



# Analysemetode: Kvantifisering av mikroplast i sediment

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Øyvind Lilleeng & Arne Pettersen

# Metoder for å kvantifisere mikroplast i sedimenter

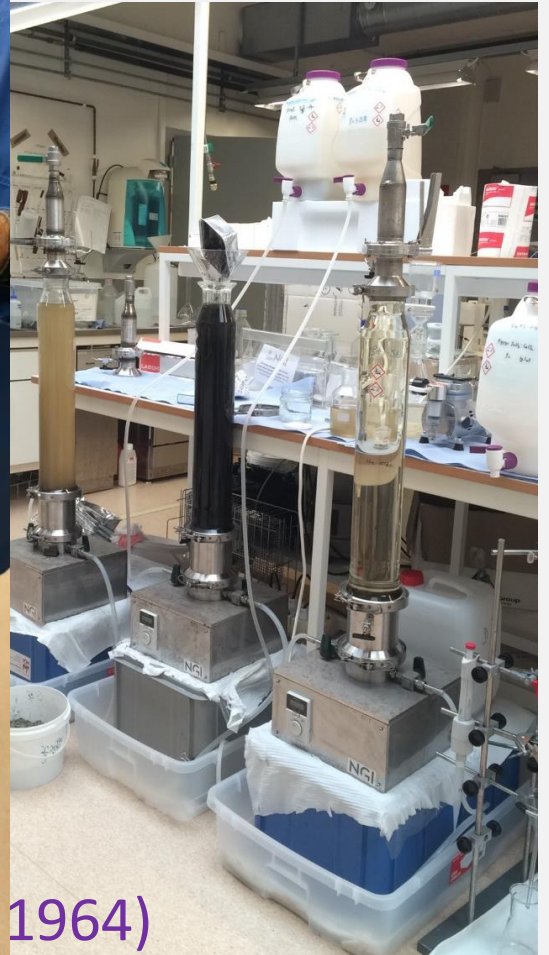
## Vanlige trinn

- 1. Prøvetaking (med eller uten sikting)
- 2. Tetthetsseparasjon for å fjerne mineraler
- 3. Filtrering
- 4. Fjerning/nedbrytning av organisk materiale som ikke er plast
- 5. Kvantifisering (vekt, antall partikler, type plast, osv.)

Alle trinn kan kontaminere prøven og ha en påvirkning på størrelse-fordeling av partiklene



# NGIs metode: te



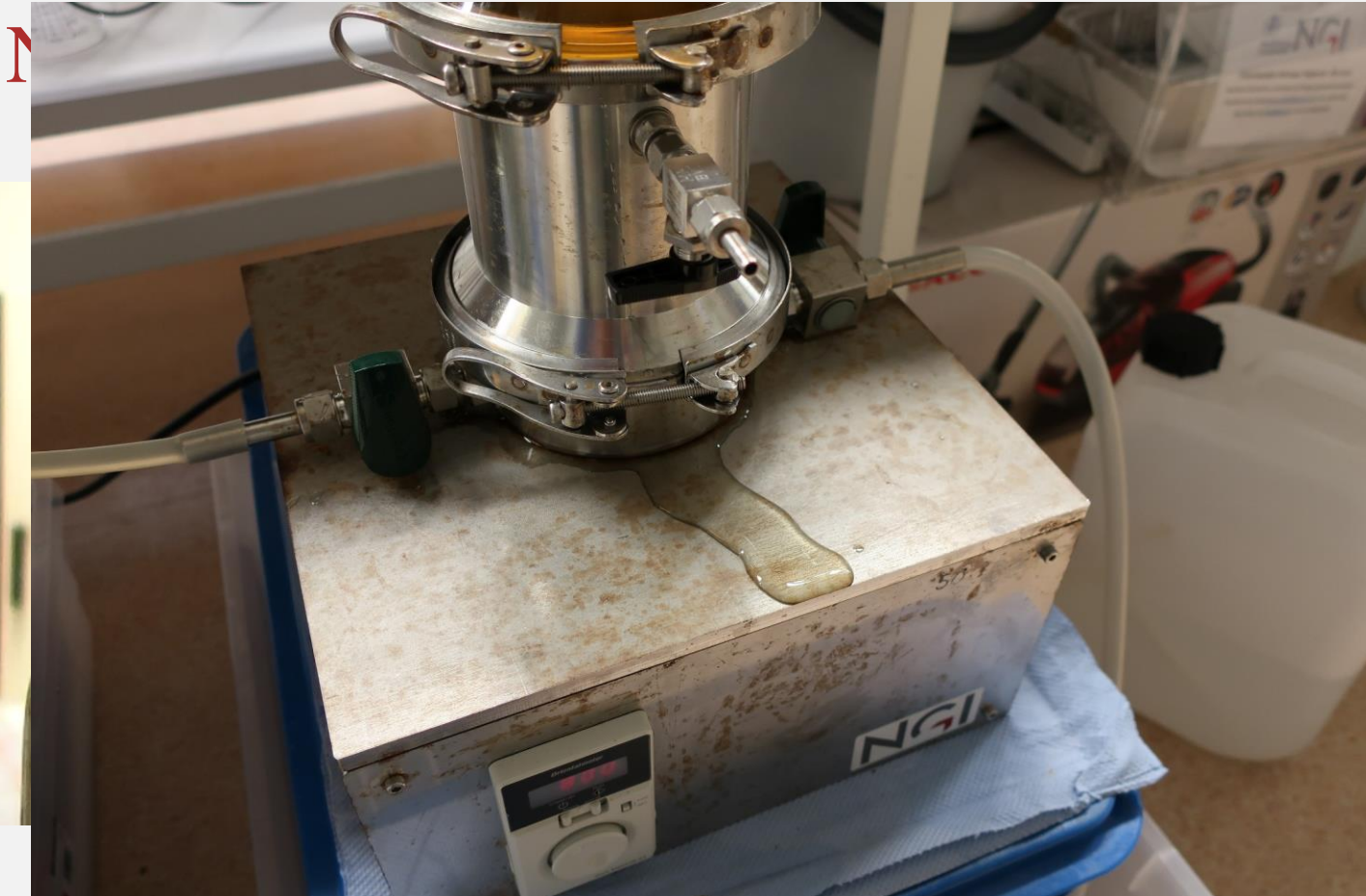
\*inspirert fra Münich  
Mikroplast Sediment  
Seperatoren (Imhof et al.)

4.4: 3.6: 2 kg ( $ZnCl_2:C$

NGI

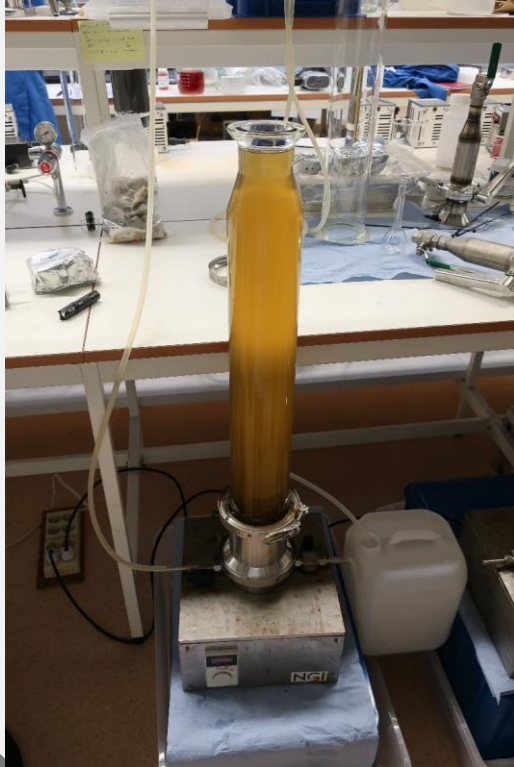
1964)

Sabnam Mahat M.Sc. 2017



al and  
float,  
s sink

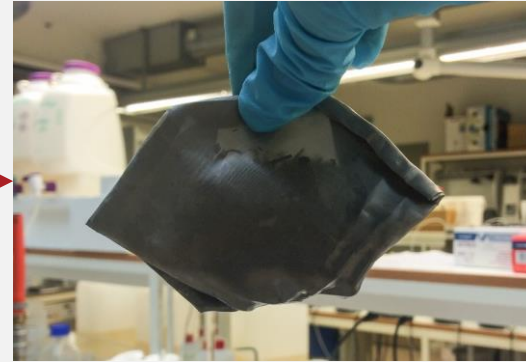
# Filtrering: Stålfiler (45 $\mu\text{m}$ )



# Filtrering av materiale med lav tetthet



rett fra Bauta til  
et stålfiler



↗ stålfiler (45  $\mu\text{m}$ )

# NGIs metode: 2 trinns våtkjemisk oksidasjon (24 timer)



Fjerner  $98 \pm 4$  % cellulose  
0% plastpellets  
0-4% plastfibre



↗ før

↗ etter

*hemmelig oppskrift, kommer snart*  
Olsen et al. in prep.

# Kvantifikasjon og kvalitetskontroll

➤ **Method blanks**-samme som vanlig separasjon, men uten sediment.

Blankene vil gi en god indikasjon av:

- Forurensing på laben,
- Forurensing under rengjøring
- Kvaliteten av gjenbrukt  $\text{ZnCl}_2:\text{CaCl}_2$

➤ **Spiked blanks**-

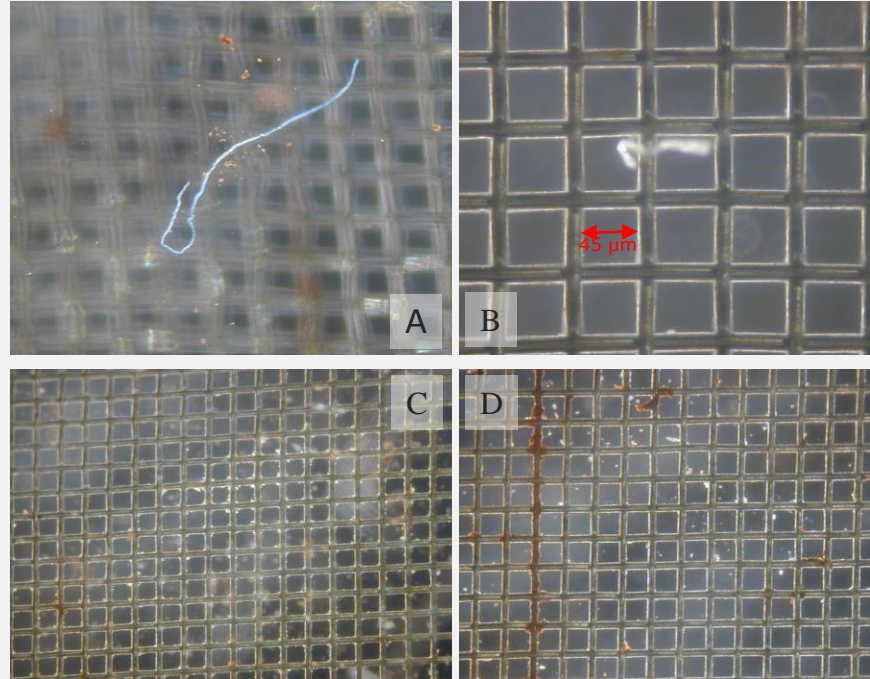
Presisjonen av metoden ble testet ved å tilføre en kjent mengde mikroplast (granulater, fibre og/eller mikropulver) til sedimentet hvor organisk materiale og mikroplast allerede var fjernet.



# Metodeblank Del 1

Kjørt 11 metodeblanker for vekt, 8 av dem brukt for telling.

Gjennomsnittlig vekt av plast  $0.0010 \pm 0.011$  g



# Metodeblank del 2, og system for telling ved mikroskopi

Mesteparten av materialet i metodeblankene var klare/hvite fibre og granulater < 100 µm

	Colour	Fibre 1D				Layer 2D				Granulat 3D			
		A	B	C	D	A	B	C	D	A	B	C	D
Average (n=8)	Clear/white	3.5	5.9	4.5	1.9	1.8	0.4	0.8	0.1	19.0	3.6	0.0	0.0
	Light brown	0.0	0.3	0.0	0.0	1.8	1.1	0.6	0.3	2.3	2.0	0.1	0.0
	Dark brown	0.0	0.1	0.0	0.0	0.3	0.0	0.0	0.0	0.9	0.3	0.3	0.0
	Black	0.1	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.4	0.0	0.0	0.0
	Blue	0.1	0.6	0.5	0.6	0.0	0.1	0.0	0.0	0.0	0.0	0.0	0.0
	Red	0.0	0.0	0.1	0.0	0.3	0.0	0.0	0.0	0.8	0.0	0.0	0.0
	Green	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Orange	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.3	0.1	0.0	0.0
	Yellow	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Sum*	3.8	6.9	5.1	2.5	4.0	1.6	1.4	0.4	23.5	6.0	0.4	0.0
	Fraction of total	18.3		32.9 %		7.4		13.3 %		29.9		53.8 %	
SUM										55.5			

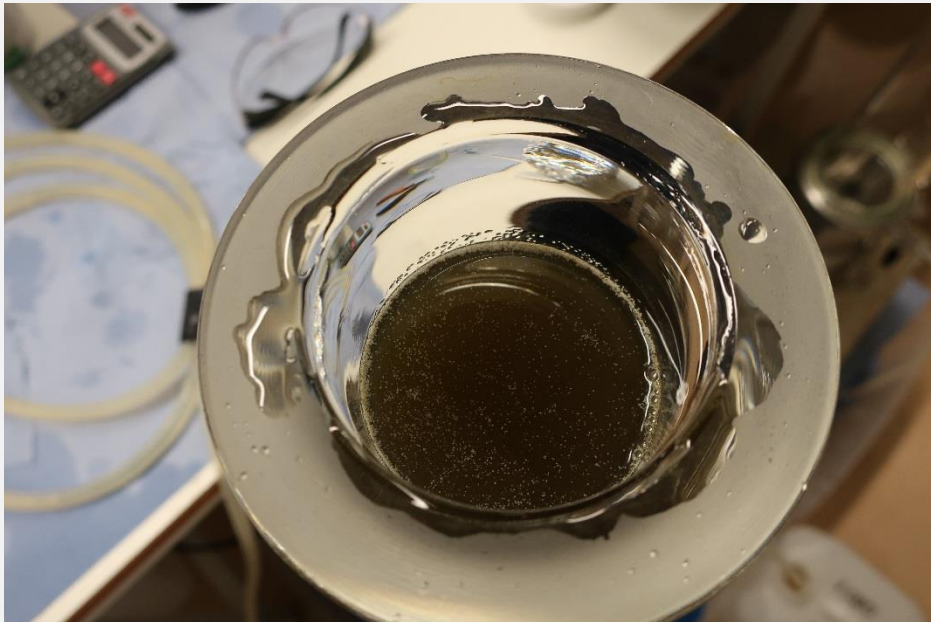
A = ≤ 45 – 100 µm

B = 100 – 300 µm

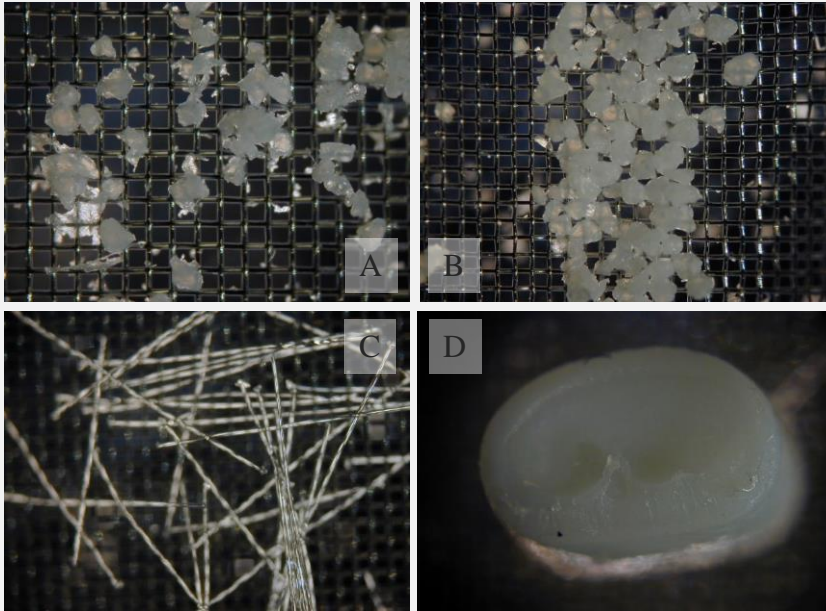
C = 300 – 1000 µm

D = 1000 – 5000 µm

# Spiked blanks

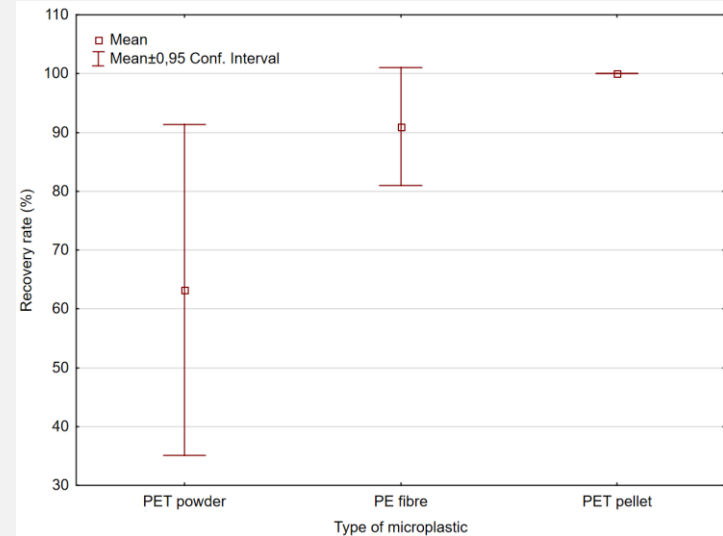


# Recovery (spiking) blank



Photos of spiking material (x10 magnification). A and B: PET powder (75-250 µm); C: PE fibre (1 cm x 16 µm); D: PET pellet (3 mm).

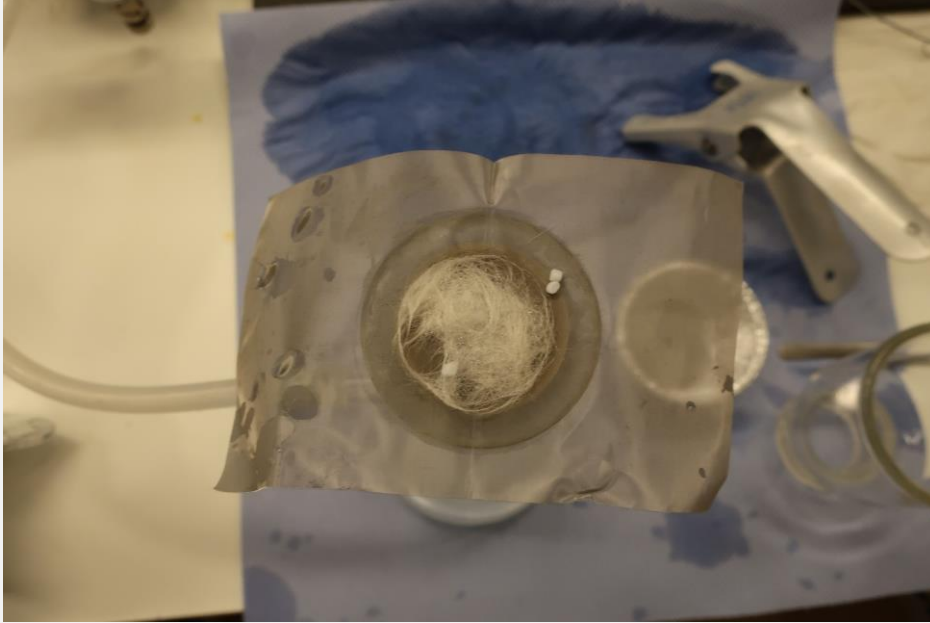
Recovery blank: kjent mengde plast tilsatt til en «ren» sedimentprøve (dvs. en sediment prøve etter tetthetsseperasjon)



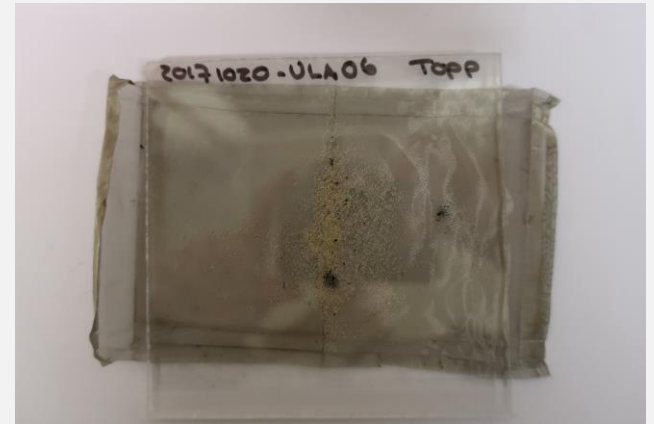
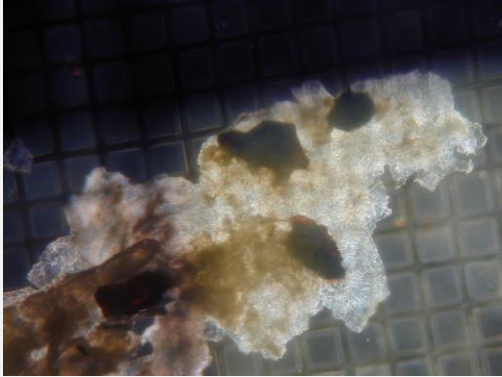
Brukt til å korrigere data:

$f_{\text{recovery}}$  for granulat og fiber = 0.77

## Mikroplast granulater (PET) (1.4 kg/L) og fibre



# Mikroskopi



- Potensiell mikroplast ble funnet i sedimentprøvene via visuell kvantifikasjon.
- For prøvene med relativt høy mikroplastkonsentrasjon på vekt basis, var antall partikler 'too many to count'.



**FT-IR**

Start (cm-1) 4000 End (cm-1) 600 Accumulations 16 Scans Administrator 15 Sample

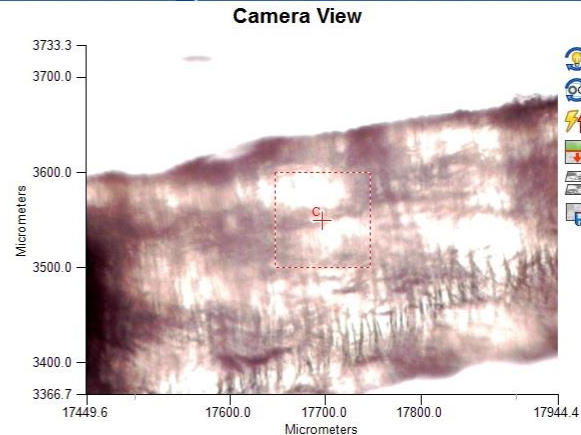
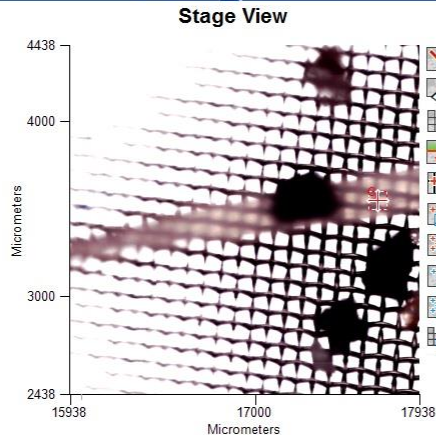


Ensure beam path is clear  
Press [Scan] to continue



## Data Explorer

- Microscope
- Sample Table
- Samples View 1
  - KV-02 Gul foam
- Samples View 2
  - Image View 1
  - Samples View 3
    - Kv-02 Gul partikkel
  - Image View 2
    - KV 02 Gul partikkel test\_(1)(1)
    - KV 02 Gul partikkel test\_(1)(2)
    - KV 02 Gul partikkel test\_(1)(3)
    - KV 02 Gul partikkel test\_(1)(4)
    - KV 02 Gul partikkel test\_(1)(5)
    - KV 02 Gul partikkel test\_(2)(1)
    - KV 02 Gul partikkel test\_(2)(2)
    - KV 02 Gul partikkel test\_(2)(3)
    - KV 02 Gul partikkel test\_(2)(4)
    - KV 02 Gul partikkel test\_(2)(5)
    - KV 02 Gul partikkel test\_(3)(1)
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    - KV 02 Gul partikkel test\_(3)(5)
    - KV 02 Gul partikkel test\_(4)(1)
    - KV 02 Gul partikkel test\_(4)(2)
    - KV 02 Gul partikkel test\_(4)(3)
    - KV 02 Gul partikkel test\_(4)(4)
    - KV 02 Gul partikkel test\_(4)(5)
    - KV 02 Gul partikkel test\_(5)(1)
    - KV 02 Gul partikkel test\_(5)(2)
    - KV 02 Gul partikkel test\_(5)(3)
    - KV 02 Gul partikkel test\_(5)(4)
    - KV 02 Gul partikkel test\_(5)(5)
    - KV 02 Fiber\_(1)(5)
- Samples View 4



Base Sample ID	Location (μm)			Points		Spacing (μm)		Apertures Width (μm)	Height (μm)	Rotation (°)	Display in graph	Save as FSM
	X	Y	Z	X	Y	X	Y					
Map Scan	10924	9103	-1413	5	5	4247	3712	100	100	0.00	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>

Setup Microscope Data Collection Setup Microscope Advanced Setup Microscope Basic

#### ATR

Crystal Position:

Auto-Pressure (Target: 5%)

5  100

Auto ATR

Crystal Up/Down Count: 44

#### Stage Control

Z-Axis Adjustment (μm):

1  50

5  100

10  200

Stage Lighting:

#### Sampling Mode

Reflectance

Transmittance

ATR

Illumination (39%)

## Setup

- Instrument
- Microscope
- Laboratory Scheduler
- Ready Checks
- Instrument Verification
- Export and Email
- Peak Detection
- View
- Pathlength
- Quant
- Compare
- Libraries and Search

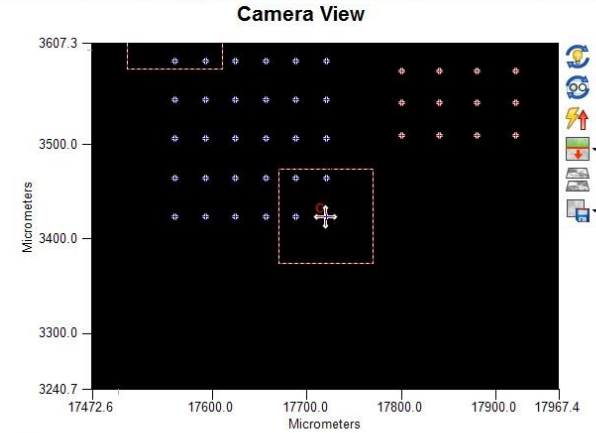
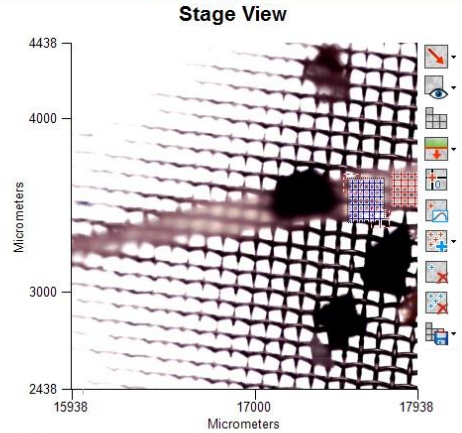
## Setup

## Spectral Libraries

## Equations



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    - Samples View 2
      - Image View 1
    - Samples View 3
      - Kv 02 Gul partikkel
      - Image View 2
        - KV 02 Gul partikkel test\_1(1)(1)
        - KV 02 Gul partikkel test\_1(1)(2)
        - KV 02 Gul partikkel test\_1(1)(3)
        - KV 02 Gul partikkel test\_1(1)(4)
        - KV 02 Gul partikkel test\_1(1)(5)
        - KV 02 Gul partikkel test\_2(1)(1)
        - KV 02 Gul partikkel test\_2(2)(1)
        - KV 02 Gul partikkel test\_2(2)(2)
        - KV 02 Gul partikkel test\_2(2)(3)
        - KV 02 Gul partikkel test\_2(2)(4)
        - KV 02 Gul partikkel test\_2(2)(5)
        - KV 02 Gul partikkel test\_2(3)(1)
        - KV 02 Gul partikkel test\_2(3)(2)
        - KV 02 Gul partikkel test\_2(3)(3)
        - KV 02 Gul partikkel test\_2(3)(4)
        - KV 02 Gul partikkel test\_2(3)(5)
        - KV 02 Gul partikkel test\_2(4)(1)
        - KV 02 Gul partikkel test\_2(4)(2)
        - KV 02 Gul partikkel test\_2(4)(3)
        - KV 02 Gul partikkel test\_2(4)(4)
        - KV 02 Gul partikkel test\_2(4)(5)
        - KV 02 Gul partikkel test\_2(5)(1)
        - KV 02 Gul partikkel test\_2(5)(2)
        - KV 02 Gul partikkel test\_2(5)(3)
        - KV 02 Gul partikkel test\_2(5)(4)
        - KV 02 Gul partikkel test\_2(5)(5)
    - Samples View 4
      - Image View 3
        - Map Scan\_(1)(1)\_001
        - Map Scan\_(1)(2)\_001
        - Map Scan\_(1)(3)\_001
        - Map Scan\_(1)(4)\_001
        - Map Scan\_(2)(1)\_001
        - Map Scan\_(2)(2)\_001
        - Map Scan\_(2)(3)\_001
        - Map Scan\_(2)(4)\_001
        - Map Scan\_(3)(1)\_001
        - Map Scan\_(3)(2)\_001
        - Map Scan\_(3)(3)\_001
        - Map Scan\_(3)(4)\_001
        - Map Scan\_(4)(1)\_001
        - Map Scan\_(4)(2)\_001
        - Map Scan\_(4)(3)\_001
        - Map Scan\_(4)(4)\_001
        - Map Scan\_(5)(1)\_001
        - Map Scan\_(5)(2)\_001



Base Sample ID	Location (µm)		Z	Points		Spacing (µm)		Apertures			Rotation (°)	Display in graph	Save as FSM
	X	Y		X	Y	X	Y	Width (µm)	Height (µm)				
Map Scan	17560	3630	-1581	6	6	32	41	100	100	0.00	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	

**ATR**

Crystal Position:

Auto-Pressure (Target: 5%):  100

Auto ATR

Crystal Up/Down Count: 44

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Z-Axis Adjustment (µm):

1  50

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Auto-Focus:

Stage Lighting:

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- Spectral Libraries
- Equations

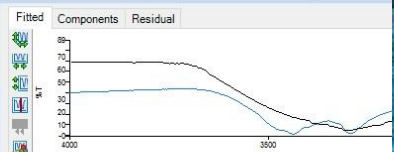
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        - KV 02 Gul partikkel test\_(4)(4)
        - KV 02 Gul partikkel test\_(4)(5)
        - KV 02 Gul partikkel test\_(5)(1)
        - KV 02 Gul partikkel test\_(5)(2)
        - KV 02 Gul partikkel test\_(5)(3)
        - KV 02 Gul partikkel test\_(5)(4)
        - KV 02 Gul partikkel test\_(5)(5)
        - KV 02 Fiber\_(1)(5)
    - Samples View 4
      - Image View 3
        - Map Scan\_(1)(1)\_001
        - Map Scan\_(1)(2)\_001
        - Map Scan\_(1)(3)\_001
        - Map Scan\_(1)(4)\_001
        - Map Scan\_(2)(1)\_001
        - Map Scan\_(2)(2)\_001
        - Map Scan\_(2)(3)\_001
        - Map Scan\_(2)(4)\_001
        - Map Scan\_(3)(1)\_001
        - Map Scan\_(3)(2)\_001
        - Map Scan\_(3)(3)\_001
        - Map Scan\_(3)(4)\_001
        - Map Scan\_(4)(1)\_001
        - Map Scan\_(4)(2)\_001
        - Map Scan\_(4)(3)\_001
        - Map Scan\_(4)(4)\_001
        - Map Scan\_(5)(1)\_001
        - Map Scan\_(5)(2)\_001

MultiSearch Results:

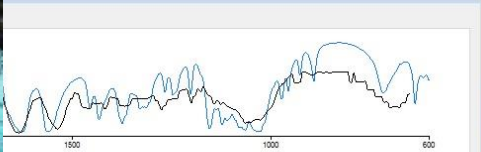
Sample Name	MultiSearch Best Hit Spectra	MultiSearch Best Hit Description	Level (%)
54 Map Scan_(5)(6)	PC0149	F02593.SP F02593POLYAMIDE 6 DF. FOR THIN LAYER CHROMATO-GRAPHY	100
55 Map Scan_(6)(1)	PC2568	F76200.SP F76200-PANTHENOL* BIOCHEMIKA C9H19NO4	100
56 Map Scan_(6)(2)	PC0149	F02593.SP F02593POLYAMIDE 6 DF. FOR THIN LAYER CHROMATO-GRAPHY	100
57 Map Scan_(6)(3)	PC2568	F76200.SP F76200-PANTHENOL* BIOCHEMIKA C9H19NO4	100
58 Map Scan_(6)(4)	PC0149	F02593.SP F02593POLYAMIDE 6 DF. FOR THIN LAYER CHROMATO-GRAPHY	100
59 Map Scan_(6)(5)	PC0149	F02593.SP F02593POLYAMIDE 6 DF. FOR THIN LAYER CHROMATO-GRAPHY	100

MultiSearch Hit List

Score	Spectra	Level (%)	Description	Cumulative Correlati
1 0.690872	PC0149	100	F02593.SP F02593POLYAMIDE 6 DF. FOR THIN LAYER CHROMATO-GRAPHY	87.1735
2 0.665552	SP0025 PC1160	98.7407 1.2593	NYLON 6/6 (POLYHEXAMETHYLENE ADIPAMIDE) F30477.SP F30477DEAE-CELLULOSE. 100-200 UM* BIOCHEMIKA	85.3707 94.2326
3 0.663066	SP0027 PC1160	97.1977 2.8023	NYLON 9/10 (POLYHEXAMETHYLENE SEBACAMIDE) F30477.SP F30477DEAE-CELLULOSE. 100-200 UM* BIOCHEMIKA	84.1068 94.8295
		7.3398 92.6602	F30477.SP F30477DEAE-CELLULOSE. 100-200 UM* BIOCHEMIKA	83.2165 94.2498
		100	NYLON 11 (POLYUNDECANOAMIDE) F76200.SP F76200-PANTHENOL* BIOCHEMIKA C9H19NO4	89.0287



Name	Description
Map Scan_(6)(6)	Sample at 17720.00 um (X), 34
Fitted spectrum from the ...	[COS:0.93313971]Fitted spectr



Name	Description	Write Pr			
Library 1					
FLUKA					
ATRSP1					
POLYMER					
TRANSM1					
				C:\pel_data\libs\POLYMER.DLB	
				C:\pel_data\libs\TRANSM1.DLB	

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Takk for  
oppmerksomheten



#påsikkergrunn