

# Use of Raman and Cold Infra-red spectroscopy for the measurement of respirable crystalline silica (RCS) and their potential application.

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# The challenge

- Limit of detection (LOD) and quantification (LOQ) of 'traditional' techniques (X-ray Diffraction and Infrared)

LODs 5 - 10  $\mu\text{g}$

LOQs 10 – 25  $\mu\text{g}$

- Part shift sampling at 2.2 l/min and attempting to control to 0.05  $\text{mg}/\text{m}^3$  Respirable Crystalline Silica

Quarter shift 2 h sampling = 13  $\mu\text{g}$

Half shift 4 h sampling = 26.4  $\mu\text{g}$

– Best practice to sample to exceed uncertainty of LOQ or LOD

- Expected measurement range from in-mask sampling

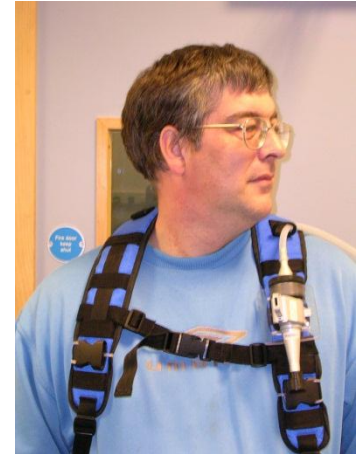
0.2 – 2.5  $\mu\text{g}$  collected

(weighing of test samples extremely difficult)

(cross certification with other techniques difficult)

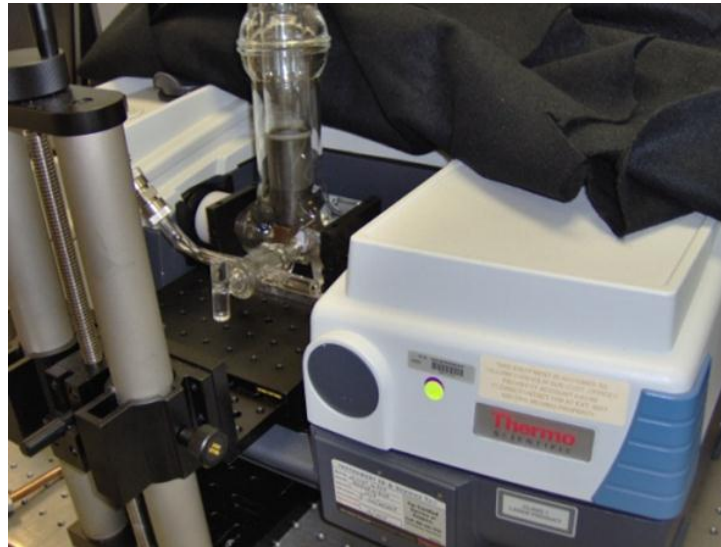
# High flow rate respirable samplers

- Operate at 4 – 10 l/min – useful option
- Battery and pumps - heavy for workers
- Samplers quite large – get in the way
- Samplers and pumps – can be expensive
- Looking at new approaches that permit use of:
  - existing samplers
  - miniature samplers for in-mask measurement



# Cold Infra-red Spectroscopy

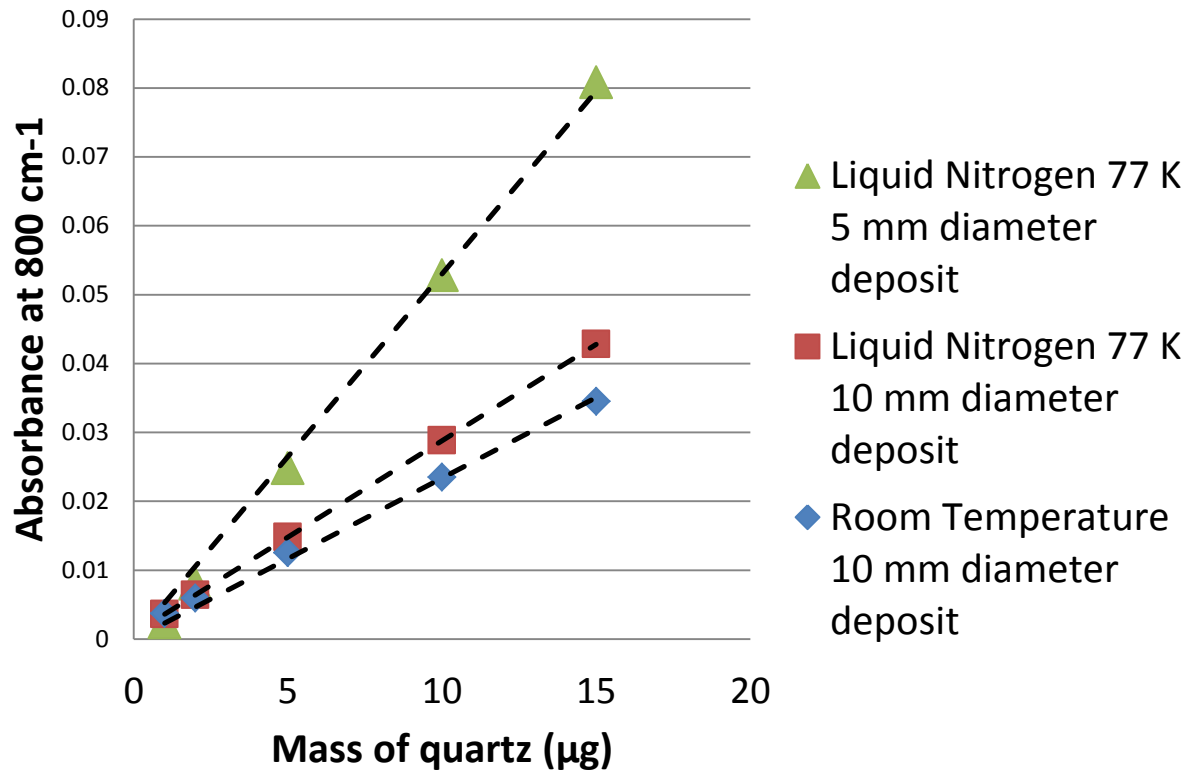
- Collaboration with National Institute for Occupational Safety and Health (USA)



- Low cost (£~5k) adaptation of existing instrument

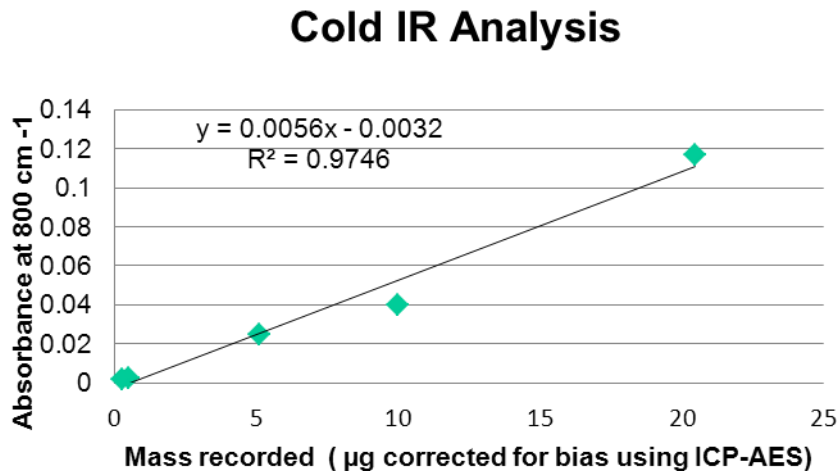


# Initial Results



# Cold Infra red spectroscopy

- Potential for routine use – analysis < 10 mins
- Lowest mass measured ~ 0.3 µg on a 5 mm diameter deposit



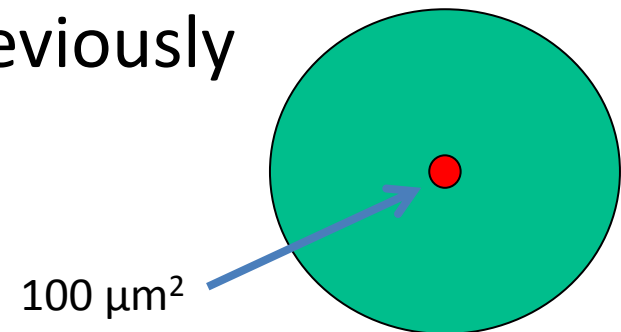
# Raman - An Opportunity ?

- Collaboration with Sheffield Hallam University (United Kingdom)
- Low concentration environments
  - e.g. inside a mask
- Low mass and matrix collection
- Concentrated into small deposit area



Permits optical techniques not previously considered for RCS measurement

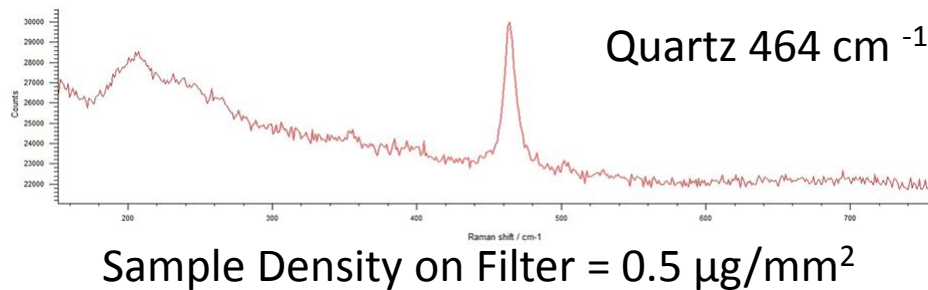
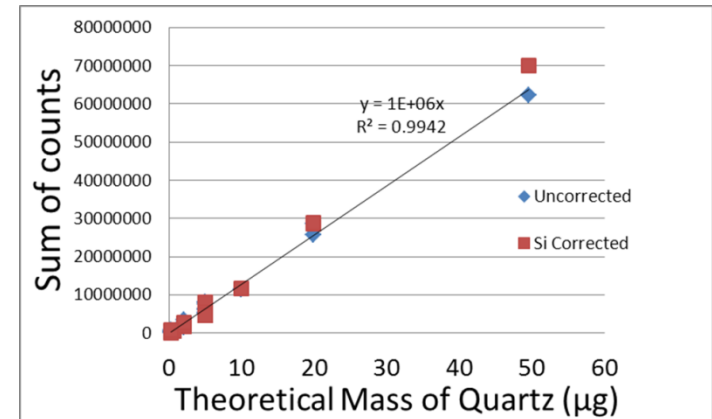
Raman Spectroscopy





# Raman – Analysis of in mask samples

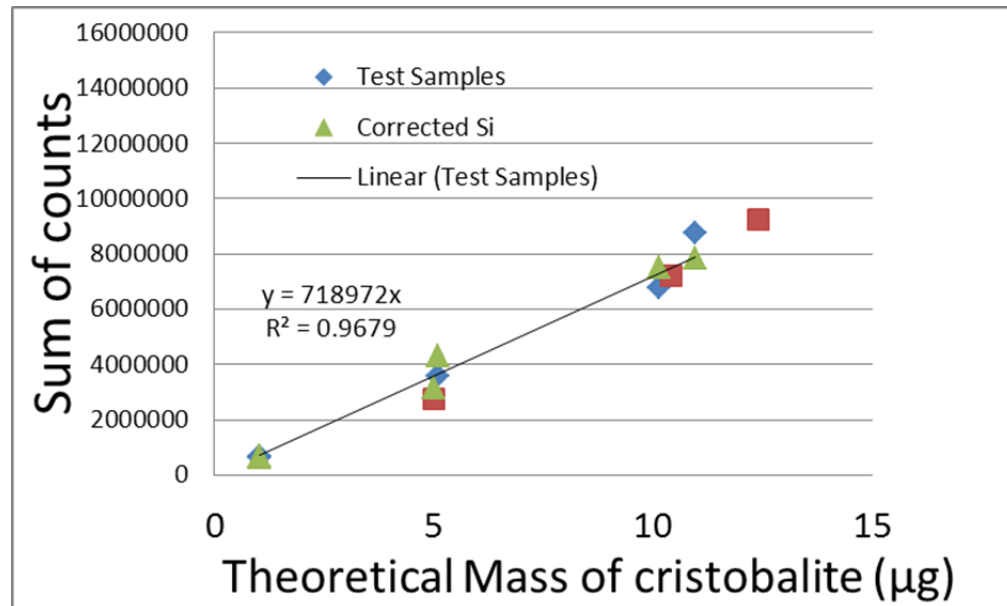
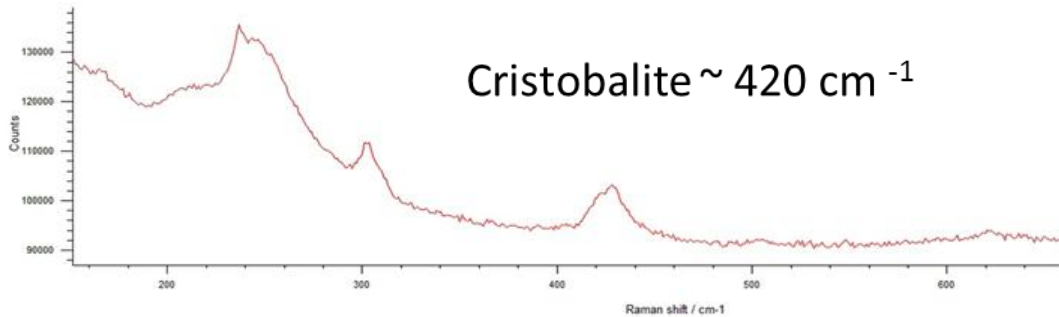
- Analysis time = TEM
- Needs small samples < 10  $\mu\text{g}$
- Can measure approx 0.15  $\mu\text{g}$
- Estimated ideal LOD in matrix ~ 1.5 %





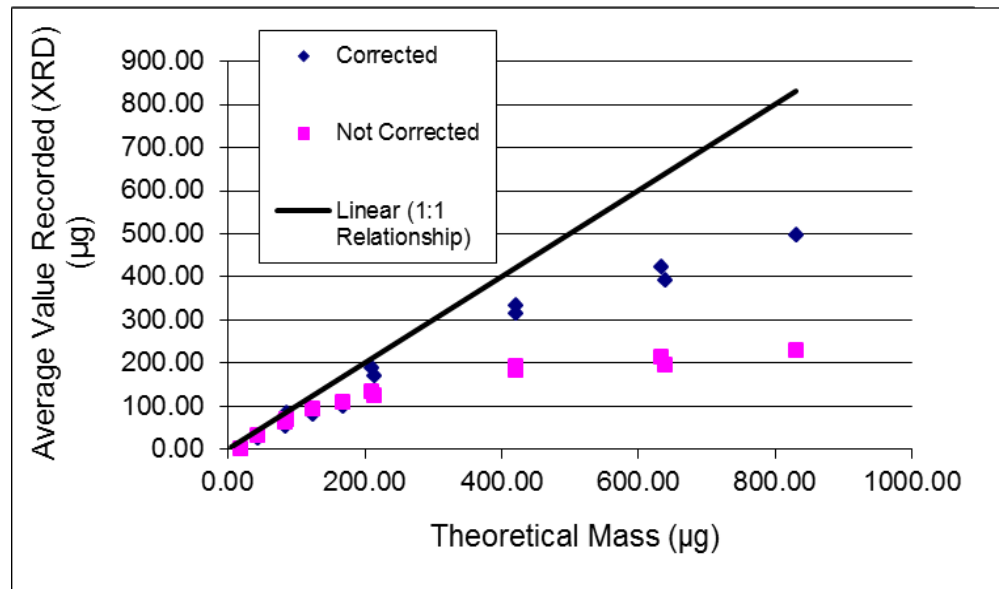


# Cristobalite - Polymorph



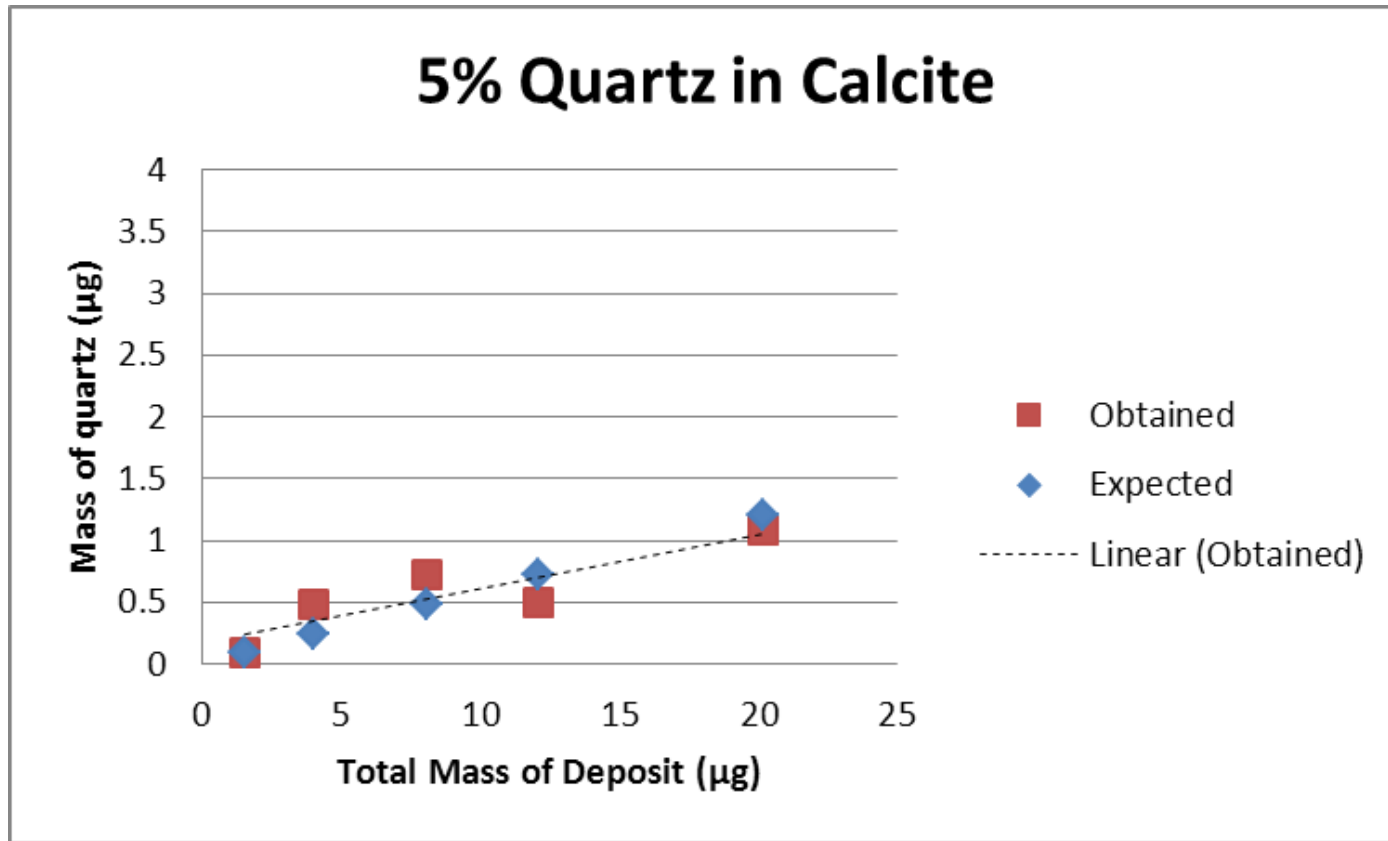
# What happens in a hematite matrix ?

Can we measure quartz in more complex mixtures ?



Mecchia M, Petorius C, Stacey P, Mattenklott M, Incocciata E, X-ray Absorption Effect in Aerosol Samples Collected on Filter Media, Silica and Associated Respirable Mineral Particles, STP 1565, Martin Harper and Taekhee Lee Eds, ASTM International

# What happens in a calcite matrix ?



# Added value research

- International and national collaboration
- Paper pending decision
- Two techniques offer potential improvements
  - specialist applications
- Next step - evaluation of real samples
- If we are to move towards miniaturisation - we first need the tools to provide meaningful data.

# Thank you

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- *Its contents, including any opinions and/or conclusions expressed, are those of the authors alone and do not necessarily reflect HSE or NIOSH/CDC policy*