Cement Solutions: Improved C-114 Qualification with WDXRF
Welcome

Today’s topics

- Chemical analysis of cement by XRF
- XRF analysis of cement and C-114 qualification
  - C-114 primer
  - How to qualify your system
  - Sample prep as key
  - Settings on the XRF
  - Making it work together
- Challenges with blended cements
- Solving the mineralogical effect
  - Fusion ABC... How to...
  - Accuracy and precision for fusion
  - When to use fusion
- High throughput laboratory?
  - Increase sample throughput and consistency
- Conclusion

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Madison, WI, USA
Today’s Panelists

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**Bruker AXS Inc.**
Madison, WI, USA

John Anzelmo
President
Claisse USA.
Madison, WI, USA
Cooperation Scientific Claisse: First in Fusion HQ in Canada
Cooperation Scientific Claisse: First in Fusion: Claisse USA
Claisse Worldwide Presence
Please use your mouse to answer the question on your screen:

In what capacities do you analyze cement? Check all that apply:

- Routine process control
- Shipping & receiving (terminal)
- External product testing or commercial lab
- DOT/DOR government agency
- Cement user – check variety of incoming materials
- Other
Chemical Analysis of Cement by XRF

- XRF as technique replaced traditional wet chemistry for routine analysis
  - Remember the gravimetric analysis of SiO2?

- Referee method is defined by C-114; XRF is just a rapid test method which needs to be qualified.

- Rapid Test Methods Qualification:
  - XRF or other instrumental methods need to be qualified as fast test methods to ensure that they comply with the requirements of C-114
  - Accuracy
  - Precision
  - By using defined reference material
  - By using adequate reference material
XRF Analysis of Cement – ASTM C-114 Qualification

Arkady Buman
Designation: C 114 – 97a

Standard Test Methods for
Chemical Analysis of Hydraulic Cement

This standard is issued under the fixed designation C 114; the number immediately following the designation indicates the year of
original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A
superscript epsilon (e) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 These test methods cover the chemical analyses of
hydraulic cements. Any test methods of demonstrated ac-
cceptable precision and bias may be used for analysis of
hydraulic cements, including analyses for referee and certi-
cation purposes, as explained in Section 3. Specific chemical
test methods are provided for ease of reference for those
desiring to use them. They are grouped as Reference Test
Methods and Alternative Test Methods. The reference test
methods are long accepted wet chemical test methods which
provide a reasonably well-integrated basic scheme of analysis
for hydraulic cements. The alternative test methods generally
provide individual determination of specific components
and may be used alone or as alternates and determinations
within the basic scheme at the option of the analyst and as
indicated in the individual method. The individual analyst
must demonstrate achievement of acceptable precision and
bias, as explained in Section 3, when these test methods are
used.

1.2 Contents:

<table>
<thead>
<tr>
<th>Section</th>
<th>Subject</th>
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<td>Reference Documents</td>
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<td>3</td>
<td>Number of Determinations and Permissible Variations</td>
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<td>3.3</td>
<td>Performance Requirements for Rapid Test Methods</td>
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<td>Precision and Bias</td>
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<td>4</td>
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<td>Interferences and Limitations</td>
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<td>Sample Preparation</td>
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<td>4.5</td>
<td>General Procedures</td>
</tr>
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<td>4.6</td>
<td>Recommended Order for Reporting Analyses</td>
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<td></td>
<td>Reference Test Methods</td>
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</table>

2. Referenced Documents

2.1 ASTM Standards:

C 25 Standard Test Methods for Chemical Analysis of
Limestone, Quicklime, and Hydrated Lime
C 115 Test Method for Fineness of Portland Cement by
the Turbidimeter
C 150 Specification for Portland Cement
C 183 Practice for Sampling and the Amount of Testing of
Hydraulic Cement
C 255 Specification for Blended Hydraulic Cements
D 1193 Specification for Reagent Water
E 29 Practice for Using Significant Digits in Test Data to
Determine Conformance with Specifications

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1 These test methods are under the jurisdiction of ASTM Committee C-1 on
Cement and are the direct responsibility of Subcommittee C01.23 on Composi-
tional Analysis.

published as C 114 – 34 T. Last previous edition C 114 – 97.

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3 Annual Book of ASTM Standards, Vol 04.01.
## ASTM C-114 Qualification
### Permissible Variations in Results

<table>
<thead>
<tr>
<th>Component</th>
<th>Max Difference between Duplicates on alternate days</th>
<th>Max Difference of the Average of Duplicates from SRM Certificate</th>
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<tbody>
<tr>
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<td>0,16</td>
<td>0,2</td>
</tr>
<tr>
<td>Al2O3</td>
<td>0,20</td>
<td>0,2</td>
</tr>
<tr>
<td>Fe2O3</td>
<td>0,10</td>
<td>0,10</td>
</tr>
<tr>
<td>CaO</td>
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<td>0,3</td>
</tr>
<tr>
<td>MgO</td>
<td>0,16</td>
<td>0,2</td>
</tr>
<tr>
<td>SO3</td>
<td>0,10</td>
<td>0,1</td>
</tr>
<tr>
<td>Na2O</td>
<td>0,03</td>
<td>0,05</td>
</tr>
<tr>
<td>K2O</td>
<td>0,03</td>
<td>0,05</td>
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<td>TiO2</td>
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<td>P2O5</td>
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<tr>
<td>ZnO</td>
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<td>0,03</td>
</tr>
<tr>
<td>Mn2O3</td>
<td>0,03</td>
<td>0,03</td>
</tr>
</tbody>
</table>
ASTM C-114 Qualification Procedure

- A sensible procedure, fully accepted by the industry
- Helps the cement manufacturer and instrument manufacturer to certify the instrument and sample preparation procedure
- From the instrumentation side, it is relatively simple for WDXRF medium- and high-power instruments
- Sample preparation part is much more difficult and complicated
- In general we can guarantee that we will meet C-114 if sample prep is OK
Please use your mouse to answer the question on your screen:

What sample preparation method do you currently use most often?

- Pressed pellet with binder
- Pressed pellet without binder
- Fusion
- None / Not Applicable
ASTM C-114 Qualification
Procedure – Sample Preparation

■ Two types of sample preparation can be used:
  • Pressed pellets: usually 5-7 g of sample and 1 g of binder are mixed and grind together for 2-3 min and then pressed into a pellet.
  • Fusion beads: 1 g of sample and 5-7 g of LiBO$_3$ are mixed together and heated to 900-1100 degrees C and then cast into a dish to make a flat bead.
The intensities from fusion beads can be 3 to 7 times less than intensities from pressed pellets, depending on dilution factor. That can dictate the power of the XRF instrument needed to meet C-114. For diffraction, the sample must be in the form of pressed powder.
Prepare seven samples of the same material (your own cement, for example).

Analyze them on the instrument in the following order:

1st sample 2nd sample
1st sample 3rd sample
1st sample 4th sample
1st sample 5th sample
1st sample 6th sample
1st sample 7th sample
Do statistical analysis of seven measurements of the same sample. That will give you the instrument repeatability.

Modern instruments will have less than .07% variation on CaO and less than .03% for SiO2 in cement.
ASTM C-114 Qualification
Procedure – Repeatability of Sample Preparation

- Do statistical analysis of measurements of the seven different samples of the same material. That will give you the instrument and sample preparation repeatability.
- On those seven samples you should meet the C-114 requirements for CaO and SiO₂.
- It is relatively easy now to see how repeatable your sample preparation is. If you are not meeting the C-114 requirements, there is no sense in preparing the NIST standards.
The following conditions are usually applied for cement applications:

**O-Mg** – multilayer crystal OVO-55, medium collimator, flow proportional counter (FPC)

**Si-K** – PET crystal, medium collimator, FPC

**K-Fe** – LiF(200) crystal, medium or fine collimator, FPC

**Fe-Cd** – LiF(200) crystal, fine or medium collimator, scintillation counter (SC)

**P, S, Cl** – Ge(111) crystal, medium collimator, FPC
The new multilayer XS-CEM was recently introduced on our new S8 TIGER XRF spectrometer.

This crystal was specifically designed for analysis of Al and Si and is much more stable than PET.

With this crystal it is possible to have a WDXRF spectrometer for cement analysis without the PET crystal by analyzing Al and Si on the XS-CEM crystal and P, S, and Cl on the Ge crystal.

This configuration drastically improves long term stability of the instrument.
ASTM C-114 Qualification
Collimators

- Sample: X-rays to all directions
- Sequential spectrometers require parallel beams
- Collimators to suppress X-rays which are not close to parallel
- For cement application, two collimators are needed: .23 and .46
In general for cement applications 30 kV voltage is needed. The current on the X-ray tube will depend on the total power of your instrument:

- 1000 W – current will be 33 mA
- 3000 W – current will be up to 100 mA
- 4000 W – current will be 135 mA up (to 170 mA)
ASTM C-114 Qualification
Precision and Counting Statistics

Precision is limited by counting statistical error

\[
\frac{\Delta c}{c} = \frac{\sqrt{N}}{N} = \frac{1}{\sqrt{N}}
\]

<table>
<thead>
<tr>
<th>N</th>
<th>(\sqrt{N})</th>
<th>(3\times \sqrt{N}/N)</th>
</tr>
</thead>
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<tr>
<td>100</td>
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<td>30%</td>
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<td>1000</td>
<td>30</td>
<td>10%</td>
</tr>
<tr>
<td>10000</td>
<td>100</td>
<td>3%</td>
</tr>
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<td>100000</td>
<td>300</td>
<td>1%</td>
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<tr>
<td>1000000</td>
<td>1000</td>
<td>0.3%</td>
</tr>
<tr>
<td>10000000</td>
<td>3000</td>
<td>0.1%</td>
</tr>
</tbody>
</table>
ASTM C-114 Qualification
Precision and Accuracy

- Precision is the ability of the instrument to produce reproducible data. It is a parameter of the instrument. WDXRF is the most precise analytical technique.

- Accuracy defines how close analytical results are to the true values. It is not a parameter of the instrument. Accuracy depends on how good your standards are, how good the sample preparation is, how you select analytical conditions and corrections, etc.
ASTM C-114 Qualification
Precise or Accurate Results?

- High Accuracy, High Precision
- High Accuracy, Low Precision
- Low Accuracy, High Precision
- Low Accuracy, Low Precision
Matrix effects depend on the composition of the sample.

After the sample is irradiated with primary X-rays from the X-ray tube, every element in the sample emits secondary fluorescence, which is unique for each element.

Depending on its energy, secondary fluorescence can be absorbed by other elements in the sample.

If the element is absorbing, it will be enhanced by this additional excitation. The element that provides the additional excitation loses some of its secondary fluorescence and will be absorbed.

As a result of this effect, some elements will have higher or lower intensities even though their concentration does not change.
Matrix effects are predictable and depend on the energy of the characteristic lines and absorption edges of the elements present in the sample.

So before you start evaluating matrix effects, a total qualitative scan of the average sample has to be done.

After you know all the elements present in the sample, the evaluation process can begin.
How to evaluate and predict interelement corrections necessary for successful empirical calibrations:

1. Absorption effect is at least three times more severe than enhancement effect.
2. The closer characteristic lines and absorption edges of analyte and interfering elements are to each other the more severe effect will be.
3. After applying the correction the standard error for the calibration of the analyte should improve at least 20%, for the correction to be valuable.
4. If the concentration of the interfering element does not change, there is no sense to apply correction for it. (In other words the dynamic range of interfering element is very important).
5. Number of standards $-N$ required for the calibration with number of corrections $-K$, should be determine from the following equation: $N=2K^2 +1$
The following empirical corrections can be used for pressed pellets if at least nine standards are available (no more than 2 corrections can be applied at the same time):

For Ca – K, S ( Fe, Si, LOI)
  Si – Mg, Al (Ca, S)
  Al – Si, Mg (Ca, Fe)
  Mg- Si, Al (Ca)
  S – Si, Ca ( Fe)
  Fe, K, Na – generally do not require corrections
ASTM C-114 Qualification Procedure

- Calibrate instrument using valid curve-fitting procedures (point-to-point saw tooth not allowed)
- Verify repeatability of sample preparation procedure
- Prepare NIST Portland-Cement SRM’s and analyze on day 1
- Next day, prepare a new set of SRM’s and analyze again
- Calculate the differences between the values, and averages of the values from the two rounds of tests
- Compare to values in Table 1 - be aware of significant digits for each value in the table
### ASTM C-114 Qualification
#### Permissible Variations in Results

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<td>0.03</td>
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<tr>
<td>ZnO</td>
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<td>0.03</td>
</tr>
<tr>
<td>Mn2O3</td>
<td>0.03</td>
<td>0.03</td>
</tr>
</tbody>
</table>
ASTM C-114 Qualification
Permissible Variations

- All within prescribed limits when <7 SRM’s are used
- 6 within, 7th no more than 2X prescribed limits
- 77% within and remainder within 2X prescribed limits
- Interelement corrections allowed for any oxide when the correction is applied to all standards used for that calibration
- Qualification data shall be made available pursuant to the Manufacturers Certification Section of C 150, or the Certification Section of Specification C 595
- Requalification by CCRL or as specified in Section 3.3.6.3 of C-114
# ASTM C-114 Qualification

## Accuracy Data

<table>
<thead>
<tr>
<th>Element</th>
<th>Concentration Range [%]</th>
<th>ASTM Max. Diff</th>
<th>S8 TIGER Max. Diff</th>
</tr>
</thead>
<tbody>
<tr>
<td>Na₂O</td>
<td>0.08 - 0.45</td>
<td>0.05</td>
<td>0.02</td>
</tr>
<tr>
<td>MgO</td>
<td>1.2 - 4.2</td>
<td>0.2</td>
<td>0.1</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>3.1 - 5.8</td>
<td>0.2</td>
<td>0.1</td>
</tr>
<tr>
<td>SiO₂</td>
<td>17.6 - 25.2</td>
<td>0.2</td>
<td>0.1</td>
</tr>
<tr>
<td>P₂O₅</td>
<td>0.02 - 0.2</td>
<td>0.03</td>
<td>0.03</td>
</tr>
<tr>
<td>SO₃</td>
<td>1.9 - 3.2</td>
<td>0.1</td>
<td>0.1</td>
</tr>
<tr>
<td>K₂O</td>
<td>0.1 - 1.5</td>
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<td>CaO</td>
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<td>MnO₃</td>
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<td>0.01</td>
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<td>Fe₂O₃</td>
<td>0.3 - 4.4</td>
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# Repeatability – 30 Measurements on Same Sample, Day 1

<table>
<thead>
<tr>
<th>Time</th>
<th>Na₂O [%]</th>
<th>MgO [%]</th>
<th>Al₂O₃ [%]</th>
<th>SiO₂ [%]</th>
<th>P₂O₅ [%]</th>
<th>SO₃ [%]</th>
<th>K₂O [%]</th>
<th>CaO [%]</th>
<th>Mn₃O₄ [%]</th>
<th>Fe₂O₃ [%]</th>
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</thead>
<tbody>
<tr>
<td>18:35:36</td>
<td>0.141</td>
<td>2.188</td>
<td>6.300</td>
<td>22.72</td>
<td>0.121</td>
<td>4.075</td>
<td>1.021</td>
<td>60.80</td>
<td>0.130</td>
<td>2.248</td>
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<tr>
<td>18:40:41</td>
<td>0.144</td>
<td>2.189</td>
<td>6.320</td>
<td>22.73</td>
<td>0.120</td>
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<td>6.290</td>
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<td>6.280</td>
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<td>4.088</td>
<td>1.024</td>
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<td>0.001</td>
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Repeatability – 30 Measurements on Same Sample, Day 3

<table>
<thead>
<tr>
<th>Time</th>
<th>Na₂O [%]</th>
<th>MgO [%]</th>
<th>Al₂O₃ [%]</th>
<th>SiO₂ [%]</th>
<th>P₂O₅ [%]</th>
<th>SO₃ [%]</th>
<th>K₂O [%]</th>
<th>CaO [%]</th>
<th>Mn₃O₄ [%]</th>
<th>Fe₂O₃ [%]</th>
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<td>1.014</td>
<td>60.73</td>
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<td>0.121</td>
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<td>1.017</td>
<td>60.79</td>
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<td>2.240</td>
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<tr>
<td>Std.Dev.</td>
<td>0.001</td>
<td>0.009</td>
<td>0.018</td>
<td>0.02</td>
<td>0.001</td>
<td>0.007</td>
<td>0.002</td>
<td>0.04</td>
<td>0.001</td>
<td>0.007</td>
</tr>
<tr>
<td>RSD.</td>
<td>0.94</td>
<td>0.39</td>
<td>0.28</td>
<td>0.10</td>
<td>0.67</td>
<td>0.17</td>
<td>0.23</td>
<td>0.07</td>
<td>1.15</td>
<td>0.33</td>
</tr>
</tbody>
</table>
Critical Components for C-114 Qualification

- Stable Instrument
- 1 kW or 4 kW for 100% of elements listed
- Proper sample preparation
- Correction capability
- Stable drift correction standards
Blended Cements
Additives

- Portland cement clinker (K)
- Granulated blast furnace slag (S)
- Puzzolan, natural (P), natural calcined (Q)
- Flyash, siliceous (V), calcareous (W)
- Burnt shale (T)
- Silica fume (D)
- Limestone, higher (L), lower TOC content (LL)
- Minor compounds, gypsum phases

⇒ 27 different cement types
Please use your mouse to answer the question on your screen:

When you analyze a sample, are you aware of which additives are present in the cement?

- Always
- Sometimes
- Seldom
- Never
High Throughput

- Have machines perform processes which need repeated accurate operation, replacing “human” action
  - Dosage and weighing
  - Fusion
  - Sample grinding and/or pressing
  - Sample transport
  - Whole process?
Automated Flux or Additive Dispenser – Example: Claisse TheAnt

- Automatic flux dispenser
- Precision 0.1 mgr
- Sample/flux ratio
- Ethernet connection to analyzer (catch weight)
Claisse "rFusion"
Modular Automation concept

TheAnt
Weighing & Flux Dispenser

Robot

TheBee Fluxer

Storage

Mixer
Requirements for Pressed Pellets
Method

- Accurate dosage of additive as well as repeatable grinding time will eliminate human factor
- No contamination issue when automated pre-grind or cleaning cycle is used
- Pressed pellet will always use same pressure, rise and hold time
Automated Preparation

- Automated operation from material in to tablet out
- Usable for process
- Or centralized sample prep when no operators can be found to do work
- Compact and silent
- Easy setup and installation by user!
- High throughput and excellent repeatability
- Does not cost arm and leg...
APM – Automated Preparation Module
Lab Automation – APM All-in-One
Integrated Mill and Press

Material in

Tablet out
POLAB® APM
Automatic Sample Preparation Module

Sample changer and automated cleaning available
XRF and XRD Analysis on Same Sample...
One Interface Does It All
AXSLAB – Push-button Operation
Lab Automation – AXSLAB
Configuration: S4 + D4 + APM\textsuperscript{plus}

Transfer of results via LAN,
e.g. to plant control system, LIMS system, Blending Software
Conclusion I

- Sequential XRF instrumentation enables more sensitive and stable operation than ever before
- C-114 certification: Sample preparation is the most difficult part
  - Case A)
    - Matrix of standards can be matched to unknown samples, matrix of standards is close to samples - not too many additives are used and fairly constant
      - e.g. cement plant production for mill control
    - Pressed pellets can be used to fulfill both C-114 or EN norms
    - Bias in accuracy will be higher than fusion, but turnaround time will be less, as well as XRD can be performed on samples.
    - Standards and unknowns will need to be ground fine enough without segregation
    - Standards can be used only from INTYPE material
Conclusion II

• Case B)
  o Matrix of standards cannot be matched to unknown samples
    *e.g.* samples might contain additives of all sorts, wide range of
different cements and products need to be measured on a curve,
accuracy is paramount.
  - *e.g.* Commercial laboratories, DOT’s and DOR’s, engineering
    companies, mining and drilling supply companies
  o Fusion preparation is the way to go when no XRD is needed and
    C-114 needs to be met with one calibration
  o All available standards and synthetic standards can be used

■ Fusion
  • Allows synthetic reference material
  • Raw material mixes, especially dry process ones, can be fused
    with same method, allowing accurate numbers for the raw mix
    control
Sample preparation is the limiting factor in the performance of the analysis.

- The more steps that can be automated and done by machines, the less variability and error is introduced.

- Using a combined press and mill reduces the variation in pellet preparation dramatically and relieves operators from the tediousness of preparation by hand. Interfacing it to further enhance throughput with a system is easy!

- Prep as pellets is fully compatible with combined XRF and full XRD analysis. The combined automated prep also yields better results in gypsum and alite when we look at XRD.
Thank you for attending!

Please type any questions you may have in the Q&A panel.

Copies of the Cement Solutions XRF and XRD presentations and related resource materials will be emailed to you.
Visit us at:

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IEEE-IAS/PCA 2008
Miami, Florida, USA

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XRF 2008 & Symposium 2008
London, Ontario, Canada

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Denver, Colorado, USA

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