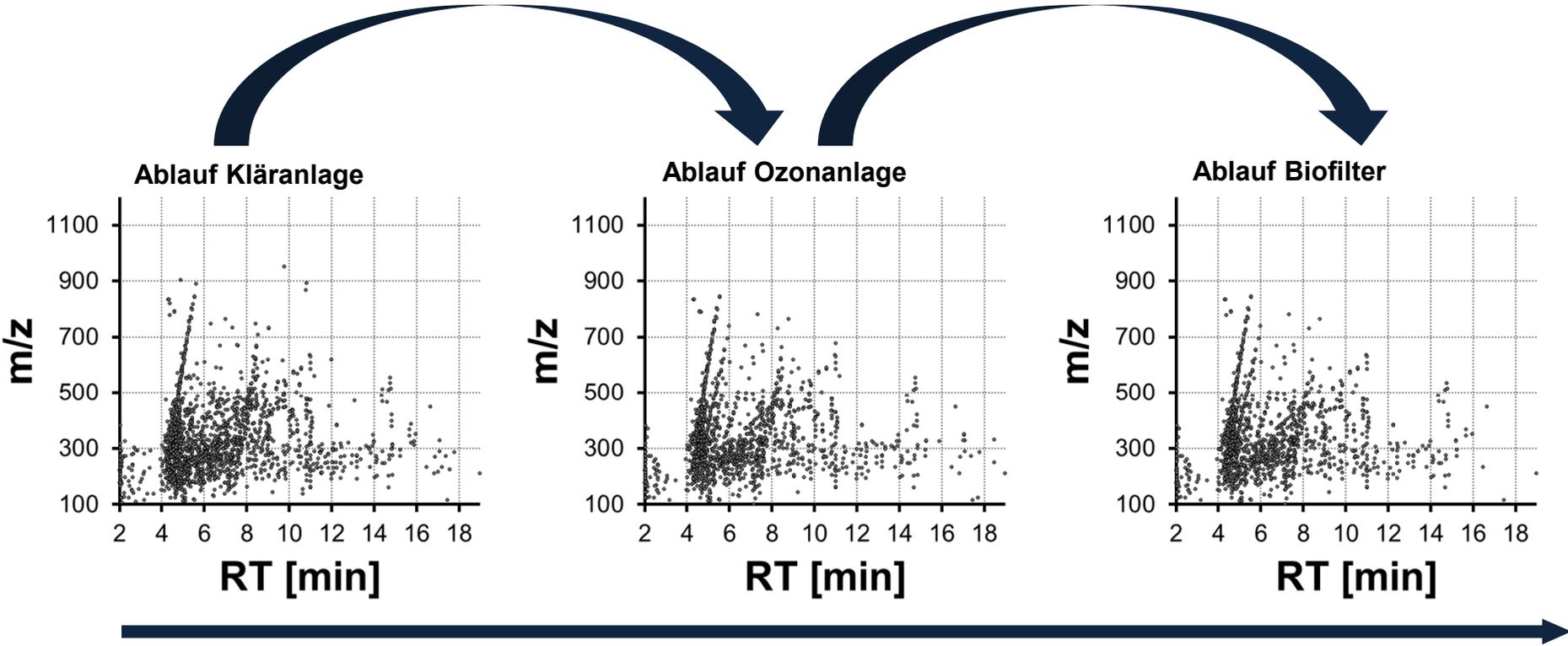
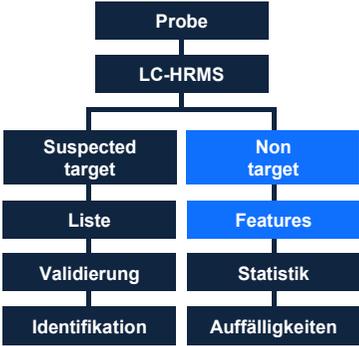
Konzentrationsverläufe für Diclofenac und 4-Hydroxydiclofenac nach Quantifizierung



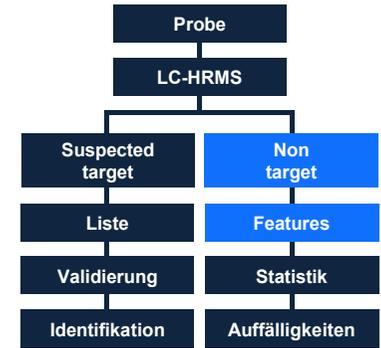
Prozessbeschreibung/Prozessvergleich



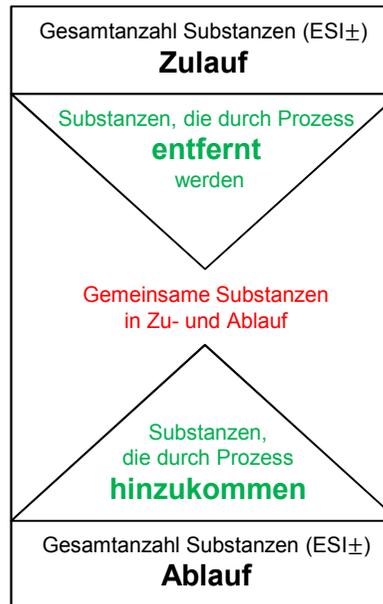
Prozessbeschreibung/Prozessvergleich

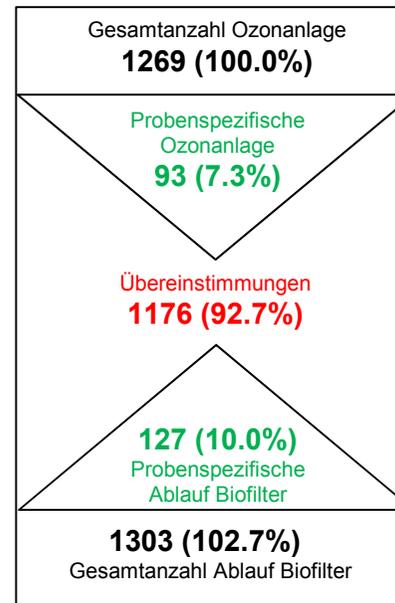
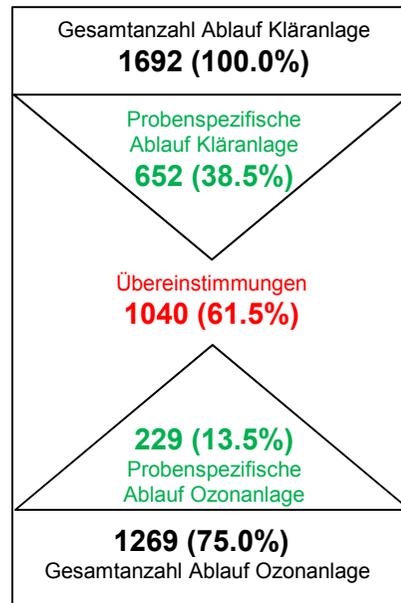
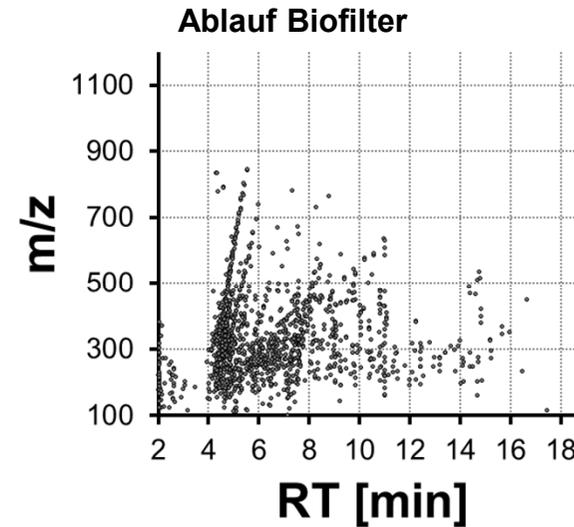
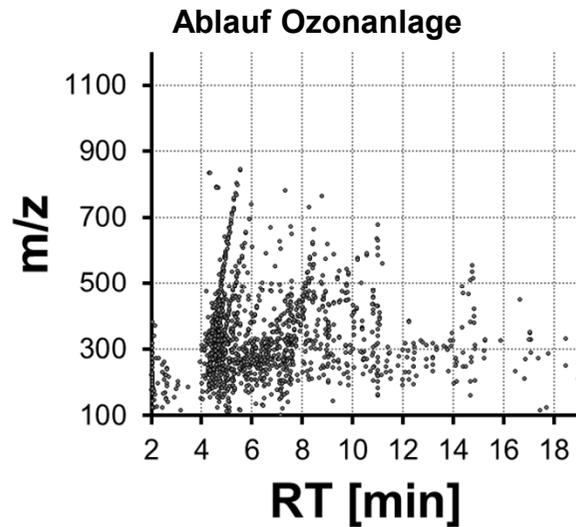
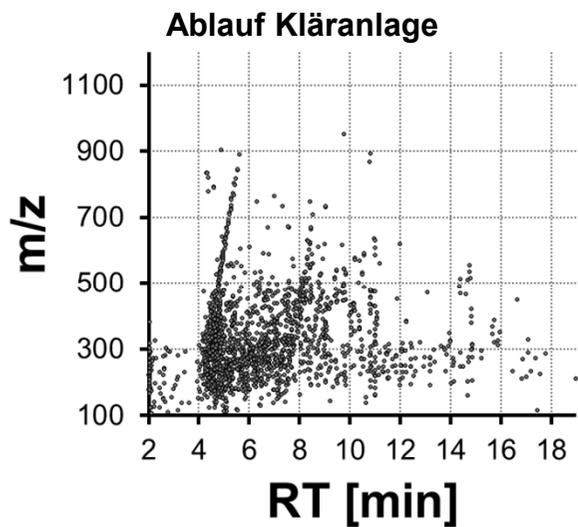


Prozessbeschreibung/Prozessvergleich Symbolik

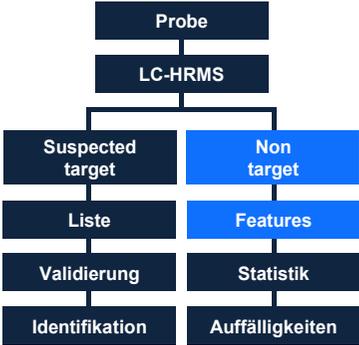


allg.
Beschreibung



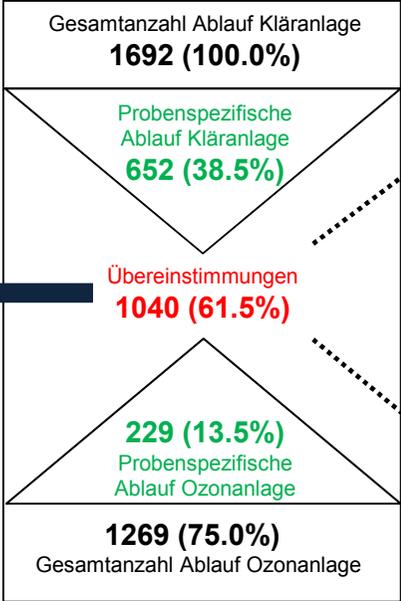


Prozessbeschreibung/Prozessvergleich inkl. Intensitätsbetrachtung



Problem:

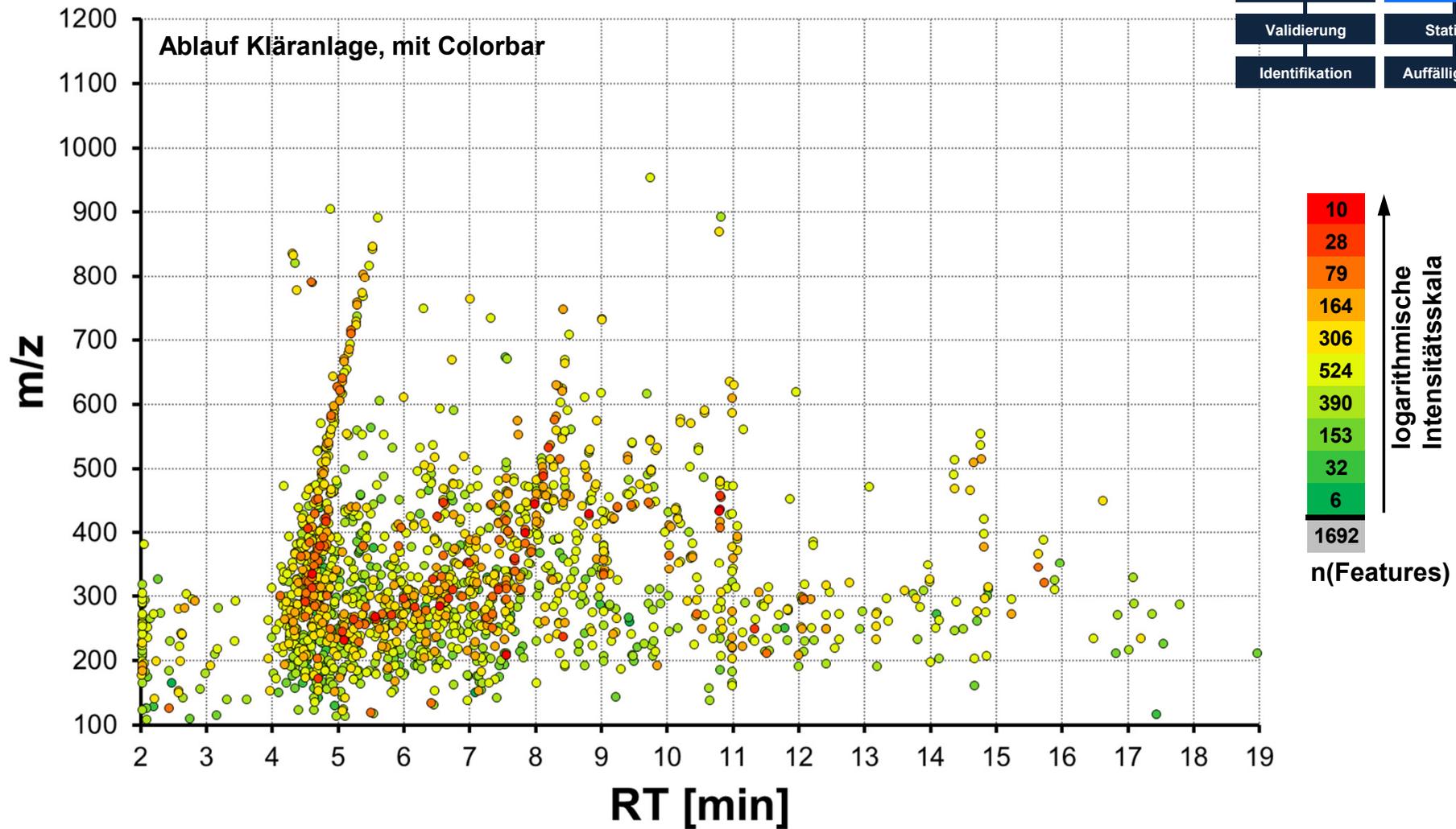
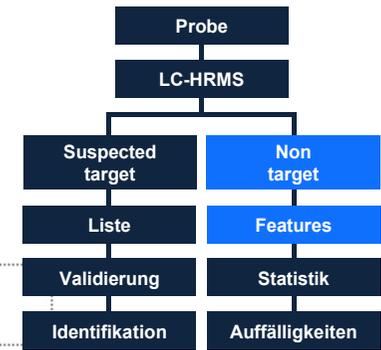
- Reduktion
- Erhöhung werden nicht erfasst



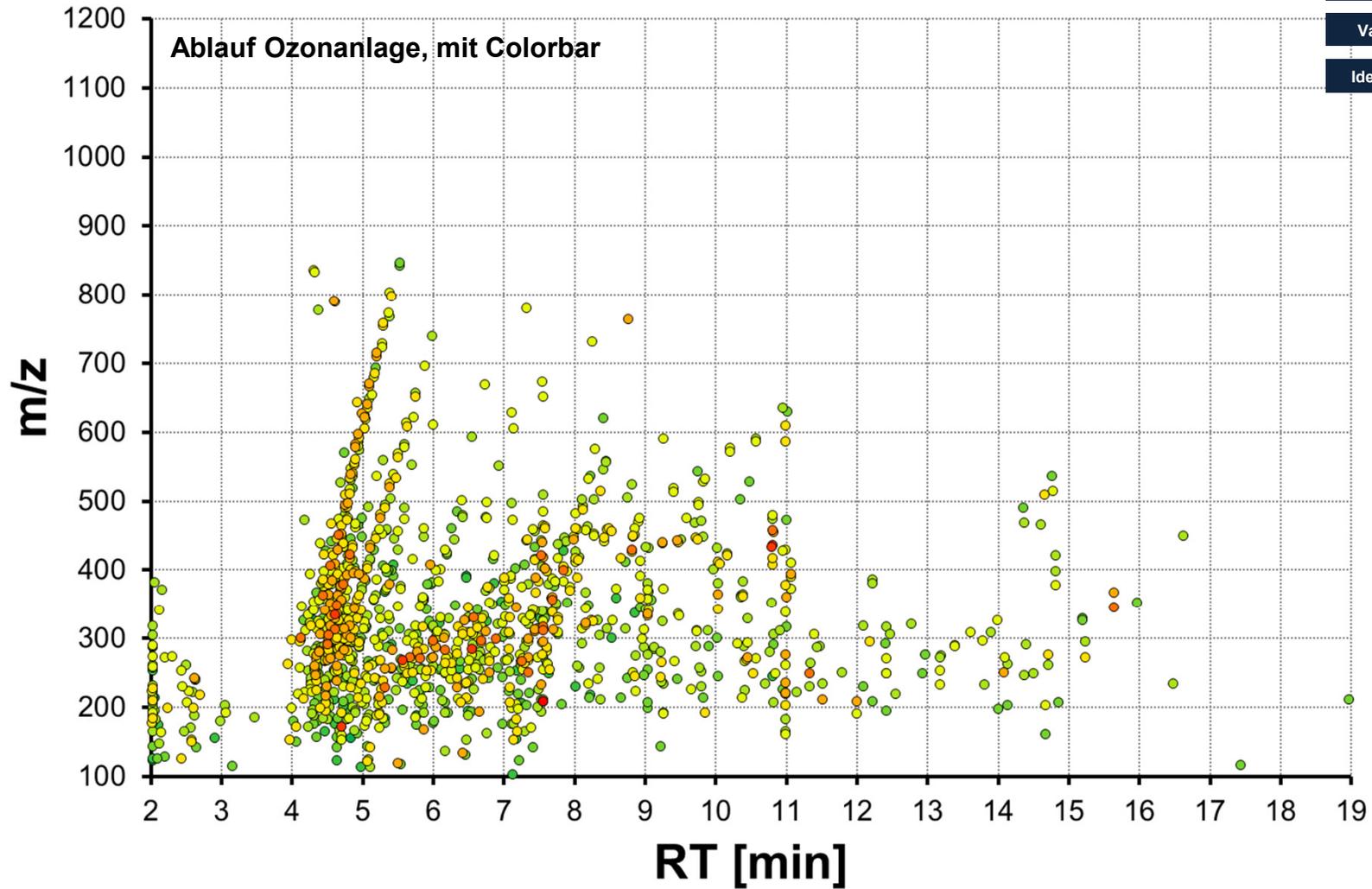
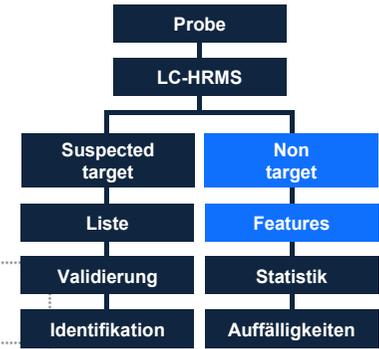
Zusätzliches Einbeziehen der Intensität!



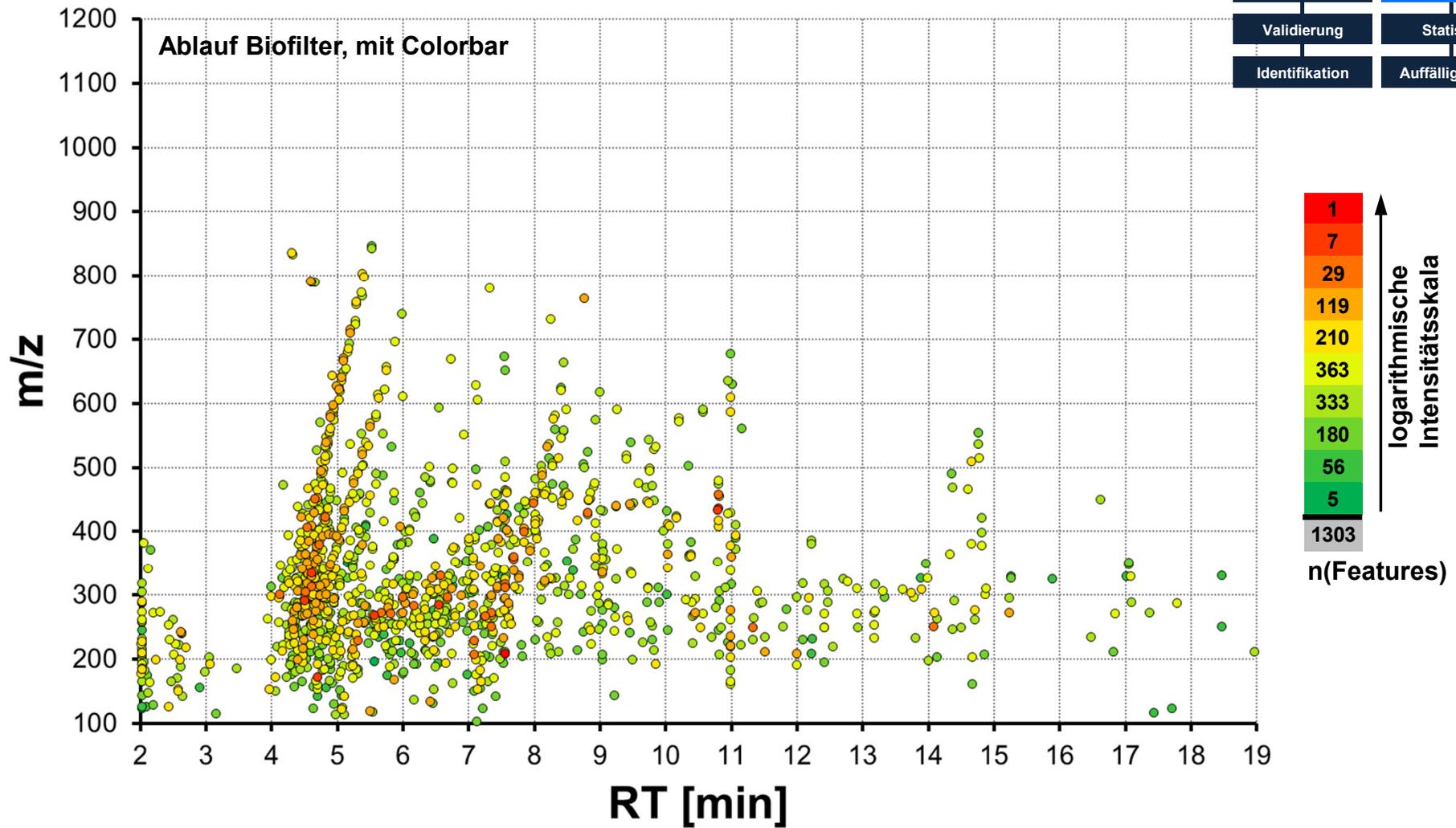
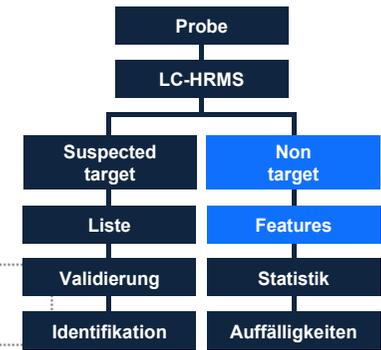
Prozessbeschreibung/Prozessvergleich inkl. Intensitätsbetrachtung



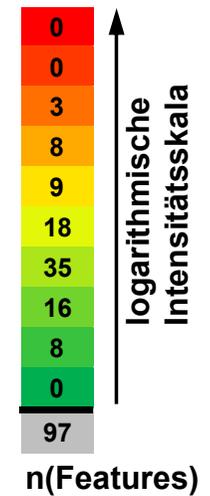
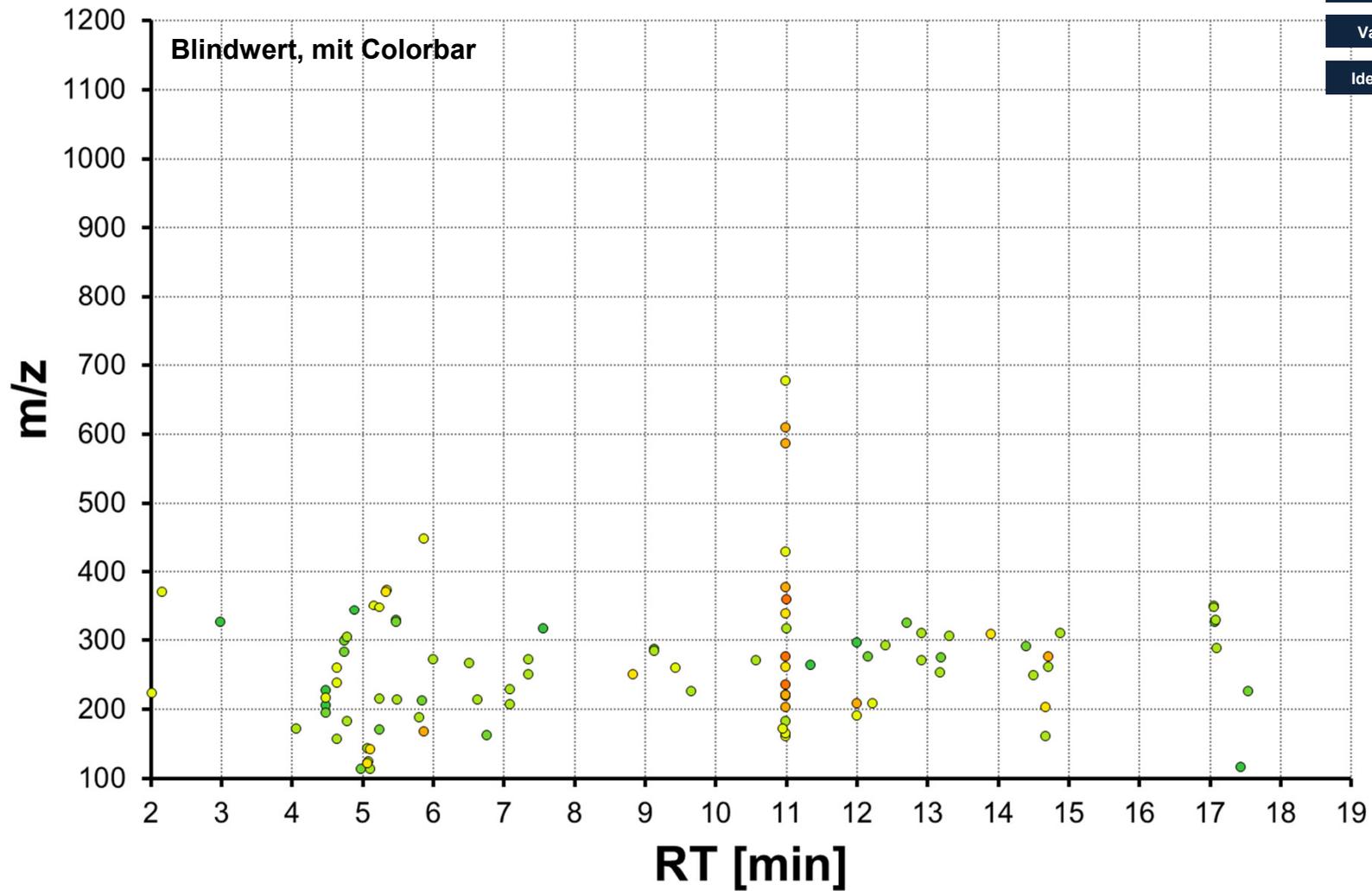
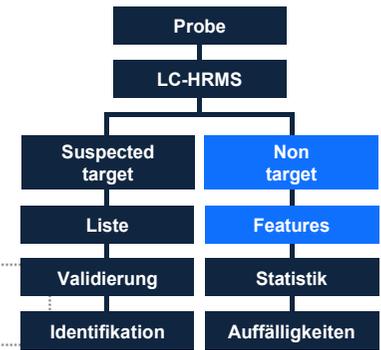
Prozessbeschreibung/Prozessvergleich inkl. Intensitätsbetrachtung



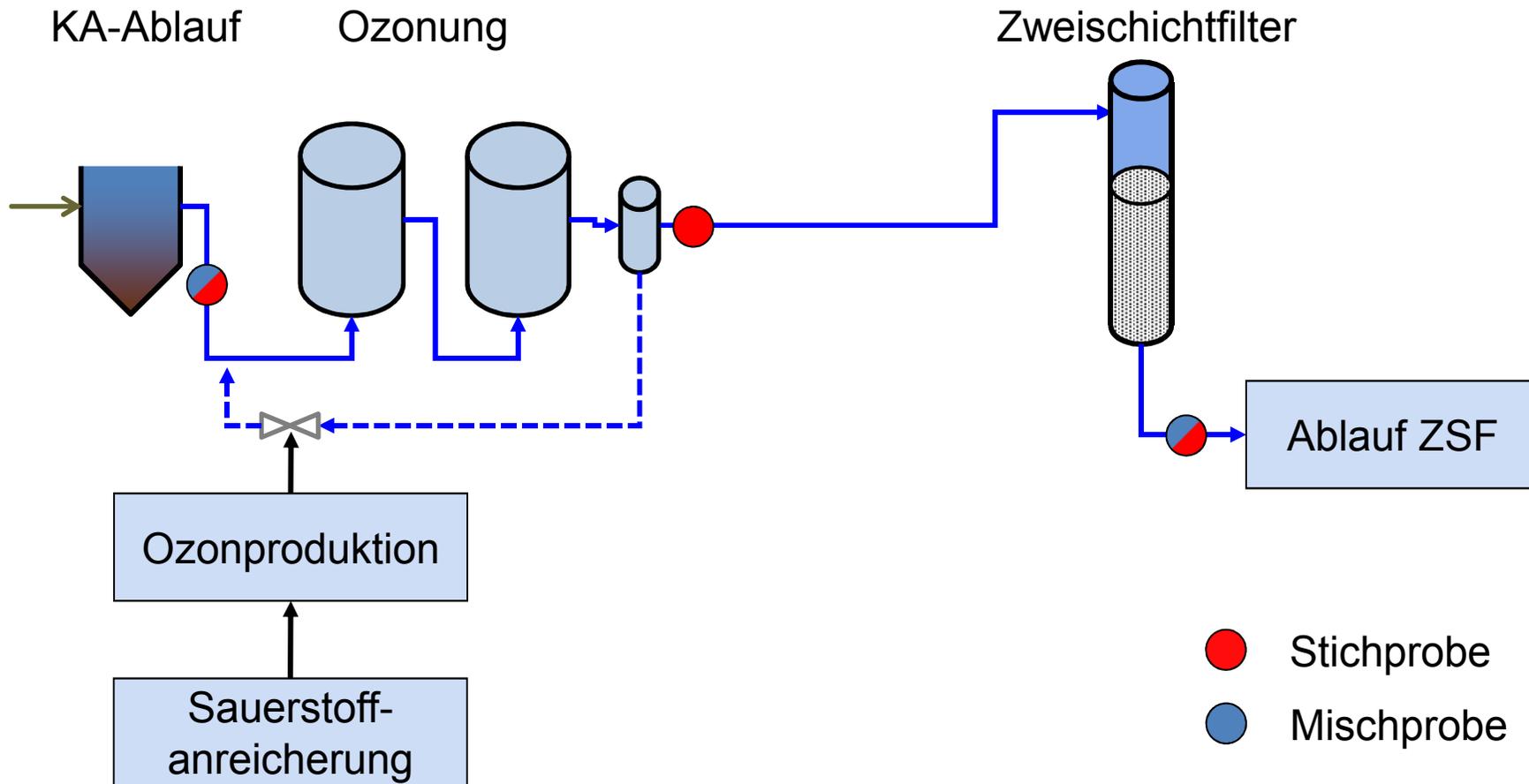
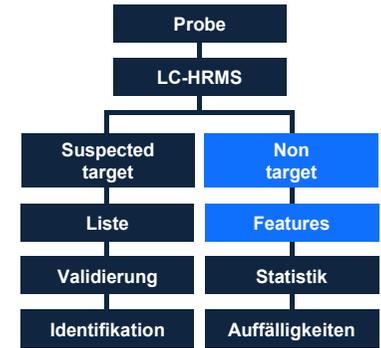
Prozessbeschreibung/Prozessvergleich inkl. Intensitätsbetrachtung



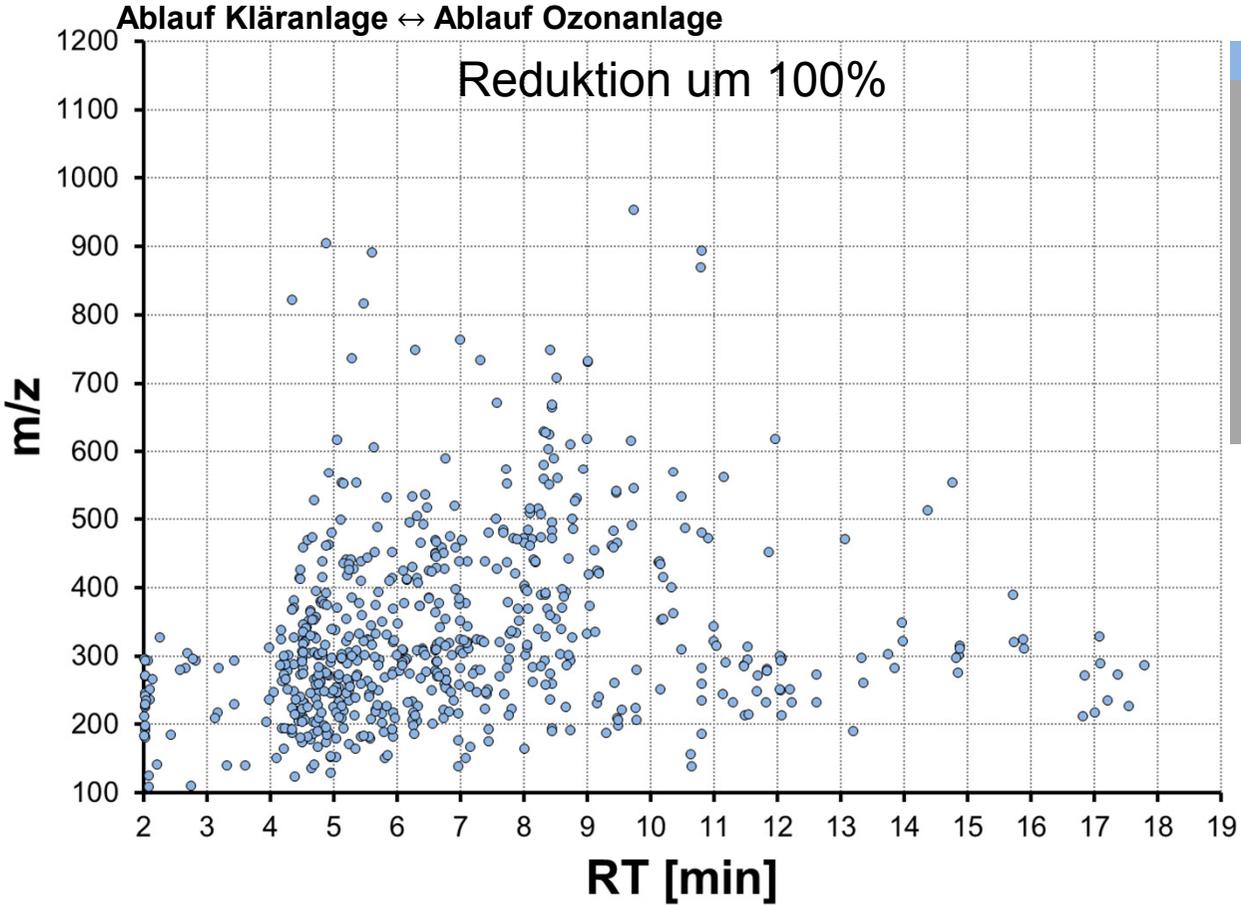
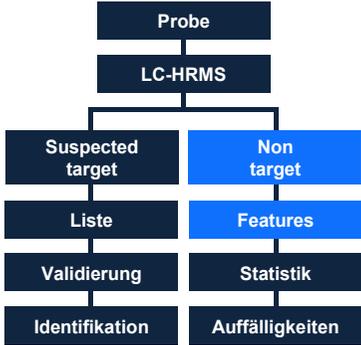
Prozessbeschreibung/Prozessvergleich inkl. Intensitätsbetrachtung



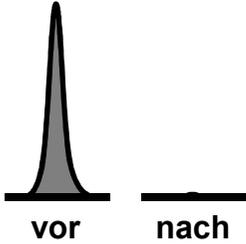
Aufbau Pilotanlage



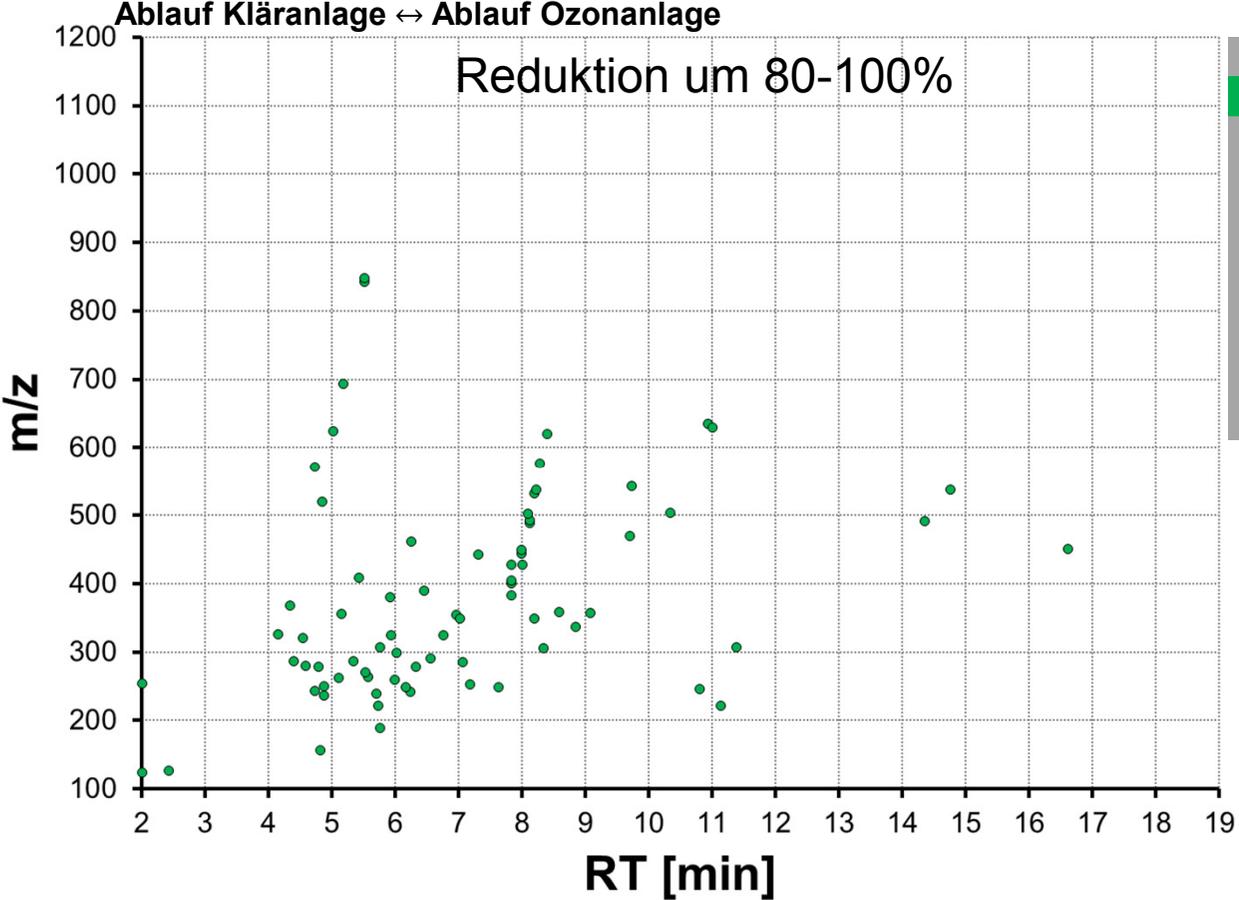
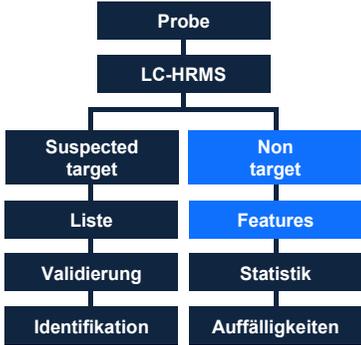
Prozessvergleich



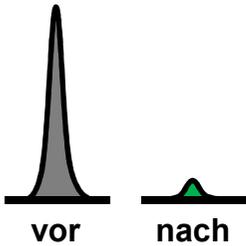
n(Features)



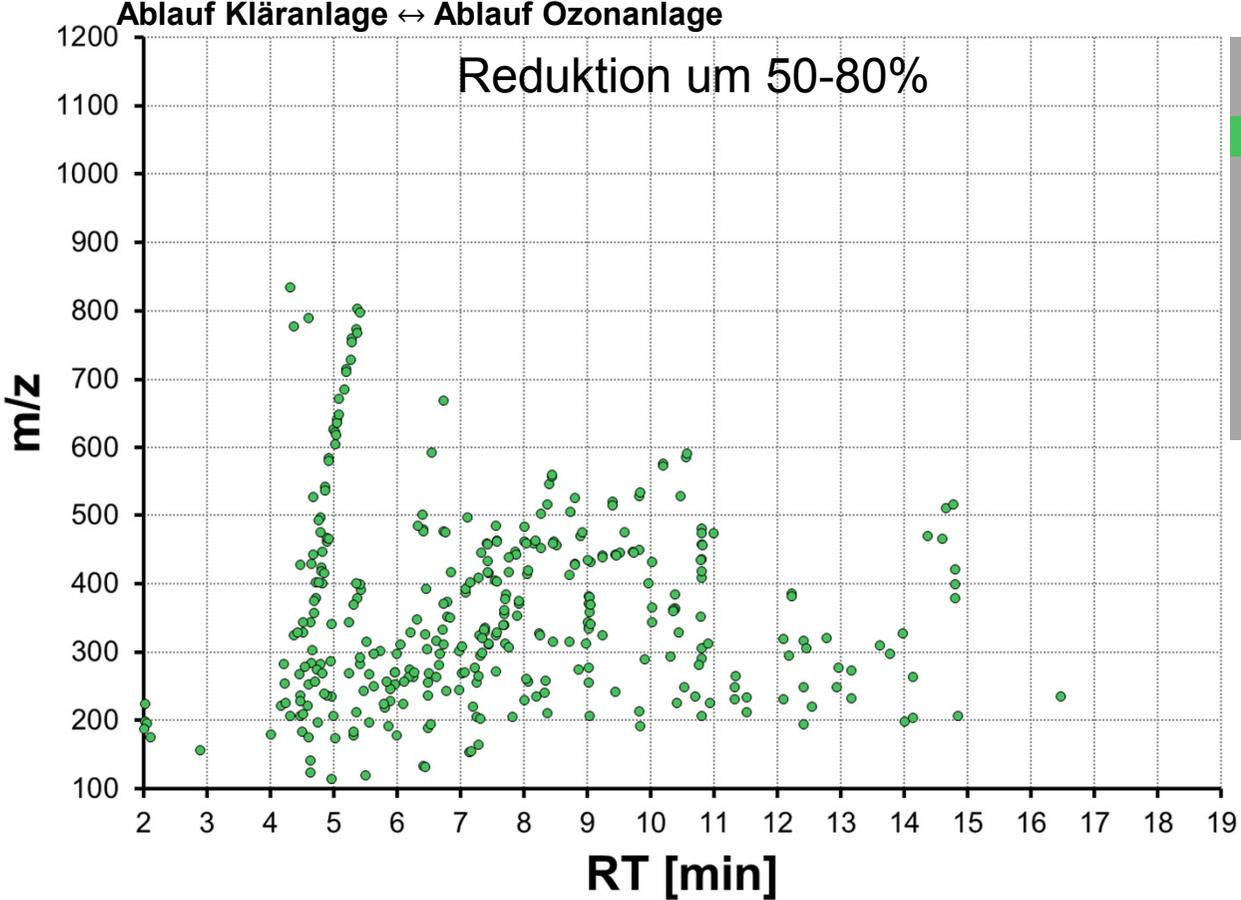
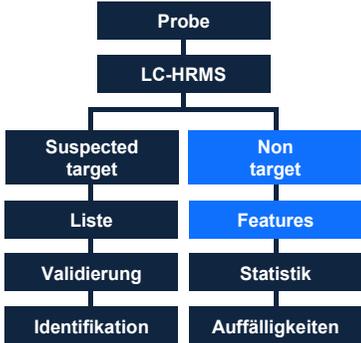
Prozessvergleich



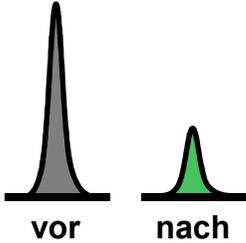
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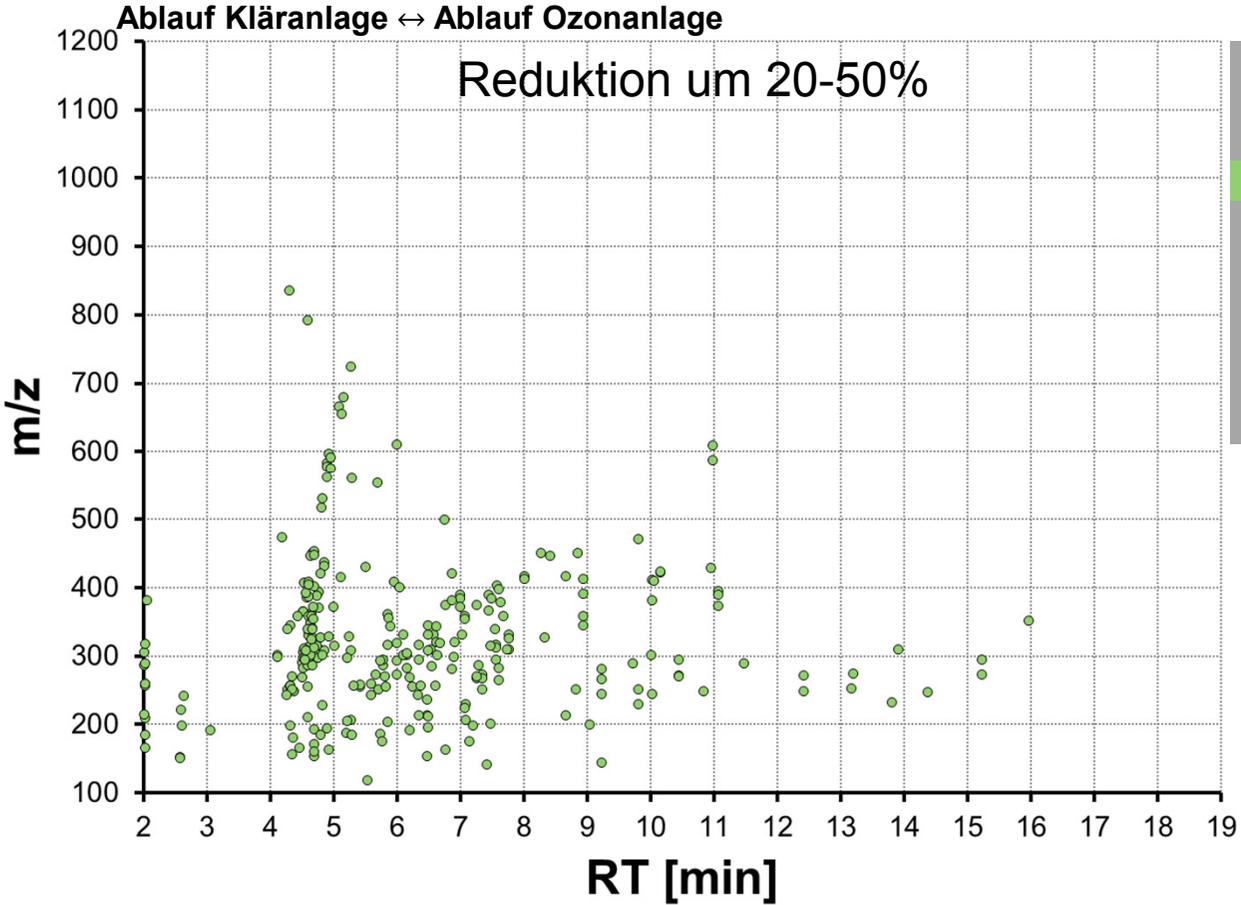
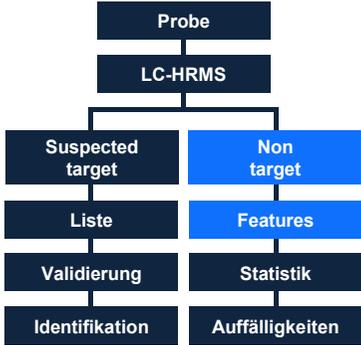
Prozessvergleich



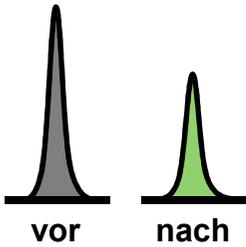
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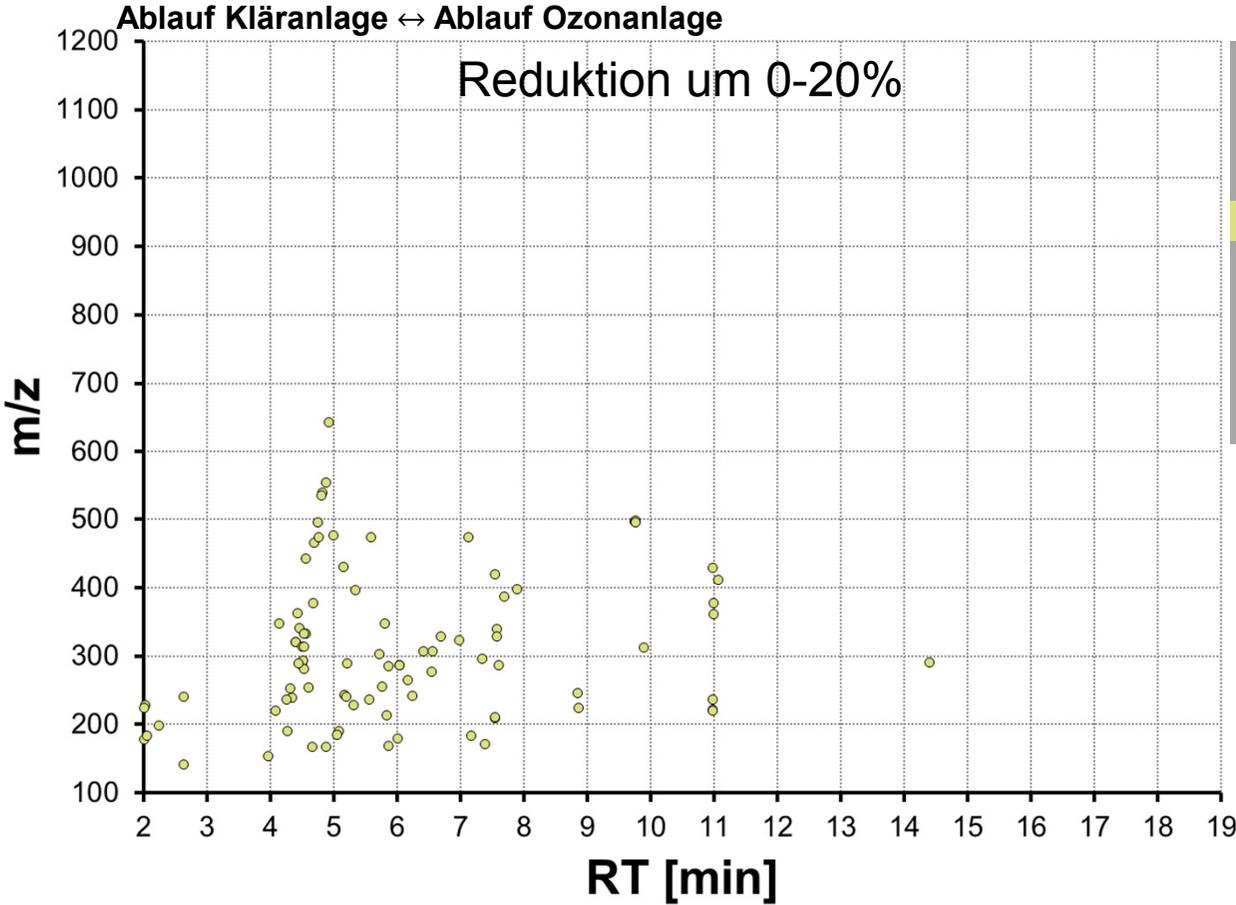
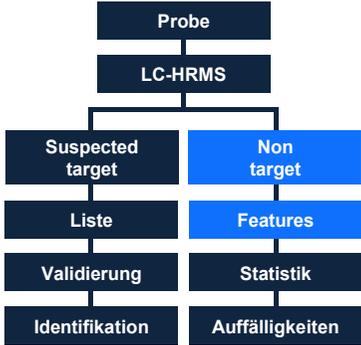
Prozessvergleich



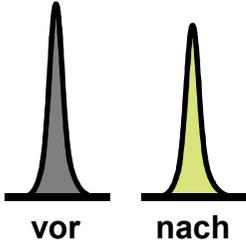
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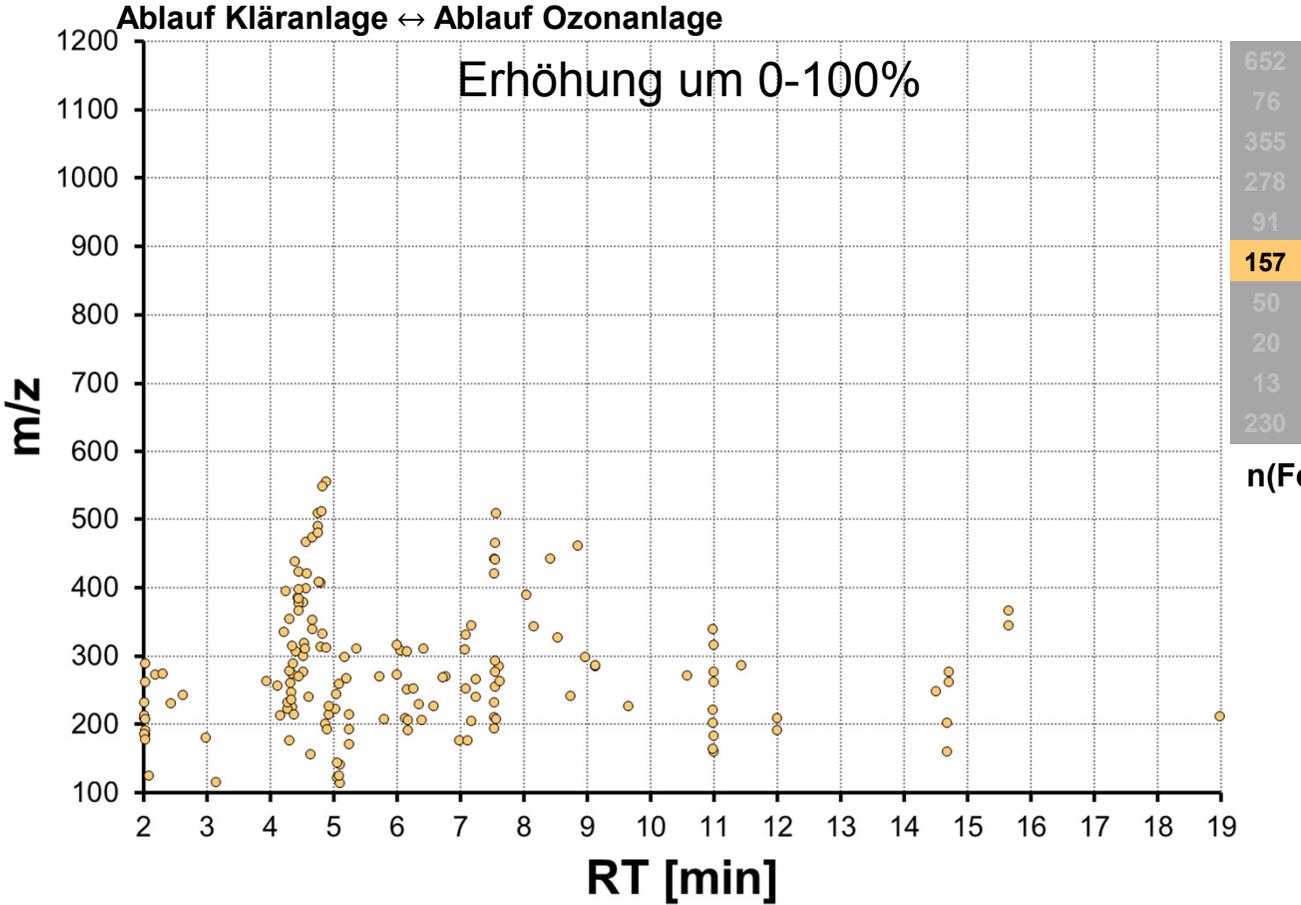
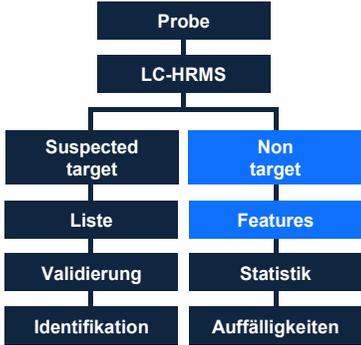
Prozessvergleich



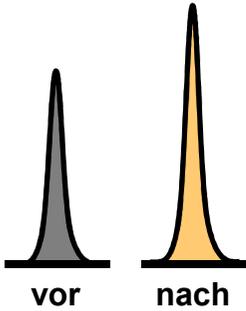
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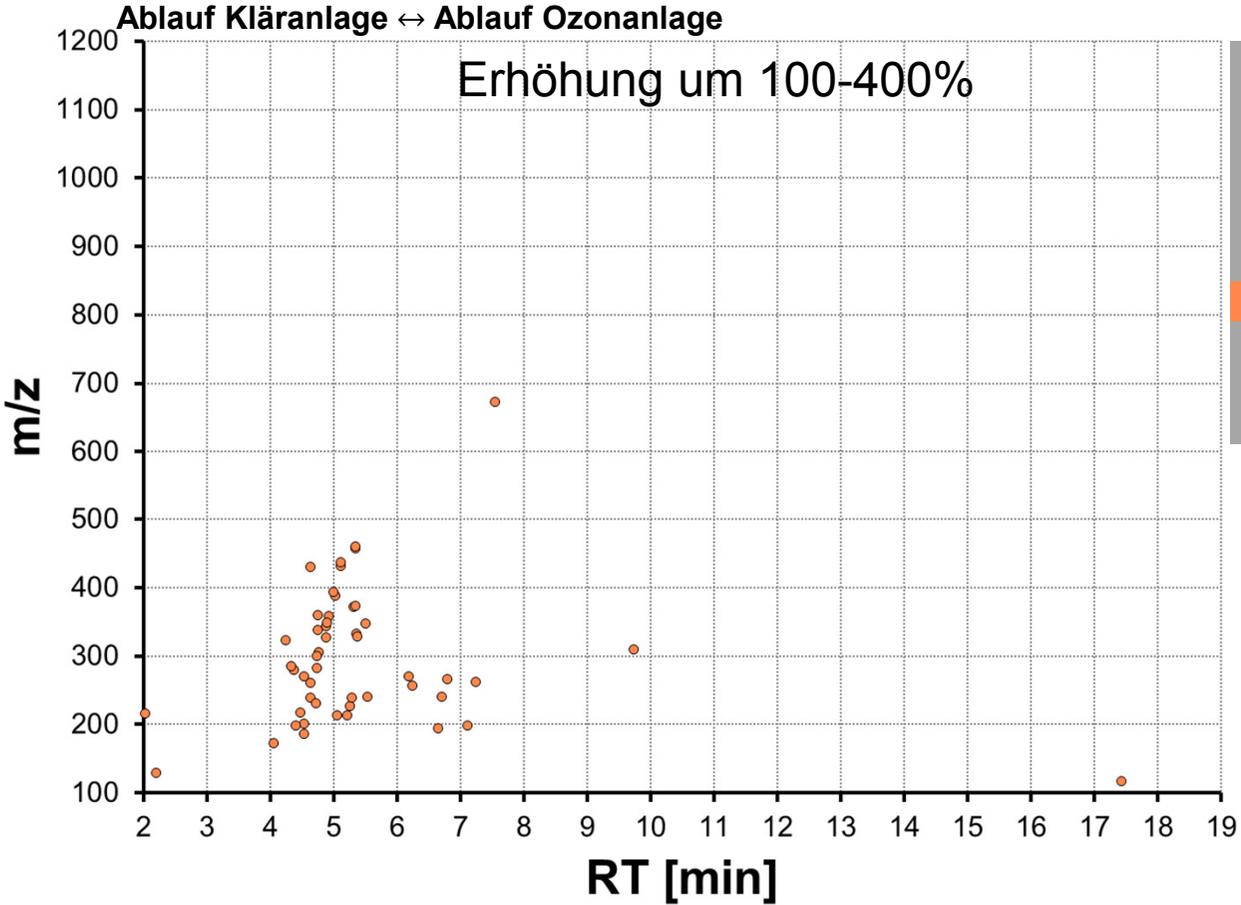
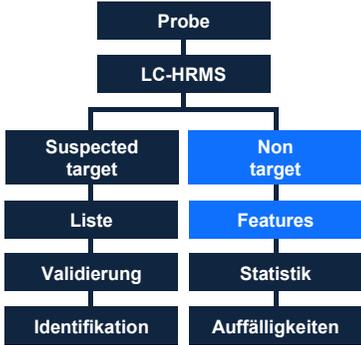
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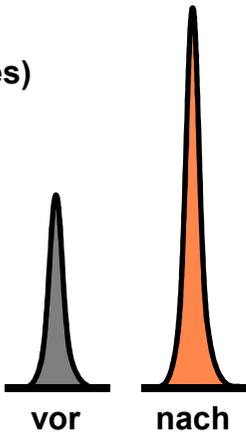
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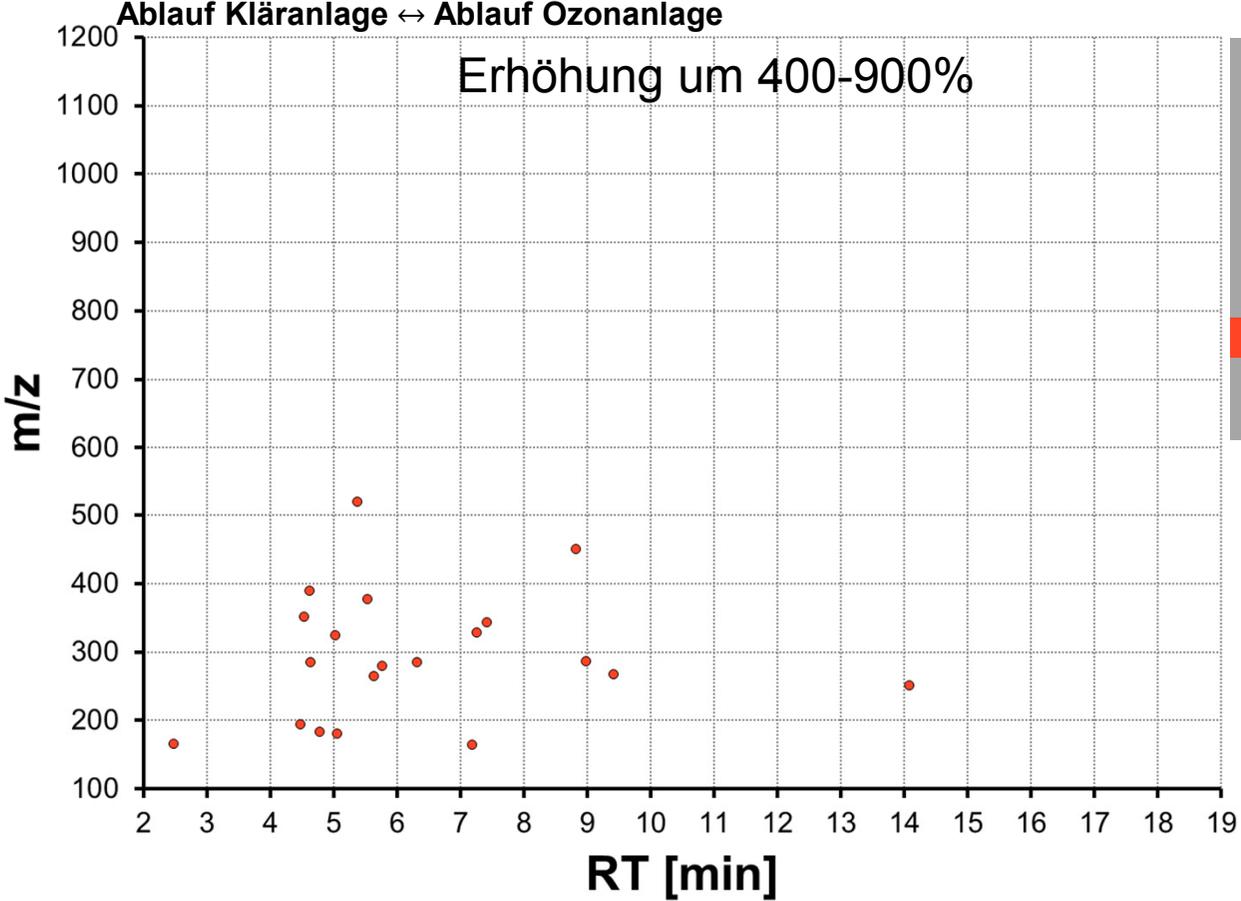
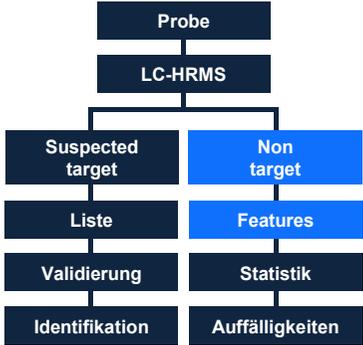
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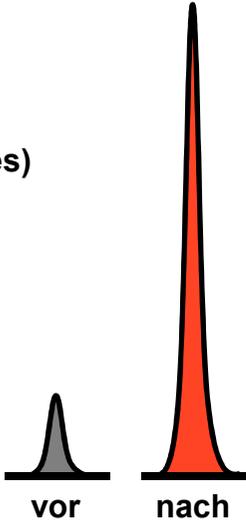
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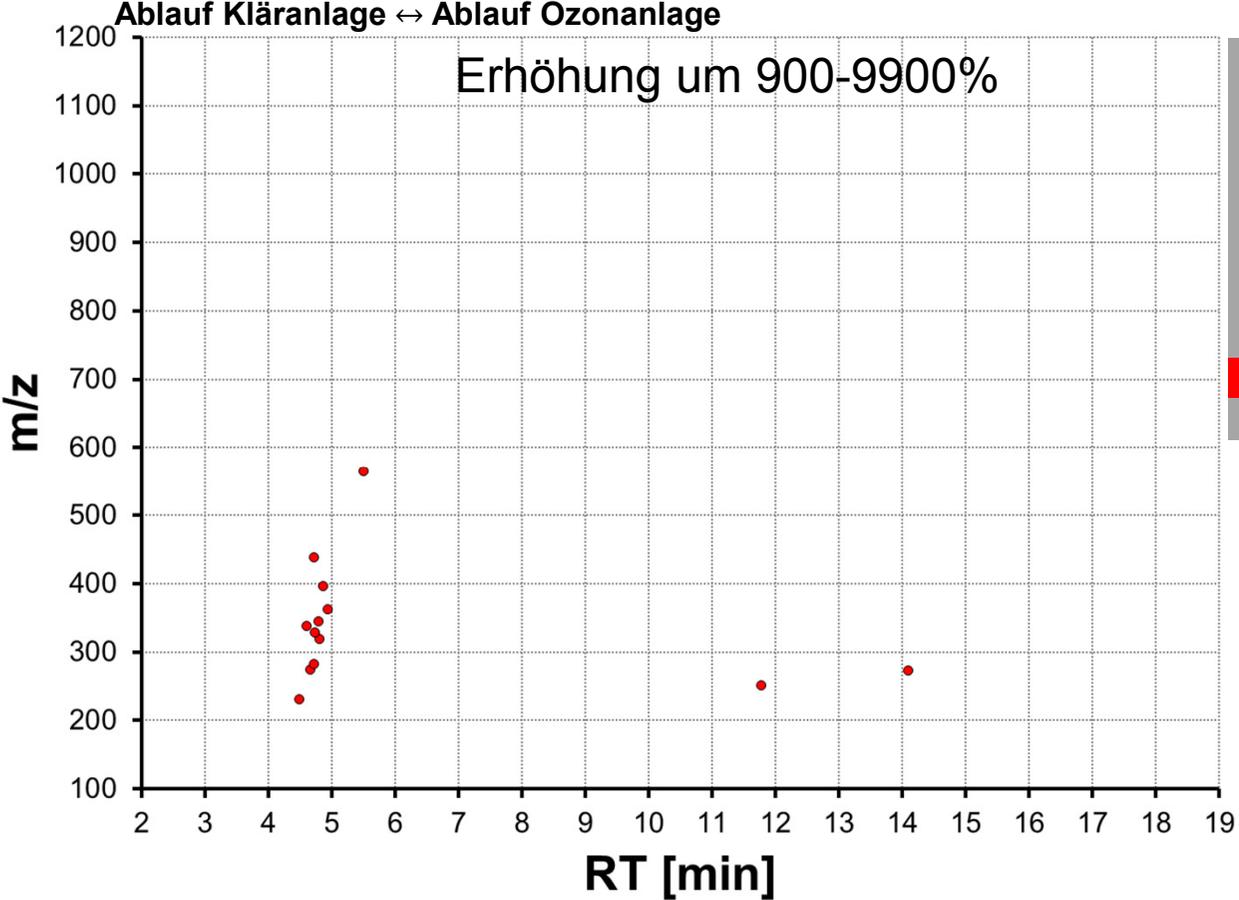
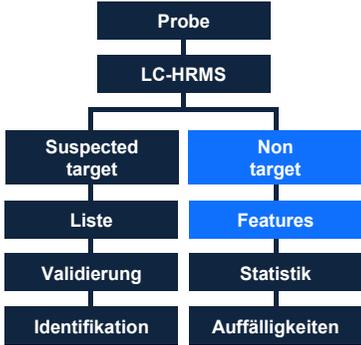
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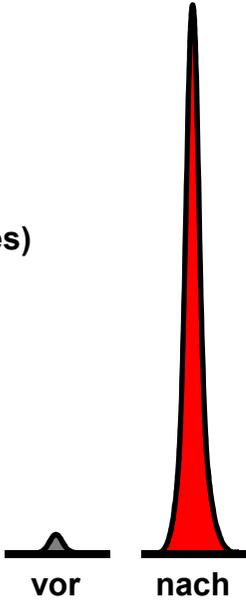
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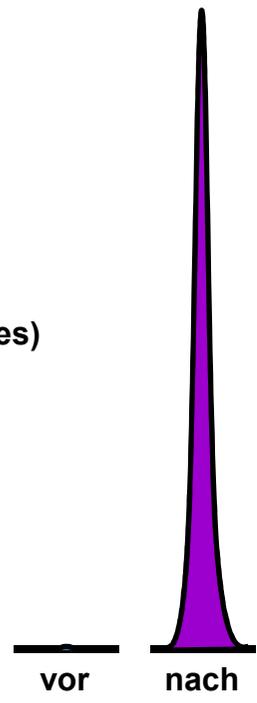
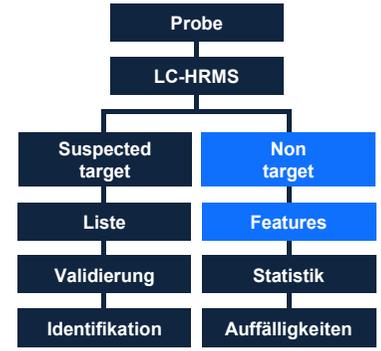
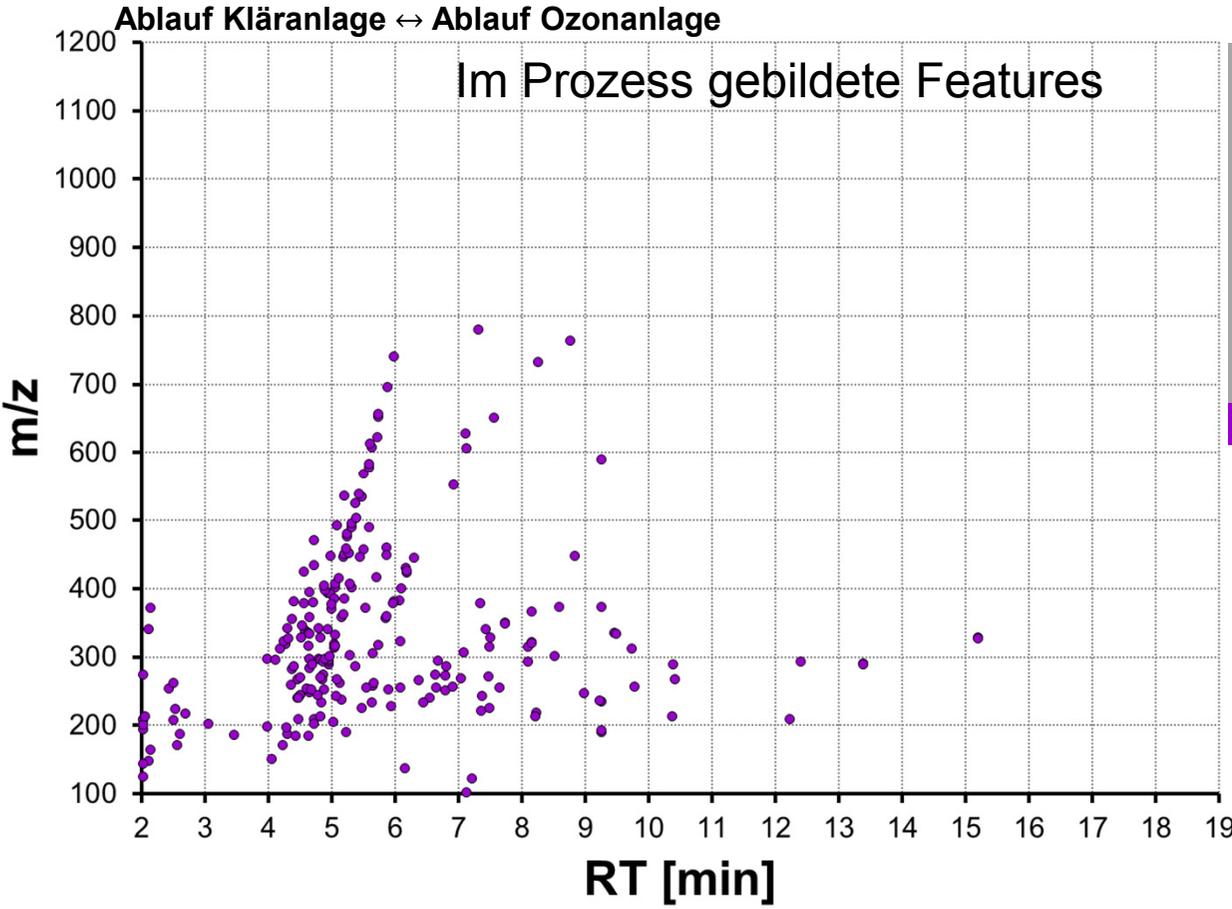
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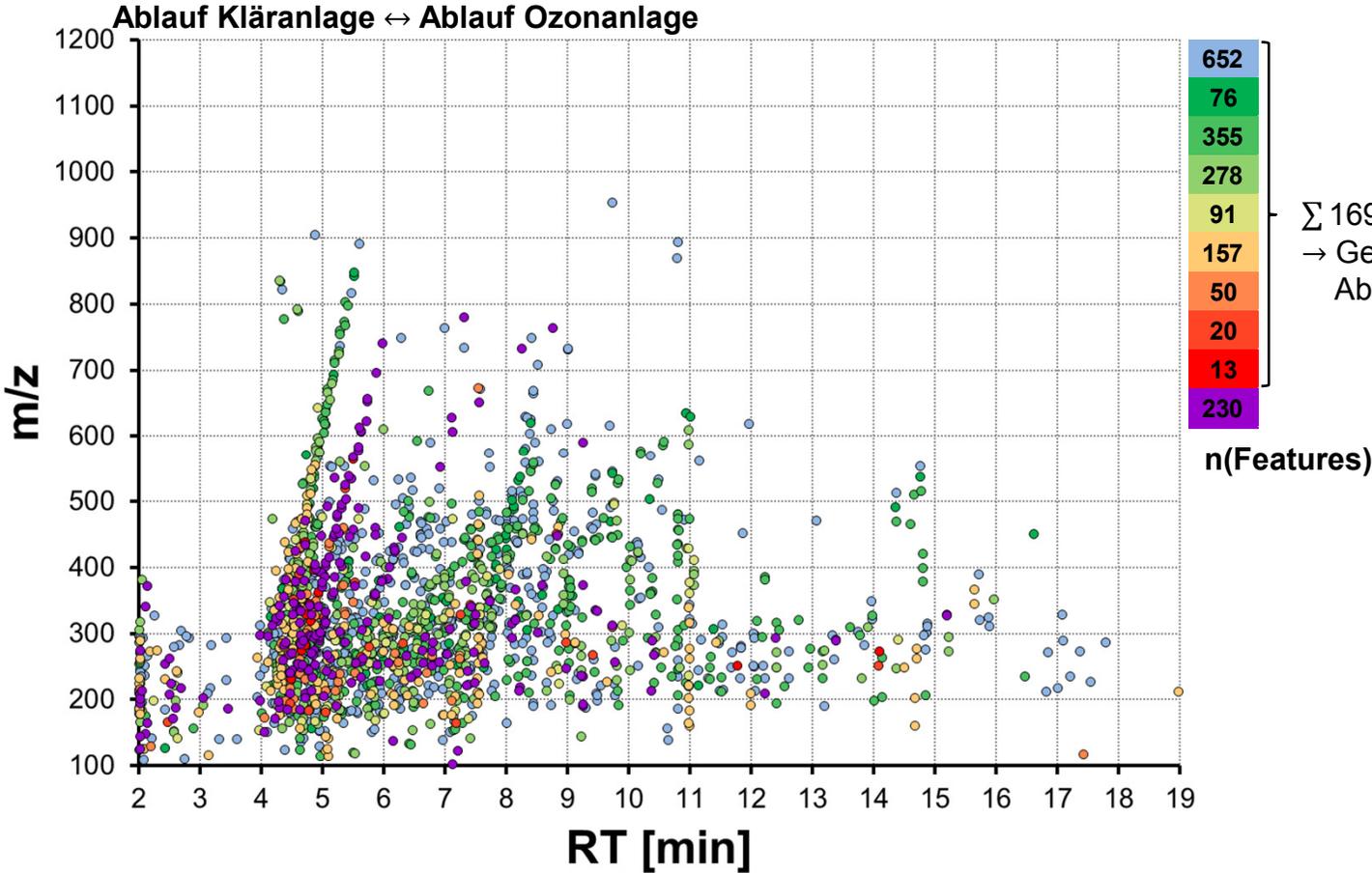
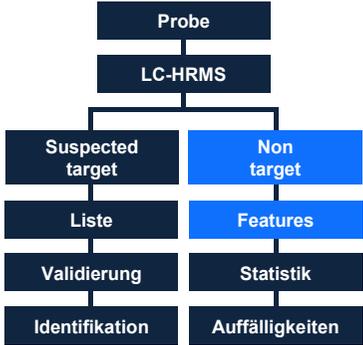
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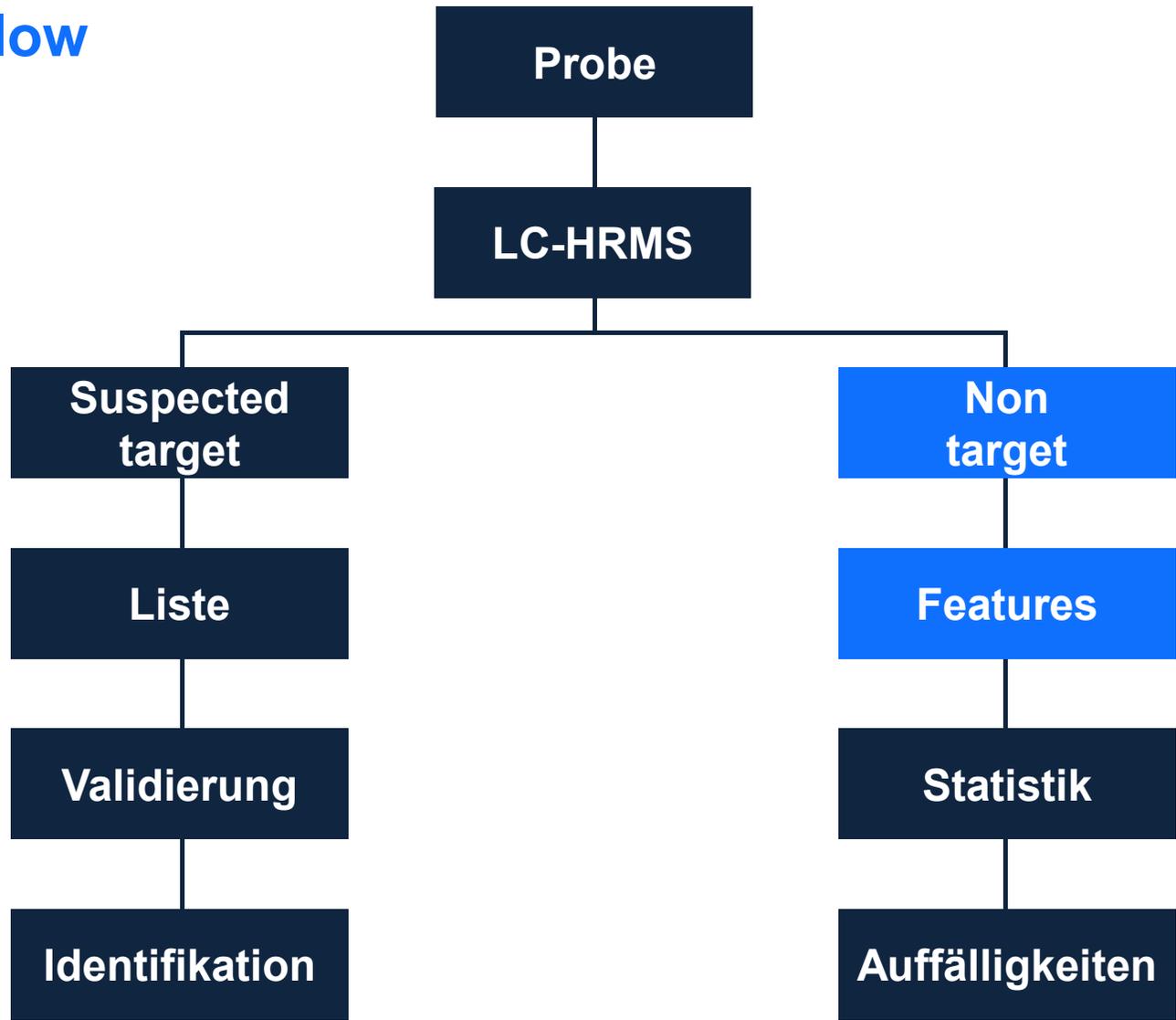
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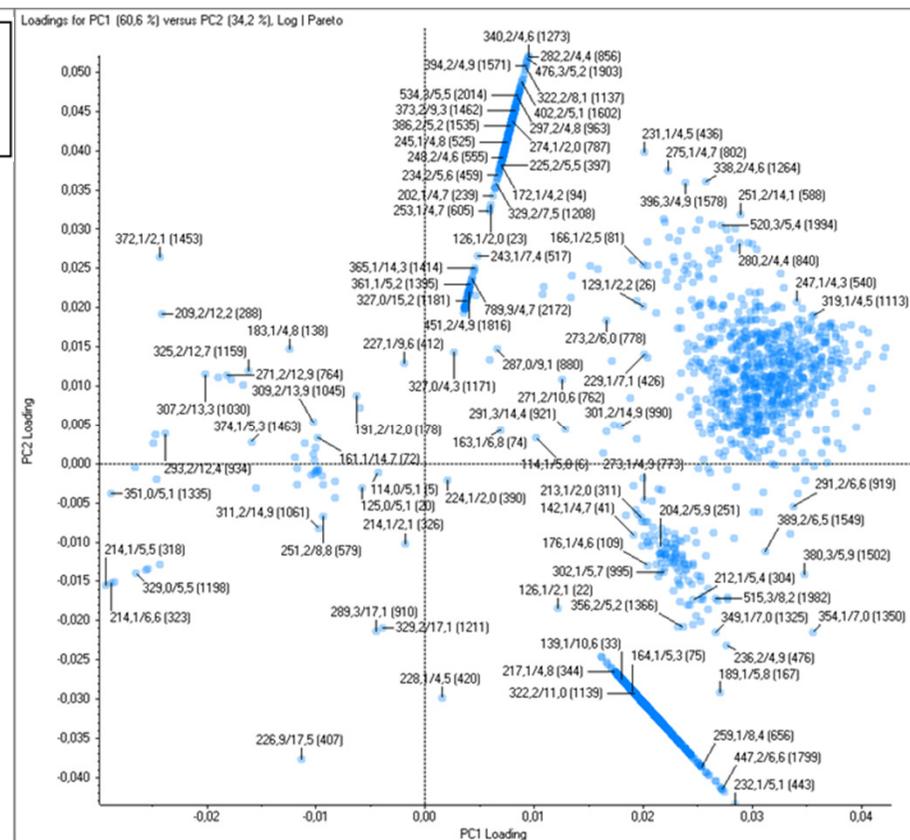
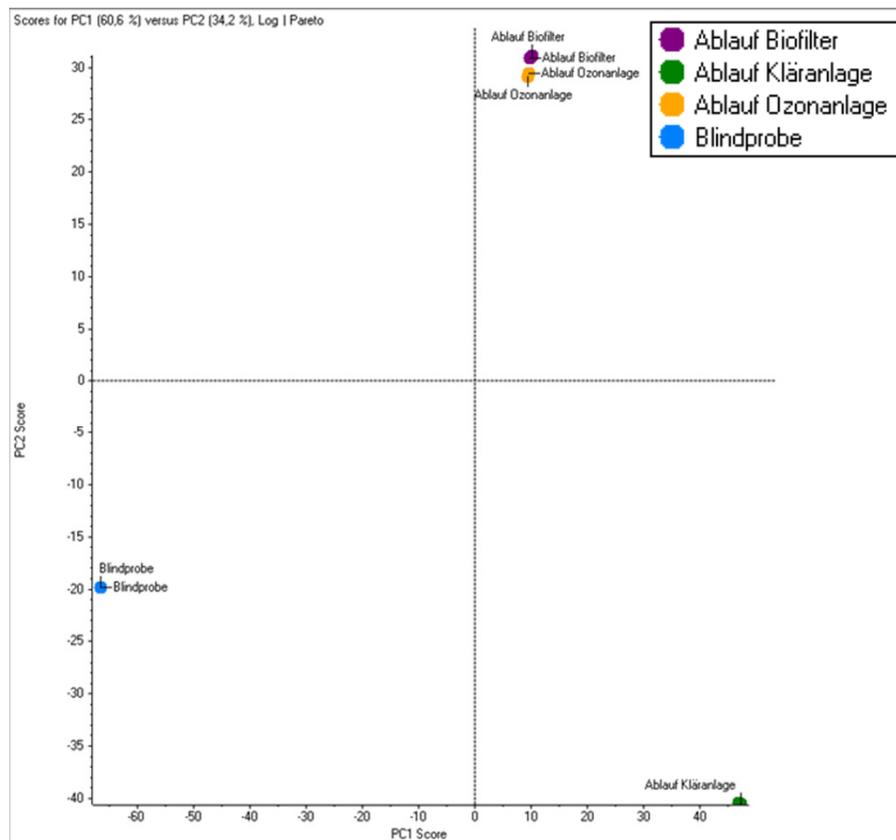
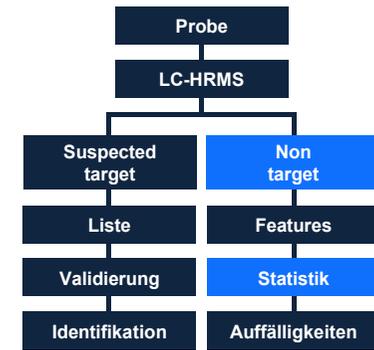
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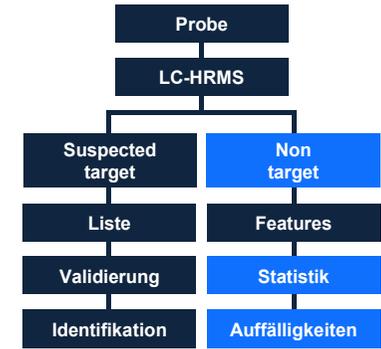
Workflow



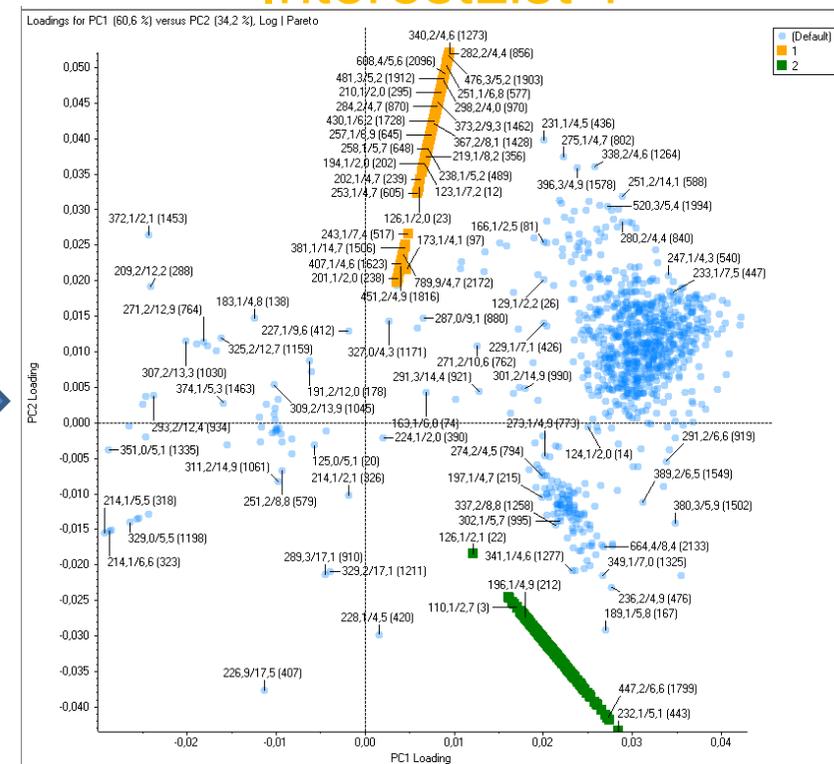
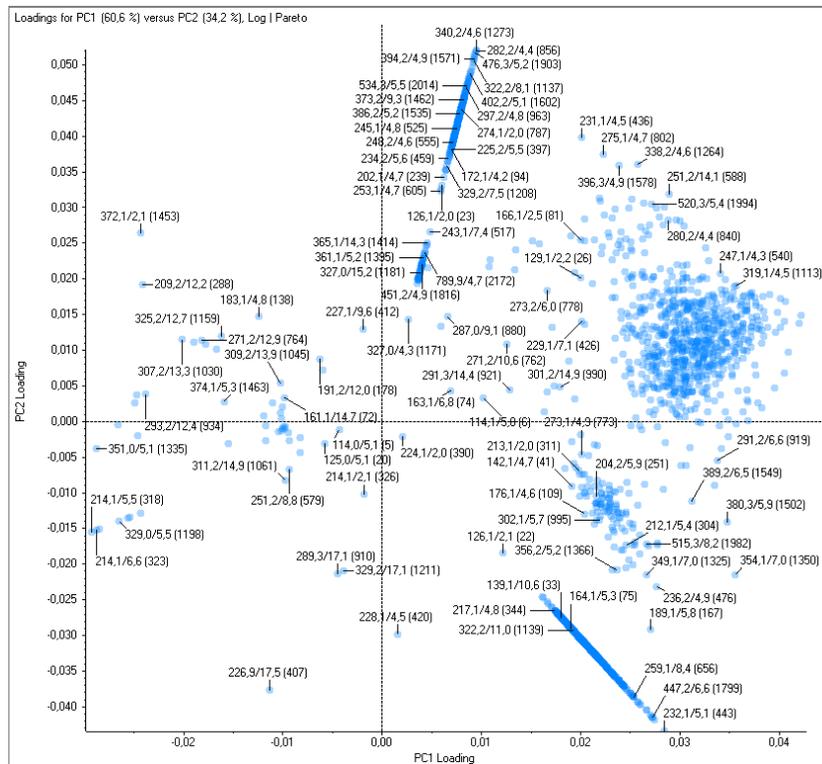
PCA Score-Plot PC1 vs. PC2 und Loading-Plot



PCA Variable Grouping

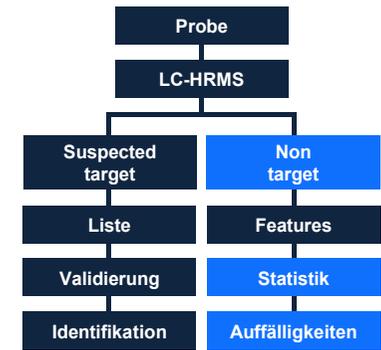


InterestList 1



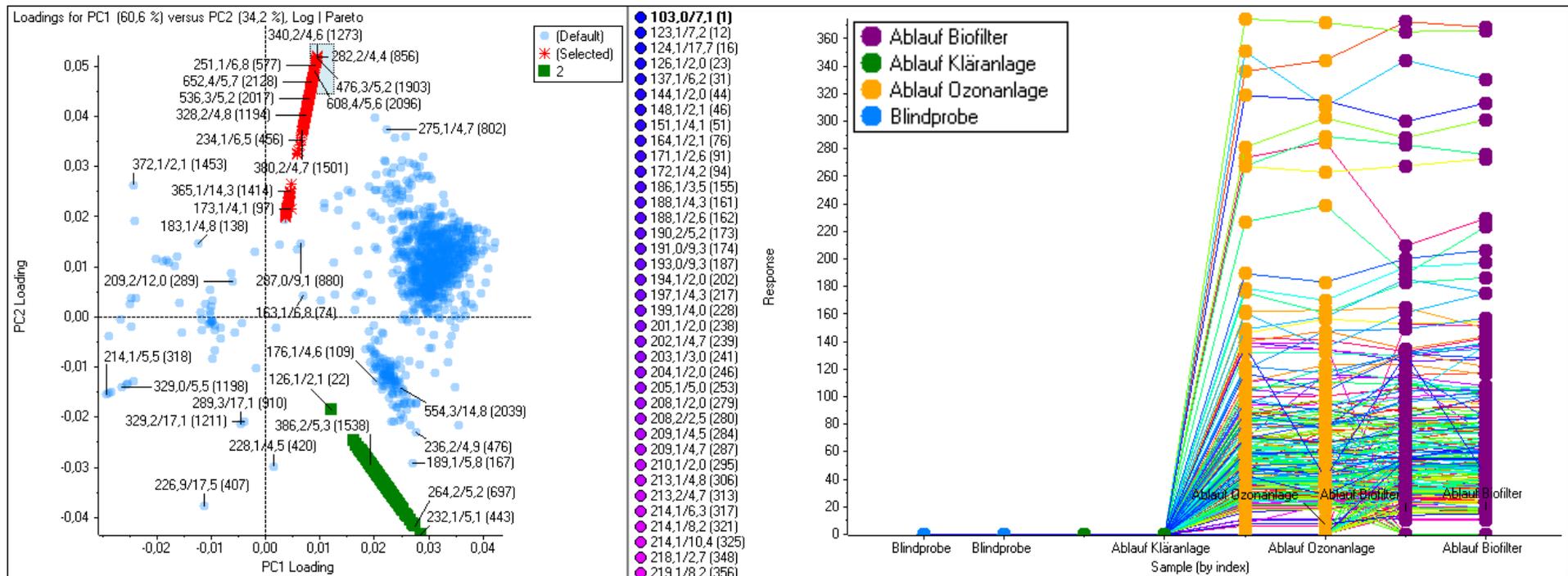
InterestList 2

Profile Plot aller Features von InterestList 1

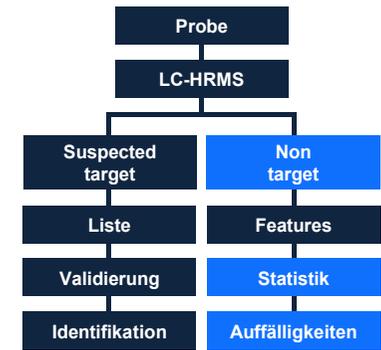


NUR in Ablauf Ozonanlage und/oder Biofilter vorhandene Features

InterestList 1

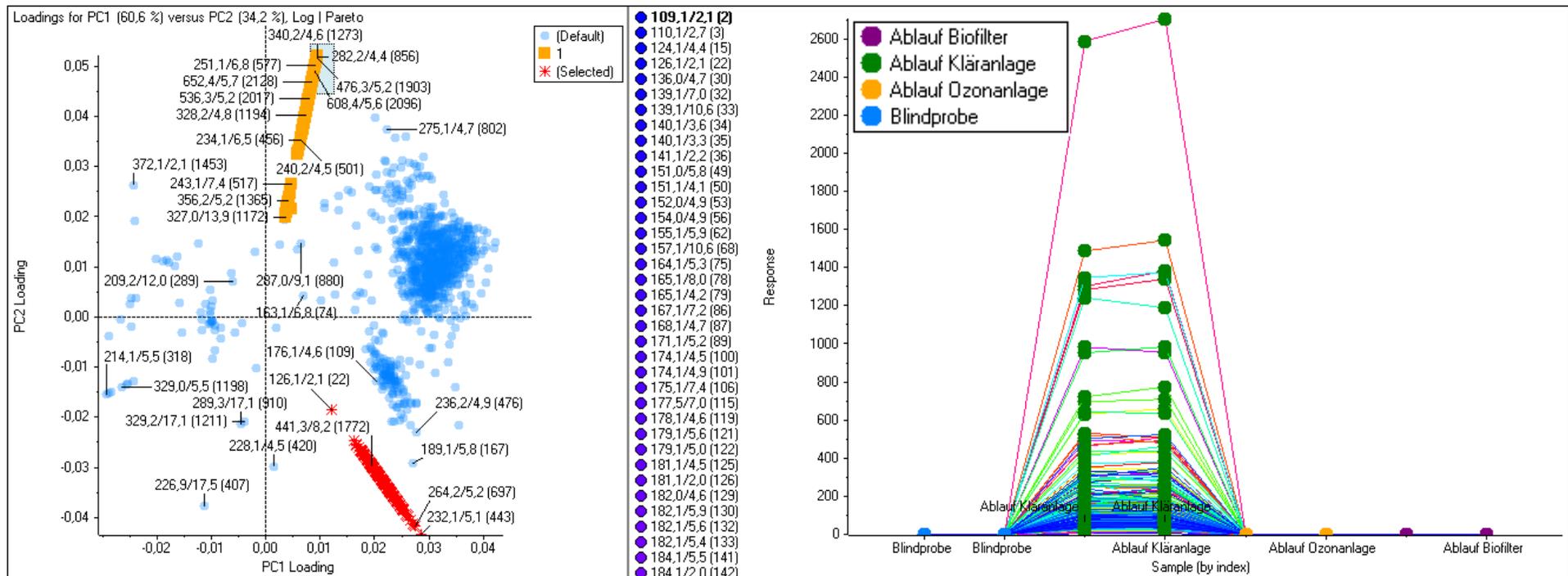


Profile Plot aller Features von InterestList 2



NUR in Ablauf Kläranlage vorhandene Features

InterestList 2



Metabolite Pilot



Potential Metabolites: 93 of 93 Peaks

Peak ID	Name	Formula	m/z	ppm	R.T. (min)	Peak Area	% Area	% Score
27	Parent	C16H25NO2	264.1953	-1.9	5.59	1.59E+03	1.0	75.0
28	M41 Di-Oxidation	C16H25NO4	296.1854		-0.6	4.80	9.58E+02	0.6
29	M76 Oxidation	C16H25NO3	280.1911		1.4	5.76	3.55E+03	2.2
30	M2 Loss of 54.0628	C8H19NO5	210.1330		-2.8	2.03	1.28E+03	0.8
31	M23 Loss of 8.0203	C11H21N5O2	256.1755		-5.0	4.62	6.39E+02	0.4

Details

Score: 75.0%

Property	Assigned Score	Maximum Score
Mass defect	40.0	40
Isotope pattern	40.0	40
MS/MS*	0.0	40
Mass accuracy	40.0	40
Total confirmation score	120.0	160

*MS/MS:
Quality: 0.40
Similarity: 0.00

Source of MS/MS
Sample .wiff file

Chromatograms

MS Sample, XIC from 264.1833 to 264.2083

Ausgangsverbindung
Tramadol

MS

+TOF MS at 5.59 min

MS/MS

+TOF MS/MS of 264.1953

Metabolite Pilot



Potential Metabolites: 93 of 93 Peaks

Peak ID	Name	Formula	m/z	ppm	R.T. (min)	Peak Area	% Area	% Score
27	Parent	C16H25NO2	264.1953		-1.9	5.59	1.59E+03	1.0
28	M41 Di-Oxidation	C16H25NO4	296.1854		-0.6	4.80	9.58E+02	0.6
29	M76 Oxidation	C16H25NO3	280.1911		1.4	5.76	3.55E+03	2.2
30	M2 Loss of 54.0628	C8H19NO5	210.1330		-2.8	2.03	1.28E+03	0.8
31	M23 Loss of 8.0203	C11H21NO2	256.1755		-5.0	4.62	6.39E+02	0.4

Details

Score: 75.0%

Property	Assigned Score	Maximum Score
Mass defect	40.0	40
Isotope pattern	40.0	40
MS/MS*	0.0	40
Mass accuracy	40.0	40
Total confirmation score	120.0	160

*MS/MS:
Quality: 0.82
Similarity: 0.00

Source of MS/MS
Sample .wiff file

Chromatograms

MS Sample, XIC from 280.1782 to 280.2032

Intensity, cps vs Time, min

oxidierte Form von Tramadol

MS

+TOF MS at 5.76 min

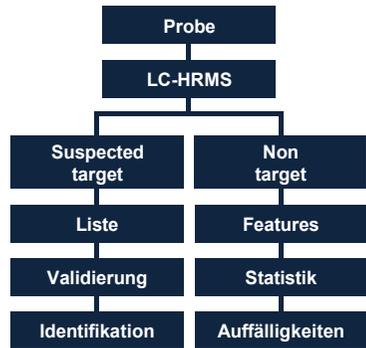
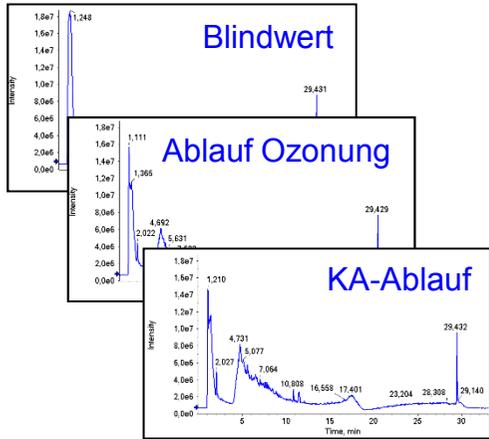
Intensity, cps vs m/z, Da

MS/MS

+TOF MS/MS of 280.1911

Intensity, cps vs m/z, Da

Zusammenfassung



- 19 der 20 ausgewählten Substanzen (vorgegebene Liste) quantifiziert via TOF-MS und MS/MS
- 60 Substanzen mittels Referenzmaterial identifiziert (RT, TOF-MS und MS/MS)
- Substanzlistenabfrage (SuMS) ergibt ca. 200 Verdachtskandidaten
- Non-Target Feature Finding in ESI positiv und negativ (ca. 1700 Features)
- Prozessbeschreibung mit Hilfe von Punktwolkenvergleich
- Multivariate Statistik (PCA) liefert auffällige Features

Dank

Thomas Lucke
Marina Bischoff
Rebecca Harsch
Tobias Bader



und Ihnen für Ihre Aufmerksamkeit