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Analytical and Characterisation Excellence

Analytical and Characterisation Excellence in nanomaterial risk assessment: A tiered approach

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Project highlights

- **Analytical innovation** in non-existent or poorly developed techniques
- **Optimisation** in existing techniques/instrumentation
- **Benchmarking/standardisation** in well developed techniques
- **Training:** Three layer model: core cohort of experts from the consortium, community training events, and online training tools
- **Data solutions:** to guide users (specially SMEs) through selection of the most appropriate methods to address their needs in risk assessment

Outline

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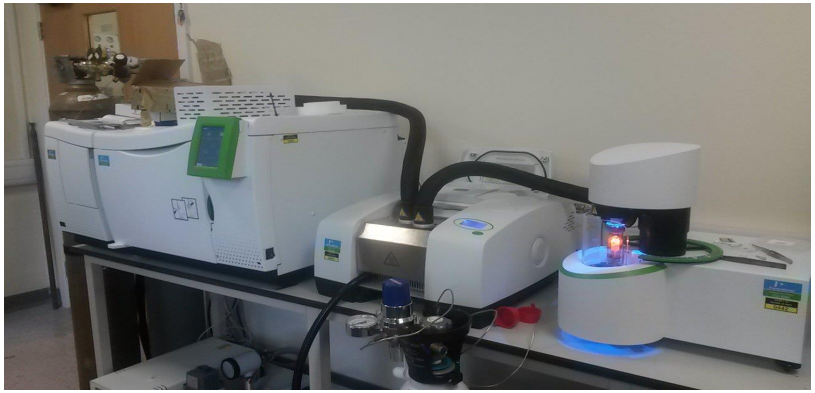
- Particle characterisation
  - spICP-MS
  - scICP-MS

- Polymer characterisation
  - TGA-FTIR-GC-MS



- Bio-nano interaction analysis
  - CE-MS/MS

Single Cell ICP-MS

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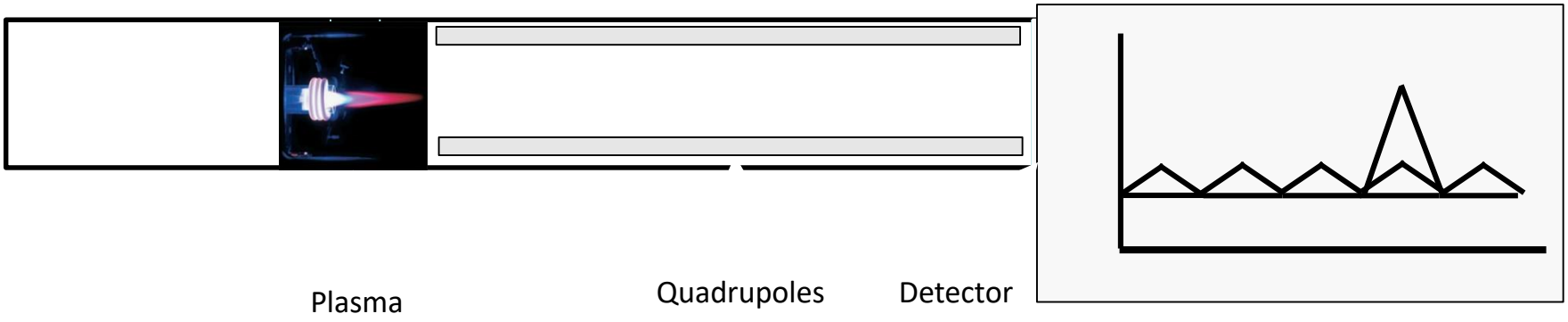
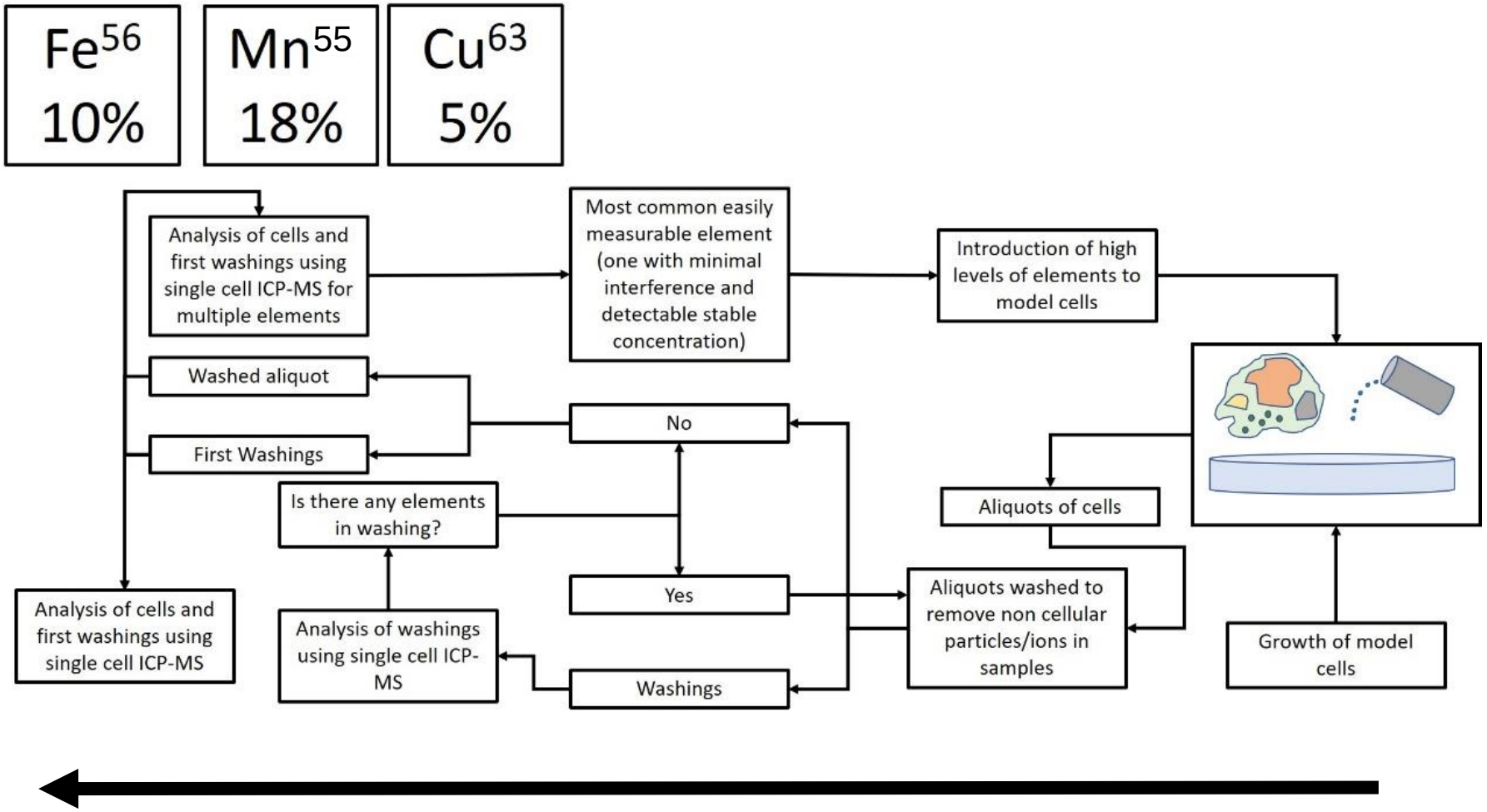


- Sc/spICP-MS is a form of ICP-MS with a rapid acquisition rate
  - Individual events at the detector treated as a NP or cell
- scICP-MS uses a novel nebuliser to maintain the integrity of the cell until it reaches the plasma

FENAC
FACILITY FOR ENVIRONMENTAL NANOSCIENCE ANALYSIS AND CHARACTERISATION

NERC
SCIENCE OF THE ENVIRONMENT

Work flow for single cell ICP-MS



Intrinsic metal analysis of A549 lung epithelial cells.

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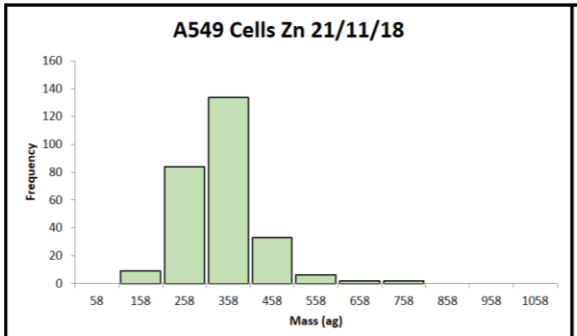
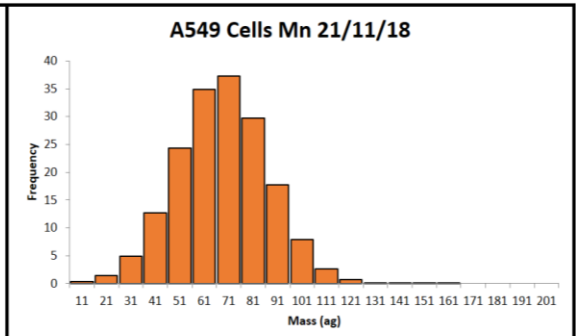
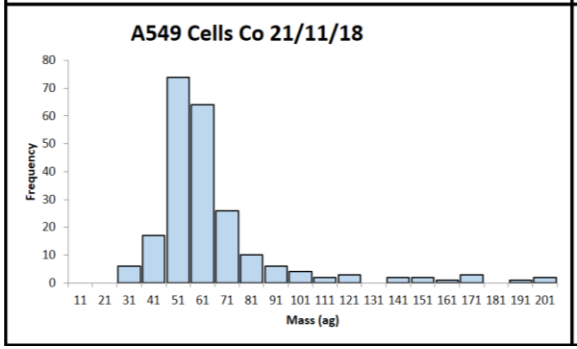
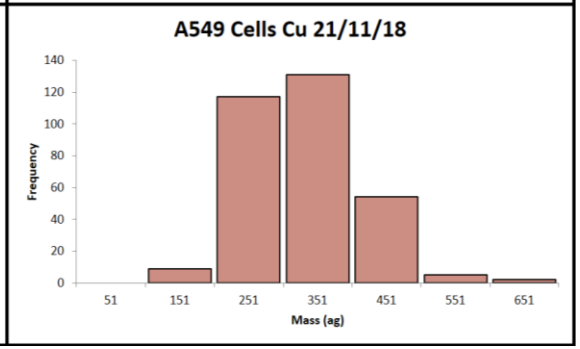
	A549 Cell Sample 21/11/18		A549 Cell Sample 26/06/19	
Element	Cell Concentration (cells per cm^3)	Mean Mean Mass (ag)	Cell Concentration (cells per cm^3)	Mean Mass (ag)
Zn 65.926	49,404	345	82,683	724
Cu 64.9278	57,973	331	N/A	N/A
Mn 54.9381	42,298	119	85,318	241
Co 58.9332	42,295	79	99,917	118
Average	47,993		89,306	
Standard Deviation	7,450		9,283	
Haemocytometer Count	42,917		92,500	









TGA-FTIR-GC-MS

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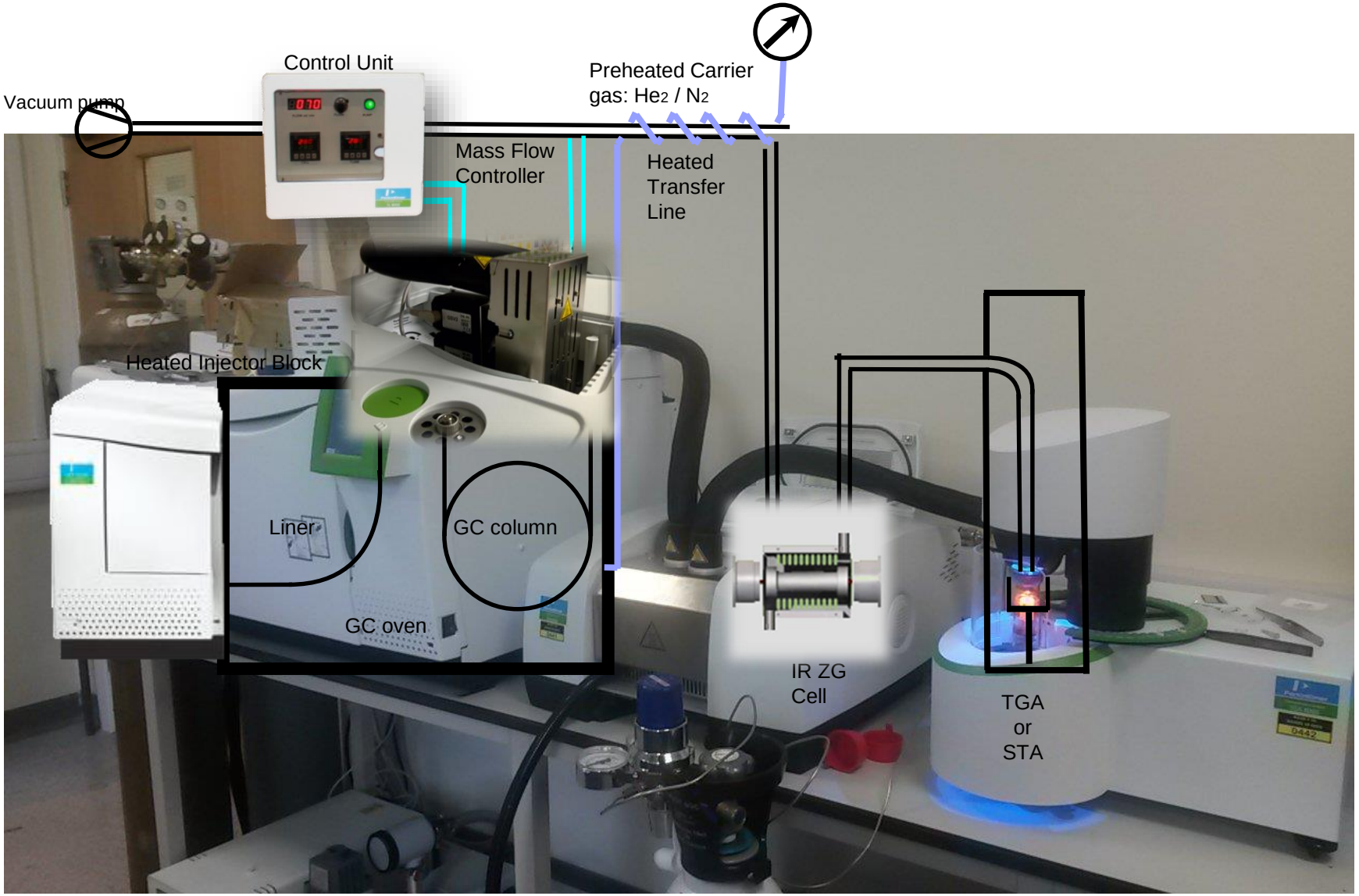
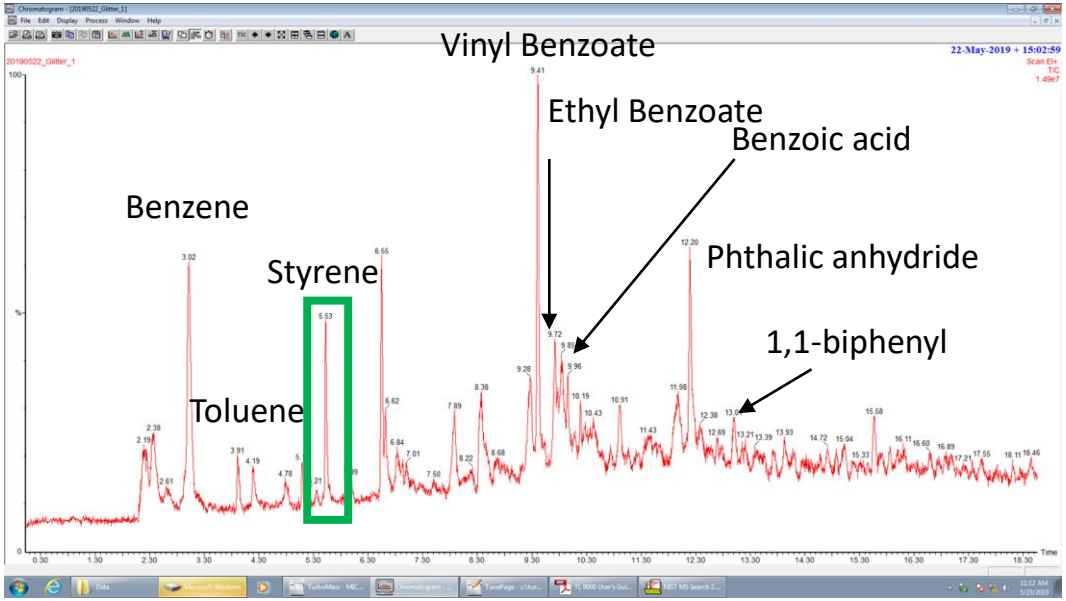
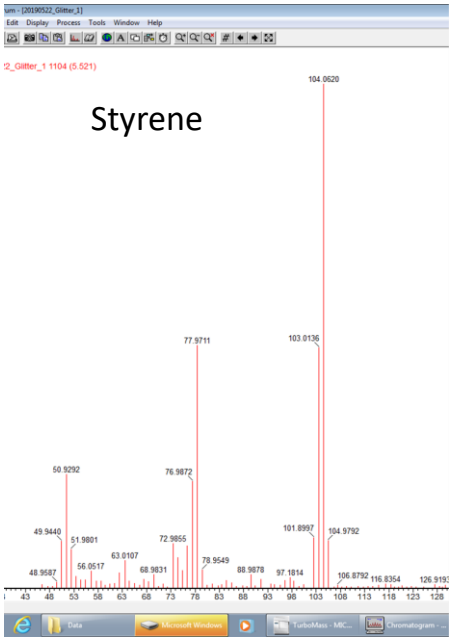


Example GC-MS chromatograms and spectra





GC-MS of glitter: Tell-tale peaks vinyl benzoate, ethyl benzoate, benzoic acid and 1,1-biphenyl confirm that the glitter is PET, care must be taken
as not all peaks are indicative of the polymer for example the styrene (benzene and toluene too)peak seen here is seen in many plastics and does
not mean polystyrene is present

Application to unknown mesoplastics

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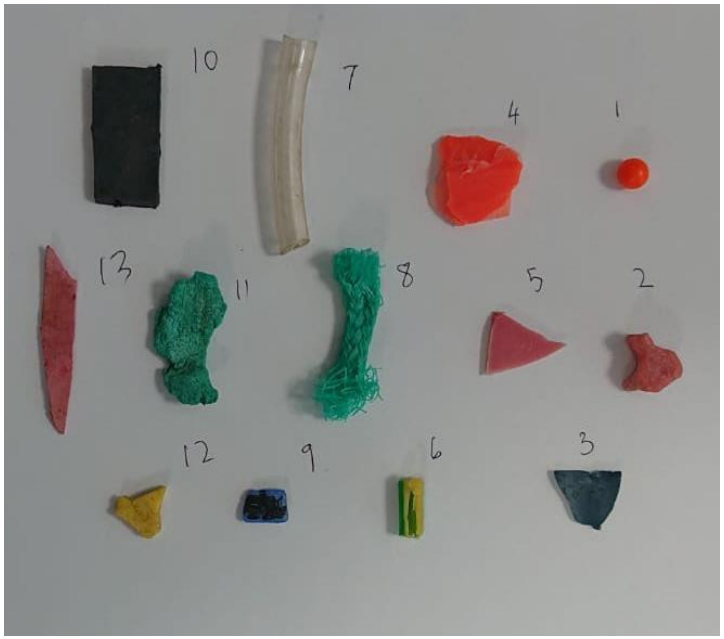
- Meso-plastic waste collected from Lowestoft beach
  - TGA was unable to definitively ID all of the plastics
  - FTIR ID'd everything apart from one non-plastic which as identified HDPE
  - GC-MS definitively identified all polymers using unique peaks
- Gas phase FTIR eliminates effect of black or fluorescent polymers

Sample ID	TGA	FTIR	GC-MS	Polymer
Beach plastic 1	No	Yes	Yes	Polystyrene
Beach plastic 2	No	Yes	Yes	Polyethylene
Beach plastic 3	Yes	Yes	Yes	Polypropylene
Beach plastic 4	No	Yes	Yes	Polystyrene
Beach plastic 5	Yes	Yes	Yes	Polypropylene
Beach plastic 6	Yes	Yes	Yes	Polyethylene
Beach plastic 7	Yes	Yes	Yes	Polyvinylchloride
Beach plastic 8	Yes	Yes	Yes	Polyethylene
Beach plastic 9	Yes	Yes	Yes	Polyethylene
Beach plastic 10	Yes	Yes	Yes	Polyethylene
Beach plastic 11	No	Incorrectly says HDPE	No	Not plastic
Beach plastic 12	Yes	Yes	Yes	Polyethylene
Beach plastic 13	No	Yes	Yes	Polyethylene

Yes and no refer to if the individual instruments in the hyphenated system can identify to polymer



Introduction to CE

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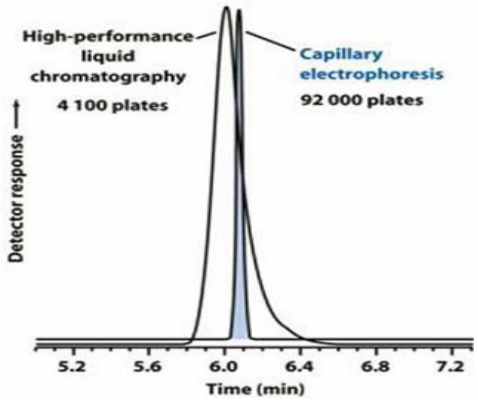
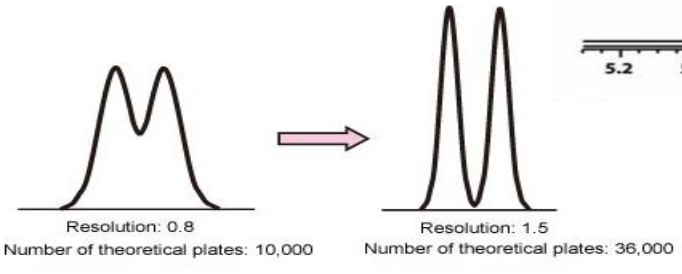
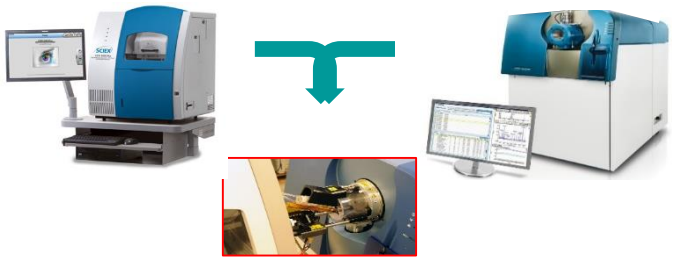
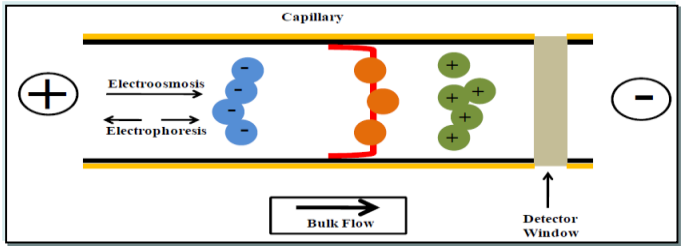
- Basis of separation is the differential migration of molecules in an applied electric field
- Electrophoresis, *not* chromatography
  - ✓ ***Orthogonal/complementary*** to LC
- Exceptional Resolving Power
  - ✓ Peak efficiencies > 1,000,000 theoretical plates
- Small nano liter injection volumes
- Separation flexibility
  - ✓ capillary environment
  - ✓ buffer selection and compatibility
- Automated, quantitative technique
- Capillary Electrophoresis is the movement of charged or polar molecules inside a capillary, filled with conductive fluid under the influence of an uniform electric field









Application to protein corona

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Protein	PS-Carb		PS		TI		TI-PVP		TI-Dispex		Silica	
	%	stdev	%	stdev	%	stdev	%	stdev	%	stdev	%	stdev
Fibrinogens	13%	0.2%	6%	1.2%	43%	1.1%	41%	0.8%	9%	0.6%	17%	2.9%
Apolipoproteins	5%	1.1%	3%	0.5%	0%	0.0%	1%	0.1%	0%	0.0%	17%	5.7%
Complement components	7%	1.5%	1%	0.6%	8%	0.7%	6%	0.3%	8%	0.3%	12%	1.9%
Immunoglobulin	1%	0.2%	2%	0.6%	22%	2.5%	20%	2.8%	7%	1.3%	12%	0.2%
Serum albumin	15%	0.6%	61%	1.7%	1%	0.0%	0%	0.0%	1%	0.1%	8%	1.4%
Vitronectin	32%	1.2%	10%	0.4%	5%	0.4%	8%	1.2%	14%	0.4%	2%	0.3%
Clusterin	1%	0.2%	10%	0.5%	0%	0.0%	0%	0.0%	0%	0.0%	1%	0.0%
Inter-alpha-trypsin inhibitor	3%	0.3%	1%	0.1%	1%	0.5%	1%	0.0%	0%	0.1%	4%	0.5%
Kininogen-1	3%	0.0%	0%	0.0%	1%	0.1%	3%	0.6%	2%	0.4%	3%	0.3%
Histidine-rich glycoprotein	7%	2.5%	0%	0.0%	1%	0.2%	1%	0.1%	0%	0.0%	1%	0.1%
Alpha-2-HS-glycoprotein	0%	0.1%	0%	0.1%	2%	0.6%	4%	0.7%	22%	2.0%	1%	0.3%
Prothrombin	0%	0.2%	0%	0.0%	5%	0.4%	7%	0.6%	21%	0.3%	0%	0.0%
Serotransferrin	0%	0.0%	0%	0.1%	0%	0.0%	0%	0.0%	0%	0.0%	4%	1.7%
Plasminogen	0%	0.1%	0%	0.1%	2%	0.0%	2%	0.2%	1%	0.1%	2%	0.5%
Gelsolin O	0%	0.2%	0%	0.0%	1%	0.3%	2%	0.1%	1%	0.0%	1%	0.2%
Beta-2-glycoprotein 1	1%	0.3%	0%	0.1%	1%	0.0%	0%	0.0%	0%	0.1%	1%	0.0%
Vitamin D-binding protein	0%	0.1%	2%	1.6%	0%	0.0%	0%	0.0%	0%	0.0%	0%	0.0%
Vitamin K-dependent protein S	0%	0.0%	0%	0.0%	1%	0.1%	1%	0.1%	1%	0.1%	0%	0.0%
Plasma kallikrein	1%	0.1%	0%	0.0%	1%	0.1%	1%	0.1%	1%	0.2%	0%	0.0%
Hemopexin	0%	0.0%	0%	0.0%	0%	0.0%	0%	0.0%	0%	0.0%	2%	0.2%
others	9%	0.3%	3%	0.5%	6%	1.0%	5%	0.0%	9%	0.5%	14%	1.3%

Colour code:	0%	5%	10%	30%	60%

Faserl et al, Nanomaterials, 2019

Acknowledgments

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- University of Birmingham
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UNIVERSITY OF BIRMINGHAM

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SCIEX

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