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LC-HRMS-based untargeted metabolomics as a tool for analytical development: In-depth assessment of exhaustive extraction protocols for pesticides-polluted soils

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Context

5 extraction protocols were tested on 3 environmental conditions (all applied on 2 different soils): control, spiked with the Glyphosate pesticide, spiked with Pelargonic acid formulation. Determining the “optimal” extraction is not an easy task, as the experimental design is large, multi-factorial and complex. Hence, an innovative LC-MS/HRMS-based untargeted approach was designed in order to evaluate the optimal protocol by using metabolomics computational tools. Those tools provided easily-interpretable plots, comprehensive quantitative indices and chemical information. They were used to assess 4 criteria defining the “optimal” conditions: 1) the ability of the extraction to cover a wide range of metabolites (qualitative), 2) its yield (quantitative), 3) its repeatability, and 4) its ability to allow for the discrimination between spiked and control samples. Therefore, the current presentation will introduce this innovative LC-MS/HRMS untargeted metabolomics-based approach as a powerful tool for methods development

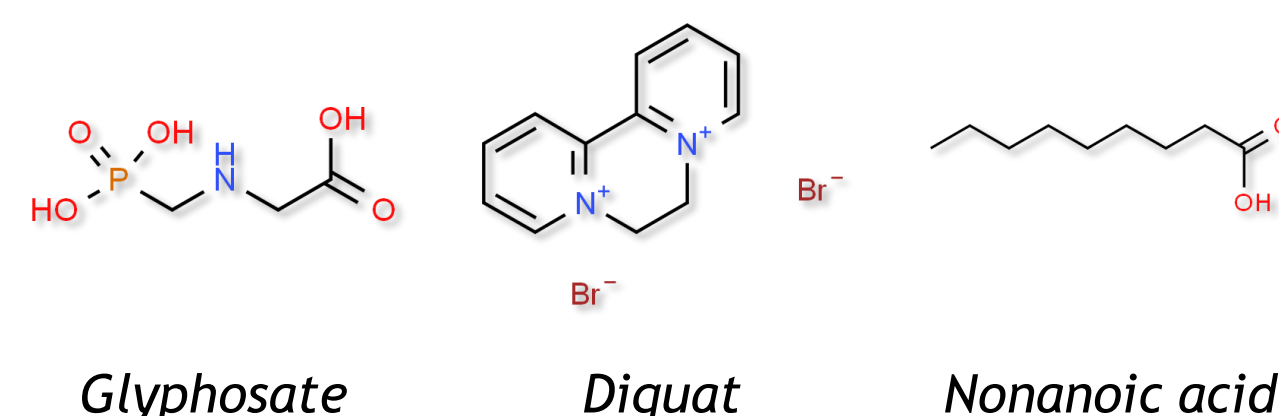
Experiment

Experimental design

Laboratory soil microcosms for 2 different soils (SP & ST)

3 environmental modalities with 5 replicates for each:

- ✓ Un-spiked Control soil (Ctr)
- ✓ Soil spiked with Formulated Glyphosate/Diquat (Gly)
- ✓ Soil spiked with Formulated Nonanoic acid (Bel)



Spiking:
1× the field dose

Extractions

5 different protocols with different solvents and reagents were applied:

- ✓ E1: Simple extraction based on acidified (HCl) Ethyl Acetate (Romdhane et al.)
- ✓ E2: Simple extraction based on Methanol (Anastasiades et al.)
- ✓ E3: Simple extraction based on Methanol + 1% FA (v/v) (Anastasiades et al.)
- ✓ E4: Two-steps extraction based on two miscible mixtures of solvents
- ✓ E5: Two-steps extraction based on two miscible mixtures of acidified solvents

Total number of samples: $2 \times 3 \times 5 \times 5 = 150$

Chemical Analysis

LC: Vanquish UHPLC⁺ Focused System (Thermo Fisher Scientific)

- Modified C18 UHPLC Column (polar analytes retention enhanced)
- 20 min/run - Elution solvents:
 - ✓ A: H₂O + 0.1 % FA
 - ✓ B: ACN + 0.1 % FA

HRMS: maXis ESI-Q/ToF System (Bruker)

- Modes: Full HRMS and AutoMS/MS (DDA)
- Scan range: *m/z* 90-1000
- Resolution: 23000-26500 at *m/z* 90.9766-974.8133

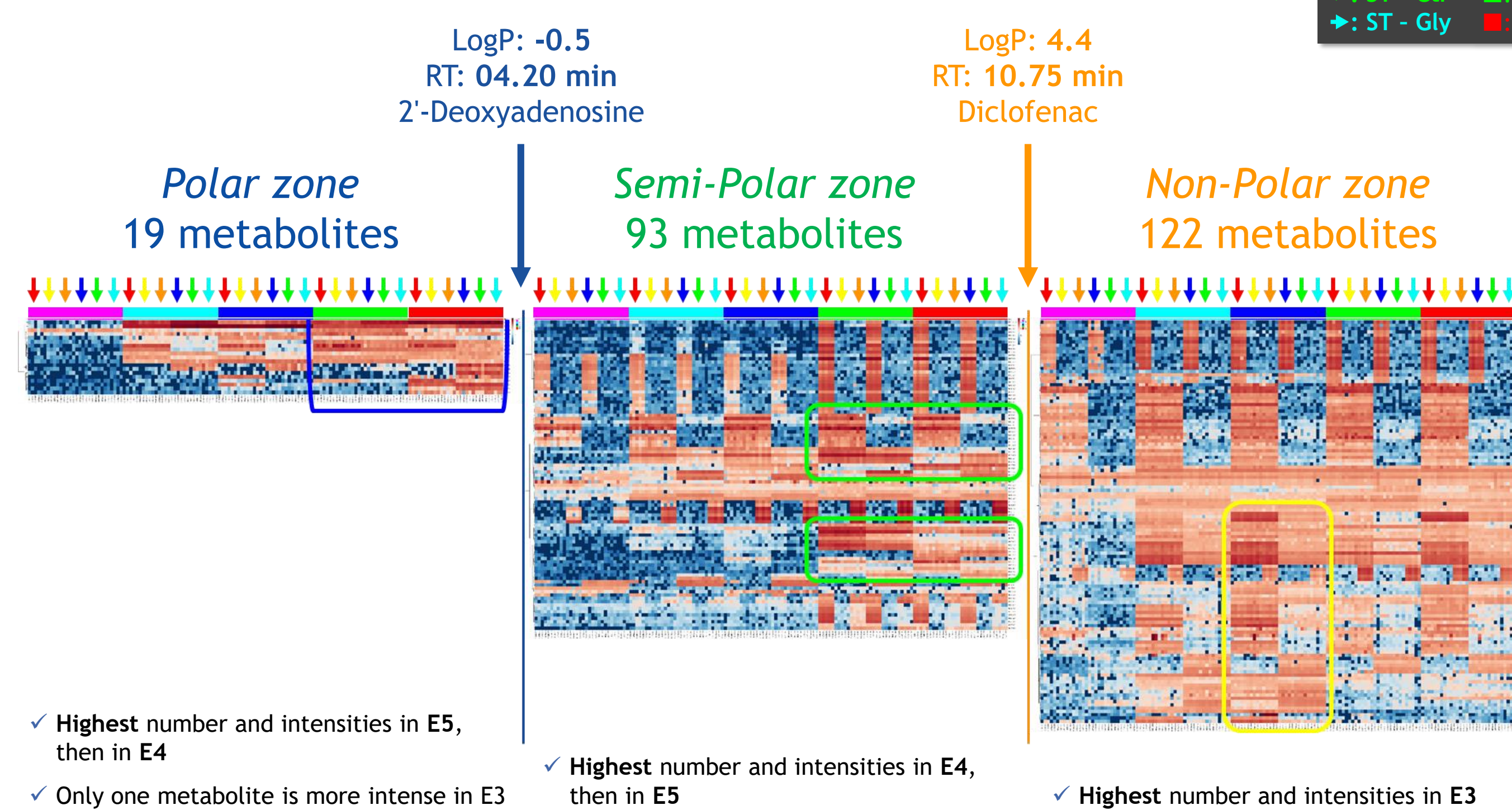


Data Analysis



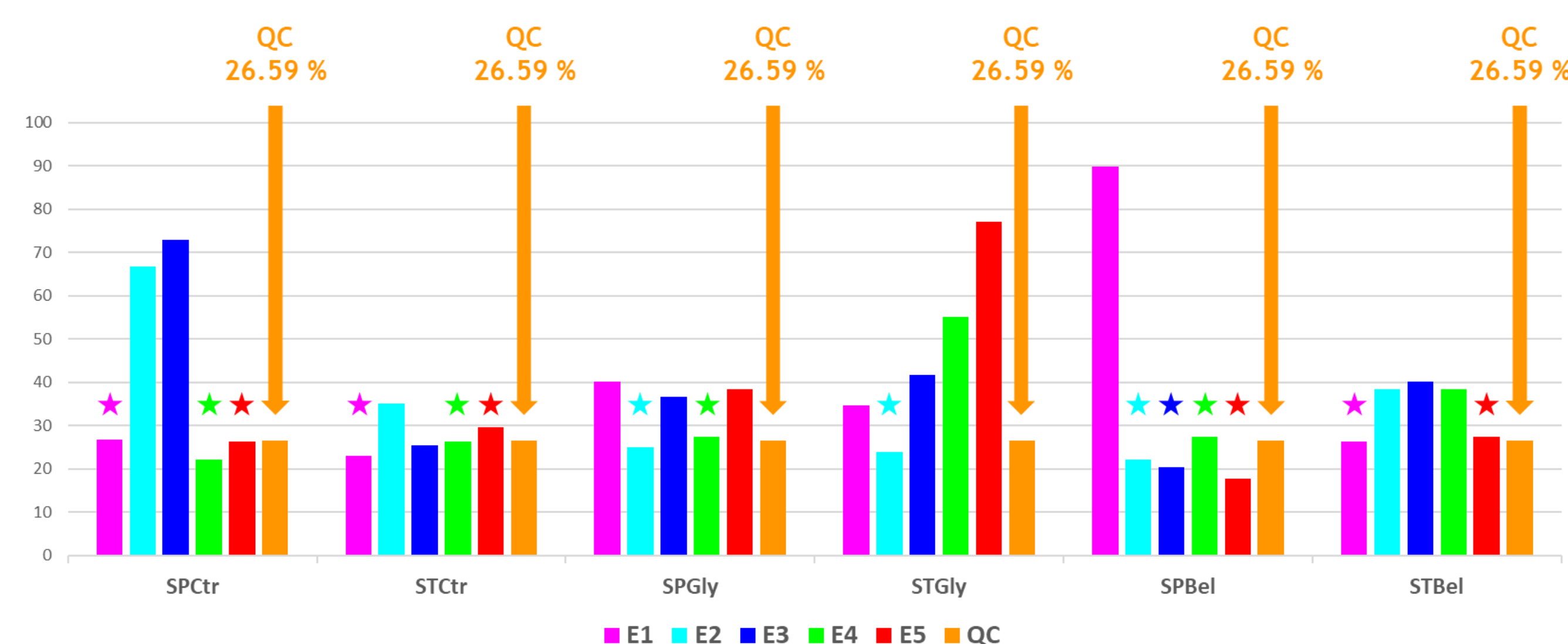
Results

Extraction Performance



Extraction Repeatability

➤ Euclidean Distances calculation: to assess extraction's “inter-replicates” reproducibility



Discrimination Power

- OPLS-DA is an explicative, supervised multivariate statistical analysis
- It allows to assess the ability and the predictivity of an extraction to discriminate between a control group of soil samples and a herbicide-spiked group of soil samples

	SP						ST					
	p1 R2X	R2Y	Q2	o1 R2X	R2Y	Q2	p1 R2X	R2Y	Q2	o1 R2X	R2Y	Q2
Gly												
E1	17.90	97.40	61.20	14.00	02.51	03.33	16.70	98.50	60.00	10.90	01.51	04.51
E2	19.30	96.40	67.10	12.40	03.21	04.34	17.70	98.10	63.00	10.50	01.74	04.21
E3	16.60	95.80	66.10	15.30	03.56	05.07	18.50	95.60	59.90	13.20	04.19	02.68
E4	24.90	97.70	80.80	10.60	02.14	02.63	20.10	97.80	72.10	13.50	02.06	05.51
E5	20.30	96.40	70.20	10.20	03.37	10.40	18.60	97.00	63.30	13.60	02.83	05.54
Bel												
E1	22.10	99.40	77.50	12.00	00.58	03.46	19.90	96.40	68.60	13.20	03.51	03.53
E2	29.80	98.60	88.40	11.90	01.31	02.56	29.40	96.50	85.60	09.71	03.40	01.34
E3	26.80	97.40	81.60	09.72	02.43	03.46	25.60	96.50	81.00	13.40	03.35	03.30
E4	37.20	98.90	93.40	10.70	01.07	01.39	33.00	98.00	90.80	15.00	01.87	02.53
E5	34.20	96.90	88.40	09.83	02.84	01.17	31.80	98.40	90.00	10.00	01.43	02.42

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Acknowledgments



Social Networks

