SEM / XRD Characterization of Cement-Based Materials

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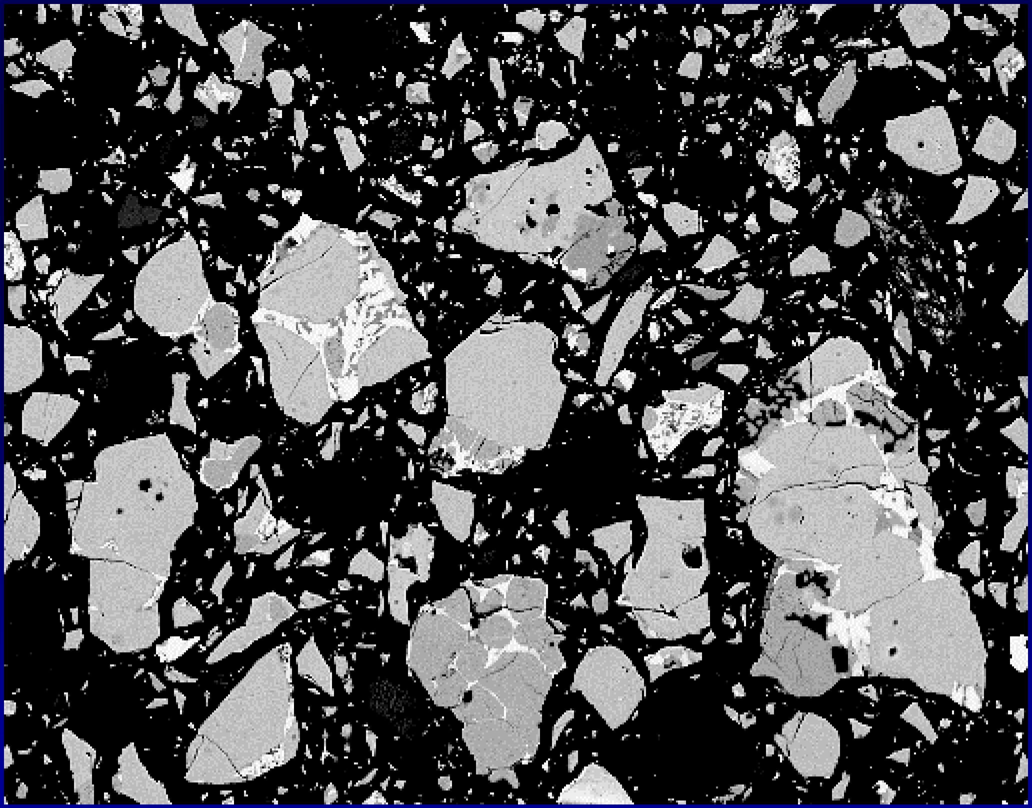
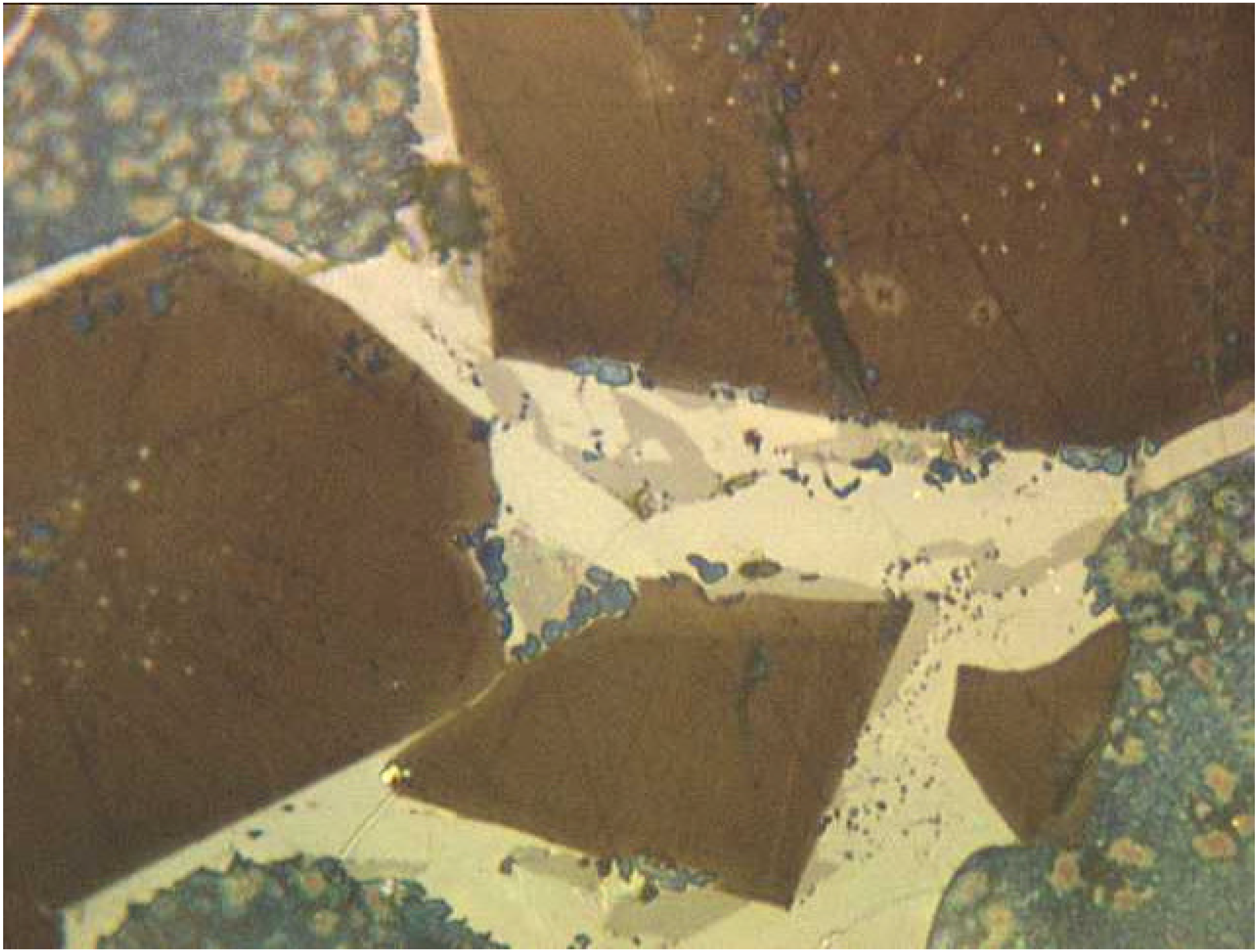
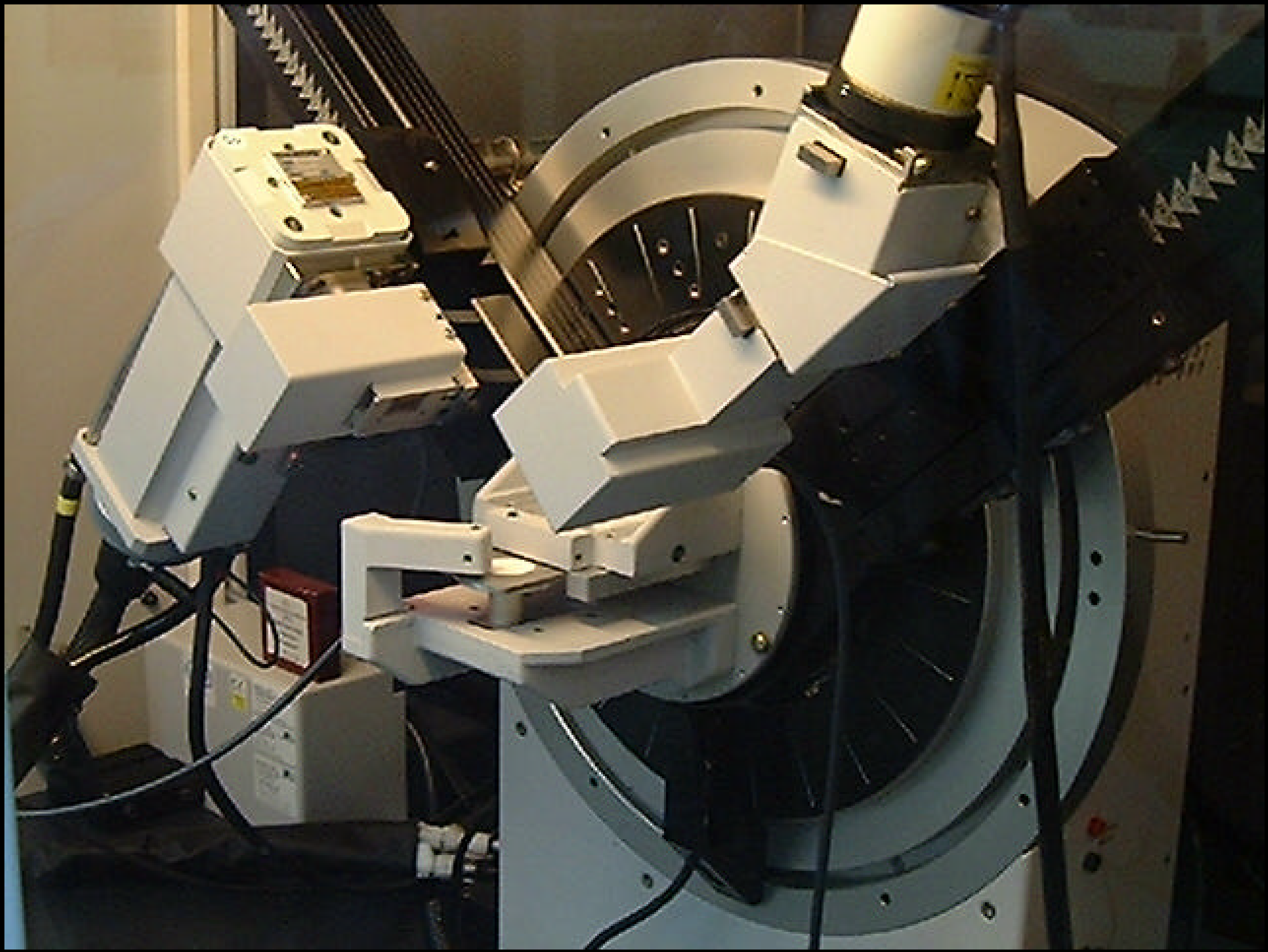




X-ray powder diffraction

Diffraction patterns of cementitious materials provide phase, chemical, and crystal structure information data that will be needed to aid understanding of cement performance and to aid selection of the optimum cements for use in HPC.



X-Ray Diffraction Analysis

•Analysis of portland cement is difficult as the large number of phases results in substantial peak overlap.

•diffraction patterns for each phase are unique,

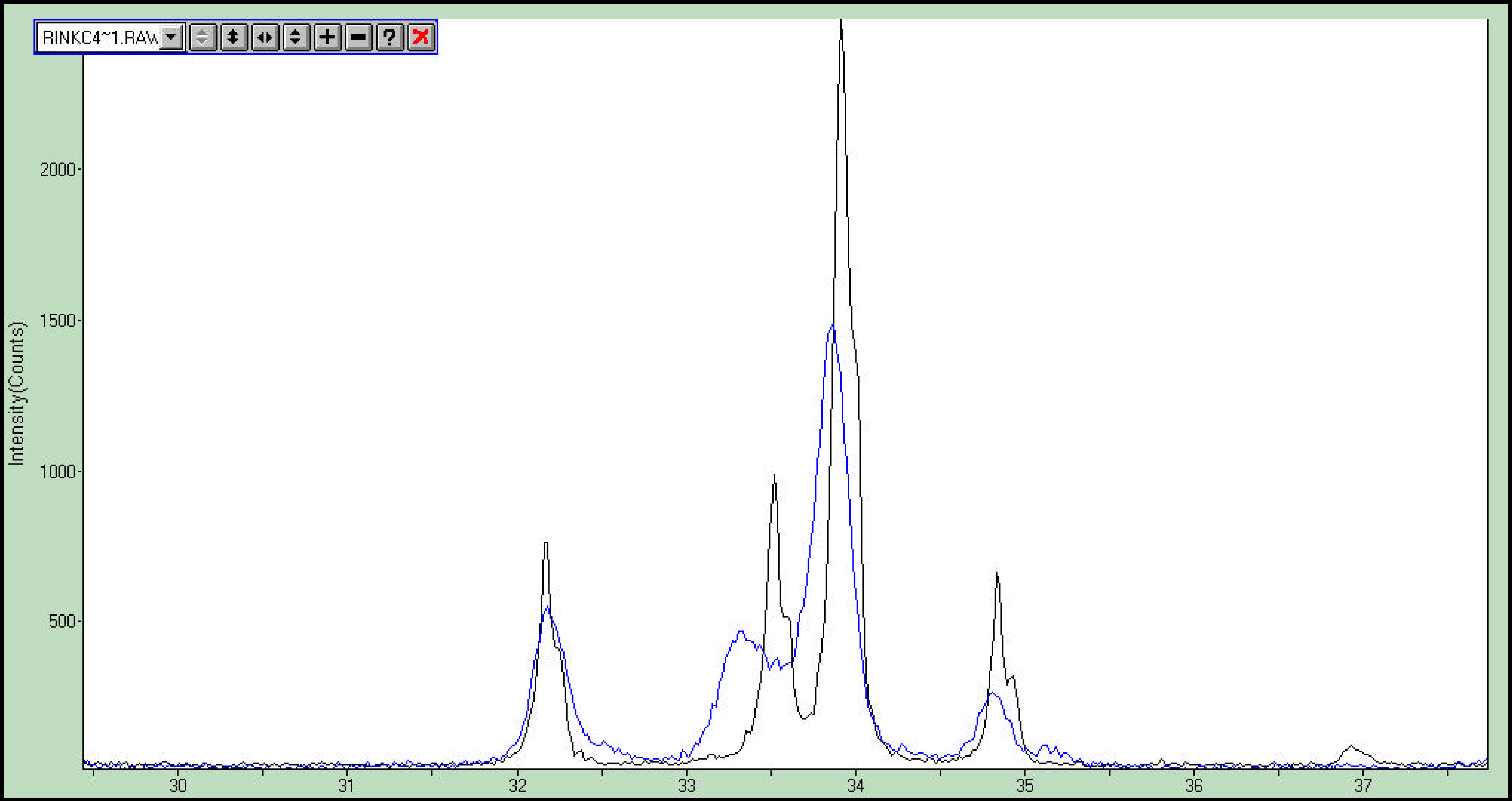
•the patterns are produced independently of others, and

•in a mixture, their intensities are proportional to phase concentration.

•Amorphous phases may be identified through use of an internal standard.

These problems are being addressed using the Rietveld method and the General Structure Analysis System (GSAS).

Laboratory-prepared Brownmillerite (black trace) vs. ferrite phase extracted from an industrial clinker (blue trace)



The Rietveld method allows standardization of powder diffraction analysis through use of calculated reference diffraction patterns based upon crystal structure models

- **Whole pattern-fitting utilizes all the diffraction pattern data,**

- **Reference models optimized for each material representing the best-fit for each phase,**

- **Compositional and structural variations may be accounted for relative to the idealized structure models in the database,**

- **Pattern scales are used in calculation of phase abundances**

Crystal Structure Database for Powder Diffraction

Phase: Brownmillerite (C_4AF)
Z: 4
Vol: 438.12 Å^3

Formula: Ca_2FeAlO_5
Space Group: Orthorhombic Ibm2
Mass, Formula Unit: 242.99

ICDD: 30-226

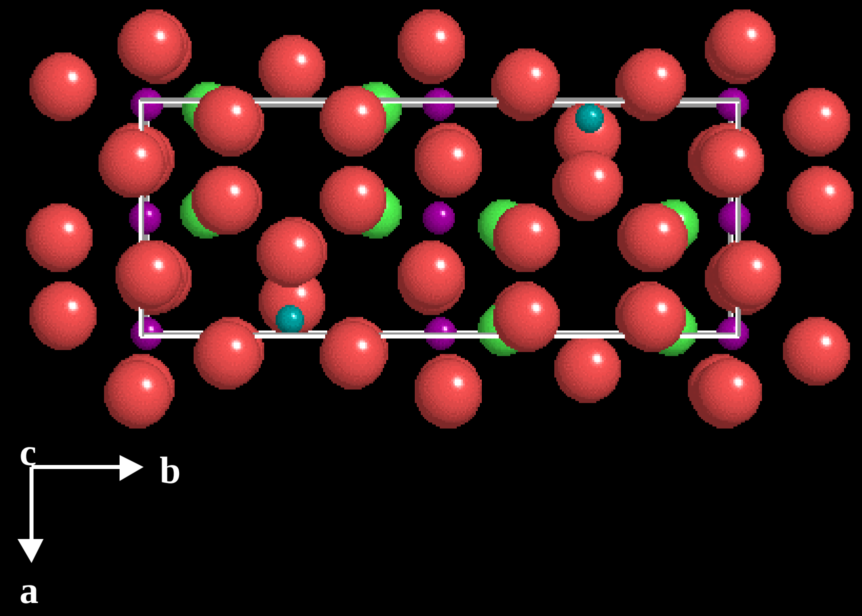
Cell Parameters (Å)

a	b	c
5.584	14.60	5.374

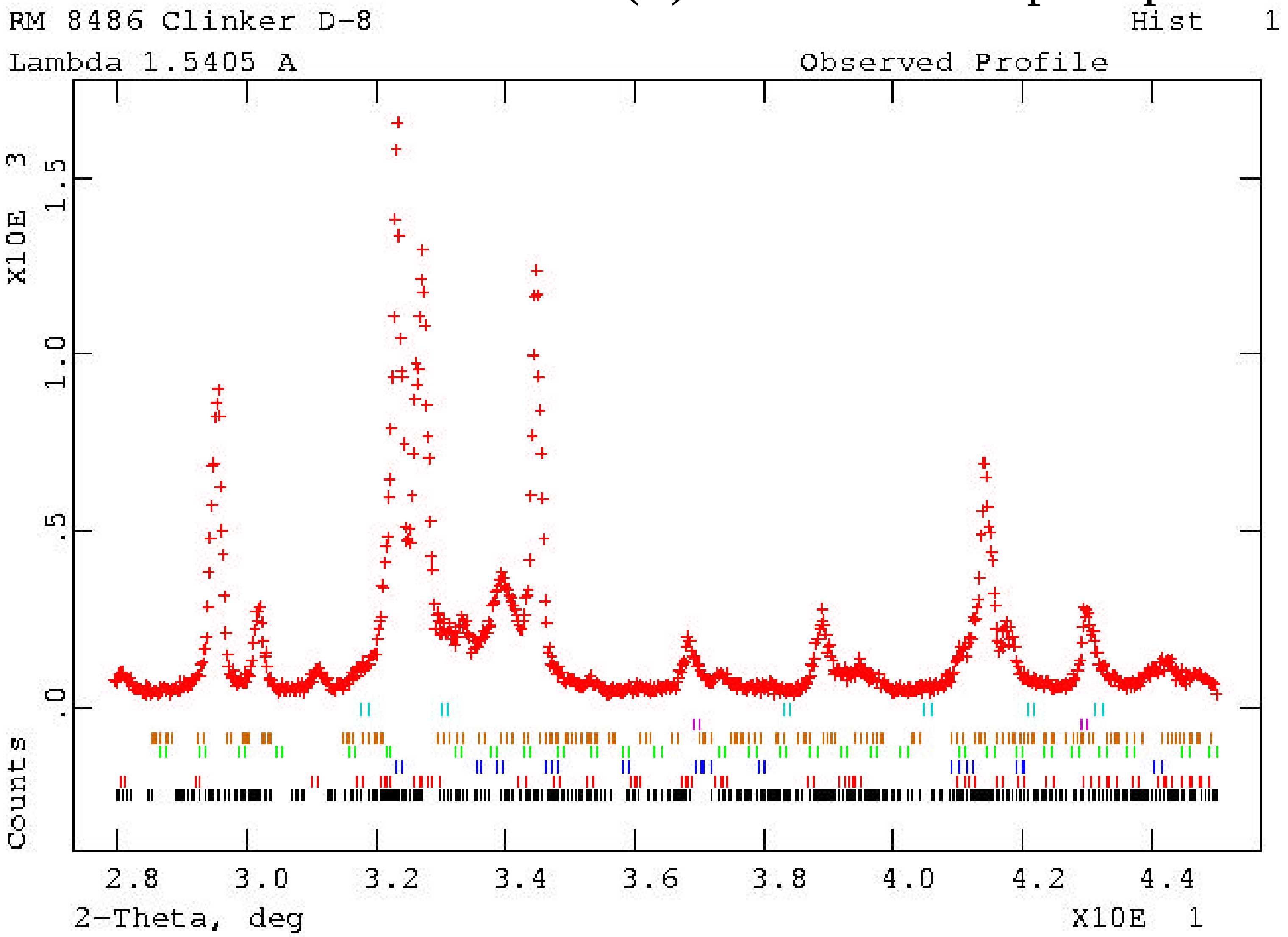
Atomic Parameters

	x	y	z	Occupancy
Ca	0.0273	0.1087	0.4920	1.00
Fe	0.0	0.0	0.0	0.76
Al	0.0	0.0	0.0	0.24
Al	0.9283	0.2500	0.9533	0.76
Fe	0.9283	0.2500	0.9533	0.24
O	0.2523	0.9861	0.2491	1.00
O	0.0680	0.1439	0.0246	1.00
O	0.8607	0.2500	0.6193	1.00

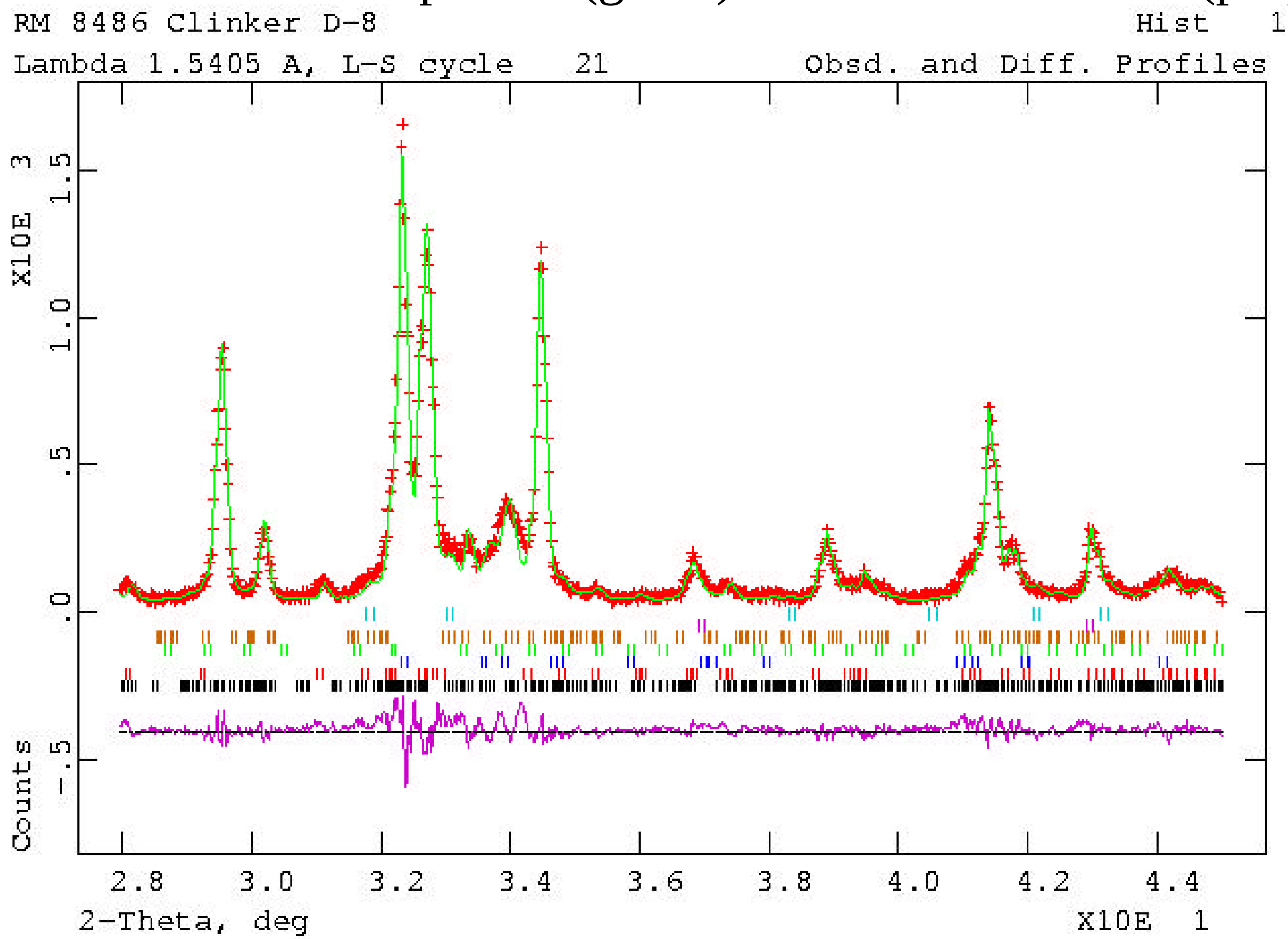
Typical composition: Taylor '97 $Ca_2AlFe_{0.6}Mg_{0.2}Si_{0.15}Ti_{0.05}O_5$



NIST Clinker 8486: raw data (+) and calculated peak positions



Calculated diffraction pattern (green) and difference curve (purple)



Simultaneous refinement of X-ray diffraction patterns of multiple phases allows quantitative analysis using the following relationship.

$$W_p = (S_p\ (ZMV)_p)\ /\ (\text{Ó}_i[S_i\ (ZMV)_i])$$

Where

W_p - the mass fraction of phase p

S - the Rietveld scale factor

Z - the number of formula units per unit cell

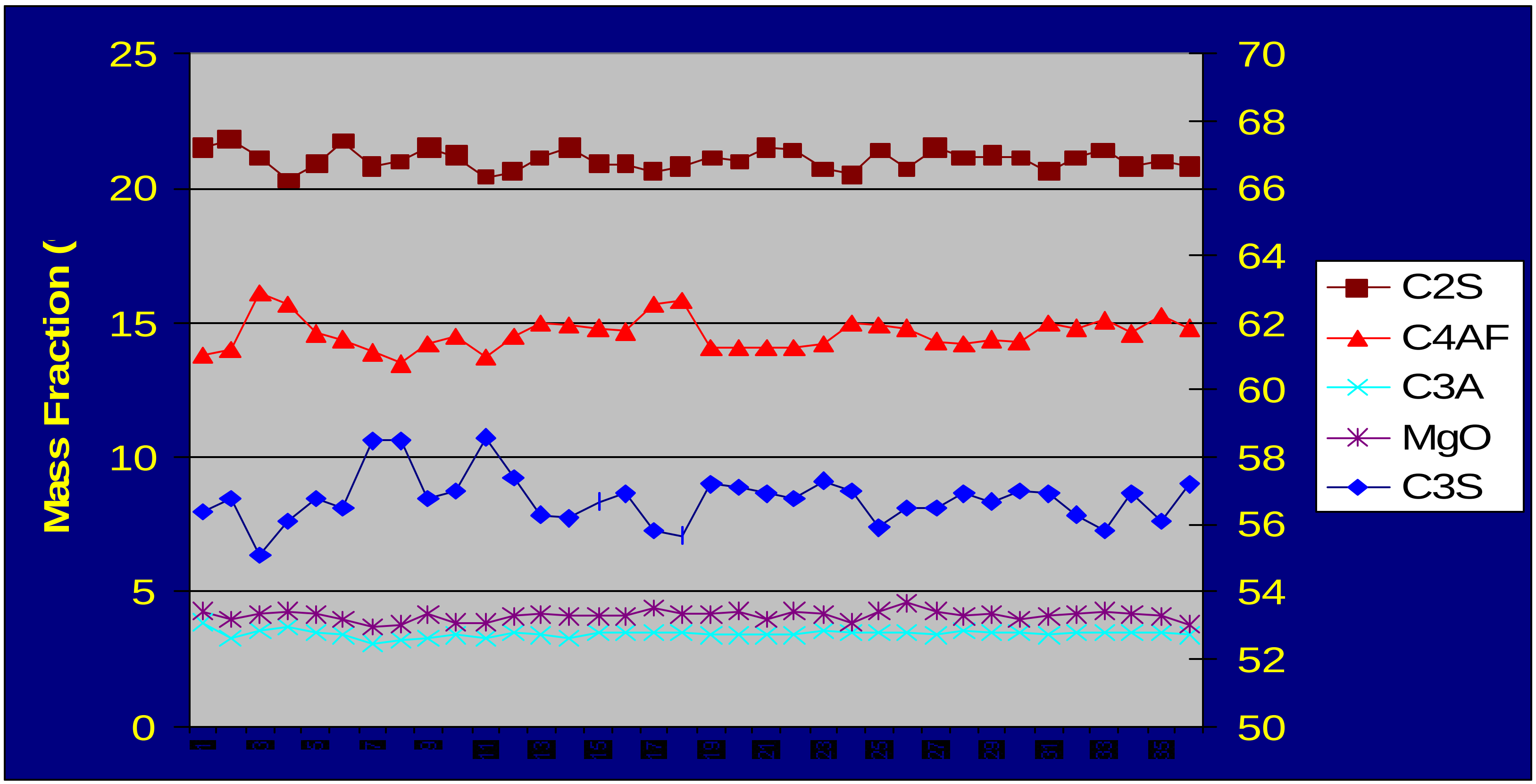
M - the mass of the formula unit

V - the unit cell volume

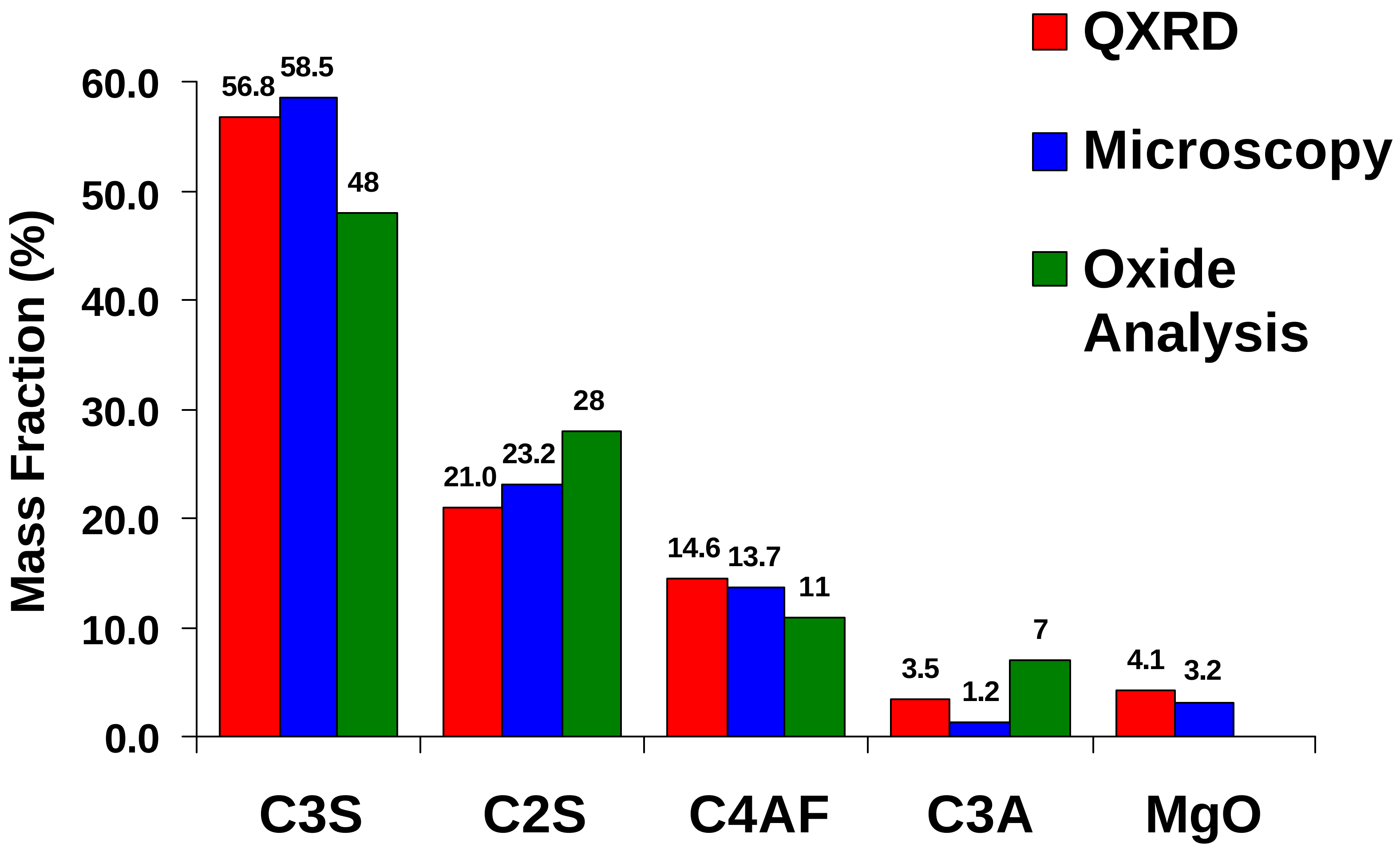
Note that microabsorption corrections are necessary for clinker and cements.

Certification of NIST Reference Clinker 8486

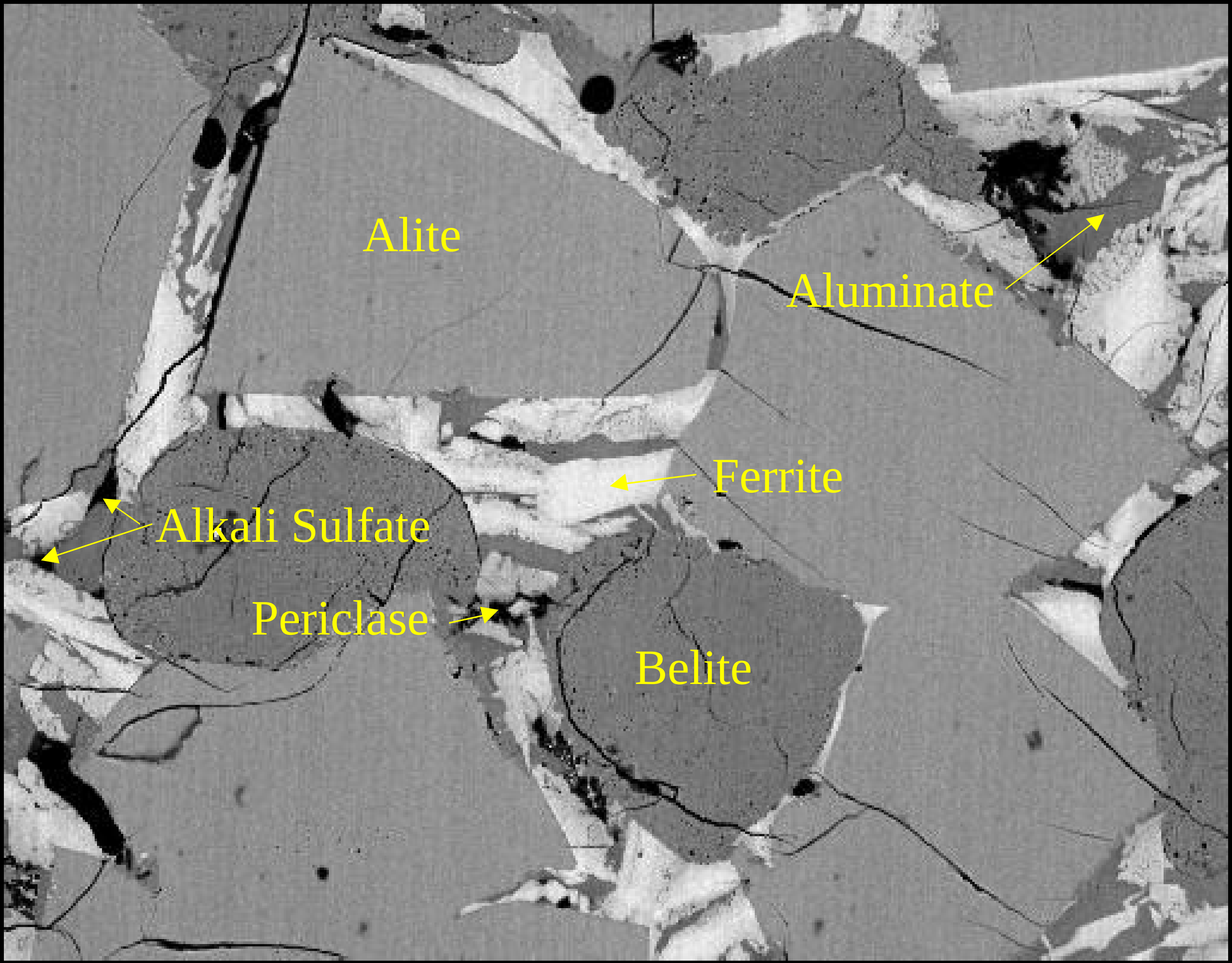
	Lower	Limits Mean	Upper
Alite	56.50	56.75	57.00
Belite	20.91	21.04	21.17
Aluminate	3.41	3.46	3.50
Ferrite	14.40	14.61	14.81
Periclase	4.07	4.13	4.20



Phase Analysis of RM 8486 by QXRD, Microscopy, and Calculation from Oxide Analysis



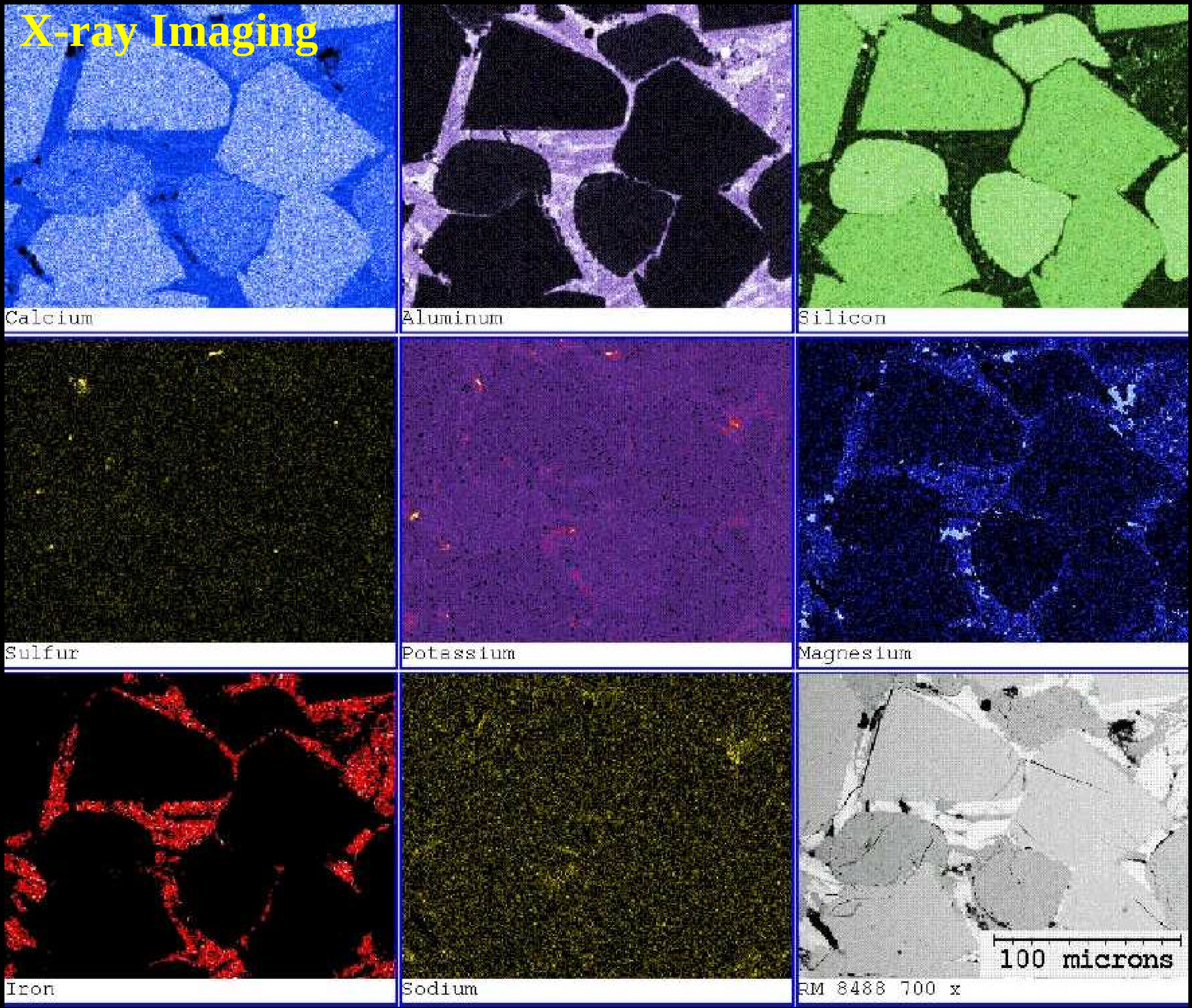
Scanning Electron Microscopy:
Imaging cement and cementitious materials microstructure to characterize phase and chemical spatial distribution.

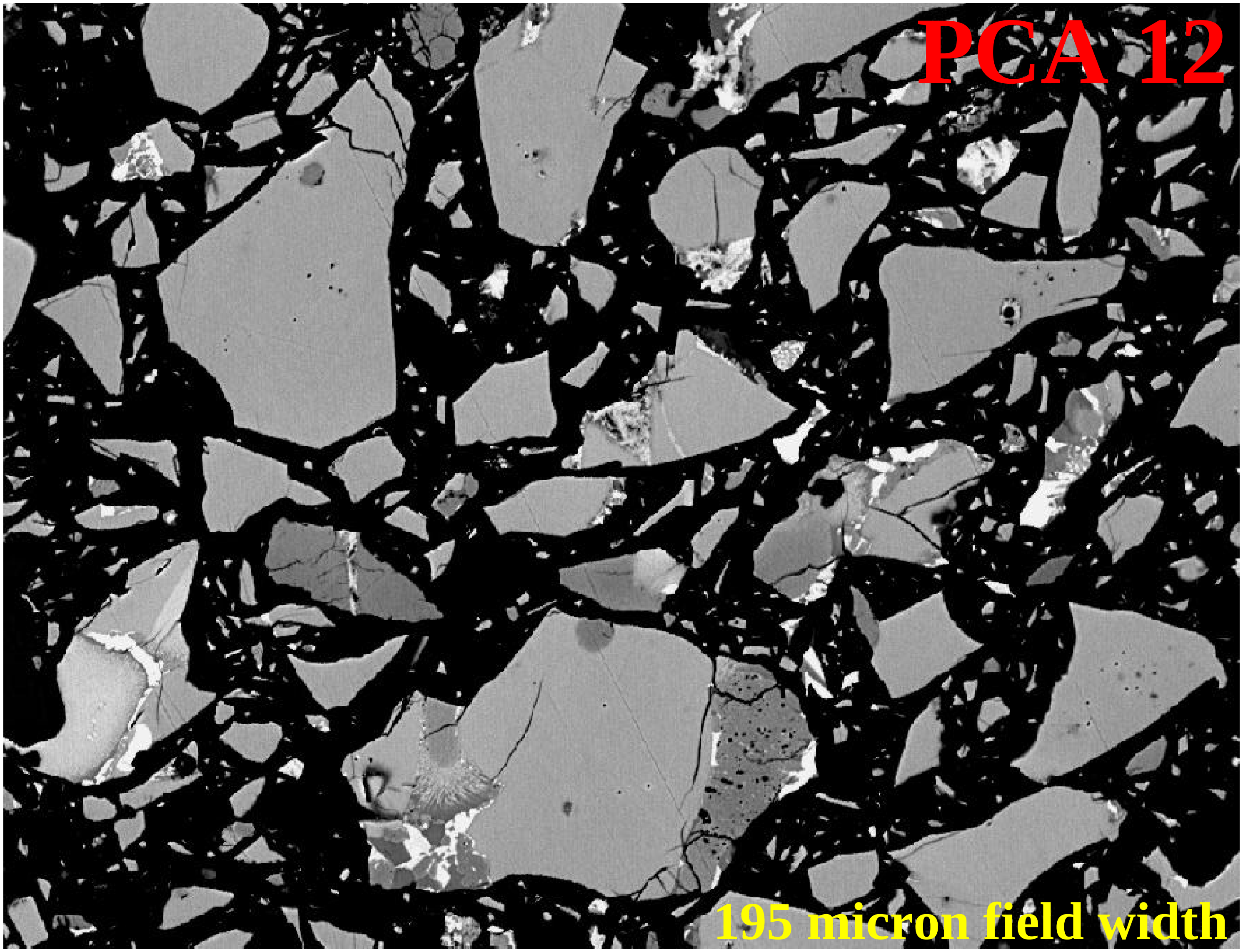


NIST RM clinker 8488

200 micron field width

X-ray Imaging



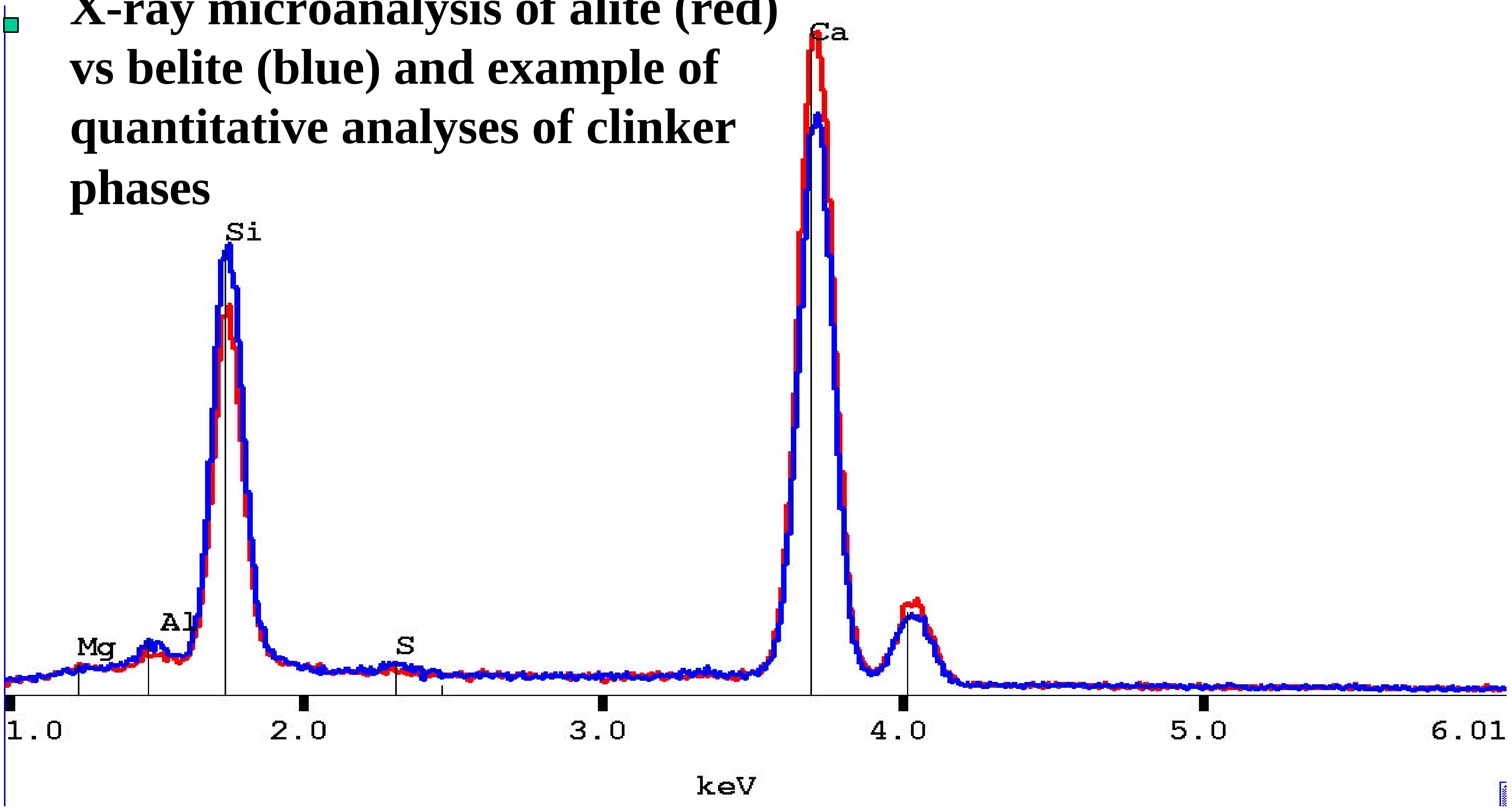


Na_2O	0.16
MgO	4.21
Al_2O_3	4.39
SiO_2	20.40
P_2O_5	0.08
SO_3	2.76
K_2O	0.48
CaO	63.60
TiO_2	0.44
Mn_2O_3	0.09
Fe_2O_3	2.64
V_2O_5	0.01
Cr_2O_3	0.02
ZnO	0.04
SrO	0.04
ZrO_2	0.02
BaO	0.07

Microscopical imaging of cement microstructure to help relate composition and texture to performance properties

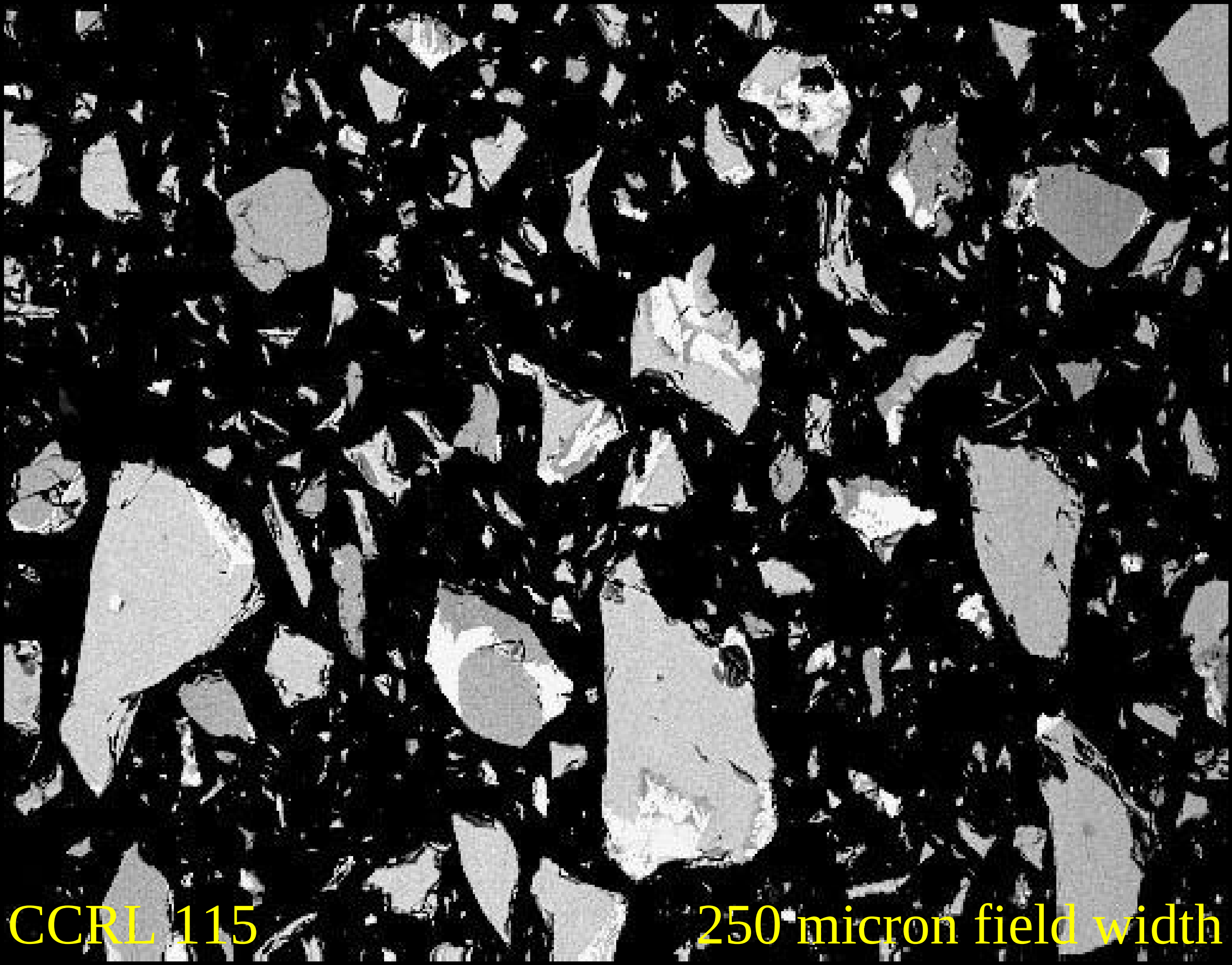
7.9% C_3A

X-ray microanalysis of alite (red) vs belite (blue) and example of quantitative analyses of clinker phases

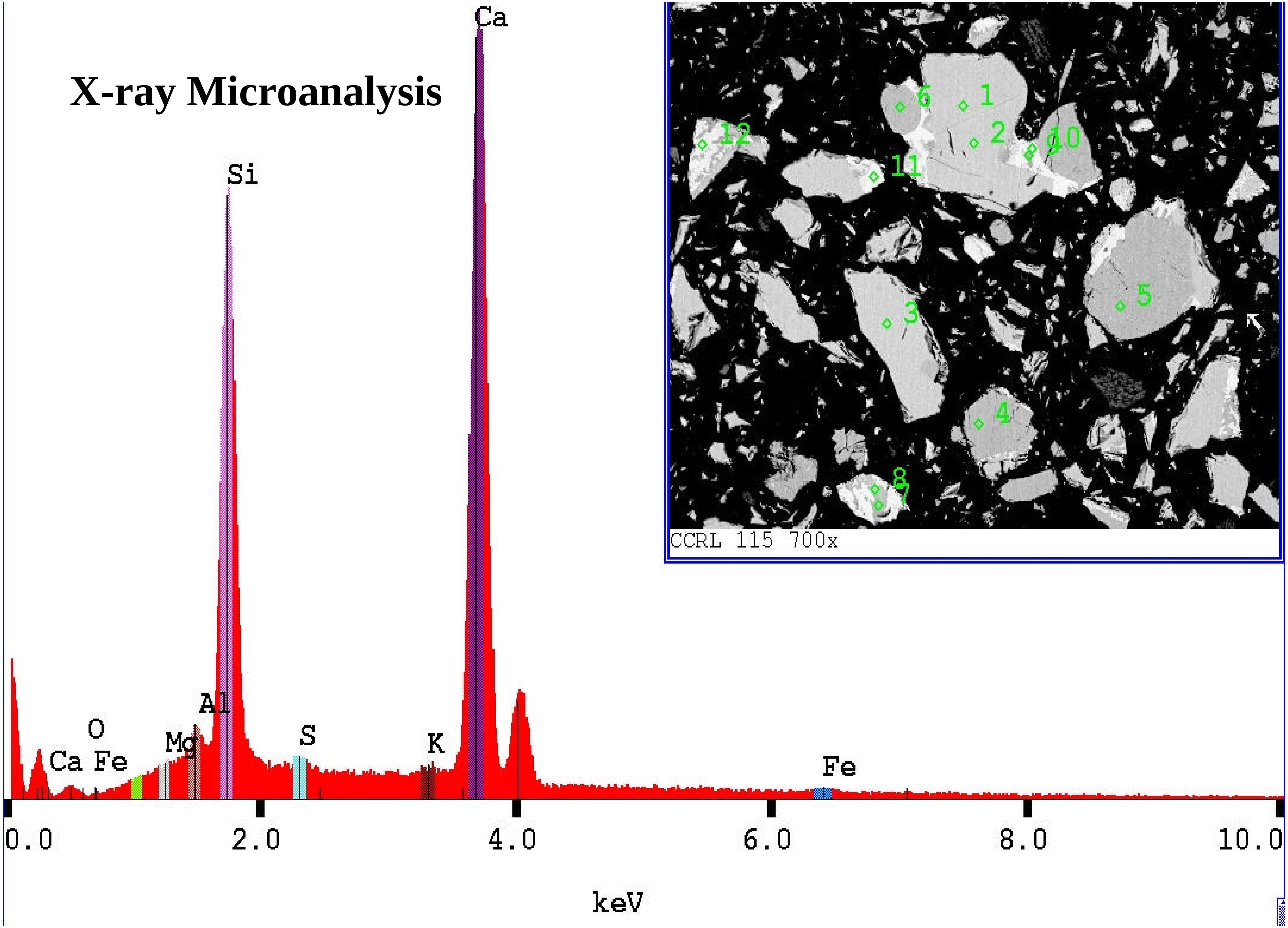


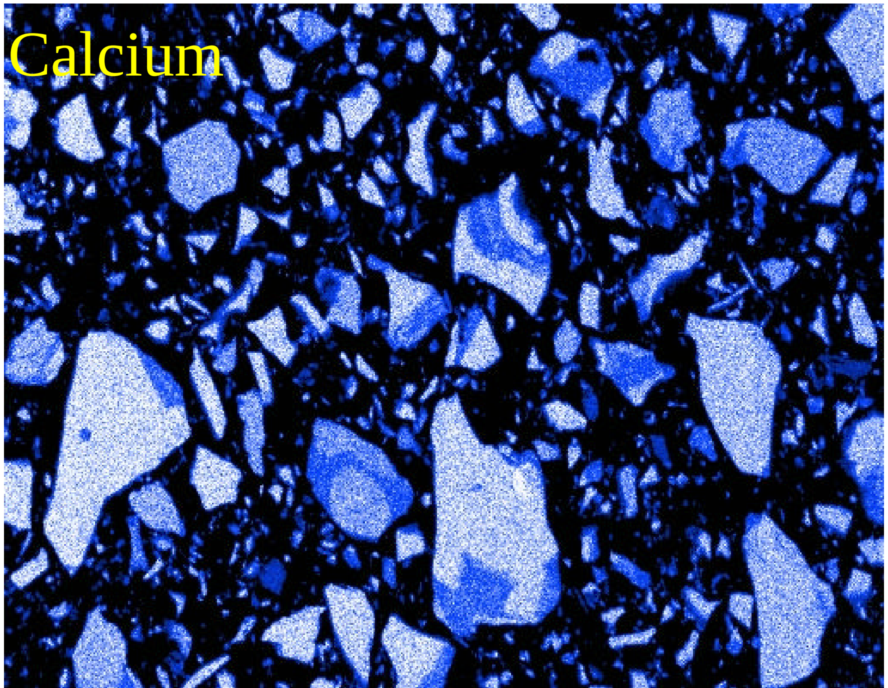
	CaO	SiO_2	Al_2O_3	Fe_2O_3	MgO	K_2O	Na_2O	TiO_2	Mn_2O_3
C3S	72.6	25.1	0.7	0.0	1.6	0.0	0.0	0.0	0.0
C2S	64.6	31.8	1.0	1.0	0.5	0.7	0.0	0.0	0.0
C3A	57.7	4.3	31.7	3.6	0.3	1.6	0.8	0.0	0.0
C4AF	49.2	4.1	20.4	21.6	2.6	0.0	0.0	1.0	1.1

Backscattered Electron Imaging of Polished Sections



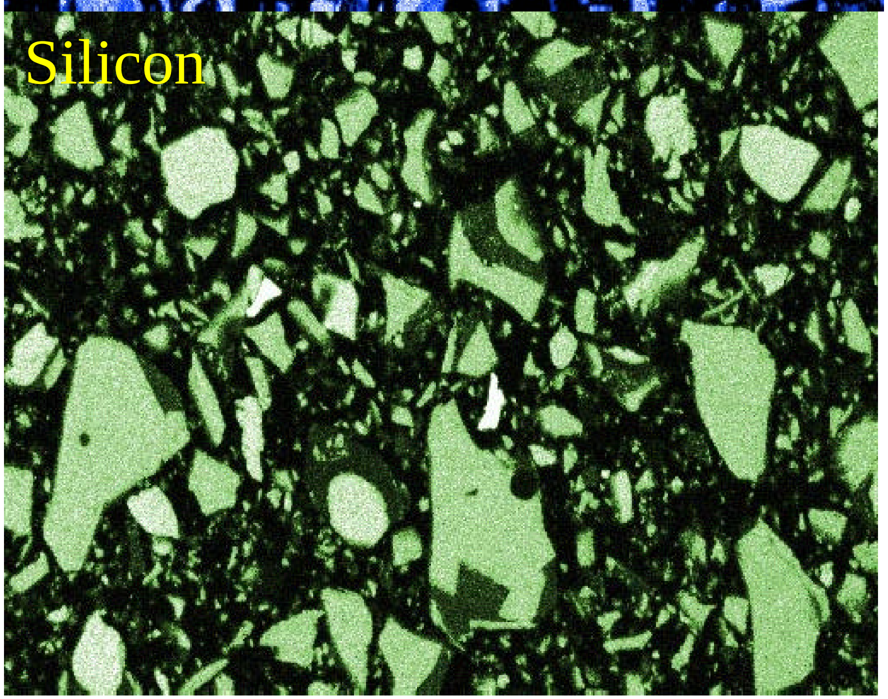
X-ray Microanalysis

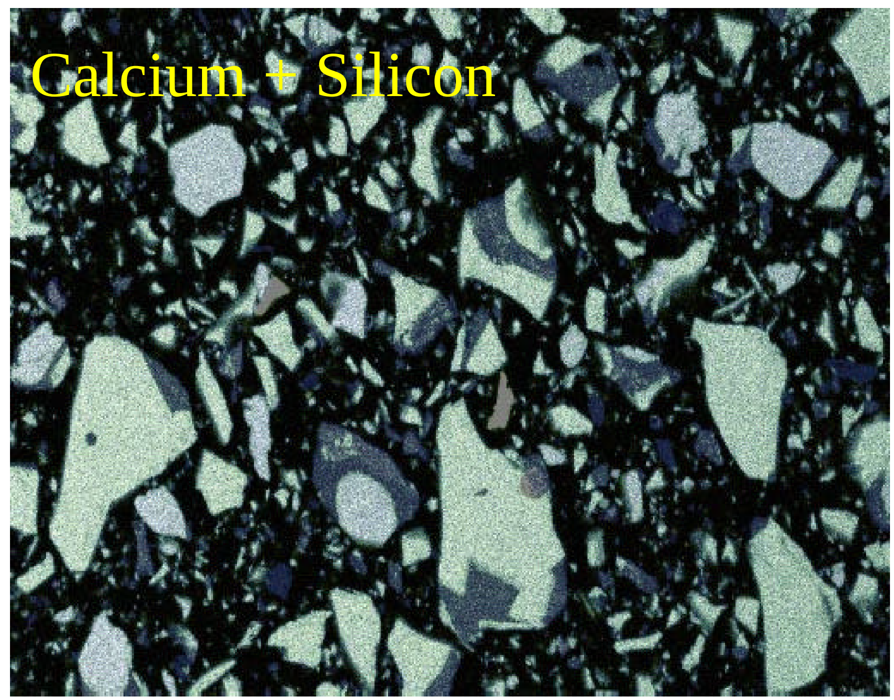


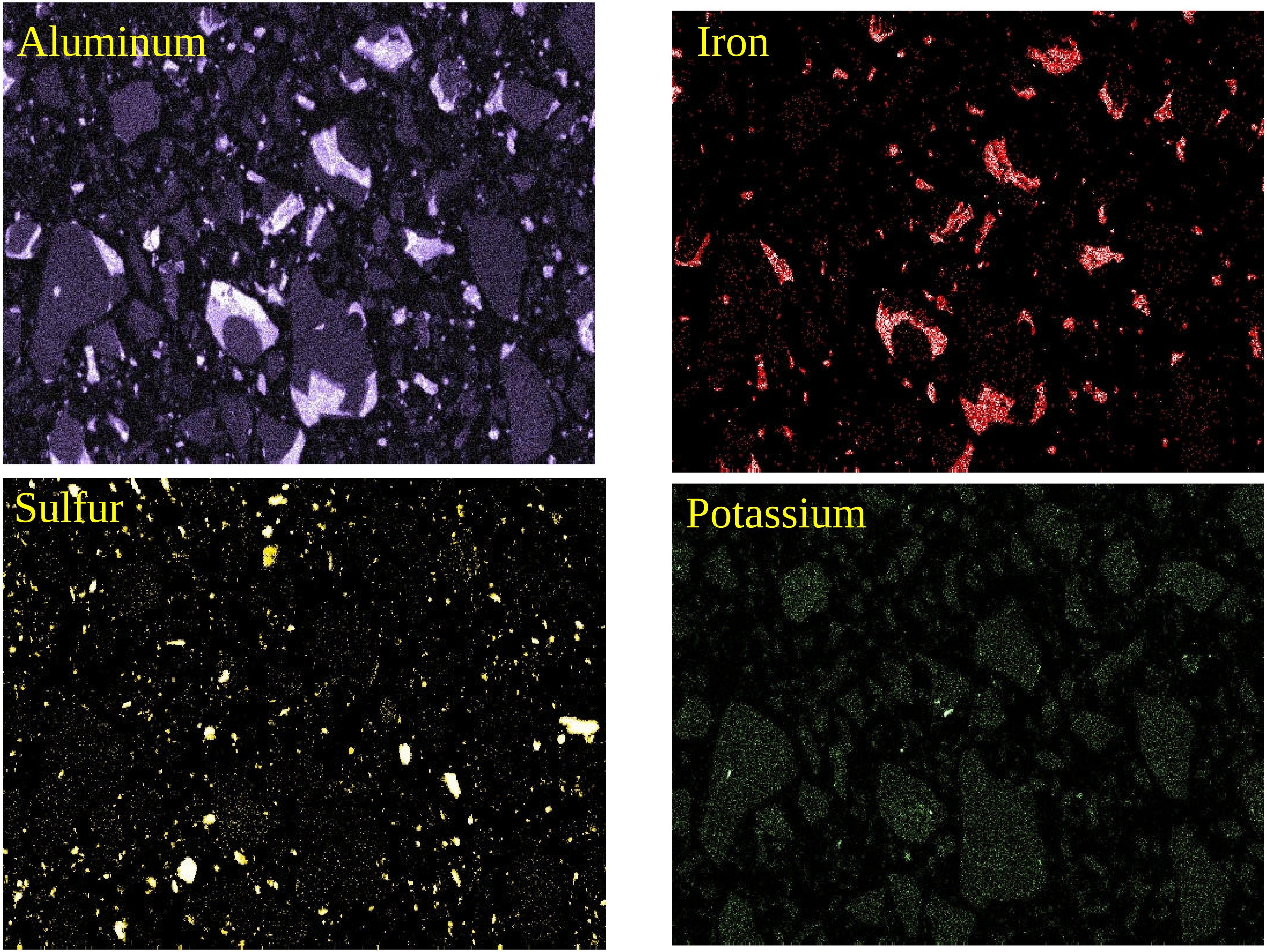


X-Ray Imaging

•to define spatial distribution of selected elements, and
•to provide a set of images to allow identification of the constituents






Aluminum
Iron
Sulfur
Potassium

Composite image with color-coded phases

Alite
Belite
Aluminate
Ferrite
Periclase
Gypsum