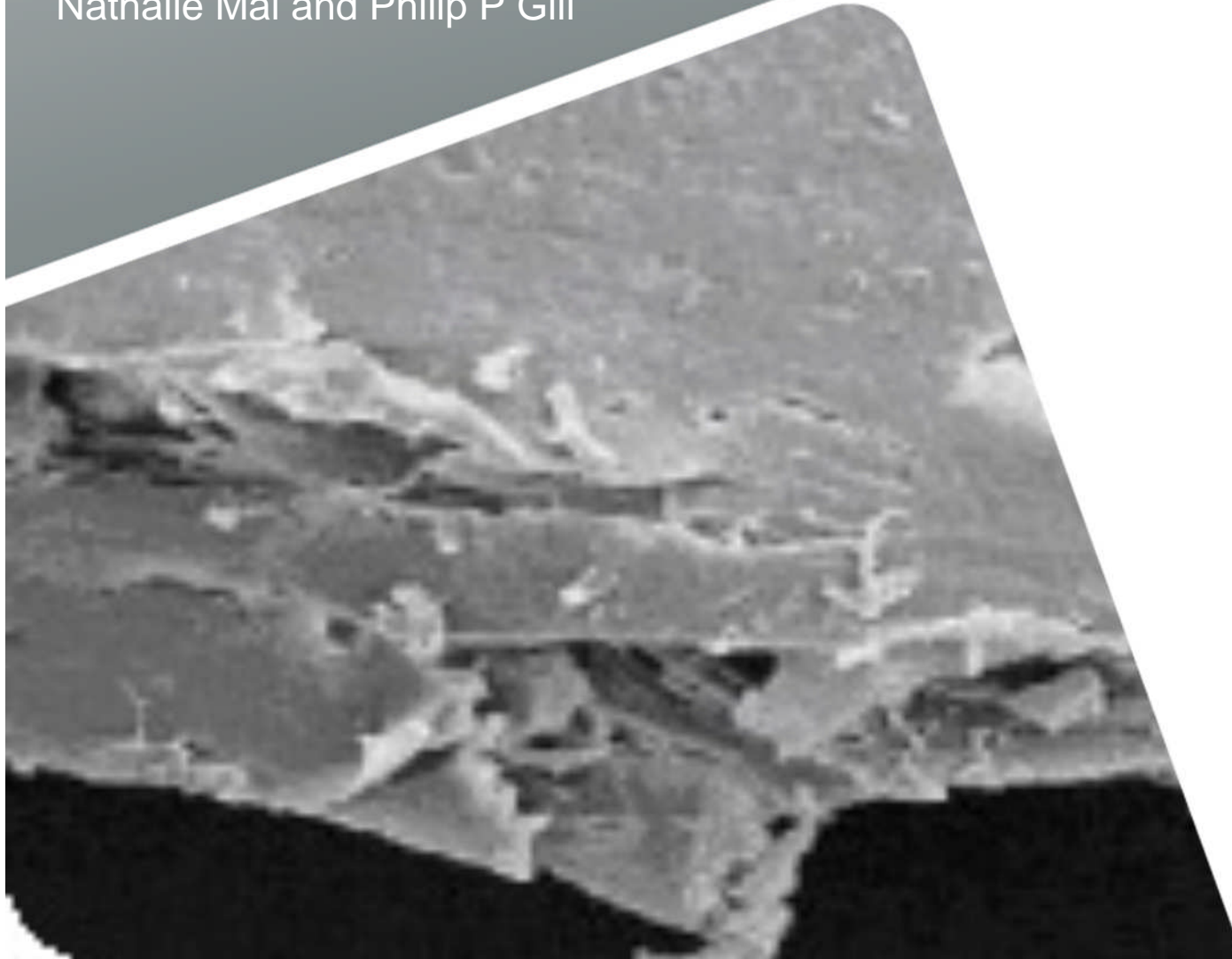


ISO/IEC 17025: GPC analysis of NC

18th April 2012

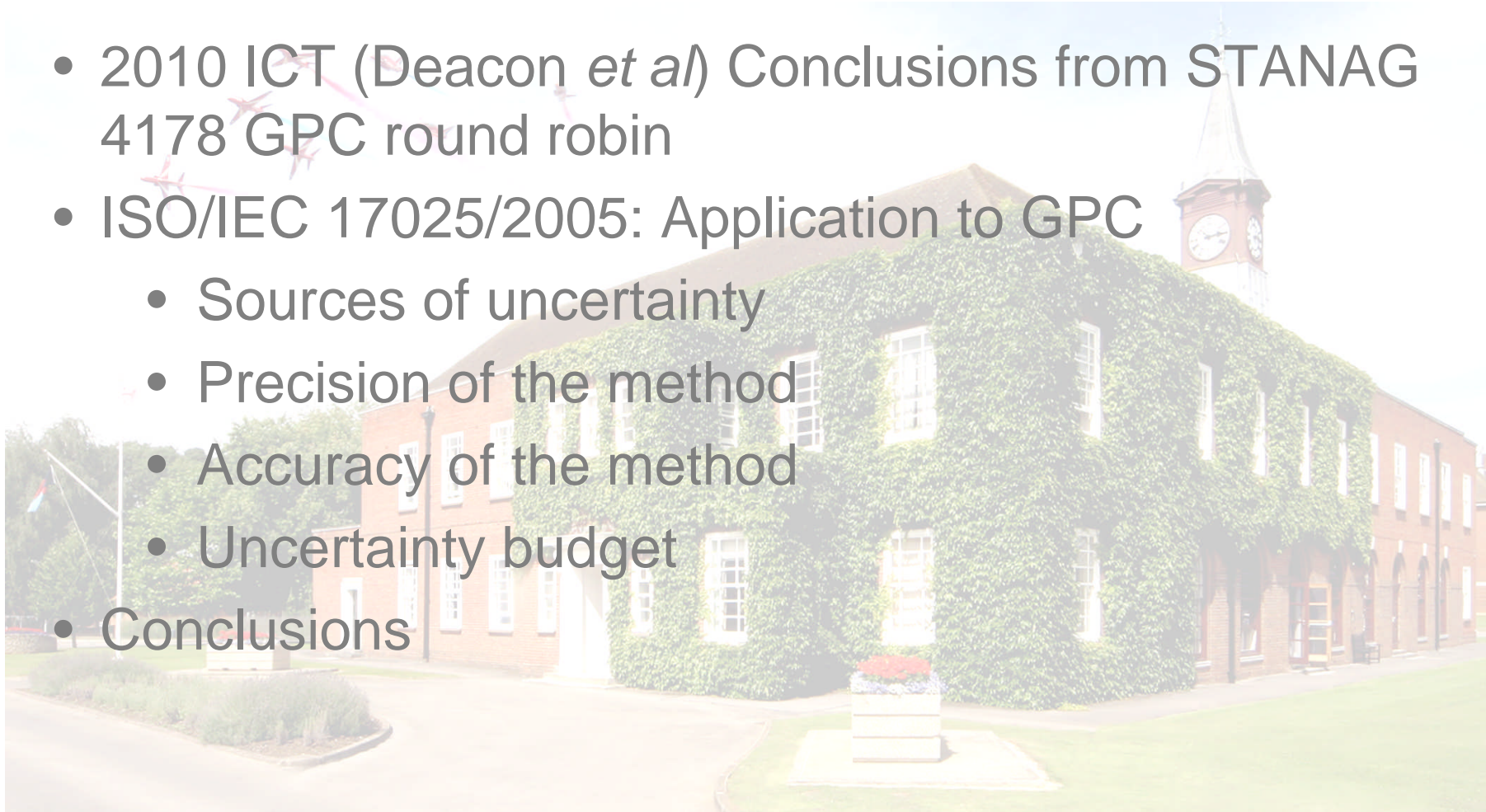
Nathalie Mai and Philip P Gill

Cranfield
UNIVERSITY



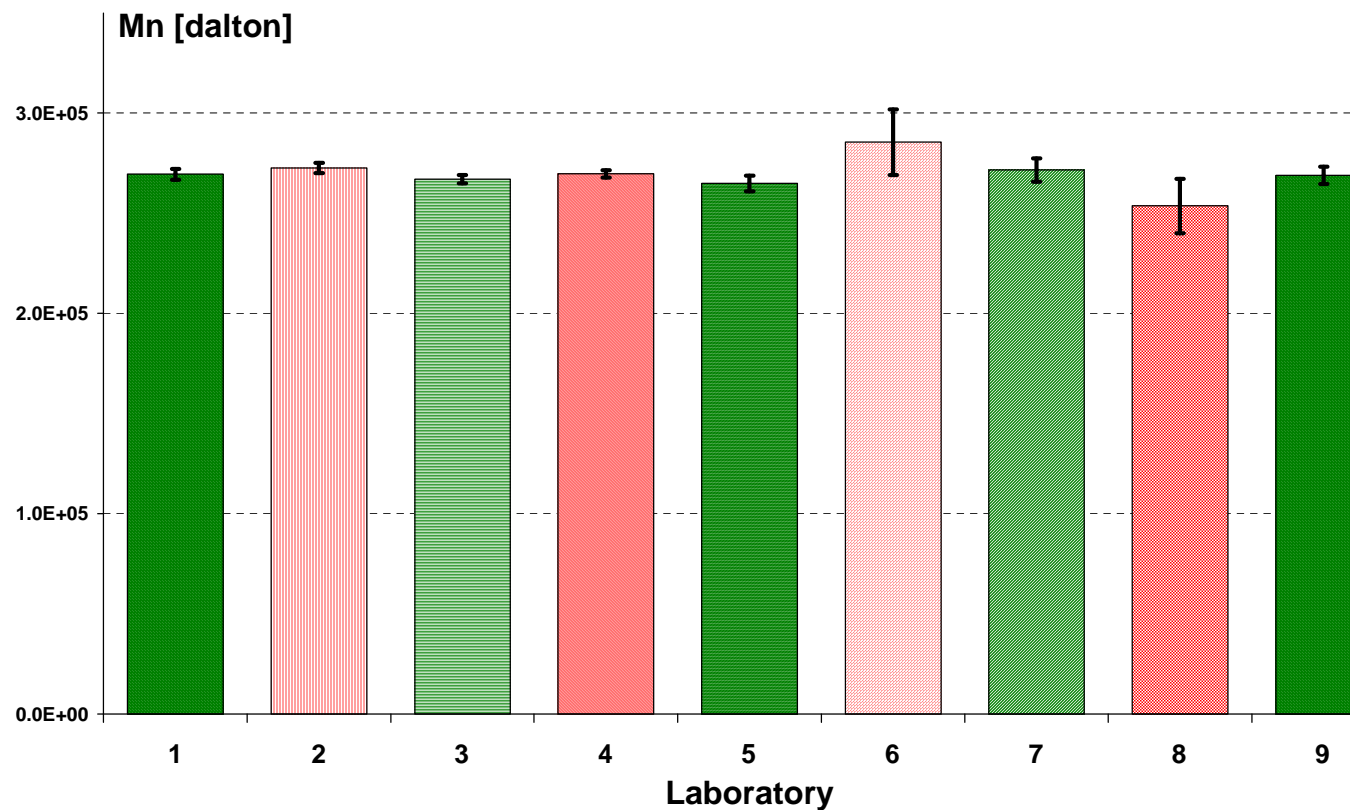
Contents

- 2010 ICT (Deacon *et al*) Conclusions from STANAG 4178 GPC round robin
- ISO/IEC 17025/2005: Application to GPC
 - Sources of uncertainty
 - Precision of the method
 - Accuracy of the method
 - Uncertainty budget
- Conclusions



GPC-RR: Polystyrene Std (NBS 706)

- Good reproducibility: RSD 2.8% (9 Laboratories)

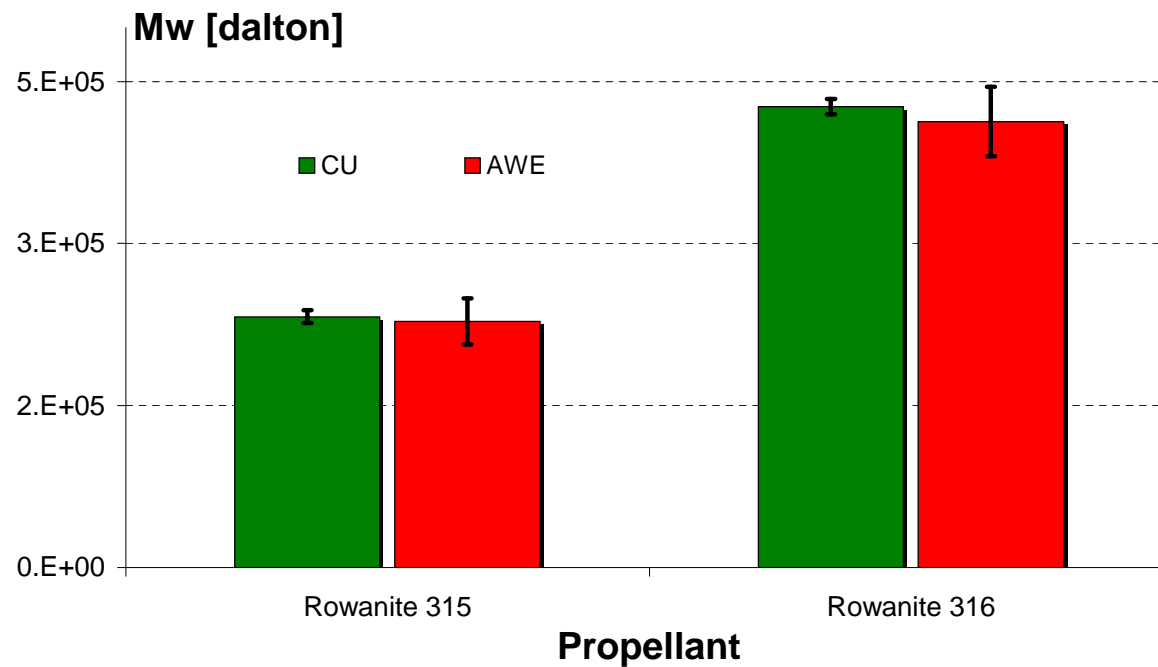


GPC-RR: Nitrocellulose

- Good repeatability but **Poor reproducibility**
- What are affecting the results?
 - Sample prep
 - Drying method
 - Concentration
 - Solvent (stabilised/unstabilised)
 - Shaking/stirring/Temp
 - Integration of the peak
 - Sensitivity of detector

UK Propellant analysis

- Good reproducibility between 2 labs



ISO/IEC 17025:2005 Requirements/Outputs:

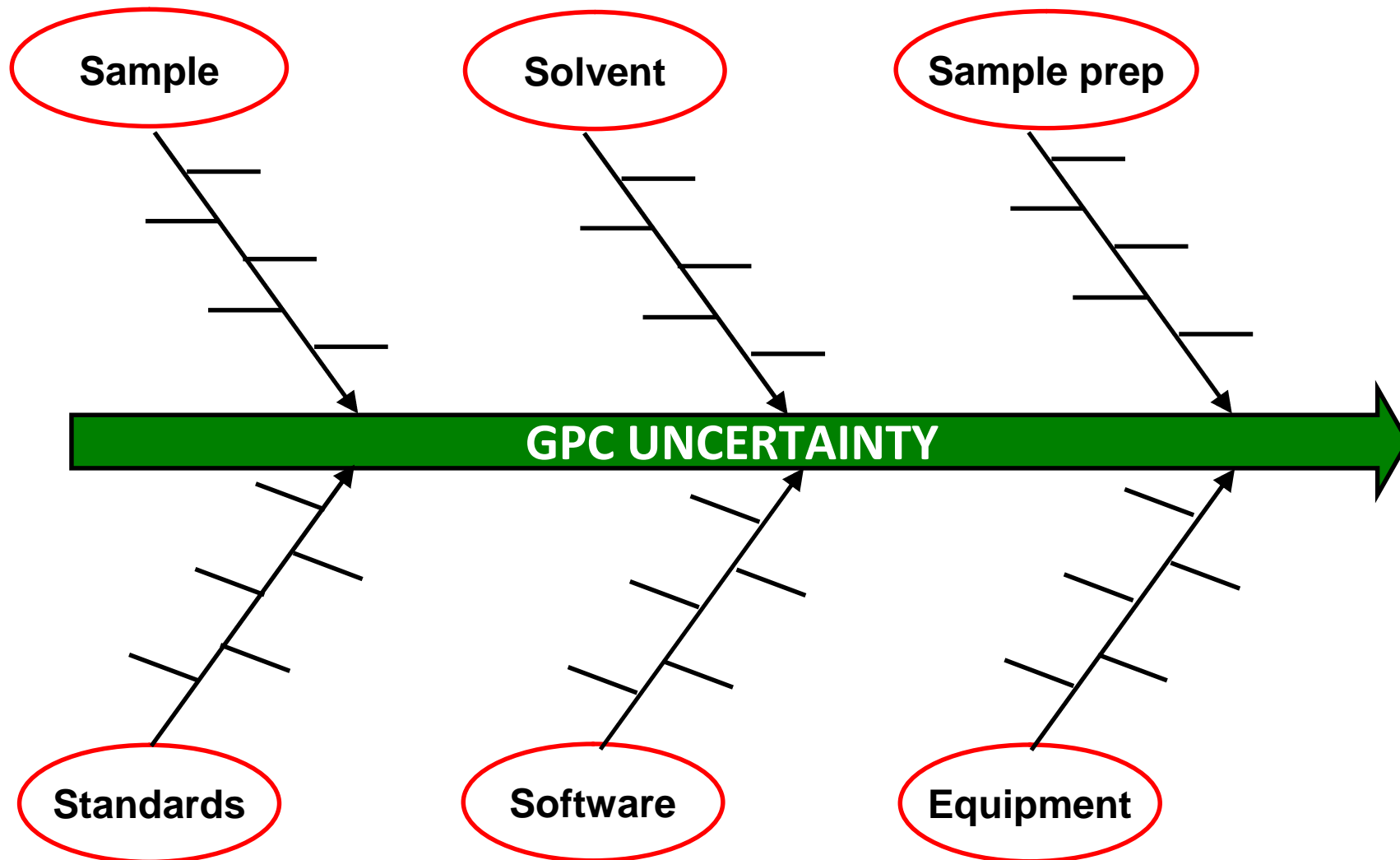
1. Demonstrate an effective quality management system
2. Technical competence
3. Ability to generate technically valid results
4. Compatibility with ISO 9001
5. Demonstration of proficiency
6. Attests to the competence of the lab

Valuable tool to truly understand and evaluate the precision, accuracy and weakness of the method

The validity of absolute measurement is insignificant if the uncertainty of the equipment is not know

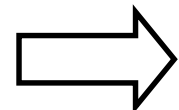
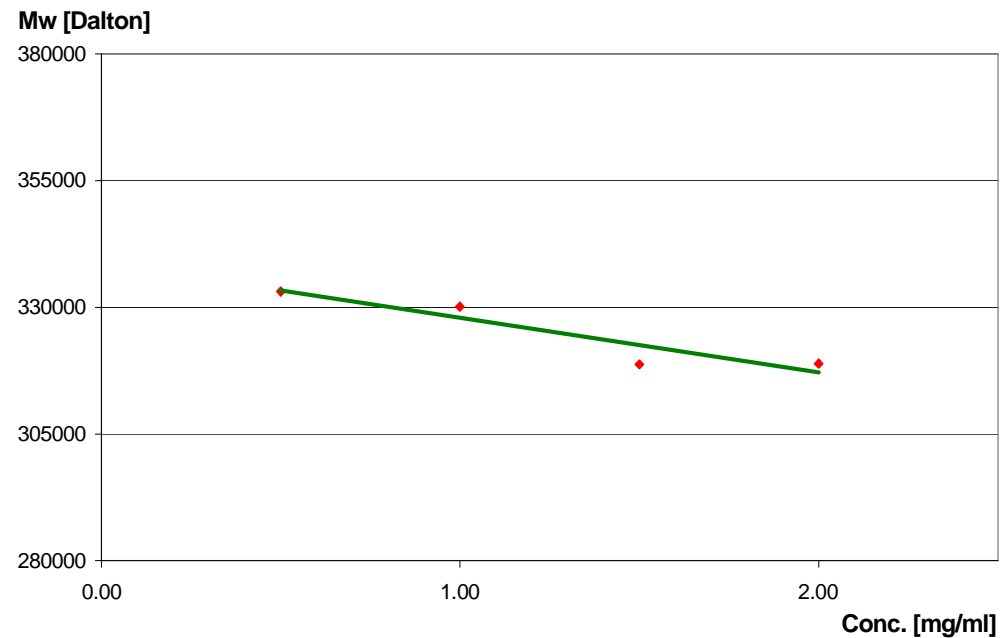
Ref: Excel Partnership (UK)

Sources of uncertainty for GPC



Sample preparation contribution

- Concentration
- Temperature
- Time in solution
- Drying (for pure NC)



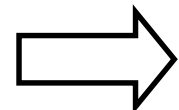
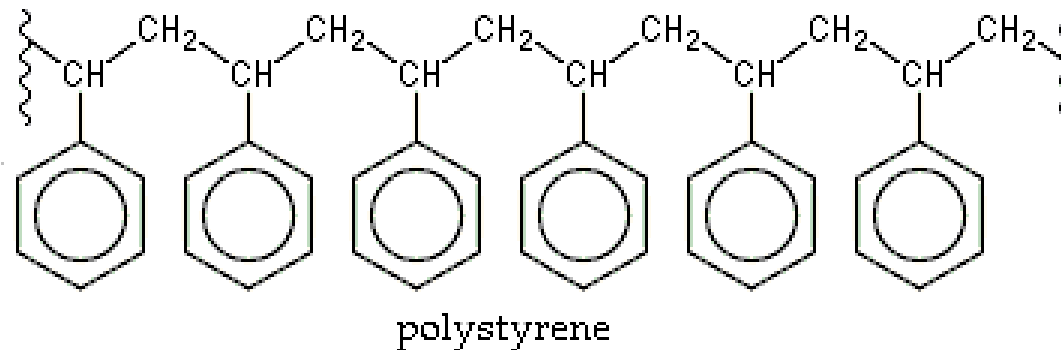
Calibration standards (Easical from Agilent)



- Concentration of narrow std
 - No effect (calibration curve not affected by conc.)
- Uncertainty budget of certified value
 - Unavailable
 - *3% according to Agilent (oral communication)*

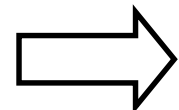
External standards (Broad PS)

- Concentration
- Uncertainty budget of certified value




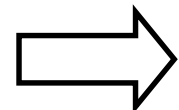
Data contribution

- Calibration (1st or nth order)
- Integration
 - Start end of integration
 - Repeatability
- Certificate of software



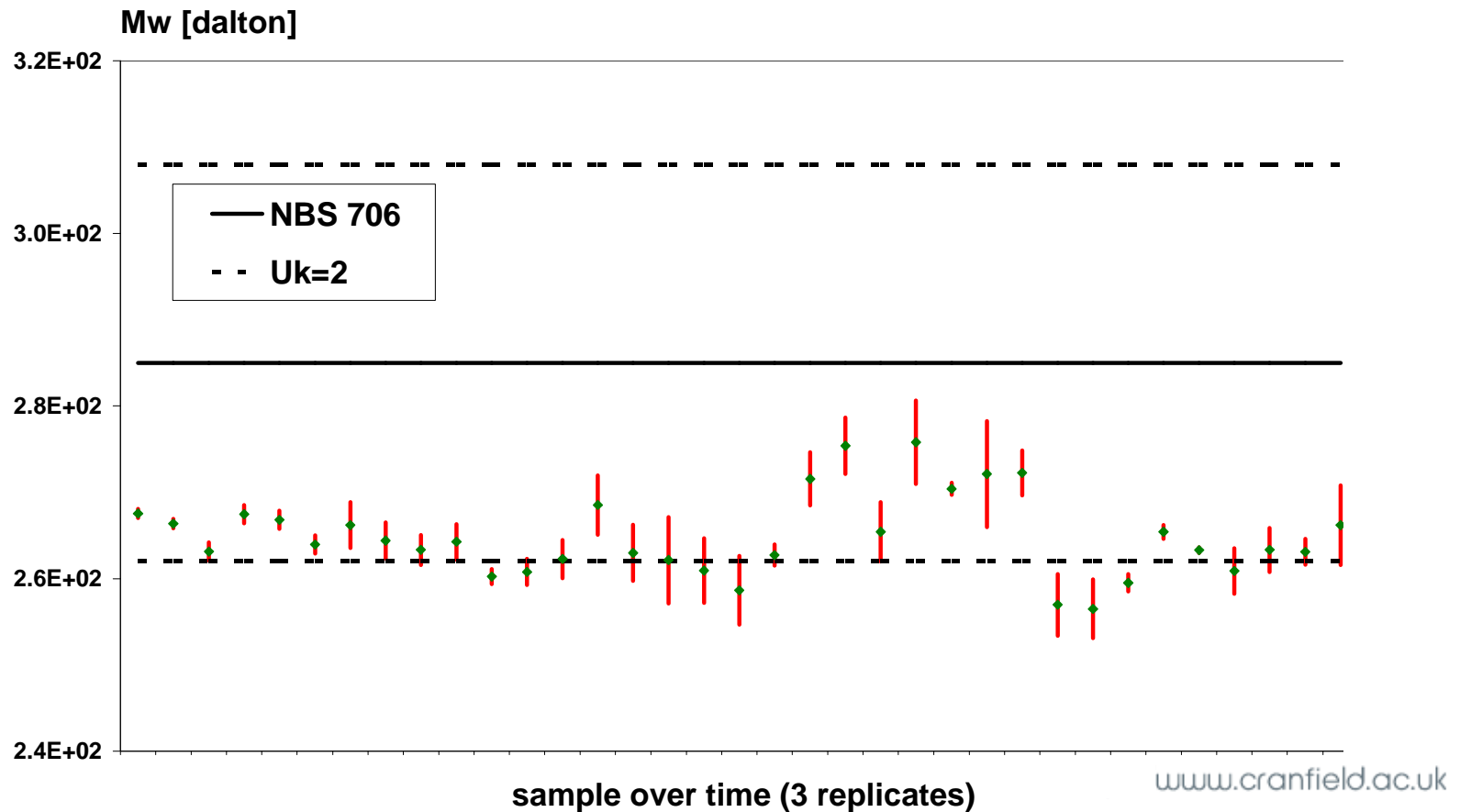
Equipment contribution

- Flow rate
 - 0.2ml/min variation  1.3% Da
- Injection volume
- Baseline stability
- Column ageing (eliminated in RR)
- Lab temperature (air conditioned)
- Sensitivity of detector

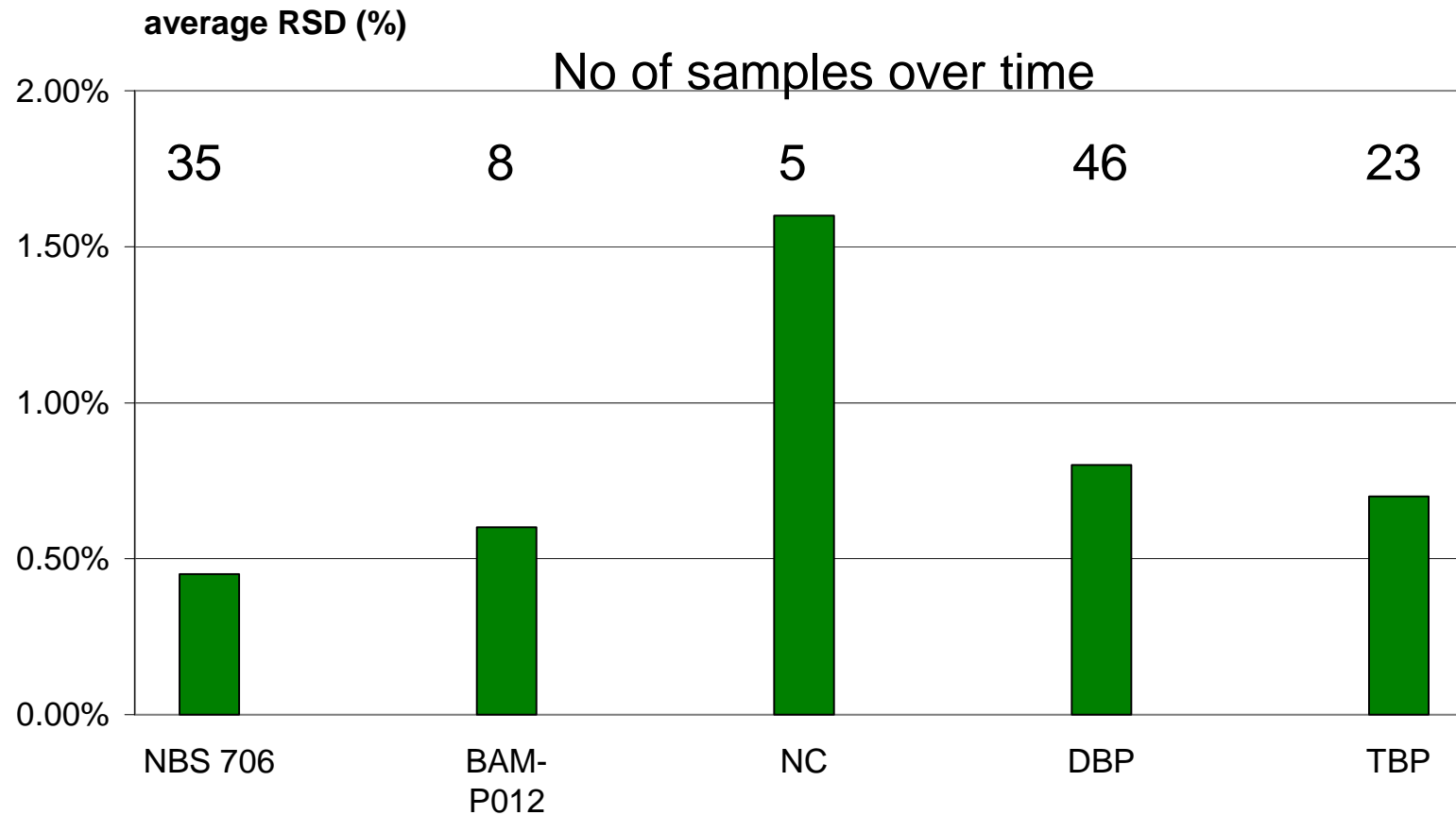


NBS706 accuracy & precision

- Good repeatability (RSD 0.45%)
- Reasonable precision (Mean 264900) but no traceability of material

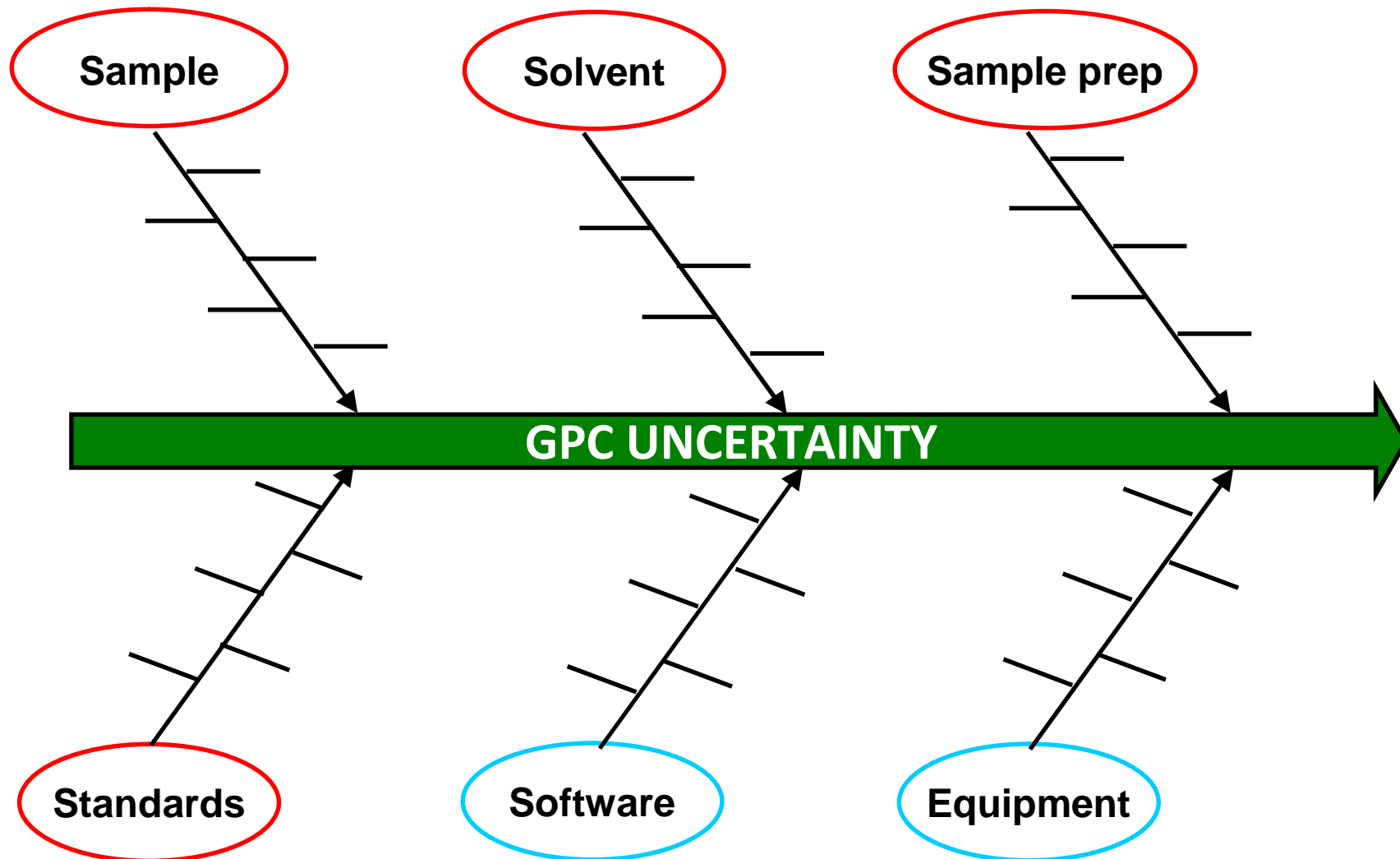


Precision of the method



Sample sets of 3 or 5 replicates (20 for NC)

Uncertainty budget



Conclusion

- Precision is good
- Accuracy?
- Reproducibility
 - PS <3% (9 labs)
 - Propellant <2% (only 2 labs)
 - NC <?? (9 labs)

Acknowledgement

- DOSG (Carole Baker)
- AWE (Paul Deacon and Ruth Knight)



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QUESTIONS?

DEFENCE ACADEMY

- DEFENCE COLLEGE OF MANAGEMENT AND TECHNOLOGY
- JOINT SERVICES COMMAND AND STAFF COLLEGE
- DEVELOPMENT, CONCEPTS AND DOCTRINE CENTRE
- RESEARCH ACQUISITION ORGANISATION