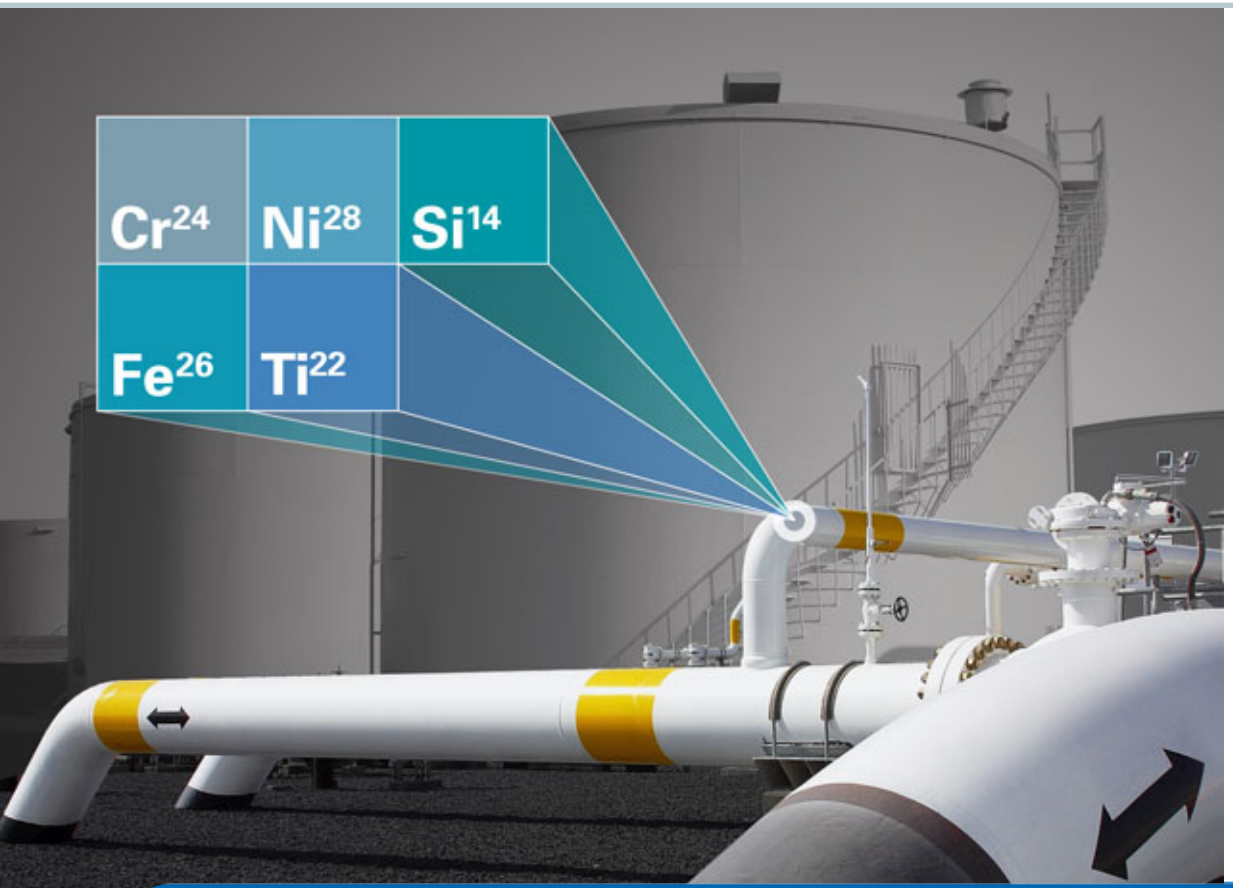
Are You Making the Grade?
Hedging the Risk with Handheld XRF

February 21, 2013



Our Panelists:

Alexander Seyfarth
Sr. Product Manager HH-XRF
Bruker Elemental
Kennewick, WA

Raymond Lindeen
Houston Analytical Instrumentation
Houston, TX

Raymond's Houston Analytical has been in the business as an instrumentation provider for over 30 years. He will introduce us to the need for PMI as well as to the unique role of XRF in it.

Mark Robert
INTEK~SOUTH

INTEK~SOUTH has been serving the refinery industry since 1982. Mark, President of INTEK~SOUTH, will discuss accuracy and measurement strategies he has been applying in the refinery segment.

Agenda

- Introduction to Positive Material identification (PMI)
- The tools of the trade… and the right way to use them
- The inspection cycle
  - When to inspect, what and how?
  - Speed vs accuracy: important facts you need to know!
- Conclusion
- Q & A session
- Meets us in person

Material Testing
Positive Material Identification (PMI)

- Over 75% of refinery failures are caused by installation of incorrect alloy
- PMI originated in the refinery industry
- PMI ensures plant safety
- Refineries require 100% inspection and verification of critical components

PMI =
Positive Material Identification

- Provides Grade and Chemistry in seconds
- Provides quick check of chemistry during metal manufacturing

PMI is a Necessity

- Accidents occurred in the past (and present) resulting in loss of a total refinery, airplane, etc. traceable to incorrect parts used
- The pressure to reduce costs has resulted in an increasing number of material/component suppliers, thus reducing the quality level
- Auditing requirements (recommended practice) API-RP-578 as part of OSHA requirements and following:
  - API 570 Piping Inspection Code
  - API 510 Pressure Vessel Inspection Code
  - API 652 Storage Tank Inspection Code

- **Selection of method is a balance of RISK MITIGATION, cost, time, and analytical requirement**

More Applications

- EU PED (The European Union Pressure Equipment Directive) demands the verification of material in one way or another (HH-XRF and OES are potential approaches)

- Pipeline systems, builders and inspection contractors are users, especially for the compressor stations

- Corrosion monitoring of cooling circuits in power plants, process installations, but also in refinery segment
  - Corrosion potential monitoring e.g. low Cr and Mn in piping indicates high corrosion potential

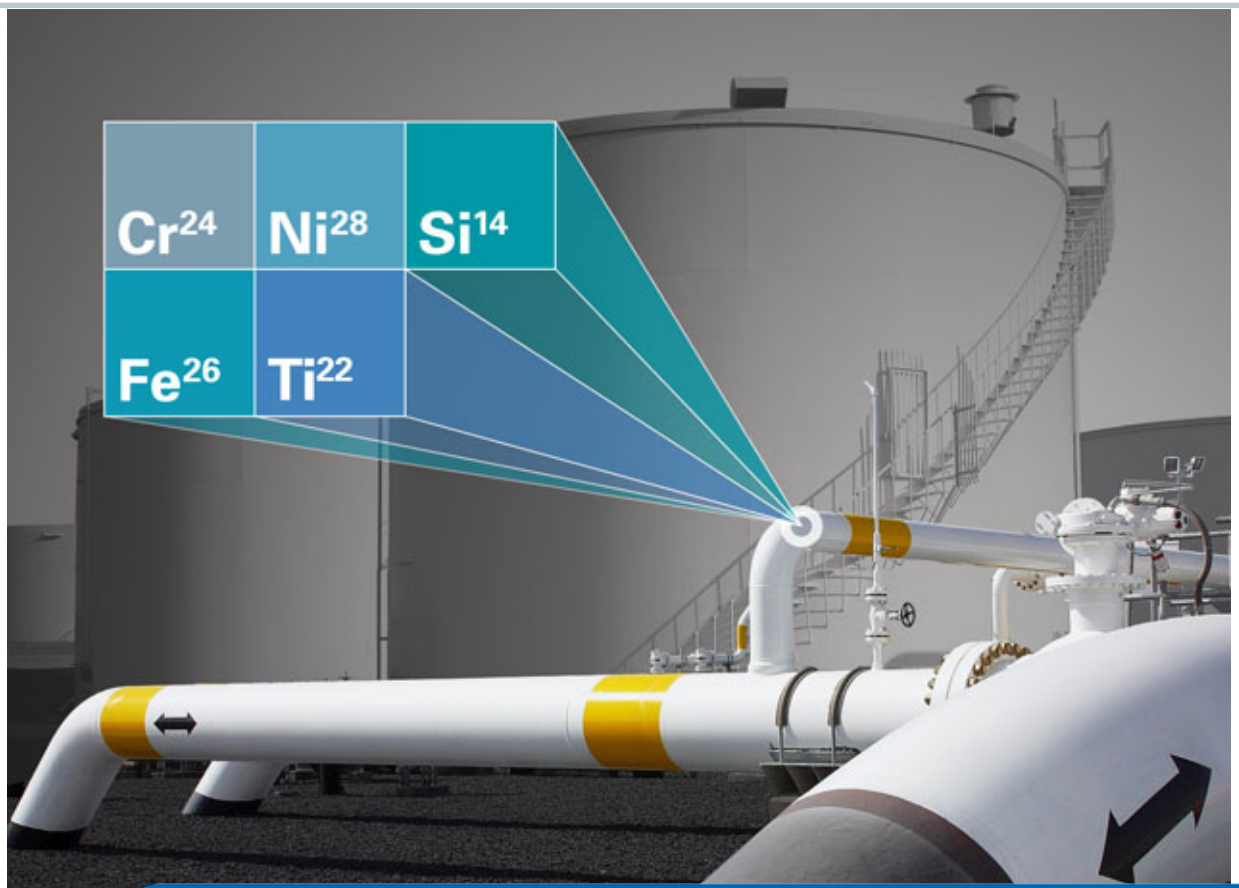
Challenge: Get Instruments to the Samples!

- Industry need for ELEMENTAL ANALYSIS for ID and CLASSIFICATION
  - Non-destructive (material to be used)
  - Fast (shortest time to result)
  - ONSITE, ON THE JOB
  - Easy to use
- “Pay back”
  - Regulatory and/or safety requirements
  - RISK MITIGATION OF UTMOST CONCERN
  - Avoid material “confusion”; mislabeling
  - SOP’s and documentation to prove diligence

- PMI is more than just tools…

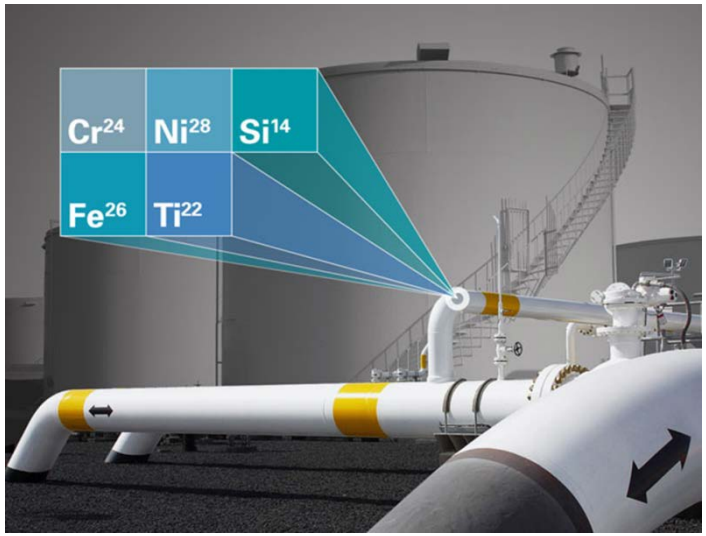
PMI: The Analytical Task

Mark Robert



PMI: The Analytical Task

- Identify a metal alloy by GRADE (GRADE ID)
- Identify OFF SPEC within a GRADE
- How can we do this?
- What tools are available?
- Two analytical techniques are used today



PMI Tools of Today - What tools do we have?

- Mobile OES (Optical Emission Spectroscopy)
- Handheld XRF

Mobile / Portable OES Spectroscopy

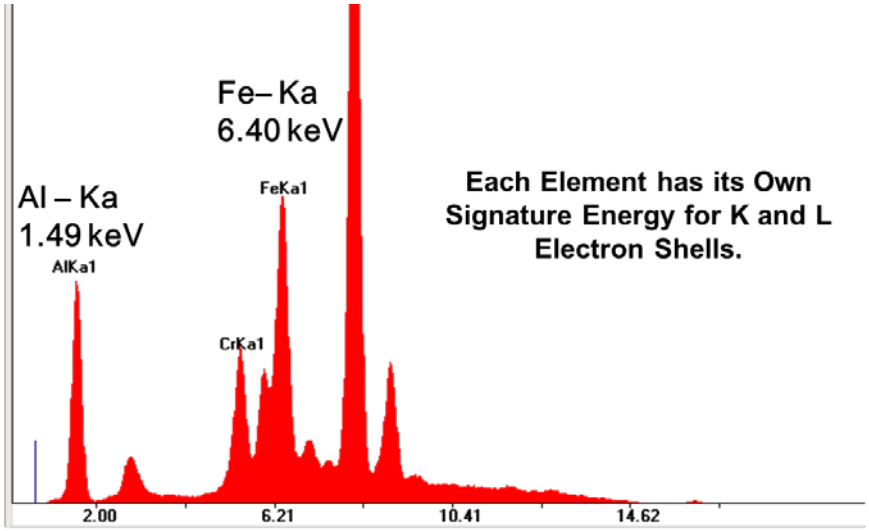
- Destructive testing (leaves burn mark)
- Requires 3-5 shots for a good analysis
- Can be used onsite and with long probe, also on scaffolding

PMI Tools of Today - Optical Emission Spectroscopy OES

- When the element “C” Carbon is required and to ID “L” Grades and “H” Grades Alloys (examples: 316L, 304L)
- Extensive Aluminum Alloy ID, may also require an OES
- Mobile or Portable lab units

HH-XRF

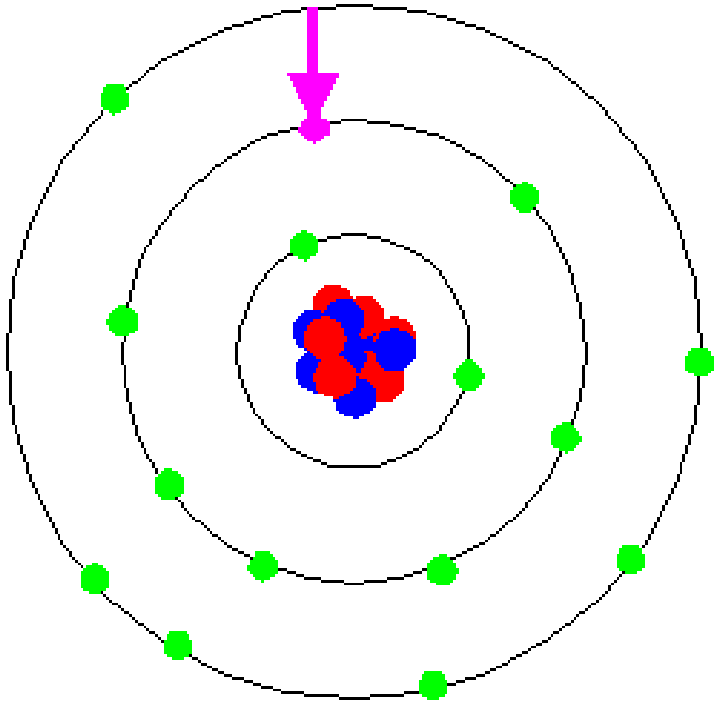
X-ray Fluorescence Spectroscopy XRF is based on excitation of elements in the sample and detection of their characteristic X-ray emission.



Each Element has its Own Signature Energy for K and L Electron Shells.

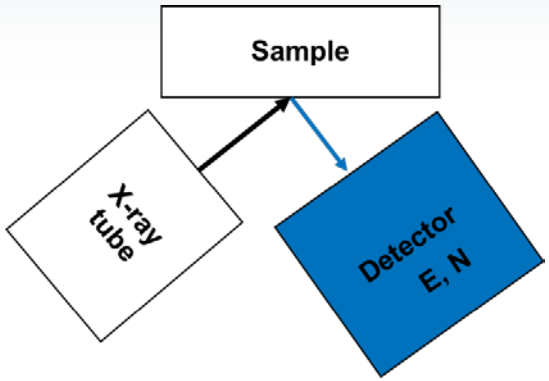
X-ray energy tells you what element it came from

Number of x-rays tells you how much is present



HH-XRF

- General setup of HH-XRF analyzer
  - Excitation tube-sample-detector
- **SiPIN** detectors (Ti – U)
  - Separates signal with < 200eV at 30,000 cps
- **Silicon Drift Detectors** SDD (Mg – U), “Light Elements” Mg, Al, Si, P, and S
  - Very good resolution, typically less than 147 eV at 100,000 cps



Detector + Electronics is the key to the analyzer performance.

Coupling of tube, sample and detector crucial

XRF is TRUE Non-Destructive Testing

PMI Tools of Today: Handheld XRF Analyzers

Silicon Drift Detectors (SDD)

- XRF Analyzers with a SDD have the ability to measure the “Light Elements” Mg, Al, Si, P, and S (Mg 0.2%) along with the standard elements normally seen.
- Also, with the 100,000 cps and a resolution of less that 147 eV, we are able to get better peak separation with precision and accurate results much quicker than the SiPIN units.
- The use of a Thin Ultralene (Prolene) Window is required on an SDD unit. This allows the LE X-rays to pass through to the detector.
- Can measures samples up to 302°F (150°C)

El	Min	%	Max	+/-
Mg	0.800	1.017	1.200	0.178
Al	95.000	97.167	99.000	0.414
Si	0.400	0.739	0.800	0.029
Ti	0.000	0.049	0.150	0.001
V		0.013		0.000
Cr	0.040	0.181	0.350	0.001
Mn	0.000	0.043	0.150	0.001
Fe	0.000	0.335	0.700	0.002
Ni		0.045		0.001
Cu	0.150	0.314	0.400	0.002
Zn	0.000	0.076	0.250	0.001
Pb		0.021		0.002

PMI Tools of Today:
Handheld XRF Analyzers

Silicon Drift Detectors – SDD

KEY NOTE: SDD analyzers can be used for “In-Service” hot pipe testing,

but must first replace the thin Ultralene window with a Kapton window.

On the downside, the Kapton window will not allow the lighter elements to pass through to the detector, so you will not be able to see Mg, Al, Si, P or S.

PMI Tools of Today: Handheld XRF Analyzers

SiPIN Detectors (Ti – U)

SiPIN XRF Analyzers make up the majority of HH-XRF's in the field today

Used to analyze Stainless Steels, Low Alloy Steels, Cu Alloys, Inconels, Monels, Ti Alloys, etc.

When your analytical needs do not require the "Light Element"

A Kapton window is used, which gives you the ability to measure samples up to 900°F (482°C)

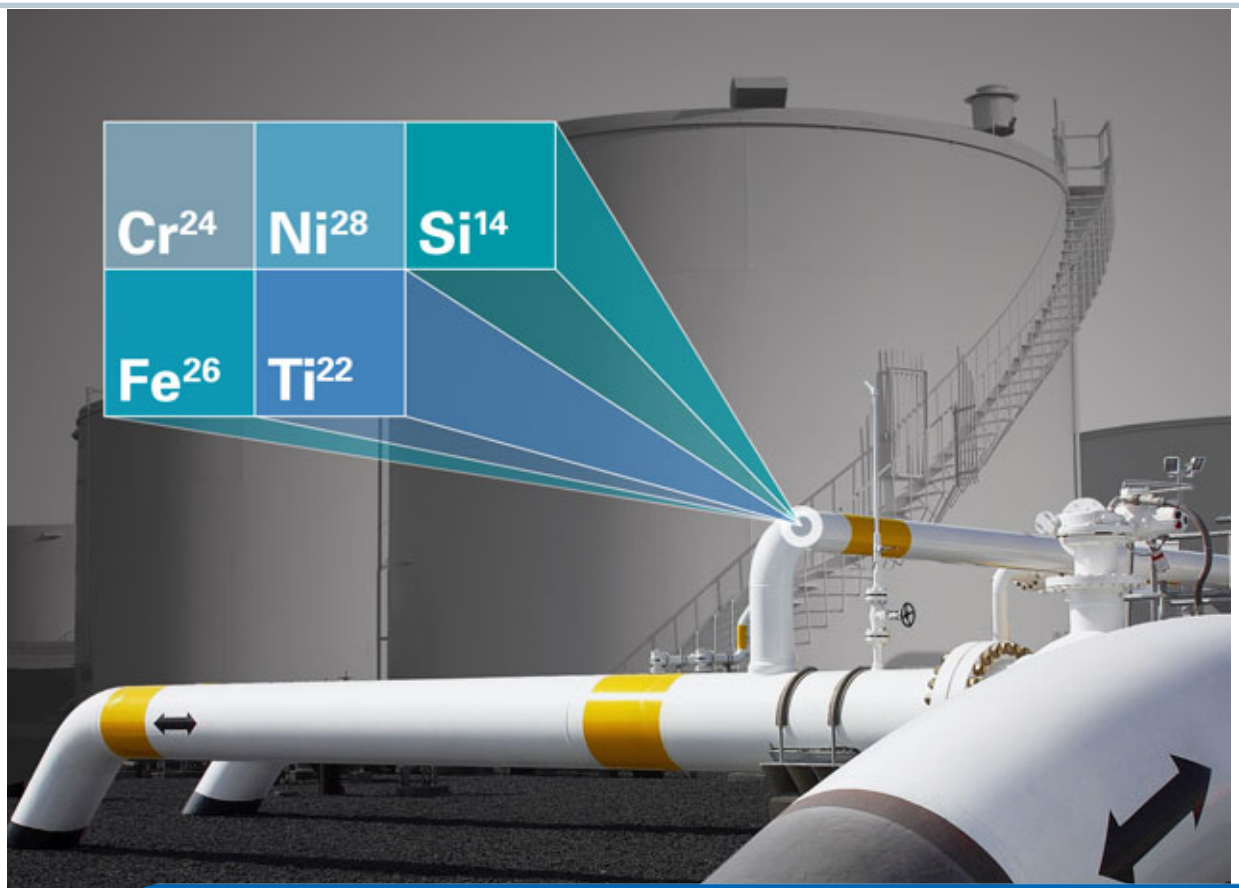
Also Non-Destructive

Summary Comparison

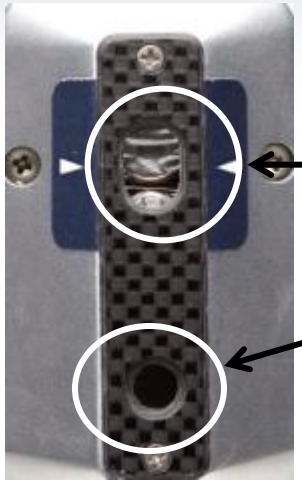
mobileOES	Capabilities	XRF
C,Mg,Al,Si, S, P,…	**Elemental Range**	Ti to U, Mg to U
Maj, Minors and Traces	**Sensitivity**	Majors Minors
0.000X	**Detection Limit**	.0X to .00X for high z
Moderate	**Speed**	Fast
Substantial	**Sample Preparation**	Minimal to Moderate
Yes (spark burn)	**Sample Surface Marking**	non destructive (none!)
Moderate	**Ease if use**	Easy
Yes (56lbs/ 25kG)	**Portability**	Hand Held < 3 lbs /1.4 kg

How to Measure Samples?

Raymond Lindeen



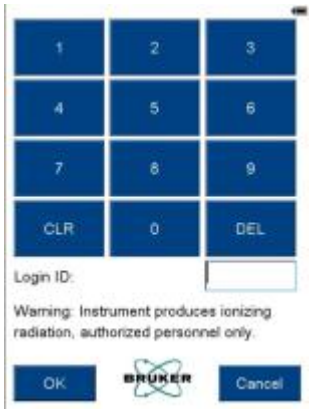
Radiation Safety



Backscatter Shut-off

IR Sample Sensor

Warning Lights

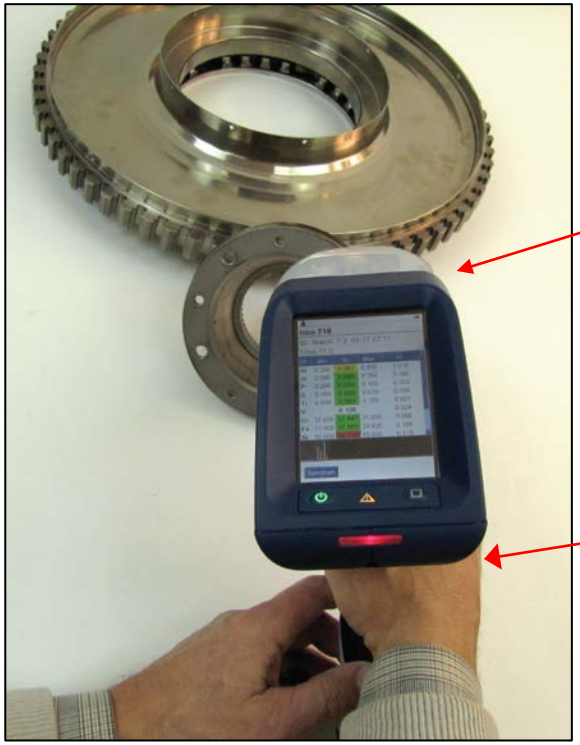


Multilevel User Login
- User
- Supervisor

X-Ray Tube Based

- Contains no radioactive materials
  - No licensing required
  - No wipe test required
  - Easy transportation
  - No disposal problems
- Complete control of excitation
  - Allows adjustment of flux
  - Allows adjustment of energy
  - Optimum excitation conditions

How it works (2)
Making Measurements



Ensure the spot you want to measure is fresh and not corroded.

1. Place sample in front of the S1. (Be sure the IR sensor is covered.)

2. Pull trigger to activate X-rays.

How it works (3)
Get Results

Alloy ID

Match Number
10 = perfect
match

Inco 792

42 Match 9.8 01-04 19:38

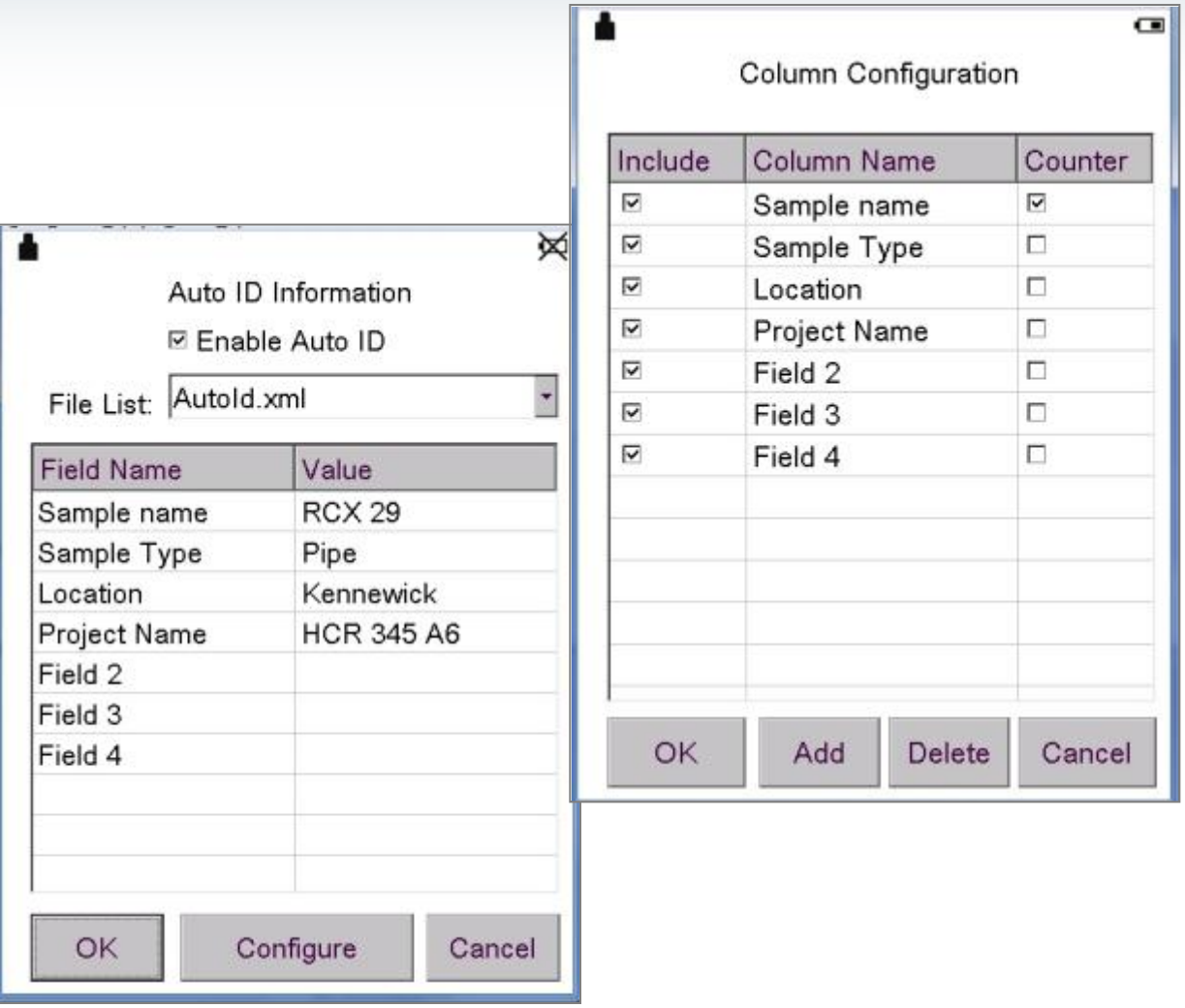
Time 3.0

El	Min	%	Max	+/-
Ni	60.00	62.50	69.00	1.76
Cr	11.00	12.36	13.00	0.32
Co	8.00	8.94	10.00	0.28
W	3.60	3.98	4.59	0.17
Ti	3.50	3.75	4.50	0.20
Ta	3.50	3.60	4.50	0.15
Mo	1.60	2.00	2.40	0.09

Grade
Range

2 Standard
Deviation

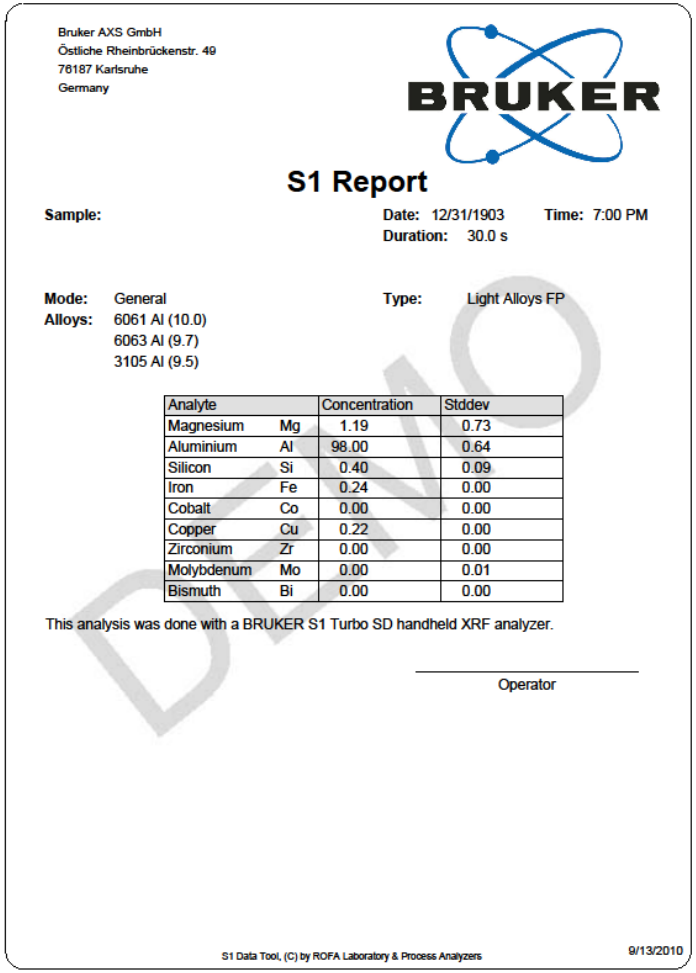
Sample Identification



Sample naming

- Multiple fields for sample naming
- Can add fields
- Can include batch and unique ID
- Input via keyboard
- Input via Barcode reader

Report Generation Software



Bruker AXS GmbH
Östliche Rheinbrückenstr. 49
76187 Karlsruhe
Germany

S1 Report

Sample:

Date: 12/31/1903 **Time:** 7:00 PM
Duration: 30.0 s

Mode: General **Type:** Light Alloys FP
Alloys: 6061 Al (10.0)
6063 Al (9.7)
3105 Al (9.5)

Analyte		Concentration	Stddev
Magnesium	Mg	1.19	0.73
Aluminium	Al	98.00	0.64
Silicon	Si	0.40	0.09
Iron	Fe	0.24	0.00
Cobalt	Co	0.00	0.00
Copper	Cu	0.22	0.00
Zirconium	Zr	0.00	0.00
Molybdenum	Mo	0.00	0.01
Bismuth	Bi	0.00	0.00

This analysis was done with a BRUKER S1 Turbo SD handheld XRF analyzer.

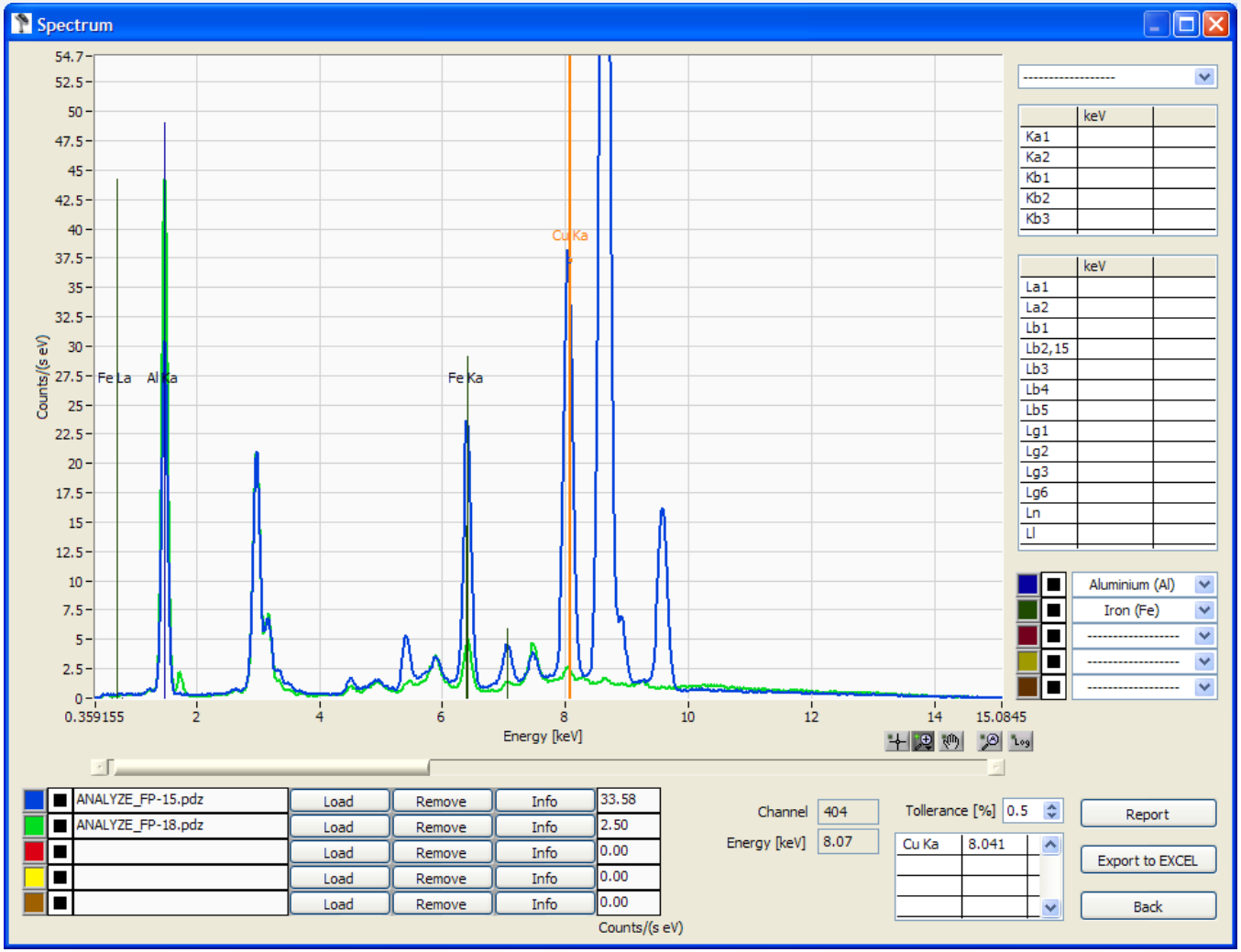
Operator

S1 Data Tool, (C) by ROFA Laboratory & Process Analyzers 9/13/2010

Using the Reporting tool

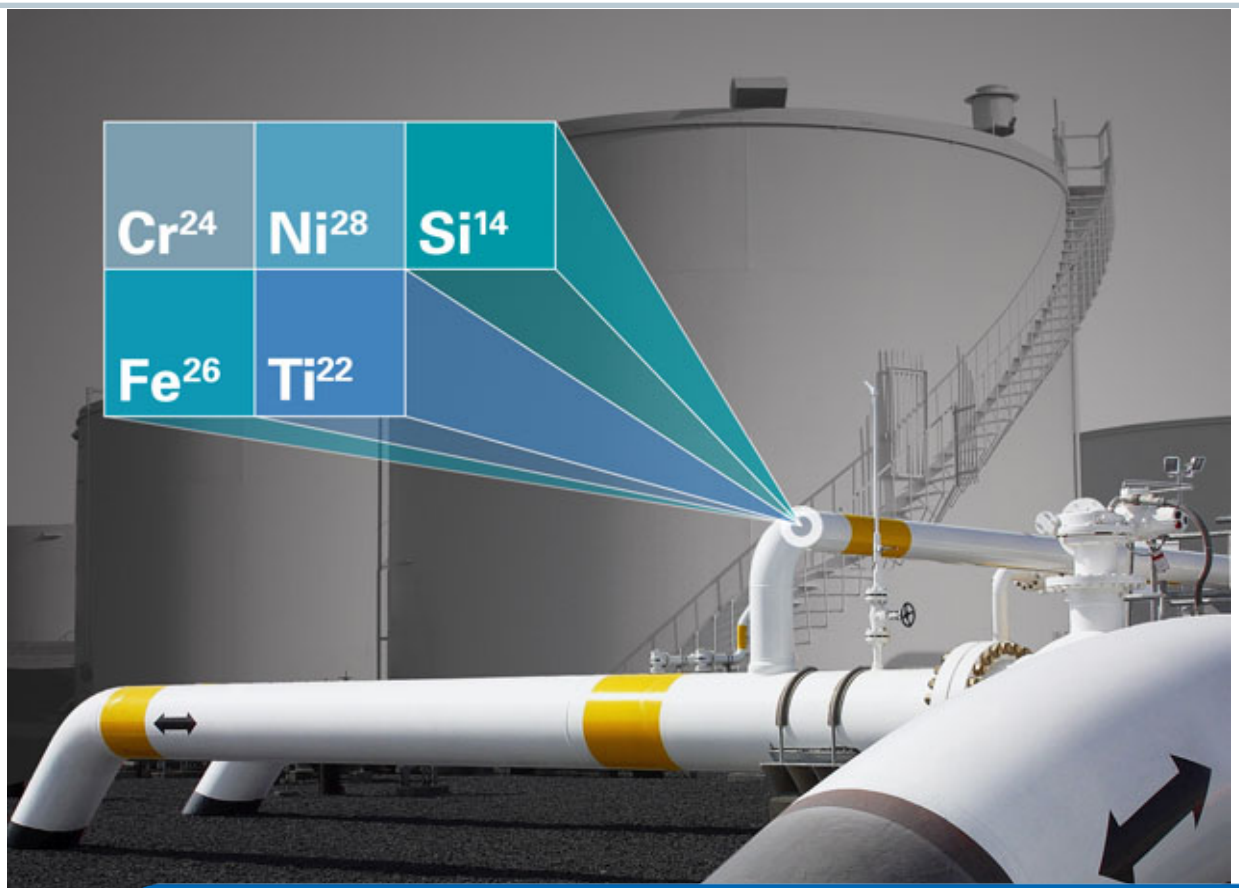
- Documents the assay traceable to the secure data storage
- Take USB stick and make report
- Data can also be imported into Plant Management Software

Include the ULTIMATE proof (the spectra!) for elemental content



What needs to be tested?

Raymond Lindeen



OSHA Safety Directive in 2007

OSHA INSTRUCTION: Directive number CPL 03-00-004, effective June 7, 2007, which is the "Petroleum Refinery Process Safety Management Emphasis Program". The purpose: "This instruction describes policies and procedures for implementing a National Emphasis Program (NEP) to reduce or eliminate the workplace hazards associated with the catastrophic release of highly hazardous chemicals at petroleum refineries.

API RP 578 as a procedure to ensure implementation

PMI of Critical Components

Critical Components shall be defined as those components whose integrity is essential to the performance and safe operation of the system. The Contractor shall submit a detailed PMI plan that identifies all piping and equipment that will be subjected to 100%, random and no PMI. The Owner/User shall review and modify as required and approve the PMI plan. The following list provides guidance ***by One Owner/User*** and can be supplemented by the Owner/User at any time. Such integrity critical components (and their recommended inspection frequency) include the following:

PMI of Critical Components

1. Pressure Vessels (shells, nozzles, flanges, and welds in or on pressure-retaining parts, linings) - 100% PMI.

2. Facility Piping (pipe, fittings, flanges, blinds, couplings, bolts, and welds) – 100% PMI for no category D piping and where service conditions require the inclusion and exclusion of 100% PMI, shall be approved by the Company.

3. Pipelines - 20% PMI (Project team may require up to 100% PMI if the pipeline in question represents a high-risk situation).

4. Weld heads and Christmas trees - 100% PMI.

5. Hydrocarbon compressors, pumps, valves, strainers, etc. - 20% (Project team may require up to 100% PMI if the pipeline in question represents a high-risk situation).

6. Fasteners - 20% PMI (Project team may require up to 100% PMI if the application in question represents a high-risk situation).

7. Soft metal seals for valve flanges-- 100% PMI.

Inspection of materials to be used in the process

- As risk mitigation and to ensure full OSHA compliance product is inspected during
  - Manufacturing of product (Shipping)
  - Receiving of material (Receiving)
  - Before use (on site)
- This has to be non-destructive!

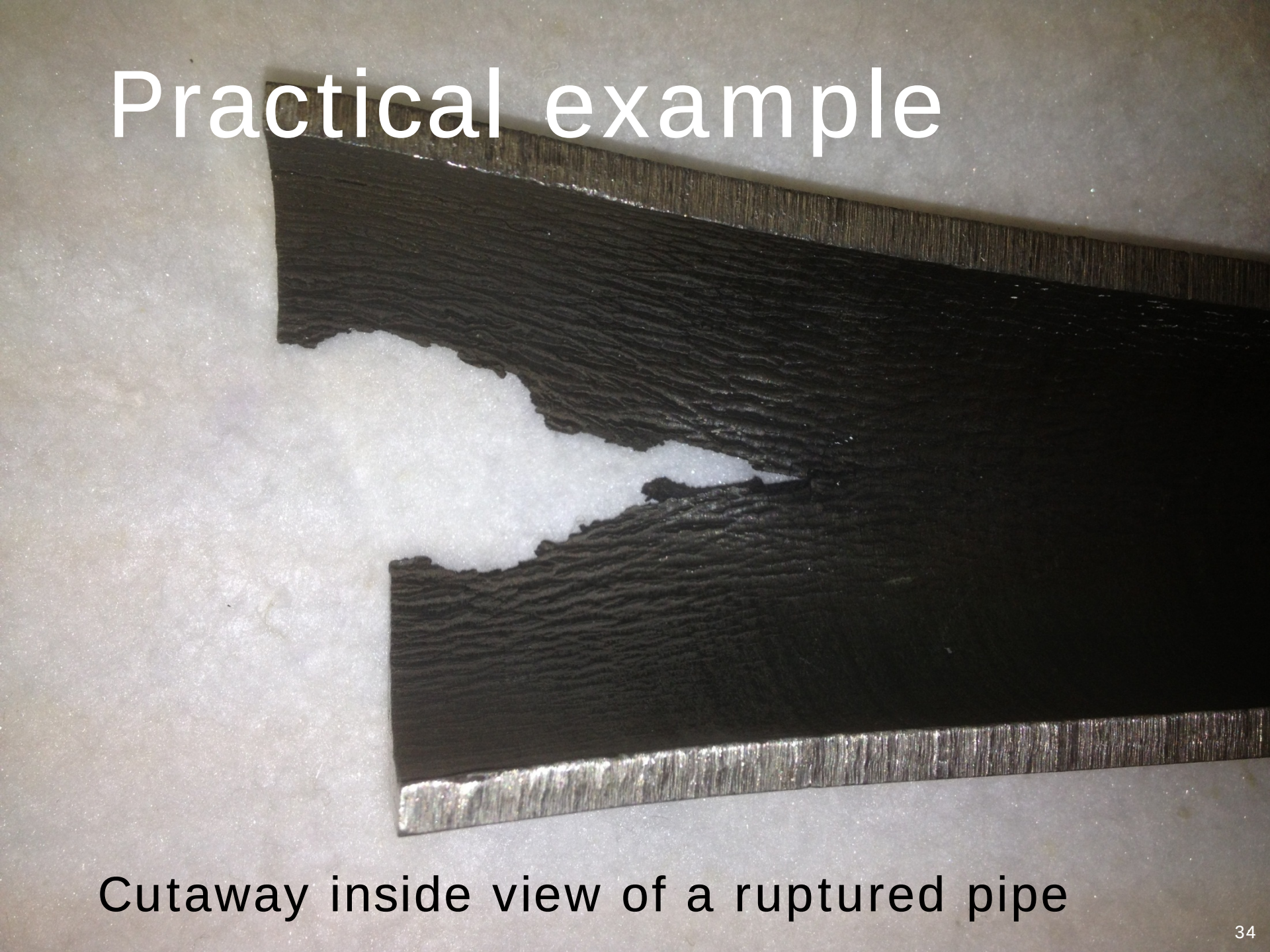
Inspection during TURNAROUND
Refinery / Pressure Vessels

- Inspection of all critical components according to API procedures (API 578, 570, 510 and 6062)
- Compliance with OSHA directive

VALIDATE PLANT ONSITE

- “Cold” pipes and equipment
- Limited sample preparation
- Predefined measurement positions

Practical example



Cutaway inside view of a ruptured pipe

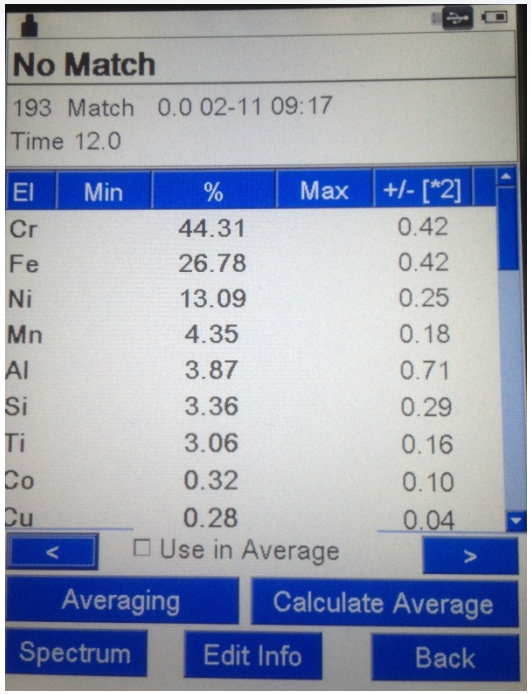
Outside view of the ruptured pipe

HH-XRF Inspection
Press button, get banana?

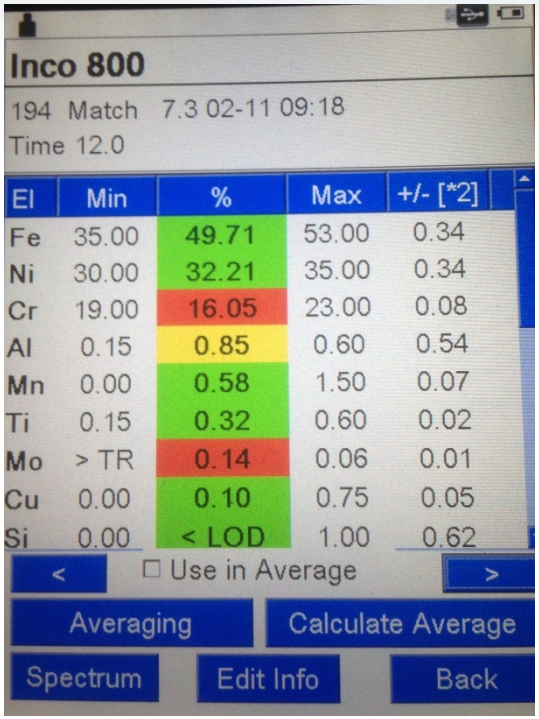
Pipe to be tested at 3 spots

1) No prep
2) Moderate prep
3) Deep sample preparation

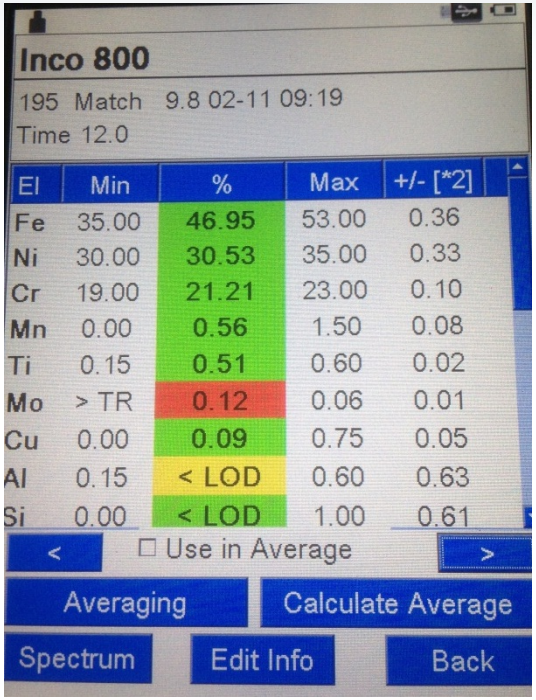
One Pipe, 3 spots, 3 results?



Not prepared
Cr content way high



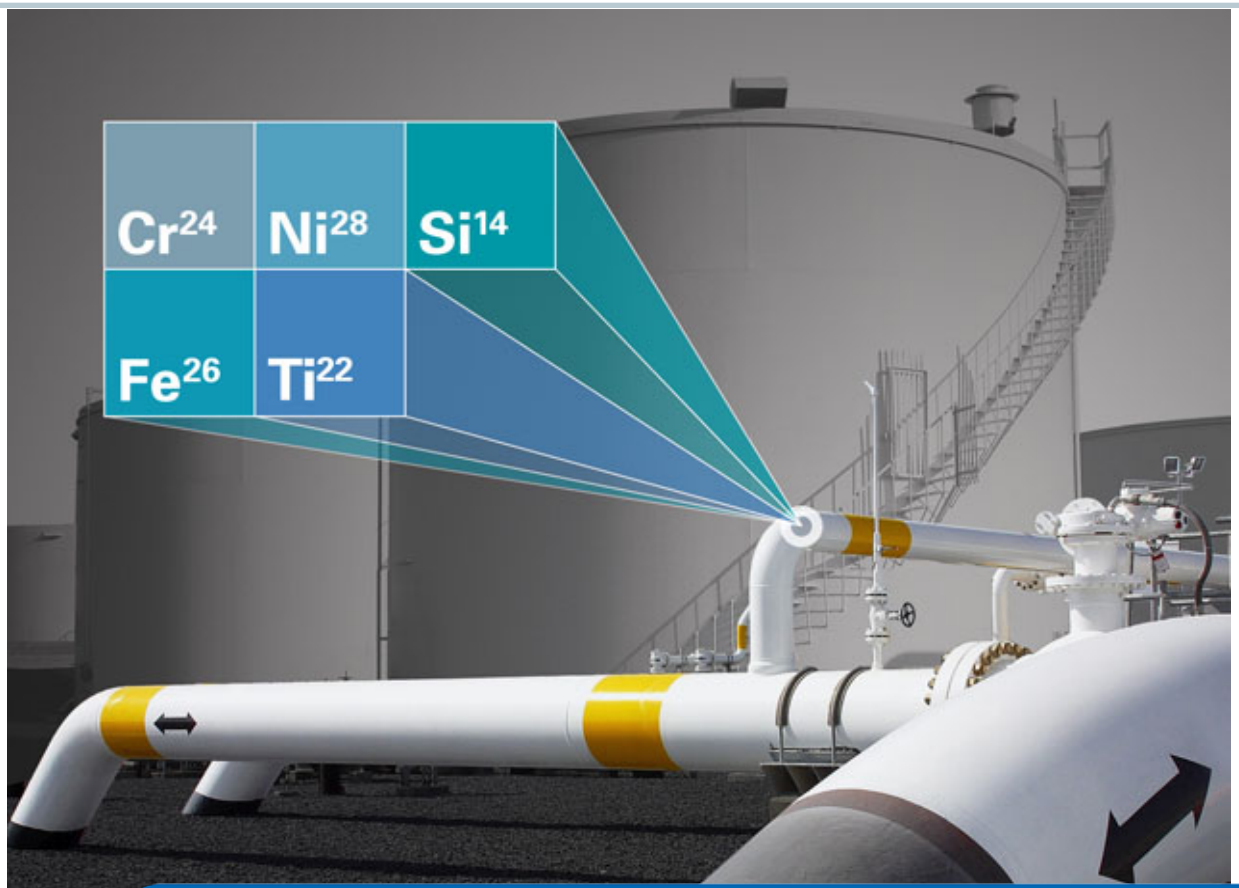
Moderate quick prep
Match poor



Surface prep ok with
high match confidence

Speed vs Accuracy

Mark Robert



PMI – Speed vs Accuracy
Knowing the difference is the key

What is more important when it comes to PMI testing, speed or accuracy?

Can we have both?

This is probably the most important question for Positive Material Identification. Although speed is nice and important for ones who need to meet deadlines, without accuracy the job is null, meaningless.

“Point and Shoot “ is a term widely used with Handheld XRF analyzers! We all use it!

But the KEY is the results. Are they correct?
Look at the alloy chemistry. Know what elements are required for that alloy.

DO NOT assume the analyzer is always right!

PMI – Speed vs Accuracy

Knowing the difference is the key

Most companies have SOP's for their PMI program's that verify the analyzer accuracy and repeatability.

This would include testing a known alloy standard such as a (NIST traceable or equivalent alloy) similar to the alloy in the plant, before a job. This ensures the analyzer is working properly. Also can establish a required minimum testing time to get the accuracy required.

API 578 states – The Contractor shall submit a detailed PMI plan that identifies all piping and equipment that will be subjected to 100%, random and no PMI. The Owner/User shall review and modify as required and approve the PMI plan.

PMI – Speed vs Accuracy

Knowing the difference is the key

In 2008, the development of the HH-XRF with Silicon Drift Detector opened a whole new world for us. Gives us the ability to see the “Light Elements,” such as Si. It also gives us the ability to see DUST. When analyzing for lighter elements in the field, make it a practice to clean the sample before testing. DUST particles will cause a significant increase in your Si values, throwing off your readings.

Is the surface clean?

Handheld XRF analyzers analyze the surface of the material. If the alloy has a coating, paint, excessive rust, this could throw off your results, rendering a no match and scratching of the head. Why did I get this for my results?

Grinding or cleaning may be required! Use caution when grinding. Possible contamination from the grinding media can occur.

Where does Speed vs Accuracy really count?

- Measuring In-Service object poses a new challenge to both tool and tool user
- Safety and production impact are of utmost importance

PMI – “In-Service” Testing
Hot Pipe Testing

- “In-Service” hot pipe testing is possible with the Handheld XRF analyzers. Learning the proper technique for this harsh environment is the key.
- Am I using the right analyzer for the job?
- Do you have the right window Kapton (yellow or mirrored) vs Ultralene (Prolene) clear window?
- Use the “TILTING METHOD.” Hold the nose of the analyzer at an angle, either the top or either side touching the pipe.

PMI – “In-Service” Testing
Hot Pipe Testing

In-Service, Hot Pipe Testing

Testing time typically 3-7 seconds – can stop test sooner, if required results are achieved.

General rule: 1 test per minute allows you and the analyzer to cool down.

Phase 1 only. Disable the “Light Element” Phase 2 when using a SDD analyzer. You will not be able to see the light elements. Elemental range from Ti up.

Conclusion

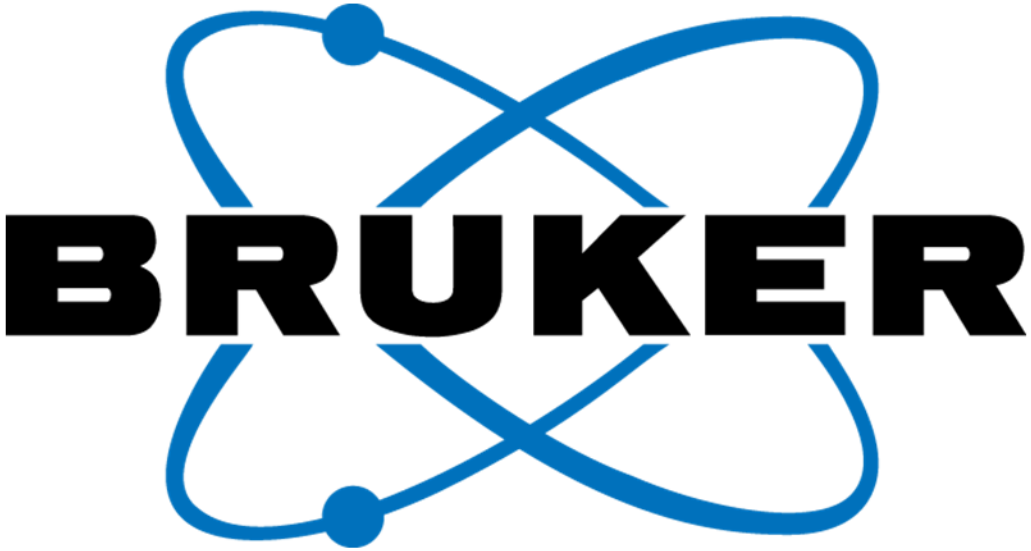
- PMI capabilities using HH-XRF have advanced dramatically in the last few years
- Instrumentation has become more capable, much LIGHTER and easier to use
- More sensitive and faster detectors can get better, more exact matches
- Important is the “correctness” of the analysis: the SOP needs to take sample preparation into account
- Doing a good job in an inspection is not just pulling the trigger of an instrument…
- Your inspection operating procedure, training of the tester and correct use of the instrument will ensure the tool works up to its specification!

Q and A

- Use the Q & A panel on the right hand side to send us your questions

Meet us in 2013

- March 18-21: NACE, Orlando, FL
- September 10-12: Quality Expo, Chicago, IL
- November 4-7: ASNDT Fall Conference, Las Vegas, NV
- November 11-15: API Fall Refining and Equipment standard meeting,
  New Orleans, LA



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