Retention-time-index (RTI) – a novel chromatographic parameter in the field of LC-MS(/MS) – An interlaboratory study

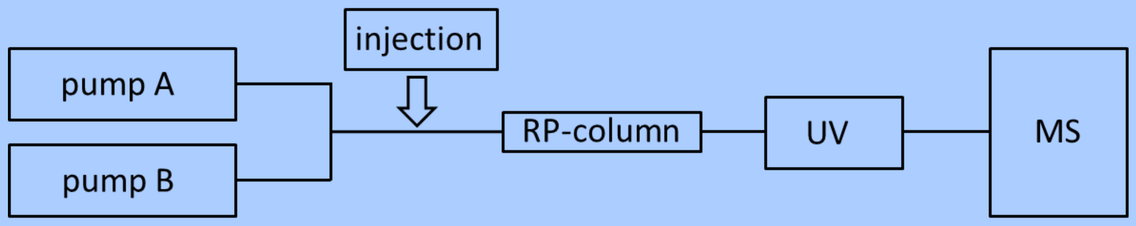
Grosse, S., Greco, G. and Letzel, T.

TU München, Analytische Forschungsgruppe am Lehrstuhl für Siedlungswasserwirtschaft, Am Coulombwall, 85748 Garching/D
Dr. Thomas Letzel, letzel@wzw.tum.de

Project

The Retention Index (RI) is well known in the field of GC-MS. To determine the RI a mixture of n-alkanes has to be injected to the GC-MS method in the own laboratory. The diversity of used columns and oven programs in GC measurements are straightforward. This is more complex for LC measurements due to much more different stationary phases, a wide range of polarity of analytes, and the use of different mobile phases. To introduce the Retention Time Index (RTI) in the field of liquid chromatography all these parameters have to be considered carefully. For the first interlaboratory study two standard mixtures (K = reference standard; U= unknown standard) were injected to the own LC-MS method on a C18 column and the RTI of the unknown molecules were calculated via software.

Experimental setup



column type:	**C18** (with and without endcapping)
type of mobile phase:	**free choice**
pH of mobile phase:	**free choice**
RTI mixture K:	Known molecules with logP range -1.36 - + 5.28 [1]
RTI mixture U:	Unknown molecules inside the logP range of K

Results

a) laboratory **1** – RTI mixture K

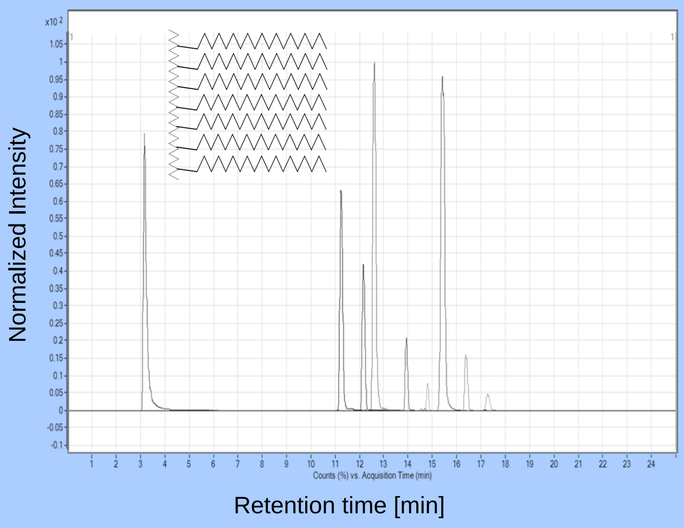


Table 1:
RTI mixture K analyzed and normalized by openMASP software tool
Laboratory 1

Name	Retention time [min]	logP [1]	RTI
Metformin	3.1	-1.36	50.0
Chloridazon	11.2	1.11	87.2
Carbetamide	12.2	1.65	95.3
Monuron	12.6	1.93	99.5
Metobromuron	13.9	2.24	104.2
Chlorbromuron	14.8	2.85	113.4
Metconazole	15.4	3.59	124.5
Diuron	16.4	4.19	133.6
Quinoxyfen	17.3	4.98	145.5
Fenofibrate	17.1	5.28	150.0

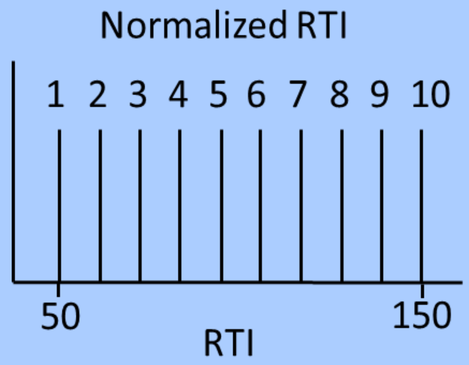


Table 3:
RTI mixture U analyzed and calculated by openMASP software tool
Laboratory 1

Name	Retention time [min]	RTI	logP calculated
Dapson	11.1	86.7	1.08
Linuron	14.7	112.4	2.78
Picoxystrobin	15.5	125.5	3.65

b) laboratory **2** – RTI mixture K

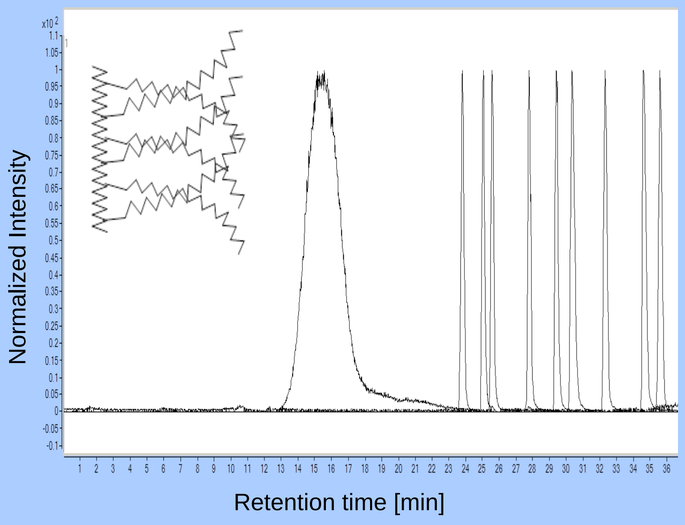
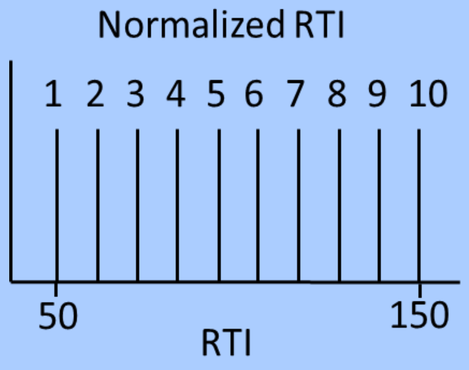


Table 2:
RTI mixture K analyzed and normalized by openMASP software tool
Laboratory 2

Name	Retention time [min]	logP [1]	RTI
Metformin	15.6	-1.36	50.0
Chloridazon	23.8	1.11	87.2
Carbetamide	25.1	1.65	95.3
Monuron	25.6	1.93	99.5
Metobromuron	27.8	2.24	104.2
Chlorbromuron	29.4	2.85	113.4
Metconazole	30.3	3.59	124.5
Diuron	32.3	4.19	133.6
Quinoxyfen	34.6	4.98	145.5
Fenofibrate	35.6	5.28	150.0



RTI values included in Table 6

Table 4:
RTI mixture U analyzed and calculated by openMASP software tool
Laboratory 2

Name	Retention time [min]	RTI	logP calculated
Dapson	24.1	89.1	1.23
Linuron	29.2	112.3	2.77
Picoxystrobin	31.3	129.1	3.89

Abb. 1:
a) chromatographic run with a C18 column measured in laboratory 1
b) chromatographic run with a C18 column measured in laboratory 2

Outside Germany:
Innsbruck (Austria)
Zurich and Dübendorf (Switzerland)
Stockholm (Sweden)
Athens (Greece)

Abb.2: map of participants [2]

Table 5:
Summary of all acquisition methods used in the first interlaboratory study

	total	In acidic condition	In neutral condition	Different column types	C18 endcapped	C18 non-endcapped	C18 polar endcapped
No. Labs	23						
No. Methods (total)	33	30	3	22	31	0	2
No. Methods (with MeOH)	16	14	2	11	15	0	1
No. Methods (with ACN)	17	16	1	11	16	0	1

The participation of 23 laboratories allow a good statistical analysis for the first interlaboratory study.
The reults show a very good standard deviation (< 6%) of the calculated RTI over all laboratories and used chromatographic methods. The sum of injections had been 52 out of 23 laboratories.

Table 6:
RTI standard deviation of all used methods in the study

	Total (all methods) s [%]	Methods with MeOH s [%]	Methods with ACN s[%]
Dapson	5.8	4.2	2.0
Linuron	1.6	1.4	1.7
Picoxystrobin	4.3	1.2	1.8

Outlook

In a next step polar embedded stationary phases will be allowed and the openMASP software toll will also be used to calculate the logP of the molecules. Therefore a second interlaboratory study will be performed .

Literature

[1] logP values out of www.chemicalize.org
[2] http://www.campion-boote.com/images/map_deutschland.jpg

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