Cormetech, Inc.

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- SCR Catalyst Chemistry
- X-Ray Fluorescence Application.
  - Analyses
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- Summary

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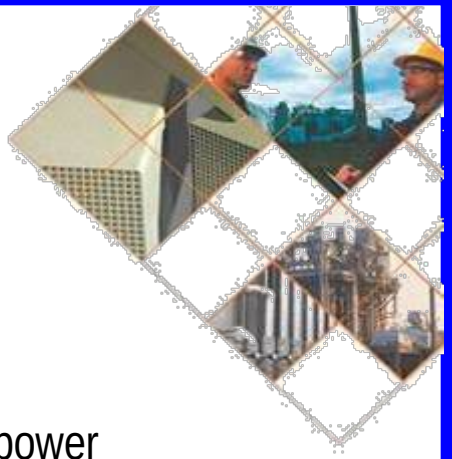
About Cormetech



- A joint equity venture of Corning and Mitsubishi Heavy Industries (MHI)
- Founded in March 1989 with headquarters in Durham, NC
- Manufactures Selective Catalytic Reduction (SCR) Catalyst for the electric power generation, petroleum refining, and chemical industries
- Manufacturing facilities in Durham, NC (1992) & Cleveland, TN (1999)

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Manufacturing Facilities

- Located in Cleveland, TN and Durham, NC
- A total of 200,000 sq. ft. manufacturing area and ~ 200,000 sq. ft. warehouse space
- Technologically advanced manufacturing processes

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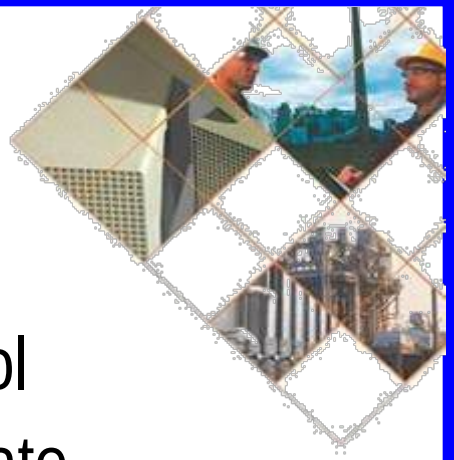
Laboratory Facilities



- State-of-the-art laboratory
- Product / process quality assurance and control
- Catalyst activity test center (lab reactors simulate power plant catalytic reactors)
- BET Surface Area
- Hg Pore Evaluation
- X-Ray fluorescence.

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The chemistry

$$4NO + 4NH_3 + O_2 \xrightarrow{\text{Catalyst}} 4N_2 + 6H_2O$$

Chemical Analyses

- Chemical composition analysis was originally done by an independent laboratories.
- Due to the need for real-time results and reduced analyses cost, Cormetech Inc, purchased an XRF-Fluorescence unit (ZSX-100e).

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X-Ray Fluorescence Application.

- Quantitative Determination of TiO_2, V_2O_5 and WO_3.
  - Routine analyses for our raw material, mid-stream and final products.
  - Sample is radiated over a 30 mm diameter.
- Semi quantitative composition analyses:
  - Bulk analysis .
  - Analysis is performed on a pressed pellet of powdered catalyst and a comprehensive scan is performed over a 30 mm diameter, while the sample is spinning.

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X-Ray Fluorescence Application.

- Micro-mapping (Surface analyses):
  - Surface analyses are performed on a sample of a single catalyst cell wall approximately 30-mm in length.
  - The sample radiation is constricted to a diameter of 1-mm
  - Multiple analyses are conducted for each sample to ensure representative results.

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XRF Analyses method

- Development of applications:
  - Calibration required for both Coal and Gas products.
  - Standard samples were precisely made with the same matrix as our normal product by our development team.
    - Standard sample composition confirmed by ICP.
    - Set up the calibration line for each type of product.
    - Validate the calibration line with ICP (multiple lab) analyses of selected gas and coal samples.
- Quality Control of the XRF units.
  - A drift correction sample is run weekly to correct for any intensity drift that may have occurs,
  - Daily check samples to monitor the integrity of the calibrations line.

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XRF-Analyses Method:

Sample Form:

- Noodles: in-process blend of ingredients
- Honeycombs: Final product
- Both are previously calcined (high temperature conversion to ceramic)

Sample Preparation (Bulk analyses):

- Mill the sample to approx 70 microns using a grinder
- Pelletize the milled powder in a 28-ton press
- Remove residual moisture using 300 C and 500 C.
  - Some samples are very hydroscopic, if place directly at 500 C it will crack, therefore the pelletized samples are first place in a 300 C furnace.
- After moisture removal, samples are immediately placed in the XRF unit for compositional analyses.

Bulk Analyses

Figure 1: Shows a unknown particles mounted on Binding material.

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Micro-mapping

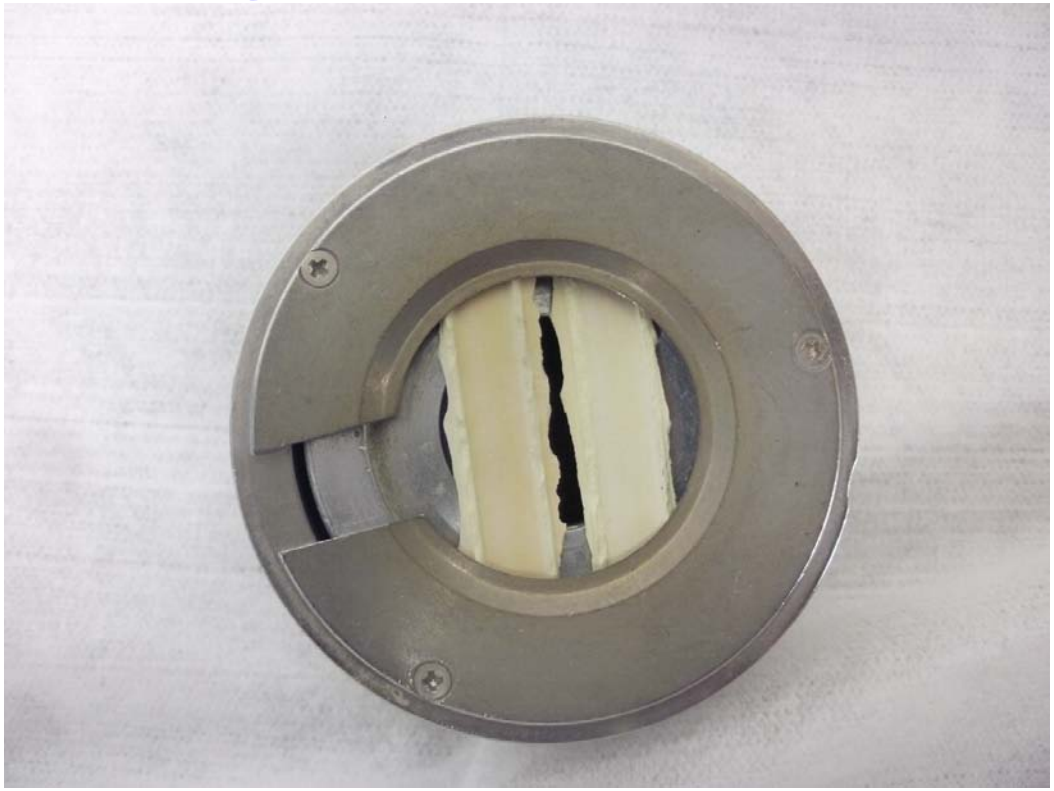

Figure 2: The first sample next to the opening represent the inlet of the catalyst while the second samples is a piece of outlet.

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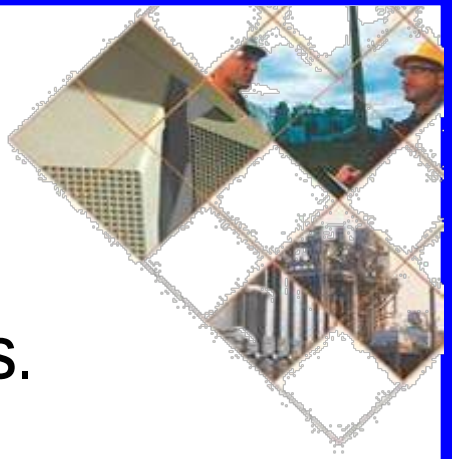
Application Challenges:



1. V_2O_5 calibration line for all our products.
   - Products are TiO_2 (> 80%) base
   - Use V-KB line, due to Ti-KB and V-KA are too close to each other.
   - A single V_2O_5 calibration line does not give accurate value for all the V_2O_5 ranges, even after matrix correction (absorption/conc).
   - At low levels, V_2O_5 value approx 25 % from expected ICP value .
   - Developed several V_2O_5 calibration line according to the product matrix (e.g., gas and coal products.)

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V2O5 Calibration Line

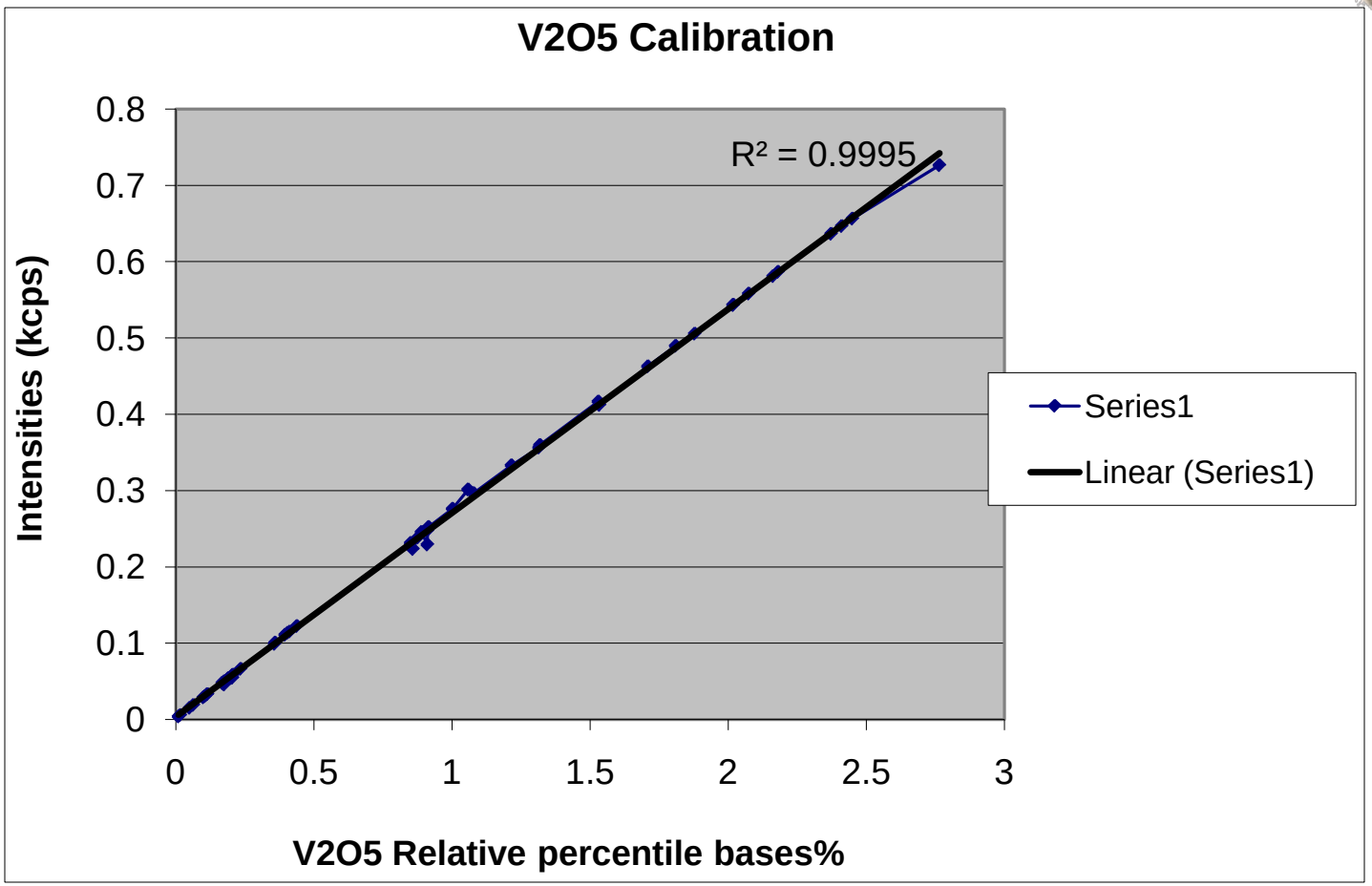

Fig: 3 Calibration line for V2O5.

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Application Challenges:

- TiO_2 Calibration line for gas products.

  Production reported an un-expected increased of TiO_2

  Lab started an investigation of root-cause.

  Reviewed the daily check

  Reviewed possible drift in calibration.

  No clear link was found to current calibration

  Action taken:

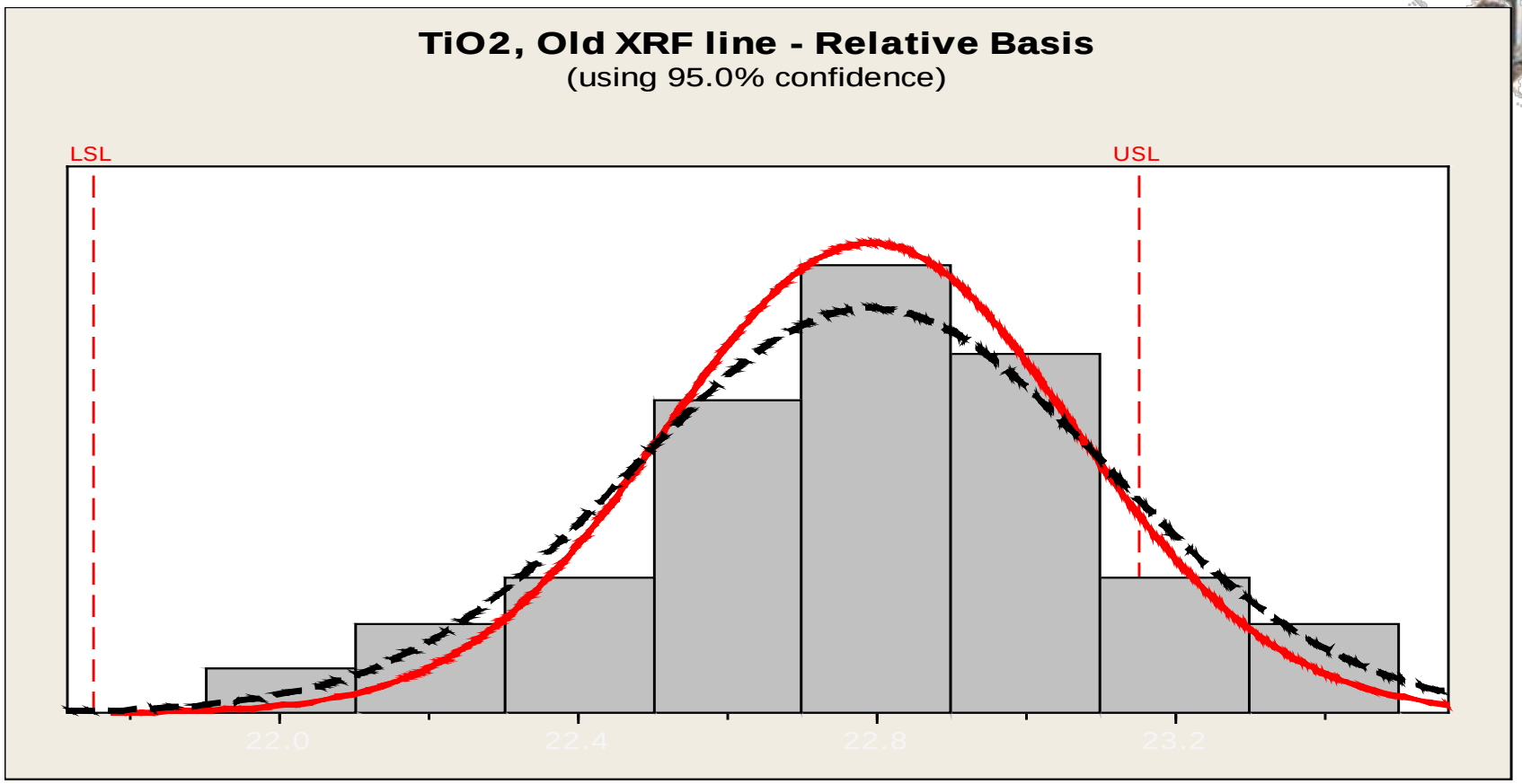
  Design a set of standard samples utilizing current production raw material

  Samples were precisely manufactured by development lab.

  Created a new calibration line, were TiO_2 was corrected for Vanadium and Tungsten absorbance.

  Challenges Developing TiO_2 calibration: Non-Reproducible data among ICP laboratories for TiO_2 sample and also comparable to expected values.

TiO_2



Relative TiO_2 Concentration

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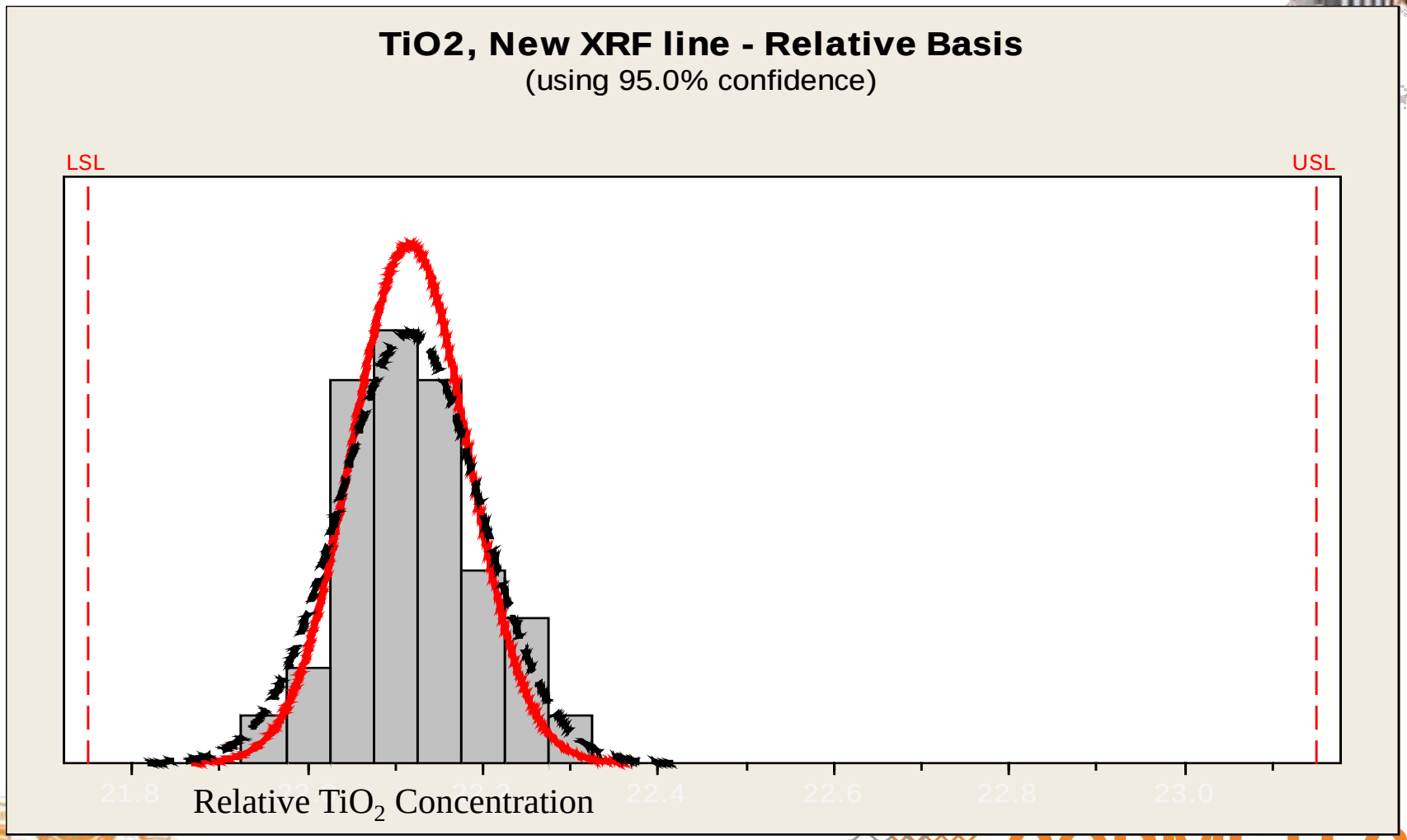
TiO_2, With mproved XRF line



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Application Challenges:

- Qualitative analyses of Cr, Pt and other trace metals.
  - Cr –KA line will interfere with V-KB, and Ti-KB interference with V-KA. Higher levels of V_2O_5 measured when Chromium is present (Fig. 4).

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Qualitative Scan

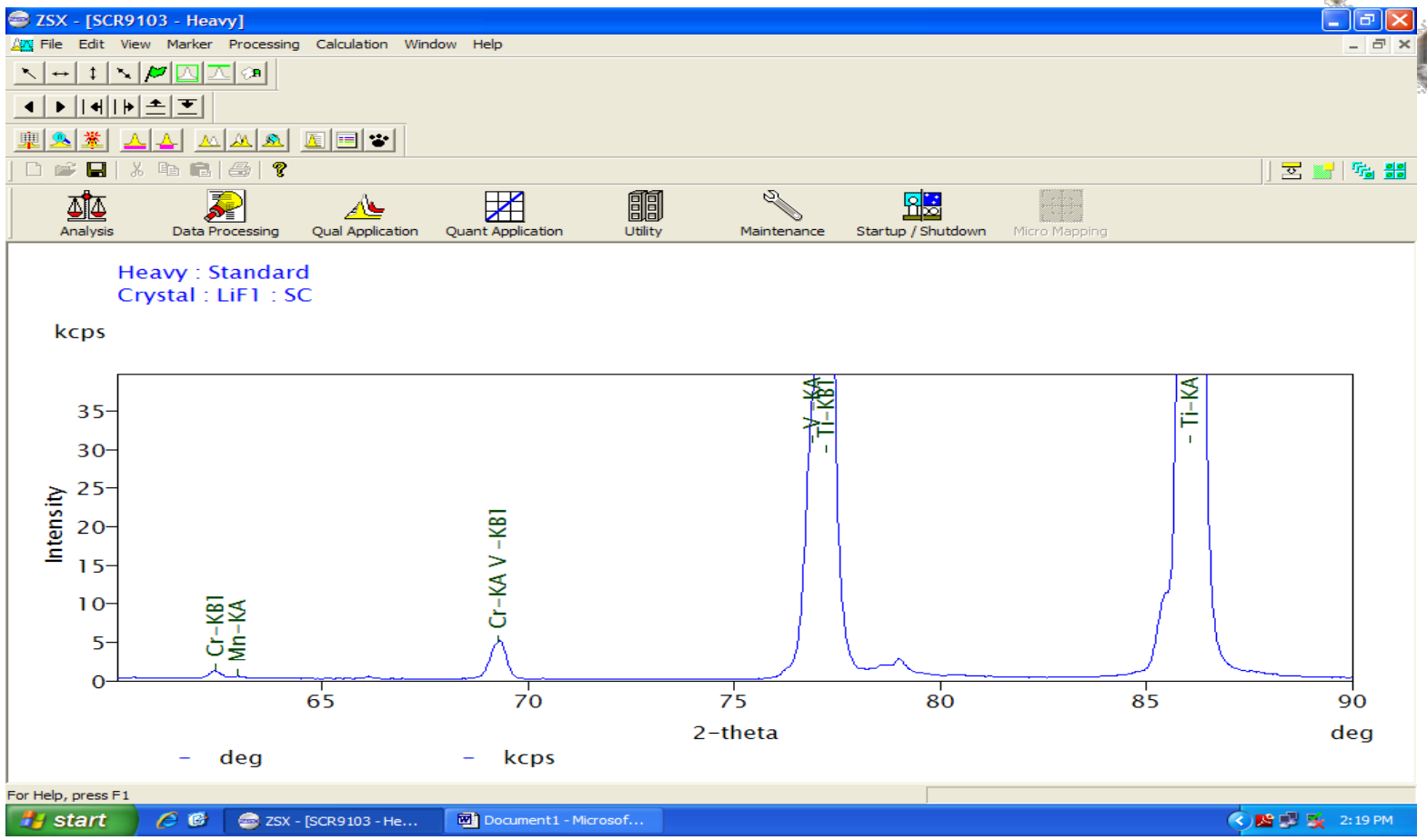

Fig 4: XRF-qualitative scan of sample containing Cr.

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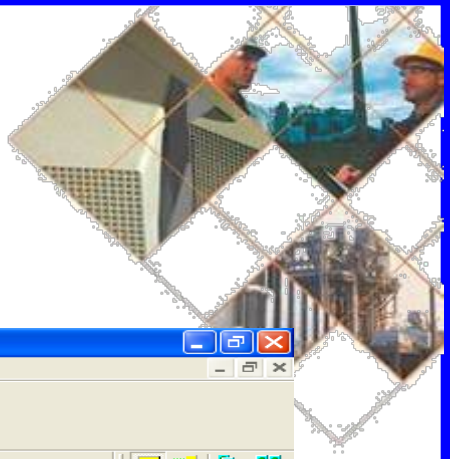
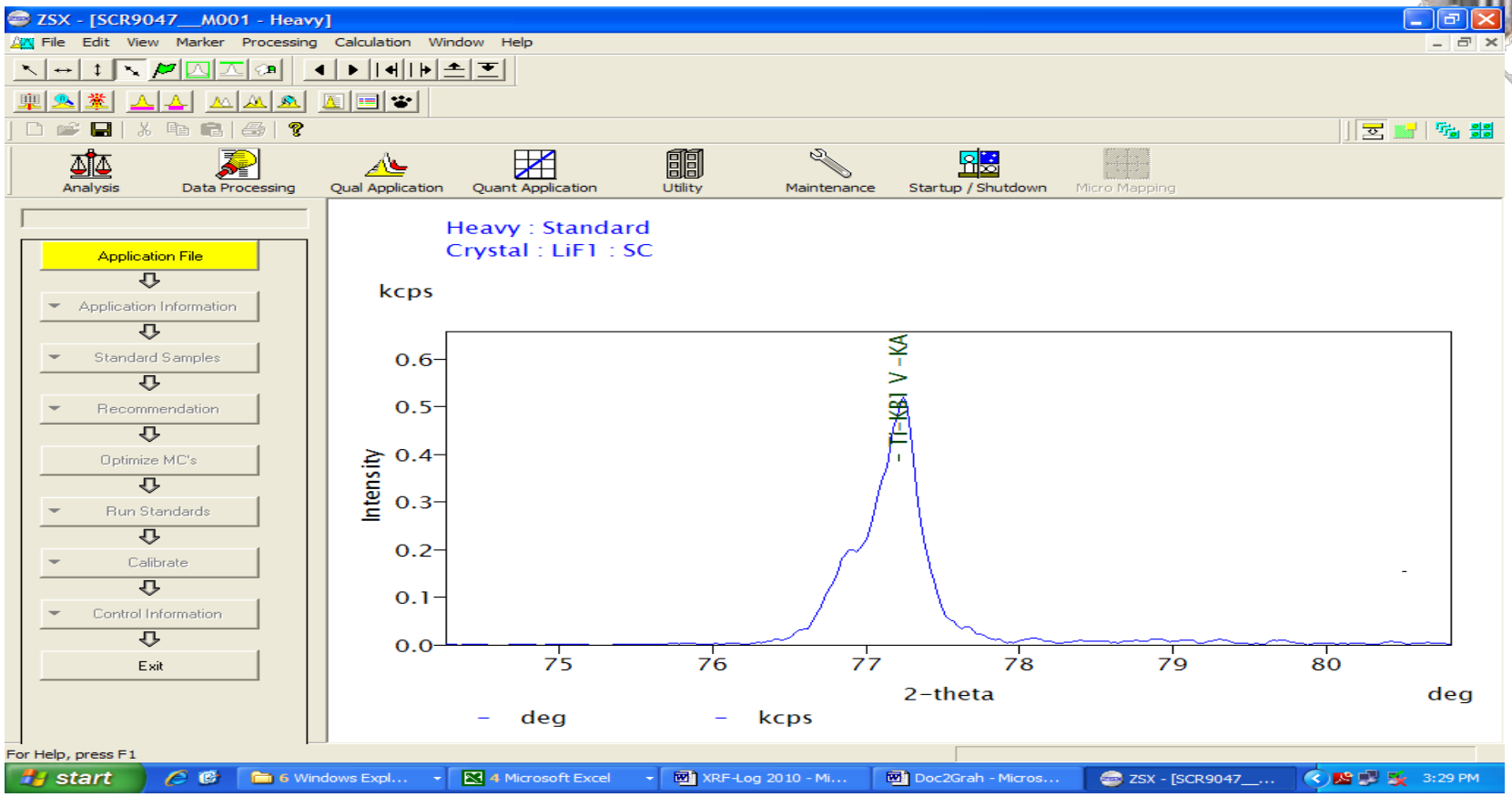
Qualitative scan Ti-V





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Summary

- Utilization of XRF-Fluorescence provides real-time results (quick feedback to production, development and commercial team).
- Decreased outside lab testing needs, therefore decreasing analysis cost.
- Improved TiO_2 testing for gas products by developing a new TiO_2 calibration line.
- The matrix make-up of each product will affect the measurement.

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Future Projects.

- Optimization and validation of a Bench-XRF fluorescence unit for lower levels of V_2O_5.
- Improve Surface analyses time.

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