Target and non-target/suspect screening analyses for emerging substances in air and dust from various indoor environments

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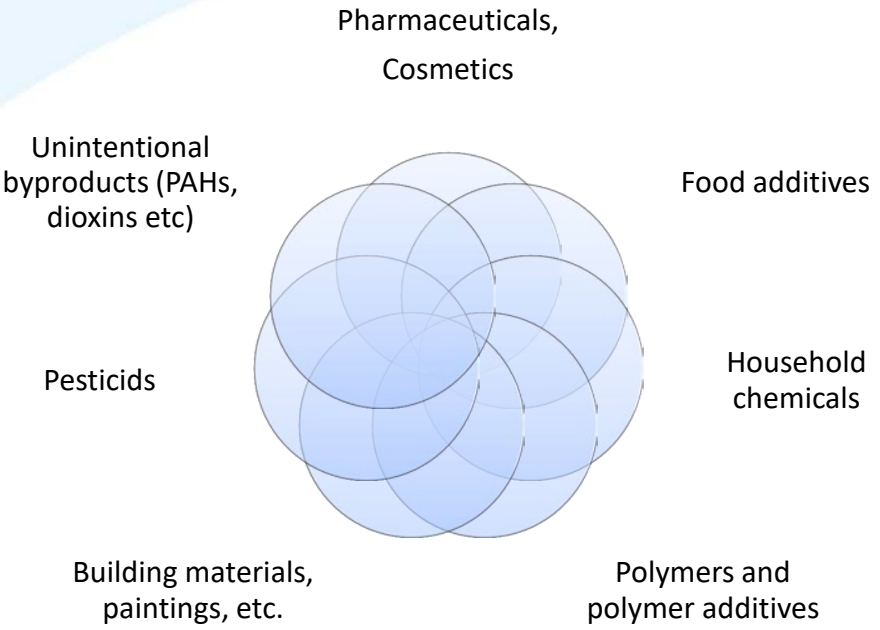
NILU Norsk institutt for luftforskning

Daily exposure to chemical cocktail

- Modern live based on chemicals
- Most chemicals are mobile and find their way to environment and humans



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Exposure

Digestive system:

food, drinks, drugs, dust , soil

Lungs: Air pollution (gases, volatile and semi-volatile compounds, dust)

Skin: Cosmetics, drugs, other.


Ingestion


Inhalation


Skin absorbtion

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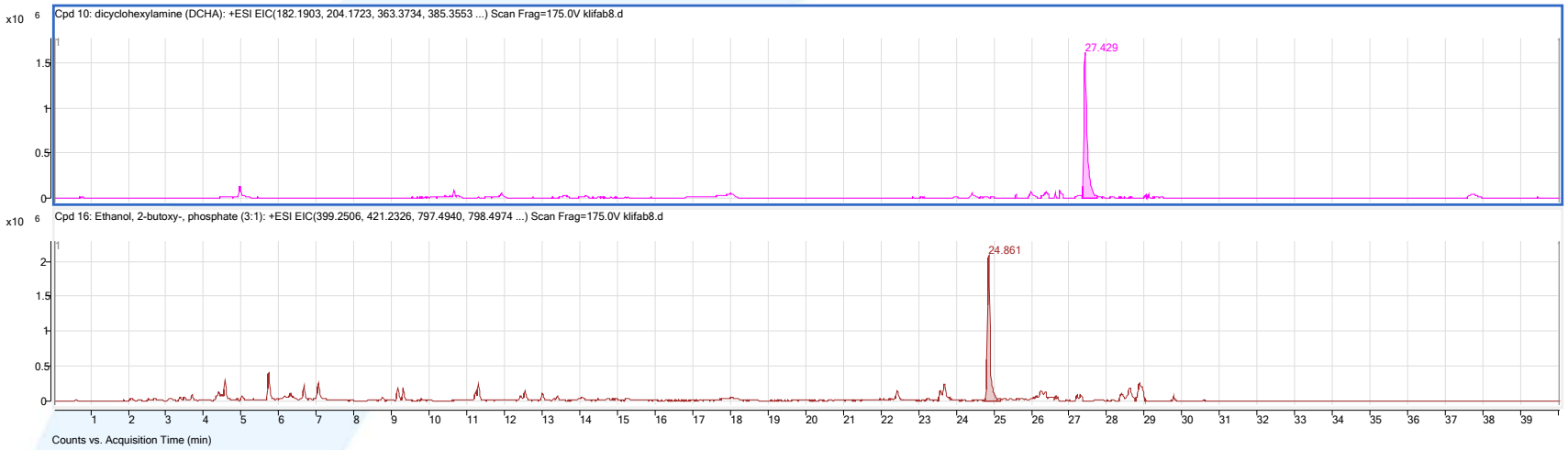
Analytical approaches

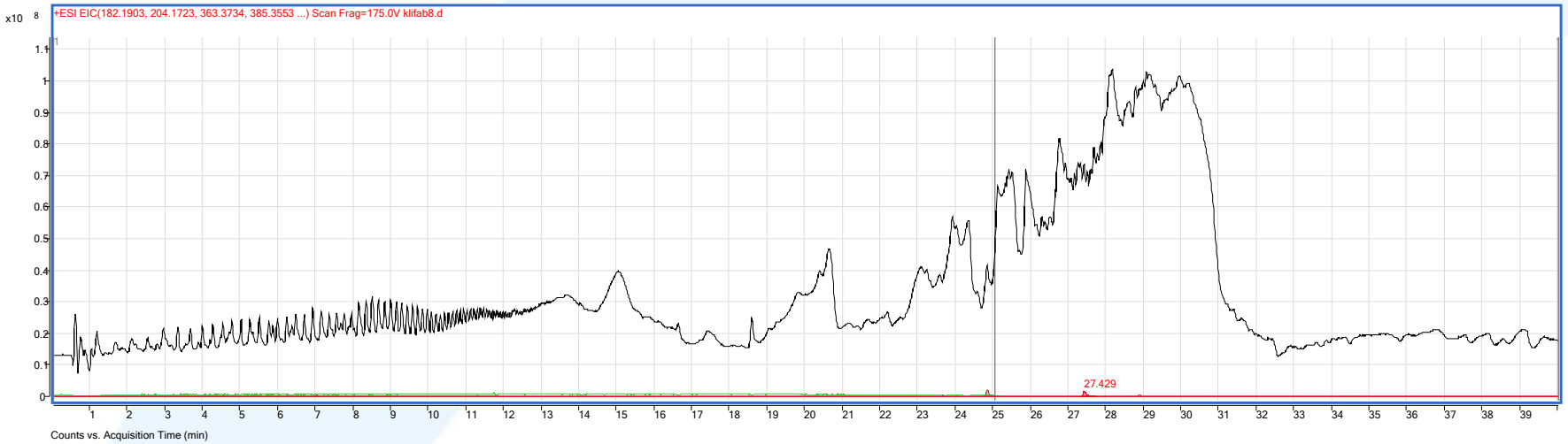
- Traditional – *targeted* screening approach

- *Non-targeted (non-specific)* screening approach

Targeted screening



Non-targeted screening



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Why non-target?

- Concentrations of known compounds are not high enough to explain some of toxic potentials of the samples

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New requirements to analytical chemistry

Rapid change in chemical products requires flexible analytical methods

”Non target” or non specific screening

New instrumental techniques available (for example: time-of-flight, Orbitrap MS)

Advantages:	Simultaneous analyses of 1000 of compounds (~100 in targeted methods)
Challenges:	1. Treatment of HUGE data files
	2. ”Separating the wheat from the chaff”

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Passive air sampling

Foto: Helene Lunder Halvorsen

Target screening of indoor air and dust

Screening 2016 – Norwegian Environment Agency

Sampling of indoor air

Adapted from Schlabach et al., 2017

Sampling of indoor dust

Adapted from Schlabach et al., 2017

Concentrations of volatile compounds in dust and indoor air samples from the Oslo area

	L3	L4	L5	1,3-di-isopropyl-benzene	1,4-di-isopropyl-benzene	4-Isopropyl-1,1'-biphenyl
Sample type	(Min – max) Average* Detection frequency			ng/g and ng/m^3		
House dust	(0,23 - 1,3) 0,46 100 %	(<0,2 - 1,6) 0,64 89 %	(<10 – 464) 98 55 %	(<0,5 - 9,2) 1,3 22 %	(<0,6 - 8,0) 1,3 11 %	(0,25 – 15) 2,3 100 %
Indoor air	(1,6 – 743) 88 100 %	(1,1 – 37) 14 100 %	(5,6 – 1460) 195 100 %	(0,45 - 4,7) 2,1 100 %	(0,51 - 3,6) 1,8 100 %	(<0,2 - <1,1) 0,22 100 %

Adapted from Schlabach et al., 2017

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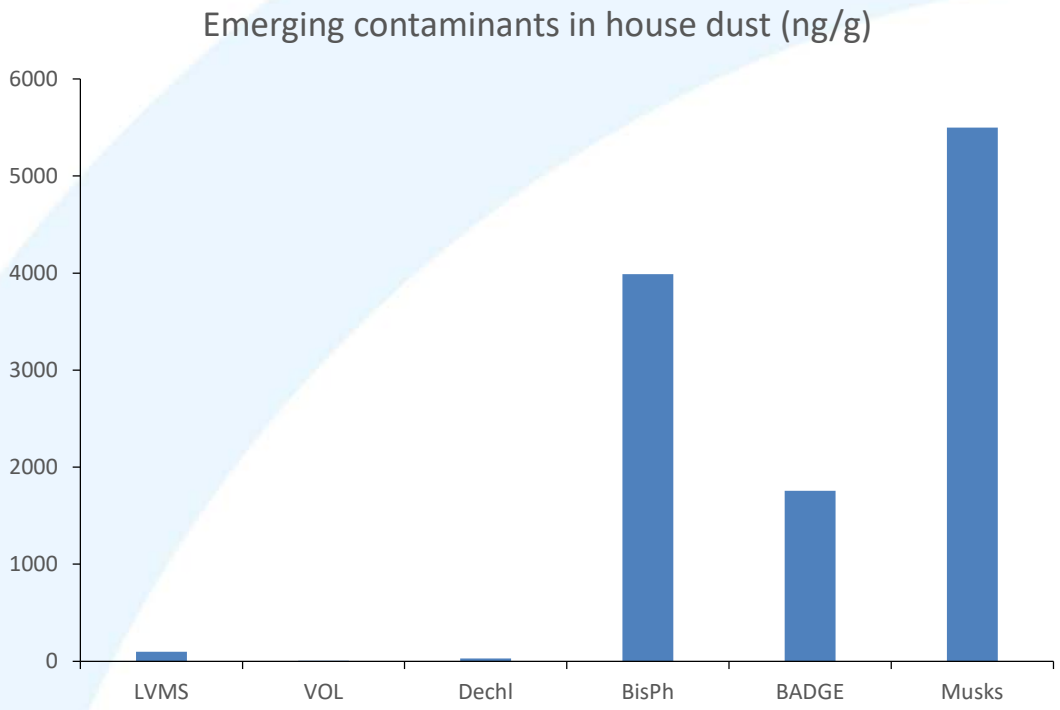
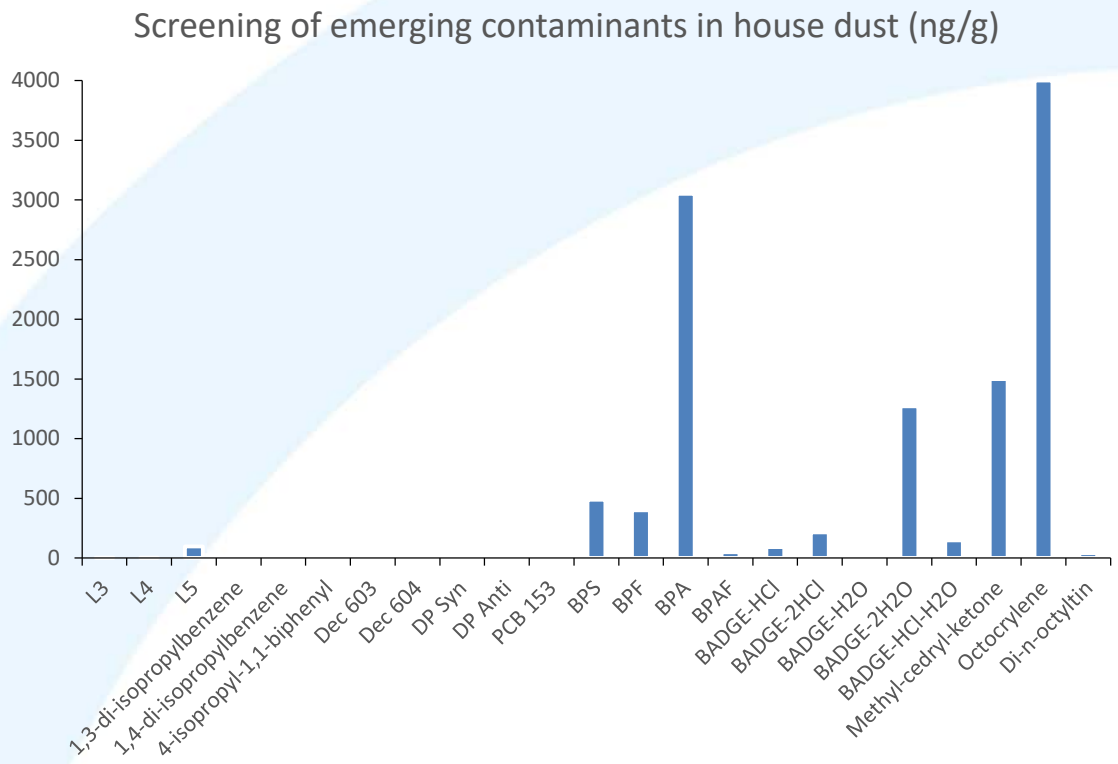
House dust



Adapted from Schlabach et al., 2017

House dust



Adapted from Schlabach et al., 2017

Norman Collaborative Trial of the Indoor dust

Rostkowski P[1], Haglund P[2], Oswald P[3], Alygizakis N[3], Thomaidis N[4], Aalizadeh R[4], Covaci A[5], Moschet Ch[6], Kaserzon S[7], Yang Ch[8], Shang D[9], Hindle R[10], Booij P[11], Ionas A[11], Grosse S[12], Arandes JB[13], Dévier MH[14], Lestremau F[15], Leonards P[16], Plassmann M[17], Magner J[18], Matsukami H[19], Jobst K[20], Ipolyi I[3], Slobodnik J[3], Reid M[21]

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Network of reference laboratories, research centers and related organizations for monitoring of emerging environmental substances

83 members, 8 working groups

https://www.norman-network.net/

Working Groups

The NORMAN network runs six Working Groups and two Cross-Working Group Activities, dealing with various issues related to emerging substances.

WG1

Prioritisation of emerging substances

WG2

Bioassays and biomarkers in water quality monitoring

WG3

Effect-directed analysis for hazardous pollutants identification

Cross-Working Group Activity: Passive sampling

Passive sampling for emerging contaminants

Cross-Working Group Activity Non-target Screening (NTS)

Non-target screening techniques for environmental monitoring

WG4

Nano-and micro scale particulate contaminants

WG5

Wastewater reuse and Contaminants of Emerging Concern

WG6

Emerging substances in the indoor environment

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REVIEW

Non-target screening with high-resolution mass spectrometry: critical review using a collaborative trial on water analysis

Emma L. Schymanski[1] · Heinz P. Singer[1] · Jaroslav Slobodnik[2] · Ildiko M. Ipolyi[2] · Peter Oswald[2] · Martin Krauss[3] · Tobias Schulze[3] · Peter Haglund[4] · Thomas Letzel[5] · Sylvia Grosse[5] · Nikolaos S. Thomaidis[6] · Anna Bletsou[6] · Christian Zwiener[7] · María Ibáñez[8] · Tania Portolés[8] · Ronald de Boer[9] · Malcolm J. Reid[10] · Matthias Onghena[11] · Uwe Kunkel[12] · Wolfgang Schulz[13] · Amélie Guillon[14] · Naïke Noyon[14] · Gaëla Leroy[15] · Philippe Bados[16] · Sara Bogialli[17] · Draženka Stipaničev[18] · Pawel Rostkowski[19] · Juliane Hollender[1,20]

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Abstract In this article, a dataset from a collaborative non-target screening trial organised by the NORMAN Association is used to review the state-of-the-art and discuss future perspectives of non-target screening using high-resolution mass spectrometry in water analysis. A total of 18 institutes from 12 European countries analysed an extract of the same water sample collected from the River Danube with either one or both of liquid and gas chromatography coupled with

Published in the topical collection *High-Resolution Mass Spectrometry in Food and Environmental Analysis* with guest editor Aldo Laganà.

Electronic supplementary material The online version of this article (doi:10.1007/s00216-015-8681-7) contains supplementary material, which is available to authorized users.

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Certificate

Most Cited in 2017

Non-target screening with high-resolution mass spectrometry: critical review using a collaborative trial on water analysis

E.L. Schymanski, H.P. Singer, J. Slobodnik, I.M. Ipolyi, P. Oswald, M. Krauss, T. Schulze, P. Haglund, T. Letzel, S. Grosse, N.S. Thomaidis, A. Bletsou, C. Zwiener, M. Ibáñez, T. Portolés, R. de Boer, M.J. Reid, M. Onghena, U. Kunkel, W. Schulz, A. Guillon, N. Noyon, G. Leroy, P. Bados, S. Bogialli, D. Stipaničev, P. Rostkowski, J. Hollender

Anal Bioanal Chem (2015) 407:6237–6255

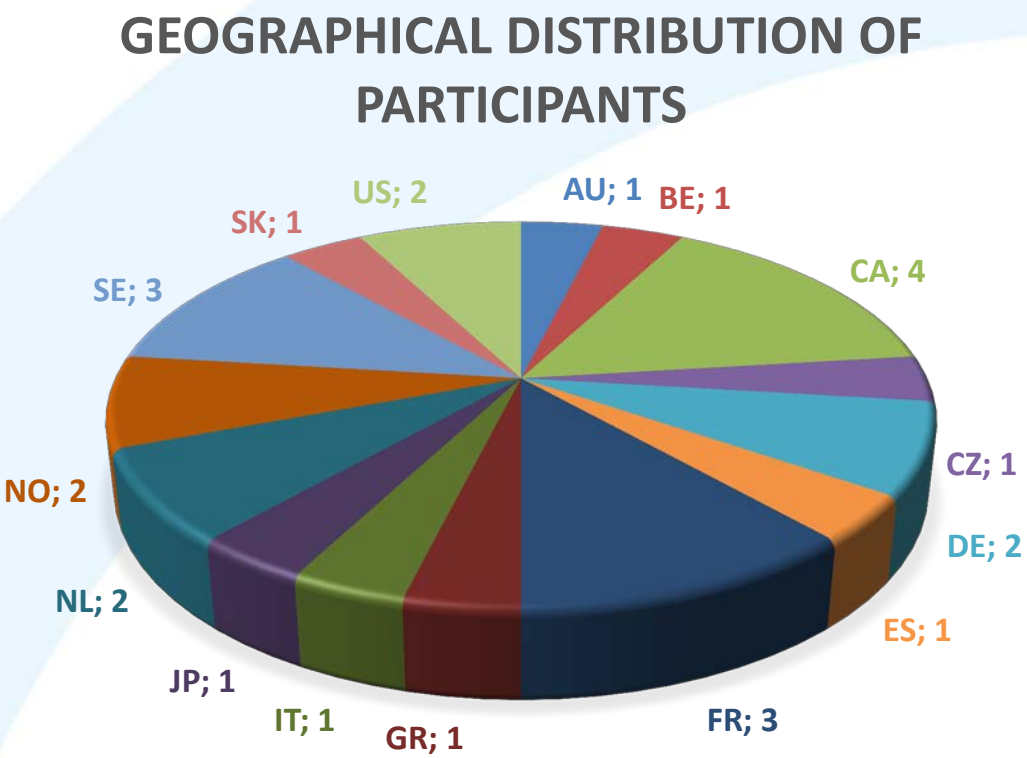
published in 'Analytical and Bioanalytical Chemistry' in 2015-2016 and one of the most cited articles in 2017.

The Editors offer their sincere congratulations and would like to thank you again for publishing your excellent work in the journal.

Nicola Oberbeckmann-Winter
Managing Editor
Analytical and Bioanalytical Chemistry

On behalf of the 'Analytical and Bioanalytical Chemistry' Editors
Hua Cui, Philippe Garrigues, Guenter Gauglitz, Emily Hilder, Gérard Hopfgartner, David C. Muddiman, Alfredo Sanz-Medel, Stephen A. Wise, Adam T. Woolley, Lihua Zhang

27 participants from 26 organisations from 15 countries



Participation GC-MS vs LC-MS

17 participants – registered for both techniques

3 participants GC-MS only

7 participants LC-MS only

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Samples

- 250mg of the homogenized, sieved dust obtained from household vacuum bags collected from homes around Toronto, Canada in 2015
- standard mixtures for use in calculation of retention time index information (for LC and GC-MS)

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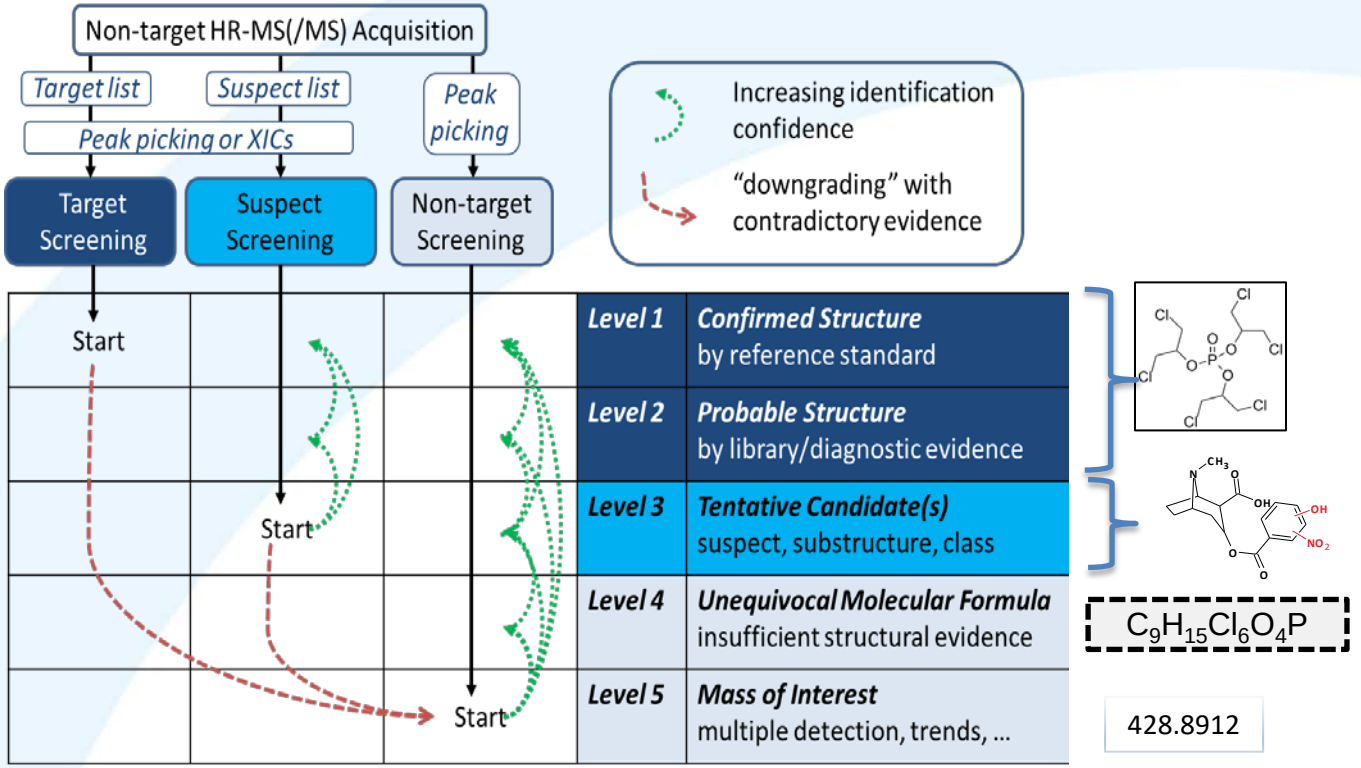
Extraction

- dichloromethane for GC-MS analysis
- dichloromethane: methanol (1:9, v/v) for LC-MS analysis.
- The extraction technique and clean-up techniques were not specified.

Workflow Norman approach



Adapted from Schymanski et al., 2014, 2015

Submission of results

14 GC/MS data sets

20 LC-MS data sets

1 participant officially withdrawn from the CT (both techniques)

1 participant withdrawn from GC

9 raw data sets uploaded

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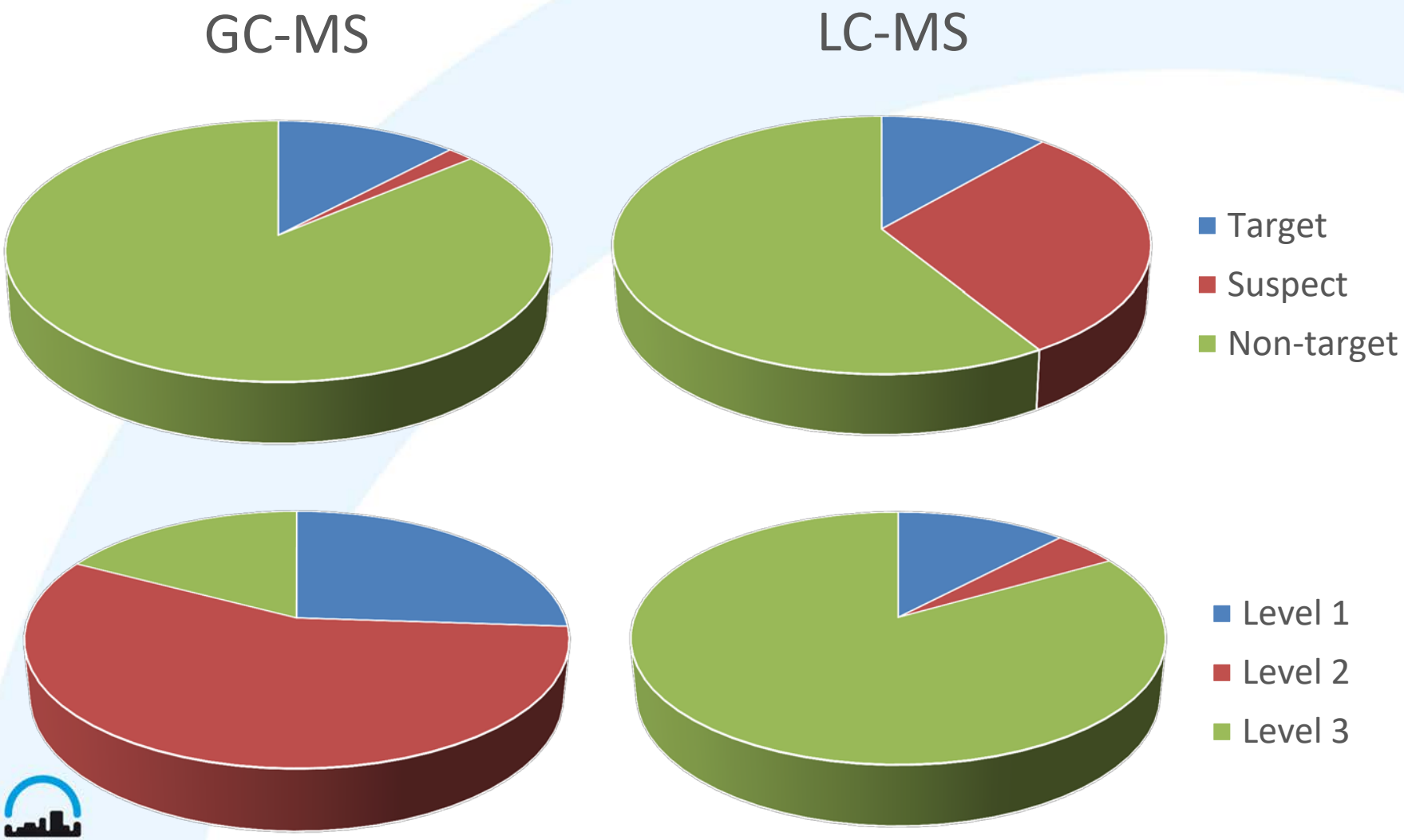
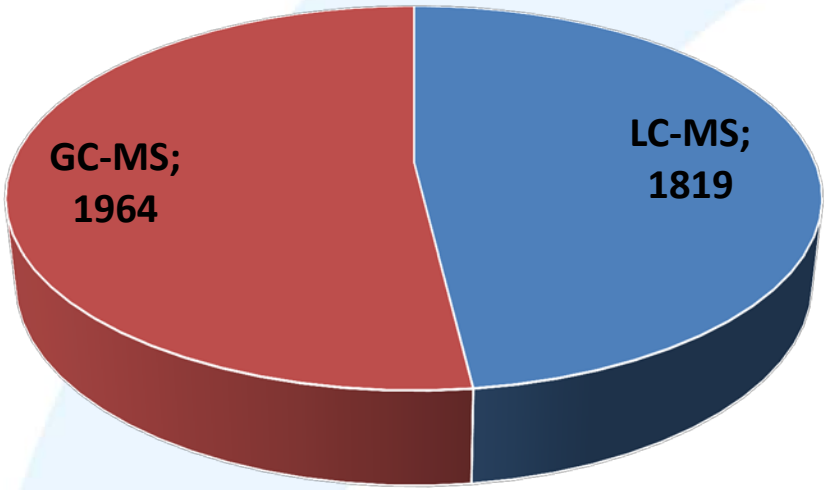
Workflows and ID confidences

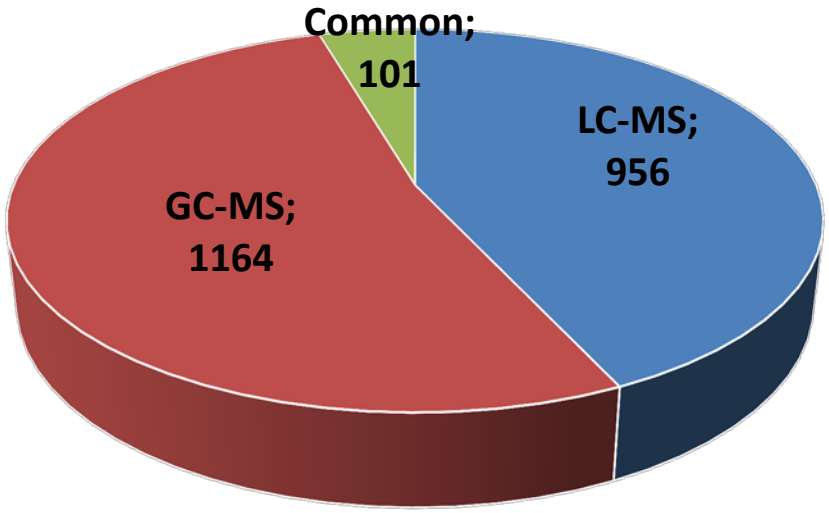


Total Number of Data and Compounds at Identification Level 1-3

Number of Data
(n= 3783)



Number of Compounds
(n=2120)



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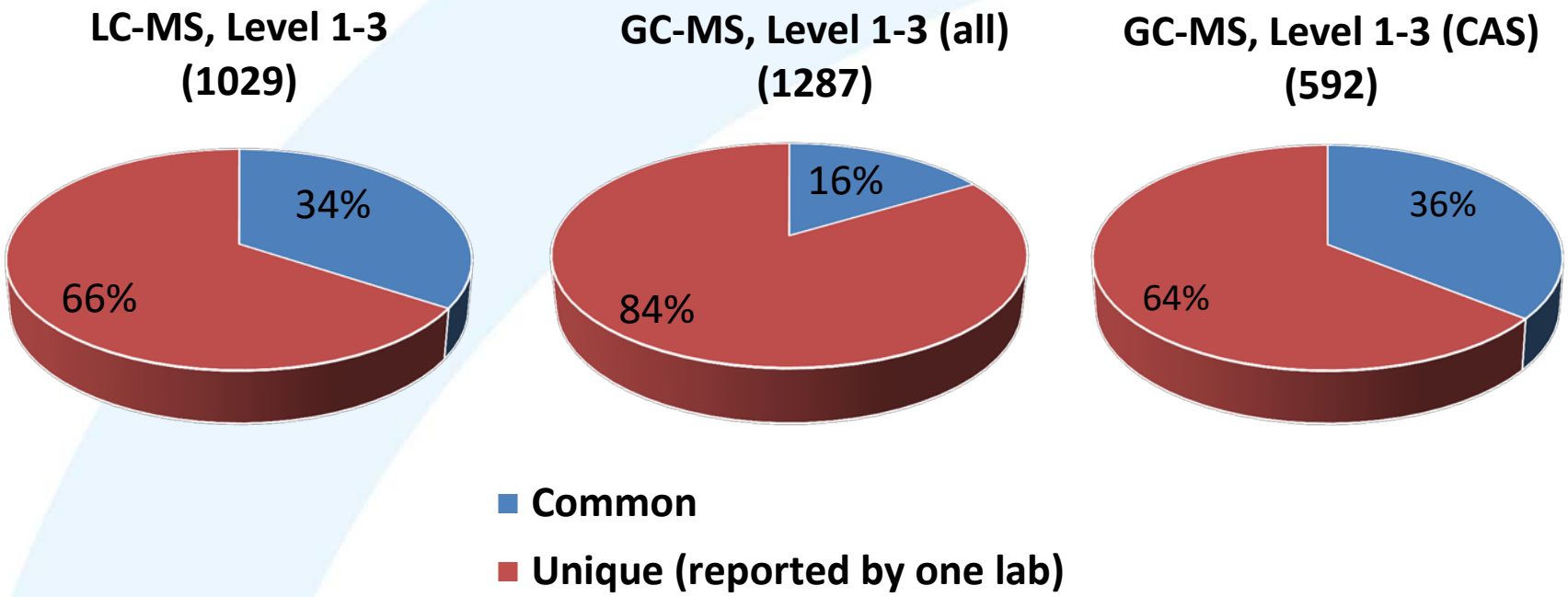
Compound Overlap Between Labs



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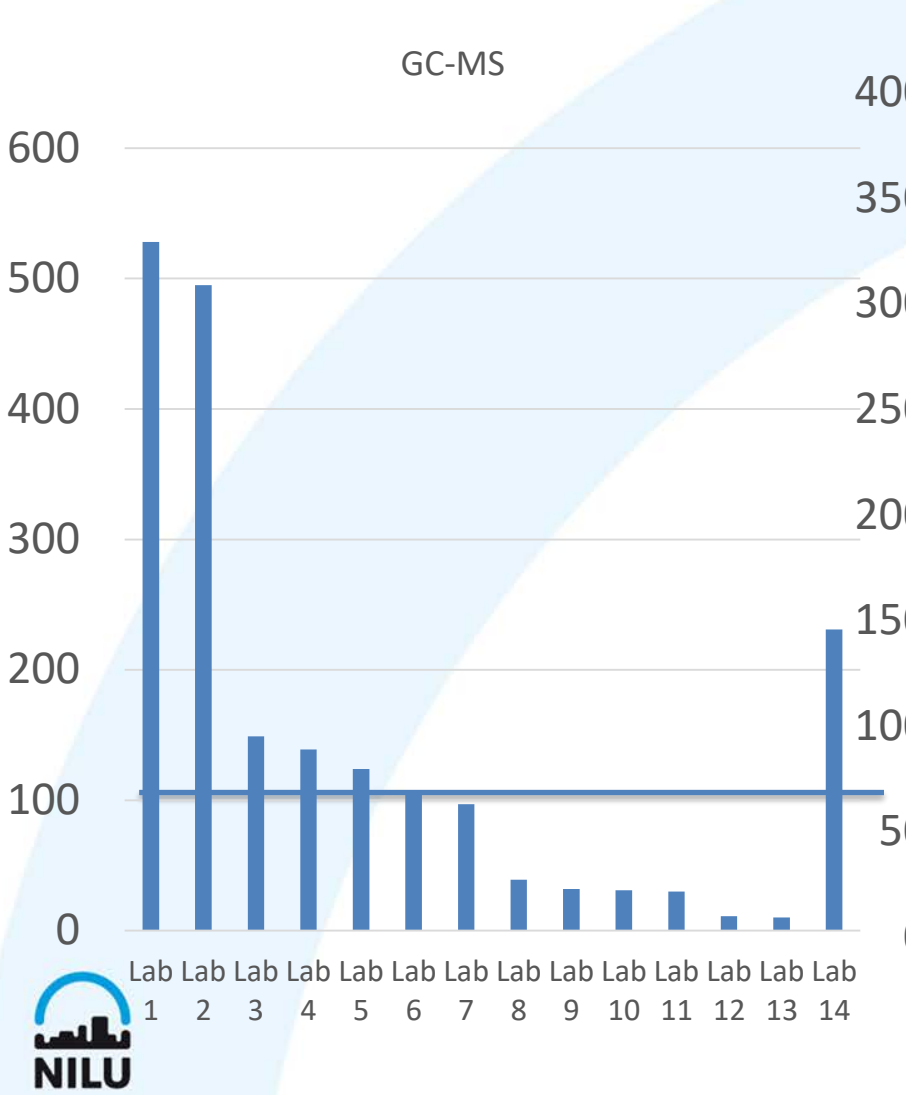
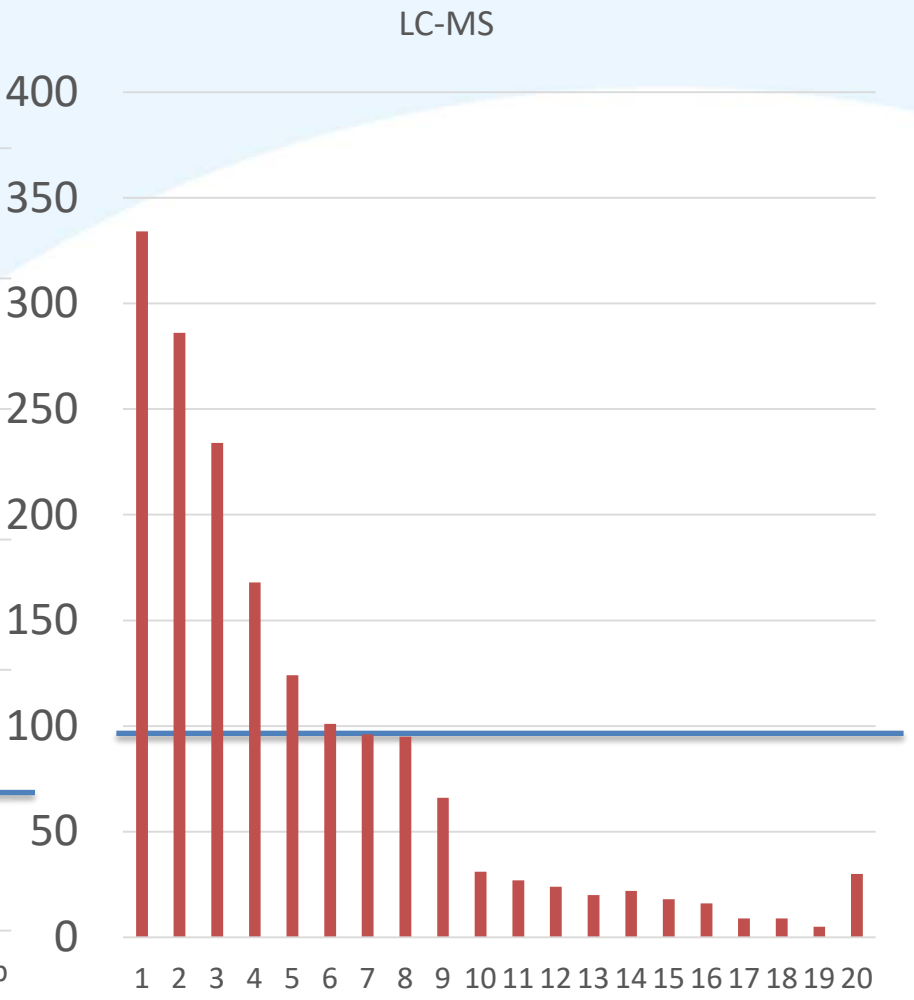
Lab contributions (Level 1-3)





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Data curation (LC-MS)

- Inspecting MS/MS data (if available)
- Library search with MassBank, NIST and Agilent PCDLs (if not included in reported workflow)
- MetFrag and CFM-ID prediction for spectra without library entries, Prediction of RTI (QSRR model) and RTI/log D correlation within the FOR-IDENT platform

Ruttkies, et al.(20 16) J Cheminform 8:3. doi:10.1186/s13321-016-0115-9
Allen, et al. (2014) Nucleic Acids Res 42 (Web Server issue):W94-99. doi:10.1093/nar/gku436
Aalizadeh, et al.(2016) J Chem Inf Model 56 (7):1384-1398. doi:10.1021/acs.jcim.5b00752

Data curation (GC-MS)

- Calculation of LRI (Van den Dool and Kratz (1963) and correlation with MW
- Abraham model
- Manual review of outliers

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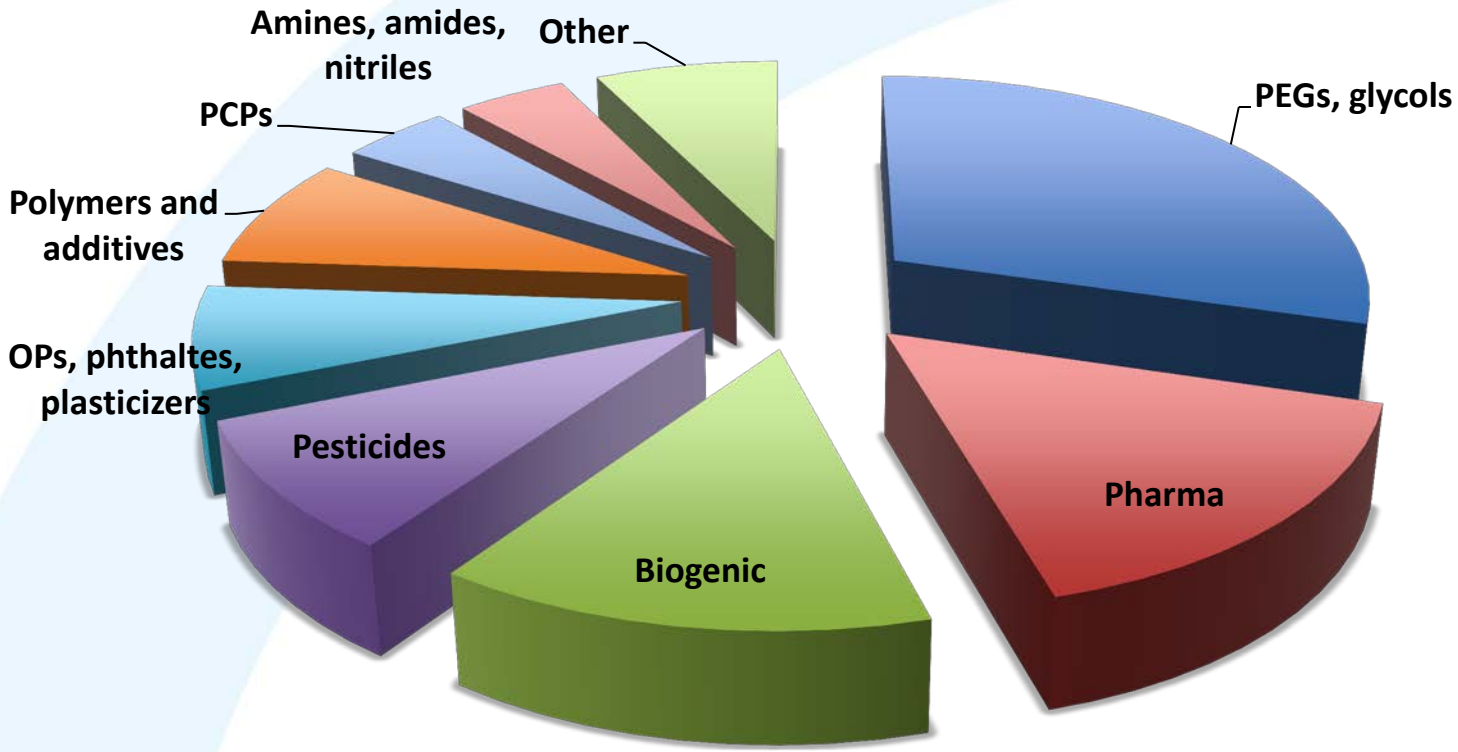
LC Compounds (tot 1029)



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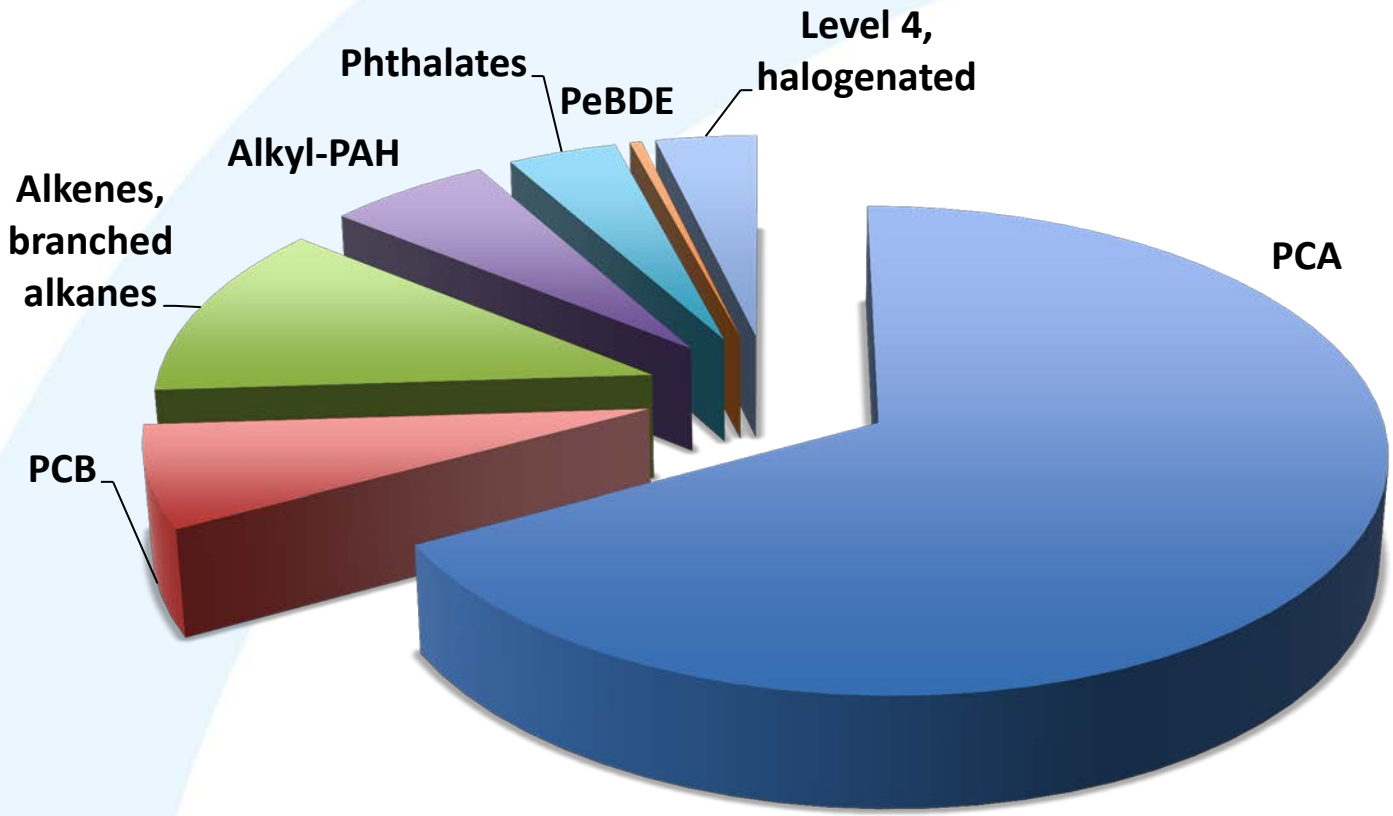
GC compounds: Positional isomers (tot 695)



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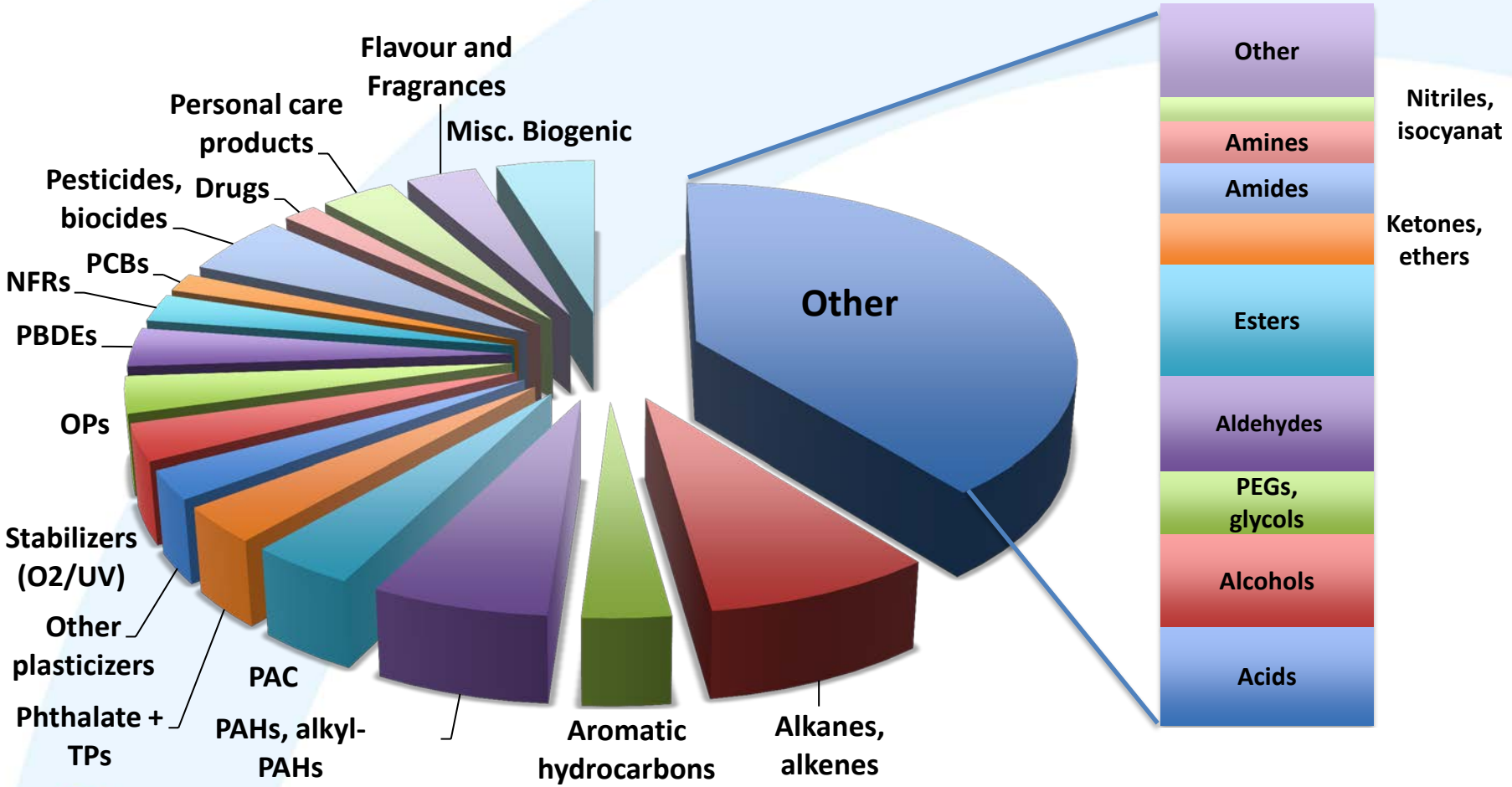
GC compounds: with CAS (tot 592)



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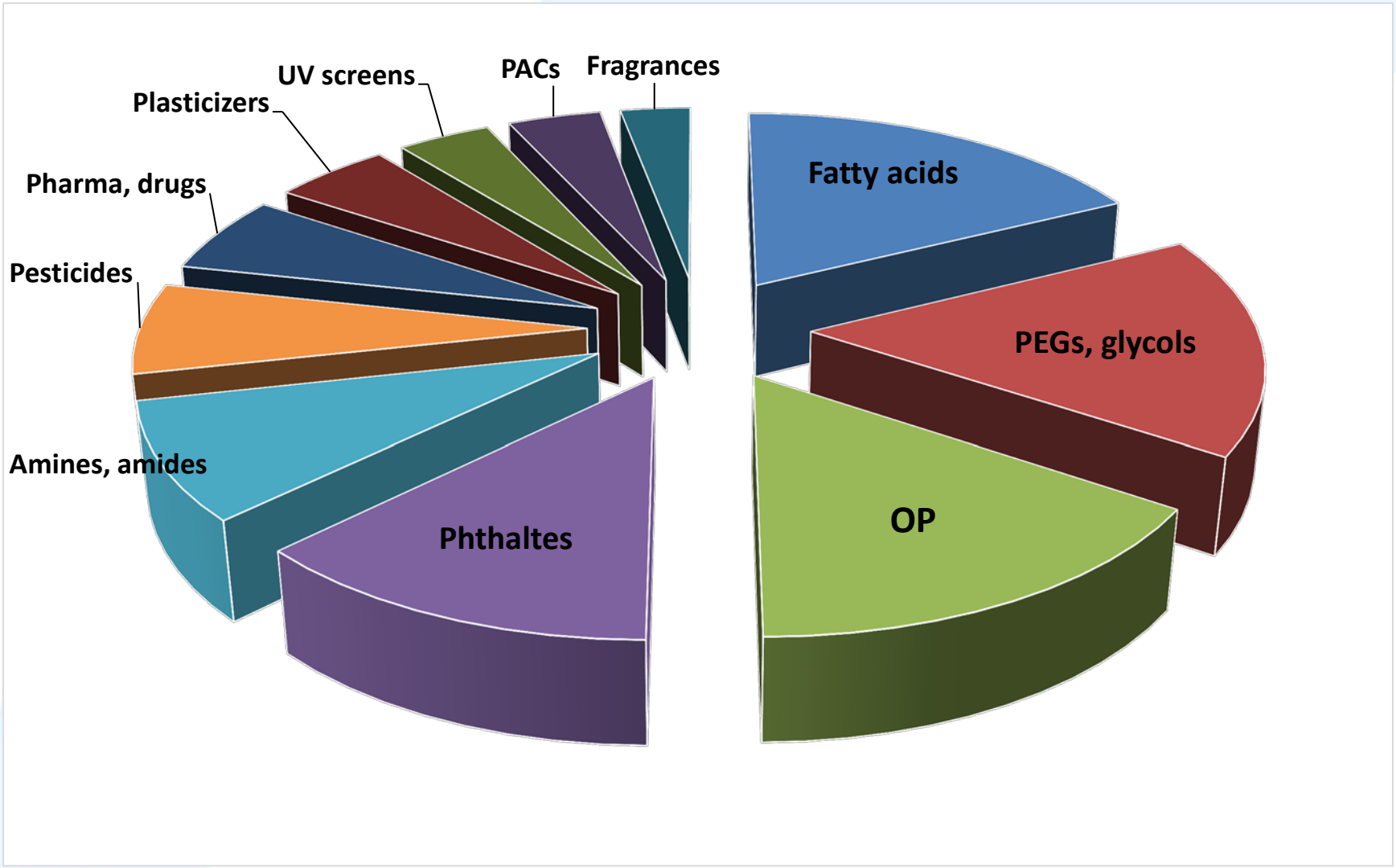
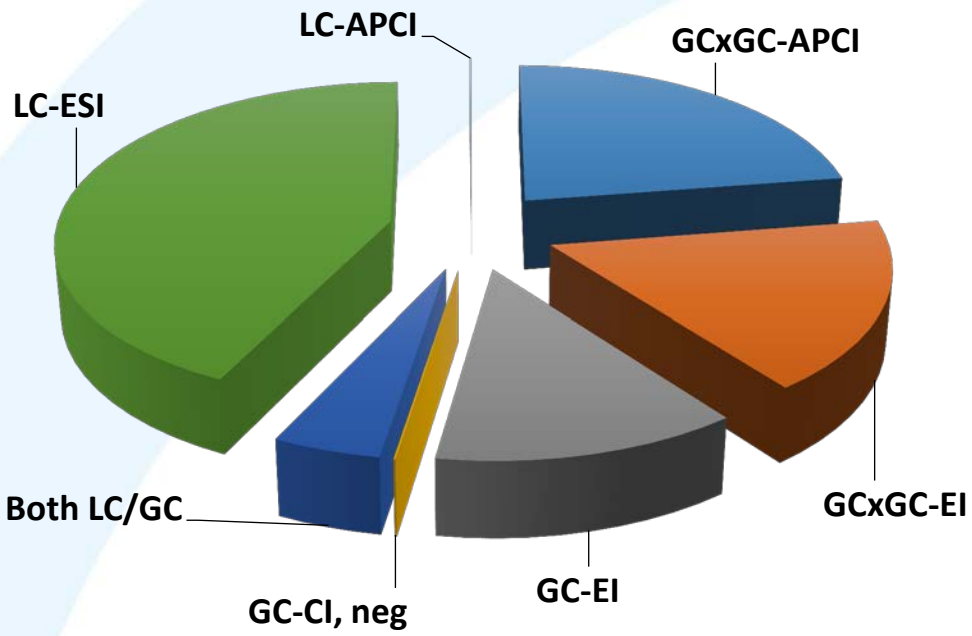
Compounds detected with LC-MS and GC-MS



Contribution of instrument platforms to identification of compounds



Summary

- **>2000 compounds detected** (indoor dust DB)
- **Over 80% unique** (detected by one lab only)
- **Approx. 10% overlap between LC-MS and GC-MS**
- **High complementarity between techniques**
  - **GC: small non-polar and semi-polar compounds**
  - **LC: semi-polar and polar compounds**
  - **GC-ECNI and GC-APCI(–): Halogenated compounds**
  - **GC-PCI: Confirmation of molecular ions**
  - **GC×GC homologous series of non/semi-polar compounds**
  - **LC-ESI(+) compounds with high proton affinity (ca 40% amines/amides)**

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Acknowledgments

Pernilla Bohlin Nizetto

Martin Schlabach

Anders Borgen

Nicholas Warner

Other members of NILU team

Participants of Norman Trial

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Thank you for your attention!

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