Standardisation of terminology and measurement for graphene and related 2D materials

Dr Andrew J. Pollard

National Physical Laboratory (NPL), UK

Outline

- The National Physical Laboratory (NPL)
- Metrology for graphene and related 2D materials
  - Example Case Study
- International Standardisation
  - Past and Current Landscape
  - Graphene Terminology
  - Standardisation of Measurement

About NPL …

The UK's national standards laboratory

- Founded in **1900**

- World leading **National Measurement Institute**

- 600+ specialists in **Measurement Science**

- State-of-the-art laboratory facilities

- The heart of the UK's **National Measurement System** to support business and society

- Experts in **Knowledge Transfer**
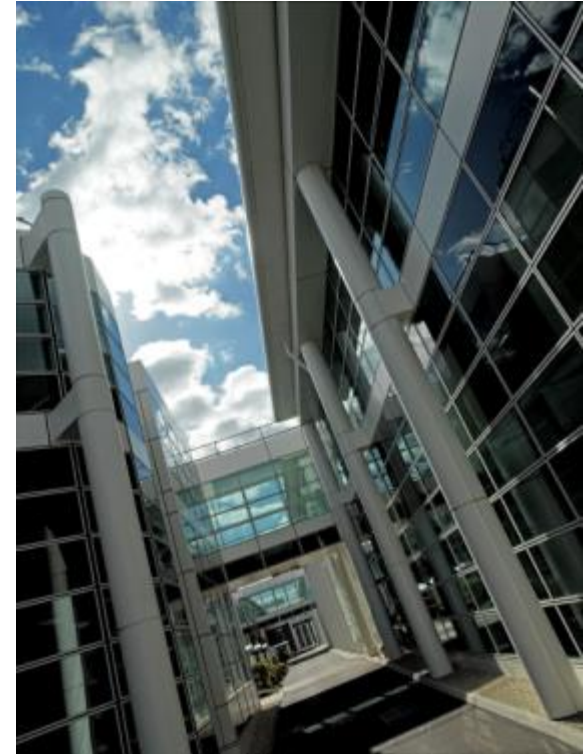
Graphene at NPL

- NPL has 20+ scientists and engineers working on 2D material related projects (and 10+ students)

- Improved access for Industry, Academia and Government to 2D material capability across NPL:

  - Characterisation
  - Metrology
  - Standardisation
  - Quality Control

- Different properties of materials require different approaches

- Formalised strategic partnering with key organisations
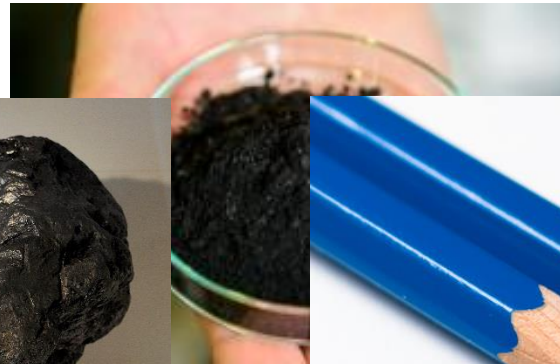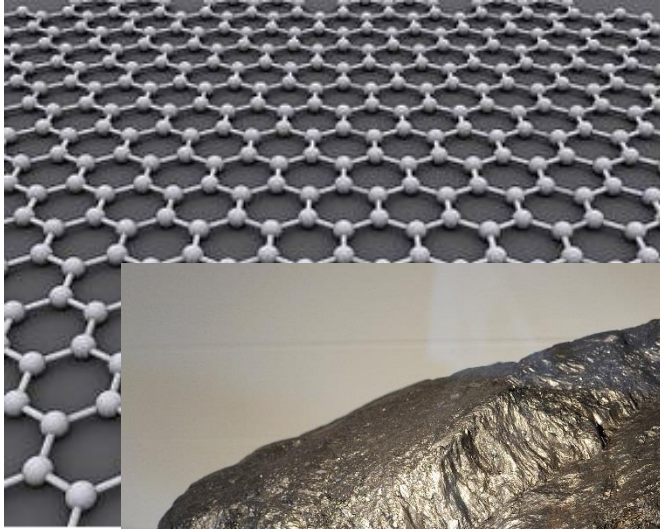
What is this stuff?!

Graphenea

Graphene Oxide
water dispersion

250 mL
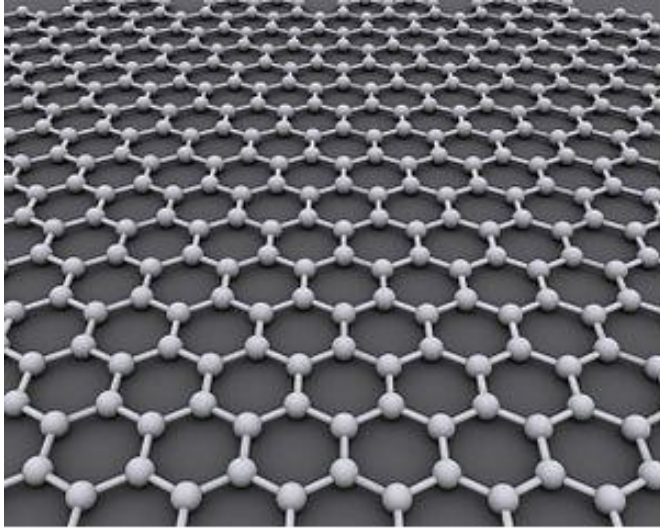
www.graphenea.com
www.appliedgraphenematerials.com
DerHexer, Wikimedia Commons, CC-by-sa 4.0
www.davidkelly.me

What is this stuff?!

What is this stuff?!

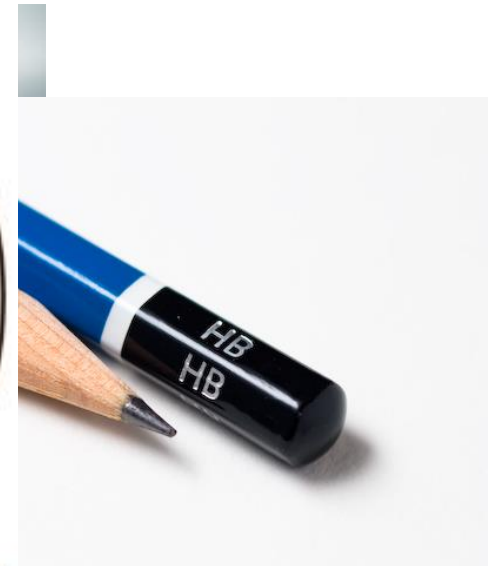SNAKE OIL PROMOTIONS

Graphenea
Graphene Oxide
water dispersion
250 mL

HB
HB
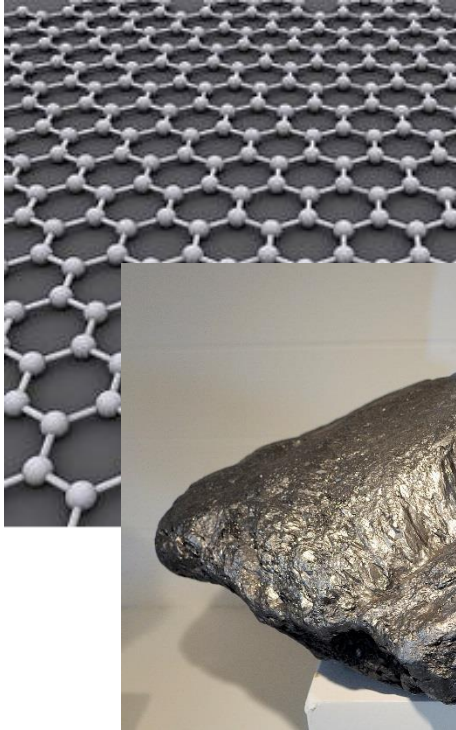
Characterisation of Printed Graphene Films

- Commercial company using graphene powders to make printed graphene electrodes
  - Different polymer binders/composites used

Brennan *et al.* App. Surf. Sci. **403** 403-412 (2017)
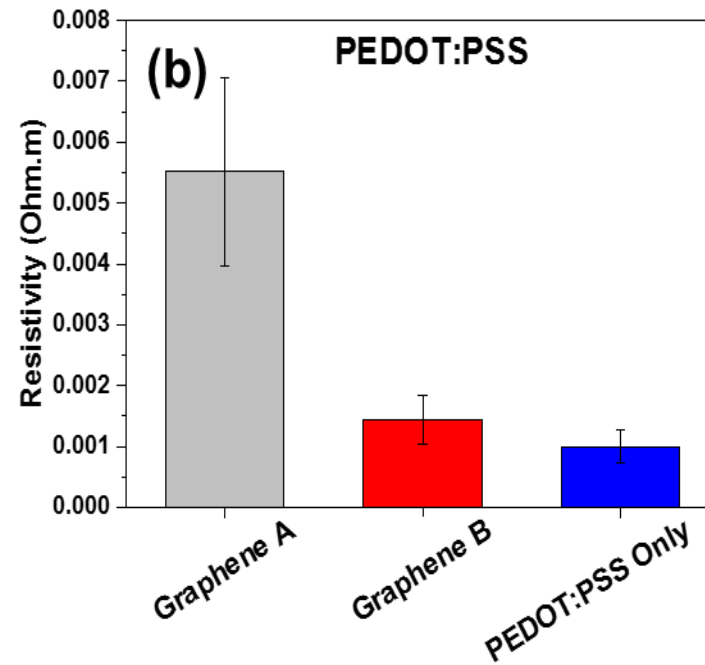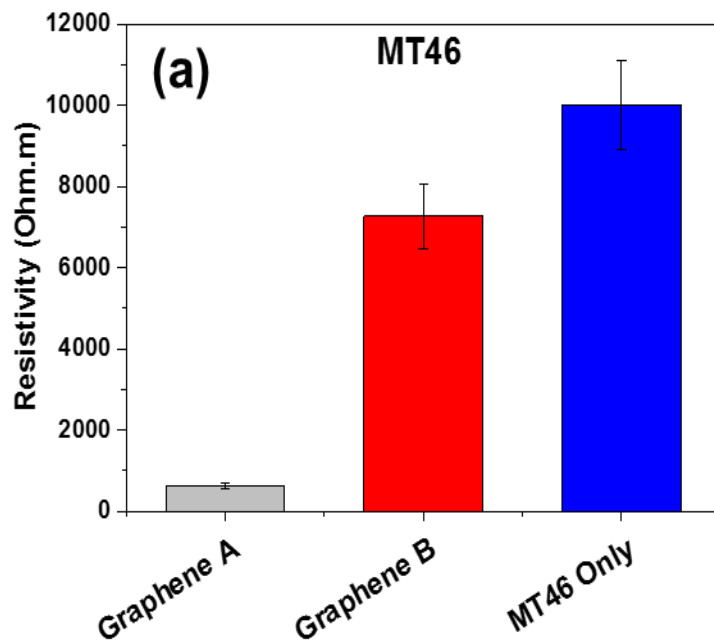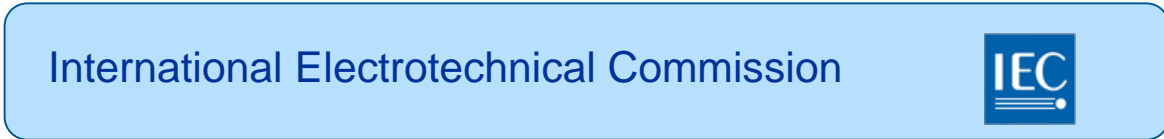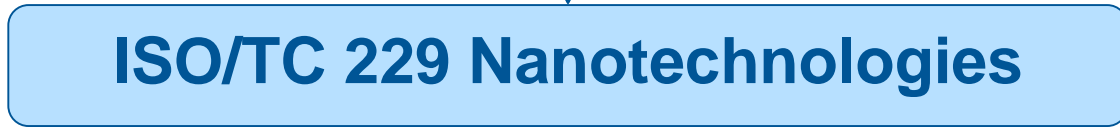
International Standardisation

International Standardisation

NPL
National Physical Laboratory

2 GRAPHENE STANDARDS PUBLISHED!!
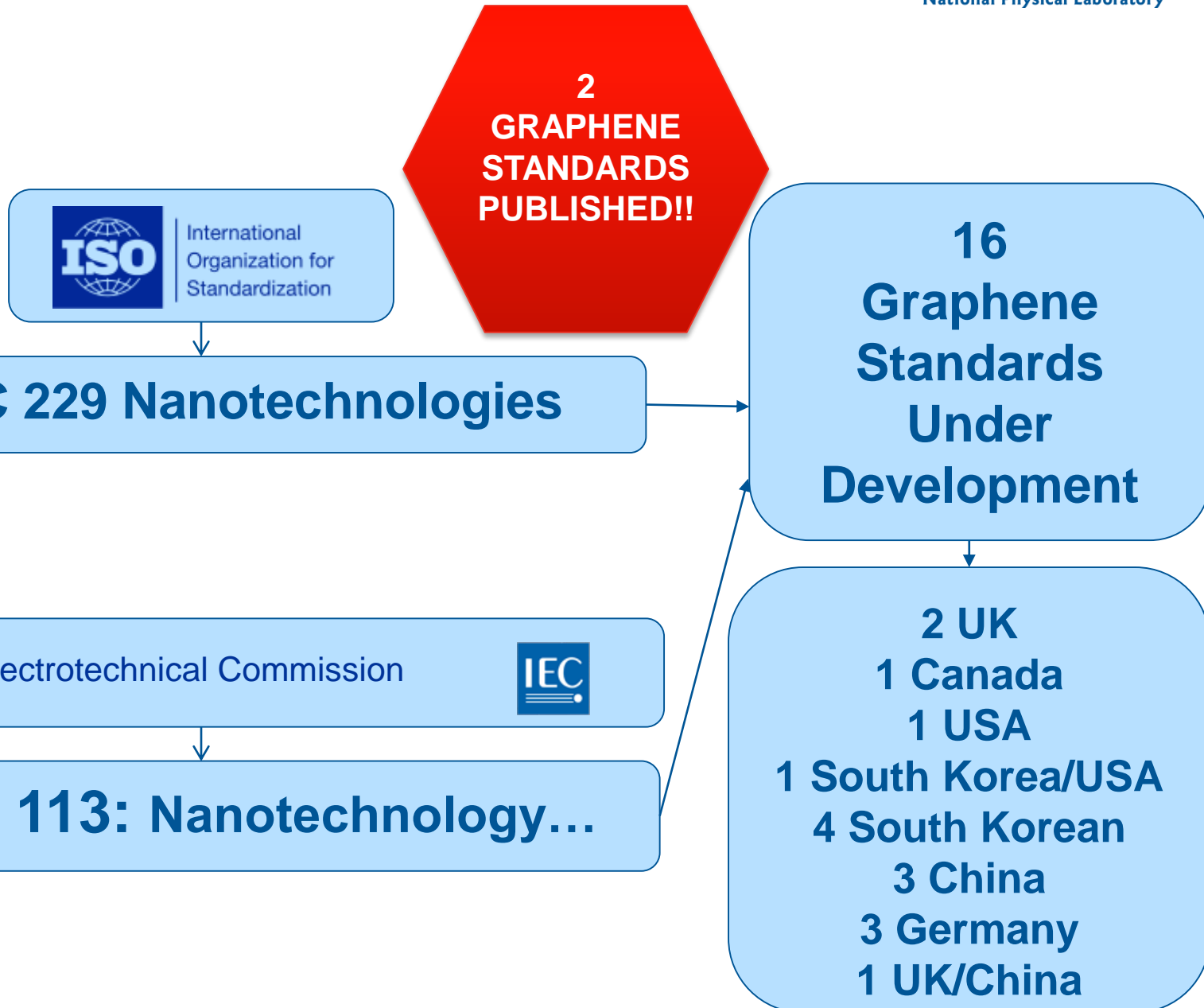
International Organization for Standardization (ISO)

↓

ISO/TC 229 Nanotechnologies →

16 Graphene Standards Under Development

↓

International Electrotechnical Commission (IEC)

↓

IEC TC 113: Nanotechnology…

**2 UK
1 Canada
1 USA
1 South Korea/USA
4 South Korean
3 China
3 Germany
1 UK/China**

International Standards Published

- **IEC TS 62607-6-4:2016**
- Nanomanufacturing - Key control characteristics - Part 6-4: Graphene - Surface conductance measurement using resonant cavity
  - USA

- **ISO/TS 80004-13:2017**
- Nanotechnologies - Vocabulary - Part 13: Graphene and related two-dimensional (2D) materials
  - UK

Terminology Standard

3.1.1.1
two-dimensional material
2D material
material, consisting of one or several layers with the atoms in each layer strongly bonded to neighbouring atoms in the same layer, which has one dimension, its thickness, in the nanoscale or smaller and the other two dimensions generally at larger scales

Note 1 to entry: The number of layers when a two-dimensional material becomes a bulk material varies depending on both the material being measured and its properties. In the case of graphene layers, it is a two-dimensional material up to 10 layers thick for electrical measurements, beyond which the electrical properties of the material are not distinct from those for the bulk [also known as graphite].
Note 2 to entry: Interlayer bonding is distinct from and weaker than intralayer bonding.
Note 3 to entry: Each layer may contain more than one element.
Note 4 to entry: A two-dimensional material can be a nanoplate.

3.1.2.1
graphene
graphene layer
single layer graphene
monolayer graphene
single layer of carbon atoms with each atom bound to three neighbours in a honeycomb structure

Note 1 to entry: It is an important building block of many carbon nano-objects.
Note 2 to entry: As graphene is a single layer, it is also sometimes called monolayer graphene or single layer graphene and abbreviated as 1LG to distinguish it from bilayer graphene (2LG) and few-layered graphene (FLG).
Note 3 to entry: Graphene has edges and can have defects and grain boundaries where the bonding is disrupted.

3.1.2.10
few-layer graphene
FLG
two-dimensional material consisting of three to ten well-defined stacked graphene layers

3.1.2.13
graphene oxide
GO
chemically modified graphene prepared by oxidation and exfoliation of graphite, causing extensive oxidative modification of the basal plane

Note 1 to entry: Graphene oxide is a single-layer material with a high oxygen content, typically characterized by C/O atomic ratios of approximately 2,0 depending on the method of synthesis.

4 Abbreviated terms

1L	monolayer/single-layer
1LG	monolayer/single-layer graphene
2D	two-dimensional
2L	bilayer
2LG	bilayer graphene
3L	trilayer
3LG	trilayer graphene
CVD	chemical vapour deposition
FL	few-layer
FLG	few-layer graphene
GNP	graphene nanoplatelet
GO	graphene oxide
hBN	hexagonal boron nitride
rGO	reduced graphene oxide

Terminology Standard

3.1.1.1
two-dimensional material
2D material
material, consisting of one
neighbouring atoms in the
or smaller and the other tw

Note 1 to entry: The number
depending on both the materi
two-dimensional material up
properties of the material are
Note 2 to entry: Interlayer bon
Note 3 to entry: Each layer ma
Note 4 to entry: A two-dimensi

3.1.2.1
graphene
graphene layer
single layer graphene
monolayer graphene
single layer of carbon ato
structure

Note 1 to entry: It is an import
Note 2 to entry: As graphene
layer graphene and abbreviat
graphene (FLG).
Note 3 to entry: Graphene ha
disrupted.

3.1.2.10
few-layer graphene
FLG
two-dimensional material c

3.1.2.13
graphene oxide
GO
chemically modified graph
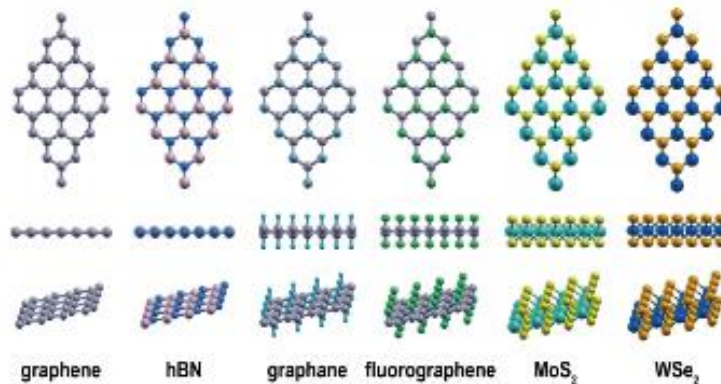extensive oxidative modification of the basal plane

Note 1 to entry: Graphene oxide is a single-layer material with a high oxygen content, typically characterized by C/O atomic ratios of approximately 2,0 depending on the method of synthesis.

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Journal of Materials Science

Invited Viewpoint: A New ISO Standard for Graphene and Related 2D Materials

graphene   hBN   graphane fluorographene   MoS₂   WSe₂

:d terms

ayer/single-layer

ayer/single-layer graphene

mensional

graphene

r

r graphene

:al vapour deposition

/er

/er graphene

ne nanoplatelet

ne oxide

nal boron nitride

d graphene oxide

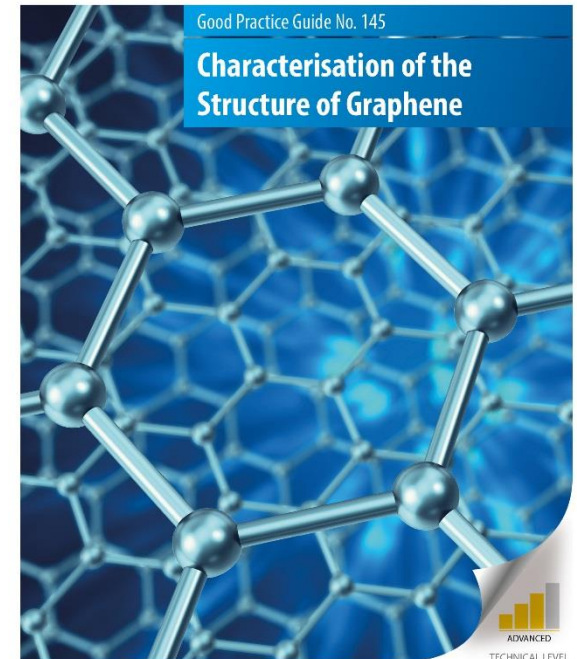Pollard *et al*. J. Mater. Sci. **52**, 13685 (2017)

Measurement Standard

- ISO/PWI 21356 'Nanotechnologies -- Structural characterization of graphene'
- Scope:

*"…best practice for characterising the structural properties of graphene using a range of suitable measurement techniques. …both samples consisting of **a layer of graphene** on a substrate and graphene in **dispersion/powder form** …flowchart of technique combinations. The sample preparation…data analysis [included].*
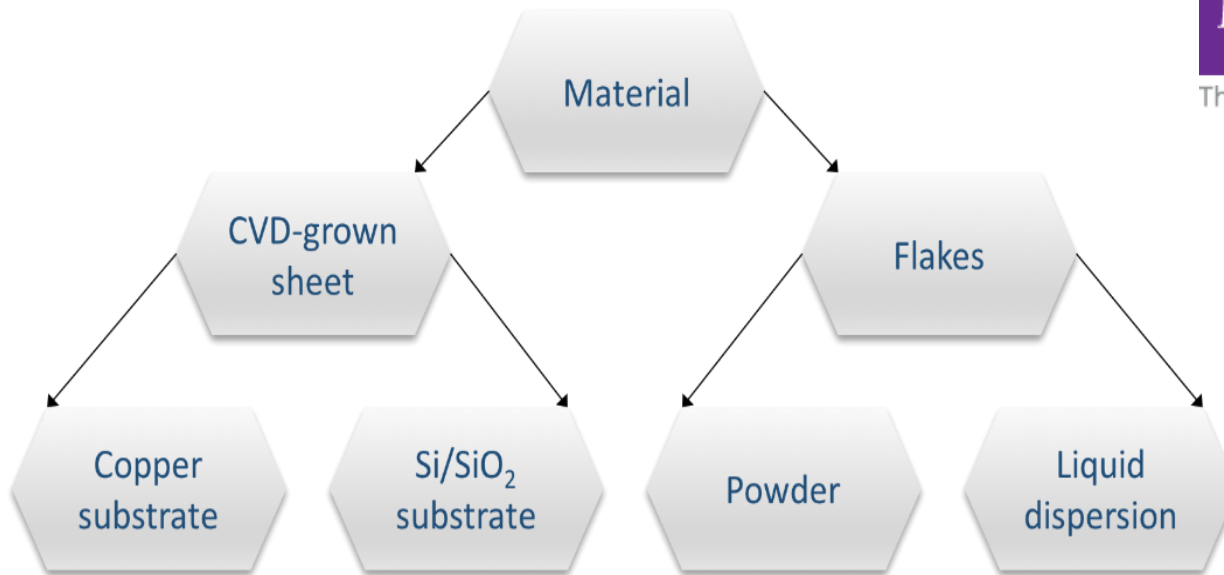
Properties such as the number of layers (thickness), lateral flake size, lateral domain size, the level of disorder, layer stacking, surface area, as well as the distribution and relation of these properties, will be included."

Good Practice Guide

- Good practice guide (GPG) series released by NPL

- Joint NPL and NGI (Manchester) GPG
  - www.npl.co.uk/graphene-guide

- Technique, issues, protocol, data analysis

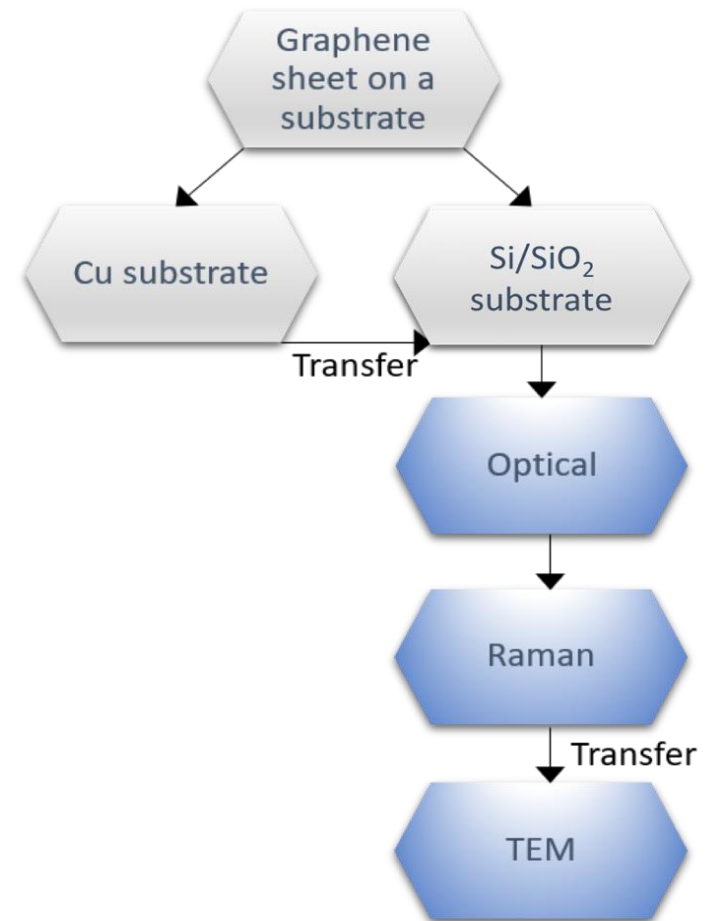- GPG can be used as interim source as international standard developed

Good Practice Guide No. 145

Characterisation of the Structure of Graphene

ADVANCED
TECHNICAL LEVEL

Type of material

Material

CVD-grown sheet → Copper substrate, Si/SiO$_2$ substrate
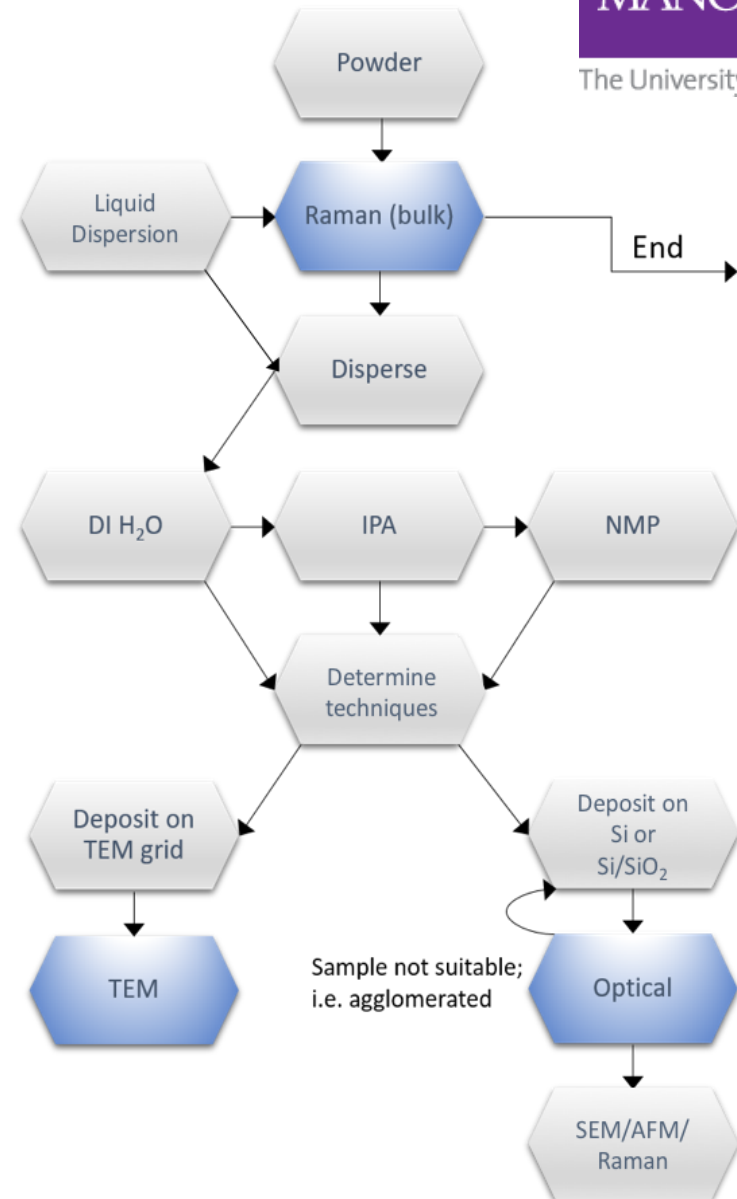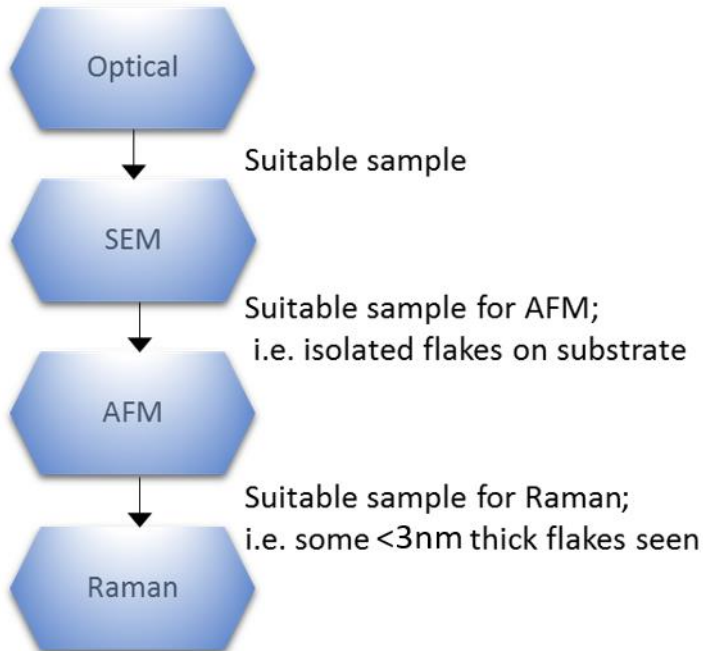
Flakes → Powder, Liquid dispersion

CVD-grown Graphene

Powders/Dispersions

Next Steps In Metrology

- Rigorous scientific investigation required to understand measurement
  - Several years timeframe
- Develop to be applicable to Industry
- Using the VAMAS framework
  - International collaboration for measurement and material comparison
- New Area now initiated 'TWA 41: Graphene and Related 2D Materials
  - Co-chaired by NPL and NIM, China
  - XPS, ICP-MS, Raman and AFM

Summary

- Metrology investigations of graphene and related 2D materials required for commercialisation
- Many standards currently in development with activity growing
- **Graphene Terminology Standard now published**
- Graphene Measurements Standards being developed
- **Good Practice Guide on measurement of graphene structure now published**
- Interlaboratory comparisons through VAMAS

NGMC@NPL

Barry Brennan
Keith Paton
Charles Clifford
Elizabeth Legge

University of Manchester

Antonios Oikonomou
Sarah Haigh
Cinzia Casiraghi
Lan Nguyen
Daniel Kelly

Dr Andrew Pollard
National Physical Laboratory
andrew.pollard@npl.co.uk
www.npl.co.uk/graphene

DZP Technologies

Zlatka Stoeva
Harry Cronin
Tsegie Faris

ISO International Organization for Standardization

IEC

Department for Business, Energy & Industrial Strategy

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