

Routine XRD analysis on anode material - our experience

Dr. Elke Thisted
Elkem Solar AS Research
Kristiansand, Norway



Mosjøen Anode ANS (MA)



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2/3 to Iceland (Alcoa)

1/3 used in Mosjøen

How are we involved?

All product analyses are done at Elkem Carbon AS in Kristiansand, Norway:

- A lot physical measurement
- Elemental analysis with XRF
- Measurement of L_c by XRD



ASTM D5187 – 912002

Standard Test Method for Determination of Crystallite Size (L_c) of Calcined Petroleum Coke by X-Ray Diffraction

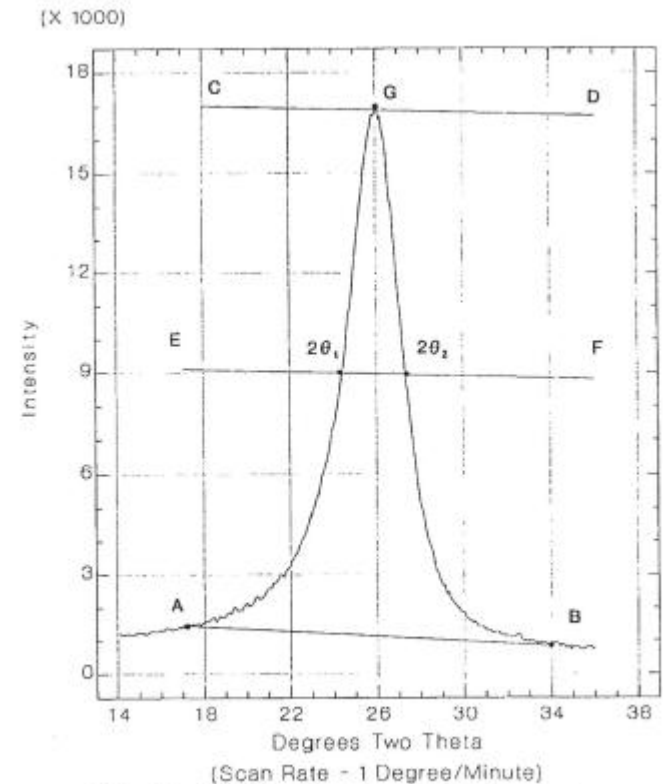


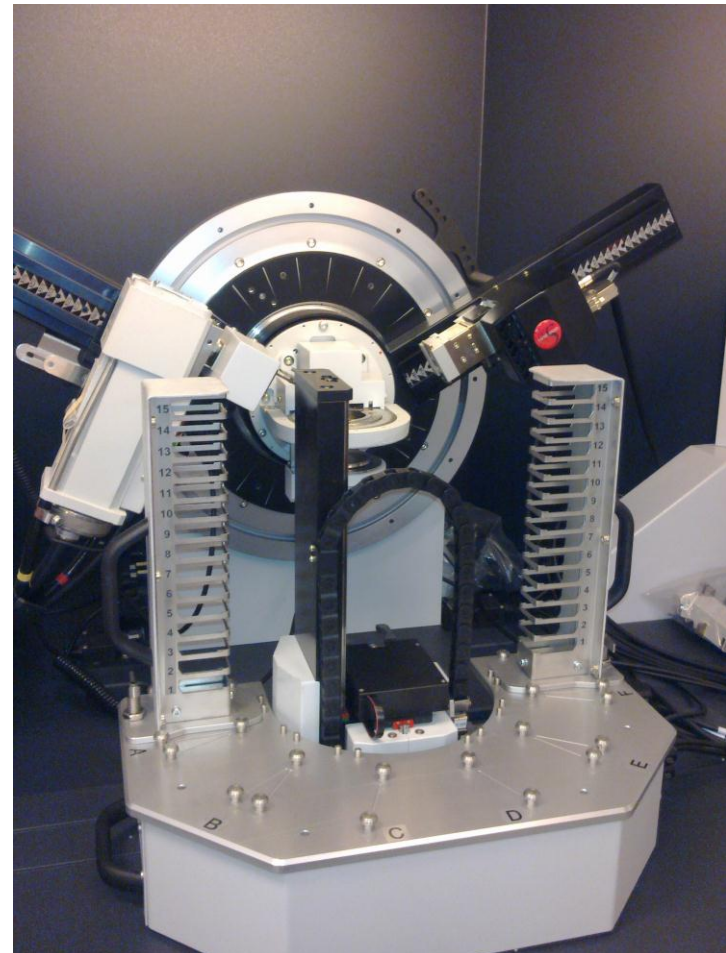
FIG. 1 Typical Diffraction Scan of Petroleum Coke

Picture taken from ASTM standard

OBS: Background linear in ASTM std!

New instrumentation was needed:

D8 Advance from Bruker, bought 2007



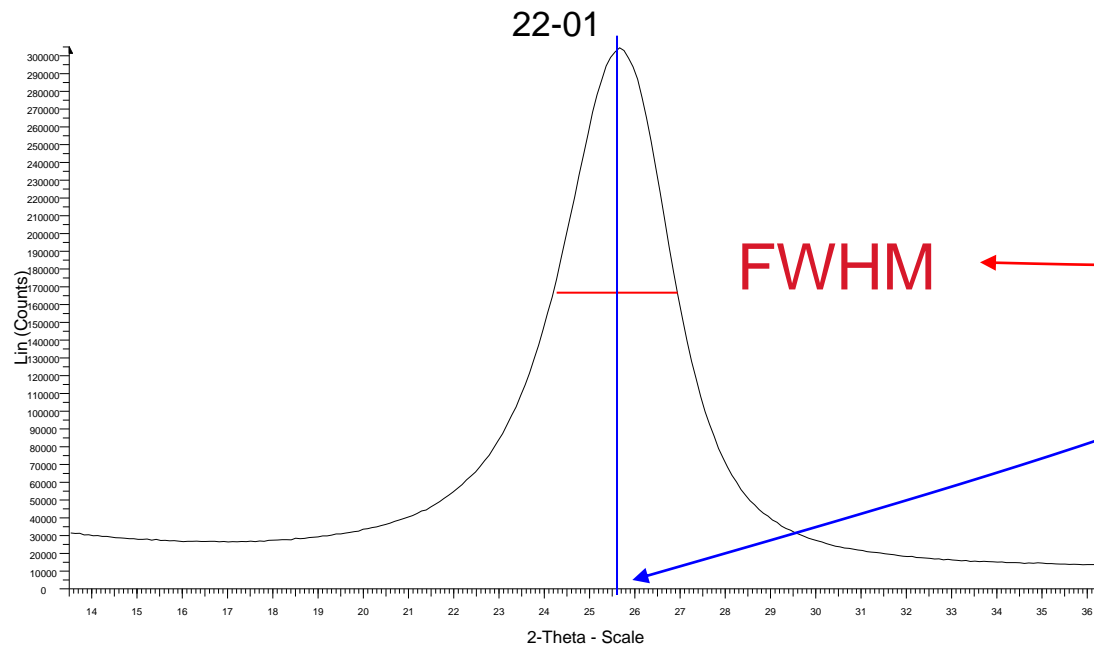
What do we measure to get the Lc value?

$$n \lambda = 2 d \sin\theta$$

Scherrer equation

$$L_c = (K\lambda)/(\beta \cos\theta)$$

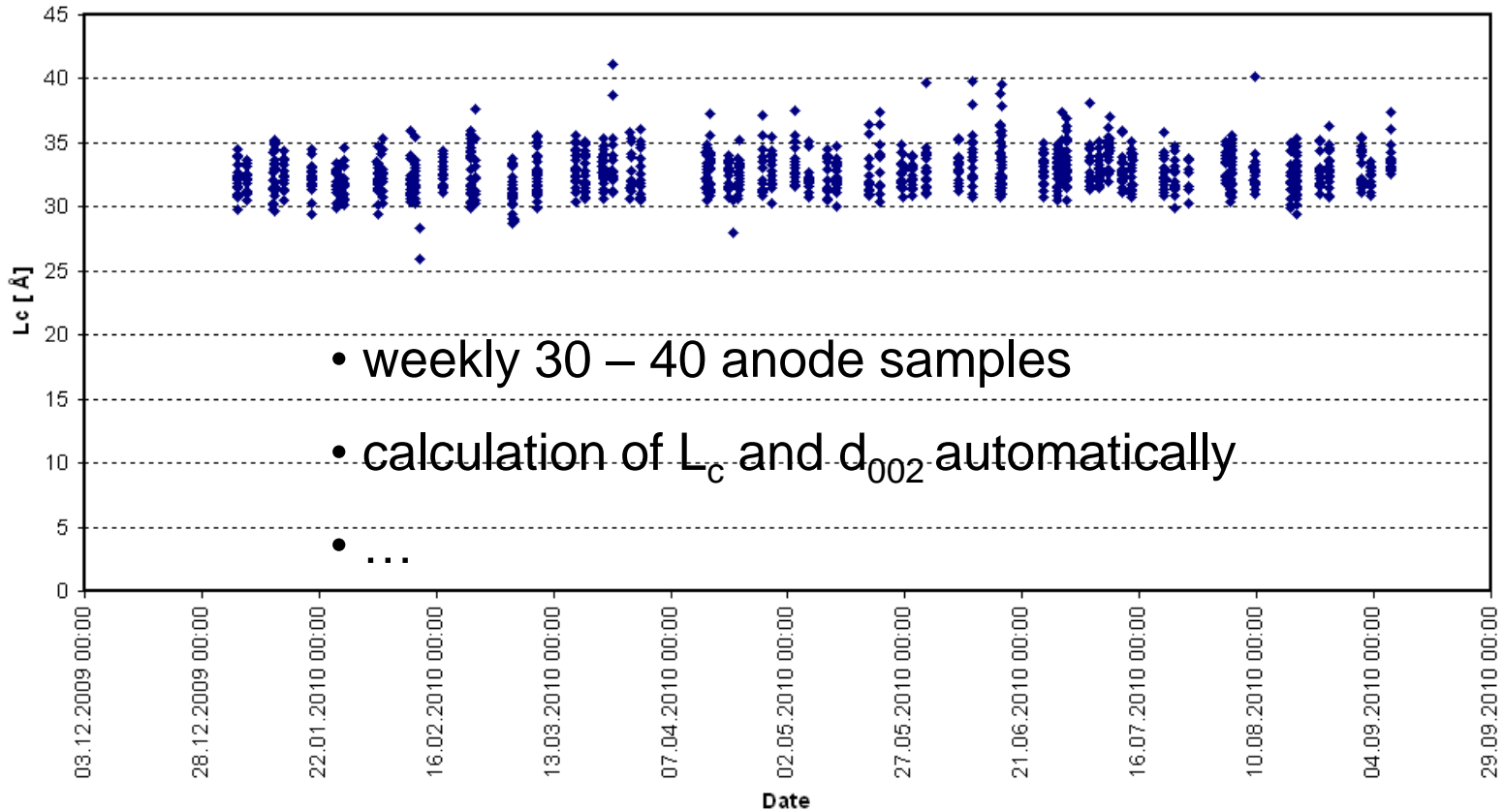
$$d_{002} = \lambda/(2 \sin\theta)$$



22-01 - File: 22-01.raw - Type: 2Th/Th locked - Start: 13.500 ° - End: 36.327 ° - Step: 0.100 ° - Step time: 184. s - Temp.: 25 °C (Room) - Time Started: 19 s - 2-Theta: 13.500 ° - Theta: 6.750 ° - Chi: 0.00 ° - Phi: 0.00 °
Operations: Import

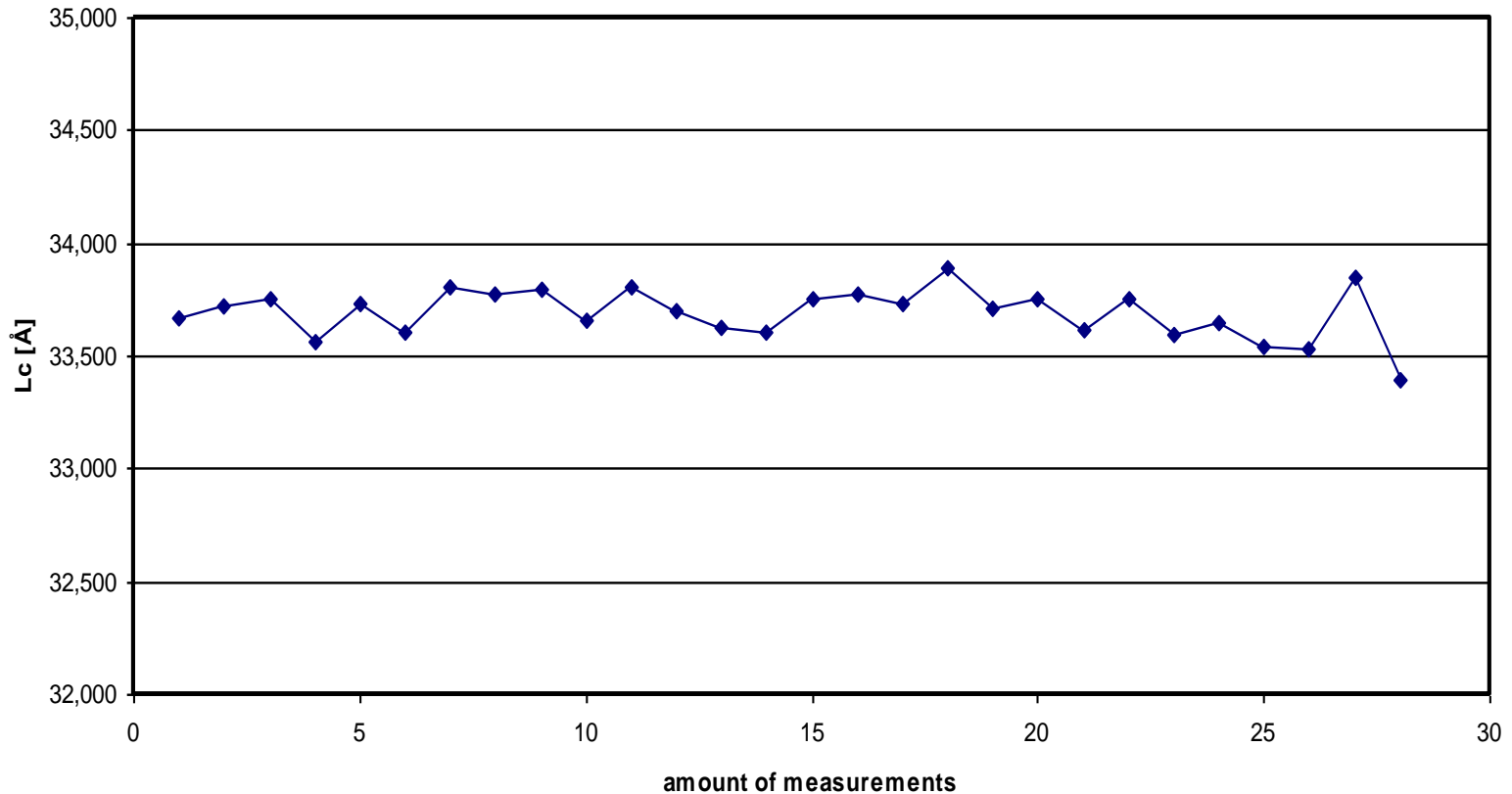
Analysis of Lc in 2010

Analysis of Lc during 2010

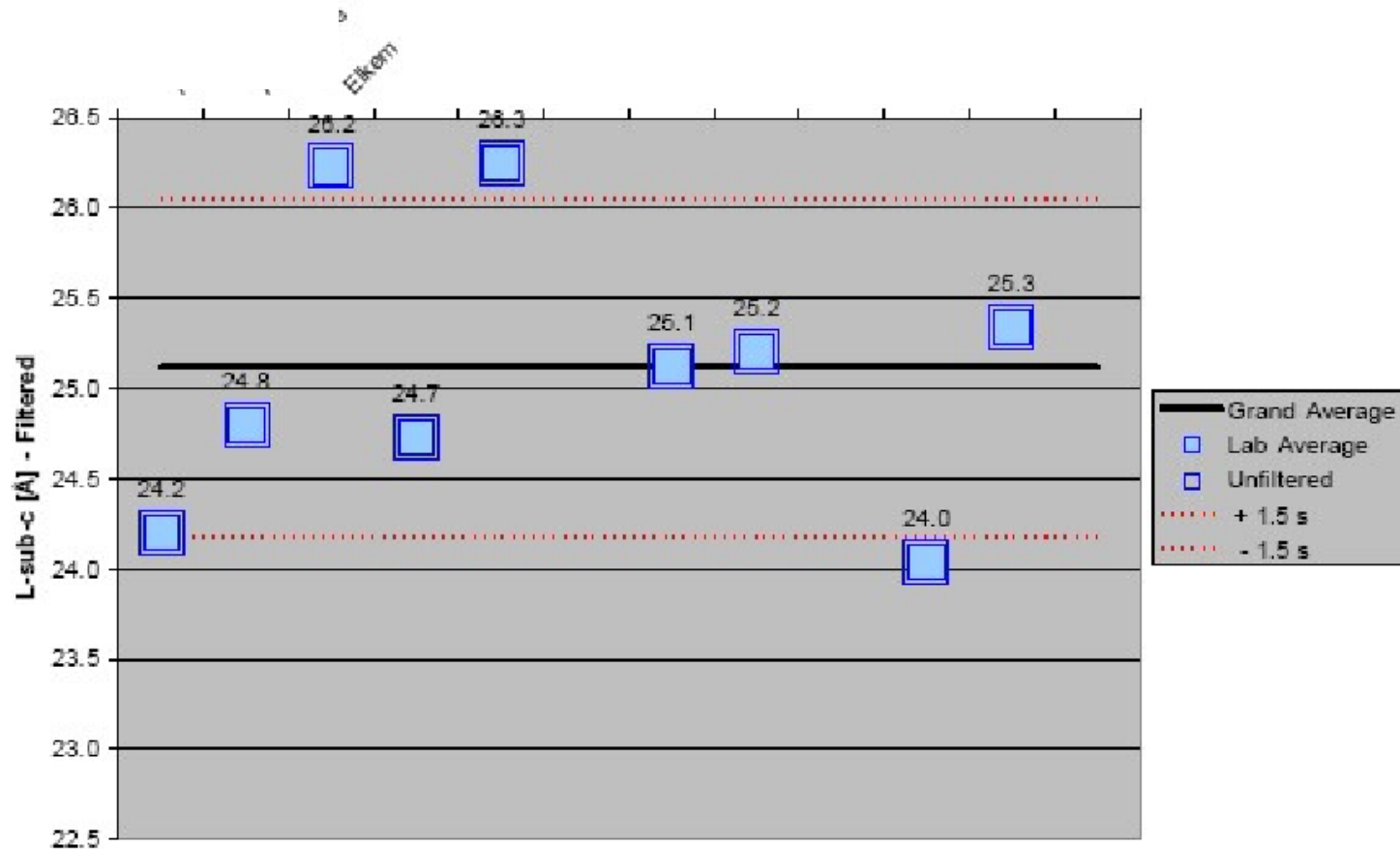


Running of the control sample

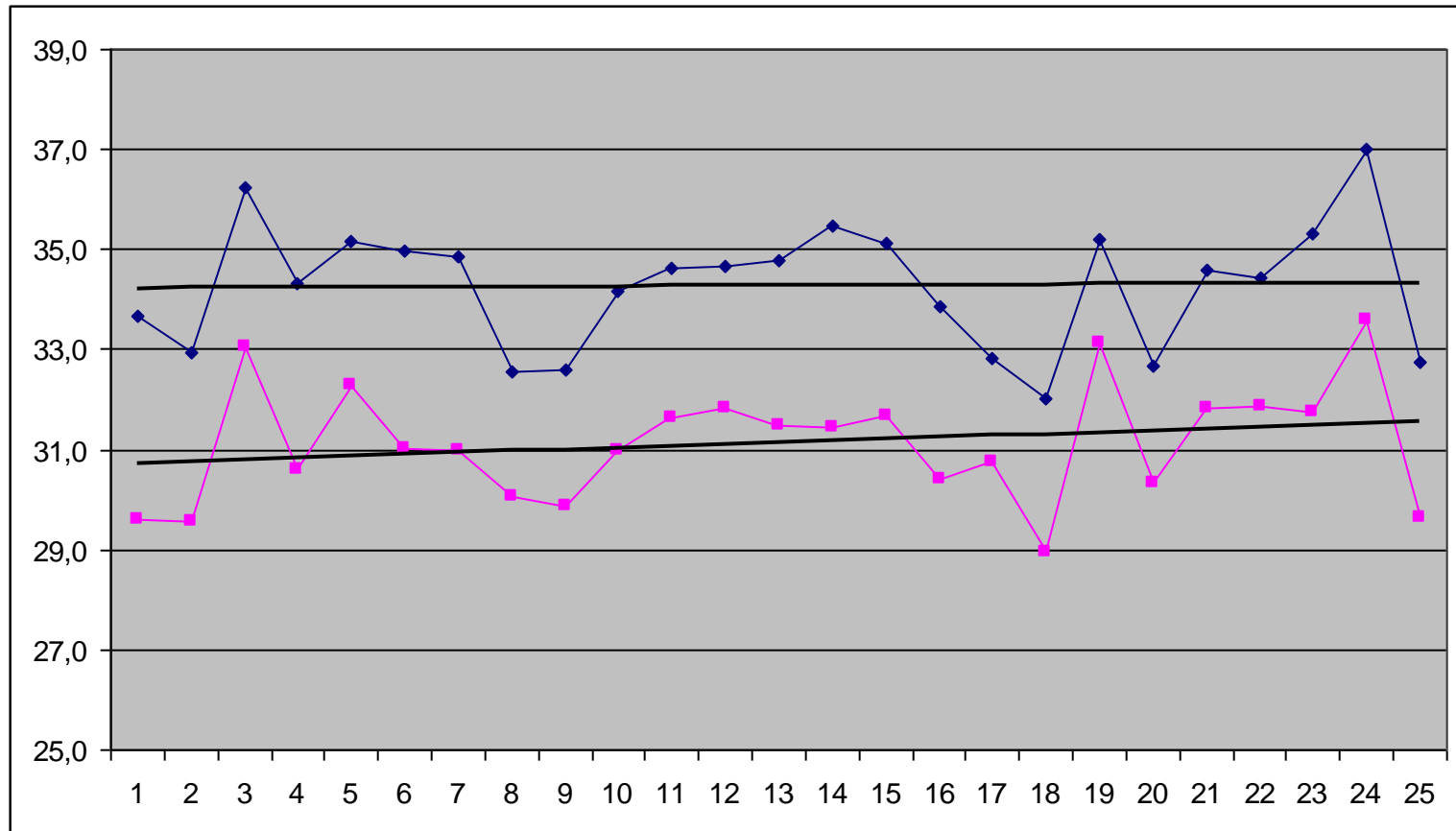
Lc of a control sample



Deviations in RR



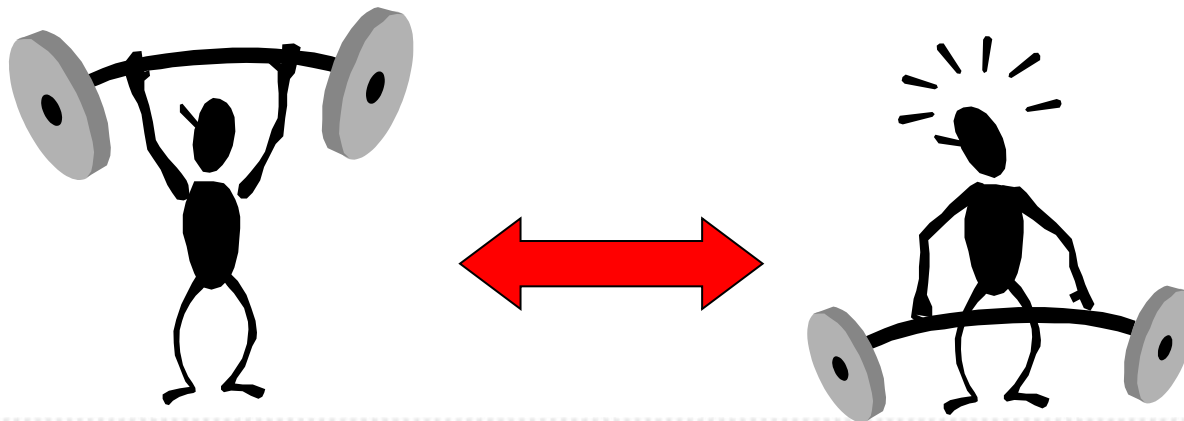
Clearly a systematic error



Elkem, other lab

Strength and weakness – our D8

- Stable (apart from start-up after unwarned power failure at the factory) "bra oppetid"
- In the beginning trouble with the sample changer arm – but after exchanging the part -> no problem
- Easy to use – nice for the operator (compared to what we had before) – automatic calculation of the L_c and d_{002}



Further work - Strength and weakness at ESR

- "make more time for XRD" – need a better/deeper understanding of our instrument
- find out why there is a systematic error when analysing Lc and fix it

Thanks for listening!