Do the results of respirable dust samples obtained from direct-on-filter X-ray diffraction, direct-on-filter Infrared and indirect Infrared (KBr pellet) methods correlate?

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Respirable dust and Silica

- Dust particles < 10 µm enters the gas exchange region of the lungs (alveoli)
- Silicosis: caused by respirable crystalline silica
- Silicosis can not be stopped, but can be prevented
- International drive to eliminate Silicosis
- Occupational Exposure Limit (OEL) in South Africa is 0.100 mg/m^3

Sampling for Silica

- Filter in a cassette and sampling pump
- Sampling over entire shift of worker (TWA exposure)
- Gravimetric weighing to determine the amount of dust
- Analysis to determine the amount silica (quartz)
- Determine exposure level in mg/m^3

Analysis methods for Silica

- Direct-on-filter (DoF) X-ray Diffraction (XRD);
- DoF Fourier-Transform Infrared (FTIR); and
- Indirect FTIR through the preparation of a potassium bromide (KBr) pellet.
- Based on internationally recognised methods:
  - DoF XRD and FTIR: MDHS 101 (HSE UK); and
  - KBr pellet method: NIOSH 7602 (USA).

Objective

To determine whether the silica results obtained from DoF XRD, DoF FTIR and KBr pellet methods correlate – within the South African context

Background

- International studies were done to compare these methods but were based on the use of PVC filters;
- In South Africa mixed cellulose ester (MCE) filters are mainly used; and
- This study took into account that blank filters are not necessarily available to the testing laboratory prior to dust sampling.

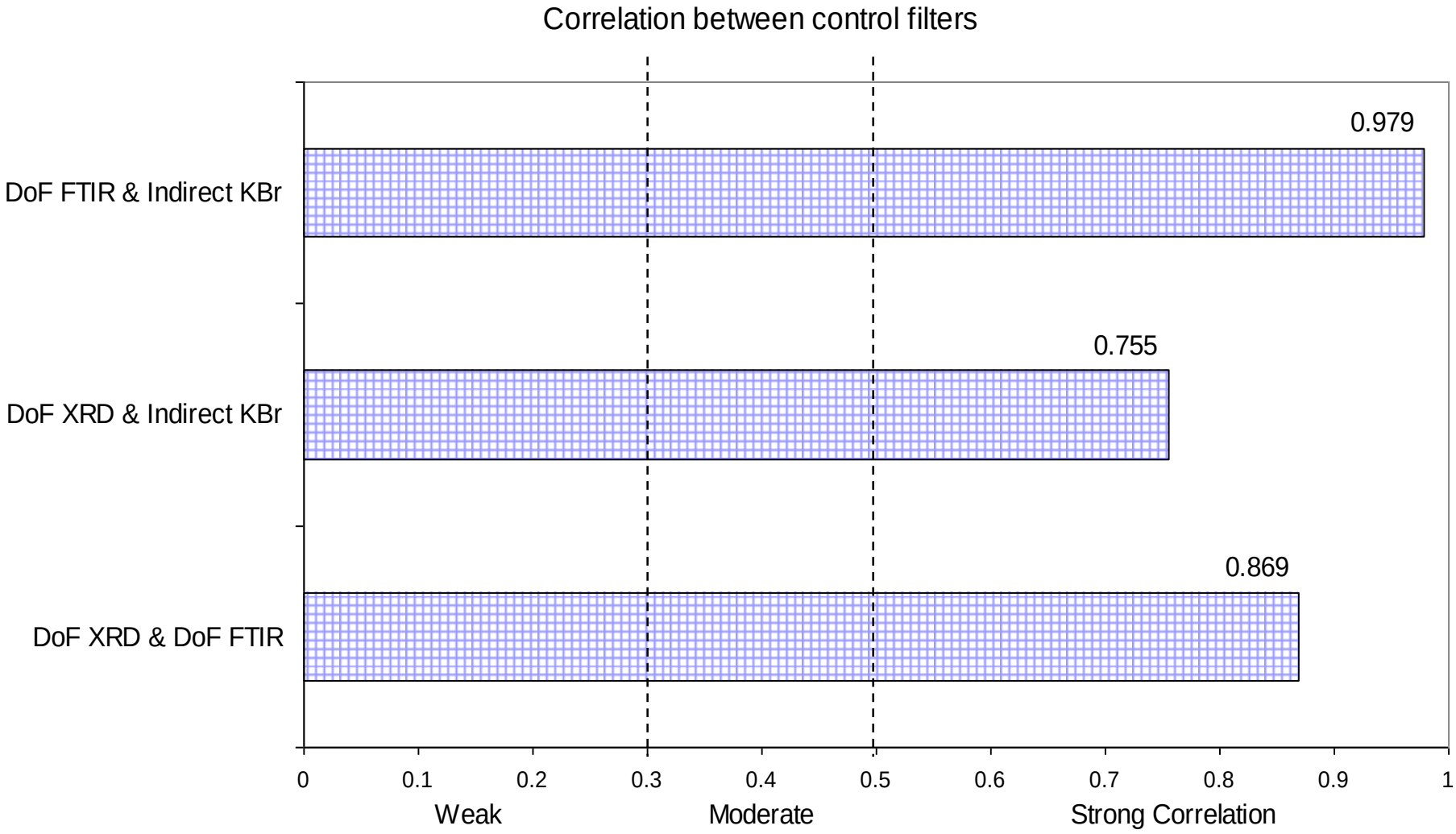
Methodology

- Filters from an international proficiency testing scheme were used as controls;
- 245 Samples from different commodities that were taken for another research project was used (gold, coal, diamond, quarries and ready-mix plants);
- Samples were analysed using all three methods;
- KBr pellets were individually prepared if the quantities of dust were sufficient (i.e. “Individual Group”); and
- Five filters were grouped and KBr pellets prepared – convention (i.e. “KBr Group 1 – 49”).

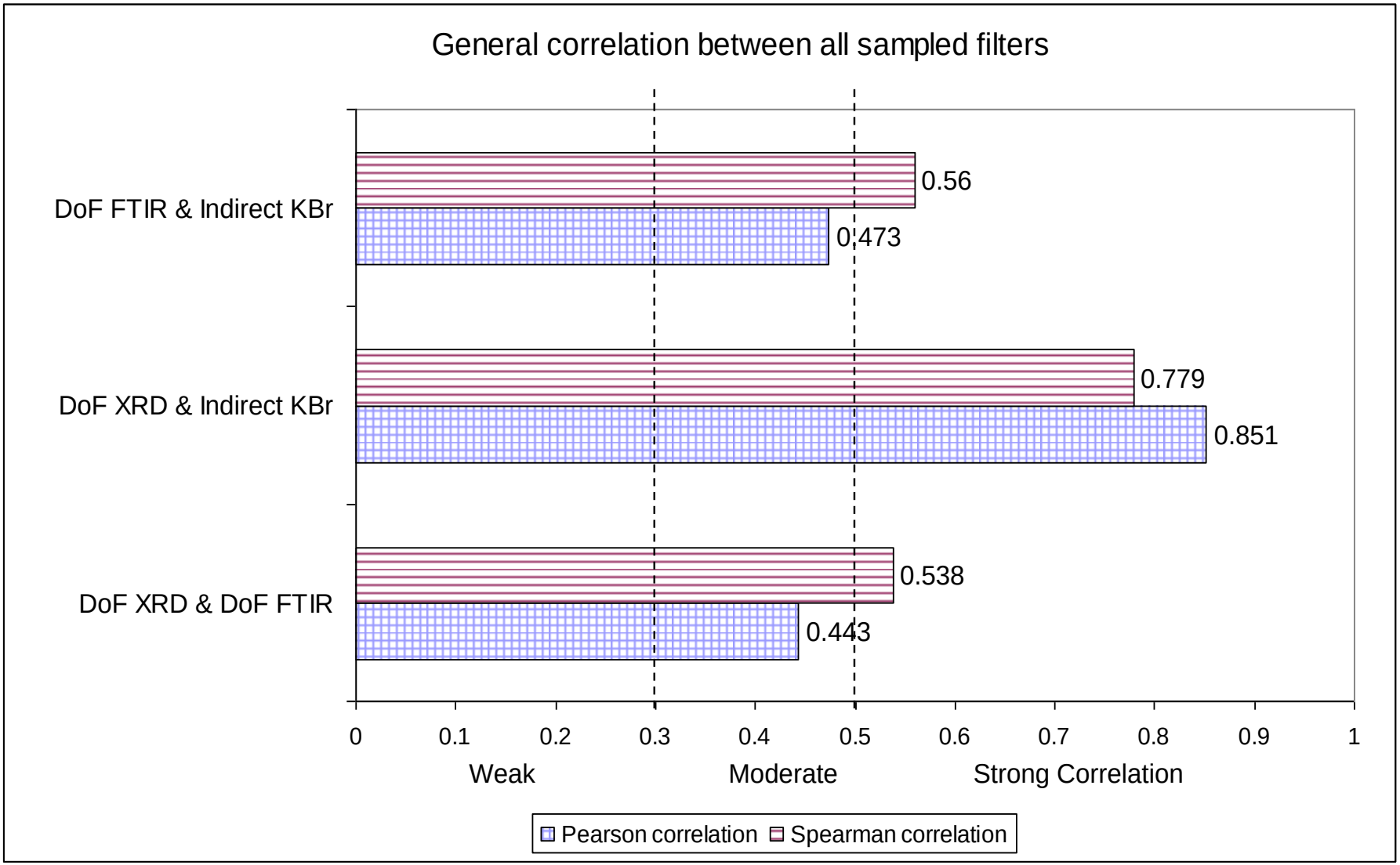
Methodology

- Calibration standards were prepared according to the methods;
- Certified reference materials were used;
- Measuring conditions were optimised according to the instrument parameters;
- Blank reference filters were analysed as the background for DoF FTIR;
- DoF FTIR scanning range was adopted to 820 – 500 cm^{-1} to compensate for the MCE filter background; and
- Limit of detection was 0.010 mg quartz

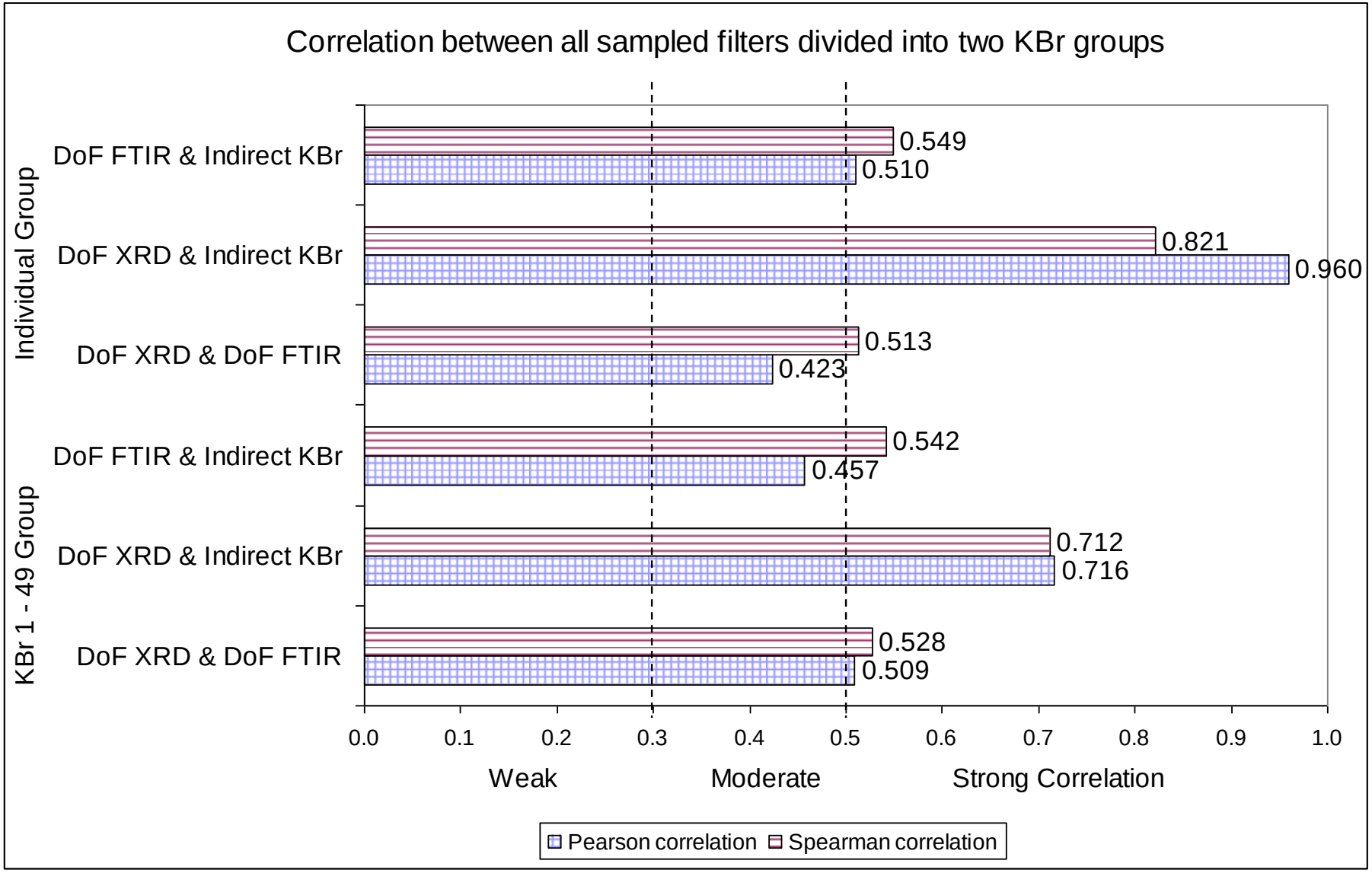
Results for control filters



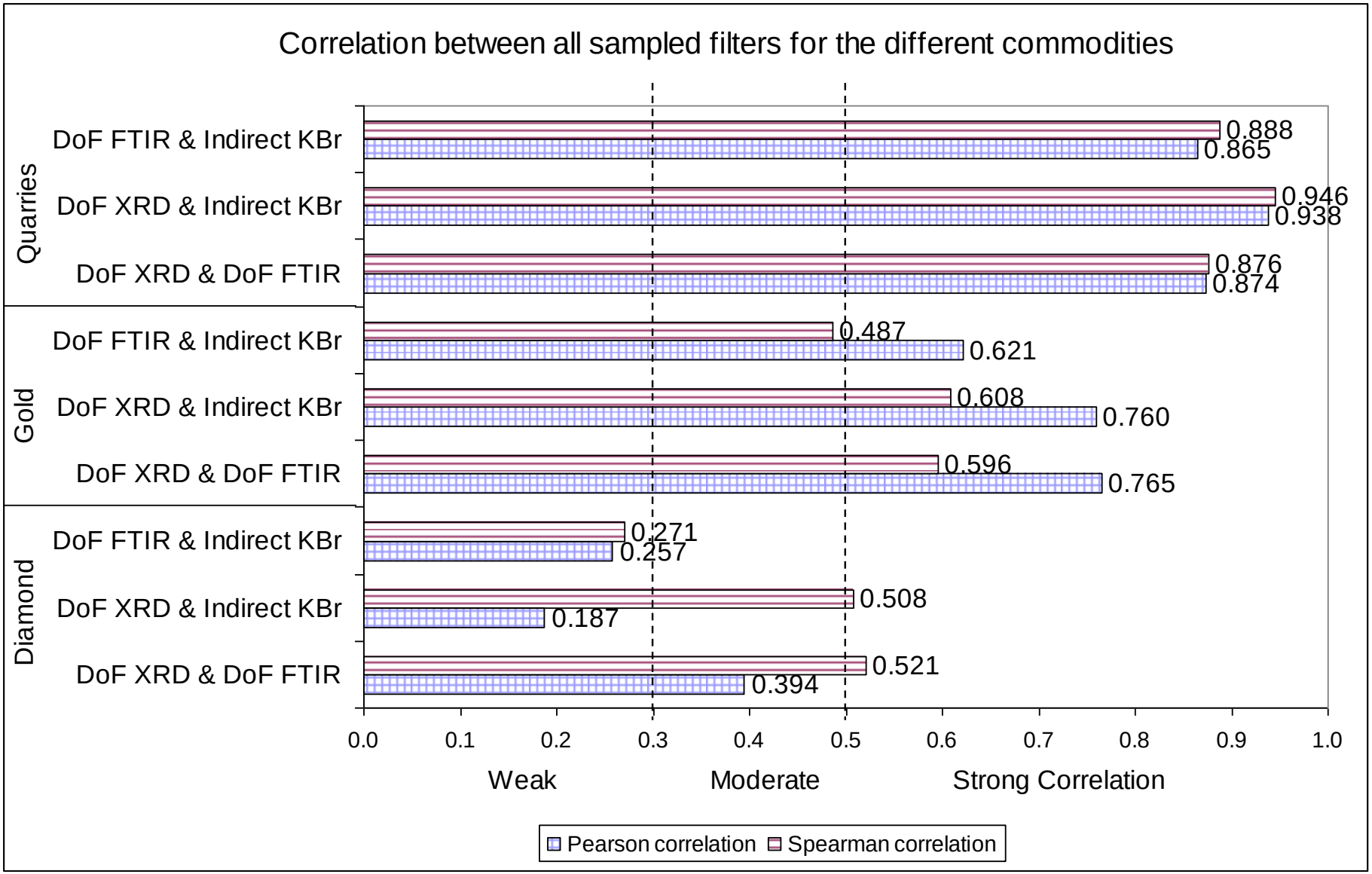
Results for entire data set



Results for each KBr group

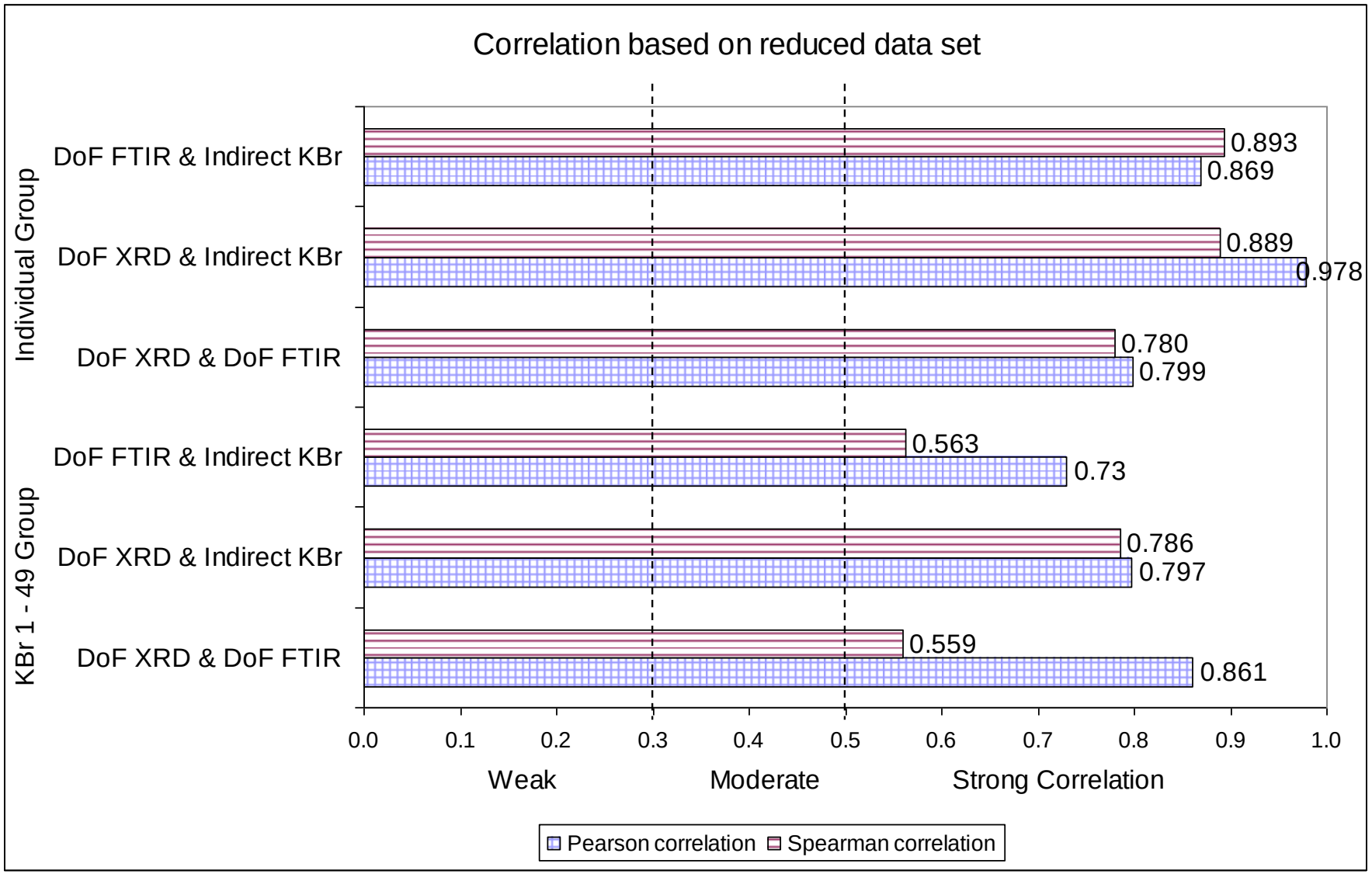


Results by commodity



Results on reduced data set

All results > 0.010 mg



Results

- Scope of study was to determine correlations only
- Control filters showed an average of 8% difference
- Field samples showed significant differences
- Further work is currently underway to determine actual differences between individual results and within each commodity

Reasons for differences

Each method is affected by different factors:

- DoF XRD: dust distribution & particle size distribution;
- DoF FTIR: background of filter & particle size distribution;
- KBr pellet: physical sample loss, chemical loss of silica, grouping of filters

Conclusions

- Strong positive correlation between the three methods for the controls filters;
- Moderate to strong positive correlation between the three methods for all the filters;
- Also strong positive correlation within commodities for gold and quarries; not for diamonds – correlation sensitive to mineral composition
- Strong positive correlation on the reduced data set

Recommendations

- Expand study to include other commodities as well;
- Determine the actual differences for the three methods; and
- Determine which factors are the cause of these differences within the South African environment.

Current research

- Determine the effect of the sampler performance on XRD response;
- Sampler performance determines particle size distribution of dust collected; and
- Sampler determines how the dust is distributed on the filter (analysis area of XRD vs FTIR).

Acknowledgements

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Questions?

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