Institute of Geologists of Ireland

Pyrite Course

I.S. 398-1: What's in it and the role of the Geologist

Michael L.J. Maher

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Responsibilities of Geologist

- You're only the messenger!
- Classification of infill not an exact science
- Ensure decisions are independent and free of bias
- Err on the side of caution
- Lithology is key to sample classification
- Adhere to the standard, but additional testing may be needed
- Document all observations
- Remember hardcore placed without control – random variations are possible

Beware of Mixed Hardcore!

Objective is Categorisation

- **Category A – Negligible Risk**
  - DCR=0, Hardcore not susceptible
  - DCR= 1 or 2, Hardcore not susceptible and alternative cause for damage
- **Category B – At Risk**
  - DCR=0 or 1 (w/o progression), Hardcore susceptible to limited expansion → Low Potential
- **Category C – At Risk**
  - DCR=0 or 1 (w/o progression), Hardcore susceptible to significant expansion → Significant Potential
- **Category D – Significant Pyritic Damage**
  - DCR=1 (w progression) or 2, Hardcore susceptible to significant or limited expansion.

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Concepts behind IS 398.1

- Rigorous process
- Consider history and location
- Damage Condition Rating is critical to classification
- Susceptible hardcore on its own not sufficient for a Red Cert – must have confirmed damage
- Minimal testing where no damage and non-susceptible aggregate type
- Minimal testing where severe damage and highly susceptible aggregate type
- More extensive testing where aggregate susceptibility in 'grey' area
- Must have at least two samples for Green Cert

Test Suites (Table 1, IS 398-1)

Test Suite	Tests
Test Suite 0	i) Geological inspection, ii) Chemical testing.
Test Suite 1	i) Geological inspection, ii) Chemical testing, iii) Mineralogical analysis by quantitative X-ray Diffraction (XRD).
Test Suite 2 **(Performed following Test Suite 0 or Test Suite 1)**	i) Thin section petrographic analysis, ii) Water Absorption.

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Test Suite 0 – Quick Route to Green

Test Parameter	Pass
Acid-soluble sulfate (AS)	≤ 0,2 %
Water-soluble sulfate (WS)	≤ 500 mg/L SO_4
Total sulfur (TS)	≤ 0,3 % S
Proportion fine grained sedimentary rock (muddy limestone, calcareous mudstone and shale)*	≤ 10 %
Evidence of secondary crystallisation (including gypsum precipitate)*	N

* See I.S. EN ISO 14689-1 (Table A1) for further information.

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Test suite 1 – Quick Route to Red

Test Parameter	Pass	Fail
Acid-soluble sulfate (AS)	≤ 0,2 % SO_4	> 0,2 % SO_4
Water-soluble sulfate (WS)	≤ 500 mg/L SO_4	> 1500 mg/L SO_4
Presence of Gypsum (from XRD)	N	Y
Total sulfur (TS)	≤ 0,3 % S	> 1,0 %S
Proportion fine grained sedimentary rock (muddy limestone, calcareous mudstone and shale)	≤ 10 %	> 30 %
Evidence of secondary crystallisation (including gypsum precipitate)*	N	Y

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Interpretation of hardcore results for Test Suite 2

Factor	Derived from	When assessing the susceptibility for expansion consider:
Pyrite content	Chemical Tests and calculations Petrographic examination	Presence and distribution
Presence of pyrite framboids	Petrographic examination	Form and distribution
Evidence of oxidised pyrite	Petrographic examination	Extent of oxidised pyrite confirms chemical activity within aggregate
Presence of clay minerals	XRD Petrographic examination	The higher the clay content, then the higher the susceptibility to pyrite-induced heave
Presence of calcite (as a source of calcium)	XRD Petrographic examination	Presence of calcite confirmed.
Presence of gypsum growth	XRD Petrographic examination Geological Inspection	Primary or secondary Extent, distribution and form of gypsum. Whether it is in the form of coatings as well as infill in fractures. Presence of clusters of gypsum rosettes.
Presence of mudstone and other fine grained sedimentary rock (muddy limestone, calcareous mudstone and shale) in bulk sample.	Geological Inspection	Proportion
Structure of aggregate particles	Geological Inspection Petrographic Examination	Particles are friable, extent of open laminations and fractures within particles.
Porosity of rock	Petrographic Examination EN 1097-6	Water absorption > 2,0 %
Moisture in sample	Geological Inspection Hardcore sample record	Sample is damp.
Presence of sulfur-bearing minerals	XRD Petrographic Examination	Presence of other sulfur – bearing minerals may decrease the total sulfur present as pyrite.

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Categorisation

		Damage Condition Rating from Building Condition Assessment			
		0	**1 without progression**	**1 with progression**	**2**
Professional Geologist's Classification of Hardcore	Pass (Not susceptible to expansion)	Category A	Category A*	Category A*	Category A*
	Inconclusive (Susceptible to limited expansion)	Category B	Category B	Category D*	Category D*
	Fail (Susceptible to significant expansion)	Category C	Category C	Category D	Category D

* In these cases the Engineer shall consider alternative probable causes for the damage other than pyritic heave.

Case Study #1

Evidence of Crystallisation

Evidence of Crystallisation

Evidence of Crystallisation

Evidence of Crystallisation

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Evidence of Crystallisation

Evidence of Crystallisation

Evidence of Crystallisation

Lithological Description

Lithology 1 (95%):

Dark grey to black, calcareous *siltstone/mudstone*. The particles are platy, moderately weak, laminated and sub-angular. The particles show outer surface coatings consisting of fine material and recrystallisation deposits. The particles reacted with 10% HCl, indicating the presence of calcite. Gypsum crystals were observed on freshly opened laminations.

Lithology 2 (5%):

Dark grey, medium grey massive *limestone*, showing similar coatings to those described in lithology 1.

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Chemical Test Results

Sample No.	Total Sulphur (% S)	Acid Soluble Sulphate (% SO_4)	Water Soluble Sulphate (mg/L)
A	1.40	0.99	1700
B	1.40	2.00	1800

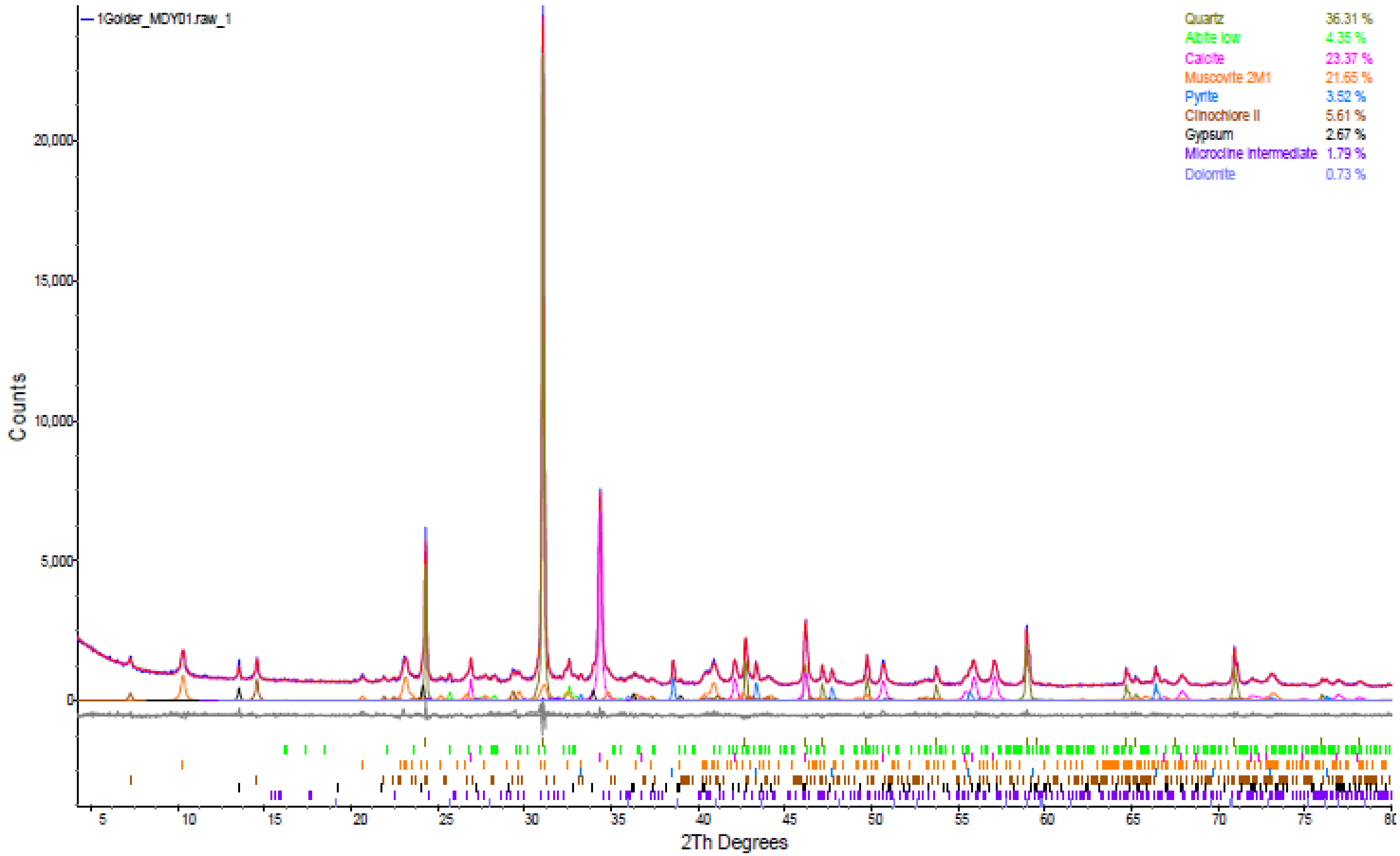
Sample No.	Sulphur present as Sulphate (% S)	Estimated Equivalent Pyrite Present (%)	Estimated Original % of Pyrite Present	Inferred % of Pyrite Already Oxidised
A	0.33	2.00	2.62	24
B	0.67	1.37	2.62	48

Physical Test Results

Sample No.	Water Absorption (%)	MicroDeval
IS EN 13242:2002* SR21:2004+A1:2007**	Max 2%	Not Specified
A	2.7	50
B	2.8	48

Not required under Suite 1

XRD



Gypsum: 2.67%

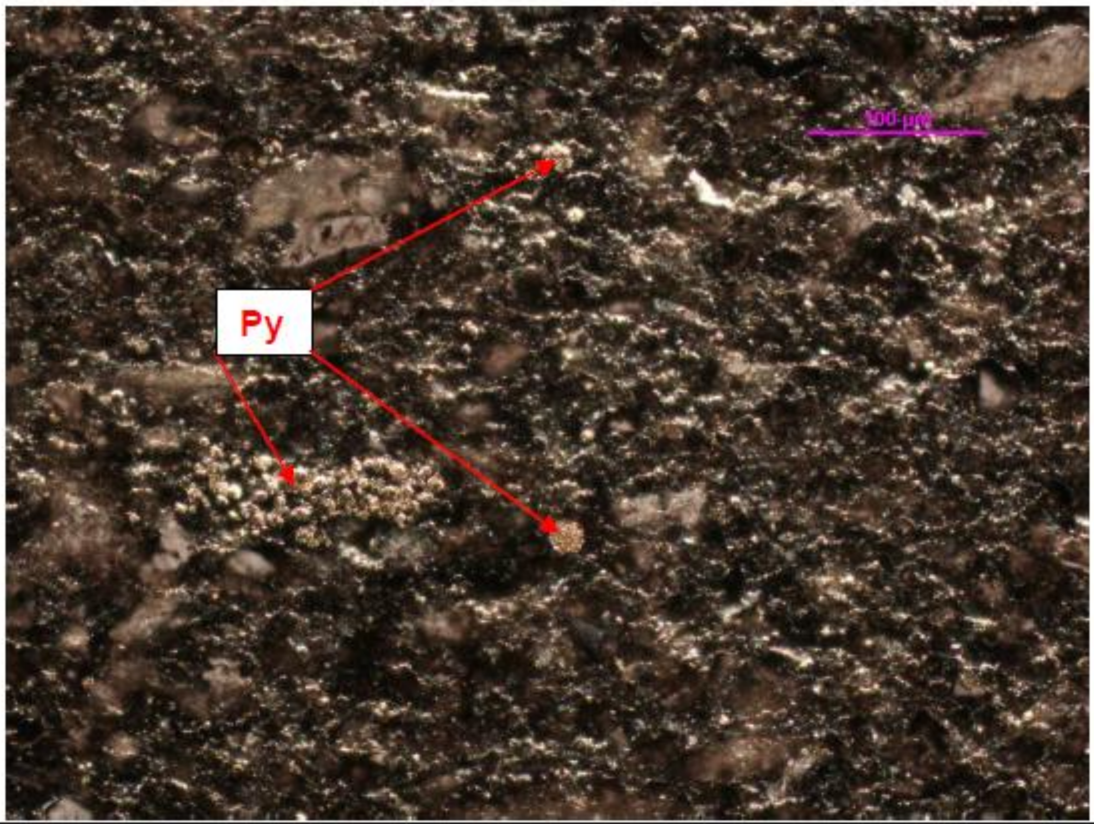
Interpretation

Test Parameter	Pass	Fail	Sample
Acid Soluble Sulfate AS	<0,2% SO_4	>0,2% SO_4	0.99%
Water Soluble Sulfate WS	<500 mg/L SO_4	>1500 mg/L SO_4	1700
Presence of Gypsum (from XRD)	N	Y	Y
Total Sulfur TS	<0,3% S	>1.0% S	1.4%
Proportion of fine grained sedimentary rock	<10%	>30%	95%
Evidence of secondary crystallization	N	Y	Y

Note: Consider worst of multiple samples for categorisation

Thin Section View

Photomicrograph 3														
	1	2	3	4	5	6	7	8	9	10	11	12	13	14
A														
B														
C														
D														
E														
F														



Category C or D

		Damage Condition Rating from Building Condition Assessment			
		0	1 without progression	1 with progression	2
Professional Geologist's Classification of Hardcore	Pass (Not susceptible to expansion)	Category A	Category A*	Category A*	Category A*
	Inconclusive (Susceptible to limited expansion)	Category B	Category B	Category D*	Category D*
	Fail (Susceptible to significant expansion)	Category C	Category C	Category D	Category D

* In these cases the Engineer shall consider alternative probable causes for the damage other than pyritic heave.

Case Study #2

Washed Samples

Sample D

Lithology 1 (85%):

Dark grey, fine grained *limestone*. The particles are sub-rounded to sub-angular, medium strong, fresh, massive and blocky to semi-blocky. They are stable and highly calcareous. No outer surface coatings or recrystallisation coatings or deposits seen.

Lithology 2 (15%):

Pale grey, fine to medium grained *limestone*. Some minor amounts of fossil fragments seen. The particles are strong, sub-angular to sub-rounded, semi-blocky and massive. They are stable, highly calcareous and fresh. No outer surface recrystallisation deposits are seen.

Chemical Test Results

Sample No.	Total Sulphur (% S)	Acid Soluble Sulphate (% SO_4)	Water Soluble Sulphate (mg/L)
C	0.04	0.03	24
D	0.04	0.06	140

Sulphur present as Sulphate (% S)	Estimated Equivalent Pyrite Present (%)	Estimated Original % of Pyrite Present
0.01	0.06	0.07
0.02	0.04	0.07

Interpretation

Test Parameter	Pass	Sample
Acid Soluble Sulfate AS	<0,2% SO_4	0.06%
Water Soluble Sulfate WS	<500 mg/L SO_4	140
Total Sulfur TS	<0,3% S	0.04%
Proportion of fine grained sedimentary rock	<10%	0%
Evidence of secondary crystallization	N	N

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Category A

		Damage Condition Rating from Building Condition Assessment			
		0	1 without progression	1 with progression	2
Professional Geologist's Classification of Hardcore	Pass (Not susceptible to expansion)	Category A	Category A*	Category A*	Category A*
	Inconclusive (Susceptible to limited expansion)	Category B	Category B	Category D*	Category D*
	Fail (Susceptible to significant expansion)	Category C	Category C	Category D	Category D

* In these cases the Engineer shall consider alternative probable causes for the damage other than pyritic heave.

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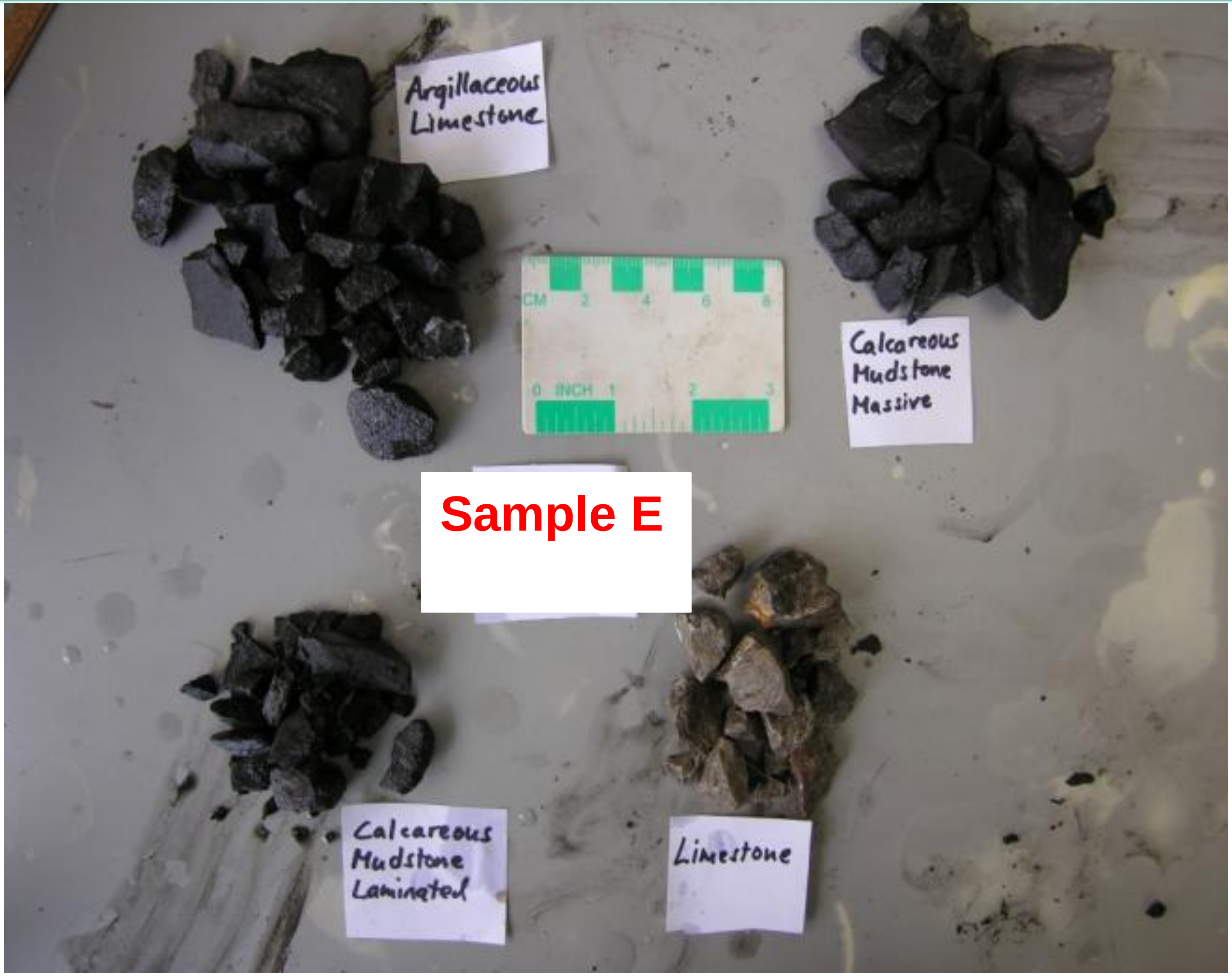
Case Study #3

Sorting of Sample



Lithological Descriptions

ROCK TYPE	% by mass
Mudstone 1: Very dark grey, calcareous, massive. Pyrite occurred as framboids 0.005 – 0.015 mm in size; anhedral grains that were typically finer than 0.002 mm were less common. Total pyrite was visually estimated to be 4-5% by volume.	41
Mudstone 2: Very dark grey, calcareous, clay- to silt-sized constituents, flat/elongated particle shapes, laminated, “slides” apart along laminations rather than breaks when struck with the geological hammer, gypsum observed on fracture surfaces. Pyrite was dominantly framboidal and anhedral and finer than 0.001 mm to 0.01 mm in size. Total pyrite was visually estimated to be 3-4% by volume in the examined thin section.	9
Argillaceous limestone: Dark grey, calcareous, silt- to clay-sized constituents. Laminae composed of argillaceous matrix, sometimes with a significant component of clay-sized carbonate, defined the structure of the rock. A fracture that may have contained gypsum was observed in one such lamina. Pyrite occurred primarily as framboids measuring 0.002 – 0.02 mm across; anhedral pyrite that was finer than 0.002 mm was less common. Total pyrite was visually estimated to 4-5% and 1-2% by volume in the examined thin sections.	35
Limestone: Light to medium brown, fossiliferous, some oxidation staining. The rock was composed of crystals of calcite and fossil fragments that were in the range of 0.1 – 0.8 mm. Trace amounts of hematite, rutile and pyrite were noted.	15
Total	100.0

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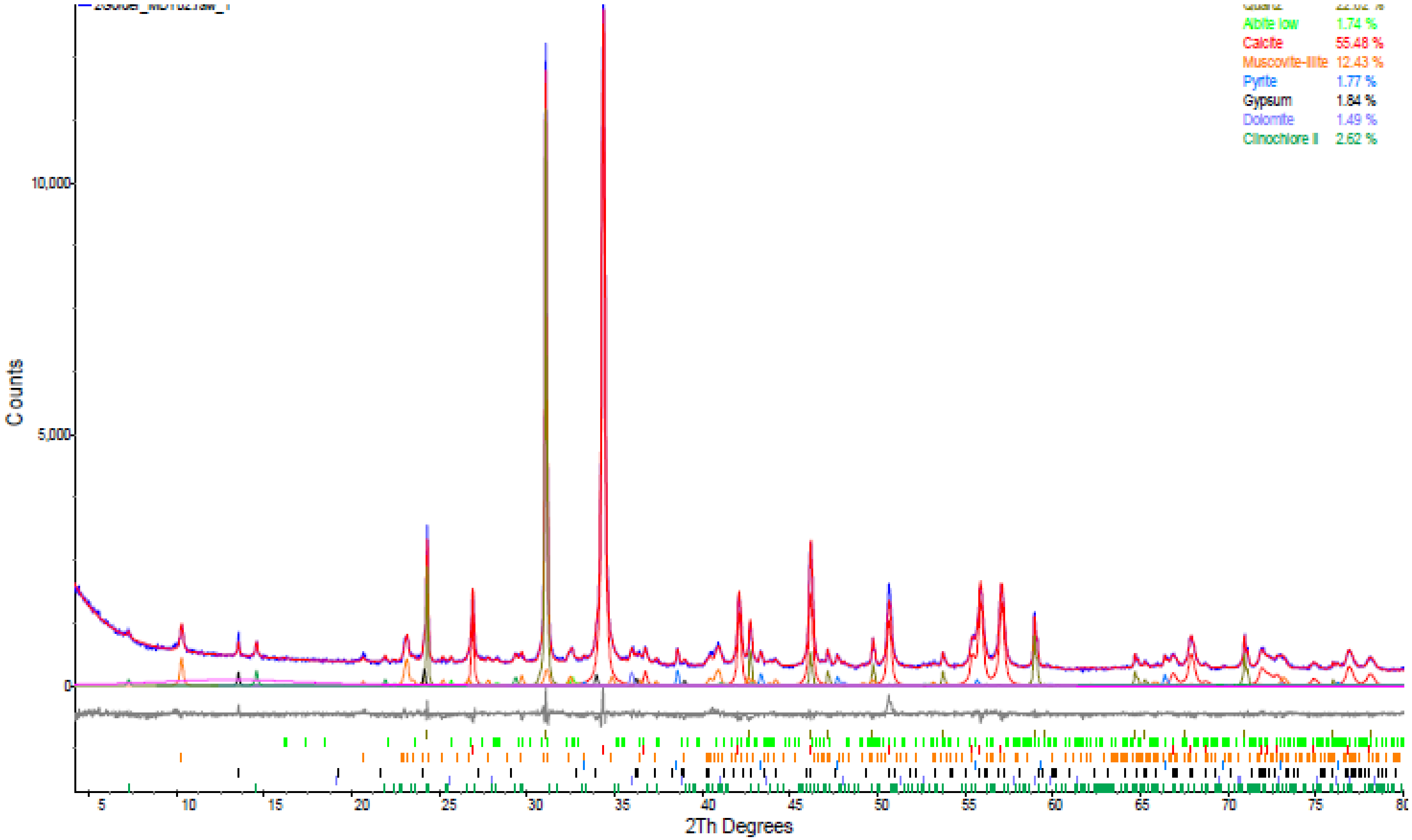
Chemical Test Results

Total Sulphur (%S)	Sulphur present as Sulphate (%SO_4)	Sulphur present as Sulphate (%S)	Sulphur Present as Sulphide (%)	Equivalent Pyrite Present (%)	Original % of Pyrite Present	% of Pyrite Already Oxidised	Water Soluble Sulphate (mg/L)
0.92	0.83	0.28	0.64	1.20	1.72	30	2600

XRD



Gypsum: 1.84%

XRD Analysis

Mineral	Ideal Formula		SAMPLE E
Quartz	SiO_2		23
Clinochlore	$(Mg,Fe^{2+})_5Al(Si_3Al)O_{10}(OH)_8$		3
Muscovite 2M-Illite	$KAl_2AlSi_3O_{10}(OH)_2$ /$K_{0.65}Al_{2.0}Al_{0.65}Si_{3.35}O_{10}(OH)_2$		12
Plagioclase	$NaAlSi_3O_8$ – $CaAl_2Si_2O_8$		2
Calcite	$CaCO_3$		56
Ankerite-Dolomite	$CaMg(CO_3)_2$		2
Gypsum	$CaSO_4 \cdot 2H_2O$		2
Pyrite	FeS_2		2
		Total	100

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Interpretation

Test Parameter	Pass	Fail	Sample E
Acid Soluble Sulfate AS	<0,2% SO_4	>0,2% SO_4	**0.83%**
Water Soluble Sulfate WS	<500 mg/L SO_4	>1500 mg/L SO_4	**2600**
Presence of Gypsum (from XRD)	N	Y	**Y**
Total Sulfur TS	<0,3% S	>1.0% S	**0.92%**
Proportion of fine grained sedimentary rock	<10%	>30%	**85%**
Evidence of secondary crystallization	N	Y	**Y**

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Need to Perform Suite 2 Testing

- Thin Section petrographic Analysis
- Water Absorption: 3.8% and 3.2%

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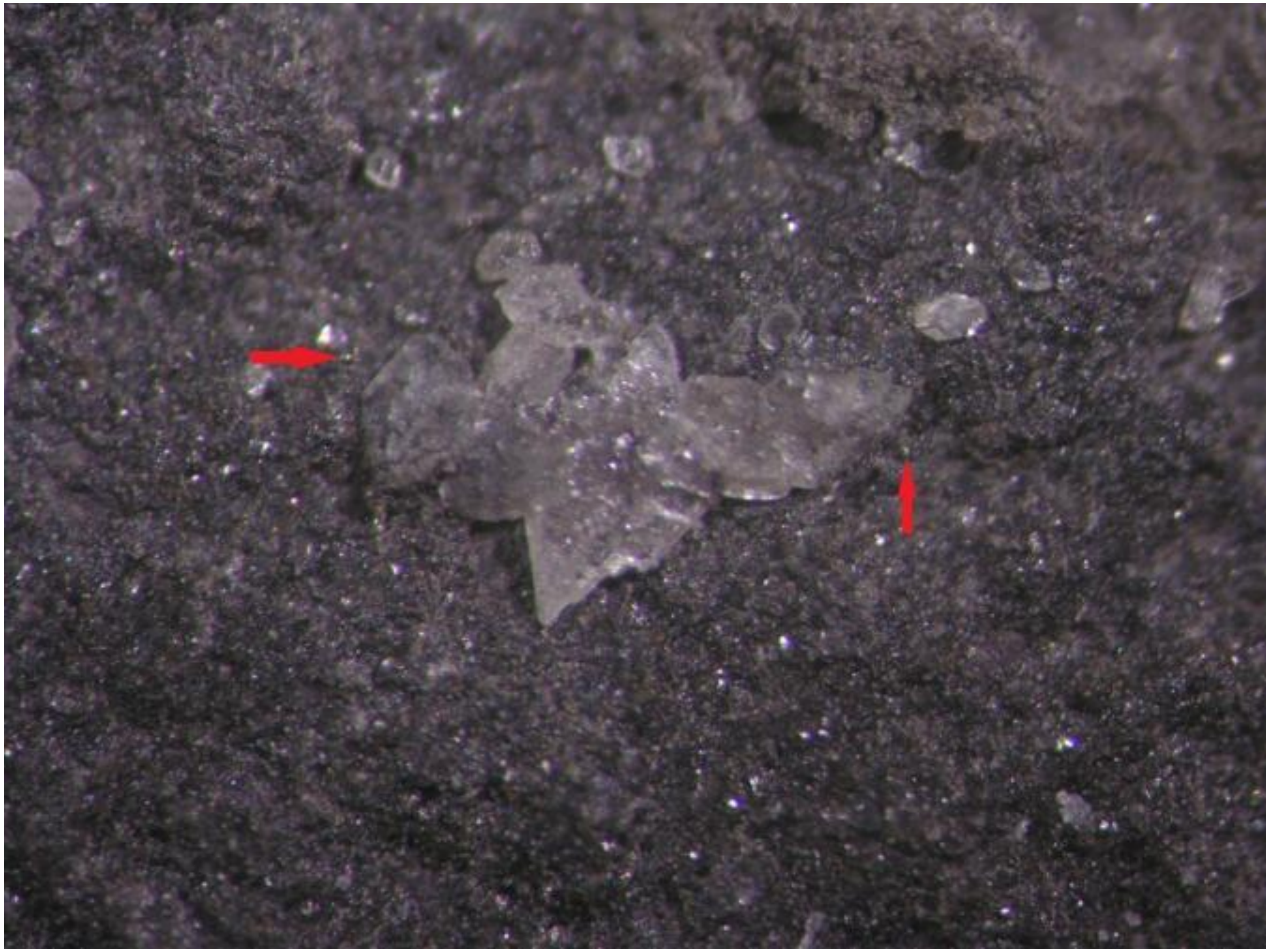
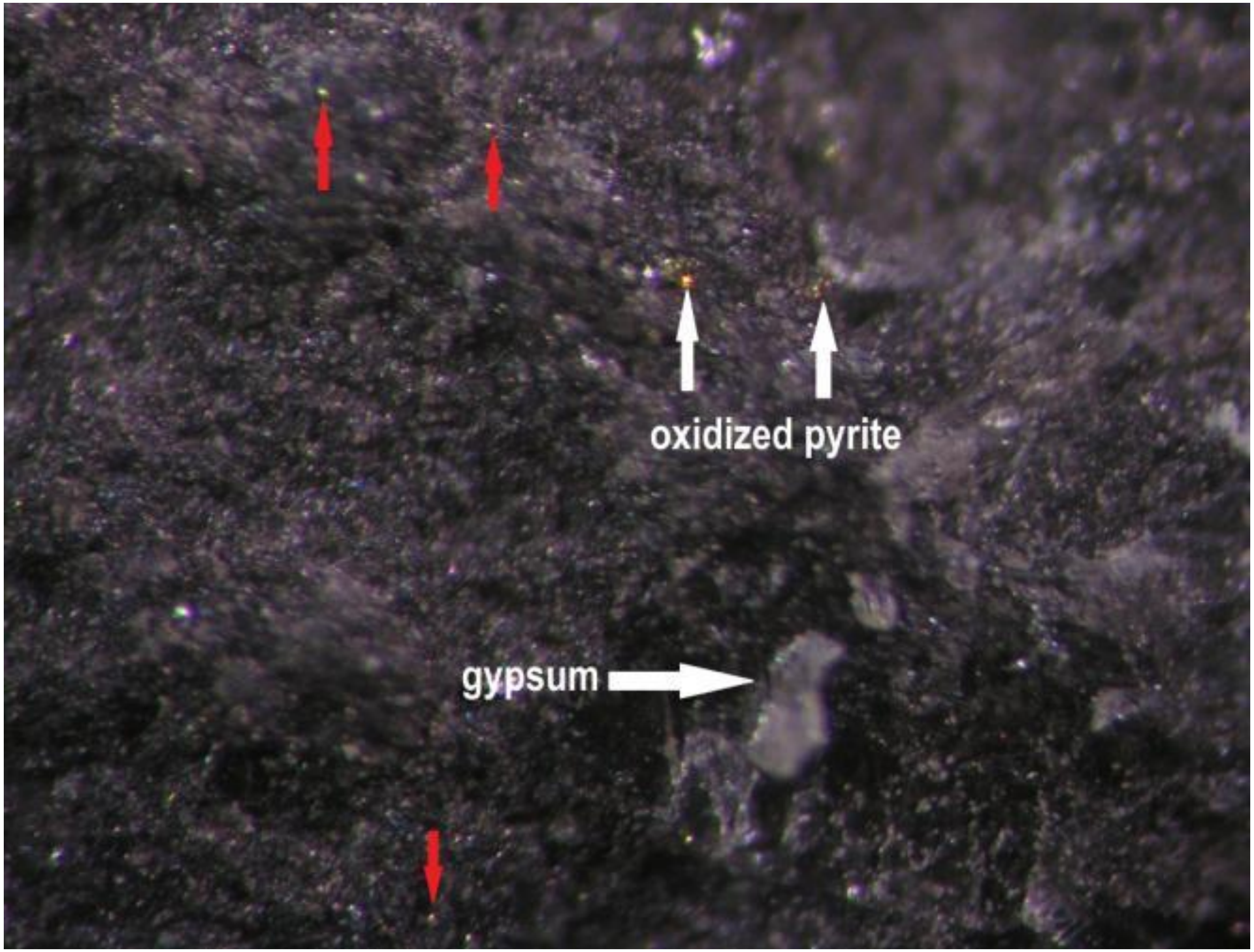
Binocular Microscope Images

Gypsum Cluster



Gypsum Clusters

Thin Section Image



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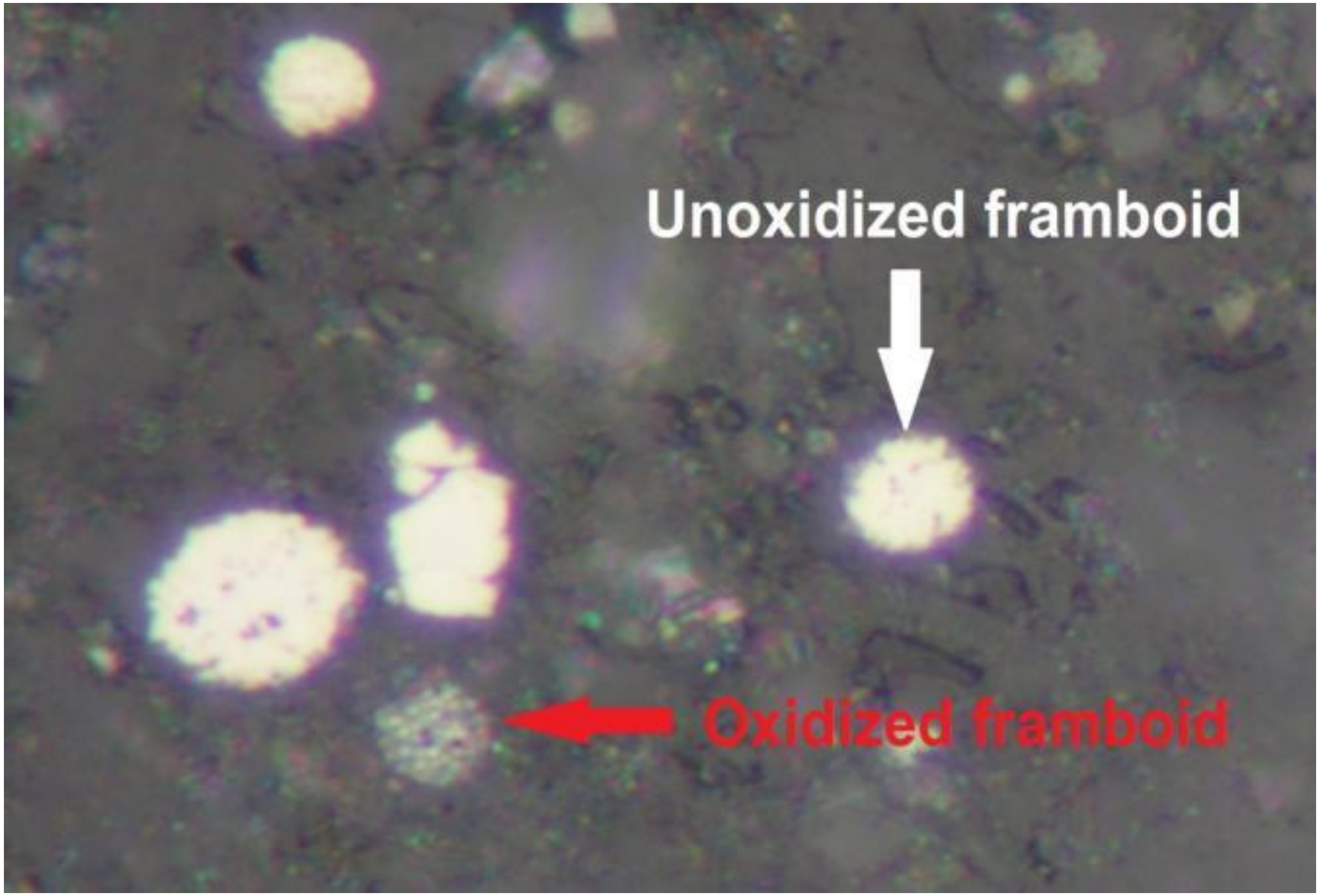
Thin Section Image



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Need to modify table to match sample

Factor	Derived from	Comments	Risk Factor Identified	
			E	F
Pyrite Content	Chemical Tests, Petrographic examination	Both E and F had high concentrations of pyrite: Chemical testing indicated 1.20% and 1.50% pyrite respectively. Pyrite contents estimated to be 3.0% and 3.2% by volume in the thin sections examined.	x	x
Presence of pyrite framboids	Petrographic examination	The petrographic examination showed framboidal pyrite to be abundant and widespread in the thin sections.	x	x
Evidence of oxidised pyrite	Petrographic examination	Oxidised pyrite identified in thin section.	x	x
Presence of clay minerals	X-Ray diffraction (XRD), Petrographic examination	Clay minerals identified by XRD and petrographic examination.	x	x
Presence of calcite	XRD, Petrographic examination	Presence of calcite confirmed by XRD and petrographic examination.	x	x
Presence of gypsum growth	XRD, Petrographic examination, Geological inspection	Presence of gypsum growth confirmed by Petrographic examination, geological inspection and XRD.	x	x

Need to modify table to match sample

Factor	Derived from	Comments	Risk Factor Identified	
			E	F
Presence of mudstone and other find-grained sedimentary rock	Geological inspection	The samples were composed of 50 to 60% fine-grained calcareous mudstone/siltstones. Smaller proportions of argilaceous limestone present	x	x
Structure of aggregate particles	Geological inspection, petrographic examination	Laminations in many particles, fractures present.	x	x
Porosity of rock	Petrographic examination, Water absorption	Water absorption of 3.8% and 3.2%	x	x
Moisture in sample	Geological inspection	Samples were damp upon retrieval from beneath house	x	x
Presence of sulphur-bearing minerals	XRD, Petrographic examination	None of the main minerals identified in XRD contain sulphur. The petrographic examination identified no significant source of sulphur other than pyrite.	x	x

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Category C or D

		Damage Condition Rating from Building Condition Assessment			
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	Inconclusive (Susceptible to limited expansion)	Category B	Category B	Category D*	Category D*
	Fail (Susceptible to significant expansion)	Category C	Category C	Category D	Category D

* In these cases the Engineer shall consider alternative probable causes for the damage other than pyritic heave.

Thank you for your attention!