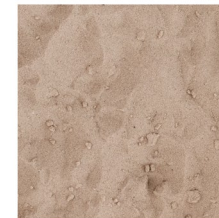# Interlaboratory Study on the Analysis of Microplastics in Environmental Matrices

WEPAL-QUASIMEME : ROUND 2024 - Development Exercise DE 17

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7 May 2025

Previous round (2022)

Test materials: Tablets (mimic water), sediment and sand

Results:

Majority report on <u>number</u> of particles, only a few on <u>mass</u> of particles

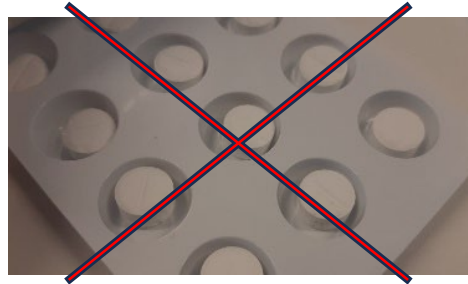➢ Mass determination: For most polymers too few data to calculate an assigned value

➢ Number of particles:

NDA Rel st. dev. : Tablets: 76-96 %
Sediment: 66-125 %
Sand: > 100 %

Goal of this Round

Take a step back in difficulty

➢ Water instead of tablets

➢ Spiked clean sand instead of natural sediment

➢ Primary focus on number of particles

Preparation of test materials

Water

Renkumse Beek

0.2 µm
Filtered

+ particles → QMP016FW

+ tablet → QMP017FW
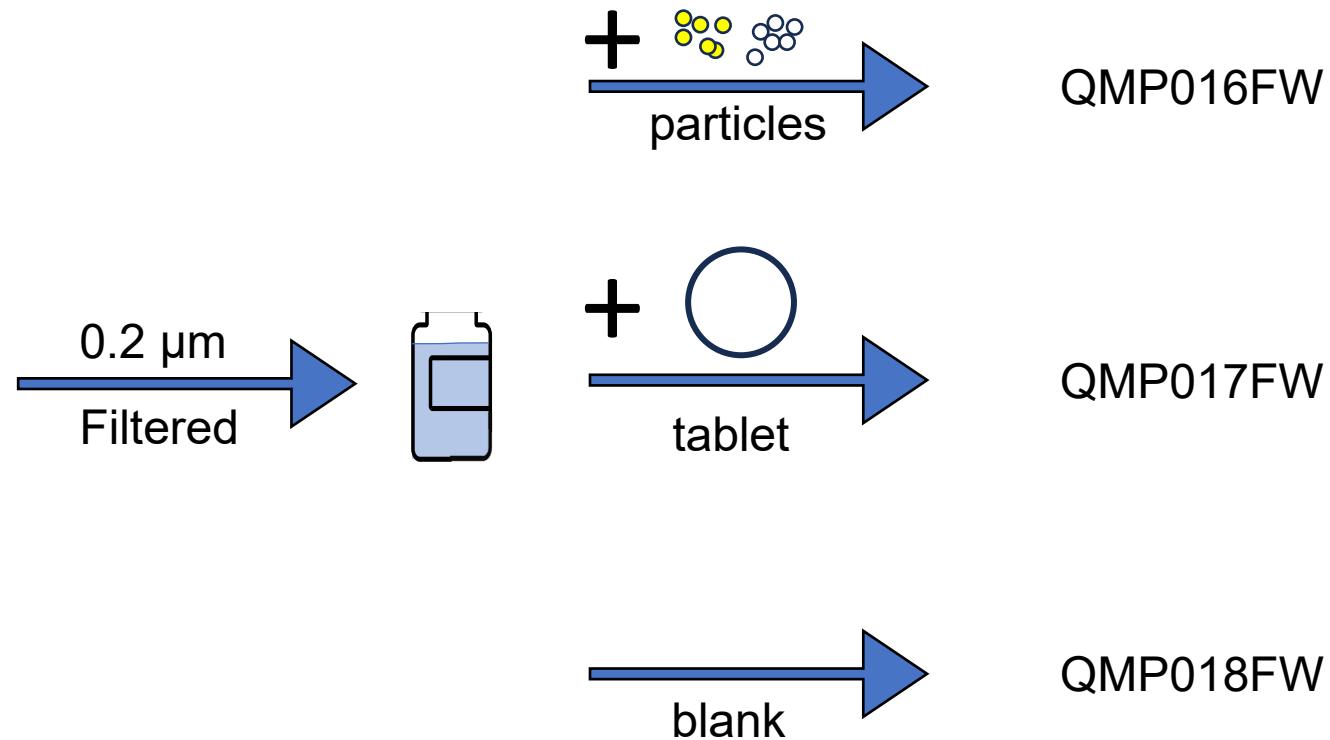
blank → QMP018FW

Preparation of test materials

Sand

construction market

600°C
4 hours

+ ○
tablet
→ QMP019MS

+ particles
→ QMP020MS   (counting)

+ particles
→ QMP022MS   (mass determination)

blank
→ QMP021MS

Test materials and polymers

Water

Sample nr	Sample type	Polymer type	Polymer Size (µm)	Conc (nr/L)	(mg/L)
QMP016FW	freshwater spiked with MP	PE (yellow) PMMA (White)	212-250 250-300	30 20	0.20 0.26
QMP017FW	freshwater spiked with MP (tablet)	PE,PET, PS	50-300	85	
QMP018FW	freshwater unspiked	X			

Sand

Sample nr	Sample type	Polymer type	Polymer Size (µm)	Conc (nr/kg)	(mg/kg)
QMP019MS	wet sand, spiked with MP (tablet)	PP, PVC, PC	50-300	1308	
QMP020MS	wet sand, spiked with MP	PE (yellow) PMMA (white) PE (red)	212-250 250-300 500-600	308 231 123	
QMP022MS (mass)	wet sand, spiked with MP	PE (yellow) PMMA (white) PMMA (white)	212-250 250-300 90-106		1.01 2.03 0.16
QMP021MS	wet sand, unspiked	X			

Participants

Round	Submitted (nr)		Submitted (mass)	
	Water	Sediment/sand	Water	Sediment/sand
2022	54	45	11	10
2024	34	25	7	6

Results - NDA rel. St.dev%

spike	number				mass			
	water		sand		water		sand	
	QMP016FW particcles	QMP017FW tablet	QMP019MS tablet	QMP020MS partcicles	QMP016FW partcicles	QMP017FW tablet	QMP019MS tablet	QMP022MS partcicles
PE (50-299 µm)	54	117		55	NAV	NAV		NAV
PE (300-5000 µm)				17				
PMMA (50-299 µm)	57			67	NAV			NAV
PET (50-299 µm)		102				NAV		
PS (50-299 µm)		69				NAV		
PP (50-299 µm)			140				NAV	
PVC (50-299 µm)			87				NAV	
PC (50-299 µm)			73				NAV	

➢ 2022: Number of particles:

NDA Rel st. dev. : Tablets: 76-96 %

Sediment: 66-125 %

Sand: > 100 %

Results - NDA rel. St.dev%

spike	number				mass			
	water		sand		water		sand	
	QMP016FW	QMP017FW	QMP019MS	QMP020MS	QMP016FW	QMP017FW	QMP019MS	QMP022MS
	partcicles	tablet	tablet	partcicles	partcicles	tablet	tablet	partcicles
PE (50-299 µm)	54	117		55	NAV	NAV		NAV
PE (300-5000 µm)				17				
PMMA (50-299 µm)	57			67	NAV			NAV
PET (50-299 µm)		102				NAV		
PS (50-299 µm)		69				NAV		
PP (50-299 µm)			140				NAV	
PVC (50-299 µm)			87				NAV	
PC (50-299 µm)			73				NAV	
PE (total)					87	183		NAV
PMMA (total)					107			NAV
PET (total)						191		
PS (total)						83		
PP (total))							129	
PVC (total)							199	
PC (total)							137	

n=4-6

Result vs spiked value (counting)

	number (p/kg)							
	water				sand			
	QMP016FW		QMP017FW		QMP019MS		QMP020MS	
	AV/Model		Model		Model			
	Mean	spiked	Mean	spiked	Mean	spiked	AV	spiked
PE (50-299 µm)	23	30	9				217	308
PE (300-5000 µm)							118	123
PMMA (50-299 µm)	11	20					163	231
PET (50-299 µm)			18					
PS (50-299 µm)			15					
PP (50-299 µm)					126			
PVC (50-299 µm)					138			
PC (50-299 µm)					156			
total				84		1308		

NAV-> model mean

Result vs spiked value (mass)

	mass (mg/kg)						
	water				**sand**		
	QMP016FW		**QMP017FW**				
	Model Mean	spiked	Model Mean	spiked	Model Mean	spiked	Model Mean	spiked
PE (total)	0.09	0.2	0.04				1.00	1.01
PMMA (total)	0.06	0.26					NA	2.19
PET (total)			0.01					
PS (total)			0.04					
PP (total))					0.79			
PVC (total)					0.19			
PC (total)					1.48			
total				≈ 0.27		≈ 4.11		

NAV-> model mean

Blank samples

water

PE (50-299 μm) ((No. p/kg)) $^\$$

Q101	-	-	1.00
Q102	40.0	40.0	20.0
Q104	25.0	2.00 <	2.00 <
Q110	24.0	4.00	-
Q130	17.0	20.0	2.00
Q134	34.0	2.00	-
Q153	9.00	7.00	-
Q221	68.0	8.00	2.00
Q3028	1.00	-	15.0
Q3759	-	12.0	-
Q3785	22.0	6.00	-
Q3800	5.00	1.00	1.00
Q3872	20.0	-	1.00
Q3873	188	10.0	-
Q3875	47.0	7.00	-
Q3878	29.0	24.0	5.00
Q3913	28.0	53.0	-
Q4026	13.0	9.00	18.0
Q4040	23.0	29.0	2.00
Q4057	18.0	-	26.0
Q4080	29.0	-	-
Q4132	22.0	-	-
Q4134	24.0	5.00	-
Q4135	64.0	35.0	-
Q4136	1.00	-	1.00
=================	Summary Statistics	======	
NDA mean	-	-	-
NDA st dev	11.93	10.136	1.438
Coeff Var (%)	54.2	117.1	93.3
N	25	19	12
Spiked Value	**30**	**+**	**0**

Blank samples

water

PE (50-299 µm) ((No. p/kg)) $^{\$}$

Q101	-	-	1.00
Q102	40.0	40.0	20.0
Q104	25.0	2.00 <	2.00 <
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Q130	17.0	20.0	2.00
Q134	34.0	2.00	-
Q153	9.00	7.00	-
Q221	68.0	8.00	2.00
Q3028	1.00	-	15.0
Q3759	-	12.0	-
Q3785	22.0	6.00	-
Q3800	5.00	1.00	1.00
Q3872	20.0	-	1.00
Q3873	188	10.0	-
Q3875	47.0	7.00	-
Q3878	29.0	24.0	5.00
Q3913	28.0	53.0	-
Q4026	13.0	9.00	18.0
Q4040	23.0	29.0	2.00
Q4057	18.0	-	26.0
Q4080	29.0	-	-
Q4132	22.0	-	-
Q4134	24.0	5.00	-
Q4135	64.0	35.0	-
Q4136	1.00	-	1.00
=================		Summary Statistics	======
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N	25	19	12
Spiked Value	30	+	0

Sample material issues

QMP017FW

Fresh water sample
-> + humic acid
-> filtered 0,2 µm

Originating from the tablets?

Sample issues

QMP016FW

"contains ideal yellow spheres (diam. approx. 0.2 mm) that could not be captured in transmission mode because they rolled during measurement"

Selection/ preparation of material issues

➢ Material for proficiency tests preferably naturally contaminated

→ not possible for MP because of inhomogeneity

➢ Manually spiking only possible for "bigger" particles (>200 µm)

➢ Limited types of polymers commercially available

➢ Spiking with tablets ->
- o Expensive
- o Not clear how much the tablet contains of each <u>individual</u> polymer
- o Mass of polymers not known
- o Formation of floating stuff in water

Conclusion

➤ Mass determination: low number of participants
- → Too low (4-6) to calculate assigned values
- → RSDs (total size class) 87-193 %

➤ Counting methods:
- → Performance for manually spiked better than for tablet spiked.
- → Performance for manually spiked test samples improved (NDA RSDs <70%)

➤ Recovery of spiked particles approx. 32-77% (except for the bigger particles (96%))

➤ Challenge to prepare proper test materials for MP.

Today: Online workshop to discuss the results and wishes for follow up study