Infrared and *still* green:
Applying the Spotlight 400 FT-IR system to microplastics research within an environmental NGO

Dr David Santillo

Greenpeace Research Laboratories

University of Exeter

Greenpeace Research Laboratories: analytical capabilities

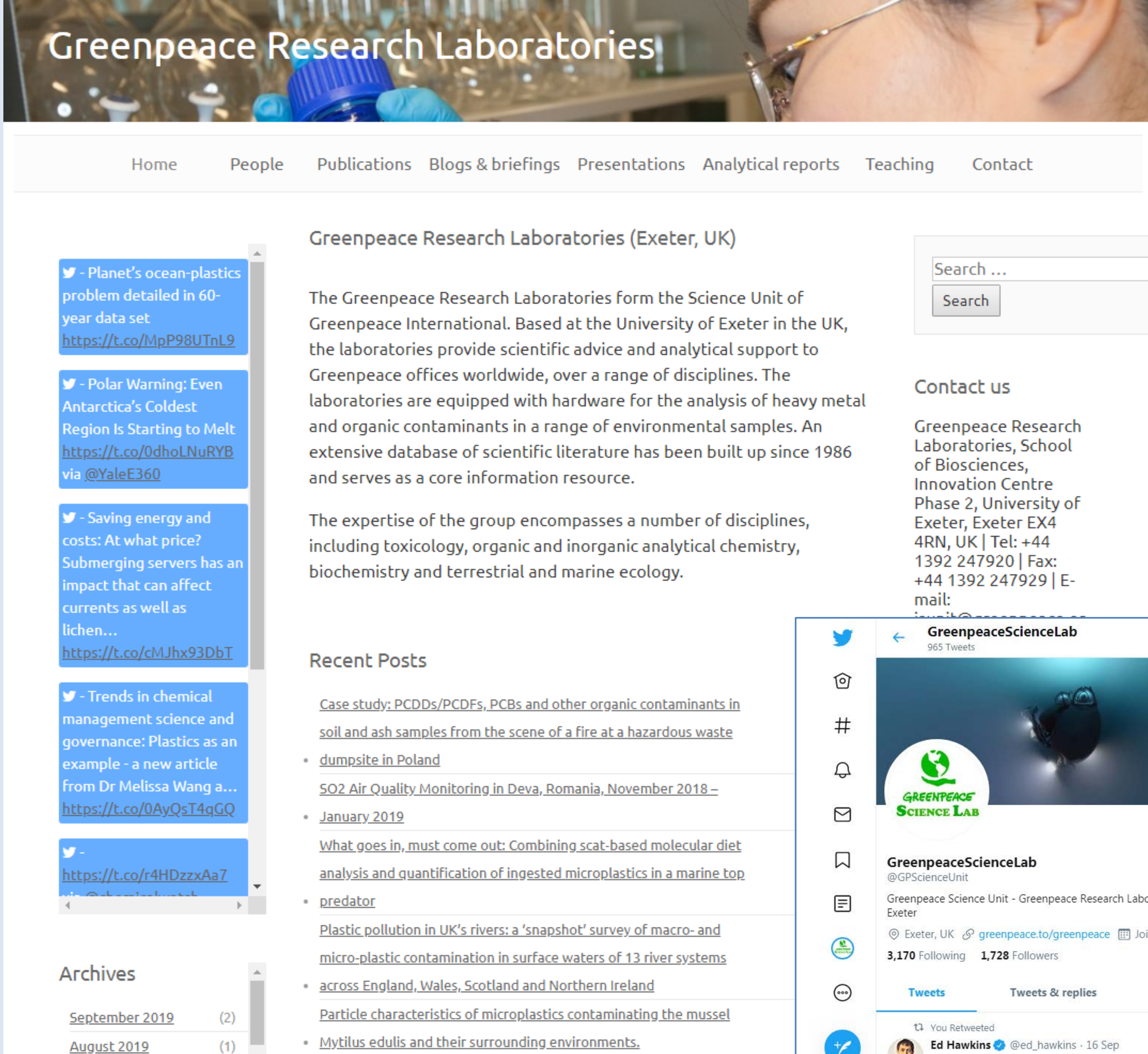
- GC-MS (persistent organic pollutants)
- LC-MS (POPs and pesticides)
- ICP-MS (toxic metals)
- FT-IR (plastics)
- Field equipment
- Radiation protection equipment and advice
- Working relationships with many leading laboratories

Greenpeace Research Laboratories (Science Unit) Mission Statement

- provide scientific advice, research and analytical support
- oversee best scientific practice, quality control and scientific communications
- to engage with the wider scientific community
- to help identify and respond to new and emerging issues/risks
- to represent Greenpeace at the science-policy interface

To conduct scientific research to inform Greenpeace's campaigns…

…'*bearing witness' through science*


Greenpeace Research Laboratories

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- Planet's ocean-plastics problem detailed in 60-year data set https://t.co/MpP98UTnL9
- Polar Warning: Even Antarctica's Coldest Region Is Starting to Melt https://t.co/0dhoLNuRYB via @YaleE360
- Saving energy and costs: At what price? Submerging servers has an impact that can affect currents as well as lichen… https://t.co/cMJhx93DbT
- Trends in chemical management science and governance: Plastics as an example - a new article from Dr Melissa Wang a… https://t.co/0AyQsT4qGQ
- https://t.co/r4HDzzxAa7

Archives

September 2019 (2)
August 2019 (1)

Greenpeace Research Laboratories (Exeter, UK)

The Greenpeace Research Laboratories form the Science Unit of Greenpeace International. Based at the University of Exeter in the UK, the laboratories provide scientific advice and analytical support to Greenpeace offices worldwide, over a range of disciplines. The laboratories are equipped with hardware for the analysis of heavy metal and organic contaminants in a range of environmental samples. An extensive database of scientific literature has been built up since 1986 and serves as a core information resource.

The expertise of the group encompasses a number of disciplines, including toxicology, organic and inorganic analytical chemistry, biochemistry and terrestrial and marine ecology.

Recent Posts

- Case study: PCDDs/PCDFs, PCBs and other organic contaminants in soil and ash samples from the scene of a fire at a hazardous waste dumpsite in Poland
- SO2 Air Quality Monitoring in Deva, Romania, November 2018 – January 2019
- What goes in, must come out: Combining scat-based molecular diet analysis and quantification of ingested microplastics in a marine top predator
- Plastic pollution in UK's rivers: a 'snapshot' survey of macro- and micro-plastic contamination in surface waters of 13 river systems across England, Wales, Scotland and Northern Ireland
- Particle characteristics of microplastics contaminating the mussel Mytilus edulis and their surrounding environments.

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Contact us

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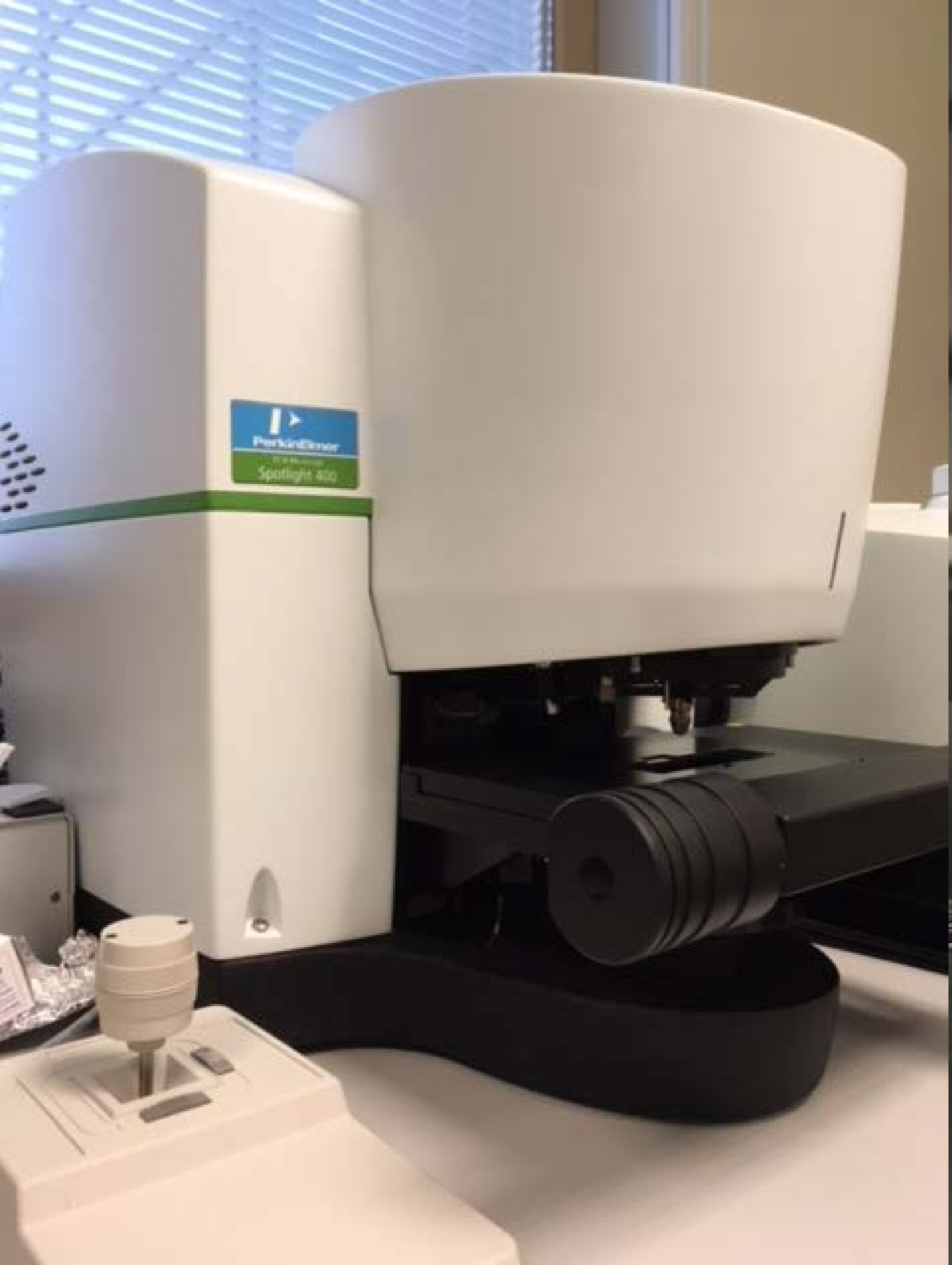
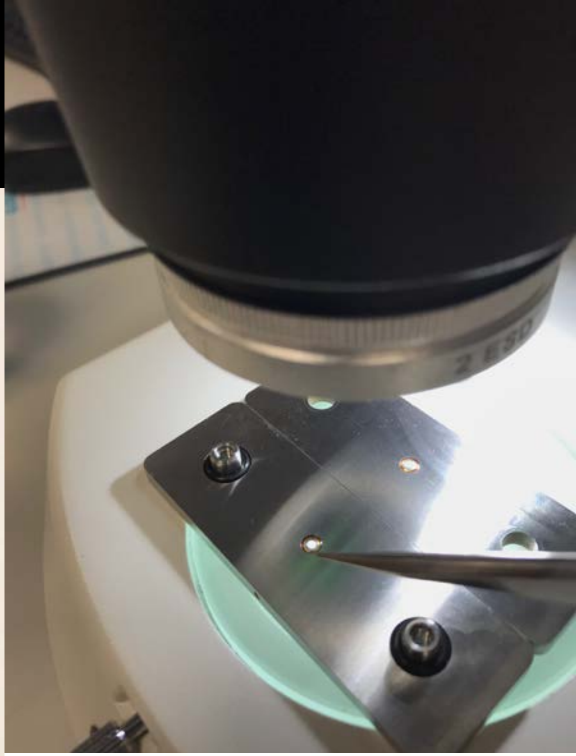
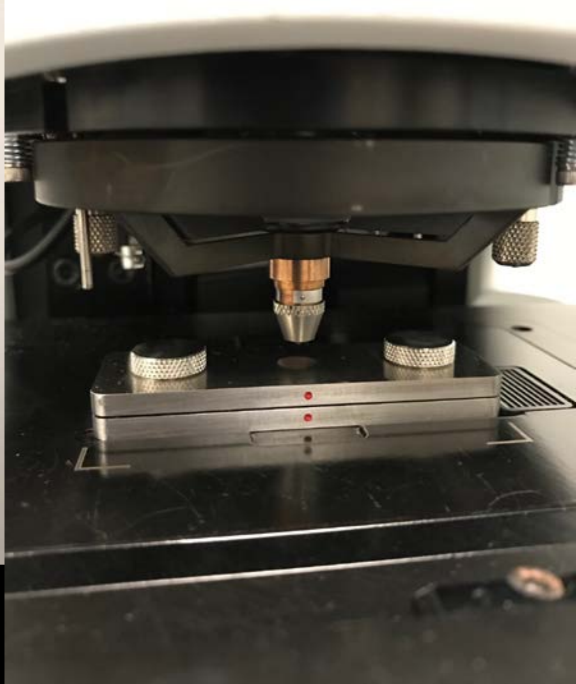

PerkinElmer
Spotlight 400


CRYSTAL S/N 30
HEIGHT 14090

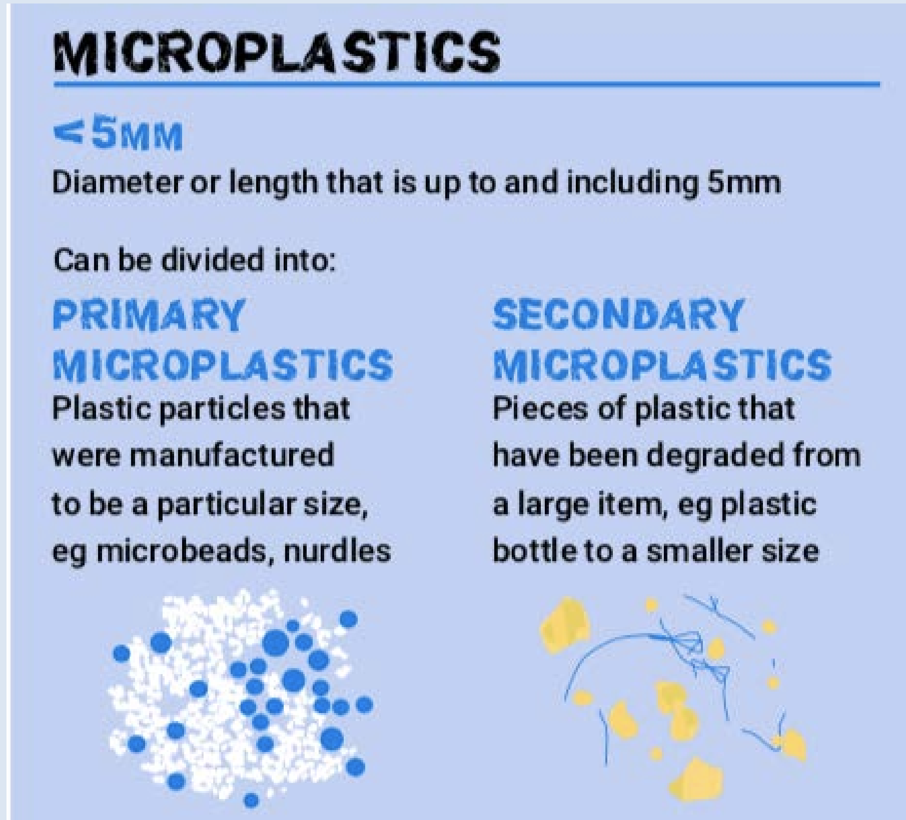
Diamond compression cell






Kimcare
Ethanol

Plastics pollution: from macro to micro…



(…to nano…though not with FT-IR)

Some key challenges in FT-IR analysis of environmental samples #1

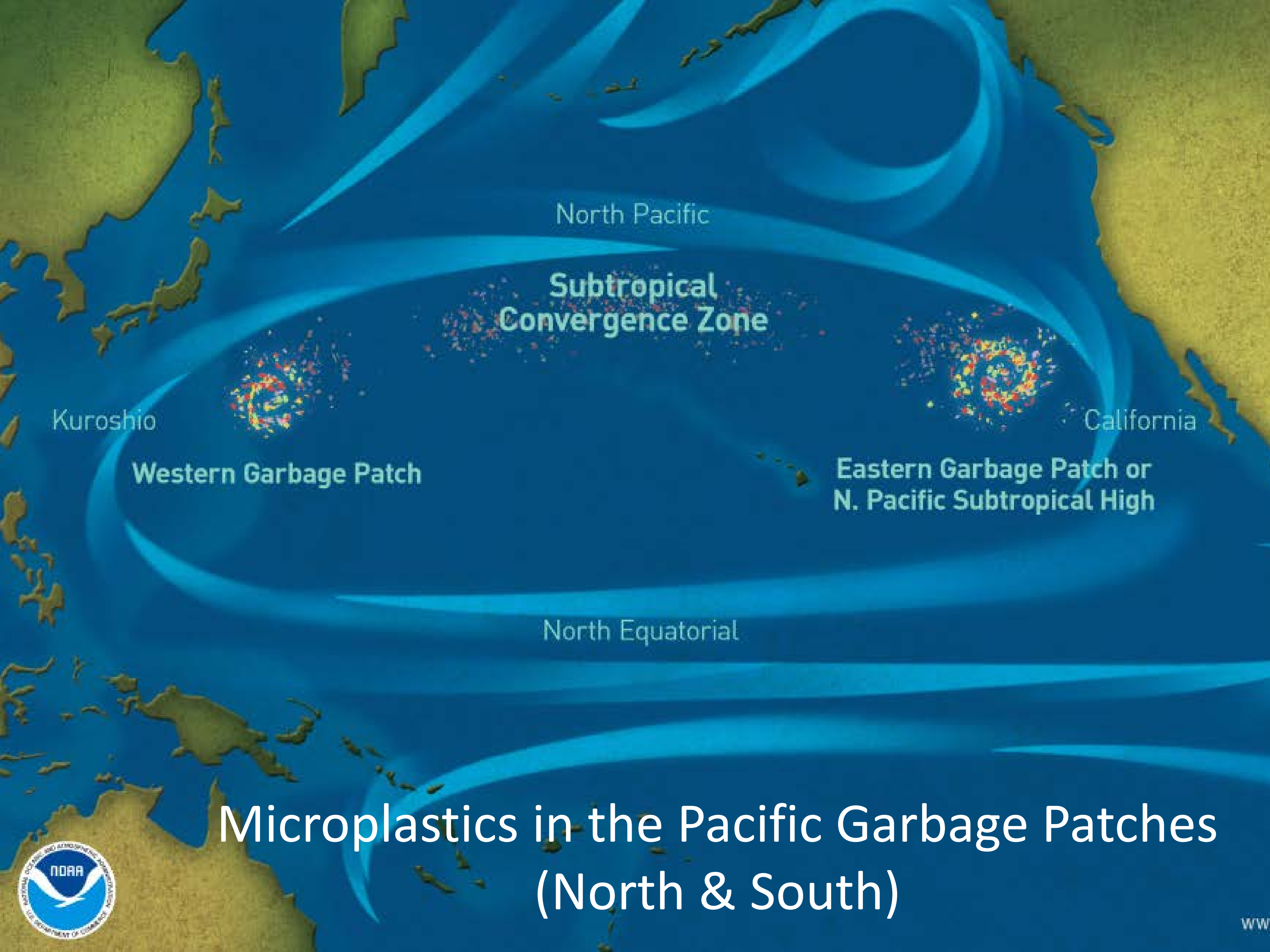
- Representative sample collection
- Difficulty in replication and sub-sampling (every sample is discrete and non-homogenous)
- Separation of plastics from other materials (biological matter and sediments)
- Interference from surface biofouling

Some key challenges in FT-IR analysis of environmental samples #2

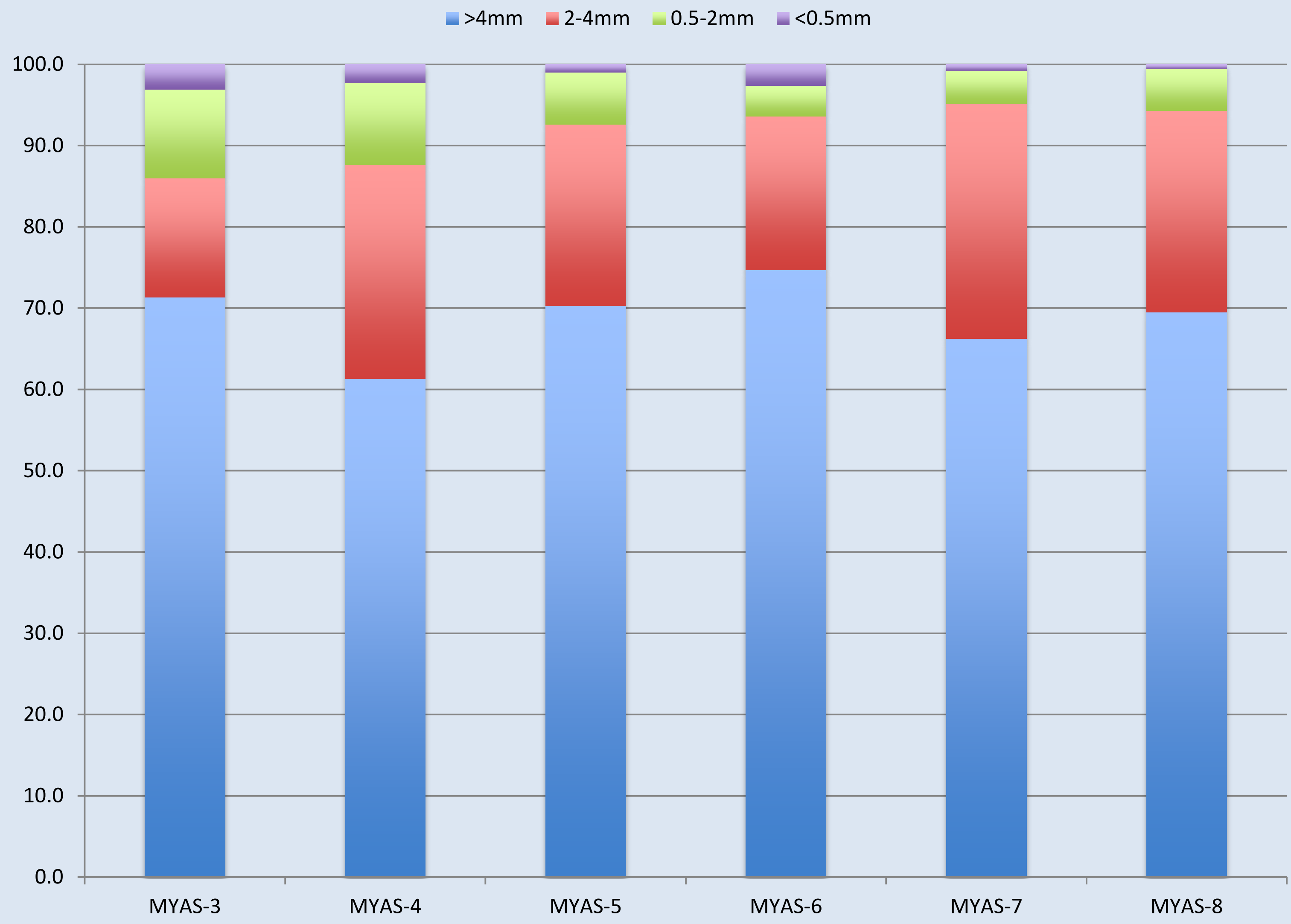
- Variable extent of polymer degradation
- Presence of pigments and other additives
- Sample contamination during collection, storage and analysis (especially fibres and paint fragments)
- Contamination of sampling equipment

(…even before we start!)

Microplastics in the Pacific Garbage Patches (North & South)

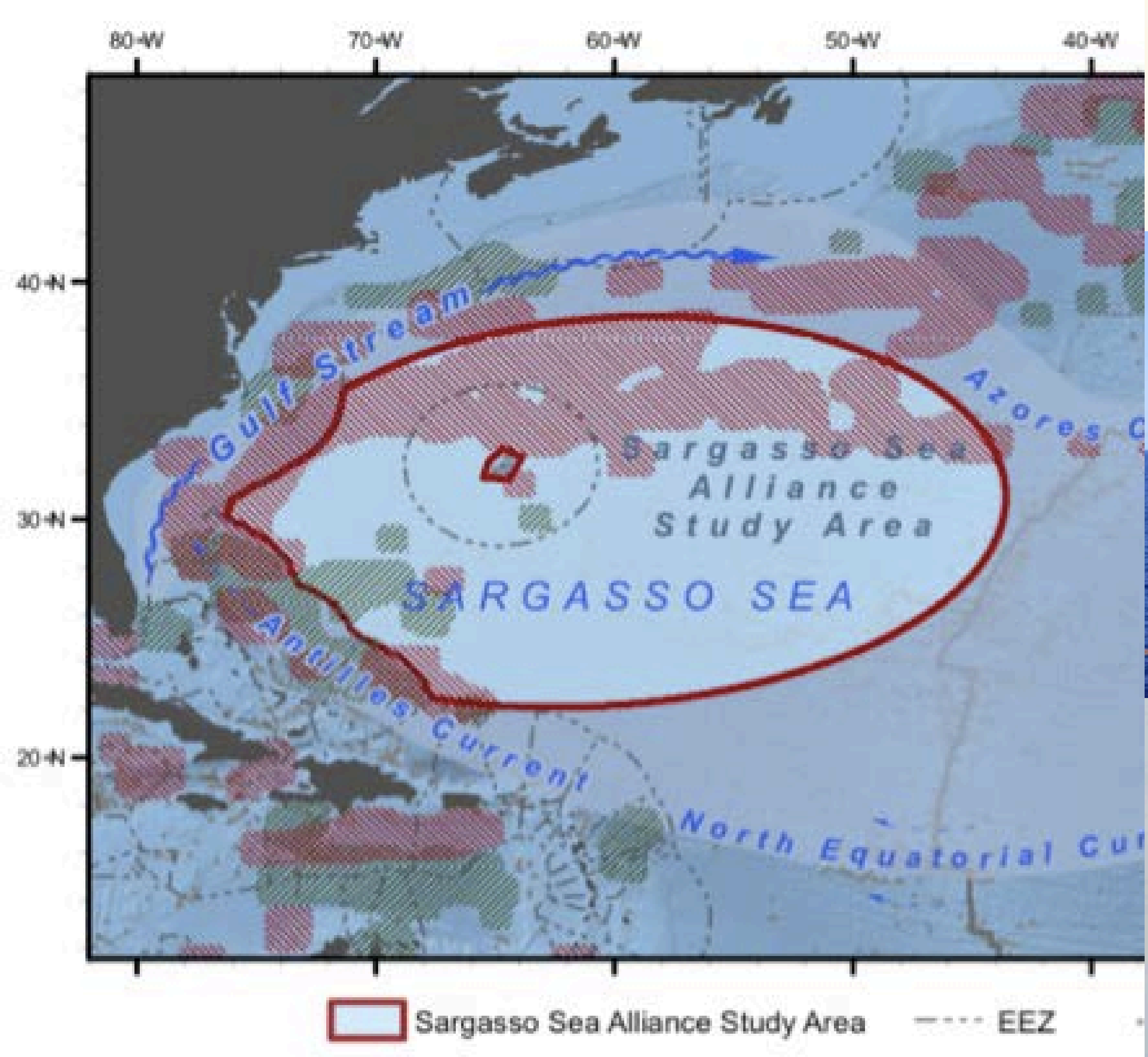


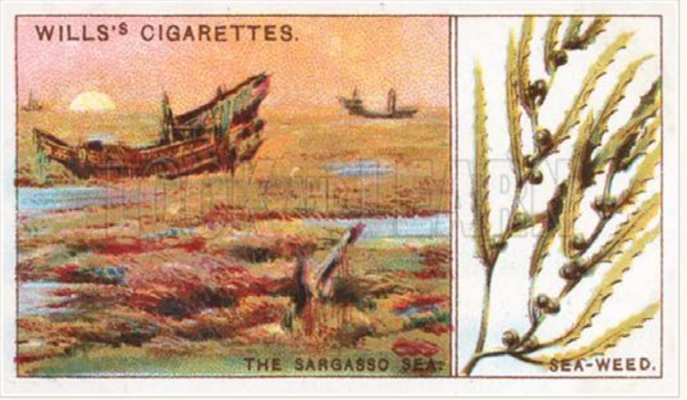

MYAS-8
<0.5mm
MYAS-3
>4mm
MYAS-3
<0.5mm
MYAS-6
MYAS-6
<0.5mm
MYAS-5
MYAS-5
<0.5mm



>4mm
2-4mm
0.5-2mm
<0.5mm
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0.0
MYAS-3
MYAS-4
MYAS-5
MYAS-6
MYAS-7
MYAS-8

Plastics associated with *Sargassum* weed





Surveys of beach plastics

Durban nurdle spill, October 2017


DRIZIT


Microplastics and
persistent fluorinated
chemicals in the Antarctic
GREENPEACE
PROTECT THE
ANTARCTIC
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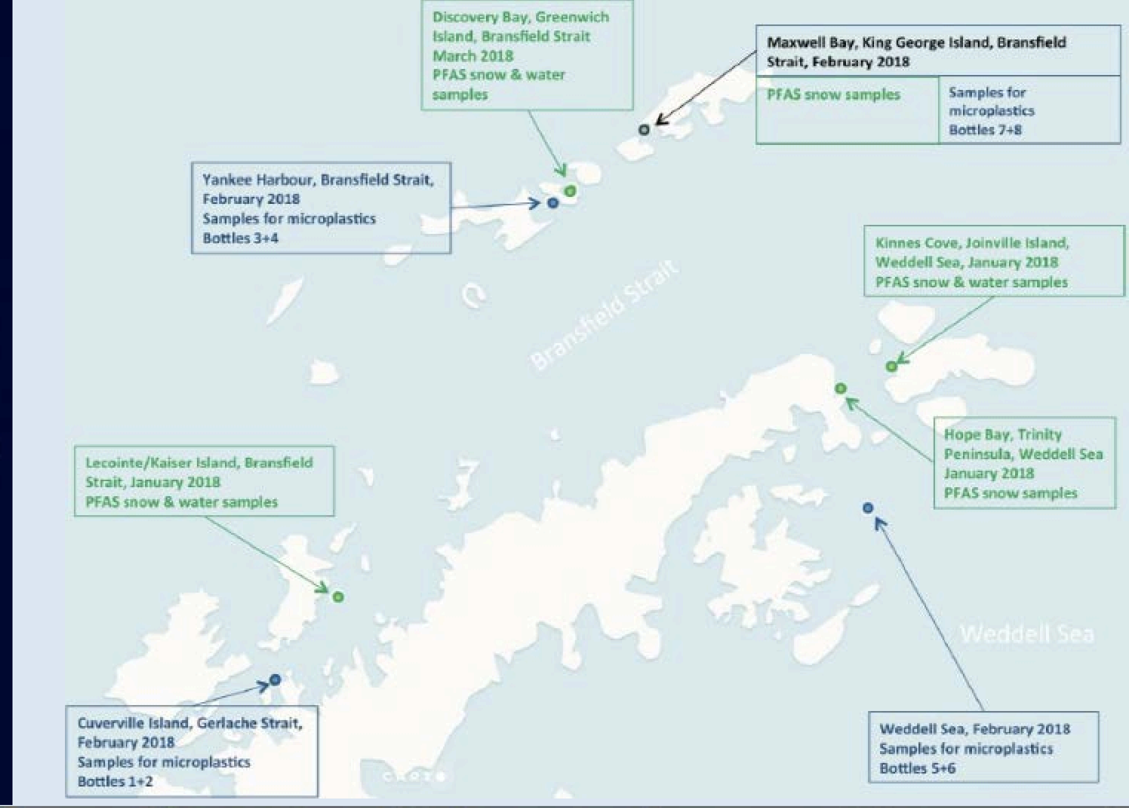
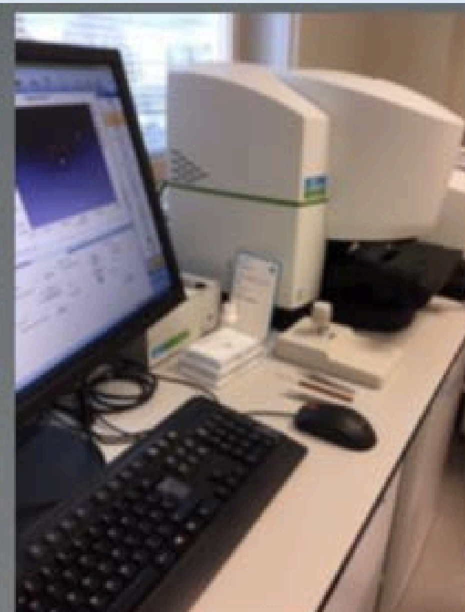
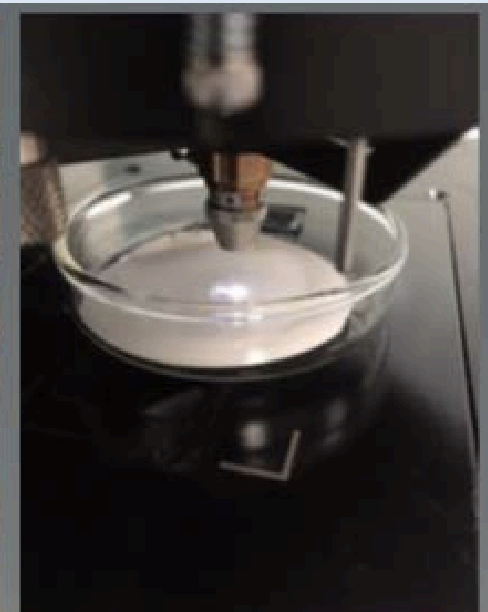
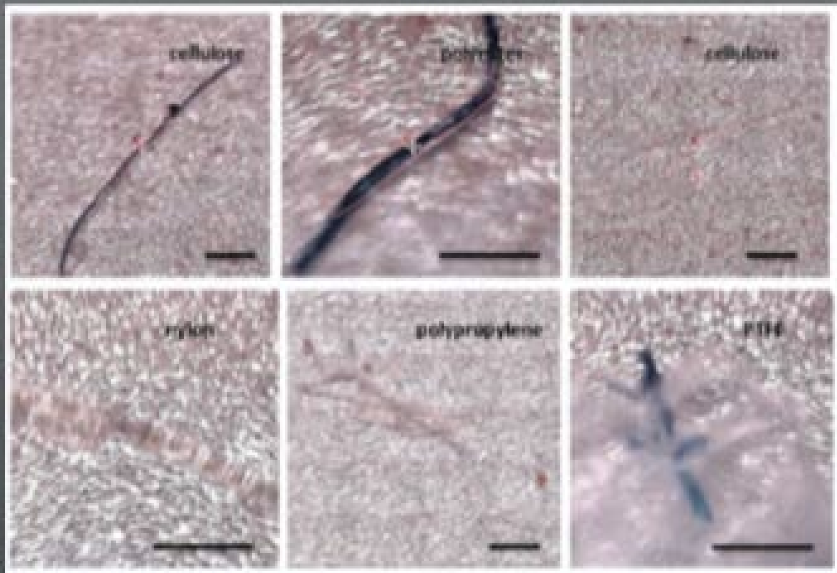


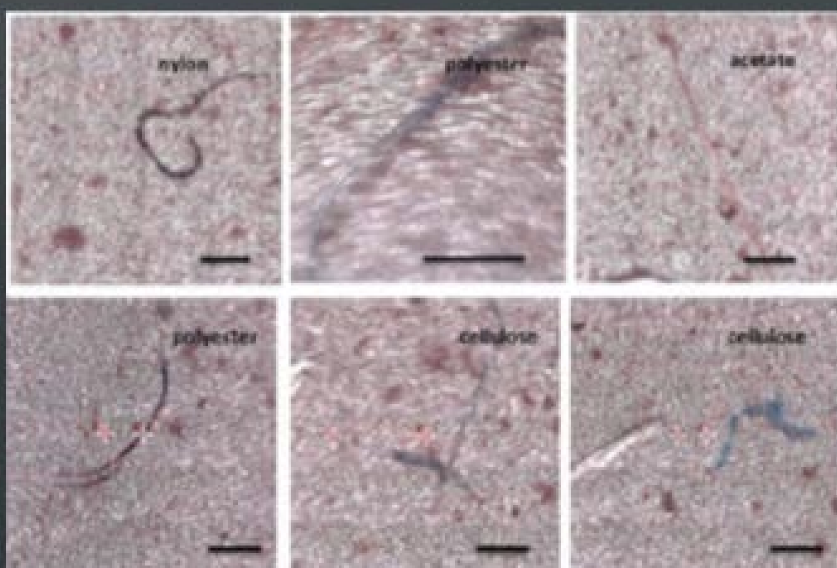
Discovery Bay, Greenwich Island, Bransfield Strait
March 2018
PFAS snow & water samples
Maxwell Bay, King George Island, Bransfield Strait, February 2018
PFAS snow samples
Samples for microplastics
Bottles 7+8
Yankee Harbour, Bransfield Strait, February 2018
Samples for microplastics
Bottles 3+4
Kinnes Cove, Joinville Island, Weddell Sea, January 2018
PFAS snow & water samples
Bransfield Strait
Hope Bay, Trinity Peninsula, Weddell Sea
January 2018
PFAS snow samples
Lecointe/Kaiser Island, Bransfield Strait, January 2018
PFAS snow & water samples
Weddell Sea
Cuverville Island, Gerlache Strait, February 2018
Samples for microplastics
Bottles 1+2
Weddell Sea, February 2018
Samples for microplastics
Bottles 5+6



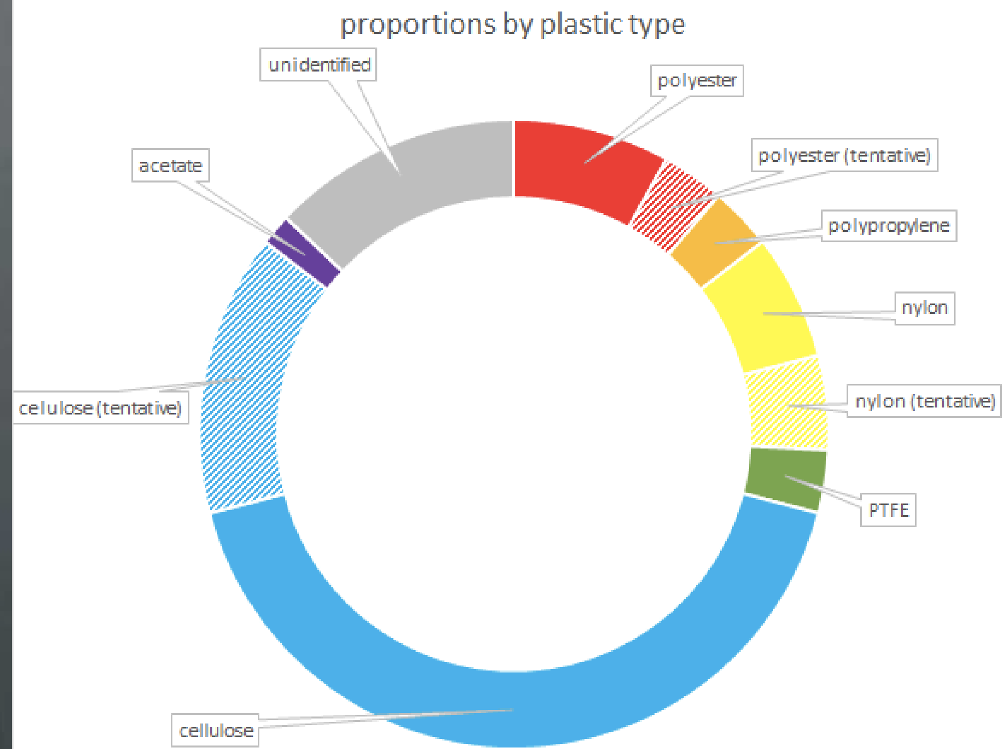


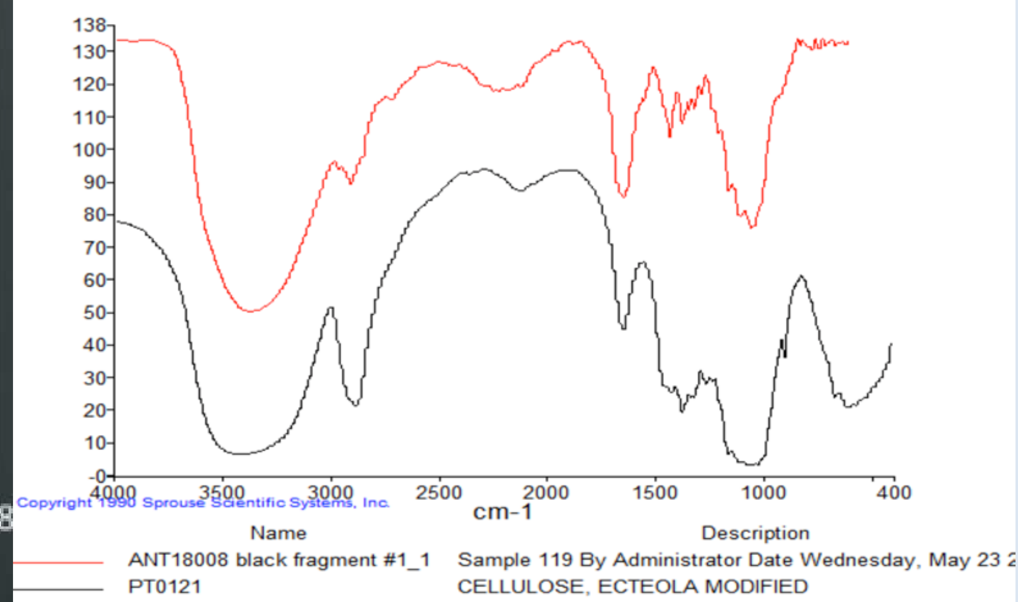


Weddell Sea (63"54.053 S; 056"42.496 W) 22/02/18
2.8 - 3.2 fibres/litre



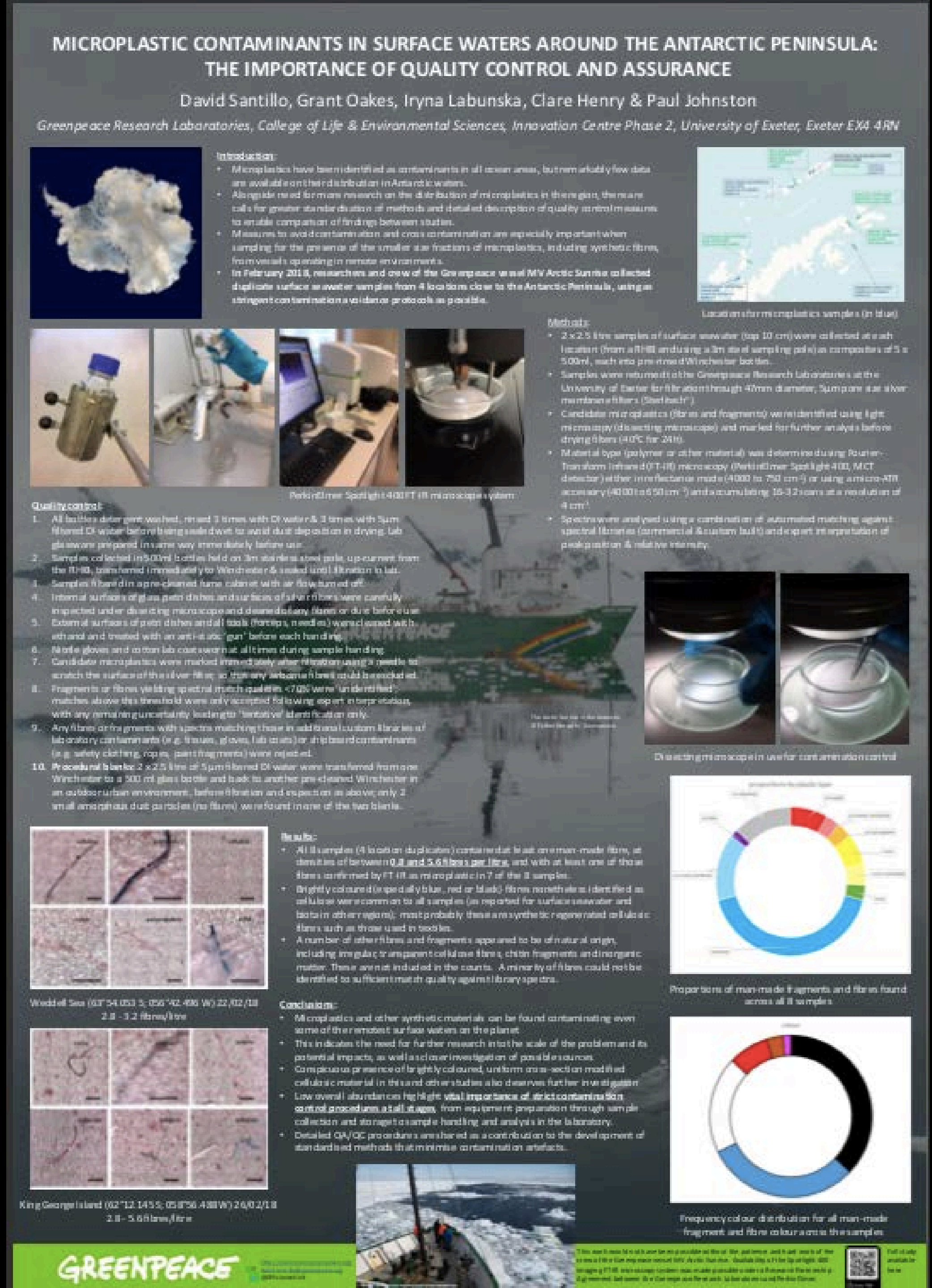
King George Island (62"12.145 S; 058"56.488 W) 26/02/18
2.8 - 5.6 fibres/litre





10 steps to quality assurance for environmental microfibre work

- Pre-wash of all glassware with 5 micron filtered water
- Use of glass & steel equipment for sample collection
- Filtration in clean fume cabinet with air flow off
- Cleaning of outer surfaces of glass petri dishes with ethanol and anti-stat gun
- Careful inspection of internal surfaces of petri dishes and filters before use
- Use of cotton lab coats & nitrile gloves
- Marking of candidate microplastics immediately after filtration (to exclude later ‘settlers’)
- Strict controls on acceptance of spectral matching
- Rejection of ship and laboratory contaminants using custom libraries
- Use of procedural blanks (and next time also field blanks)


MICROPLASTIC CONTAMINANTS IN SURFACE WATERS AROUND THE ANTARCTIC PENINSULA:
THE IMPORTANCE OF QUALITY CONTROL AND ASSURANCE
David Santillo, Grant Oakes, Iryna Labunska, Clare Henry & Paul Johnston
Greenpeace Research Laboratories, College of Life & Environmental Sciences, Innovation Centre Phase 2, University of Exeter, Exeter EX4 4RN
Introduction:
Microplastics have been identified as contaminants in all ocean areas, but remarkably few data are available on their distribution in Antarctic waters.
Alongside need for more research on the distribution of microplastics in the region, there are calls for greater standardisation of methods and detailed description of quality control measures to enable comparison of findings between studies.
Measures to avoid contamination and cross contamination are especially important when sampling for the presence of the smaller size fractions of microplastics, including synthetic fibres, from vessels operating in remote environments.
In February 2018, researchers and crew of the Greenpeace vessel MV Arctic Sunrise collected duplicate surface seawater samples from 4 locations close to the Antarctic Peninsula, using as stringent contamination avoidance protocols as possible.
Locations for microplastics samples (in blue)
Methods:
2 x 2.5 litre samples of surface seawater (top 10 cm) were collected at each location (from a RHIB and using a 3m steel sampling pole) as composites of 5 x 500ml, each into pre-rinsed Winchester bottles.
Samples were returned to the Greenpeace Research Laboratories at the University of Exeter for filtration through 47mm diameter, 5µm pore size silver membrane filters (Sterlitech®).
Candidate microplastics (fibres and fragments) were identified using light microscopy (dissecting microscope) and marked for further analysis before drying filters (40°C for 24h).
Material type (polymer or other material) was determined using Fourier-Transform Infrared (FT-IR) microscopy (PerkinElmer Spotlight 400, MCT detector) either in reflectance mode (4000 to 750 cm-1) or using a micro-ATR accessory (4000 to 650 cm-1) and accumulating 16-32 scans at a resolution of 4 cm-1
Spectra were analysed using a combination of automated matching against spectral libraries (commercial & custom built) and expert interpretation of peak position & relative intensity.
PerkinElmer Spotlight 400 FT-IR microscope system
Quality control:
1. All bottles detergent washed, rinsed 3 times with DI water & 3 times with 5µm filtered DI water before being sealed wet to avoid dust deposition in drying. Lab glassware prepared in same way immediately before use.
2. Samples collected in 500ml bottles held on 3m stainless steel pole, up-current from the RHIB, transferred immediately to Winchester & sealed until filtration in lab.
3. Samples filtered in a pre-cleaned fume cabinet with air flow turned off.
4. Internal surfaces of glass petri dishes and surfaces of silver filters were carefully inspected under dissecting microscope and cleaned of any fibres or dust before use.
5. External surfaces of petri dishes and all tools (forceps, needles) were cleaned with ethanol and treated with an anti-static 'gun' before each handling.
6. Nitrile gloves and cotton lab coats worn at all times during sample handling.
7. Candidate microplastics were marked immediately after filtration using a needle to scratch the surface of the silver filter, so that any airborne fibres could be excluded.
8. Fragments or fibres yielding spectral match qualities <70% were 'unidentified'; matches above this threshold were only accepted following expert interpretation, with any remaining uncertainty leading to 'tentative' identification only.
9. Any fibres or fragments with spectra matching those in additional custom libraries of laboratory contaminants (e.g. tissues, gloves, lab coats) or shipboard contaminants (e.g. safety clothing, ropes, paint fragments) were rejected.
10. Procedural blanks: 2 x 2.5 litre of 5µm filtered DI water were transferred from one Winchester to a 500 ml glass bottle and back to another pre-cleaned Winchester in an outdoor urban environment, before filtration and inspection as above; only 2 small amorphous dust particles (no fibres) were found in one of the two blanks.
Dissecting microscope in use for contamination control
Results:
All 8 samples (4 location duplicates) contained at least one man-made fibre, at densities of between 0.8 and 5.6 fibres per litre, and with at least one of those fibres confirmed by FT-IR as microplastic in 7 of the 8 samples.
Brightly coloured (especially blue, red or black) fibres nonetheless identified as cellulose were common to all samples (as reported for surface seawater and biota in other regions); most probably these are synthetic regenerated cellulosic fibres such as those used in textiles.
A number of other fibres and fragments appeared to be of natural origin, including irregular, transparent cellulose fibres, chitin fragments and inorganic matter. These are not included in the counts. A minority of fibres could not be identified to sufficient match quality against library spectra.
Weddell Sea (63°54.053 S; 056°42.496 W) 22/02/18
2.8 - 3.2 fibres/litre
Proportions of man-made fragments and fibres found across all 8 samples
Conclusions:
Microplastics and other synthetic materials can be found contaminating even some of the remotest surface waters on the planet.
This indicates the need for further research into the scale of the problem and its potential impacts, as well as closer investigation of possible sources.
Conspicuous presence of brightly coloured, uniform cross-section modified cellulosic material in this and other studies also deserves further investigation.
Low overall abundances highlight vital importance of strict contamination control procedures at all stages, from equipment preparation through sample collection and storage to sample handling and analysis in the laboratory.
Detailed QA/QC procedures are shared as a contribution to the development of standardised methods that minimise contamination artefacts.
King George Island (62°12.145 S; 058°56.488 W) 26/02/18
2.8 - 5.6 fibres/litre
Frequency colour distribution for all man-made fragment and fibre colours across the samples
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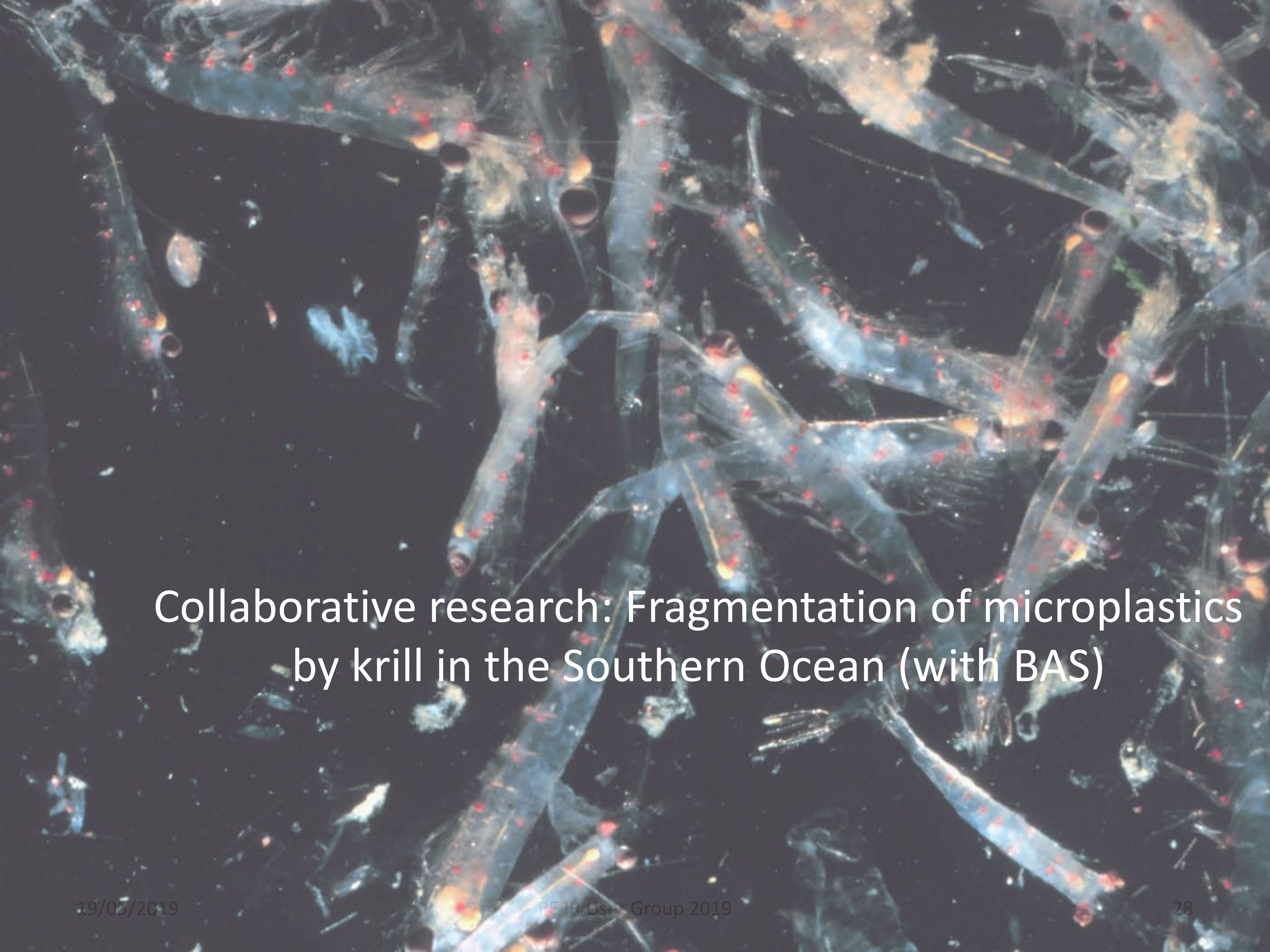


Collaborative research: Fragmentation of microplastics by krill in the Southern Ocean (with BAS)

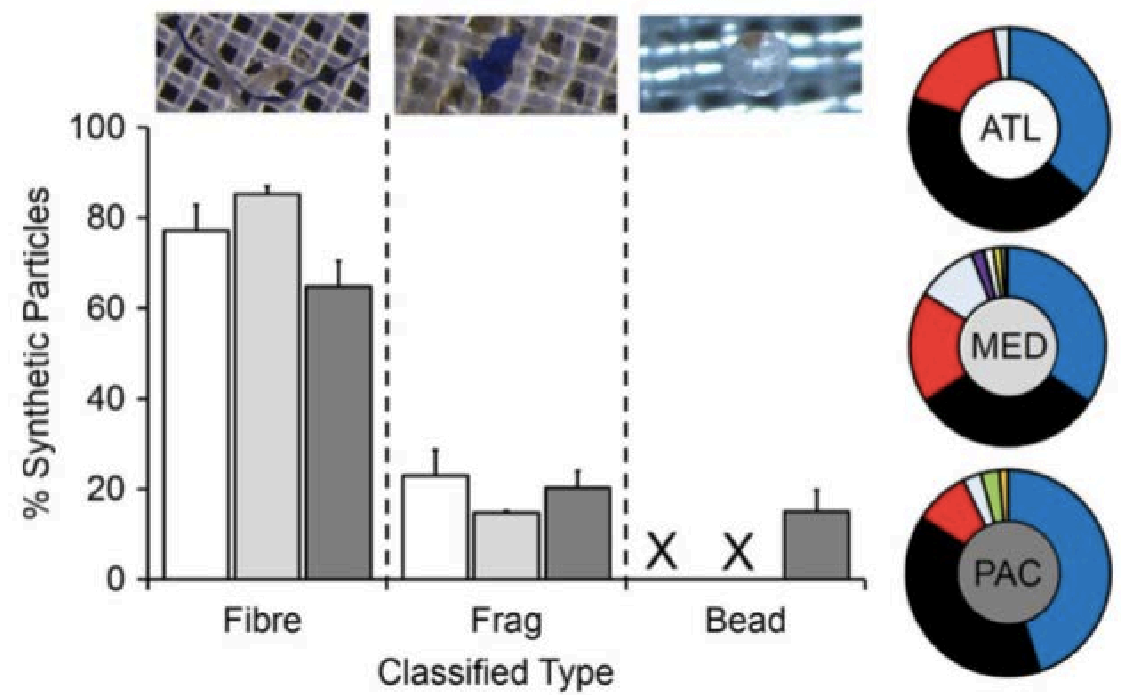
Received: 27 August 2018 | Accepted: 15 October 2018
DOI: 10.1111/gcb.14519

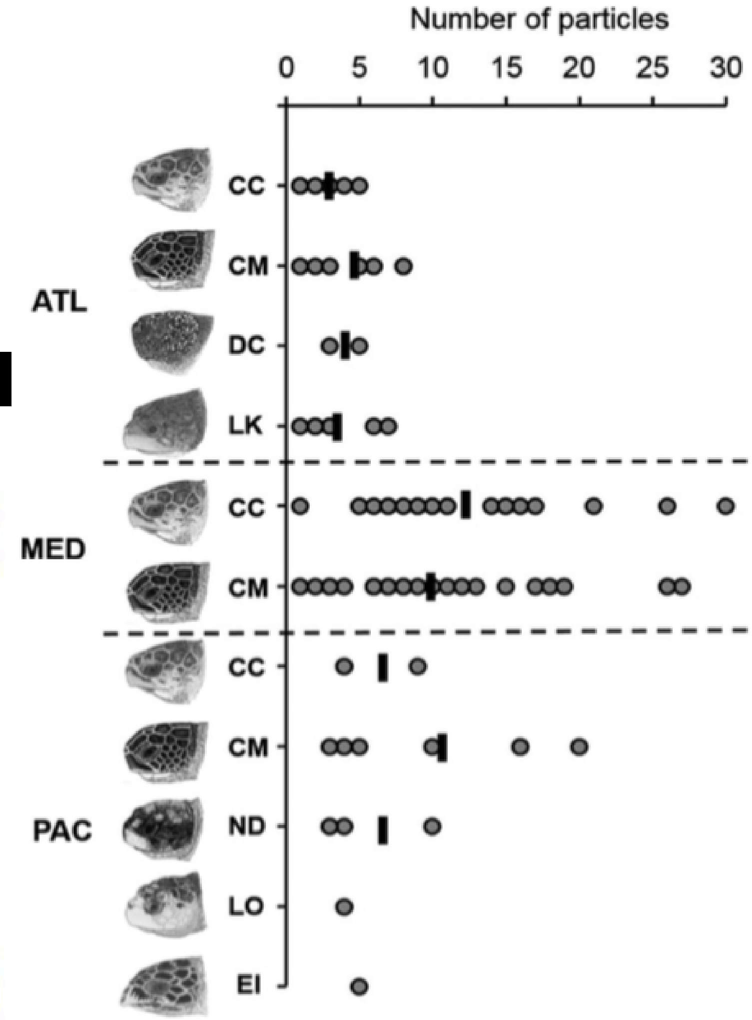
PRIMARY RESEARCH ARTICLE

WILEY Global Change Biology

Microplastic ingestion ubiquitous in marine turtles

Emily M. Duncan[1,2,3] | Annette C. Broderick[1] | Wayne J. Fuller[1,4,5] | Tamara S. Galloway[2] | Matthew H. Godfrey[6] | Mark Hamann[7] | Colin J. Limpus[8] | Penelope K. Lindeque[3] | Andrew G. Mayes[9] | Lucy C. M. Omeyer[1] | David Santillo[10] | Robin T. E. Snape[1,5] | Brendan J. Godley[1]





www.nature.com/scientificreports

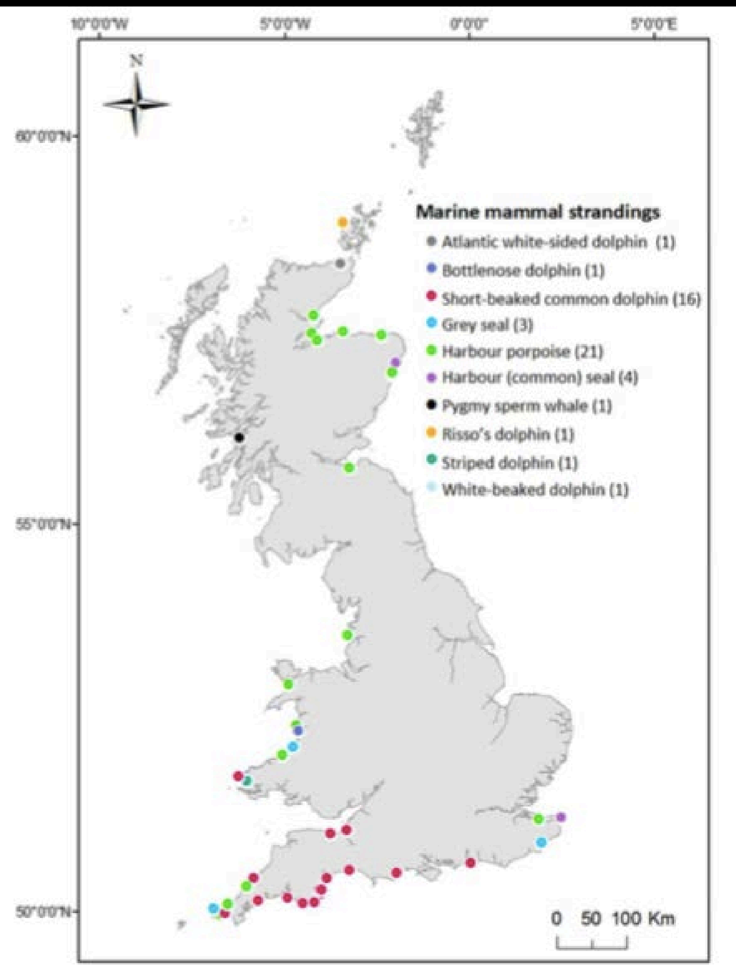
SCIENTIFIC REPORTS

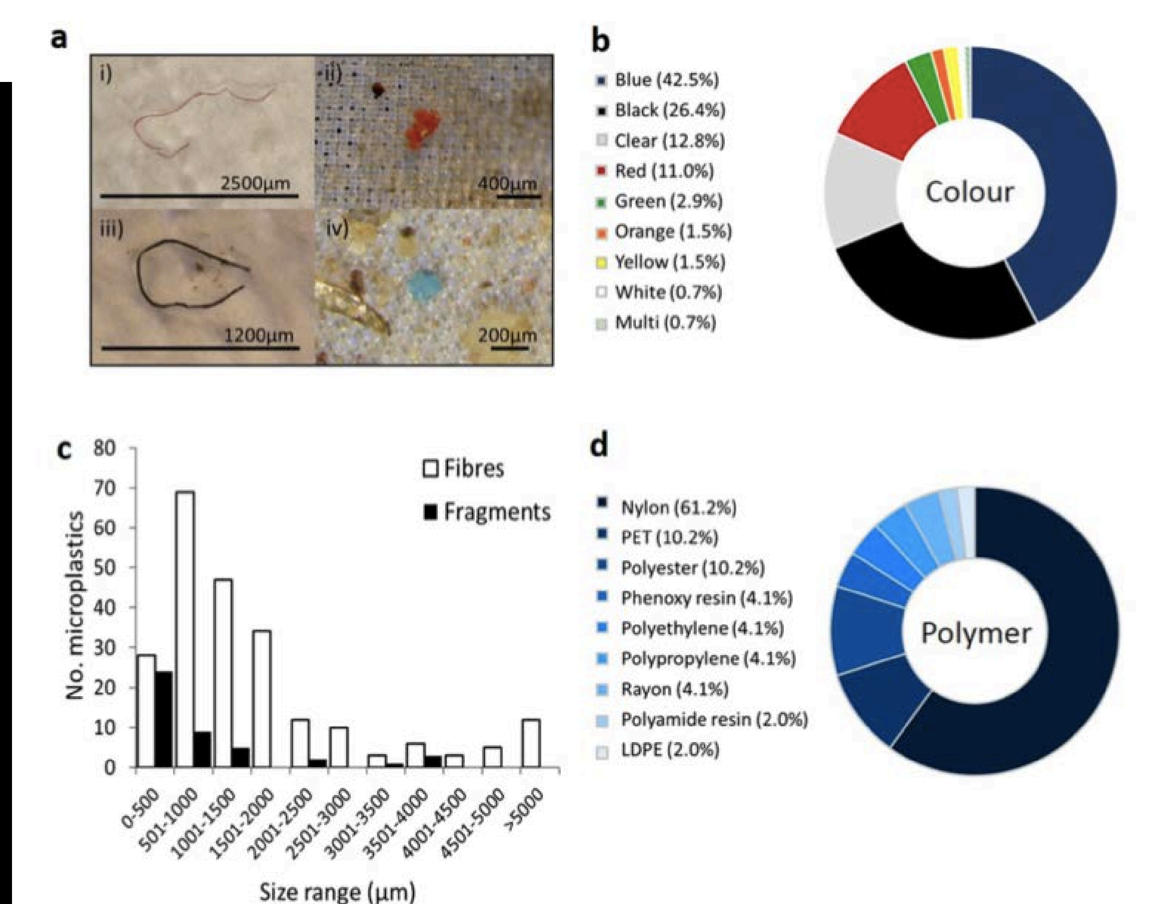
OPEN

Microplastics in marine mammals stranded around the British coast: ubiquitous but transitory?

Received: 7 August 2018
Accepted: 30 November 2018
Published online: 31 January 2019

S. E. Nelms[1,2], J. Barnett[3], A. Brownlow[4], N. J. Davison[4], R. Deaville[5], T. S. Galloway[6], P. K. Lindeque[1], D. Santillo[7] & B. J. Godley[2,3]





Received: 7 December 2018 | Accepted: 5 July 2019
DOI: 10.1111/2041-210X.13271

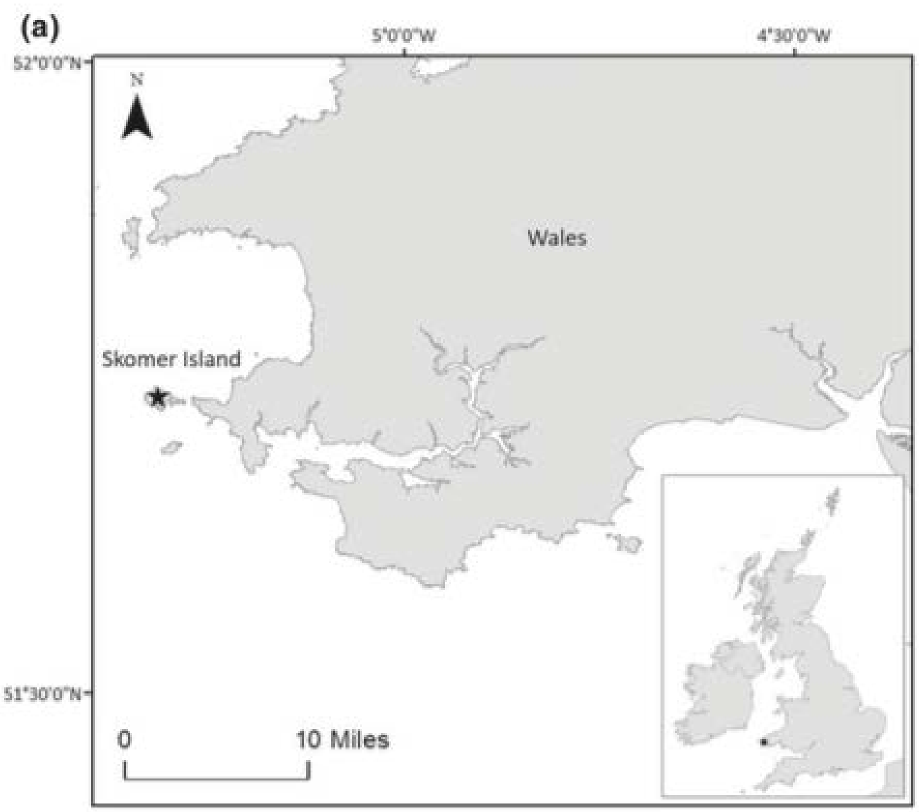
RESEARCH ARTICLE

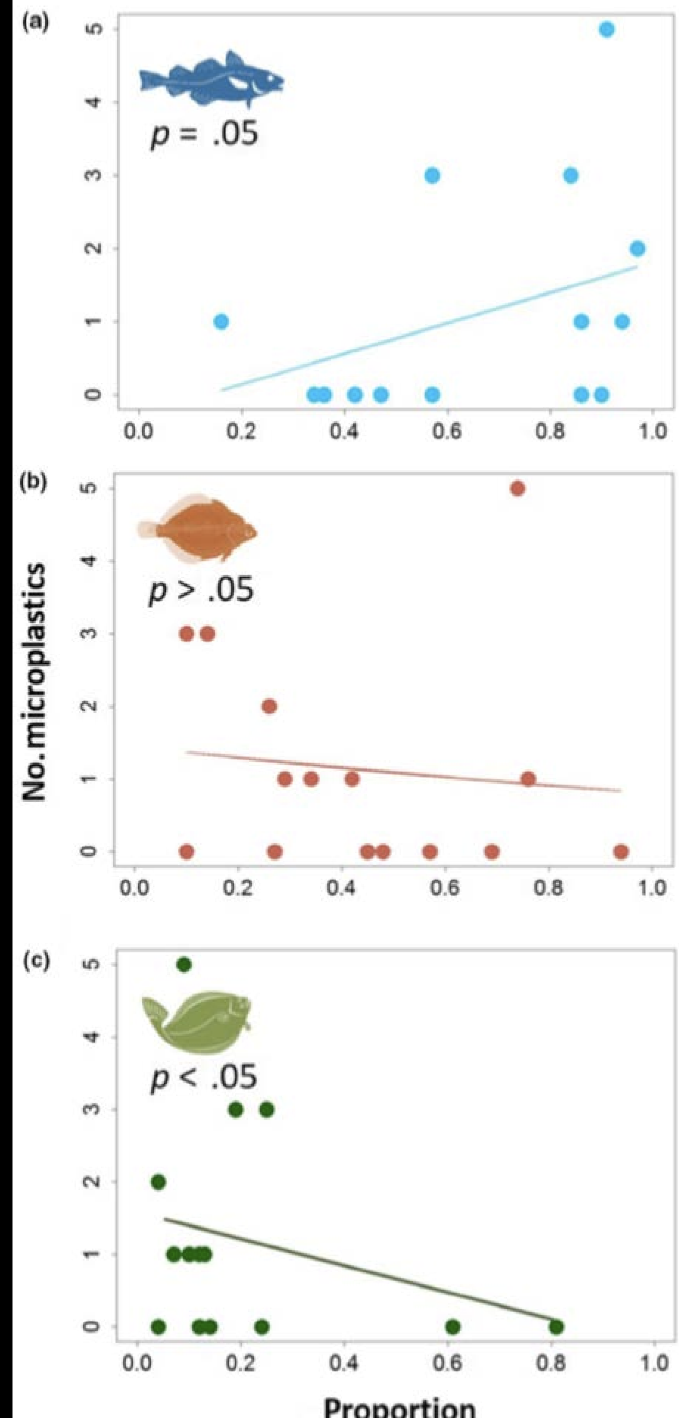
Methods in Ecology and Evolution

What goes in, must come out: Combining scat-based molecular diet analysis and quantification of ingested microplastics in a marine top predator

Sarah E. Nelms[1,2] | Helen E. Parry[1] | Kimberley A. Bennett[3] | Tamara S. Galloway[4] | Brendan J. Godley[2,5] | David Santillo[6] | Penelope K. Lindeque[1]

[1]Plymouth Marine Laboratory, Plymouth, UK; [2]Centre for Ecology and Conservation, University of Exeter, Cornwall, UK; [3]School of Science, Engineering & Technology, Abertay University, Dundee, UK; [4]Biosciences, Geoffrey Pope Building, University of Exeter, Devon, UK; [5]Environment and Sustainability Institute, University of Exeter, Cornwall, UK and [6]Greenpeace Research Laboratories, Innovation Centre Phase 2, University of Exeter, Devon, UK





Marine Pollution Bulletin 146 (2019) 125–133

Contents lists available at ScienceDirect

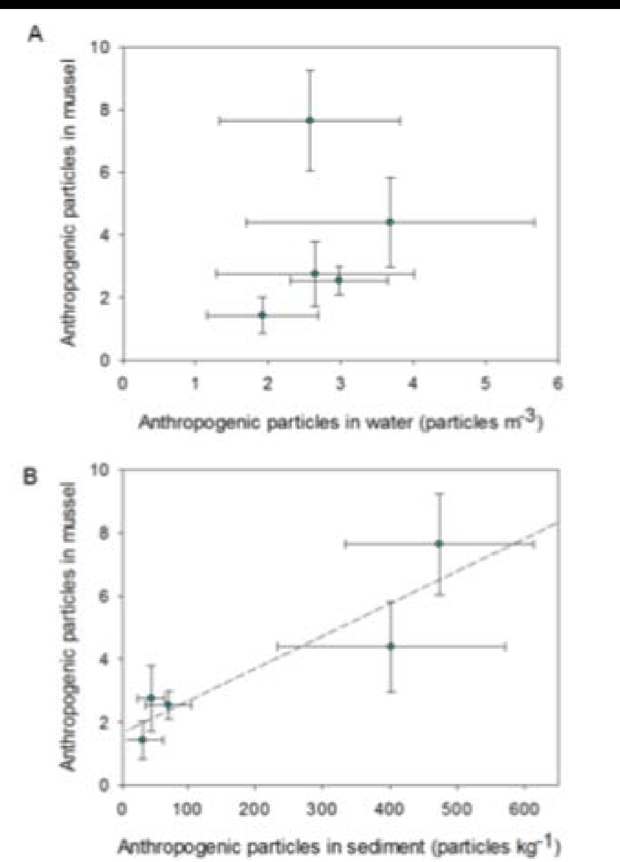
Marine Pollution Bulletin

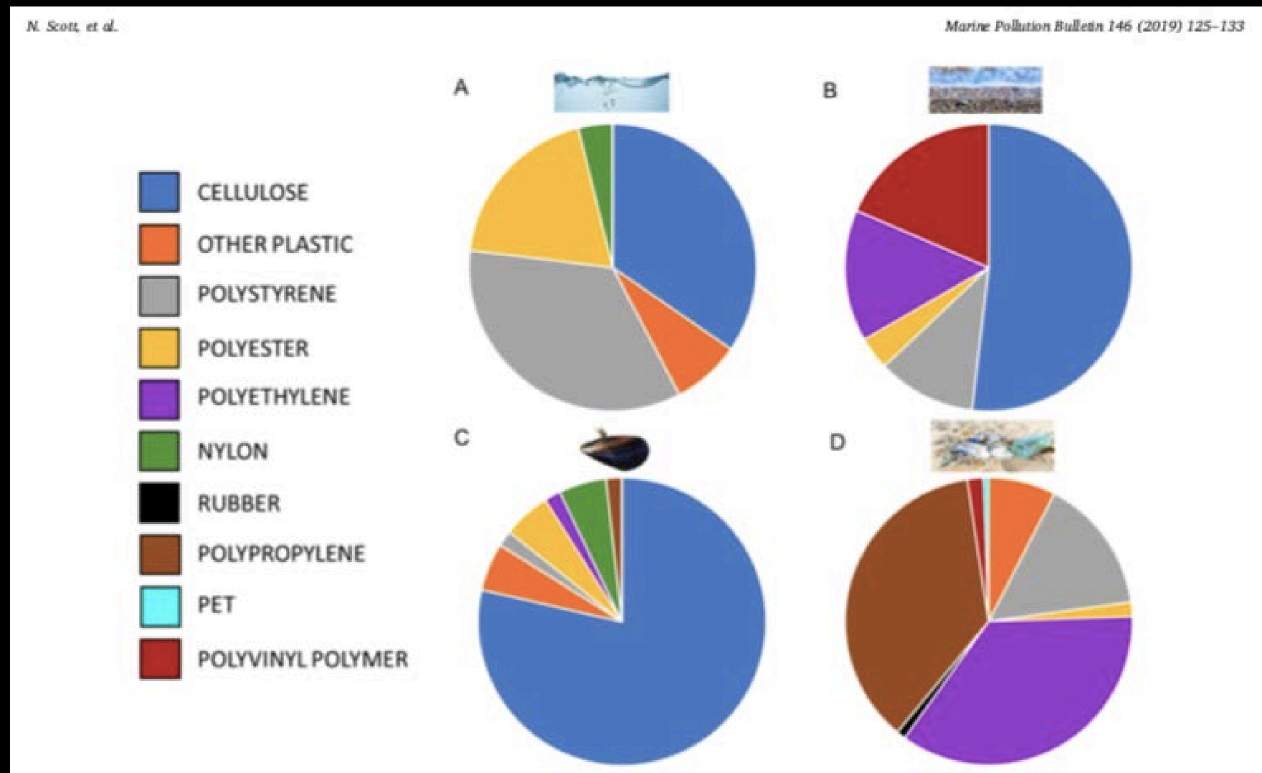
journal homepage: www.elsevier.com/locate/marpolbul

Particle characteristics of microplastics contaminating the mussel *Mytilus edulis* and their surrounding environments

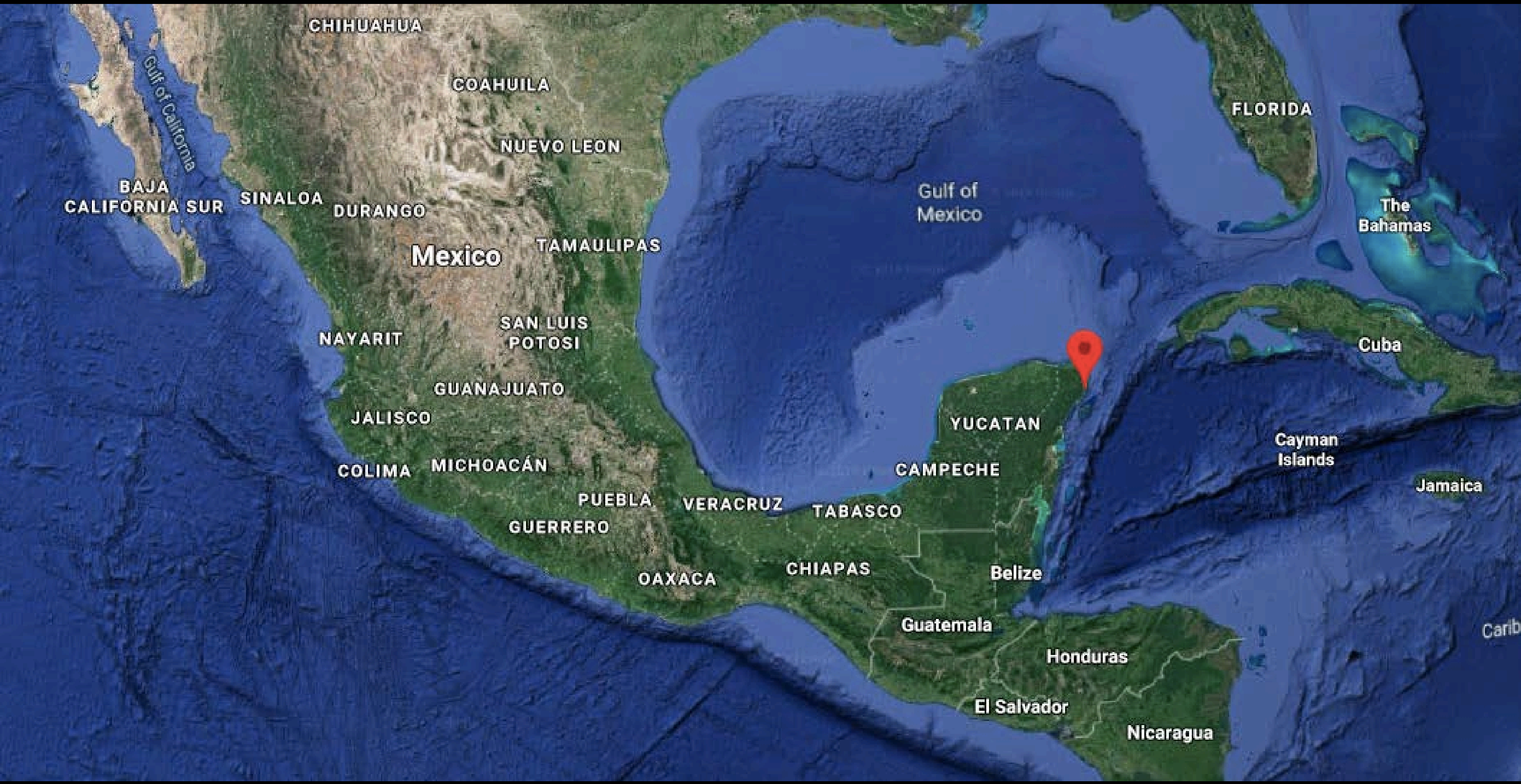
Nicholas Scott[a], Adam Porter[a], David Santillo[b], Holly Simpson[a], Sophie Lloyd-Williams[a], Ceri Lewis[a,*]

[a] College of Life and Environmental Sciences: Biosciences, Geoffrey Pope Building, University of Exeter, Stocker Road, Exeter EX4 4QD, United Kingdom.
[b] Greenpeace Research Laboratories, Innovation Centre Phase 2, University of Exeter, Rennes Drive, Exeter EX4 4RN, United Kingdom.





Coming up: Microplastics in fish from Mexican waters (Atlantic & Pacific)



Coming up: microplastics (including paint chips) on the beaches of Galapagos

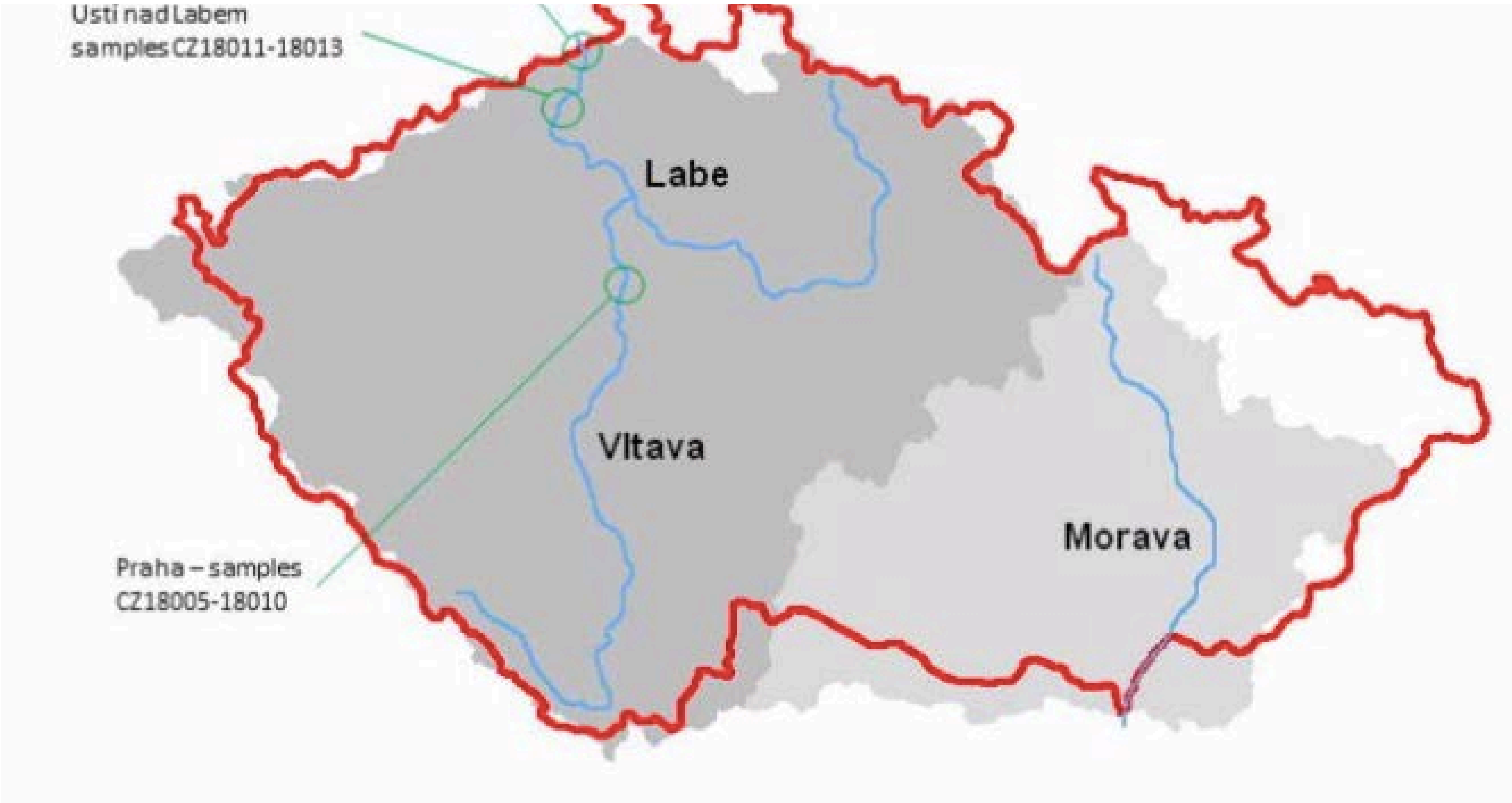

MICROPLASTICS
IN RIVERS

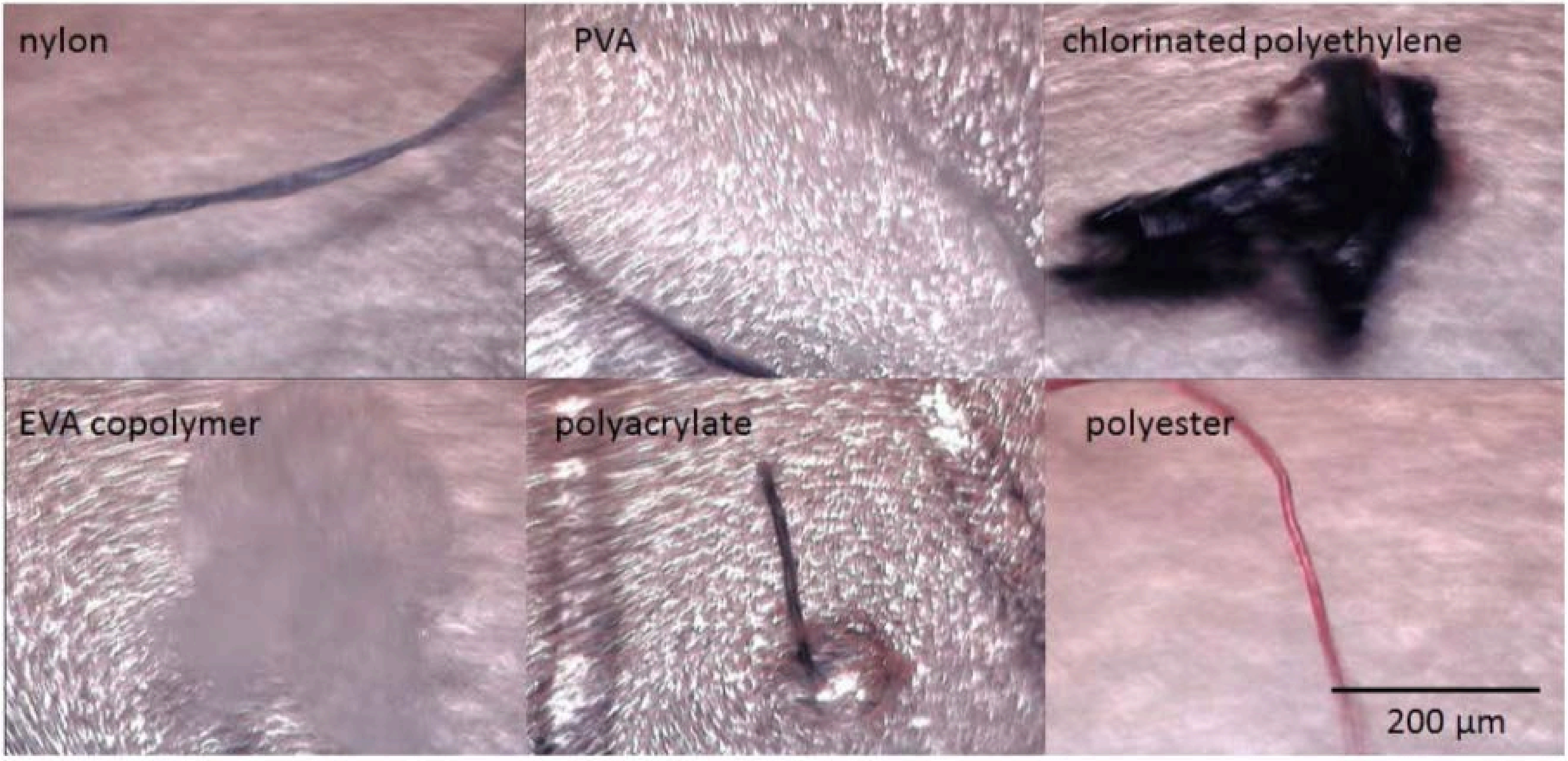
Microplastics in the Vltava and Elbe Rivers, Czech Republic

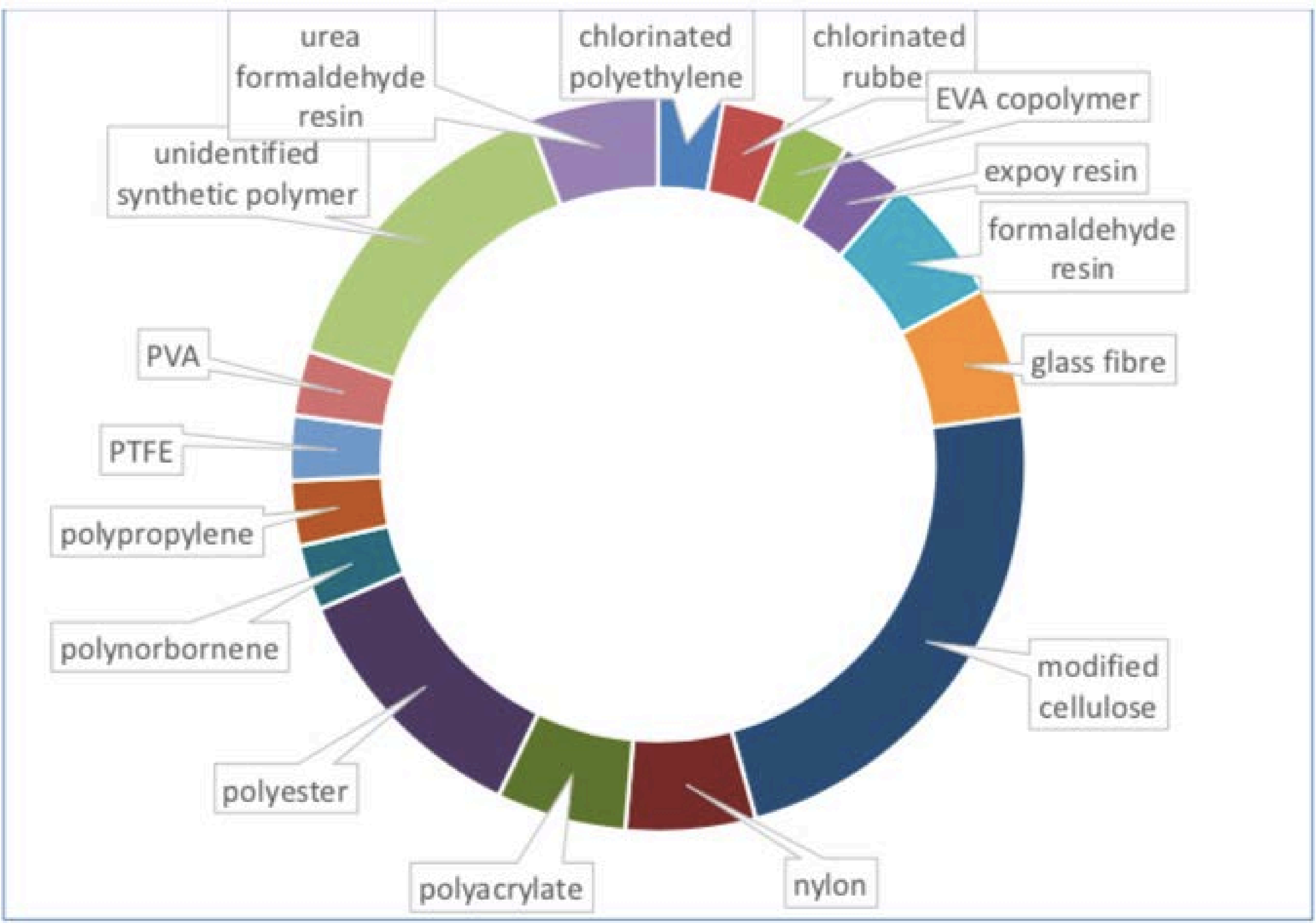
Greenpeace Research Laboratories Analytical Results 2019-01

A 'snapshot' survey of microplastics in surface waters of the Vltava and Labe (Elbe) Rivers in the Czech Republic

March 2019

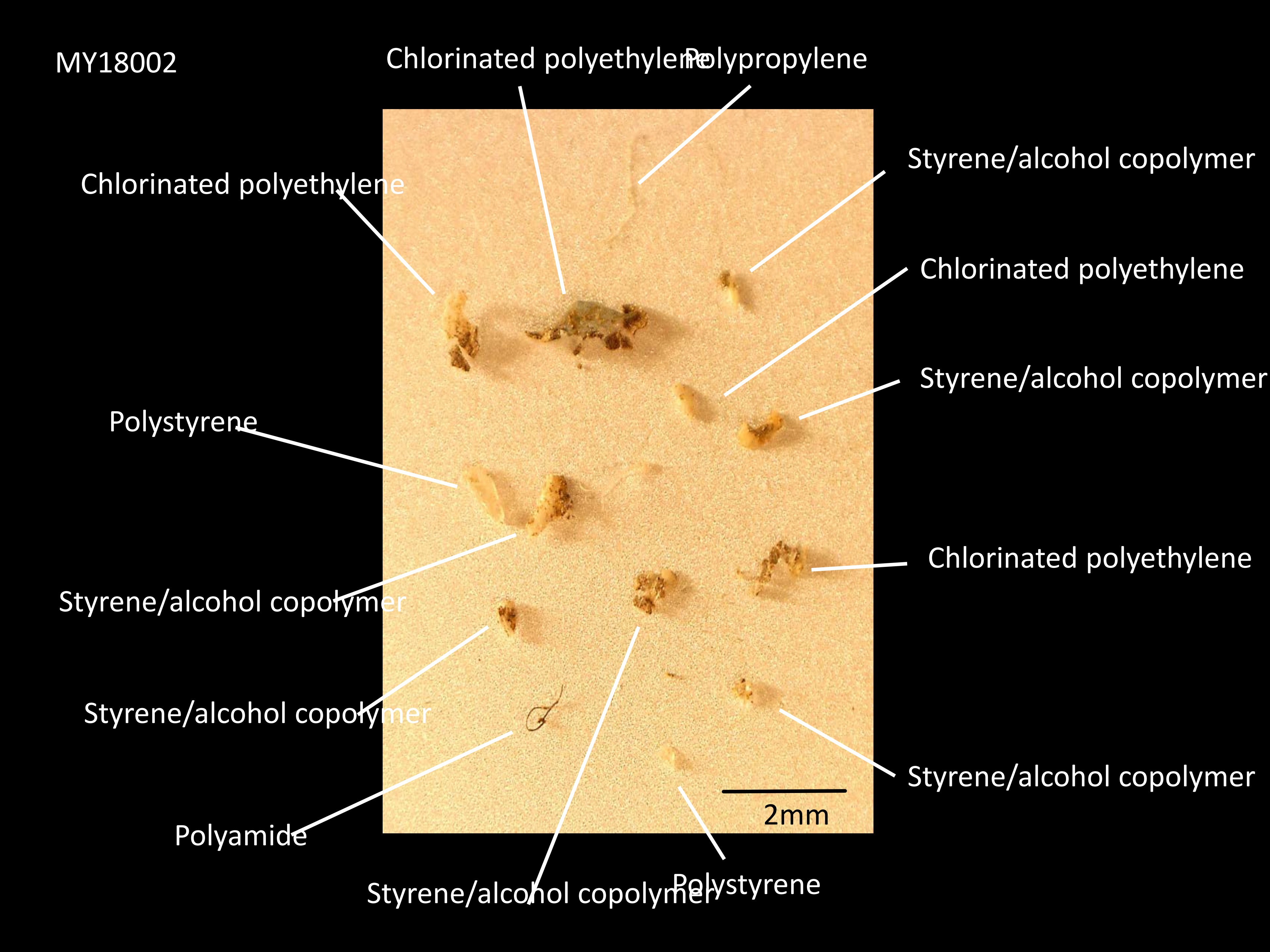



nylon
PVA
chlorinated polyethylene
EVA copolymer
polyacrylate
polyester
200 μm


urea formaldehyde resin
chlorinated polyethylene
chlorinated rubbe
EVA copolymer
expoy resin
formaldehyde resin
glass fibre
modified cellulose
nylon
polyacrylate
polyester
polynorbornene
polypropylene
PTFE
PVA
unidentified synthetic polymer

Implications of the plastic waste trade in Malaysia


Angel
Delight
Butterscotch


MY18002
Chlorinated polyethylene
Polypropylene
Styrene/alcohol copolymer
Chlorinated polyethylene
Chlorinated polyethylene
Styrene/alcohol copolymer
Polystyrene
Chlorinated polyethylene
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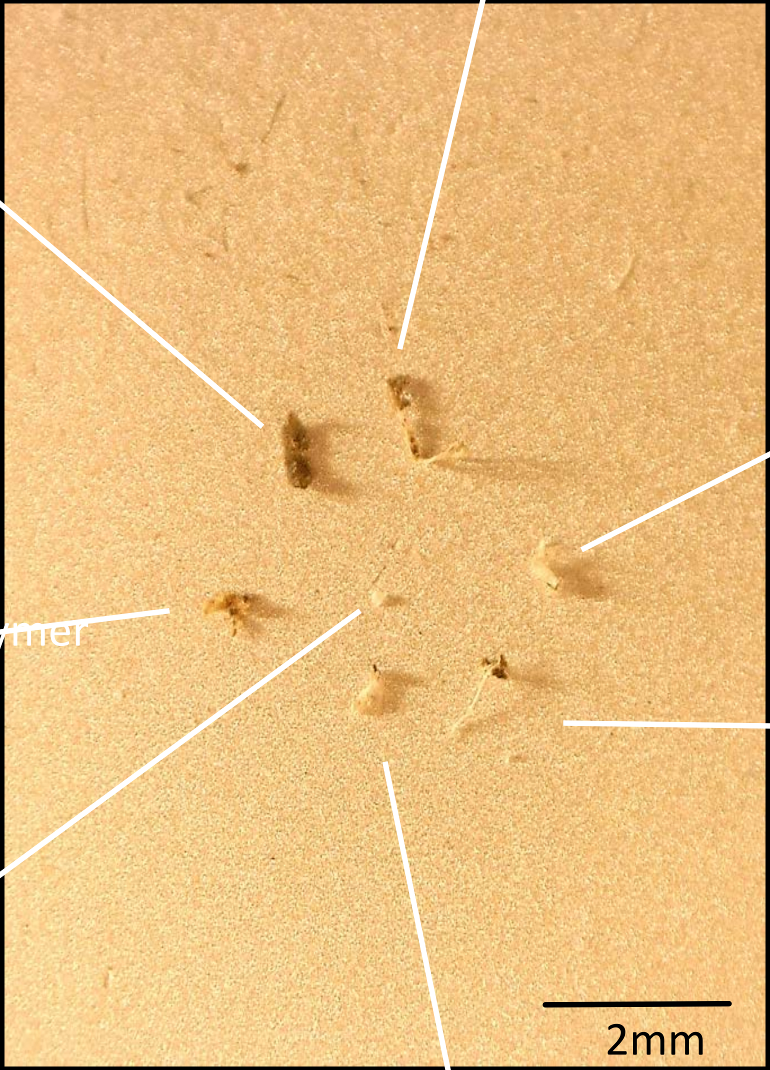
Polyamide
2mm
Styrene/alcohol copolymer
Polystyrene

MY18004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Polyethylene
Styrene/alcohol copolymer
Polypropylene
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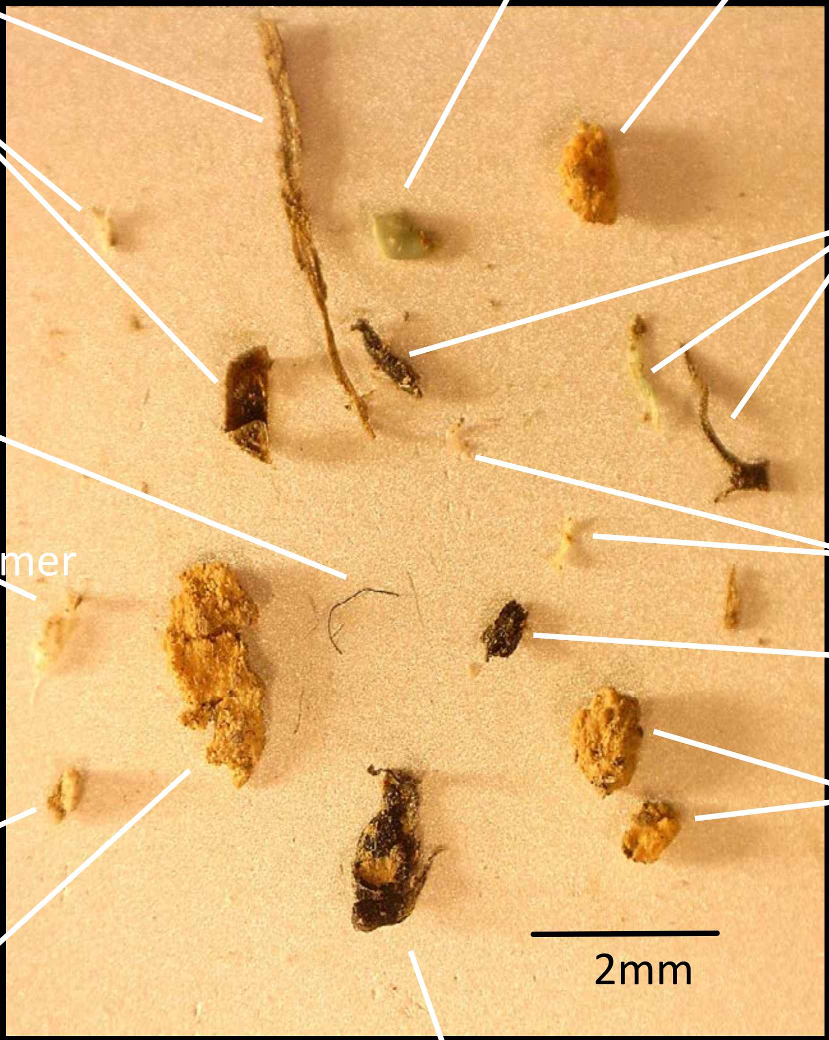
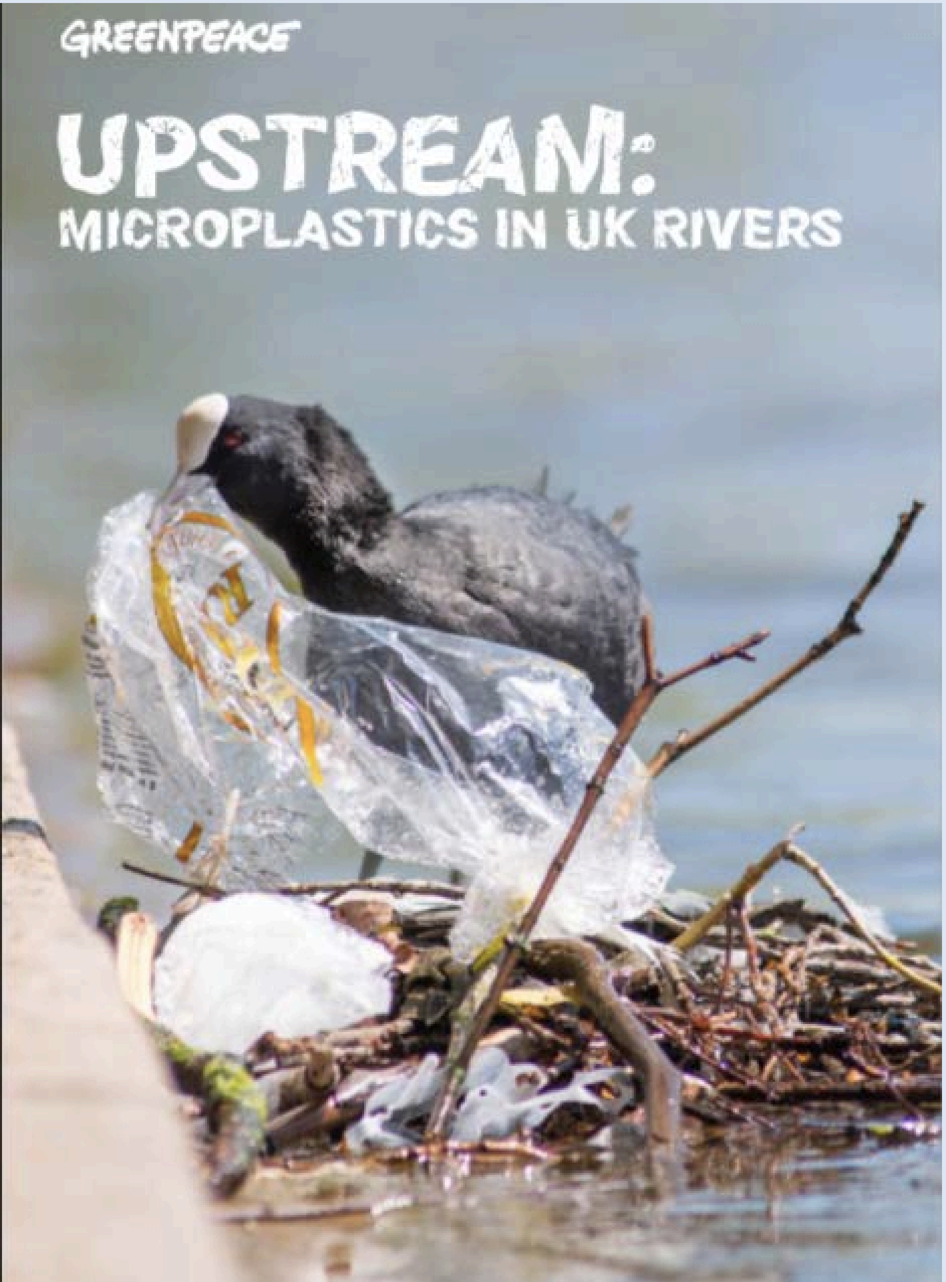

MY18005
Chlorinated polyethylene
Ethylene/vinyl acetate copolymer
Styrene/alcohol copolymer
Polyethylene
Polyethylene
Polyester
Polypropylene
Styrene/alcohol copolymer
Polyethylene
Polystyrene/vinylidene copolymer
Polyethylene
2mm
Polystyrene/vinylidene copolymer
Polyethylene



Plastic pollution in UK's rivers: a 'snapshot' survey of macro- and micro-plastic contamination in surface waters of 13 river systems across England, Wales, Scotland and Northern Ireland

David Santillo, Kevin Brigden, Veronica Pasteur, Fiona Nicholls, Paul Morozzo & Paul Johnston

Greenpeace Research Laboratories Technical Report 04-2019, June 2019

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Summary

Although the problem of plastic pollution at sea is widely documented, the situation in rivers that carry much of that pollution has been far less well studied to date. As a contribution to greater understanding of the problem, in February and March 2019, Greenpeace UK (supported by the People's Postcode Lottery) carried out a geographically widespread 'snapshot' survey of levels of plastics, including microplastics, in 13 rivers across the UK (9 in England, 2 in Wales, 1 in Scotland and 1 in Northern Ireland) using a floating 'manta' net placed mid-stream. Plastics were counted, weighed and sized and their identities determined using forensic infrared analysis (FT-IR).

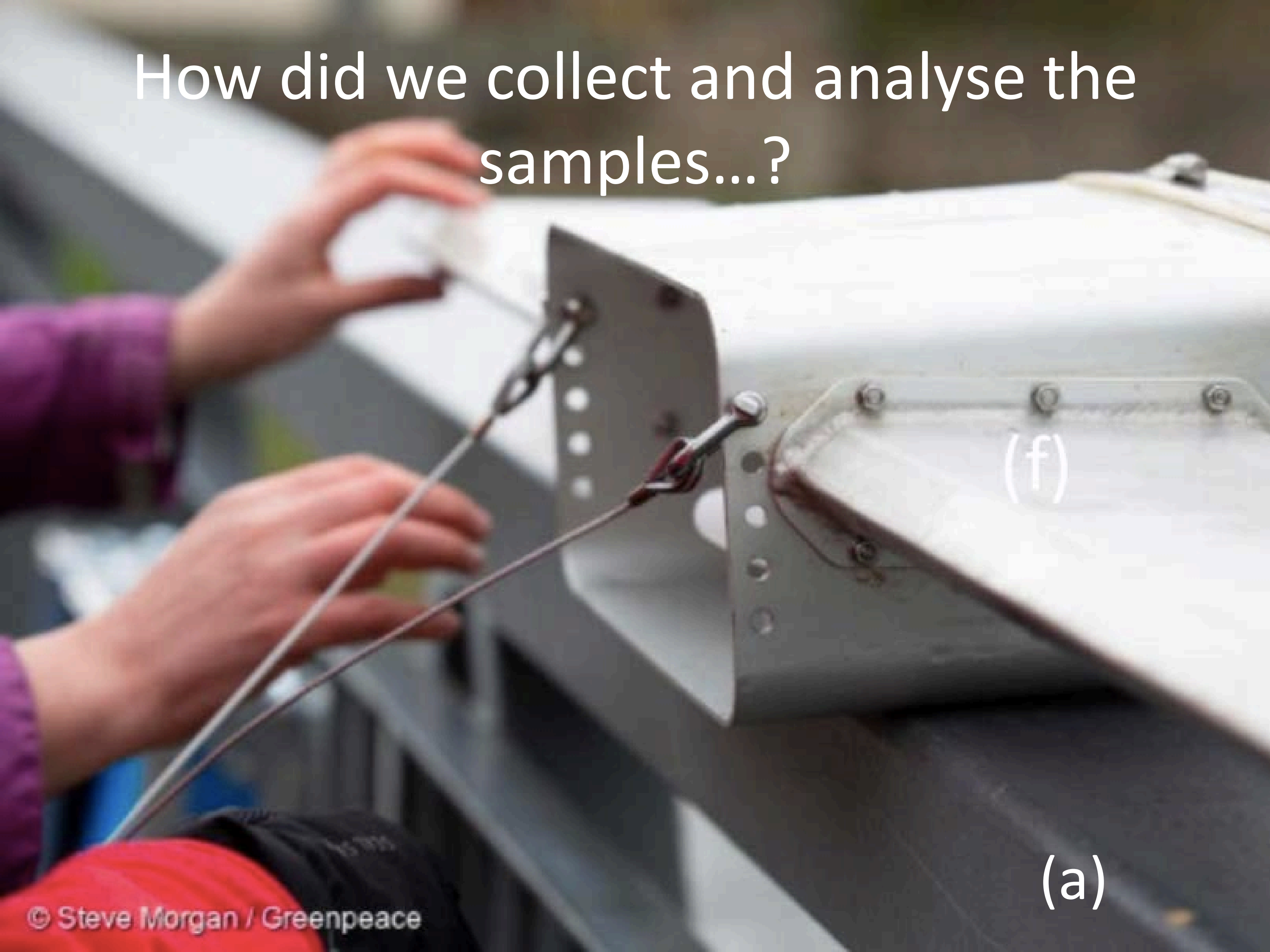
At least one piece of plastic (microplastics <5mm in all dimensions and/or larger items) was found in samples from 28 of the 30 locations, and in samples from at least one of the locations on each river. Across all sampling locations, a total of 1271 pieces of plastic were captured in the nets, ranging in size from plastic straw and bottle top fragments down to tiny microbeads less than 1mm across. Plastic fragments and microbeads less than 2mm in size were the most commonly found, followed by fragments and pellets between 2mm and 5mm. Although concentrations per unit volume or per unit surface area of river water varied widely between locations, on average our results fall in a similar range to those reported in studies of individual rivers in other parts of Europe.

Acknowledgments: The collection of samples for this project was supported by funding from The People's Postcode Lottery. Special thanks to Dr Kirsten Thompson and Kathryn Miller for the preparation of a literature review on which the design of this research project was based.

Where did we look…?

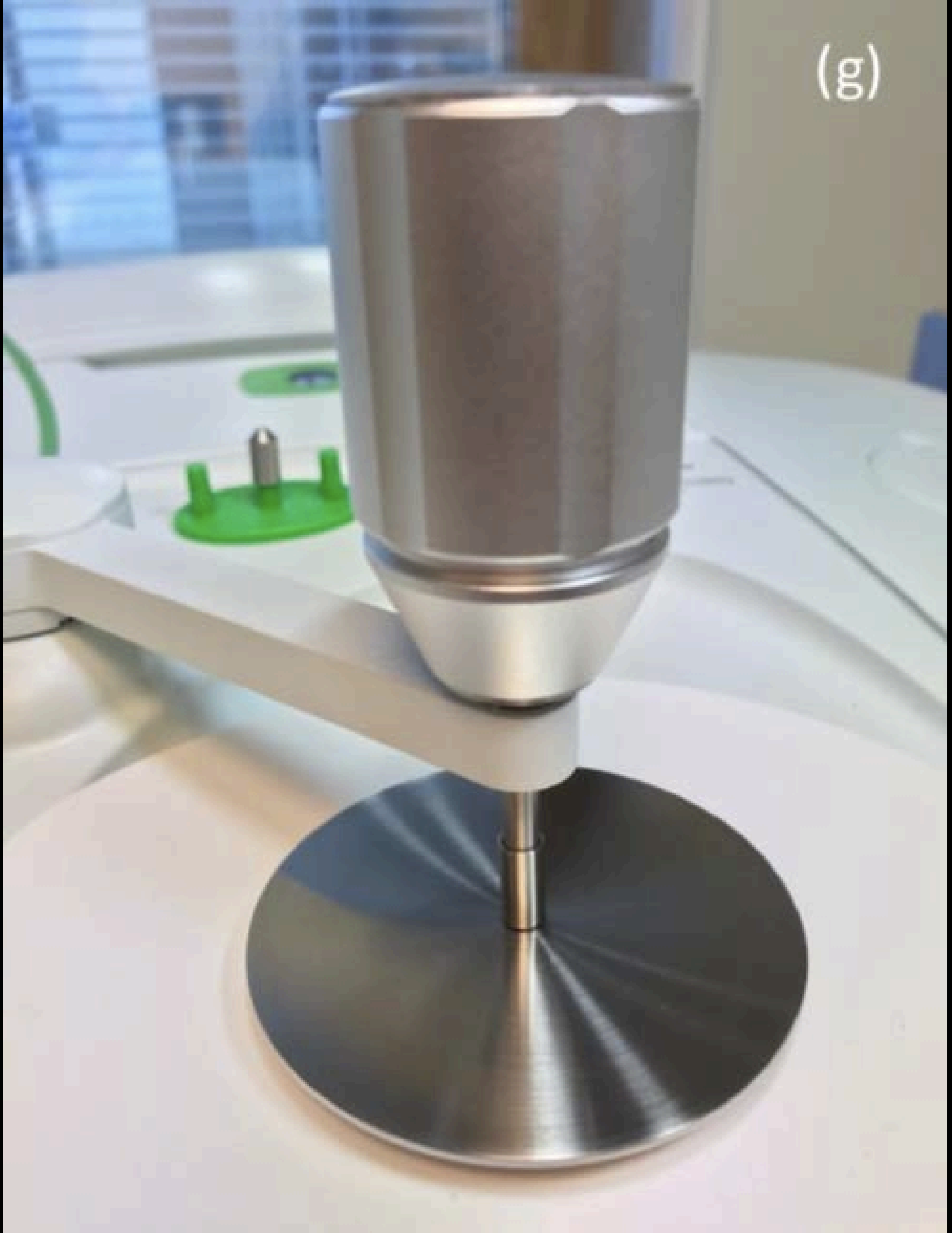
- 13 rivers across the UK…
  - 9 in England
  - 2 in Wales
  - 1 in Scotland
  - 1 in Northern Ireland
- 30 locations in total
- Feb-Mar 2019

How did we collect and analyse the samples…?



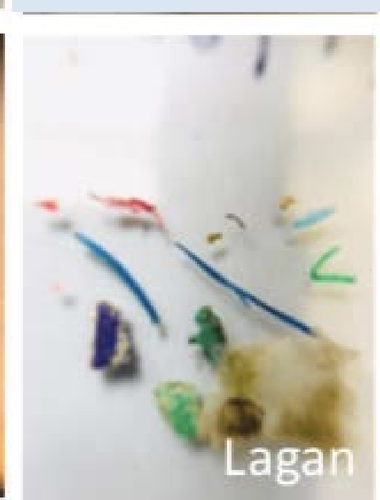
(b)


(d)
© Vanessa Miles / Greenpeace

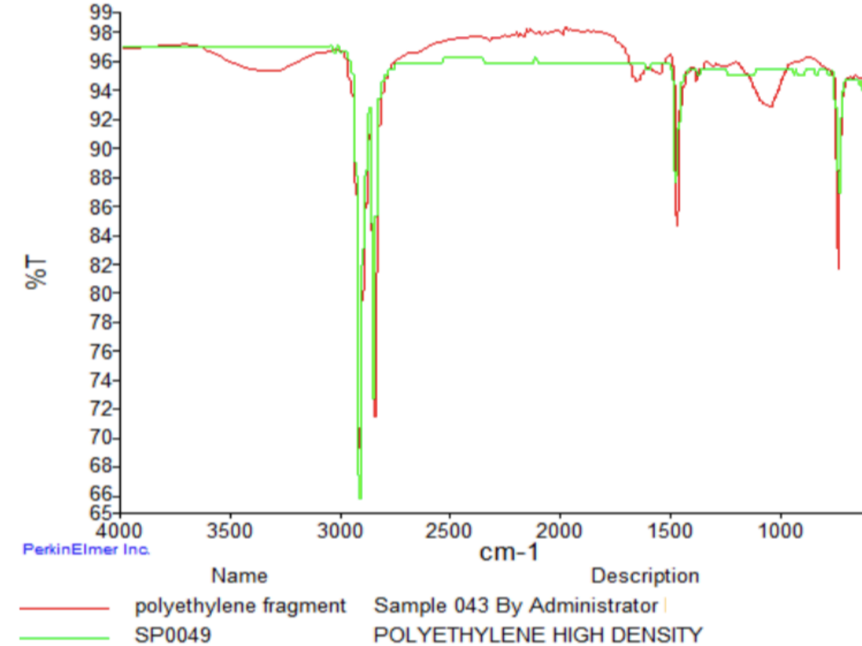

(g)

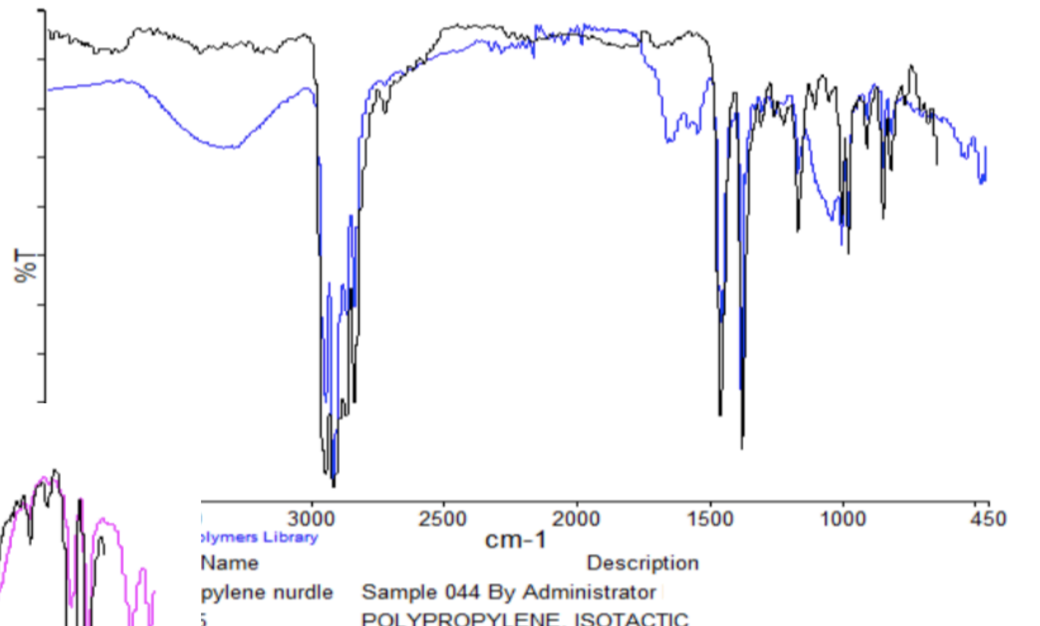

UK 19019
(with #20)
UK 19004
UK 19005
(with #4)

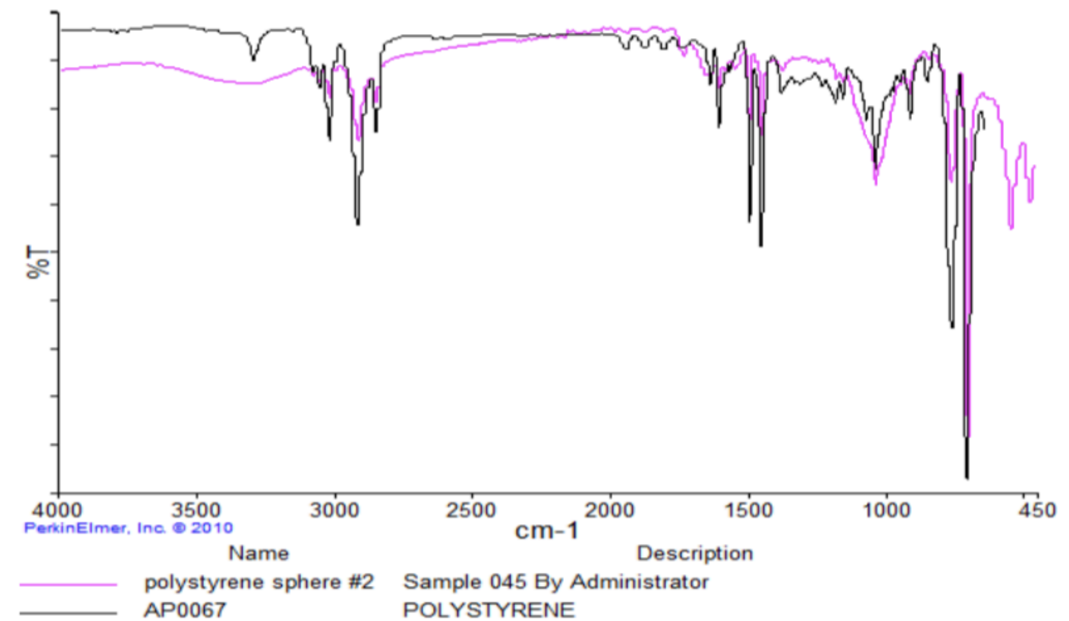
At least one piece of plastic was found at 28 of the 30 locations sampled



Microplastics <2mm (fragments & microbeads) were most abundant, followed by fragments and pellets between 2mm & 5mm

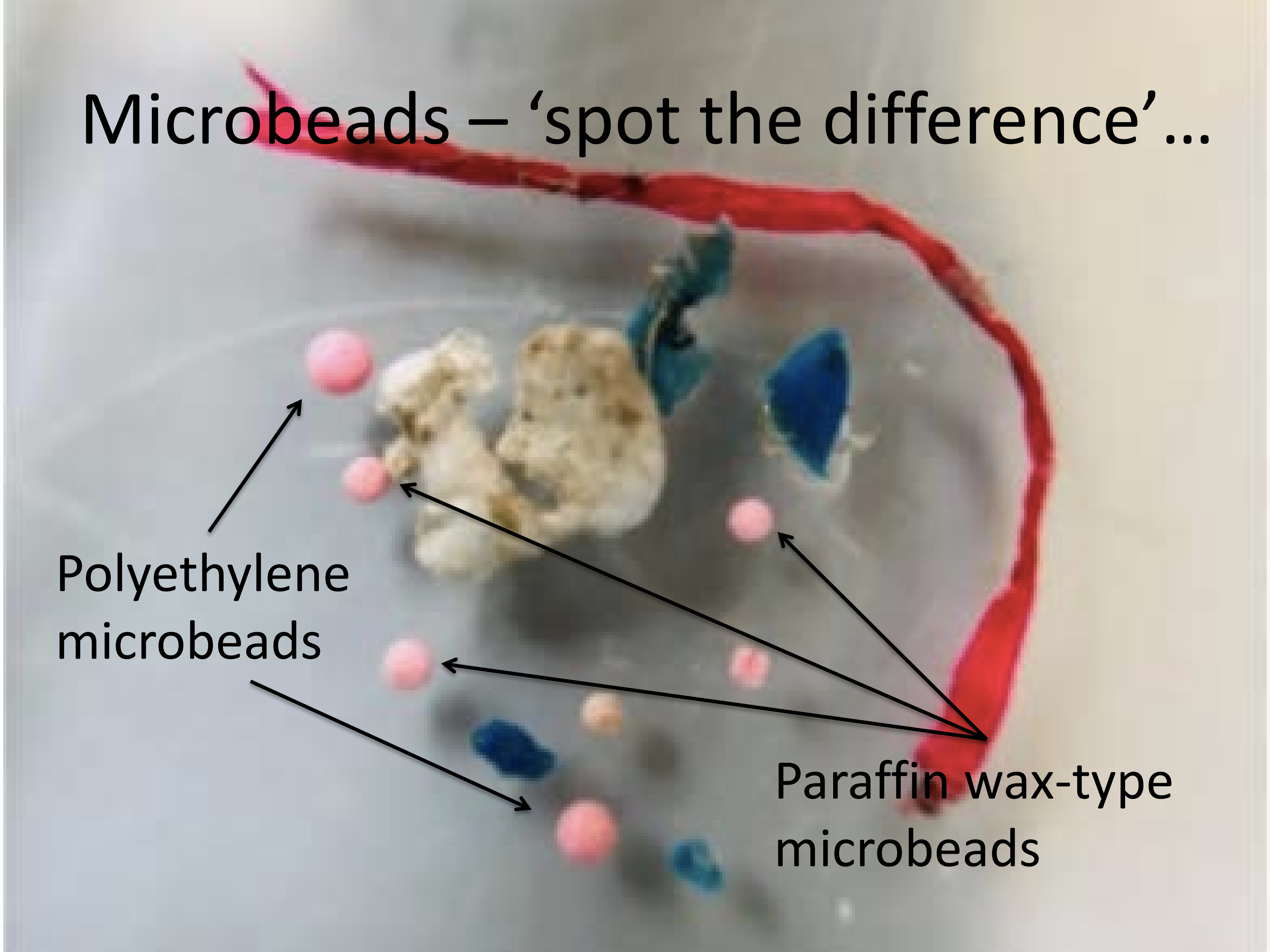

99
98
96
94
92
90
88
86
84
82
80
78
76
74
72
70
68
66
65
%T
4000
3500
3000
2500
2000
1500
1000
cm-1
PerkinElmer Inc.
Name
Description
polyethylene fragment
Sample 043 By Administrator
SP0049
POLYETHYLENE HIGH DENSITY


%T
3000
2500
2000
1500
1000
450
cm-1
olymers Library
Name
Description
pylene nurdle
Sample 044 By Administrator
POLYPROPYLENE, ISOTACTIC


%T
4000
3500
3000
2500
2000
1500
1000
450
cm-1
PerkinElmer, Inc. © 2010
Name
Description
polystyrene sphere #2
Sample 045 By Administrator
AP0067
POLYSTYRENE

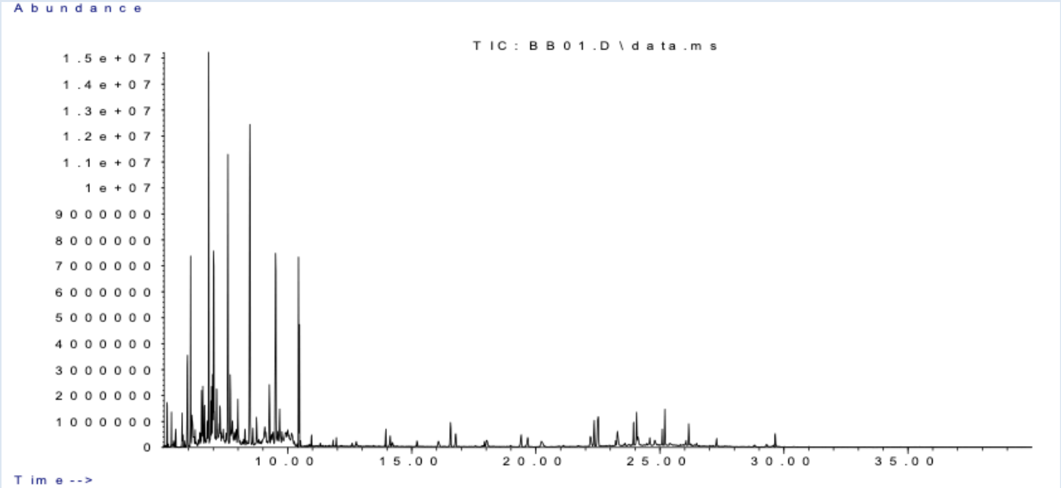
- ‘Nurdles found at 7 locations
- Microbeads found at 5 locations
- ‘Biobeads’ found at 4 locations

Microbeads – 'spot the difference'…



Microplastics as chemical carriers

Microplastics can also carry chemical contaminants…such as these found on the surface of a single biobead from the Mersey…



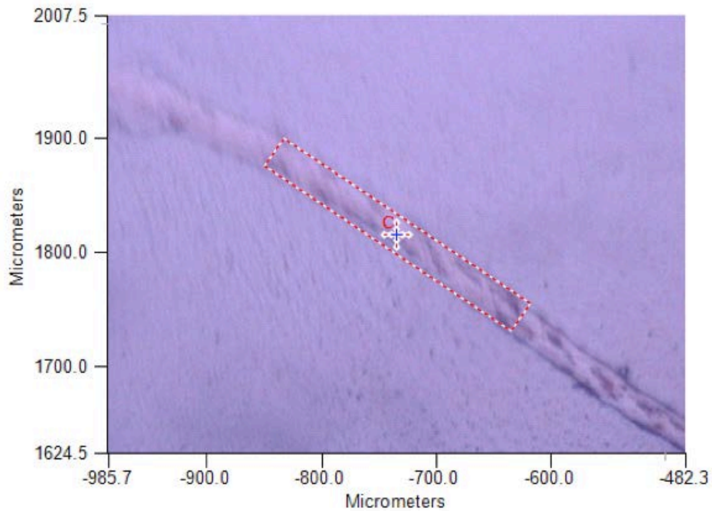
000117-81-7	1,2-Benzenedicarboxylic acid, bis(2-ethylhexyl) ester
004537-15-9	Benzene, (1-butylheptyl)-
002719-62-2	Benzene, (1-pentylheptyl)-
002719-64-4	Benzene, (1-propylnonyl)-
014949-23-6	Cholest-24-ene, (5.beta.)-
000150-60-7	Disulfide, bis(phenylmethyl)-
000057-10-3	Hexadecanoic acid
005129-61-3	Heptadecanoic acid, 16-methyl-, methyl ester
000112-39-0	Hexadecanoic acid, methyl ester
000000-00-0	Linear alkanes & alkenes (C16-C35), 36 compounds
000000-00-0	Branched cyclic and linear alkanes (C15-C20), 4 compounds
000207-08-9	Benzo[k]fluoranthene
000205-99-2	Benzo[b]fluoranthene
000050-32-8	Benzo[a]pyrene

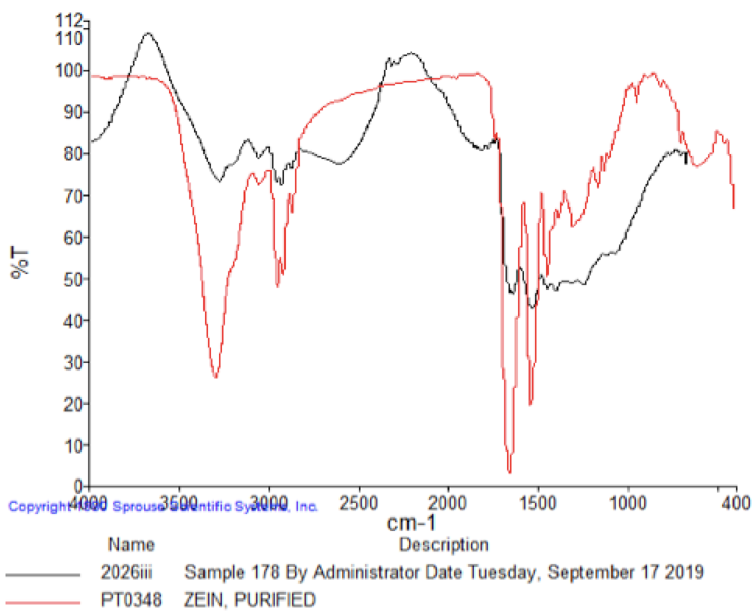
What does it mean for wildlife…?

…for that, the research is only just beginning…

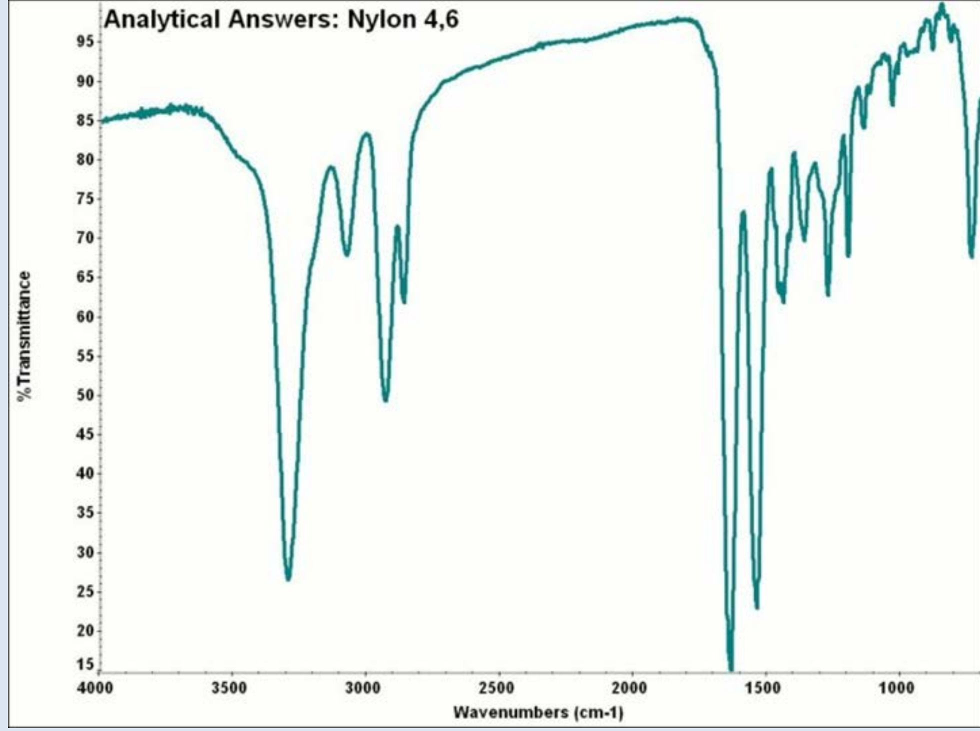
Coming up: microplastics in otter stomachs (with Cardiff University)

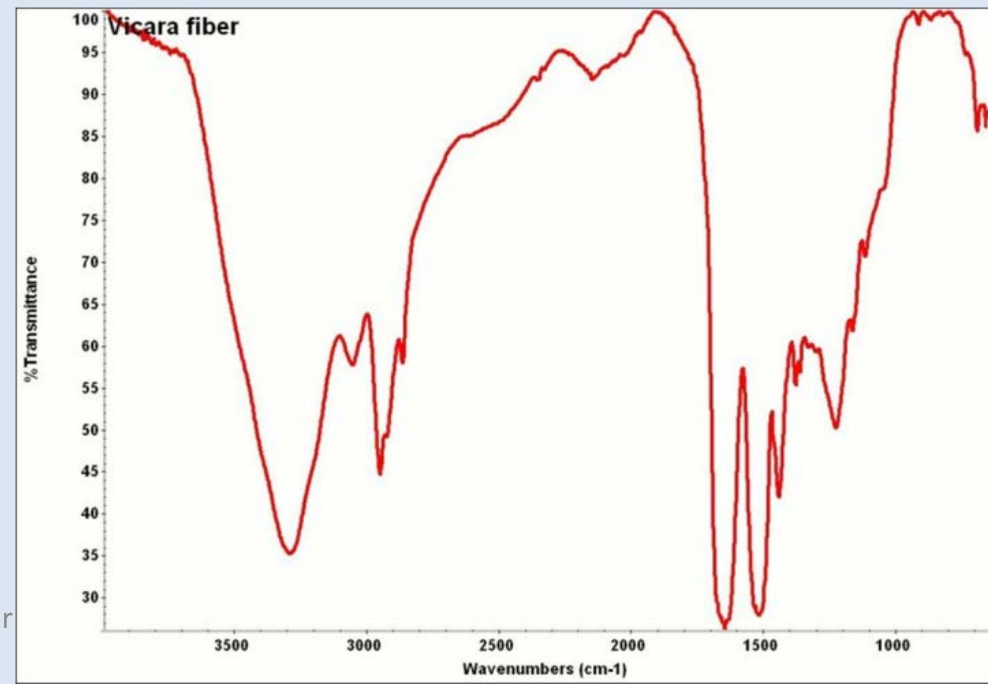
Nylon…or is it protein…?





IR User





Conclusions #1

- FT-IR is a powerful technique in environmental forensics research for plastics
- It is also a valuable tool for visualisation of the problem and public/political engagement
- Environmental samples will always present difficulties for characterisation
- Need a mix of techniques and combined lines of evidence (IR spectrum, physical properties, pyrolysis GC-MS, Raman, others…?)

Conclusions #2

- Plastic (and microplastic) pollution is ubiquitous (though far worse in some areas than in others)
- Understanding of environmental fate and effects of microplastics remains limited
- Techniques for rapid screening of sediments are still something of a holy grail
- Cellulose is a common component of microfibre contamination – but where is it all coming from?