CONSTRUCTION MATERIALS CONSULTANTS, INC.

Investigation of Concrete Blocks from Michael McClelland's Residence at 7 Carraig Bridge, Bridgend County Donegal, Ireland

Prepared for:
Michael McClelland

July 17, 2024
CMC 0724158

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July 17, 2024

Michael McClelland
7 Carraig Bridge
Bridgend, Co. Donegal
Ireland F93E516

RE: Concrete Blocks from the Residence of Michael McClelland, 7 Carraig Bridge, Bridgend, County Donegal, Ireland

Dear Michael:

Construction Materials Consultants, Inc. (CMC) is pleased to provide the enclosed comprehensive report on investigation of two concrete block samples that you have provided from your residence for assessment of overall condition and evidence of any potentially deleterious reactions, if any.

Results, opinions, and conclusions presented herein are based on the information and samples provided at the time of this investigation. We reserve the right to modify the report as additional information becomes available. Neither CMC nor its employees assume any obligation or liability for damages, including, but not limited to, consequential damages arising out of, or in conjunction with the use, or inability to use this resulting information.

Sample remains will be preserved in our archive of iron sulfide projects. With your authorization we will distribute this report amongst the scientific communities interested in iron sulfide reactions in concrete. All reports are the confidential property of CMC and information contained herein may not be published or reproduced pending CMC's written approval.

Please feel free to contact us with any additional questions. We look forward to providing our services again for your fellow homeowners who are in need of similar services of their homes for assessment of potential iron sulfide related distress.

Sincerely Yours,

CONSTRUCTION MATERIALS CONSULTANTS, INC.

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EXECUTIVE SUMMARY

Standing in the graveyard of an estimated 5000+ severely distressed concrete masonry homes in County Donegal, Ireland, the present home represents an epitome of success in proper choice of 'sound' construction materials during manufacturing of concrete blocks so that the home can stand sound during 20 years of its existence and hence offer an opportunity to examine the blocks and investigate the secrets behind its sound performance.

Background - As a background for large-scale cracking and crumbling of concrete masonry homes across County Donegal, two different mechanisms are offered (McCarthy et al. 2017, Leemann et al. 2023, Brough et al. 2023, Jana 2023 a, b, 2024 a). One mechanism, known as the 'mica crisis' is reported to be from the use of defective concrete blocks containing excessive "free mica" flakes in the mortar fractions (grains < 63-μm in size) derived from abrasion of micaceous aggregates (mostly phyllite with subordinate mica schist, etc., McCarthy et al. 2017) from the mixing process of aggregates and paste during manufacturing of blocks. Excess mica (e.g., > 5% of paste volume, Eden and Sandberg 2019) from abraded phyllite and/or mica schist aggregate in the paste has, reportedly, caused many known mica-related issues, e.g., increased water demand at a given workability, increased water absorption, increased microporosity, loss of compressive strength, reduced resistance to frost attack from high water demand, increased leaching, etc.

Subsequent studies have established evidence of iron sulfides mostly in the form of pyrrhotite and/or framboidal pyrite in the aggregates, which have caused oxidation and related expansions in the presence of moisture and oxygen followed by internal sulfate attacks (ISA) from reactions between sulfates released from pyrrhotite oxidation and cement hydration products resulting in formation of gypsum, ettringite, and/or thaumasite causing expansions and cracking to softening and crumbling of paste e.g., from decomposition of calcium silicate hydrate (CSH) in paste from thaumasite attack and severe carbonation and leaching of paste (Leemann et al. 2023, Brough et al. 2023, Jana 2024 a, b). Relatively open microstructures of blocks, use of dominant iron sulfide bearing phyllite aggregates, oxidation of pyrrhotite grains in phyllite, paste carbonation and leaching, and internal sulfate attacks from reactions between the sulfates released from iron sulfide oxidation and cement hydration/carbonation products are found responsible for the catastrophic distress (Jana 2024a).

The key player amongst all potential factors is found to be the presence of reactive sulfide minerals mostly in phyllite aggregate in aggregates in the form of framboidal pyrite, fine-grained disseminated pyrite, and pyrrhotite that have demonstrated their destructive powers as in cases from adjacent County Mayo (McCarthy et al. 2017) as well as other parts of the world including the classic Mundic problems in Cornwell and Devon (Hawkins 2014), classic pyrite heave problems from Dublin, Ireland to Ottawa, Canada (Hawkins 2014, Eden 2014, Eden and Sandberg 2019, Maher 2023) or pyrrhotite oxidation related cracking of concrete foundations in thousands of residential and commercial properties from Trois-Rivières Québec, Canada (Duchesne et al. 2021, 2023) to eastern Connecticut (Zhong and Wille 2018, Jana 2020, 2022, 2023a, 2024b) and Massachusetts in US. Studies of 1800 distressed properties from 1995 to 2010 in County Donegal found deleterious roles of pyrrhotite-bearing phyllite as the main cause of distress (Brough et al. 2023). Studies of some of the most severely distressed homes from County Donegal by Brough et al. 2023 and the author (Jana 2024 a, b) have seen the deleterious role of pyrrhotite in the dominant pyrrhotite-bearing phyllite aggregates to be the main culprit.

Samples & Methodologies - Two small, drilled cores of concrete masonry blocks (40 mm diameter, 30 mm long, identified as 'RHG' from right-hand gable inside garage from outer leaf, and 'LHG' from left-hand gable, outer leaf) were provided. Despite the inherent porous, typical popcorn-type texture of blocks (void contents calculated from sectioned surfaces by *Image J* to be less than 10%), both drilled cores were received in intact conditions. Both samples were examined and processed for various laboratory testing e.g., from total sulfur content analysis, optical microscopy, scanning electron microscopy and energy-dispersive X-ray microanalysis (SEM-EDS), X-ray fluorescence spectroscopy (XRF), and X-ray diffraction (XRD) by following various industry standards (e.g., ASTM C 856, ASTM C 1723, ASTM D 4239, EN-12620, I.S. 465:2018+A1:2020, BS 1881, CSA A23.1, Eden 2010).

Total Sulfur - According to most industry specifications, total sulfur content (S_T) of aggregate should be less than 0.1% for it to be considered innocuous; for S_T >1% aggregate should be discarded, and for S_T between 0.1 and 1% aggregate should be further tested by petrography for the presence of potentially deleterious iron sulfide minerals. Total sulfur contents of two samples measured by combustion-IR (Leco) method are 0.08% for LHG and 0.12% for RHG, average 0.1%, which, after conversion for S_T for aggregates from aggregate contents in blocks are higher than the 0.1% threshold (within 0.1% and 0.2%) for it to be subjected to further examinations by petrography. S_T values, however, are noticeably lower than the values found for severely distressed blocks from other houses in County Donegal tested in author's laboratory, which has provided the firsthand indication for the time-tested sound condition of the house for 20 years.

Petrography - Petrography has provided the most important information for the overall sound condition of house -

a. First, pyrrhotite-bearing phyllite, the most common aggregate found in more than 80% of distressed homes across Donegal (Brough et al. 2023) is virtually absent in both samples; instead, aggregates in both samples are found to be compositionally similar and made using major amount of *micaceous quartzite* coarse aggregate and only minor amount of phyllite. Micaceous quartzite grains are dense, hard, angular, ½ in. (12.5 mm) in maximum size, equidimensional to a few elongated, well-graded, well-distributed, and show massive quartzitic (granoblastic) textured mass of major amount of variably strained quartz, subordinate feldspar (albite), and parallel bands of muscovite and chlorite. These are texturally somewhat different from typical mica schists where both quartzo-feldspathic and micaceous bands show elongated parallel arrangements from medium-grade regional metamorphism, or phyllite, which are characteristically much finer-grained than schist and show foliated texture from parallel arrangements of mica, chlorite, quartz, and feldspar for lower grade regional metamorphism (i.e., below the biotite isograd).

b. Second, despite potential alkali-silica reactivity of many strained quartz grains in micaceous quartzite, there is, however, no evidence of such a reaction found in the two samples.

c. Third, reflected-light (ore) microscopy of polished epoxy-impregnated solid sections of blocks showed isolated finely disseminated grains of non-reactive pyrite (<0.1 mm size, at <0.1% level) in its typical blocky crystal habits and traces of chalcopyrite but no signs of reactive sulfide grains such as pyrrhotite or framboidal pyrite to warrant further investigations. The total sulfur (S_T) results are contributed from these minor, disseminated non-reactive pyrite and chalcopyrite grains as opposed to any threat from a reactive sulfide phase, along with sulfate contribution from Portland cement.

d. Fourth, consistent with the absence of any potentially deleterious iron sulfide grains, no deleterious sulfate deposits such as secondary ettringite or thaumasite are found in the paste, even though paste has shown carbonated nature, consistent with the overall open microstructure of blocks to facilitate ready migration of atmospheric CO_2 to the blocks during 20 years of service (no render layer was found in the samples).

e. Fifth, degree of carbonation of paste is found to be less than that found in the blocks from many distressed homes in County Donegal. Carbonation varied from minor to moderate to no carbonation at all, where in the latter case, paste showed typical near-isotropic nature of calcium silicate hydrate in cross polarized light as opposed to golden yellow interference color of fine-grained calcite in a carbonate paste.
f. Sixth, even though the house showed no visible cracks (well, except only one fine, hairline crack reported to have been noticed by the homeowner in the entire house after 20 years of service), optical microscopy of fluorescent-epoxy-impregnated thin section of blocks did found some fine, hairline microcracks in both samples mostly within the paste fractions or transecting the aggregates none of which are found associated with any potentially deleterious reactions but are rather simple manifestation of drying and/or carbonation shrinkage of carbonated paste fractions. Perhaps, one such microcrack was noticed by the homeowner that has extended to the exposed surface for his attention.

It is interesting to note that the reported manufacturer of concrete blocks that has, allegedly, supplied blocks for most of the distressed homes across County Donegal has quarried the aggregates from Bancrana quarry in Donegal. In the specification of blocks, the manufacturer has indicated use of metaquartzite aggregate in the blocks, and not phyllite seen in most distressed homes. Metaquartzite, or micaceous quartzite as in the present case are characteristically denser, harder, and equigranular (granoblastic) textured monomineralic (or dominantly quartz and subordinate feldspar e.g., from metamorphic arkosic sandstone) rock of variably strained quartz and sometimes minor feldspar that do not provide pathways for migration of moisture into the rocks during service as the phyllite aggregate does for its characteristic foliated (phyllitic) texture of fine-grained parallelly arranged mica (muscovite) and chlorite flakes. Therefore, reactive iron sulfide grains present in phyllite will be exposed to moisture far more readily for oxidation and subsequent distress than in a denser metaquartzite or micaceous quartzite host. Clearly, portions of pyrrhotite-bearing phyllite in the quarry has caused the distress, which should have been avoided and replaced by the metaquartzite portions of the quarry as demonstrated by the present house. Therefore, quarried stones should have been first examined by petrography (e.g., *a la* ASTM C 295) prior to the incorporation into the blocks. At a minimum, an S_T test should have effectively differentiated a non-reactive iron sulfide bearing metaquartzite as seen here from a reactive iron sulfide-bearing phyllite seen in many distressed homes.

To demonstrate the negligible presence of phyllite, only one or two phyllite aggregate particles are found in each of LHG and RHG (< 5% of aggregate), which (only in RHG) did show evidence of reddish-brown oxidation products of iron sulfate even though no reactive iron sulfide grains were detected.

XRD - XRD analyses detected minor diffraction peaks for an iron sulfate hydroxide mineral parabutlerite [iron sulfate hydrate, $Fe(SO_4)(OH)(H_2O)_2$] at <1% amount from the minor phyllite grain, but no direct evidence for any potentially deleterious iron sulfide grain was found in aggregate (including one phyllite grain in 'RHG'). XRD analyses of both samples, however, failed to detect pyrite seen in reflected-light microscopy for its presence at below the detection limits of instrument. Rietveld analyses of both samples showed: (a) abundant quartz from metaquartzite, quartz plus feldspar from mica schist (quartz 59.8%/52.9%, albite 15.3%/15.4%), (b) subordinate muscovite and chlorite from mica schist and one phyllite in RHG (11.1%/16.3% muscovite, 7.6%/7.1% chlorite), and (c) calcite from carbonated paste at 5.3%/7.8% - all proportions are given for RHG/LHG, respectively.

Free Mica Contents from SEM-EDS - Cluster phase map analyses in SEM-EDS showed free muscovite contents in the mortar fractions to be 7.2% in RHG and 10.5% in LHG. These results, however, are highly variable depending on the scale and the area of examination. Area percentages of muscovite in the mortar fractions calculated from EDS image analyses in SEM-EDS, however, are not to be confused with 'bulk' muscovite contents in blocks ('bound' in aggregates plus 'free' in mortar fractions) determined from XRD. XRD result should be considered more meaningful for any mica-related durability concerns, if at all, than the free mica content in paste from EDS result collected from a small (<0.5 sq. mm) area. Close association of reactive iron sulfides with the 'bound' mica within the host aggregates are found to have boosted their reactivities from ready accessibility of moisture transported through the mica flakes (Oliveira et al. 2014, Schmidt et al. 2011), although free mica flakes in paste can also increase the potential for moisture absorption and related durability issues including a boost to oxidation of reactive iron sulfide grains in mortar. Bound mica in the schistose/foliated textured host phyllite/mica schist provide the necessary pathways for migration of oxygen and moisture for iron sulfide oxidation and resultant cracking and crumbling of aggregates than the similar effects from free (abraded) mica in the mortar fractions.

Nevertheless, mortar fractions in both samples, in general, showed flakes of muscovite mica from mica schist just like abraded phyllite flakes of intimately mixed muscovite and chlorite found in the phyllite-bearing distressed blocks in other homes. Presence of these phyllosilicates in the mortar fraction are practically unavoidable for their easily splitable nature to be ended up in the mortar fraction during mixing of ingredients at the plastic state. Unfortunately, they are taken at special attention in many literatures of Ireland (e.g., Eden and Sandberg 2019) including calculation of risk factor based on their proportion in the I.S. 465:2018+A1:2020 specification for their alleged potential deleterious reactions to affect the durability of blocks (e.g., high risk factor at > 5% free mica). Both samples showed > 5% free mica contents in the mortar fractions yet perfectly sound conditions of house after 20 years of service simply from the lack of any potentially reactive iron sulfide grains in the blocks.

The author has found no evidence of any deleterious reactions of free mica in some of the most severely distressed blocks unless and until they are found to be contaminated with reactive iron sulfide grains either in the host rock and/or in the abraded fractions. The present house stands as an ideal case for their lack of any deleterious reactions after 20 years of service to not necessarily point fingers to these so-called 'free mica' fractions in the mortar fractions of blocks until and unless some sort of reactive form of iron sulfide grains are found, which are absent in the examined blocks to stand as testament for the sound condition of house.

Paste in SEM-EDS - Similar to petrography or XRD, SEM-EDS studies of paste fractions of blocks also failed to detect any common deleterious reaction products, e.g., gypsum, ettringite, or thaumasite from iron sulfide reactions found in the other distressed homes. The entire cross-sectional areas of examination for both samples have failed to detect any noticeable sulfur signals in EDS analyses from the paste to warrant closure examination of any sulfate reaction products.

Lack of Reactive Sulfides or ISA – Petrography, along with SEM-EDS studies have failed to detect any reactive sulfide grains or evidence of internal sulfate attack (ISA) in paste that are so commonly found in the distressed homes across County Donegal (Leemann et al. 2023, Jana 2024a). This lack of ISA is the bottom-line for the sound performance of this home for past 20 years.

Conclusions – The following conclusions are dawn from the present laboratory studies of two block samples from the home at 7 Carraig Bridge in Bridgend, Co. Donegal, Ireland:

a. In-depth evaluation of two samples confirmed: (a) absence of any distress in the microstructural level, including (b) absence of any potentially deleterious reactive forms of iron sulfide constituents (e.g., pyrrhotite, framboidal pyrite, etc.) or (c) negligible amounts of aggregates that are known to cause distress in many homes across County Donegal (e.g., phyllite) – all of which are the reasons for the reported sound condition. Lack of distress are confirmed from (a) homeowner's visual assessment, (b) evaluation of field photos, and (c) subsequent detailed laboratory testing of representative samples presented here encompassing optical microscopy, SEM-EDS, chemical analyses (including total sulfur), and XRD.

b. Based on all these testing, the house is judged to be categorized under Group 1 as "**undamaged**" at "**low risk**" under I I.S.465:2018+A1:2020 (Tables 1 and 3, respectively) at the time of this examination. *Risk factor assessment should not be done based on free mica content let alone collected from < 0.5 sq. mm area of mortar in an SEM-EDS image*.

c. Mica content of aggregate, or free (abraded) mica content of interstitial mortar fraction has negligible role on the distress as the present house showed the normal expected free mica in the mortar fraction at amounts (~10%) higher than the 5% threshold limit referred by others (e.g., Eden and Sandberg 2019) for many distressed homes where it is actually not the free mica content but the presence of reactive sulfide grains that were found responsible for the distress. Mica can, at best, facilitate moisture migration to the reaction sites of reactive sulfides for oxidation, which could be the reason for dominance of phyllite aggregate in majority (> 80%) of the distressed homes across County Donegal that have produced a larger amount of free mica in the paste from abrasion during mixing than the free mica contents in homes having blocks made using e.g., metaquartzite aggregate (Brough et al. 2023).

d. Sound condition of the present house after 20 years of service is found to be due to use of sound micaceous quartzite aggregates that are free of any reactive form of iron sulfide minerals (e.g., pyrrhotite, framboidal pyrite, etc.).

e. Contrary to the reactive iron sulfide (e.g., pyrrhotite) bearing phyllite aggregate found in most of the distressed homes across County Donegal, present house showed the importance of use of proper aggregate selection that remain sound for the absence of any reactive sulfide grains.

f. A total sulfur content slightly in excess of 0.1% doesn't necessarily indicate an imminent danger if the sulfur is petrographically found to have contributed from the non-reactive forms of sulfide, as has been the case here.

g. *Risk factor assessment should be done based on petrographically determined reactive iron sulfide grains (pyrrhotite, framboidal pyrite, marcasite, etc.) in the quarried aggregates or blocks including types and proportions of reactive grains where total sulfur (at* $0.1\% < S_T < 1\%$*) can only provide an initial clue for further exploration by petrography for exact speciation of iron sulfides*.

h. Assessment of risk factor based on free mica content in I.S. 465:2018+A1:2020 specification can be seriously misleading to the actual field performance of blocks, as in the present case where despite having a field-proven 20-year performance record, the house can still be wrongfully categorized under 'high risk' based entirely on the free mica content calculated from a small area (~ < 0.5 sq. mm) in an SEM-EDS image. In fact, this house should stand as an example of potential pitfall of considering free mica as opposed to reactive sulfide as a basis for risk assessment.

i. Petrography must incorporate polished thin section and solid section examination in transmitted and reflected light microscopy in a petrographic microscope, which must be done first (following S_T test) before doing SEM-EDS studies.

j. As a general rule for new constructions in County Donegal, despite being the geologically key locality for phyllite formed in the widespread Dalradian Supergroup from a 700 Ma old mountain-building event (Grenvillian Orogeny), phyllite-bearing aggregates should be avoided for their common association with reactive iron sulfide (pyrrhotite) grains in most of the distressed homes in County Donegal. Mica schist and micaceous quartzite aggregates should be examined first for reactive sulfides, particularly for stones having total sulfate contents between 0.1 and 1 percent.

k. Therefore, a revised standard specification is needed for proper assessment of homes in Ireland and other parts of the world that have experienced iron sulfide related distress, as mentioned in Jana 2024b (Appendix 4). Laboratory testing protocols based on total sulfur, petrography, SEM-EDS, micro-XRF, and XRD are, for the most cases, highly effective in screening of quarried stones for future construction, or aggregates in existing concrete blocks for condition assessment. For aggregates having $0.1\% < S_T < 1\%$ and petrographically observed iron sulfides grains, long-term expansion tests of mortar bars of quarried stones can be done under controlled laboratory conditions of relative humidity, temperature, mix design, and other parameters for performance assessment. Petrographic detection of aggregates having reactive sulfides such as pyrrhotite, marcasite, or framboidal pyrite should be avoided as they can cause serious damage even at < 0.1% level (Jana 2023a).

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THE HOUSE

Michael McClelland's Residence at
7 Carraig Bridge, Bridgend, County Donegal, Ireland

Right front of house

Close right corner

Left front of house

Figure 1: Field photos of the home from different sides showing the overall sound condition of walls and the absence of any visible cracking.

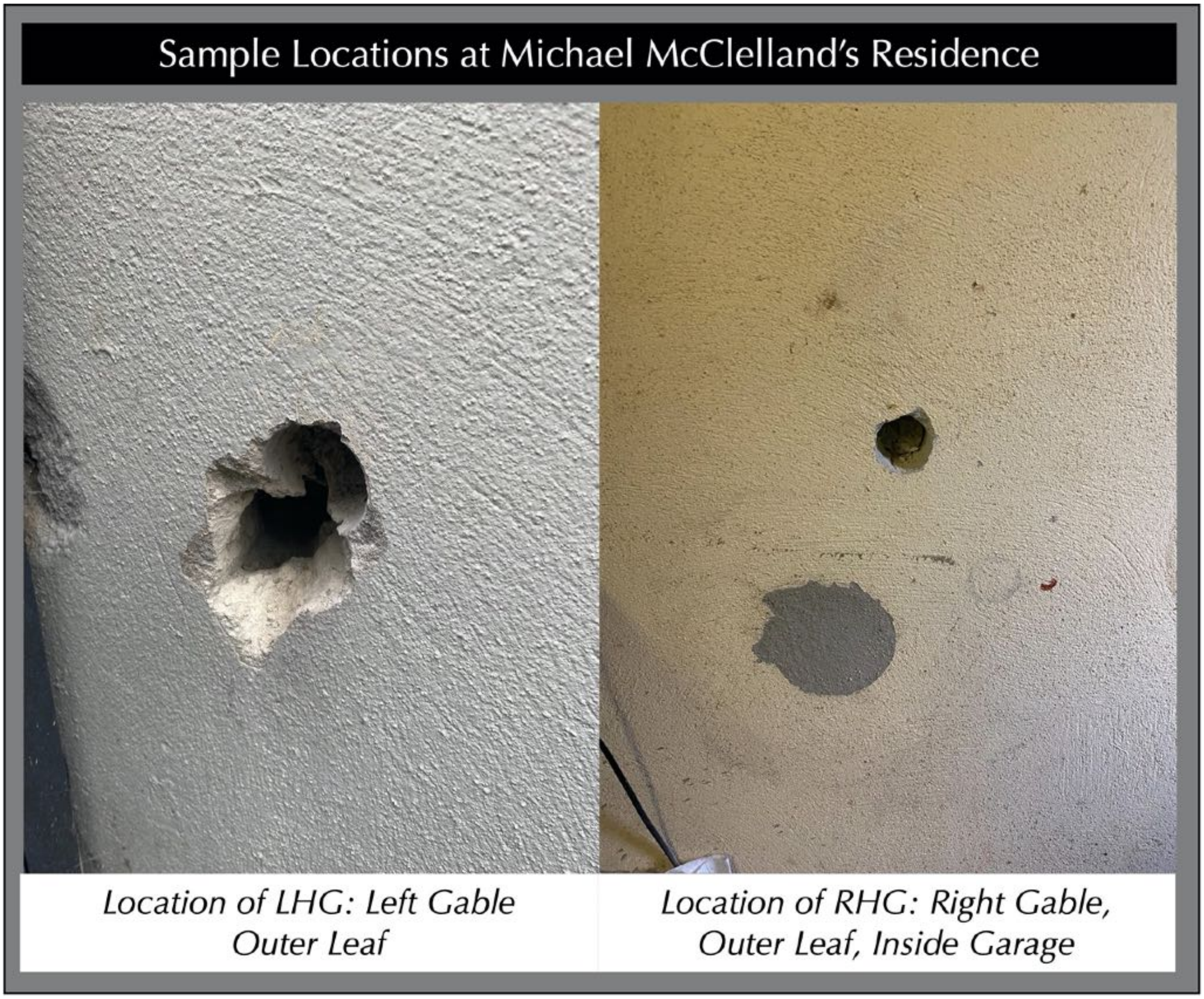
Front upstairs window

Middle corner

Back of house

Hairline crack above back door

Figure 2: Field photos of the home from different sides showing the overall sound condition of walls and the absence of any visible cracking except one fine, hairline crack marked with arrows.



Sample Locations at Michael McClelland's Residence

Location of LHG: Left Gable Outer Leaf

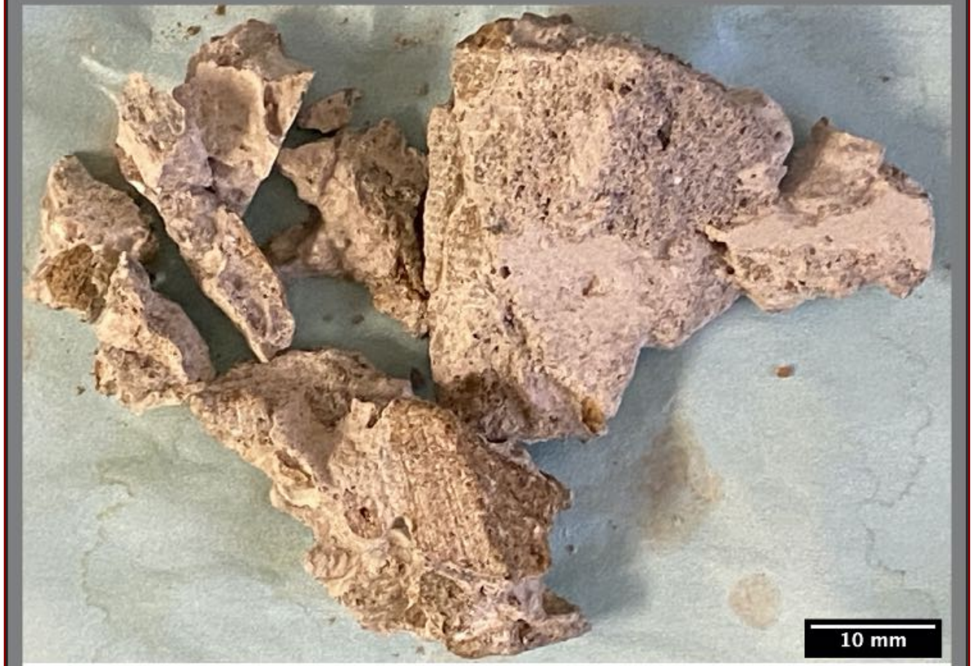
Location of RHG: Right Gable, Outer Leaf, Inside Garage

Figure 3: Locations from where two drilled core samples were provided from left gable (LHG) and right gable (RHG) – both from the outer leaf of house.

BLOCK SAMPLES

Figure 4: Cylindrical (top) and cross sectioned (bottom) surfaces of drilled core 'LHG' showing crushed micaceous quartzite aggregate, interstitial paste, and coarse voids where void contents are estimated from ImageJ to be 9.8% and 6.8% at sectioned surfaces from bottom left and right photo, respectively.

Block Samples from Michael McClelland's Residence at
7 Carraig Bridge, Bridgend, County Donegal, Ireland



Cylindrical surface of a drilled core 'RHG' 40 mm diameter 30 mm length

Sectioned surfaces of a drilled core 'RHG' 40 mm diameter 30 mm length

Figure 5: Cylindrical (top) and cross sectioned (bottom) surfaces of drilled core 'RHG' showing crushed micaceous quartzite aggregate, interstitial paste, and coarse voids.

METHODOLOGIES

TOTAL SULFUR

A portion of each block sample was pulverized for total sulfur content analyses by combustion IR (Leco) method according to the procedures described in EN 12620, I.S. 465:2018+A1:2020, CSA A23.1:19, and ASTM D 4239.

OPTICAL MICROSCOPY

Drilled cores of blocks were trimmed to multiple sections in a water-cooled diamond saw. Some sections were oven-dried, encapsulated with fluorescent dye-mixed epoxy then polished to mirror finish surfaces while others were selected for preparation of light-transparent fluorescent-epoxy-impregnated thin section by following multiple sample preparation steps described in Jana 2006 (e.g., grinding of a sectioned surface on a lapping wheel with alumina and diamond abrasives of different grain sizes, bonding of the smooth ground surface to a frosted glass slide, precision sectioning, precision grinding, and final hand grinding to bring down the final thickness to 30 microns (0.03 mm) or less so that polarized light can transmit through the thin slice).


Figure 6: The optical microscopy laboratory at CMC which houses many microscopes used for this project.

Petrographic examination was done according to the procedures described in various literatures, e.g., ASTM C 295, ASTM C 856, BS 1881-211:2016, BS 812, Part 104 (2021), BS EN 12620, BS EN 932-3 (2022), Eden (2010, 2013, 2014), CSA A23.2-15 (2009), CS TR 71 (2010), Hawkins (2014), and Poole and Sims (2016).

The steps of petrographic examinations include (Jana 2006: (a) visual examinations of samples, as received; (b) low-power stereo microscopical examinations of as-received, saw-cut, and polished cross sections of samples for evaluation of textures, and compositions; and (c) examinations of polished solid section and polished large area (50 mm × 75 mm) thin section in a petrographic microscope by reflected and transmitted polarized lights for detailed compositional and microstructural analyses. Figure 6 shows various microscopes in the optical microscopy lab some of which were used during petrographic examinations.

SCANNING ELECTRON MICROSCOPY AND X-RAY MICROANALYSES (SEM-EDS)

Figure 7: JEOL Neoscope JCM-7000 benchtop and CamScan Series 2 floor model scanning electron microscopes in CMC used to examine samples at noticeably higher magnifications than optical microscopes along with detailed microstructural and microchemical analyses.

In addition to optical microscopy, polished sections of blocks were further examined in JEOL Neoscope JCM-7000 and Camscan scanning electron microscopes (Figure 7) to determine their chemical compositions and microstructures. Both JEOL and Camscan SEMs are equipped with a high-resolution column 40Å tungsten, 15 to 40 kV electron optics zoom condenser 75° focusing lens (for Camscan) operating at 5kV to 20 kV, equipped with a variable geometry secondary electron detector, backscatter electron detectors, and Ametek EDAX EDS detectors for observations of microstructures at high-resolution, compositional analyses, and quantitative determinations of major element oxides from various areas of interest, respectively. Revolution 4Pi software in Camscan and Smileview software in JEOL Neoscope are used for digital storage of secondary electron and backscatter electron images, elemental mapping, and analysis along with point/raster mode, elemental map and cluster phase map analyses of an area of interest. Polished sections were coated with a gold-palladium alloy and used with a custom-made aluminum sample holder in the large multiported chamber with the eucentric 50 × 100 mm motorized stage in Camscan or 40 × 40 mm motorized stage in JEOL. SEM-EDS studies were done by using the methods of ASTM C 1723.

X-RAY FLUORESCENCE SPECTROSCOPY (XRF)

An X-ray fluorescence (XRF) instrument was used for determination of chemical (major element oxide) compositions of pressed pellets of pulverized portions of blocks including total sulfur contents, expressed as SO_3.

A series of standards from Portland cements, lime, slag, gypsum to various rocks, and masonry mortars of certified compositions (e.g., from USGS, GSA, NIST, CCRL, Brammer, or measured by ICP) were used to calibrate the instruments for various oxides, and empirical calculations were done from such calibrations to determine the oxide compositions of blocks.

One of the three energy-dispersive bench-top X-ray fluorescence units in CMC shown in Figure 8 was used. All units deliver rapid qualitative and quantitative determination of major and minor atomic elements in a wide variety of sample types with minimal standards. The instruments were calibrated by using various certified (CCRL, NIST, GSA, and Brammer) reference standards of cements and rocks and major element oxidation composition of unknown sample was determined from empirical calibrations of well-documented standards.

Approximately 8 grams of representative sample was pulverized for 3 minutes with alcohol in a pulverizing mill (Spex floor model pulverizer) with three wax tablets for preparation of pressed pellets. A 25-ton Spex automated hydraulic press was used for preparation of pressed pellets.

Figure 8: Rigaku NEX-CG (top), Spectro Xepos HE (middle), and Oxford X-Supreme 8000 (bottom) benchtop XRF units in CMC, all of which can perform analyses of multiple pressed pellets or fused beads of samples.

X-RAY DIFFRACTION (XRD)

A portion of pulverized block samples used for XRF was used to determine mineralogical composition by X-ray diffraction.

XRD was carried out in a floor model Siemens D 5000 or a benchtop Bruker D2 Phaser (2nd Generation, shown in Figure 9) Powder diffractometer (Bragg-Brentano geometry) employing a Cu X-ray tube (Cu k-alpha radiation of 1.54 angstroms), a primary slit of 1 mm, a receiving slit of 3 mm, a position sensitive 1D Lynxeye XE-T detector (in Bruker unit). Generator settings used are 10 to 30 kV and 10mA (100 to 300 watt).

The resulting diffraction patterns were collected by MDI Jade's Datascan (for Siemens) or Bruker's Diffrac.Measurement software (for D2 Phaser).

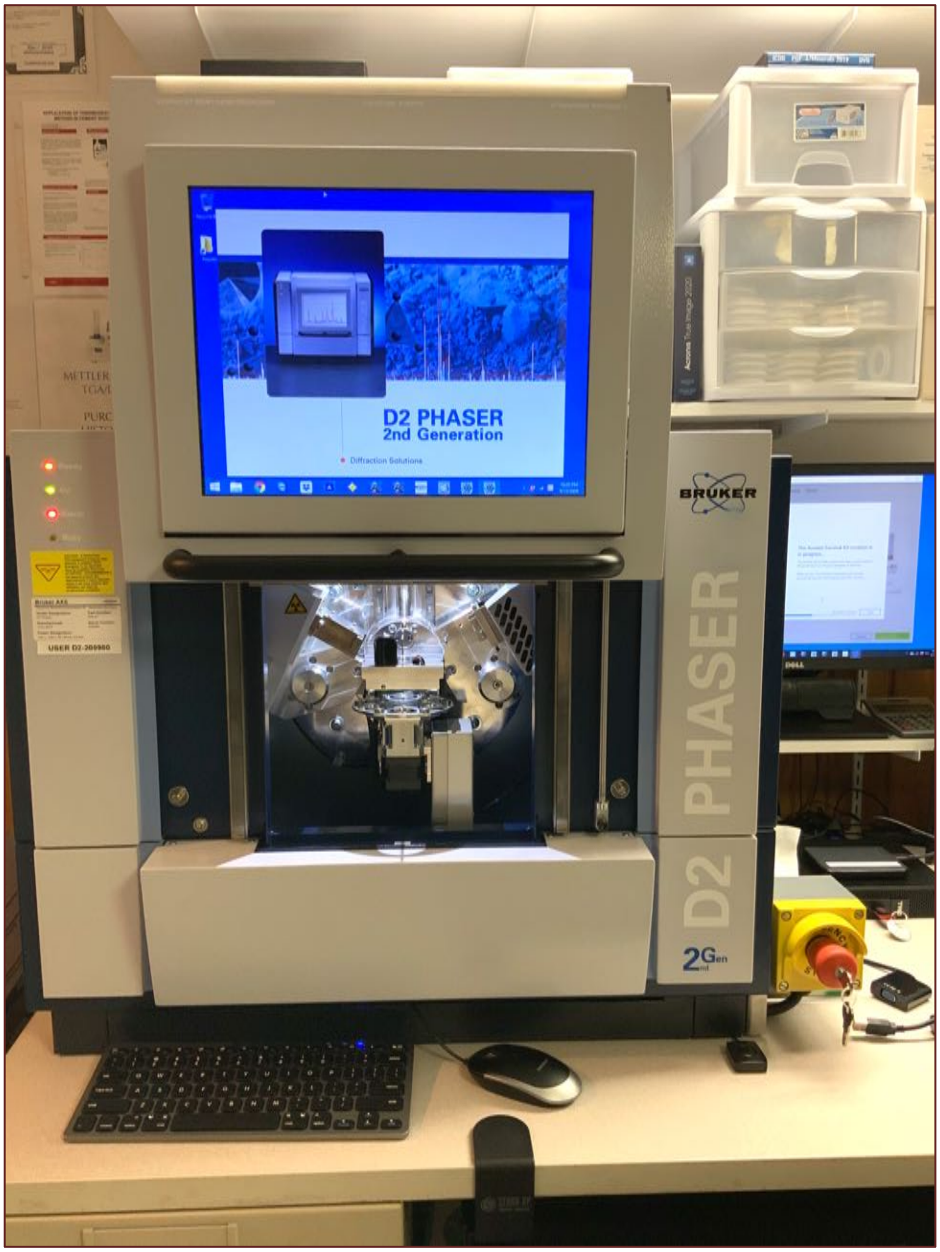


Figure 9: Bruker's D2 Phaser (2nd generation) benchtop X-ray powder diffractometer with Lynxeye 1D position sensitive detector used in X-ray diffraction studies of block samples.

Phase identification was done with Bruker's Diffrac.EVA. Match! or MDI Jade software with the search-match database from Crystallographic Open Database (COD) or ICDD's PDF4+ Minerals. Additional phase identification, and Rietveld quantitative analyses were carried out with MDI's Jade software.

OPTICAL MICROSCOPY

POLISHED SOLID SECTIONS OF BLOCKS

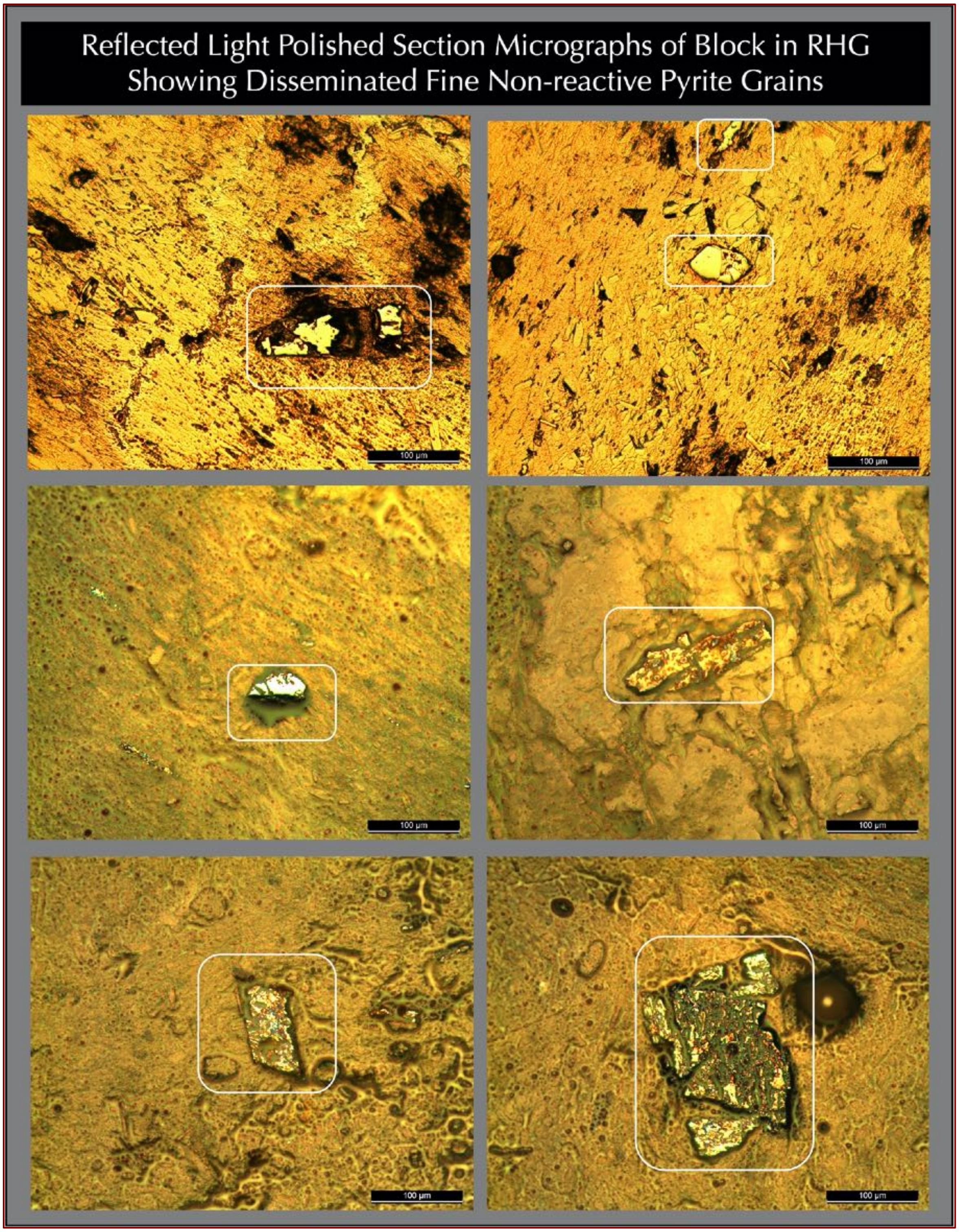
Figure 10: Polished section of sample RGH showing only one potentially deleterious phyllite coarse aggregate with reddish-brown oxidation products of iron and associated microcracks, which are also filled with the oxidation products (marked by arrows). Rest of the coarse aggregate particles are **crushed micaceous quartzite**, which are dense, hard, equigranular to elongated, massive quartzitic (granoblastic) textured mass of equigranular grains of major amount of quartz, subordinate feldspar (albite), and parallel bands of muscovite and chlorite, having a nominal maximum size of 10 mm. Interstitial paste in the mortar fraction shows beige color tone. Coarse irregular-shaped interstitial voids are calculated (from ImageJ) to be less than 10 percent.

Polished Solid Section of Sample LGH

Aggregate has major amount of metaquartzite (MQ) and mica schist (MS) and subordinate amount of Phyllite (P)

Figure 11: Polished section of sample LGH showing **crushed micaceous quartzite** coarse aggregate particles, which are dense, hard, equigranular to elongated, massive quartzitic (granoblastic) textured mass of equigranular grains of major amount of quartz, subordinate feldspar (albite), and parallel bands of muscovite and chlorite, having a nominal maximum size of 10 mm. Interstitial paste in the mortar fraction shows beige color tone. Coarse irregular-shaped interstitial voids are calculated (from ImageJ) to be less than 10 percent.

REFLECTED-LIGHT (ORE) MICROSCOPY



Reflected Light Polished Section Micrographs of Block in RHG Showing Disseminated Fine Non-reactive Pyrite Grains

Figure 12: Reflected-light micrographs of a gold-coated polished section (top row) and polished thin section (middle and bottom rows) of fragments in RHG showing typical golden yellow color tone and blocky habit of a few scattered very fine (< 0.1 mm) non-reactive pyrite grains. Photos were taken from a petrographic microscope with incident-light illumination. Scale bars are 0.1 mm.

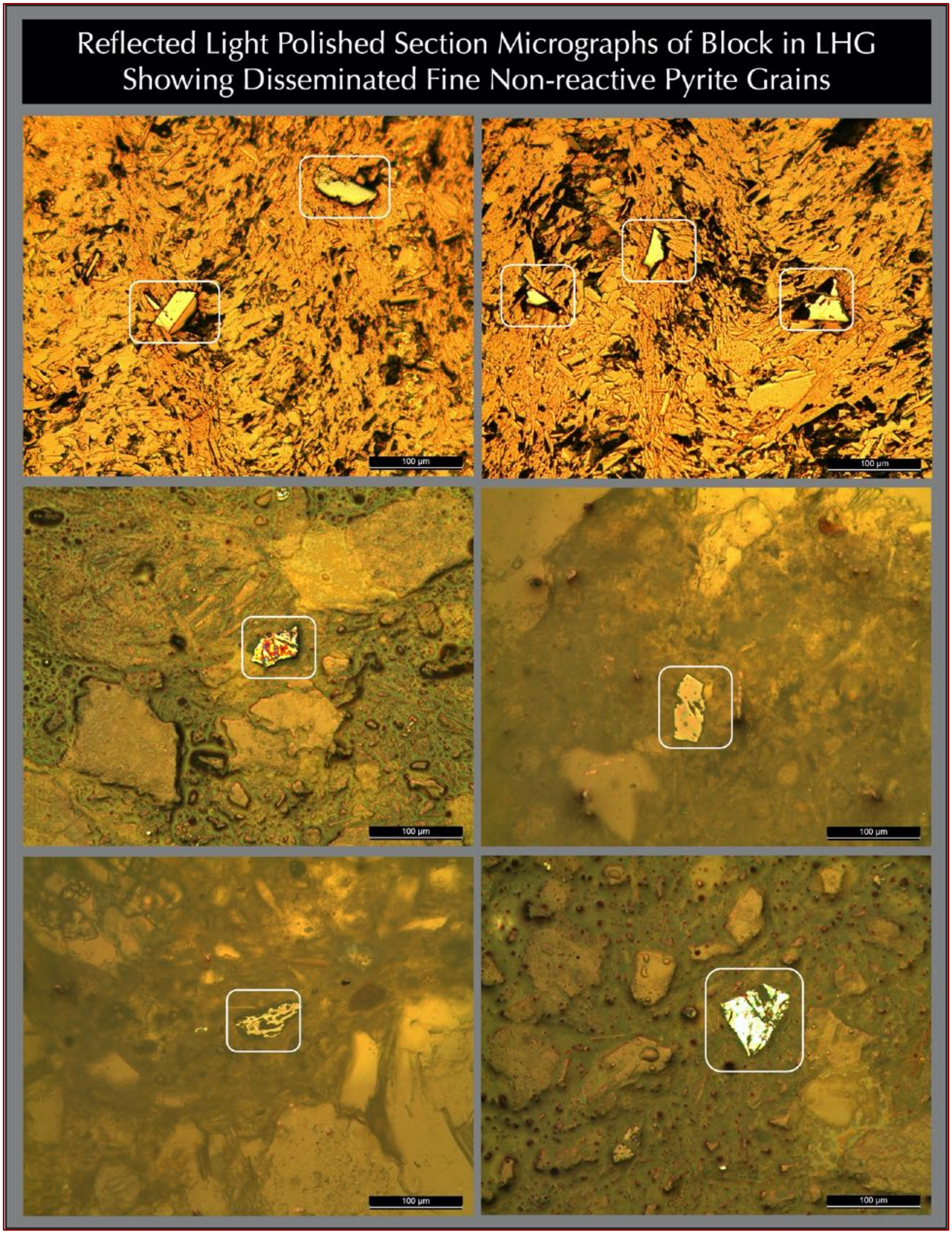
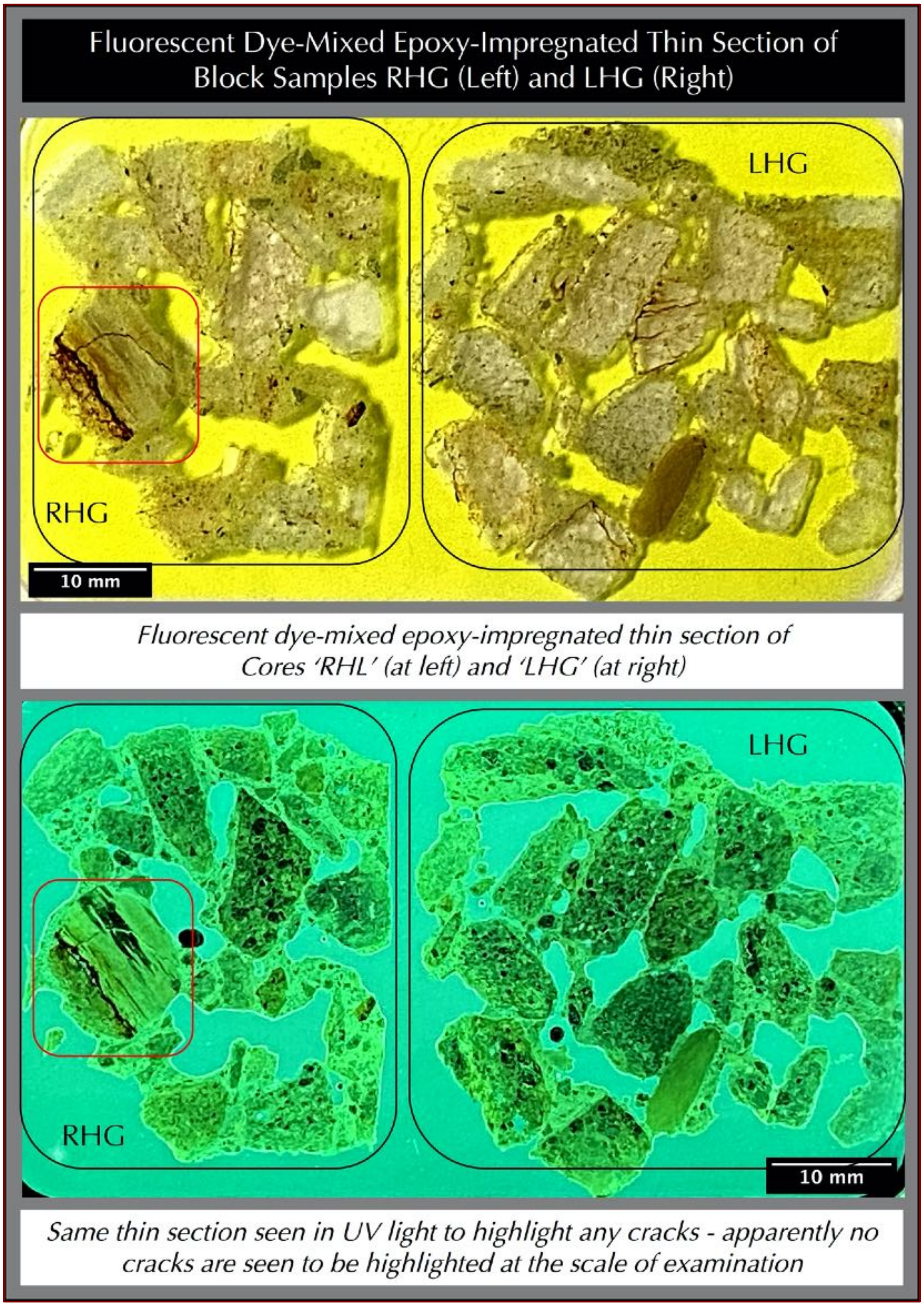


Figure 13: Reflected-light micrographs of a gold-coated polished section (top row) and polished thin section (middle and bottom rows) of fragments in LHG showing typical golden yellow color tone and blocky habit of a few scattered very fine (< 0.1 mm) non-reactive pyrite grains. Photos were taken from a petrographic microscope with incident-light illumination. Scale bars are 0.1 mm.

THIN SECTIONS OF BLOCKS

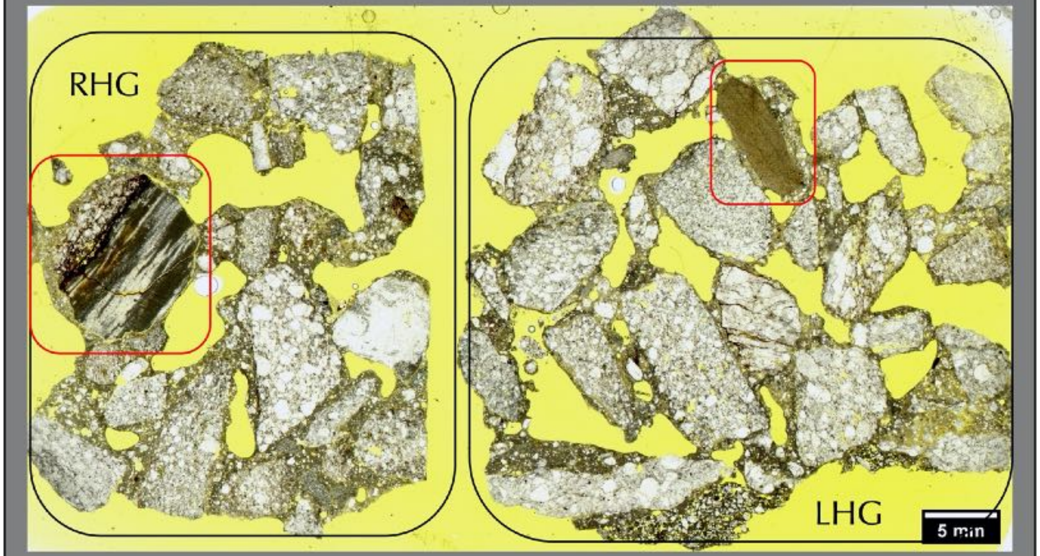

Fluorescent Dye-Mixed Epoxy-Impregnated Thin Section of Block Samples RHG (Left) and LHG (Right)

Fluorescent dye-mixed epoxy-impregnated thin section of Cores 'RHL' (at left) and 'LHG' (at right)

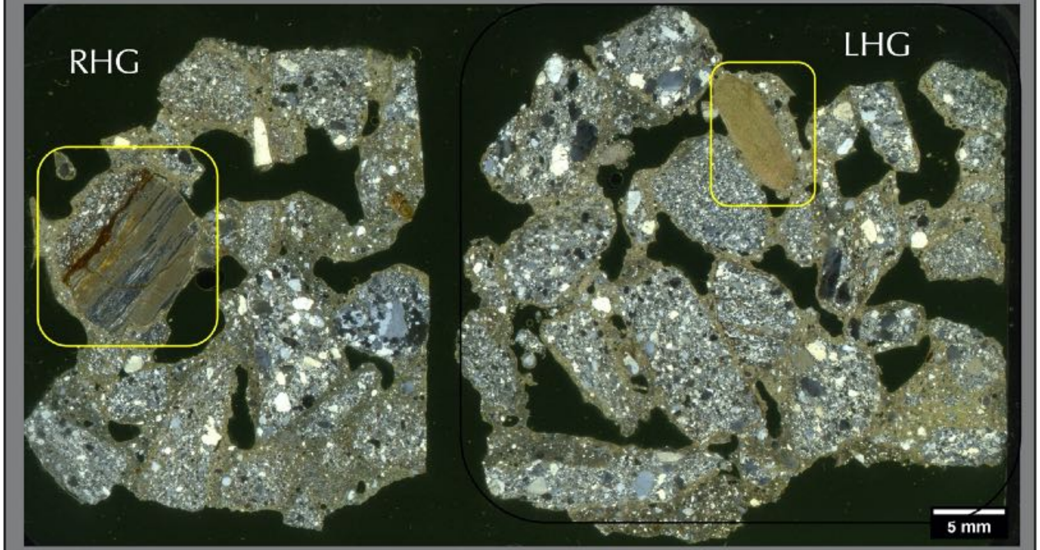
Same thin section seen in UV light to highlight any cracks - apparently no cracks are seen to be highlighted at the scale of examination

Figure 14: Fluorescent dye-mixed epoxy-impregnated polished thin section of block fragments prepared for reflected, transmitted, and fluorescent (UV) light microscopy in a high-power stereozoom and a petrographic microscope followed by SEM-EDS studies. Bottom row shows the thin section viewed in UV light where no cracking was highlighted from fluorescent epoxy. Thin section was scanned on a flatbed office scanner with LED and UV light for top and bottom images, respectively.

Fluorescent Dye-Mixed Epoxy-Impregnated Thin Section of
Block Samples RHG (Left) and LHG (Right) at PPL (Top) & XPL (Bottom)



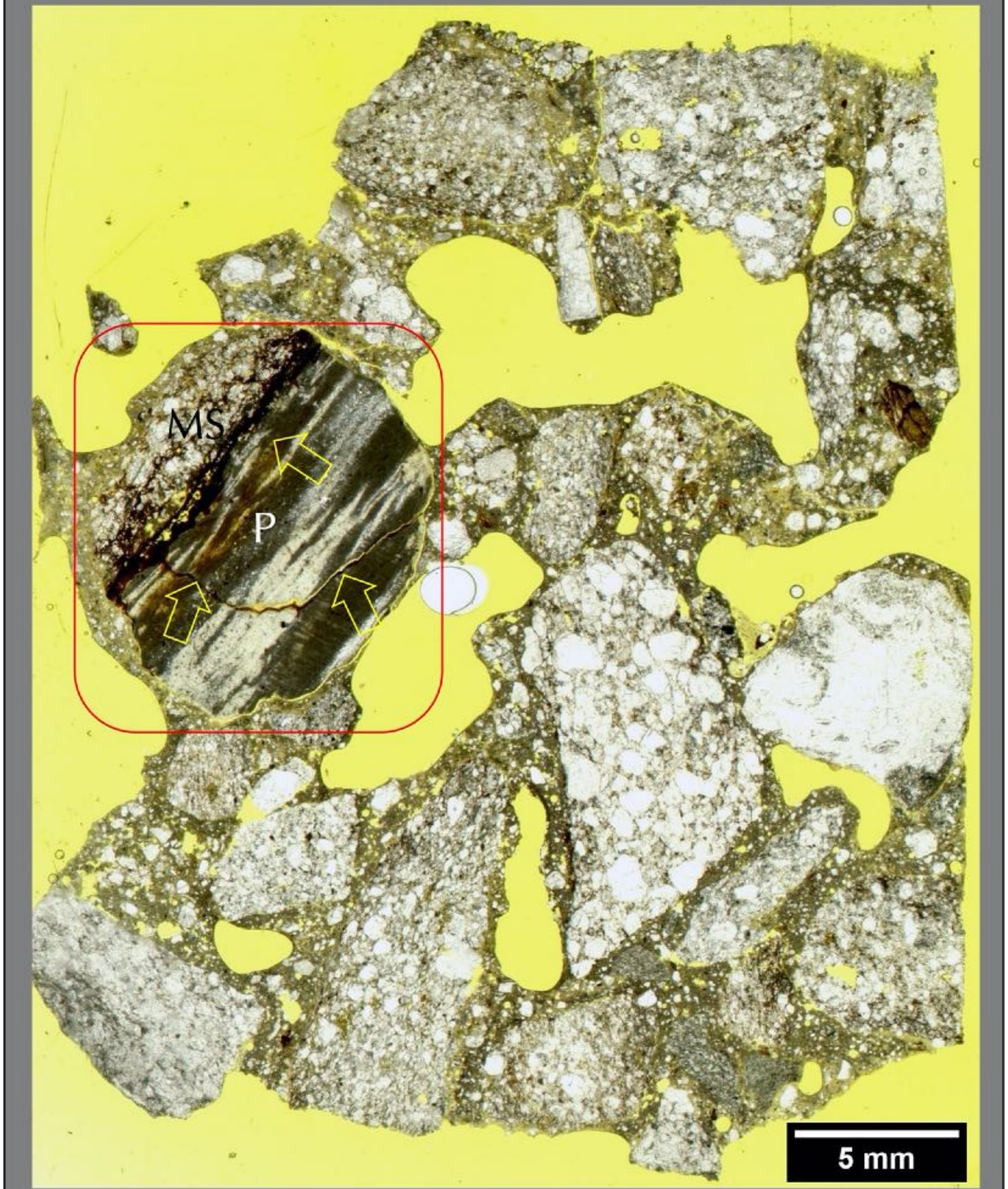
Fluorescent dye-mixed epoxy-impregnated thin section of
Cores 'RHL' (at left) and 'LHG' (at right) seen at Plane Polarized Light



Fluorescent dye-mixed epoxy-impregnated thin section of
Cores 'RHL' (at left) and 'LHG' (at right) seen at Cross Polarized Light

Figure 15: Fluorescent dye-mixed epoxy-impregnated polished thin section of blocks fragments shown at plane and cross polarized-light modes at top and bottom photos, respectively that are scanned on a flatbed office scanner with one or two perpendicular polarizing filters, respectively. Both samples show **crushed micaceous quartzite** coarse aggregate particles, which are dense, hard, equigranular to elongated, massive quartzitic (granoblastic) textured mass of major amount of quartz, subordinate feldspar (albite), and parallel bands of muscovite and chlorite, having a nominal maximum size of 12.5 mm. Interstitial paste in the mortar fraction shows carbonated nature. Coarse irregular-shaped interstitial voids are calculated (from ImageJ) to be less than 10 percent.

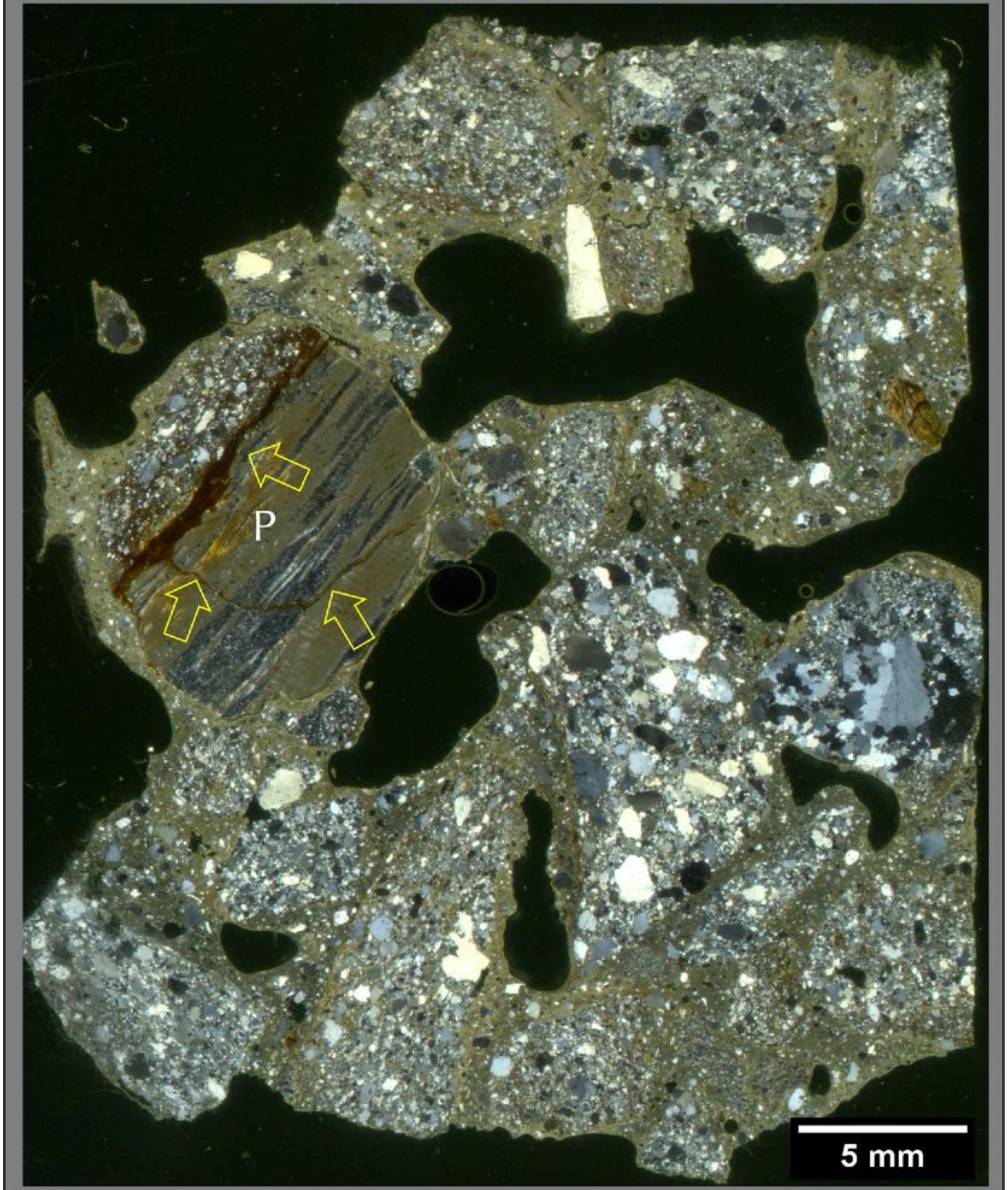
Thin Section of Sample RHG at PPL



Reddish-brown oxidation products and a crack filled with reddish-brown oxidation product in a phyllite aggregate particle (Boxed);
Rest of the coarse aggregate particles are crushed, 12.5-mm maximum size micaceous quartzite

Figure 16: Enlarged view of the plane polarized light image from Figure 15 of RHG showing one potentially deleterious phyllite (P) coarse aggregate with reddish-brown oxidation products of iron and associated microcracks, which are also filled with the oxidation products (marked by arrows). Rest of the coarse aggregate particles are **crushed micaceous quartzite**, which are dense, hard, equigranular to elongated, massive quartzitic (granoblastic) textured mass of major amount of quartz, subordinate feldspar (albite), and parallel bands of muscovite and chlorite, having a nominal maximum size of 12.5 mm. Interstitial paste in the mortar fraction shows carbonated nature. Coarse irregular-shaped interstitial voids are calculated (from ImageJ) to be less than 10 percent.

Thin Section of Sample RHG at XPL



Reddish-brown oxidation products and a crack filled with reddish-brown oxidation product in a Phyllite aggregate particle (Boxed); Rest of the coarse aggregate particles are crushed, 12.5-mm maximum size Micaceous Quartzite

Figure 17: Enlarged view of the cross polarized light image from Figure 15 of RHG showing one potentially deleterious phyllite (P) coarse aggregate with reddish-brown oxidation products of iron and associated microcracks, which are also filled with the oxidation products (marked by arrows). Rest of the coarse aggregate particles are **crushed micaceous quartzite**, which are dense, hard, equigranular to elongated, massive quartzitic (granoblastic) textured mass of major amount of quartz, subordinate feldspar (albite), and parallel bands of muscovite and chlorite, having a nominal maximum size of 12.5 mm.

Thin Section of Sample LHG at PPL (Top) & XPL (Bottom)

Aggregate has major amount of Micaceous Quartzite *and minor amount of* Phyllite *(P)*

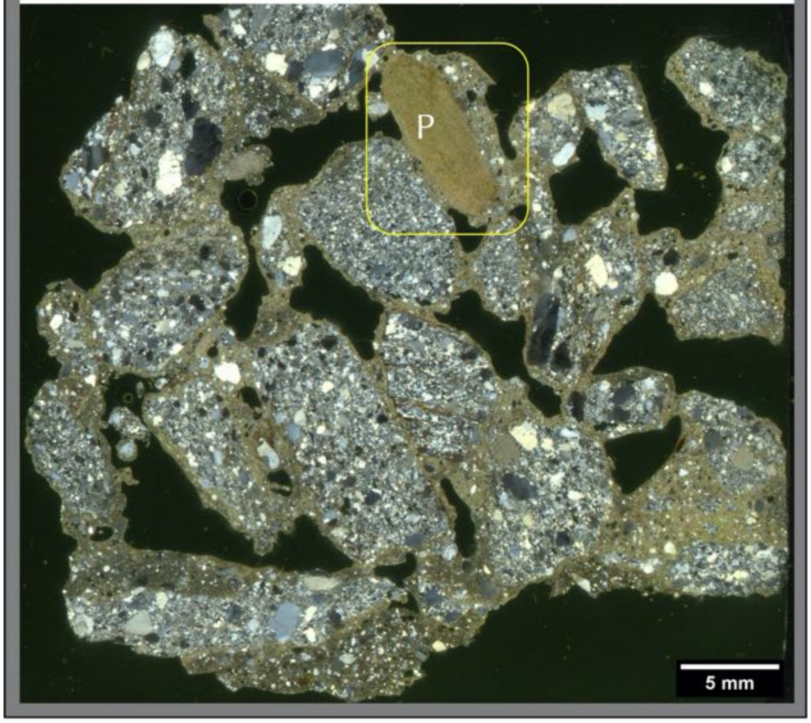


Figure 18: Enlarged view of the plane (top) and cross (bottom) polarized light images from Figure 15 of LHG showing one potentially deleterious fine-grained phyllite (P) coarse aggregate depicting foliated texture, and major amount of **crushed micaceous quartzite** coarse aggregate particles, which are dense, hard, equigranular to elongated, massive quartzitic (granoblastic) textured mass of major amount of quartz, subordinate feldspar (albite), and parallel bands of muscovite and chlorite, having a nominal maximum size of 12.5 mm. Interstitial paste in the mortar fraction shows beige color tone. Coarse irregular-shaped interstitial voids are calculated (from ImageJ) to be less than 10 percent.

TRANSMITTED-LIGHT OPTICAL MICROSCOPY

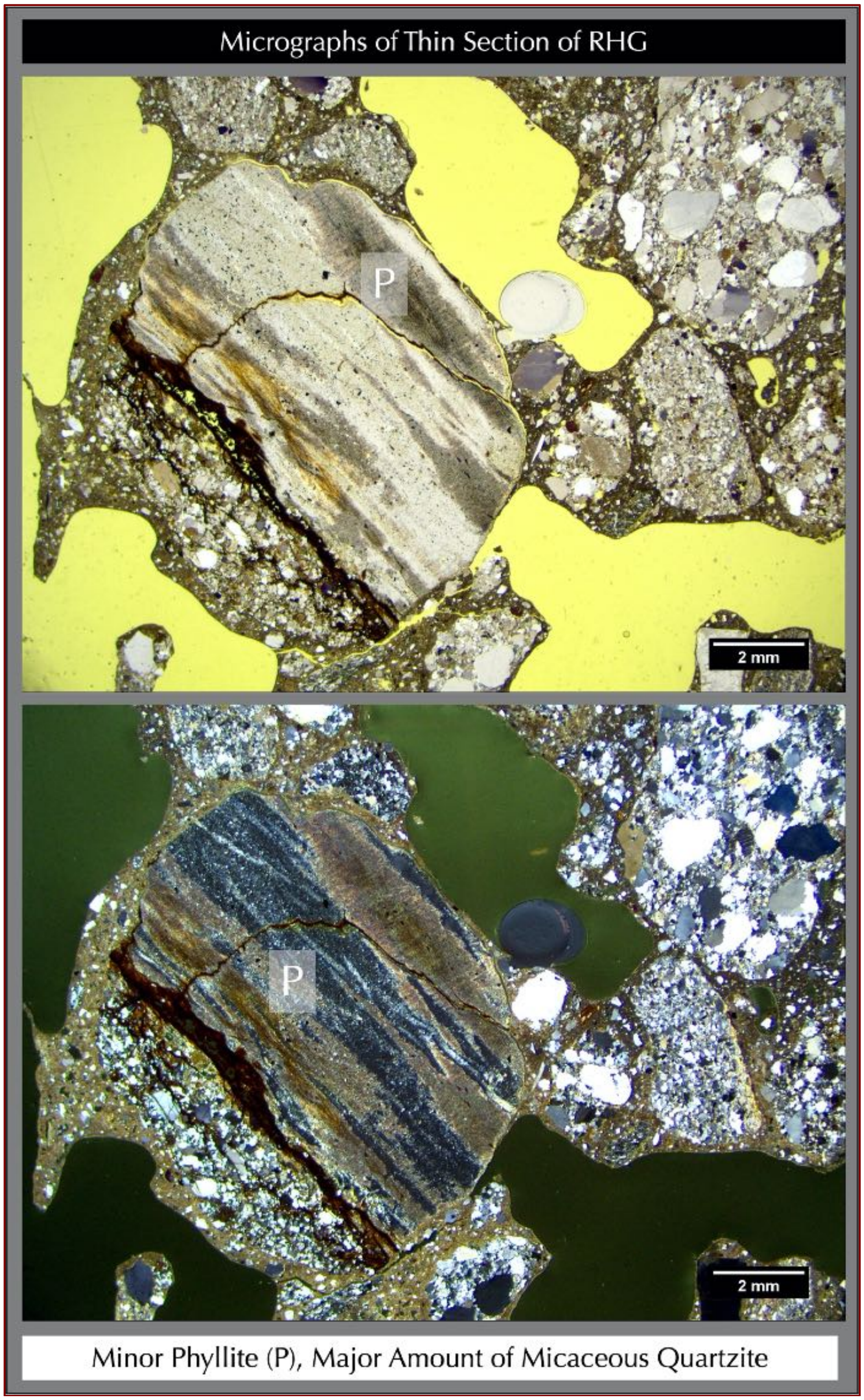
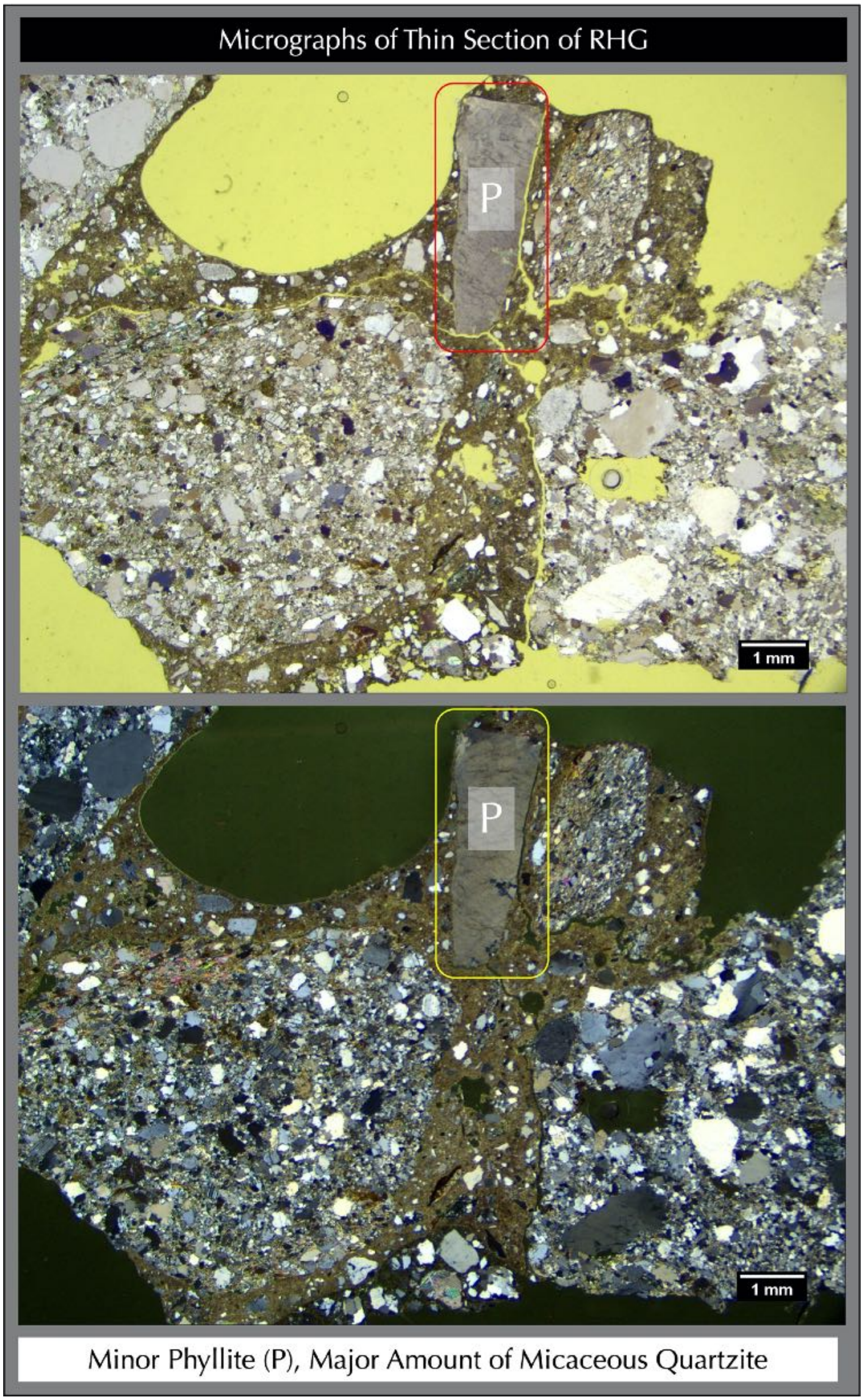


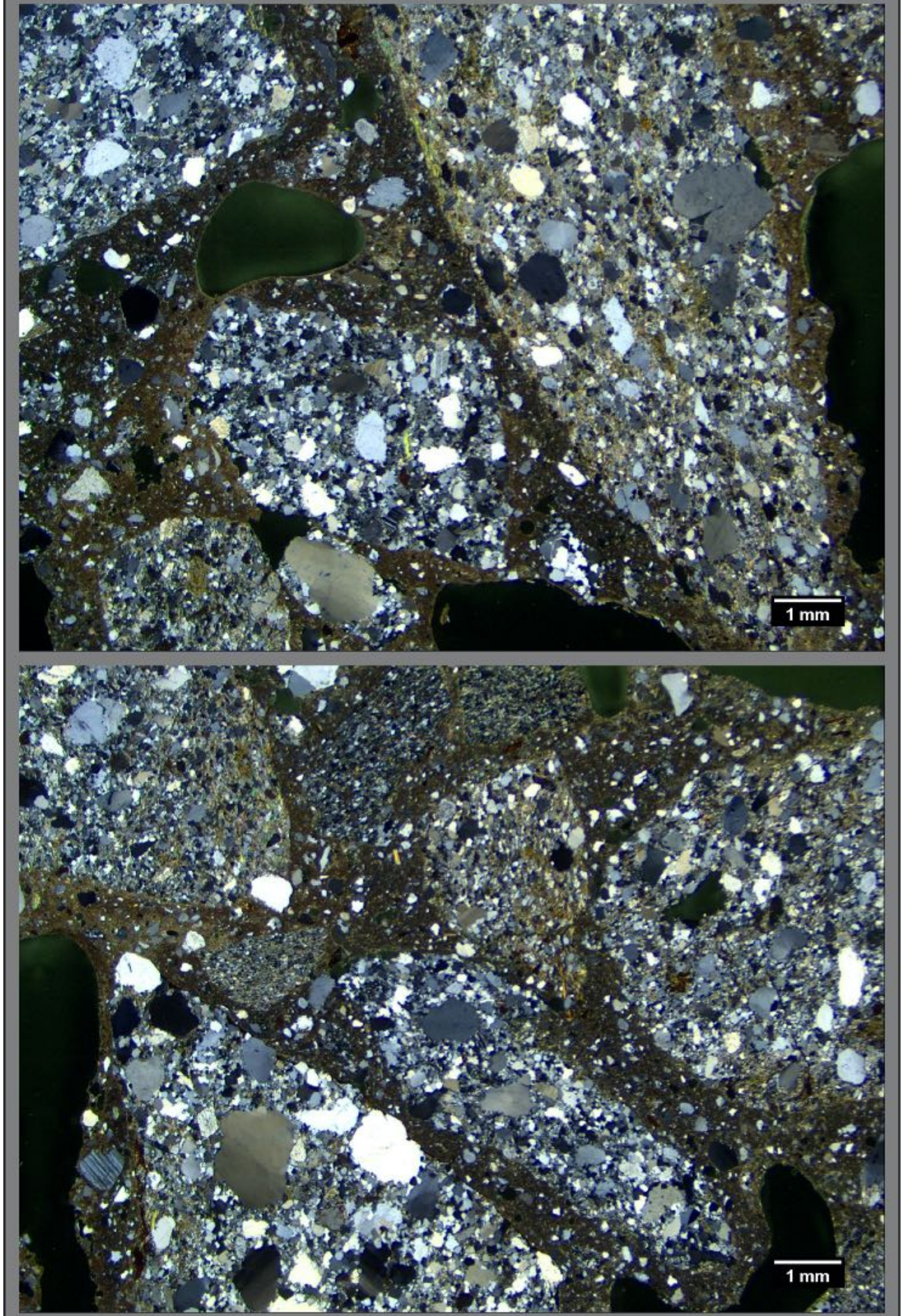
Figure 19: Thin section micrographs of block fragment RHG in PPL (top) and corresponding XPL (bottom) modes showing minor amount of phyllite (P) and major amount of micaceous quartzite grains in coarse aggregate. Phyllite shows characteristic fine-grained foliated texture, whereas dominant micaceous quartzite grains show massive quartzitic (granoblastic) textured mass of major amount of quartz, subordinate feldspar (albite), and parallel bands of muscovite and chlorite. Interstitial paste in the mortar fraction shows carbonated nature.


Micrographs of Thin Section of RHG

Minor Phyllite (P), Major Amount of Micaceous Quartzite

Figure 20: Thin section micrographs of block fragment RHG in PPL (top) and corresponding XPL (bottom) modes showing minor amount of phyllite (P) and major amount of micaceous quartzite grains in coarse aggregate. Phyllite shows characteristic fine-grained foliated texture, whereas dominant micaceous quartzite grains show massive quartzitic (granoblastic) textured mass of major amount of quartz, subordinate feldspar (albite), and parallel bands of muscovite and chlorite. Interstitial paste in the mortar fraction shows carbonated nature.

Micrographs of Thin Section of RHG



Coarse aggregate particles are crushed, 12.5-mm maximum size
Micaceous Quartzite

Figure 21: Thin section micrographs of block fragment RHG showing dominant micaceous quartzite grains in coarse aggregate depicting massive quartzitic (granoblastic) textured mass of major amount of quartz, subordinate feldspar (albite), and parallel bands of muscovite and chlorite. Interstitial paste in the mortar fraction shows carbonated nature.

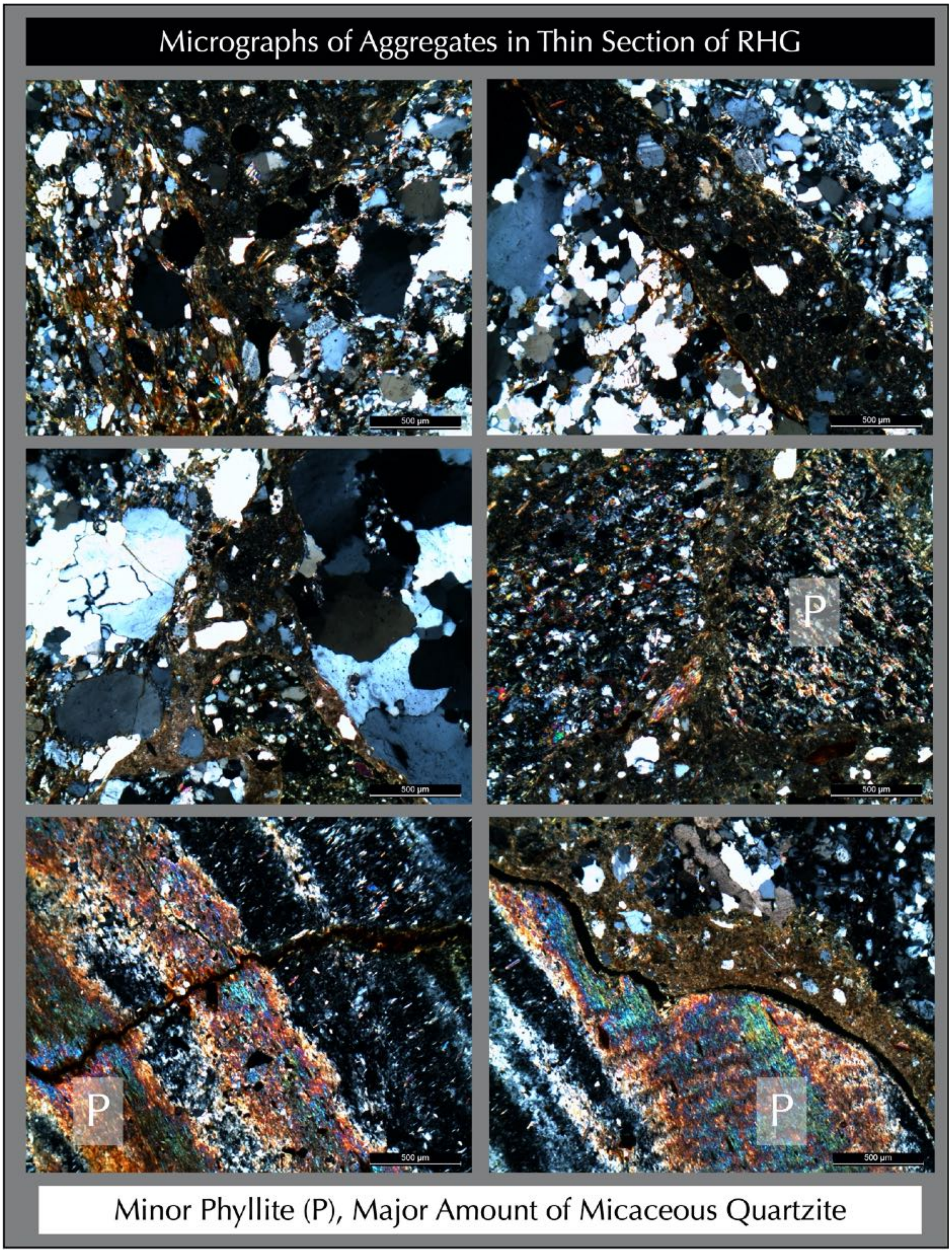


Figure 22: Thin section micrographs of block fragment RHG showing minor amount of phyllite (P) and major amount of micaceous quartzite grains in coarse aggregate. Phyllite shows characteristic fine-grained foliated texture, whereas dominant micaceous quartzite grains show massive quartzitic (granoblastic) textured mass of major amount of quartz, subordinate feldspar (albite), and parallel bands of muscovite and chlorite. Interstitial paste in the mortar fraction shows variably carbonated to non-carbonated nature.

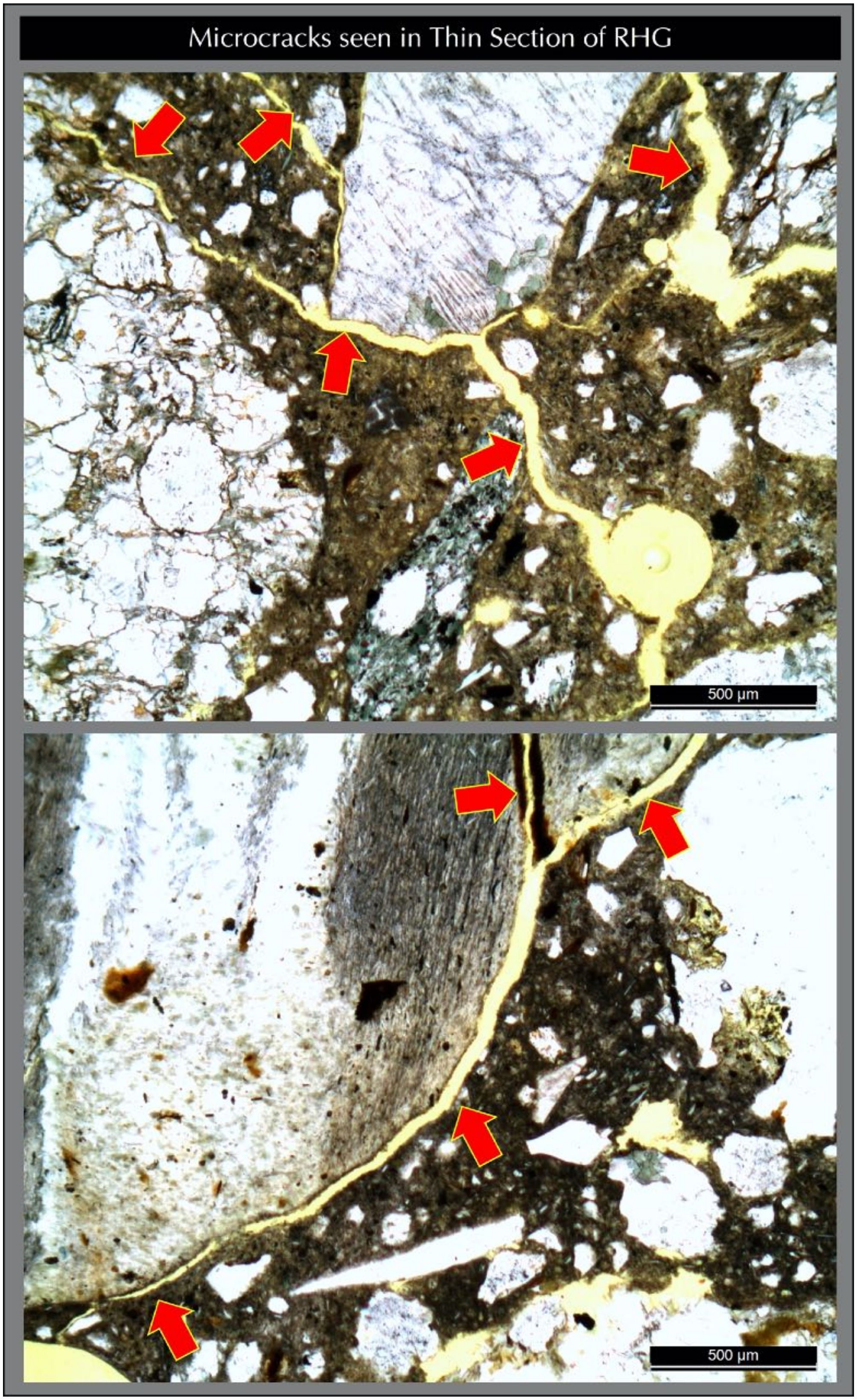


Figure 23: Thin section micrographs in PPL mode in RHG showing microcracks (marked by arrows) in mortar fraction and along the interface between a phyllite coarse aggregate and mortar fraction highlighted by fluorescent epoxy.

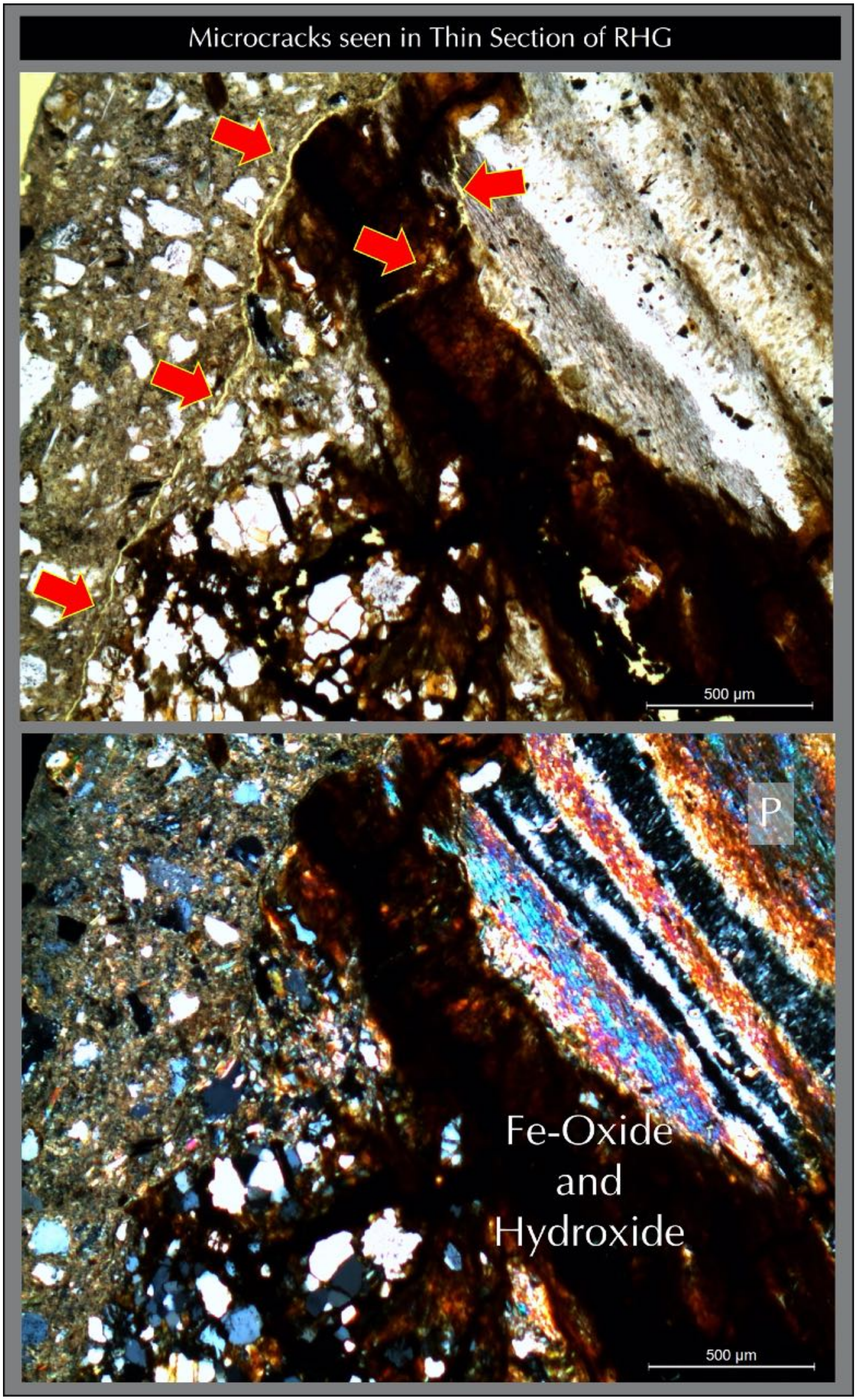


Figure 24: Thin section micrographs in PPL mode in RHG showing reddish-brown oxidation band in phyllite and microcrack (marked by arrows) at the interface to the mortar fraction.

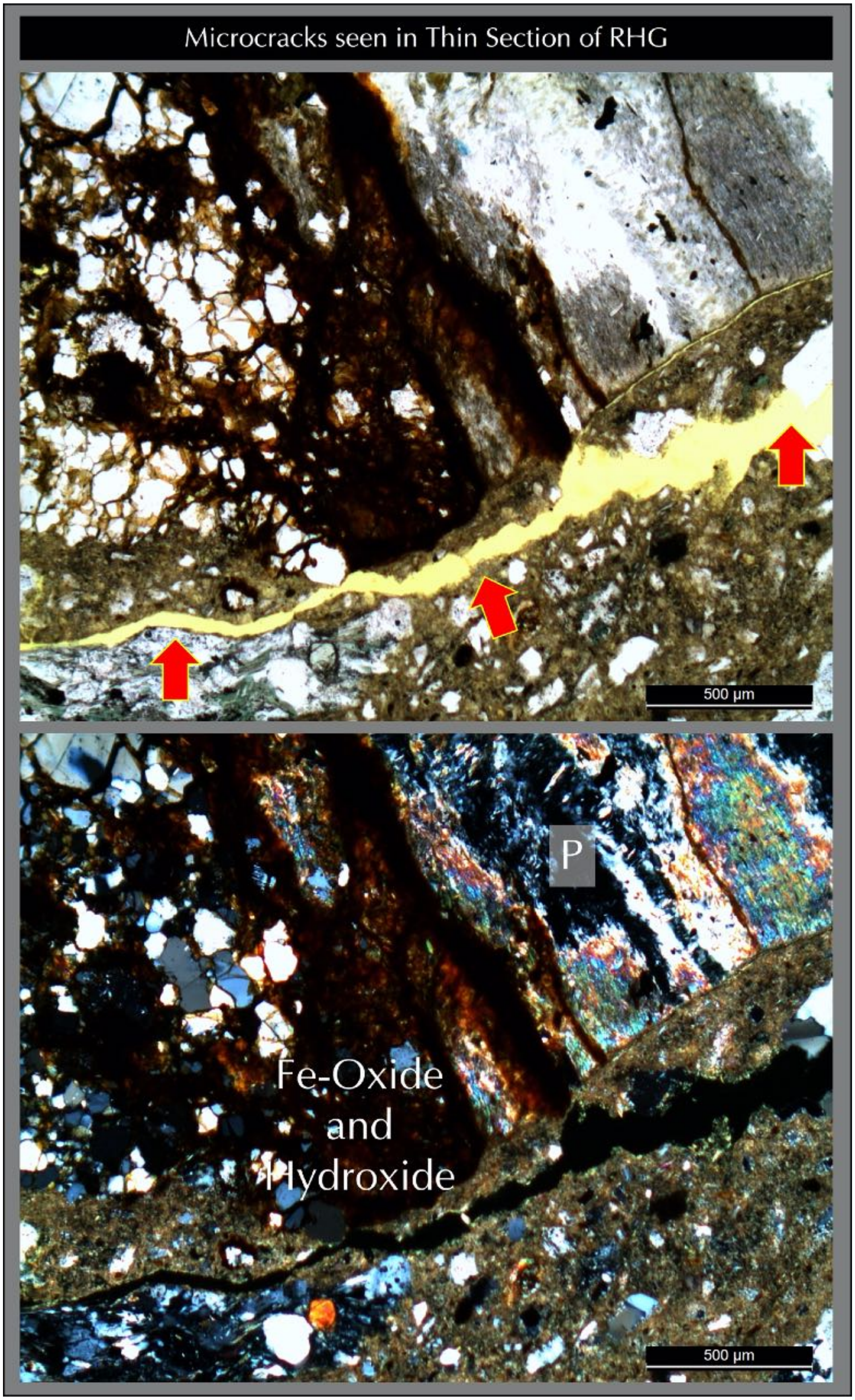


Figure 25: Thin section micrographs in PPL mode in RHG showing reddish-brown oxidation band in phyllite and microcrack (marked by arrows) at the interface to the mortar fraction.

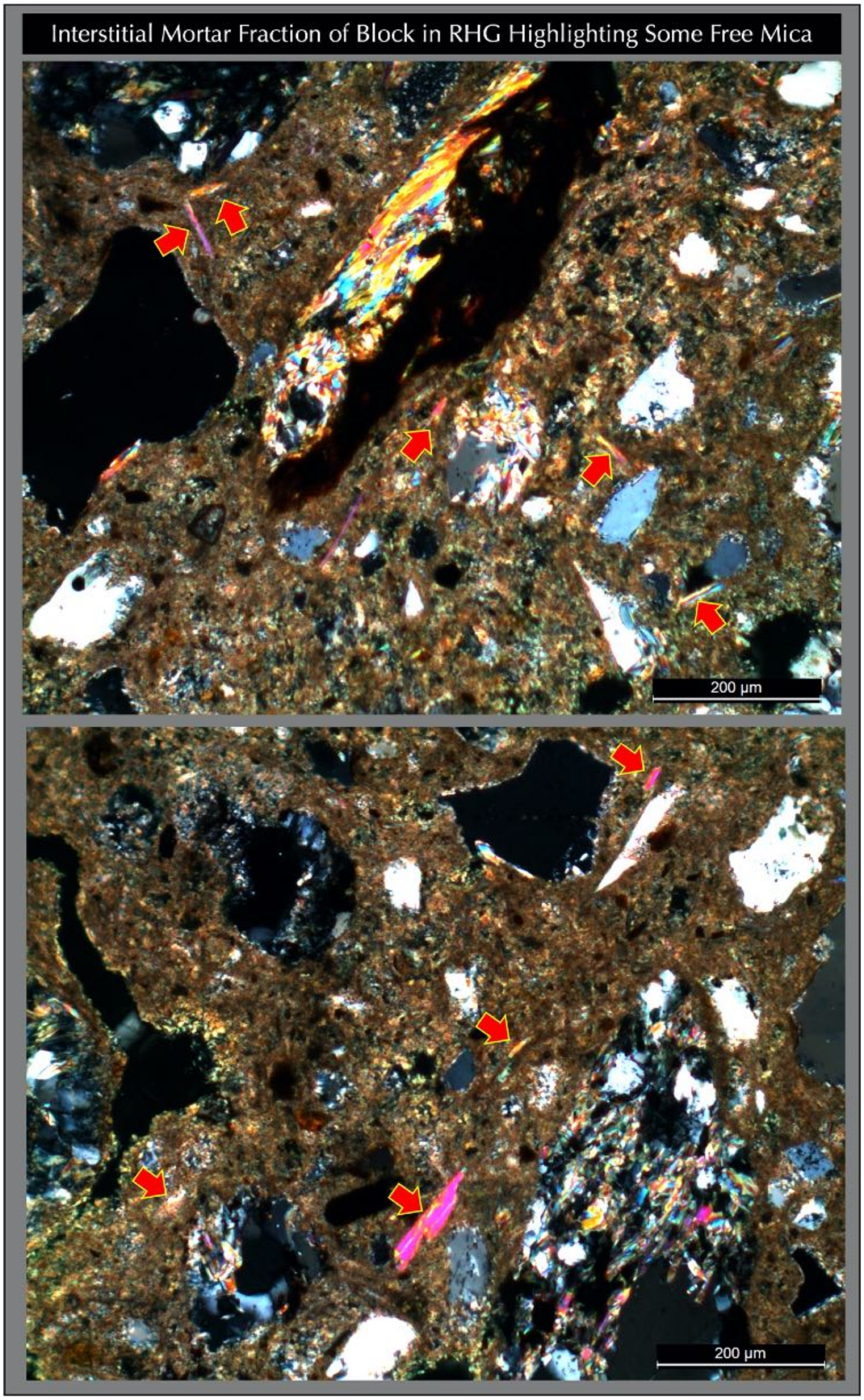
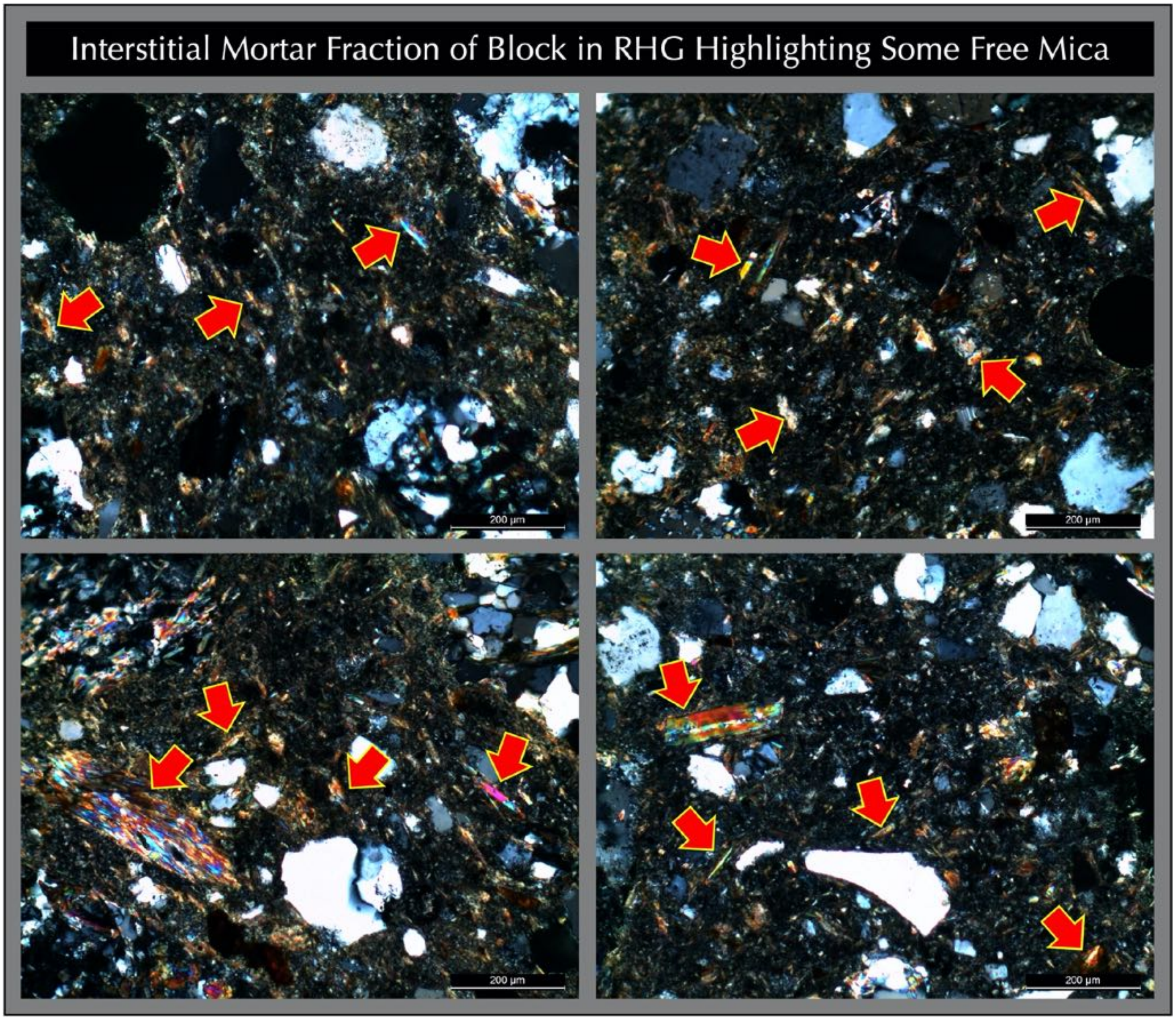


Figure 26: Micrographs of thin section of block fragment RHG showing the mortar fraction consisting of fine angular grains of quartz and feldspar, flakes of muscovite mica (free mica, a few are marked with arrows) and chlorite, and interstitial variably carbonated paste.



Interstitial Mortar Fraction of Block in RHG Highlighting Some Free Mica

Figure 27: Micrographs of thin section of block fragment RHG showing the mortar fraction consisting of fine angular grains of quartz and feldspar, flakes of muscovite mica (free mica, a few are marked with arrows) and chlorite, and interstitial non-carbonated paste (depicting typical near-isotropic nature of paste as opposed a carbonated paste of golden yellow interference color).

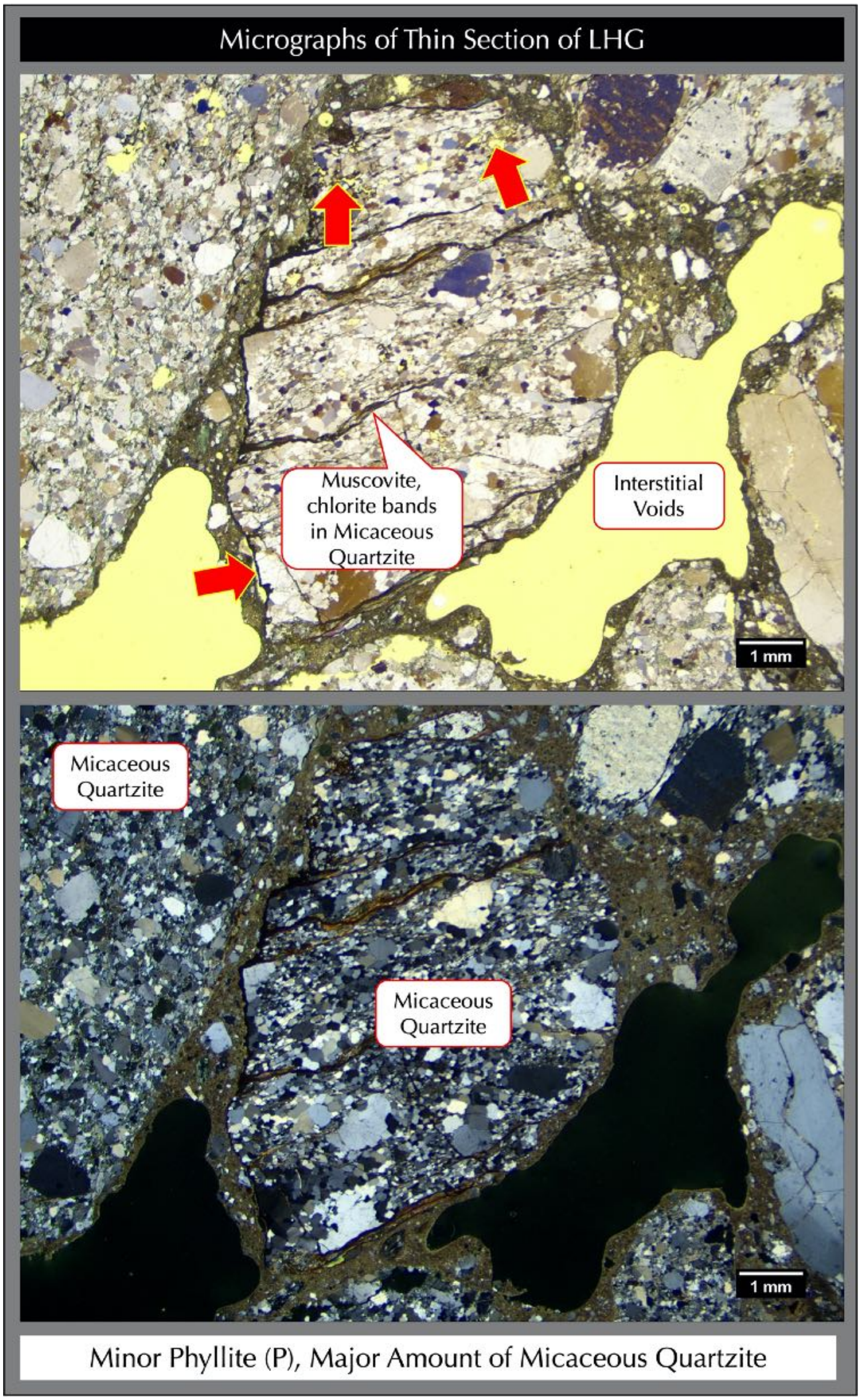


Figure 28: Thin section micrographs of block fragment LHG in PPL (top) and corresponding XPL (bottom) modes showing micaceous quartzite grains in coarse aggregate having massive quartzitic (granoblastic) textured mass of major amount of quartz, subordinate feldspar (albite), and parallel bands of muscovite and chlorite. Interstitial paste in the mortar fraction shows carbonated nature.

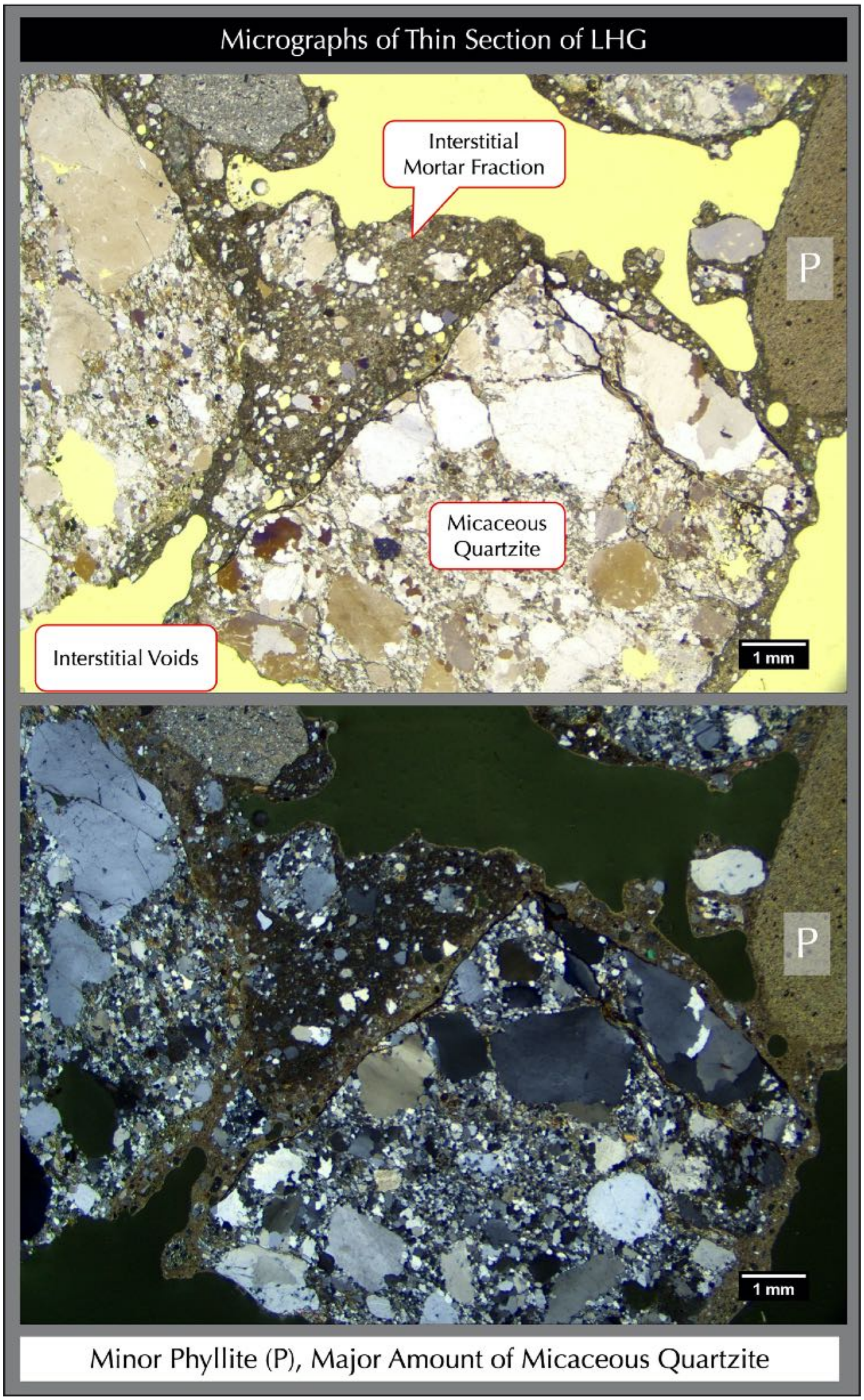


Figure 29: Thin section micrographs of block fragment LHG in PPL (top) and corresponding XPL (bottom) modes showing minor amount of phyllite (P) and major amount of micaceous quartzite grains in coarse aggregate. Phyllite shows characteristic fine-grained foliated texture, whereas dominant micaceous quartzite grains show massive quartzitic (granoblastic) textured mass of major amount of quartz, subordinate feldspar (albite), and parallel bands of muscovite and chlorite. Interstitial paste in the mortar fraction shows carbonated nature.

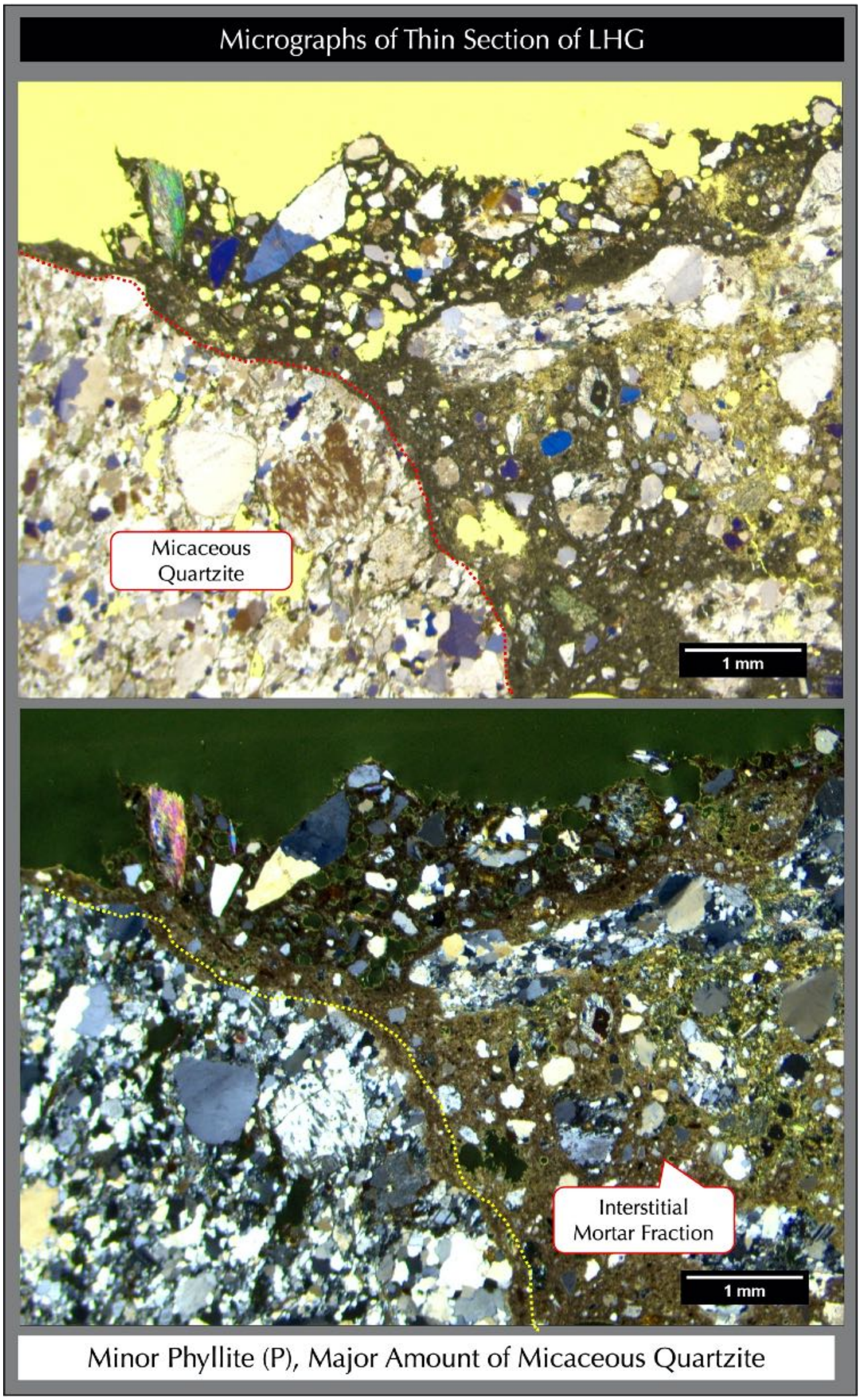
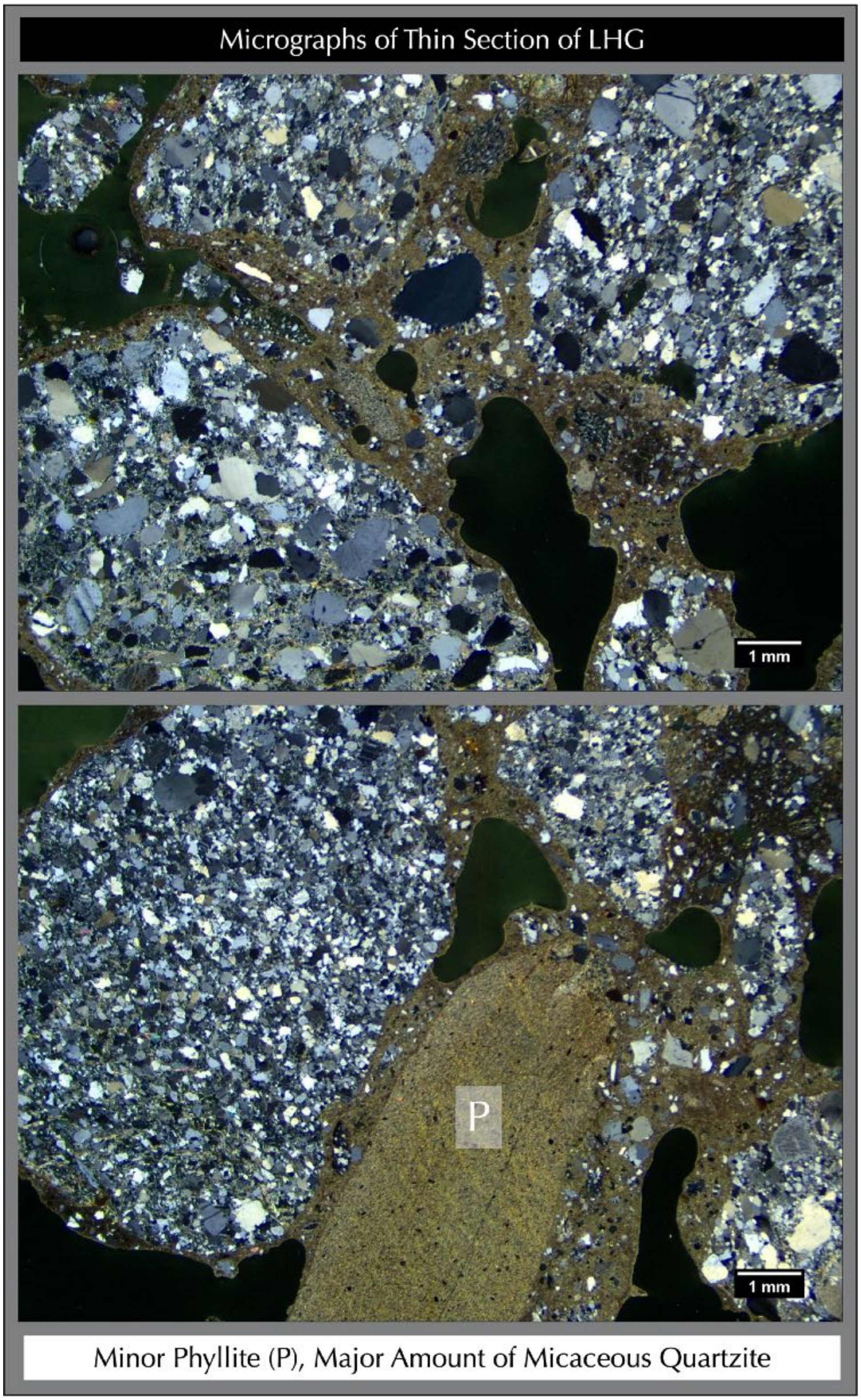


Figure 30: Thin section micrographs of block fragment LHG in PPL (top) and corresponding XPL (bottom) modes showing micaceous quartzite grains in coarse aggregate having massive quartzitic (granoblastic) textured mass of major amount of quartz, subordinate feldspar (albite), and parallel bands of muscovite and chlorite. Interstitial paste in the mortar fraction shows carbonated nature.


Micrographs of Thin Section of LHG

Minor Phyllite (P), Major Amount of Micaceous Quartzite

Figure 31: Thin section micrographs of block fragment LHG showing minor amount of phyllite (P) and major amount of micaceous quartzite grains in coarse aggregate. Phyllite shows characteristic fine-grained foliated texture, whereas dominant micaceous quartzite grains show massive quartzitic (granoblastic) textured mass of major amount of quartz, subordinate feldspar (albite), and parallel bands of muscovite and chlorite. Interstitial paste in the mortar fraction shows carbonated nature.

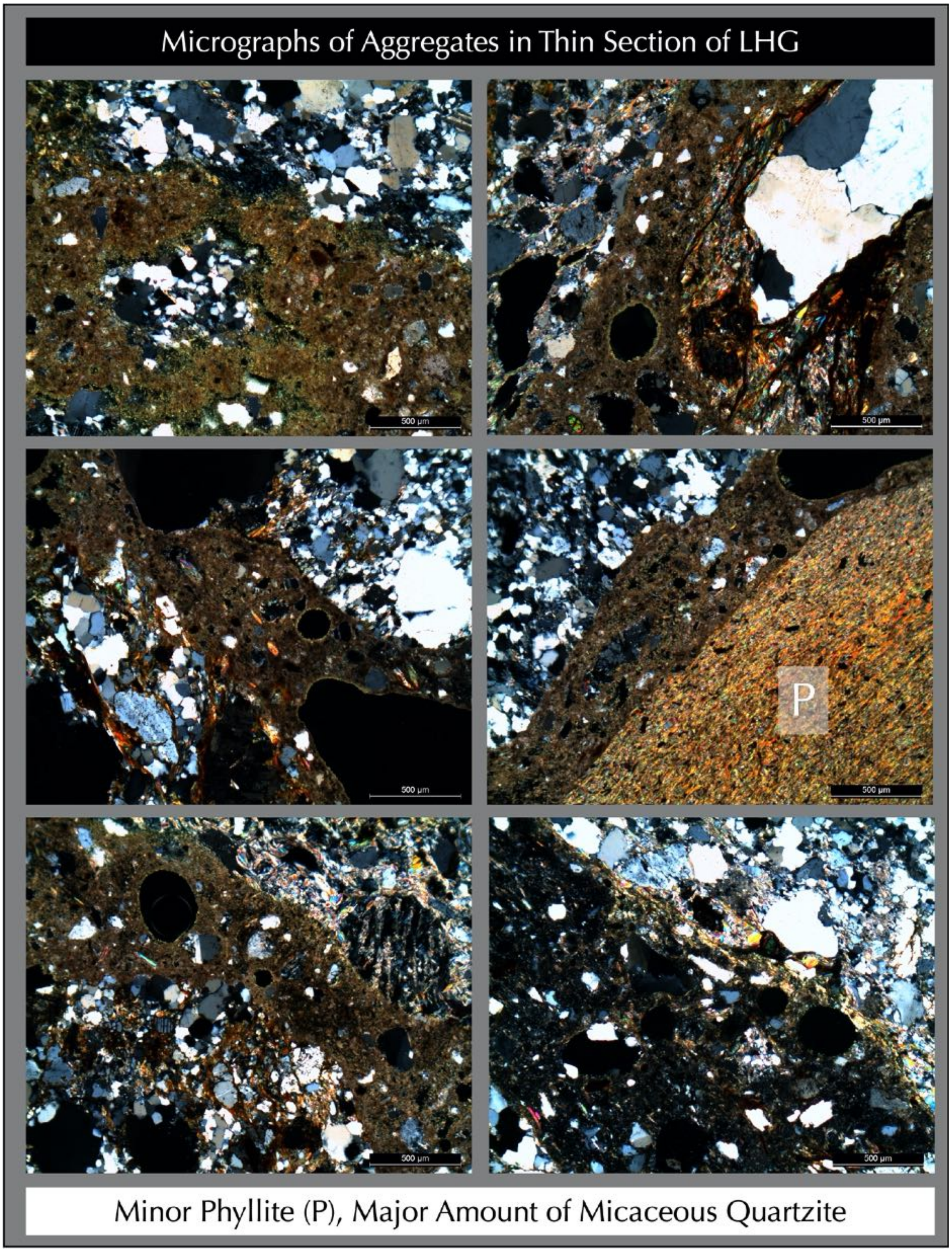


Figure 32: Thin section micrographs of block fragment LHG showing minor amount of phyllite (P) and major amount of micaceous quartzite grains in coarse aggregate. Phyllite shows characteristic fine-grained foliated texture, whereas dominant micaceous quartzite grains show massive quartzitic (granoblastic) textured mass of major amount of quartz, subordinate feldspar (albite), and parallel bands of muscovite and chlorite. Interstitial paste in the mortar fraction shows variably carbonated to non-carbonated nature.

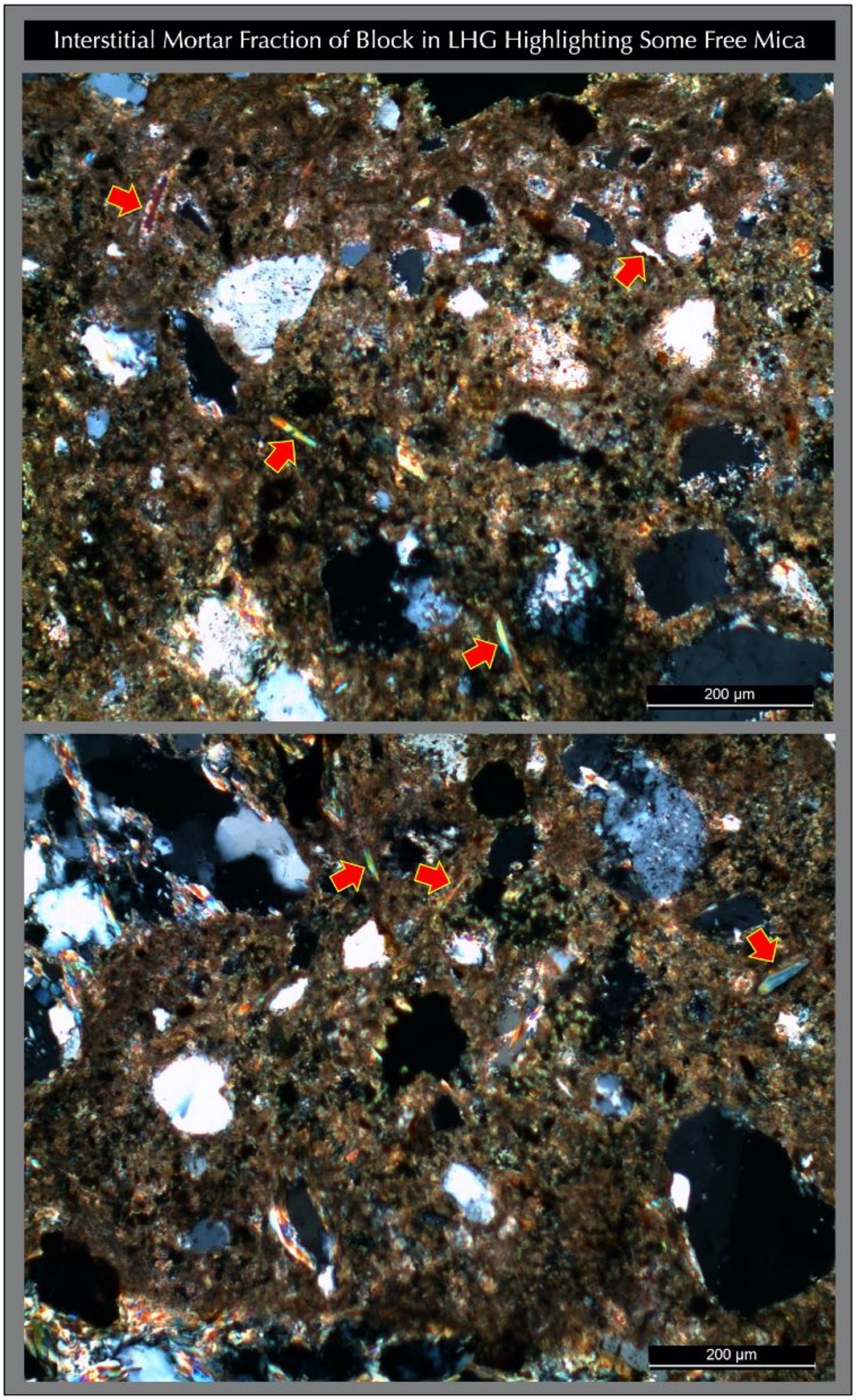


Figure 33: Micrographs of thin section of block fragment LHG showing the mortar fraction consisting of fine angular grains of quartz and feldspar, flakes of muscovite mica (free mica, a few are marked with arrows) and chlorite, and interstitial variably carbonated paste.

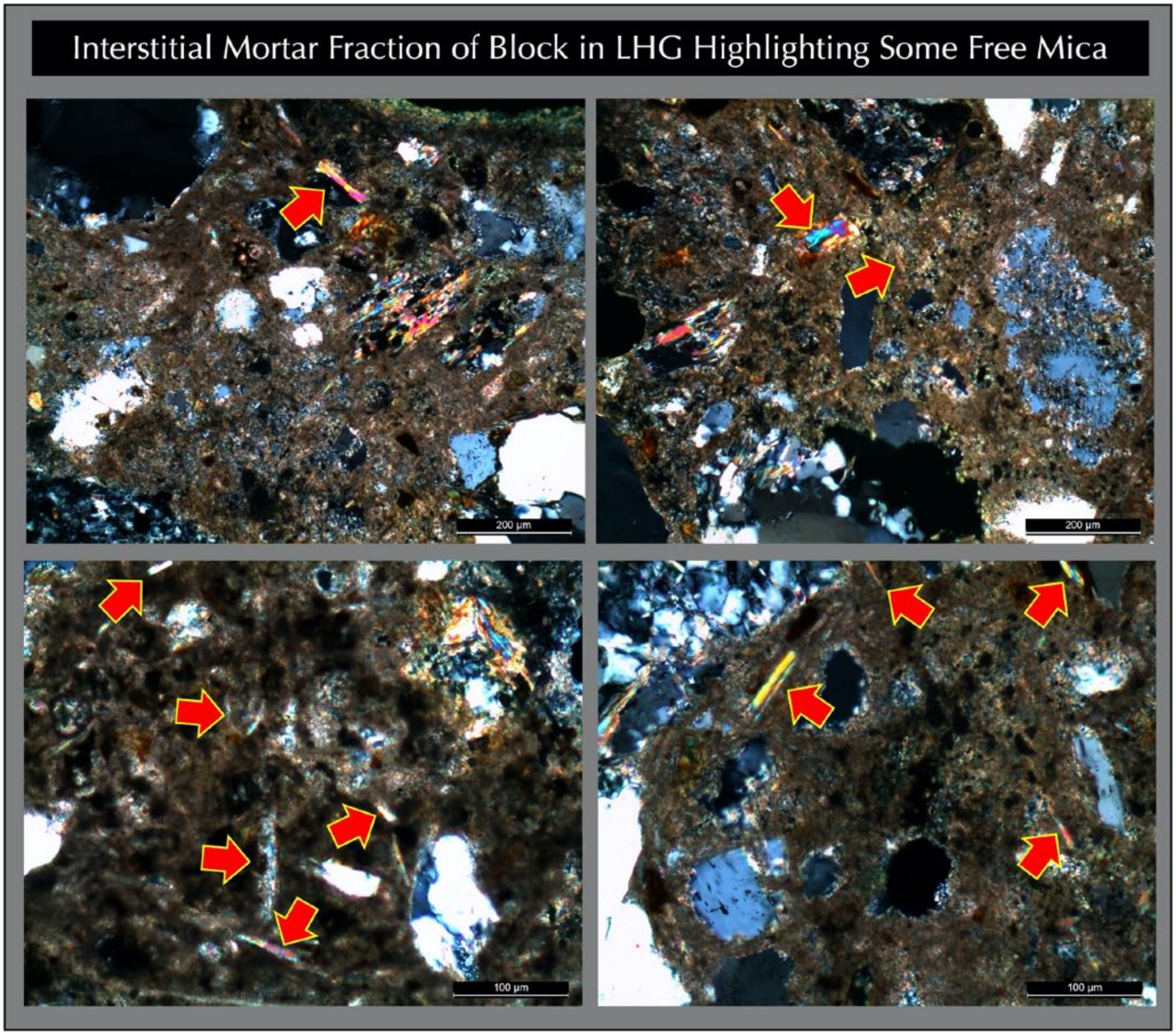


Figure 34: Micrographs of thin section of block fragment LHG showing the mortar fraction consisting of fine angular grains of quartz and feldspar, flakes of muscovite mica (free mica, a few are marked with arrows) and chlorite, and interstitial variably carbonated to non-carbonated paste (depicting typical near-isotropic nature of non-carbonated paste as opposed a carbonated paste of golden yellow interference color).

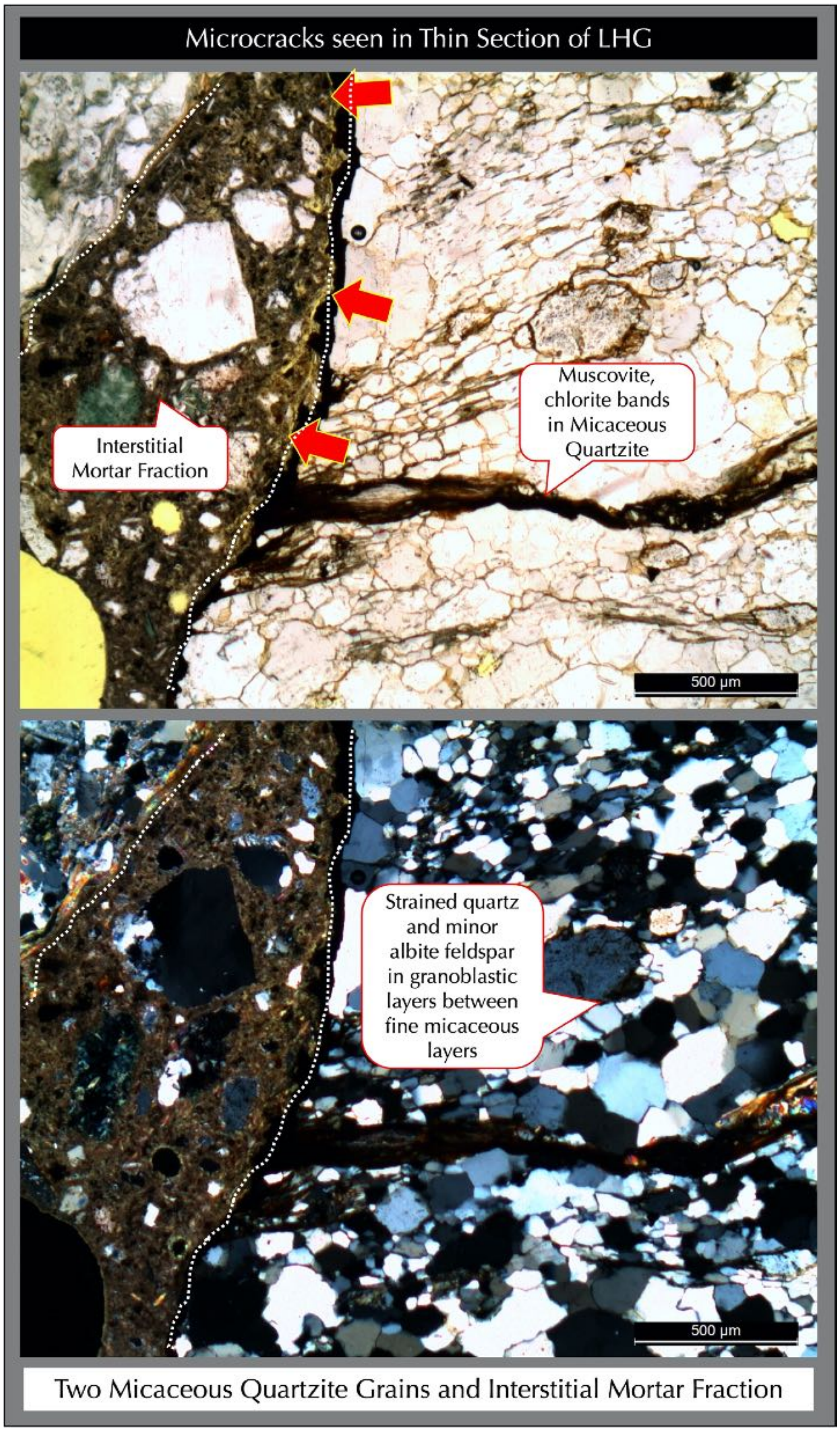


Figure 35: Thin section micrographs of block fragment LHG in PPL (top) and corresponding XPL (bottom) modes showing micaceous quartzite grains in coarse aggregate having massive quartzitic (granoblastic) textured mass of major amount of quartz, subordinate feldspar (albite), and parallel bands of muscovite and chlorite. Interstitial paste in the mortar fraction shows variably carbonated nature.

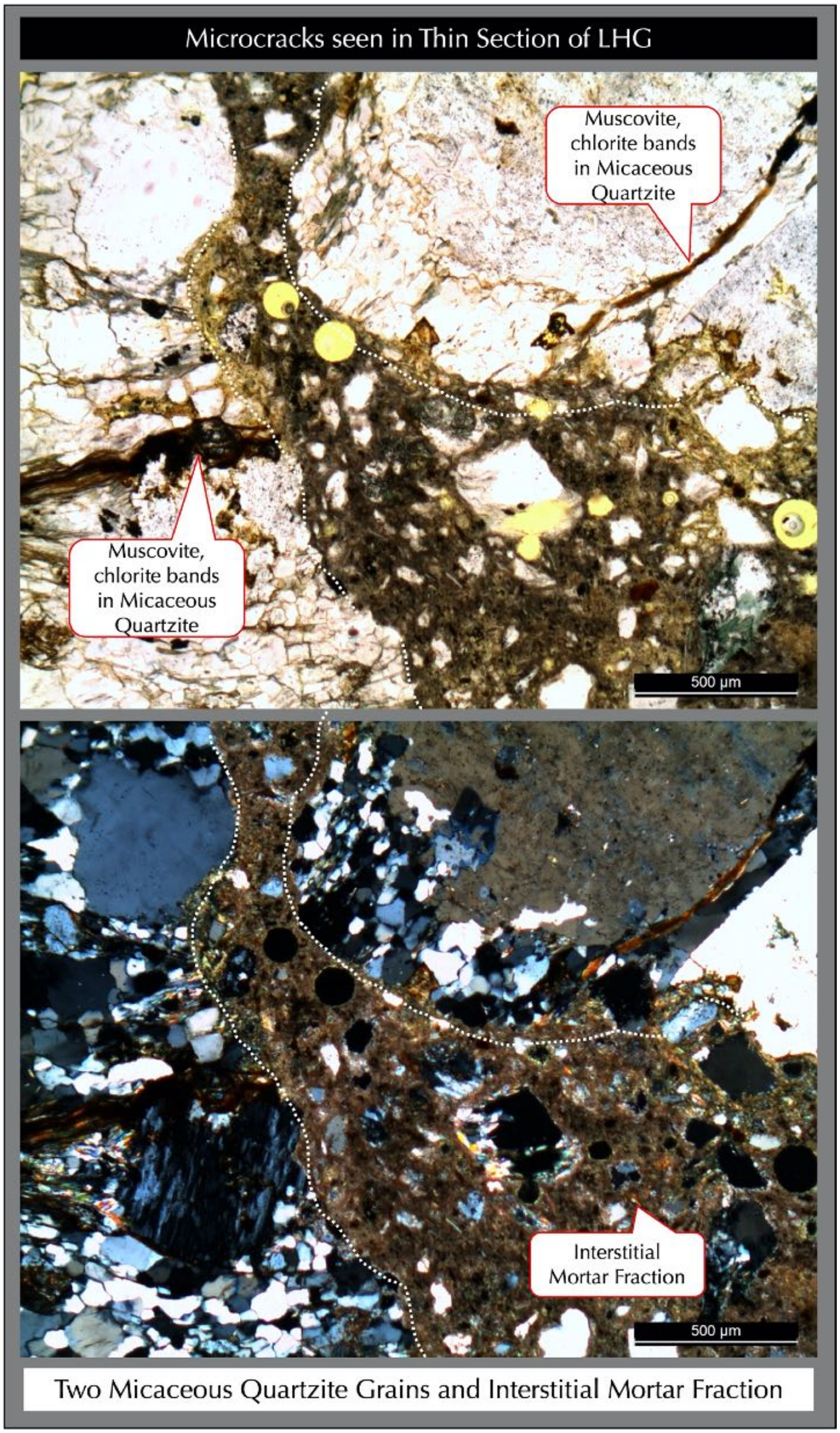


Figure 36: Thin section micrographs of block fragment LHG in PPL (top) and corresponding XPL (bottom) modes showing micaceous quartzite grains in coarse aggregate having massive quartzitic (granoblastic) textured mass of major amount of quartz, subordinate feldspar (albite), and parallel bands of muscovite and chlorite. Interstitial paste in the mortar fraction shows variably carbonated nature.

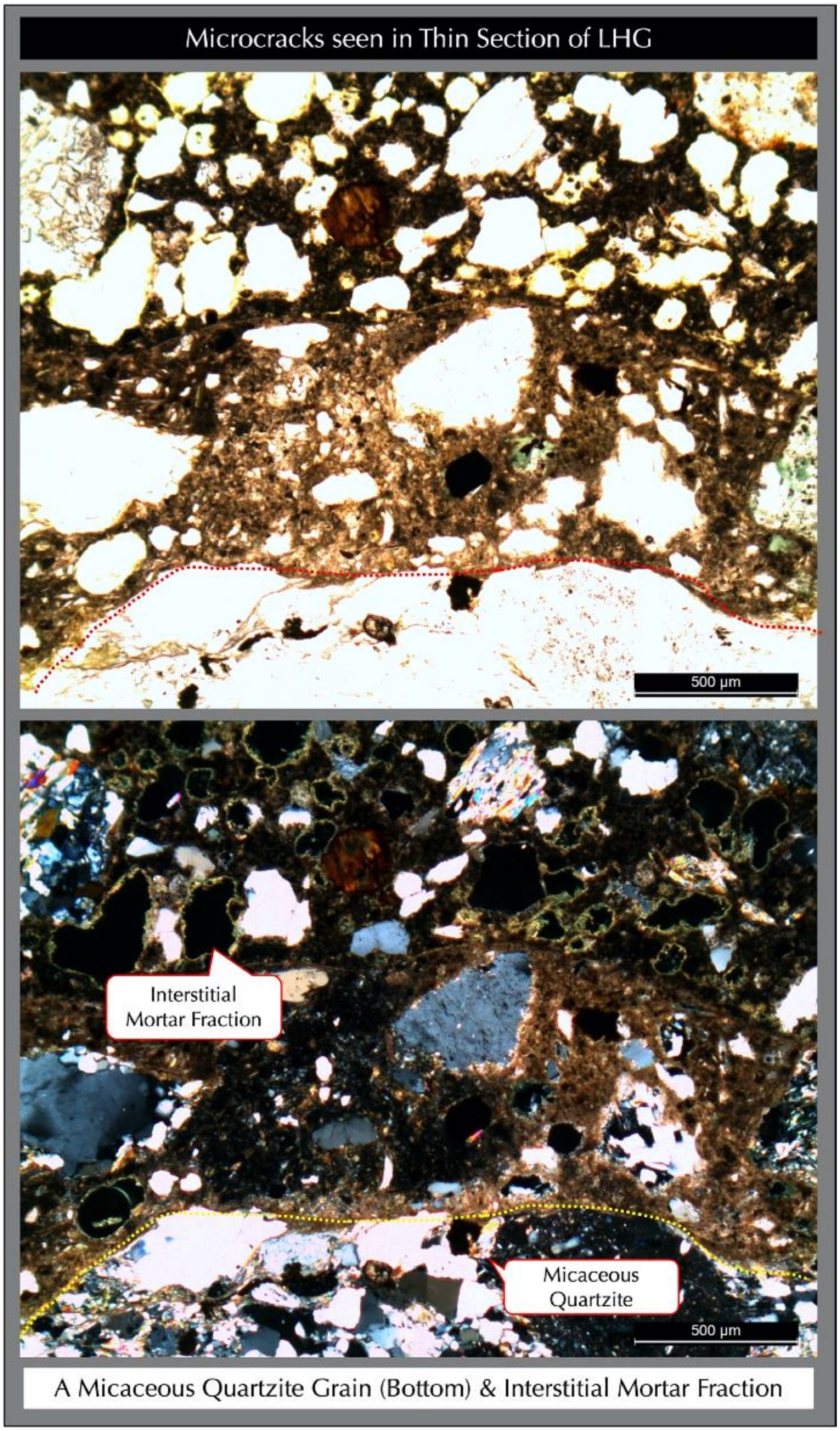


Figure 37: Thin section micrographs of block fragment LHG in PPL (top) and corresponding XPL (bottom) modes showing micaceous quartzite grains in coarse aggregate having massive quartzitic (granoblastic) textured mass of major amount of quartz, subordinate feldspar (albite), and parallel bands of muscovite and chlorite. Interstitial paste in the mortar fraction shows variably carbonated nature.

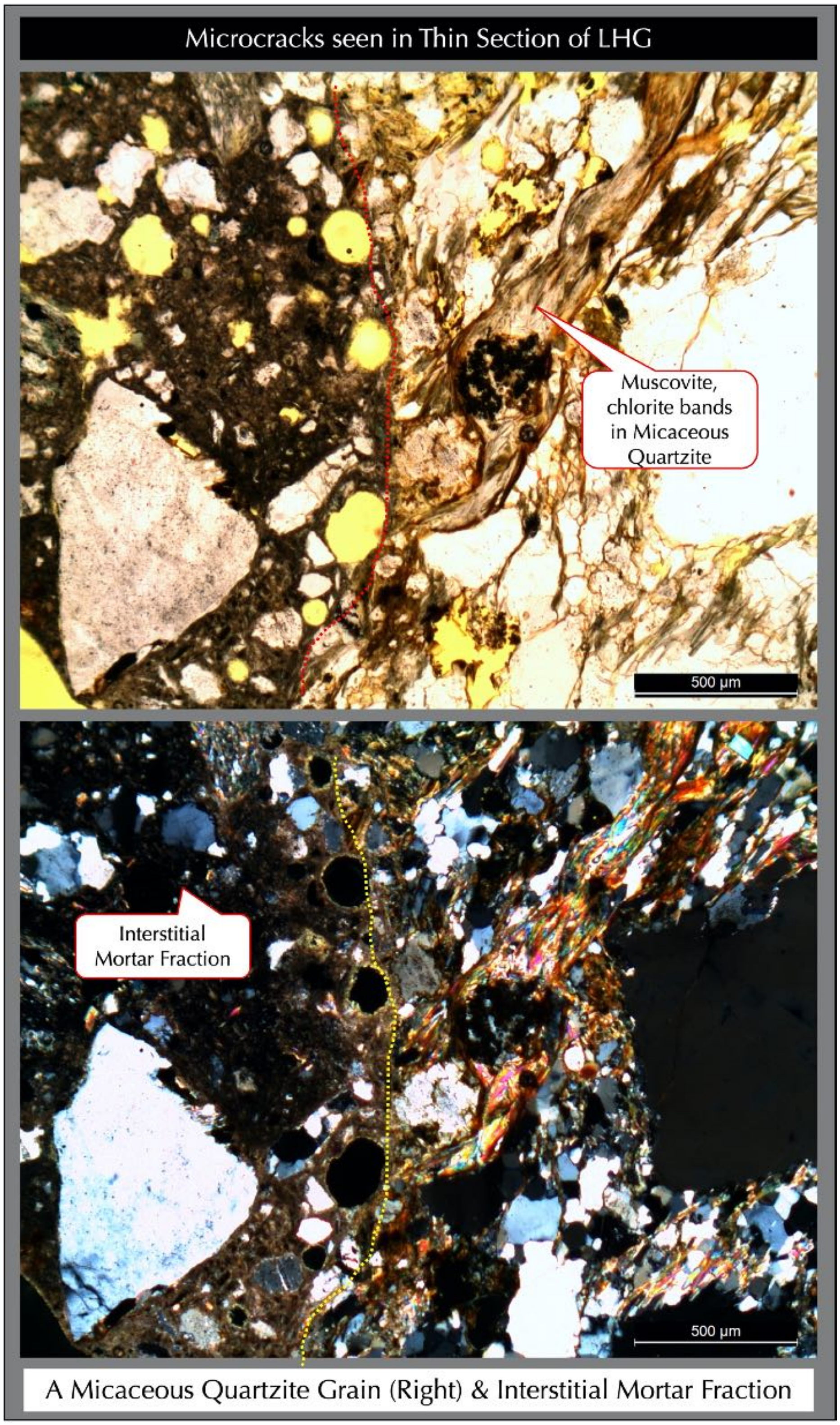


Figure 38: Thin section micrographs of block fragment LHG in PPL (top) and corresponding XPL (bottom) modes showing micaceous quartzite grains in coarse aggregate having massive quartzitic (granoblastic) textured mass of major amount of quartz, subordinate feldspar (albite), and parallel bands of muscovite and chlorite. Interstitial paste in the mortar fraction shows non-carbonated nature.

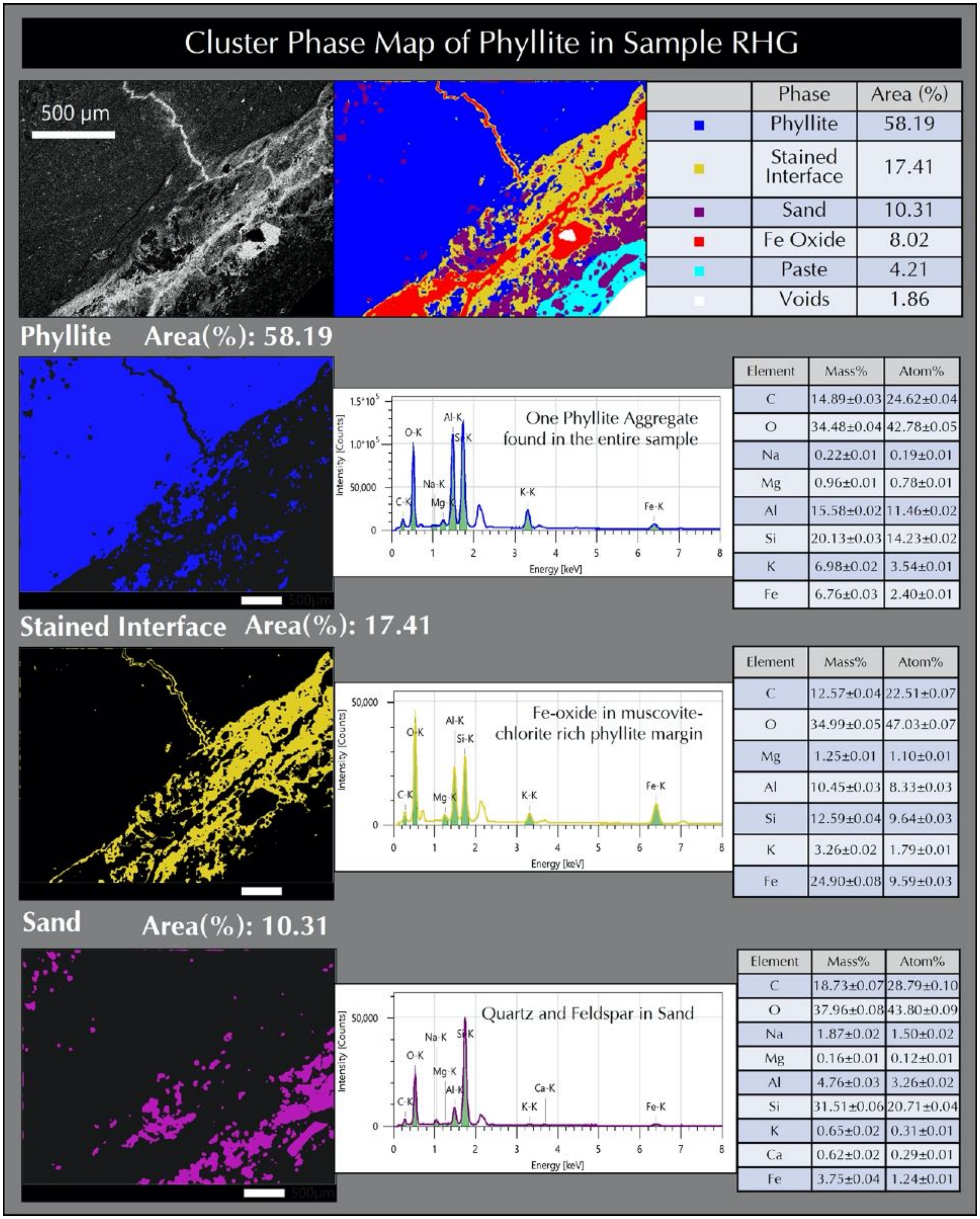
SUMMARY OF PETROGRAPHIC DATA

Properties and Compositions of Aggregates	Cores LHG and RHG
Coarse Aggregates	
Types	Crushed micaceous quartzite and minor phyllite (Figures 15-18)
Nominal maximum size (in.)	½ in. (12.5 mm, Figures 10-11, 15-18)
Rock Types	Major amount (> 95%) of micaceous quartzite grains having massive quartzitic (granoblastic) textured mass of major amount of quartz, subordinate feldspar (albite), and parallel bands of muscovite and chlorite; and minor (<5%, e.g., only 1 or 2 grains of phyllite in each sample) phyllite having fine-grained foliated textured parallel arrangement of muscovite, chlorite, quartz, and feldspar (Figures 15-18, 21-22, 31-32)
Angularity, Density, Hardness, Color, Texture, Sphericity	Angular, dense and hard for quartzite and soft and fragile for phyllite, medium gray, and equidimensional to elongated (Figures 10-11)
Cracking, Alteration, Coating	Unaltered to a few weathered, uncoated, and mostly uncracked (except some microcracks found in minor phyllite grains) (Figures 10-11, 15-18)
Grading & Distribution	Well-graded and well-distributed (Figures 10-11)
Soundness	Sound for dominant micaceous quartzite grains but unsound (cracking in some phyllite grains, Figures 19-20, 23, 25)
Alkali-Aggregate Reactivity	Despite the presence of many strained quartz particles in the aggregate that are known to be potentially alkali-silica reactive, there is, however, no evidence of such a reaction found in the aggregates
Sulfide Minerals in Coarse Aggregates	Isolated finely disseminated blocky habit pyrite (<0.1 mm grain size) and associated minor chalcopyrite mostly in the mortar fraction and preferentially concentrated in the minor phyllite grain compared to quartzite (Figures 24, 25, 32)
Fine Aggregates	
Types	Natural siliceous sand mixed with abraded micaceous quartzite (Figures 21-22, 31-34)
Nominal maximum size (in.)	¼ in. (6.5 mm)
Rock Types	Major amount of siliceous component (quartz, quartzite, strained quartzite, feldspar, and chert) and subordinate amount of abraded component of coarse aggregate that has also contributed to 'free mica' in the mortar fraction (Figures 21-22, 31-34)
Cracking, Alteration, Coating	Clear, off-white, light to medium to dark-gray, dense, hard, subrounded to rounded, equidimensional to elongated, mostly unaltered, uncoated, and mostly uncracked
Grading & Distribution	Well-graded and well-distributed (Figures 10-11, 21-22, 31-34)
Soundness	Sound
Alkali-Aggregate Reactivity	None
Sulfide Minerals in Fine Aggregates	Isolated finely disseminated blocky habit pyrite (<0.1 mm grain size) and associated minor chalcopyrite mostly in the mortar fraction and preferentially concentrated in the minor phyllite grain compared to quartzite (Figures 12, 13)

Properties and Compositions of Paste	Cores LHG and RHG
Color, Hardness, Porosity, Luster	Compositionally similar Portland cement-based paste that is moderately hard to moderately soft, porous, and depict subtranslucent and subvitreous luster
Residual Portland Cement Particles	Normal, 6 to 8 percent (mostly dark brown ferrite remains) by paste volumes in the non-carbonated portions
Calcium hydroxide from cement hydration	Normal, 8 to 10 percent by paste volume in the non-carbonated portions
Pozzolans, Slag, etc.	None
Water-cement ratios (*w/c*), estimated	0.50 to 0.55 (estimated in the non-carbonated portions)
Cement Contents (%)	Approximately 10 % by volume in both samples
Secondary Deposits	Other than occasional secondary calcite deposits in some voids most voids are found empty without any potentially deleterious ettringite, gypsum, or thaumasite deposits found in the other distressed homes
Paste Carbonation	Variable carbonation from severe carbonation at the exposed ends of core to moderate to no carbonation (Figures 17 to 20, 24 to 38)
Microcracking	Occasional shrinkage microcracks are seen in the mortar fractions of both cores particular in association with one or two phyllite grains in each sample (Figures 23 and 25 for RHG)
Aggregate-paste Bonds	Moderately Tight to Tight (around quartzite) to Weak (around Phyllite)
Chemical deteriorations	No evidence of any potentially deleterious reactions (e.g., ISA or ASR) is found
Sulfide Minerals in Mortar Fractions	Isolated finely disseminated block habit pyrite and minor chalcopyrite (Figures 12, 13)
Free Mica in Mortar Fractions	Approximately 10% (SEM-EDS images produced 7.2% in RGH and 10.5% in LHG

Air Voids	LHG	RHG
Air Entrainment	Marginally air-entrained	Marginally air Entrained
Total Void Contents (%, Estimated)	8 – 10 %	6 – 8 %
Void Distribution	Well-distributed	Well-distributed
Secondary Deposits in Air Voids	None, small spherical entrained voids are mostly free (Figures 36, 38)	None, small spherical entrained voids are mostly free (Figures 20, 23)

SEM-EDS



Cluster Phase Map of Phyllite in Sample RHG

	Phase	Area (%)
🟦	Phyllite	58.19
🟨	Stained Interface	17.41
🟪	Sand	10.31
🟥	Fe Oxide	8.02
■	Paste	4.21
⬜	Voids	1.86

Phyllite Area(%): 58.19

One Phyllite Aggregate found in the entire sample

Element	Mass%	Atom%
C	14.89±0.03	24.62±0.04
O	34.48±0.04	42.78±0.05
Na	0.22±0.01	0.19±0.01
Mg	0.96±0.01	0.78±0.01
Al	15.58±0.02	11.46±0.02
Si	20.13±0.03	14.23±0.02
K	6.98±0.02	3.54±0.01
Fe	6.76±0.03	2.40±0.01

Stained Interface Area(%): 17.41

Fe-oxide in muscovite-chlorite rich phyllite margin

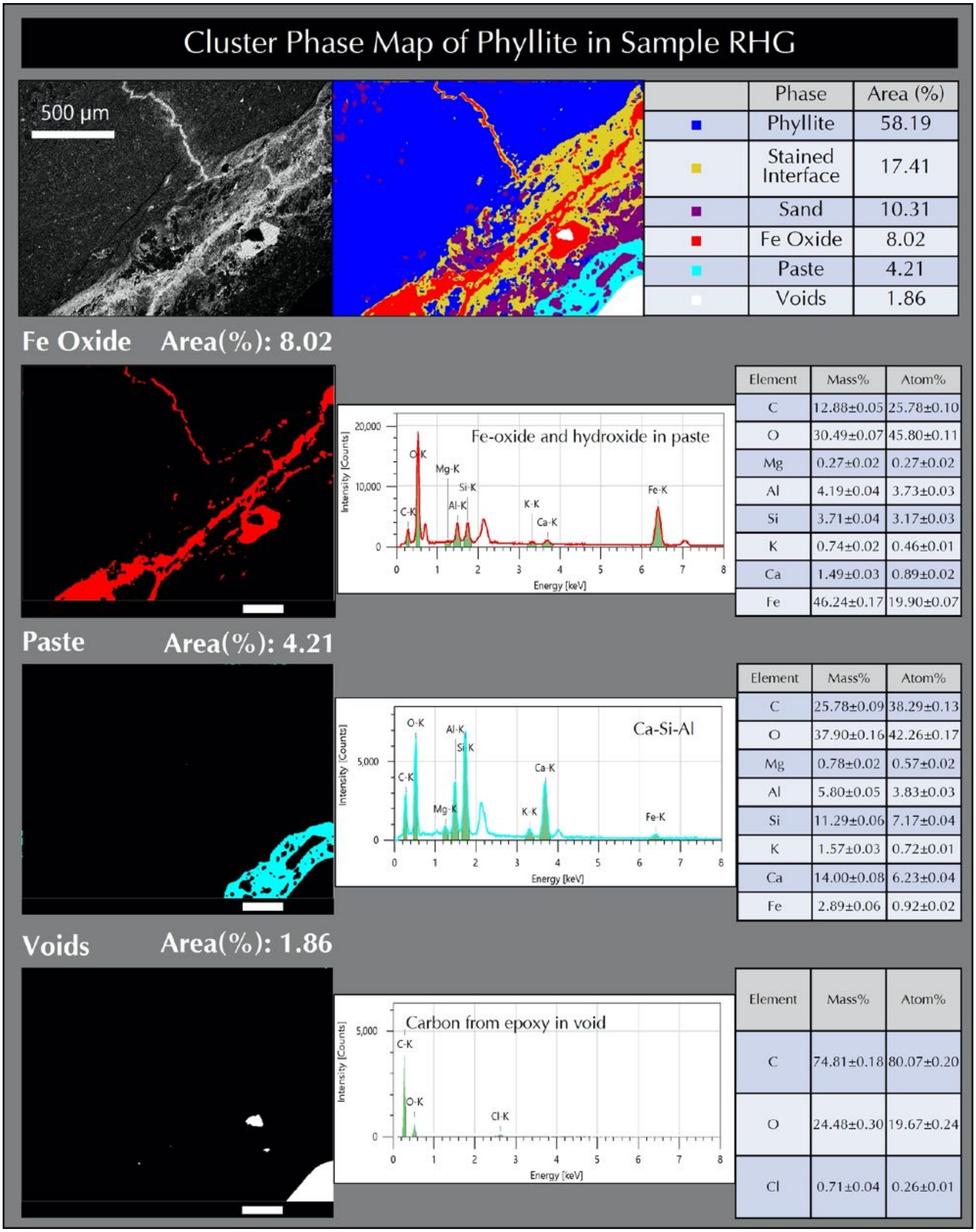
Element	Mass%	Atom%
C	12.57±0.04	22.51±0.07
O	34.99±0.05	47.03±0.07
Mg	1.25±0.01	1.10±0.01
Al	10.45±0.03	8.33±0.03
Si	12.59±0.04	9.64±0.03
K	3.26±0.02	1.79±0.01
Fe	24.90±0.08	9.59±0.03

Sand Area(%): 10.31

Quartz and Feldspar in Sand

Element	Mass%	Atom%
C	18.73±0.07	28.79±0.10
O	37.96±0.08	43.80±0.09
Na	1.87±0.02	1.50±0.02
Mg	0.16±0.01	0.12±0.01
Al	4.76±0.03	3.26±0.02
Si	31.51±0.06	20.71±0.04
K	0.65±0.02	0.31±0.01
Ca	0.62±0.02	0.29±0.01
Fe	3.75±0.04	1.24±0.01

Figure 39: Backscatter electron image (top left), cluster phase map of phyllite aggregate and adjacent mortar fraction (top middle), phase proportions calculated from phase map (top right), and segmented phase maps, corresponding EDS spectrum and oxide composition of various phases of block fragment RHG in a polished solid section. Phase colors: blue – phyllite, yellow – stained interface, and purple - sand.



	Phase	Area (%)
🟦	Phyllite	58.19
🟨	Stained Interface	17.41
🟪	Sand	10.31
🟥	Fe Oxide	8.02
■	Paste	4.21
⬜	Voids	1.86

Element	Mass%	Atom%
C	12.88±0.05	25.78±0.10
O	30.49±0.07	45.80±0.11
Mg	0.27±0.02	0.27±0.02
Al	4.19±0.04	3.73±0.03
Si	3.71±0.04	3.17±0.03
K	0.74±0.02	0.46±0.01
Ca	1.49±0.03	0.89±0.02
Fe	46.24±0.17	19.90±0.07

Element	Mass%	Atom%
C	25.78±0.09	38.29±0.13
O	37.90±0.16	42.26±0.17
Mg	0.78±0.02	0.57±0.02
Al	5.80±0.05	3.83±0.03
Si	11.29±0.06	7.17±0.04
K	1.57±0.03	0.72±0.01
Ca	14.00±0.08	6.23±0.04
Fe	2.89±0.06	0.92±0.02

Element	Mass%	Atom%
C	74.81±0.18	80.07±0.20
O	24.48±0.30	19.67±0.24
Cl	0.71±0.04	0.26±0.01

Figure 40: Backscatter electron image (top left), cluster phase map of phyllite aggregate and adjacent mortar fraction (top middle), phase proportions calculated from phase map (top right), and segmented phase maps, corresponding EDS spectrum and oxide composition of various phases of block fragment RHG in a polished solid section. Phase colors: red – Fe oxide, cyan - paste, blue – quartz, and white – voids.

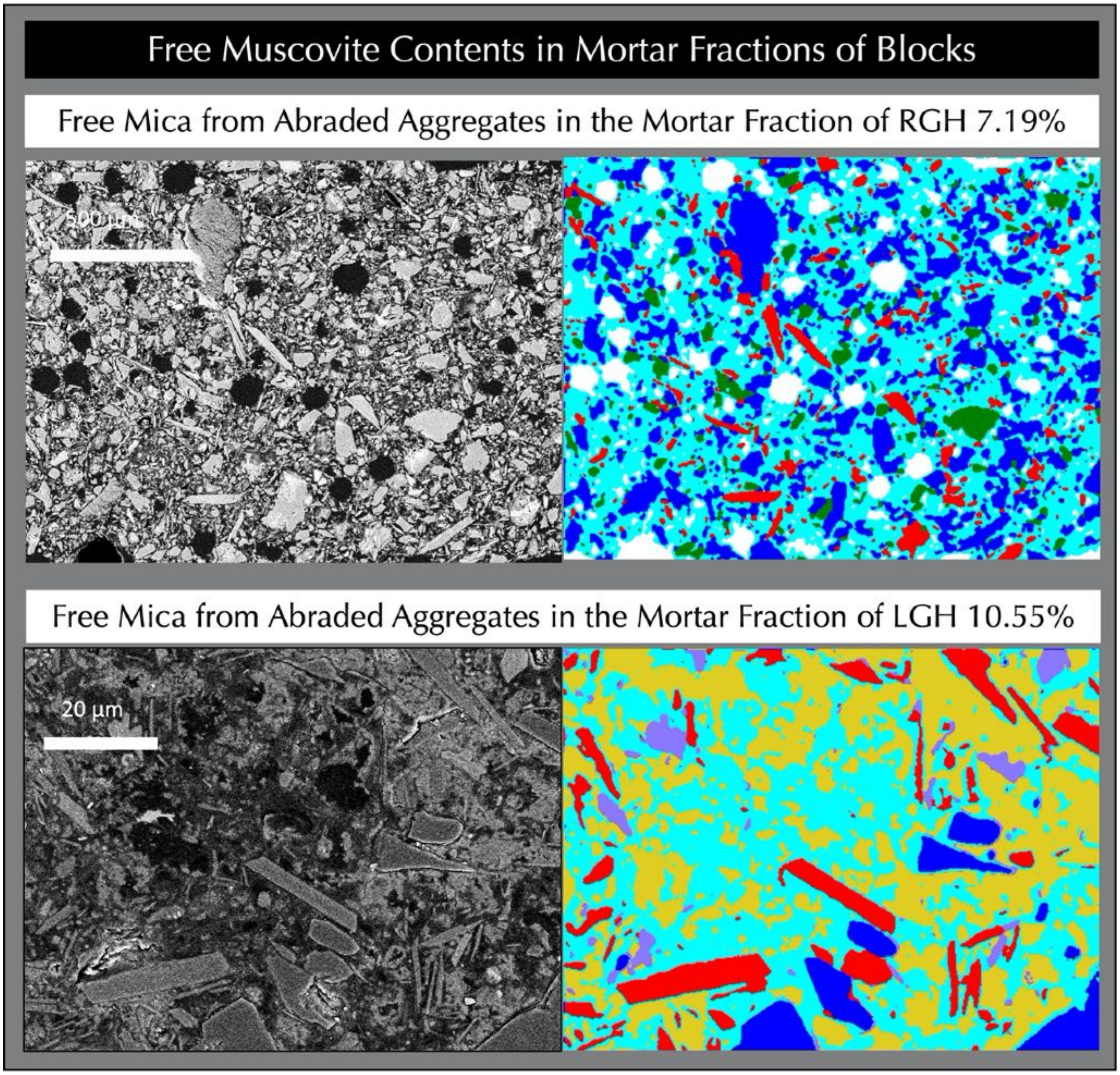
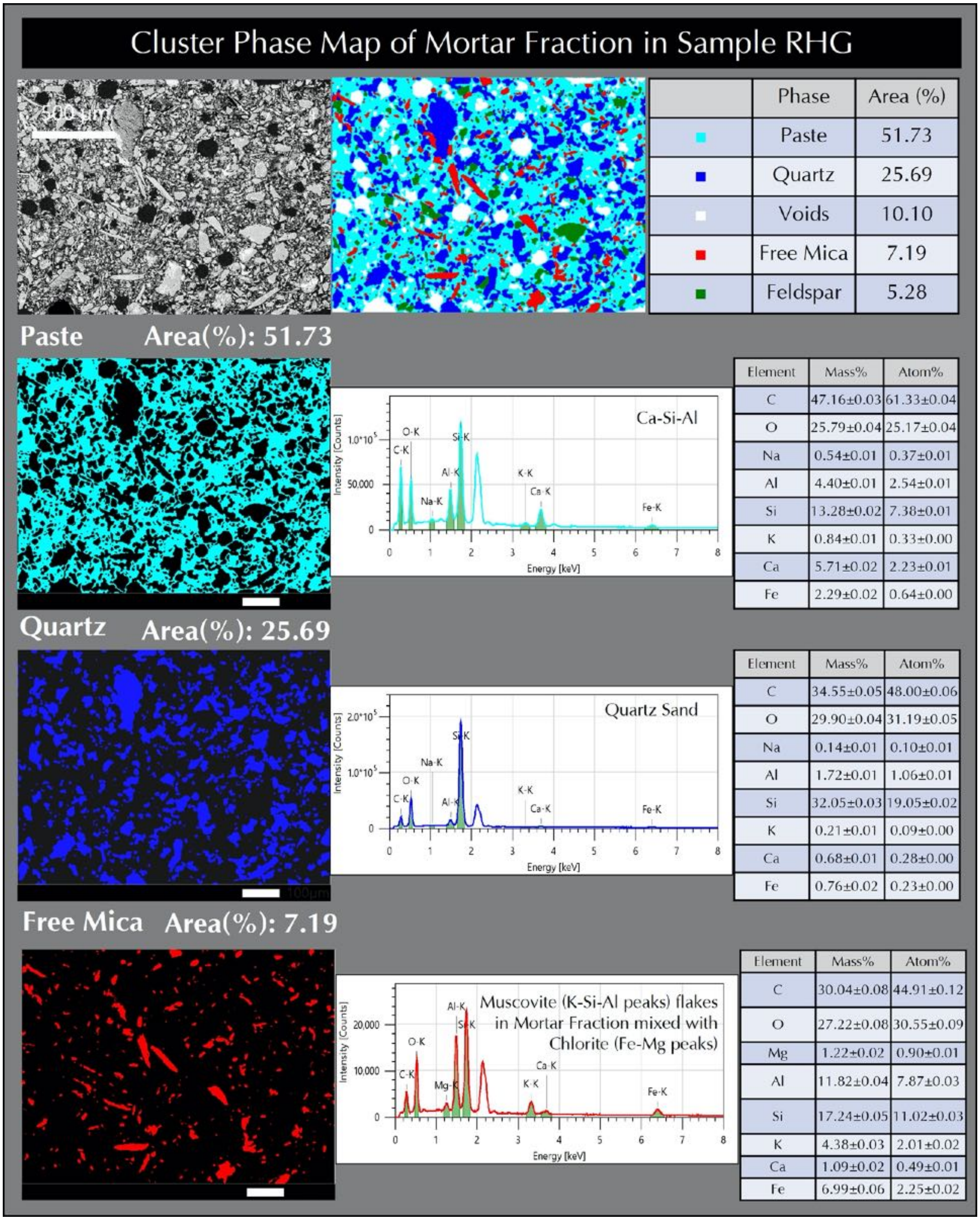


Figure 41: Free mica flakes in the mortar fractions of RGH (top) and LGH (bottom) highlighted in red in the cluster phase maps calculated from elemental maps of mica (K, Al, Si). Phase colors: cyan and yellow - paste, blue – quartz, white – voids, red – free mica, purple – chlorite, and green – feldspar (albite).



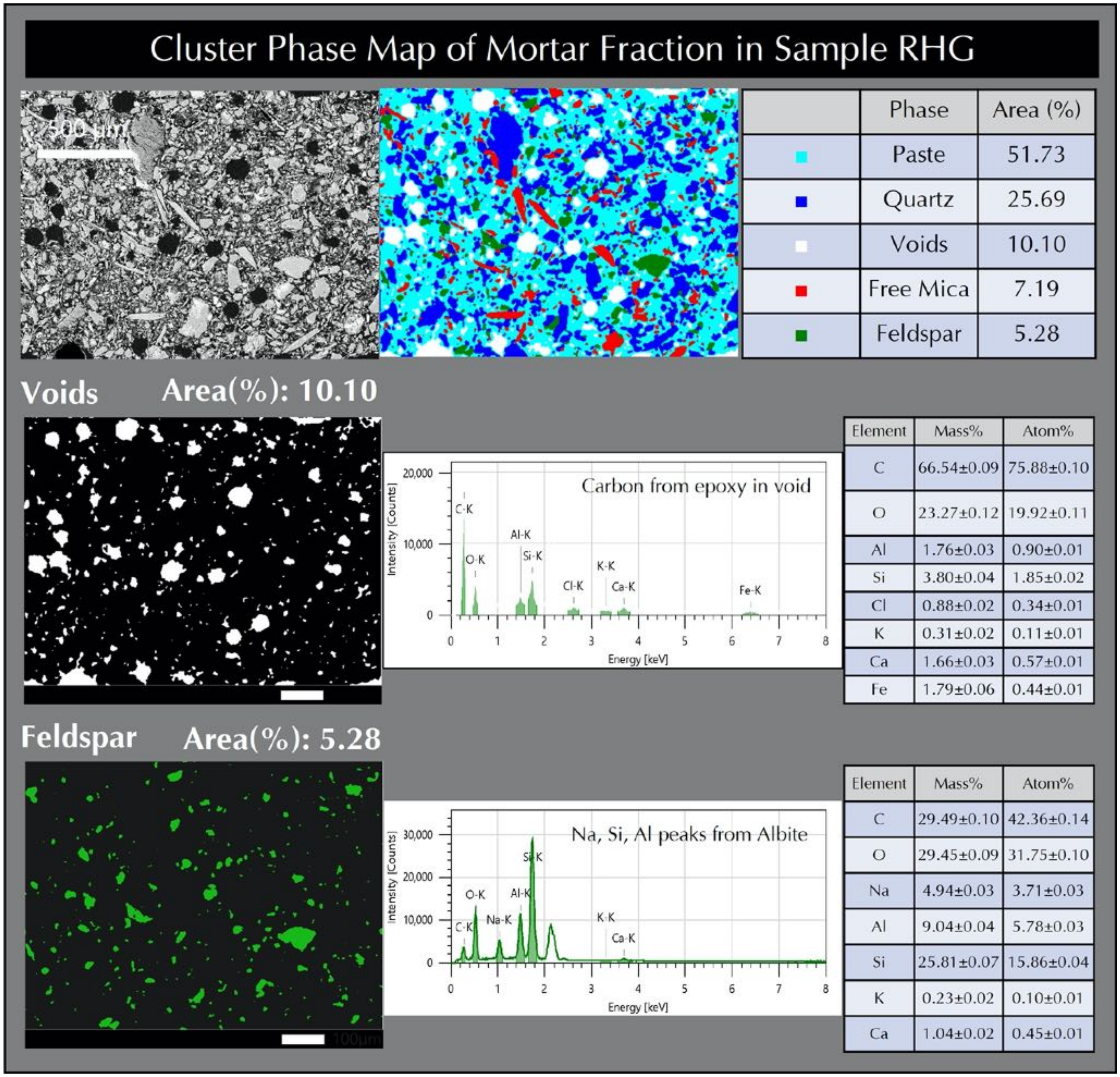
	Phase	Area (%)
■	Paste	51.73
■	Quartz	25.69
■	Voids	10.10
■	Free Mica	7.19
■	Feldspar	5.28

Element	Mass%	Atom%
C	47.16±0.03	61.33±0.04
O	25.79±0.04	25.17±0.04
Na	0.54±0.01	0.37±0.01
Al	4.40±0.01	2.54±0.01
Si	13.28±0.02	7.38±0.01
K	0.84±0.01	0.33±0.00
Ca	5.71±0.02	2.23±0.01
Fe	2.29±0.02	0.64±0.00

Element	Mass%	Atom%
C	34.55±0.05	48.00±0.06
O	29.90±0.04	31.19±0.05
Na	0.14±0.01	0.10±0.01
Al	1.72±0.01	1.06±0.01
Si	32.05±0.03	19.05±0.02
K	0.21±0.01	0.09±0.00
Ca	0.68±0.01	0.28±0.00
Fe	0.76±0.02	0.23±0.00

Element	Mass%	Atom%
C	30.04±0.08	44.91±0.12
O	27.22±0.08	30.55±0.09
Mg	1.22±0.02	0.90±0.01
Al	11.82±0.04	7.87±0.03
Si	17.24±0.05	11.02±0.03
K	4.38±0.03	2.01±0.02
Ca	1.09±0.02	0.49±0.01
Fe	6.99±0.06	2.25±0.02

Figure 42: Backscatter electron image (top left), cluster phase map of phyllite aggregate and adjacent mortar fraction (top middle), phase proportions calculated from phase map (top right), and segmented phase maps, corresponding EDS spectrum and oxide composition of various phases of block fragment RHG in a polished solid section. Phase colors: cyan – paste, blue – quartz, and red – free mica.

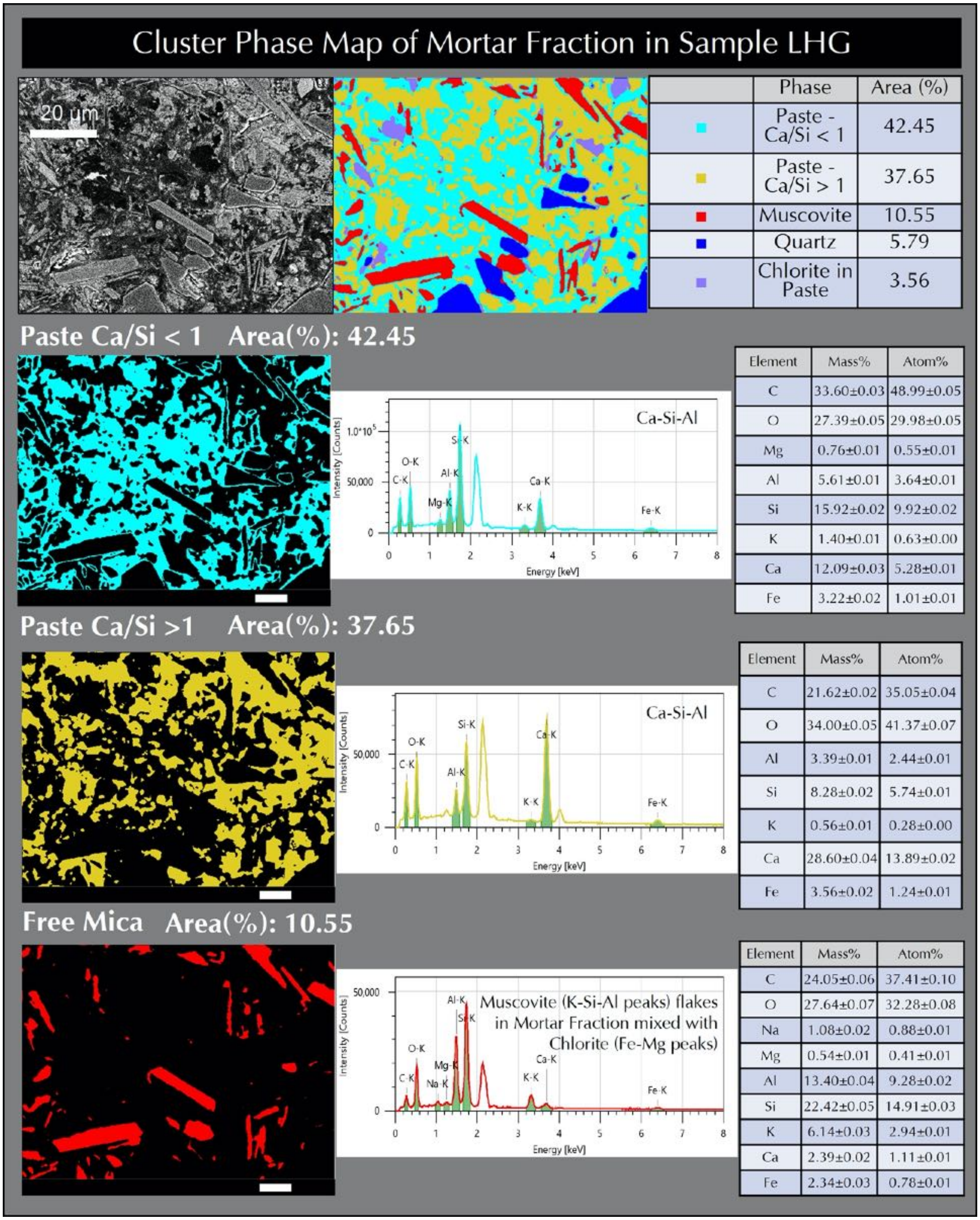


	Phase	Area (%)
■	Paste	51.73
■	Quartz	25.69
■	Voids	10.10
🟥	Free Mica	7.19
🟩	Feldspar	5.28

Element	Mass%	Atom%
C	66.54±0.09	75.88±0.10
O	23.27±0.12	19.92±0.11
Al	1.76±0.03	0.90±0.01
Si	3.80±0.04	1.85±0.02
Cl	0.88±0.02	0.34±0.01
K	0.31±0.02	0.11±0.01
Ca	1.66±0.03	0.57±0.01
Fe	1.79±0.06	0.44±0.01

Element	Mass%	Atom%
C	29.49±0.10	42.36±0.14
O	29.45±0.09	31.75±0.10
Na	4.94±0.03	3.71±0.03
Al	9.04±0.04	5.78±0.03
Si	25.81±0.07	15.86±0.04
K	0.23±0.02	0.10±0.01
Ca	1.04±0.02	0.45±0.01

Figure 43: Backscatter electron image (top left), cluster phase map of phyllite aggregate and adjacent mortar fraction (top middle), phase proportions calculated from phase map (top right), and segmented phase maps, corresponding EDS spectrum and oxide composition of various phases of block fragment RHG in a polished solid section. Phase colors: white – voids, and green – feldspar (albite).



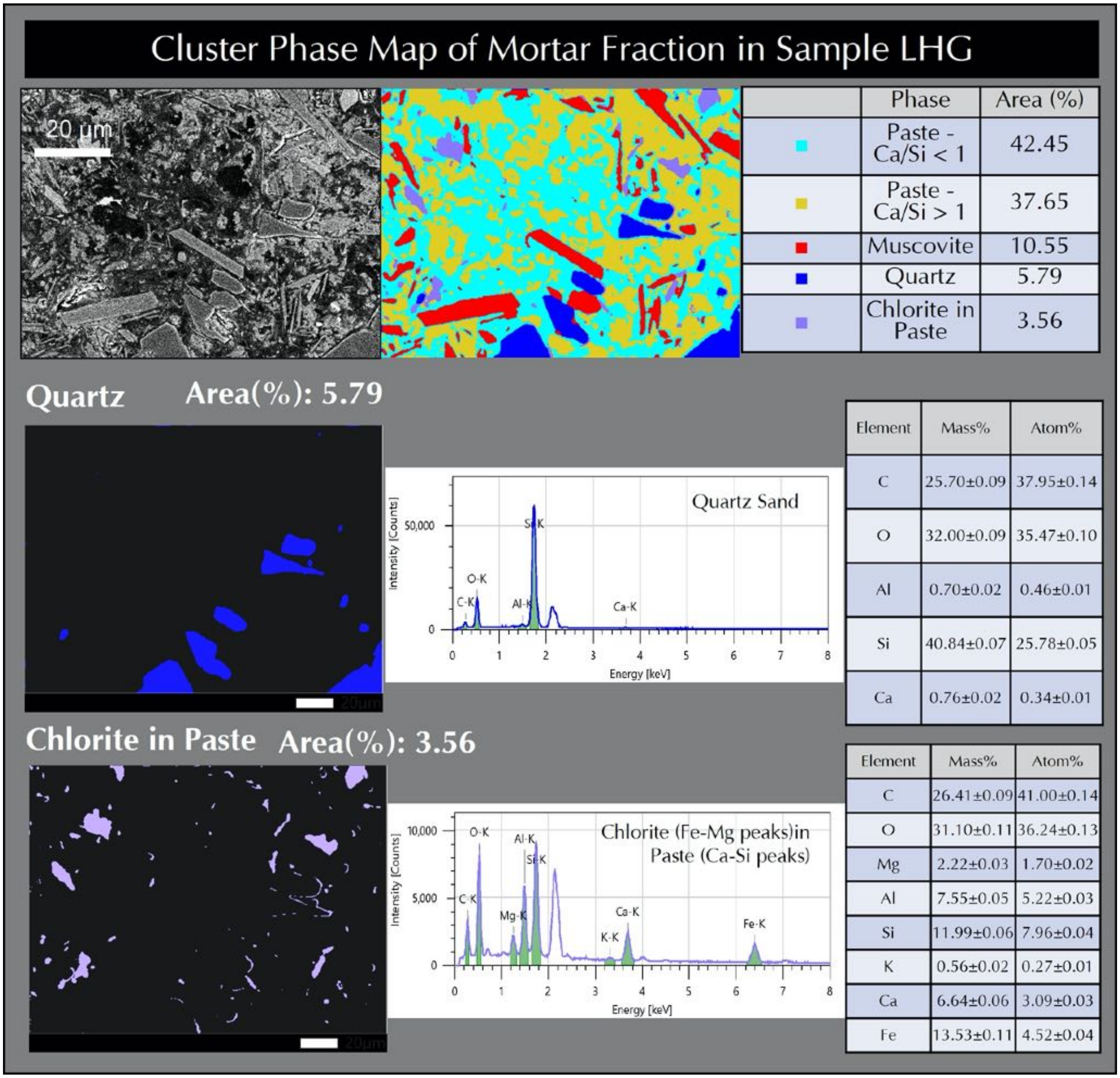
	Phase	Area (%)
■	Paste - Ca/Si < 1	42.45
■	Paste - Ca/Si > 1	37.65
■	Muscovite	10.55
■	Quartz	5.79
■	Chlorite in Paste	3.56

Element	Mass%	Atom%
C	33.60±0.03	48.99±0.05
O	27.39±0.05	29.98±0.05
Mg	0.76±0.01	0.55±0.01
Al	5.61±0.01	3.64±0.01
Si	15.92±0.02	9.92±0.02
K	1.40±0.01	0.63±0.00
Ca	12.09±0.03	5.28±0.01
Fe	3.22±0.02	1.01±0.01

Element	Mass%	Atom%
C	21.62±0.02	35.05±0.04
O	34.00±0.05	41.37±0.07
Al	3.39±0.01	2.44±0.01
Si	8.28±0.02	5.74±0.01
K	0.56±0.01	0.28±0.00
Ca	28.60±0.04	13.89±0.02
Fe	3.56±0.02	1.24±0.01

Element	Mass%	Atom%
C	24.05±0.06	37.41±0.10
O	27.64±0.07	32.28±0.08
Na	1.08±0.02	0.88±0.01
Mg	0.54±0.01	0.41±0.01
Al	13.40±0.04	9.28±0.02
Si	22.42±0.05	14.91±0.03
K	6.14±0.03	2.94±0.01
Ca	2.39±0.02	1.11±0.01
Fe	2.34±0.03	0.78±0.01

Figure 44: Backscatter electron image (top left), cluster phase map of phyllite aggregate and adjacent mortar fraction (top middle), phase proportions calculated from phase map (top right), and segmented phase maps, corresponding EDS spectrum and oxide composition of various phases of block fragment LHG in a polished solid section. Phase colors: paste – cyan and yellow for Ca/Si <1 and > 1, respectively, and red – free mica.



Cluster Phase Map of Mortar Fraction in Sample LHG

	Phase	Area (%)
■	Paste - Ca/Si < 1	42.45
■	Paste - Ca/Si > 1	37.65
■	Muscovite	10.55
■	Quartz	5.79
■	Chlorite in Paste	3.56

Quartz Area(%): 5.79

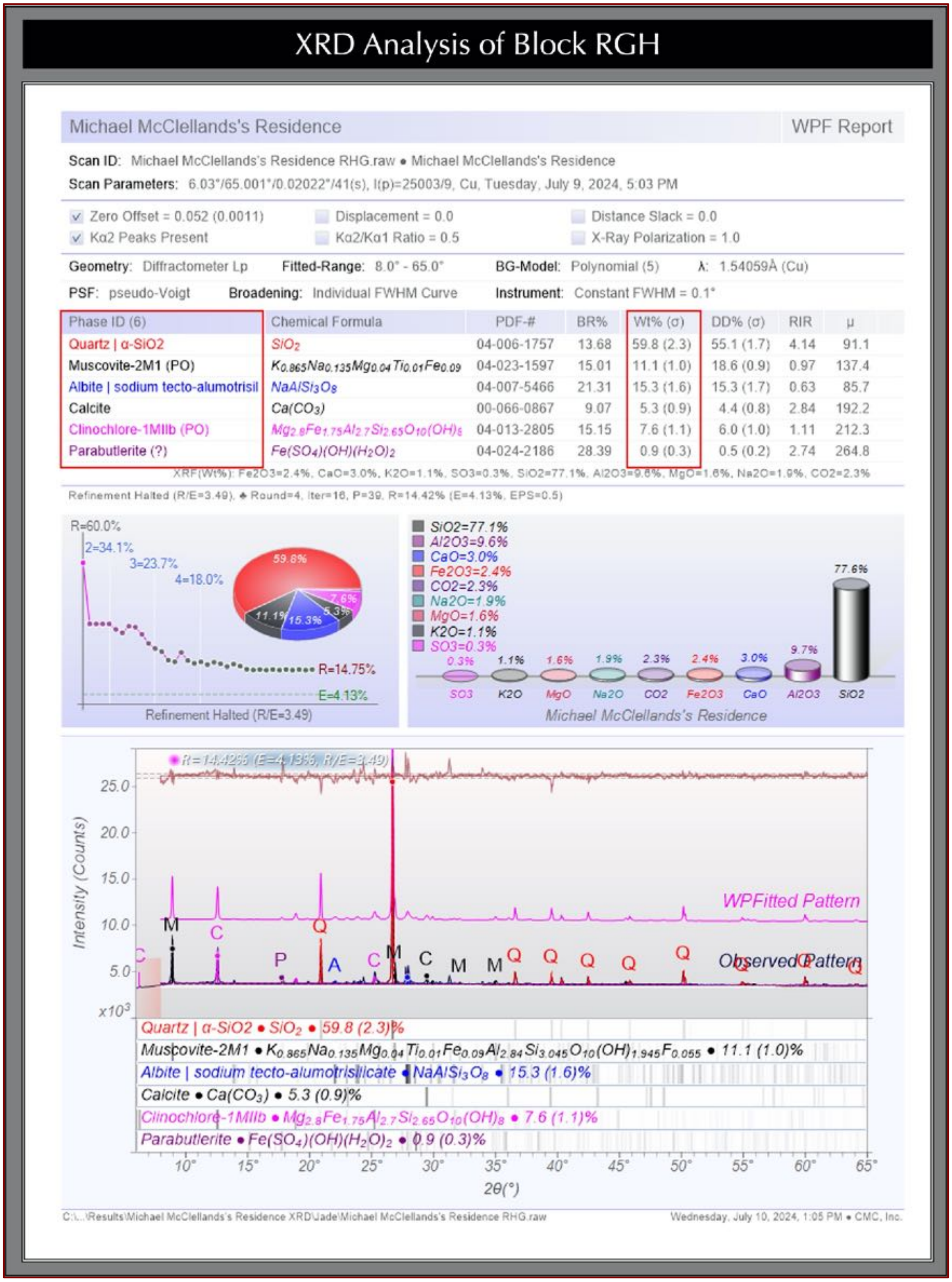
Element	Mass%	Atom%
C	25.70±0.09	37.95±0.14
O	32.00±0.09	35.47±0.10
Al	0.70±0.02	0.46±0.01
Si	40.84±0.07	25.78±0.05
Ca	0.76±0.02	0.34±0.01

Chlorite in Paste Area(%): 3.56

Element	Mass%	Atom%
C	26.41±0.09	41.00±0.14
O	31.10±0.11	36.24±0.13
Mg	2.22±0.03	1.70±0.02
Al	7.55±0.05	5.22±0.03
Si	11.99±0.06	7.96±0.04
K	0.56±0.02	0.27±0.01
Ca	6.64±0.06	3.09±0.03
Fe	13.53±0.11	4.52±0.04

Figure 45: Backscatter electron image (top left), cluster phase map of phyllite aggregate and adjacent mortar fraction (top middle), phase proportions calculated from phase map (top right), and segmented phase maps, corresponding EDS spectrum and oxide composition of various phases of block fragment LHG in a polished solid section. Phase colors: blue – quartz and purple - chlorite.

X-RAY DIFFRACTION



XRD Analysis of Block RGH

Michael McClellands's Residence WPF Report

Scan ID: Michael McClellands's Residence RHG.raw • Michael McClellands's Residence

Scan Parameters: 6.03°/65.001°/0.02022°/41(s), I(p)=25003/9, Cu, Tuesday, July 9, 2024, 5:03 PM

☑ Zero Offset = 0.052 (0.0011) ☐ Displacement = 0.0 ☐ Distance Slack = 0.0

☑ Kα2 Peaks Present ☐ Kα2/Kα1 Ratio = 0.5 ☐ X-Ray Polarization = 1.0

Geometry: Diffractometer Lp **Fitted-Range:** 8.0° - 65.0° **BG-Model:** Polynomial (5) **λ:** 1.54059Å (Cu)

PSF: pseudo-Voigt **Broadening:** Individual FWHM Curve **Instrument:** Constant FWHM = 0.1°

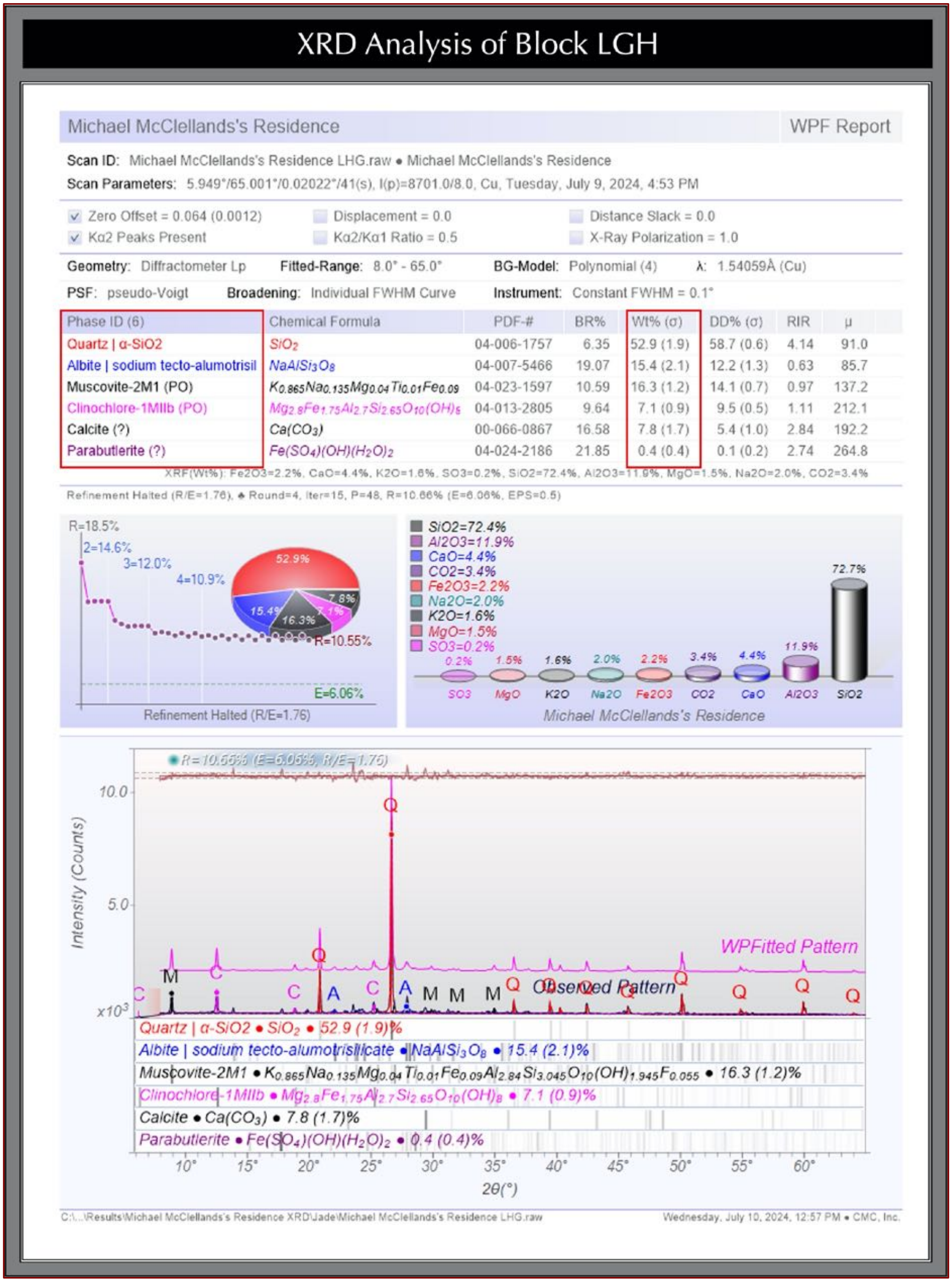
Phase ID (6)	Chemical Formula	PDF-#	BR%	Wt% (σ)	DD% (σ)	RIR	μ
Quartz | α-SiO2	SiO_2	04-006-1757	13.68	59.8 (2.3)	55.1 (1.7)	4.14	91.1
Muscovite-2M1 (PO)	$K_{0.865}Na_{0.135}Mg_{0.04}Ti_{0.01}Fe_{0.09}$	04-023-1597	15.01	11.1 (1.0)	18.6 (0.9)	0.97	137.4
Albite | sodium tecto-alumotrisil	$NaAlSi_3O_8$	04-007-5466	21.31	15.3 (1.6)	15.3 (1.7)	0.63	85.7
Calcite	$Ca(CO_3)$	00-066-0867	9.07	5.3 (0.9)	4.4 (0.8)	2.84	192.2
Clinochlore-1MIIb (PO)	$Mg_{2.8}Fe_{1.75}Al_{2.7}Si_{2.65}O_{10}(OH)_8$	04-013-2805	15.15	7.6 (1.1)	6.0 (1.0)	1.11	212.3
Parabutlerite (?)	$Fe(SO_4)(OH)(H_2O)_2$	04-024-2186	28.39	0.9 (0.3)	0.5 (0.2)	2.74	264.8

XRF(Wt%): Fe2O3=2.4%, CaO=3.0%, K2O=1.1%, SO3=0.3%, SiO2=77.1%, Al2O3=9.6%, MgO=1.6%, Na2O=1.9%, CO2=2.3%

Refinement Halted (R/E=3.49), ♣ Round=4, Iter=16, P=39, R=14.42% (E=4.13%, EPS=0.5)

C:\...\Results\Michael McClellands's Residence XRD\Jade\Michael McClellands's Residence RHG.raw Wednesday, July 10, 2024, 1:05 PM • CMC, Inc.

Figure 46: XRD pattern of block sample RGH. Rietveld analysis detected 59.8% quartz, 15.3% albite, 11.1% muscovite mica, 7.6% chlorite, 5.3% calcite, and <1% parabutlerite. Notice the absence of pyrite (for its presence at below the detection limit of XRD that was detected in reflect-light microscopy), and potentially deleterious reaction products (gypsum, ettringite, thaumasite) of internal sulfate attacks.



XRD Analysis of Block LGH

Michael McClellands's Residence — WPF Report

Scan ID: Michael McClellands's Residence LHG.raw • Michael McClellands's Residence

Scan Parameters: 5.949°/65.001°/0.02022°/41(s), I(p)=8701.0/8.0, Cu, Tuesday, July 9, 2024, 4:53 PM

☑ Zero Offset = 0.064 (0.0012) ☐ Displacement = 0.0 ☐ Distance Slack = 0.0

☑ Kα2 Peaks Present ☐ Kα2/Kα1 Ratio = 0.5 ☐ X-Ray Polarization = 1.0

Geometry: Diffractometer Lp **Fitted-Range:** 8.0° - 65.0° **BG-Model:** Polynomial (4) **λ:** 1.54059Å (Cu)

PSF: pseudo-Voigt **Broadening:** Individual FWHM Curve **Instrument:** Constant FWHM = 0.1°

Phase ID (6)	Chemical Formula	PDF-#	BR%	Wt% (σ)	DD% (σ)	RIR	μ
Quartz | α-SiO2	SiO_2	04-006-1757	6.35	52.9 (1.9)	58.7 (0.6)	4.14	91.0
Albite | sodium tecto-alumotrisil	$NaAlSi_3O_8$	04-007-5466	19.07	15.4 (2.1)	12.2 (1.3)	0.63	85.7
Muscovite-2M1 (PO)	$K_{0.865}Na_{0.135}Mg_{0.04}Ti_{0.01}Fe_{0.09}$	04-023-1597	10.59	16.3 (1.2)	14.1 (0.7)	0.97	137.2
Clinochlore-1MIIb (PO)	$Mg_{2.8}Fe_{1.75}Al_{2.7}Si_{2.65}O_{10}(OH)_8$	04-013-2805	9.64	7.1 (0.9)	9.5 (0.5)	1.11	212.1
Calcite (?)	$Ca(CO_3)$	00-066-0867	16.58	7.8 (1.7)	5.4 (1.0)	2.84	192.2
Parabutlerite (?)	$Fe(SO_4)(OH)(H_2O)_2$	04-024-2186	21.85	0.4 (0.4)	0.1 (0.2)	2.74	264.8

XRF(Wt%): Fe2O3=2.2%, CaO=4.4%, K2O=1.6%, SO3=0.2%, SiO2=72.4%, Al2O3=11.9%, MgO=1.5%, Na2O=2.0%, CO2=3.4%

Refinement Halted (R/E=1.76), ♣ Round=4, Iter=15, P=48, R=10.66% (E=6.06%, EPS=0.5)

C:\...\Results\Michael McClellands's Residence XRD\Jade\Michael McClellands's Residence LHG.raw — Wednesday, July 10, 2024, 12:57 PM • CMC, Inc.

Figure 47: XRD pattern of block sample LGH. Rietveld analysis detected 52.9% quartz, 15.4% albite, 16.3% muscovite mica, 7.1% chlorite, 7.8% calcite, and <0.5% parabutlerite. Notice the absence of pyrite (for its presence at below the detection limit of XRD that was detected in reflect-light microscopy), and potentially deleterious reaction products (gypsum, ettringite, thaumasite) of internal sulfate attacks.

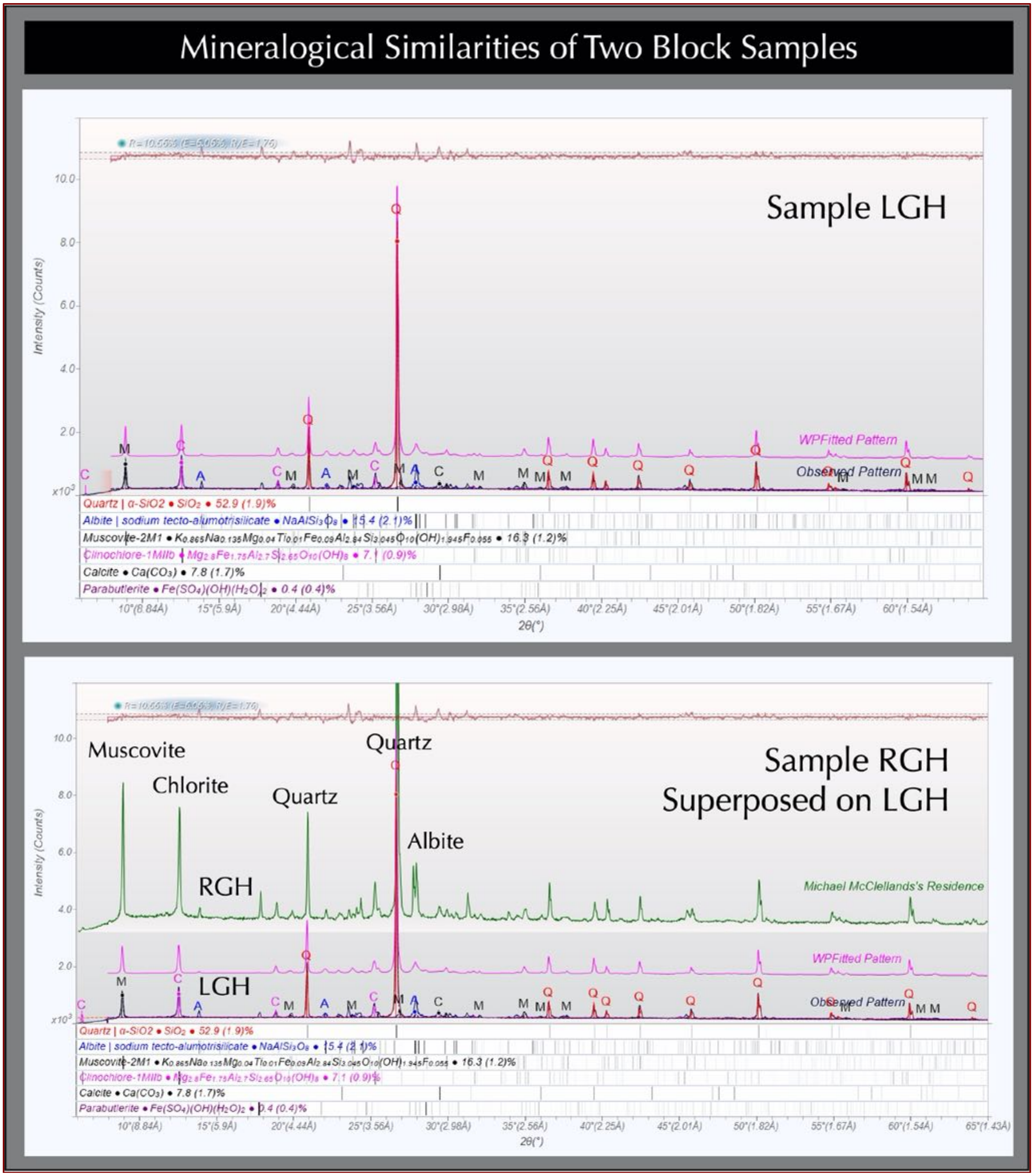
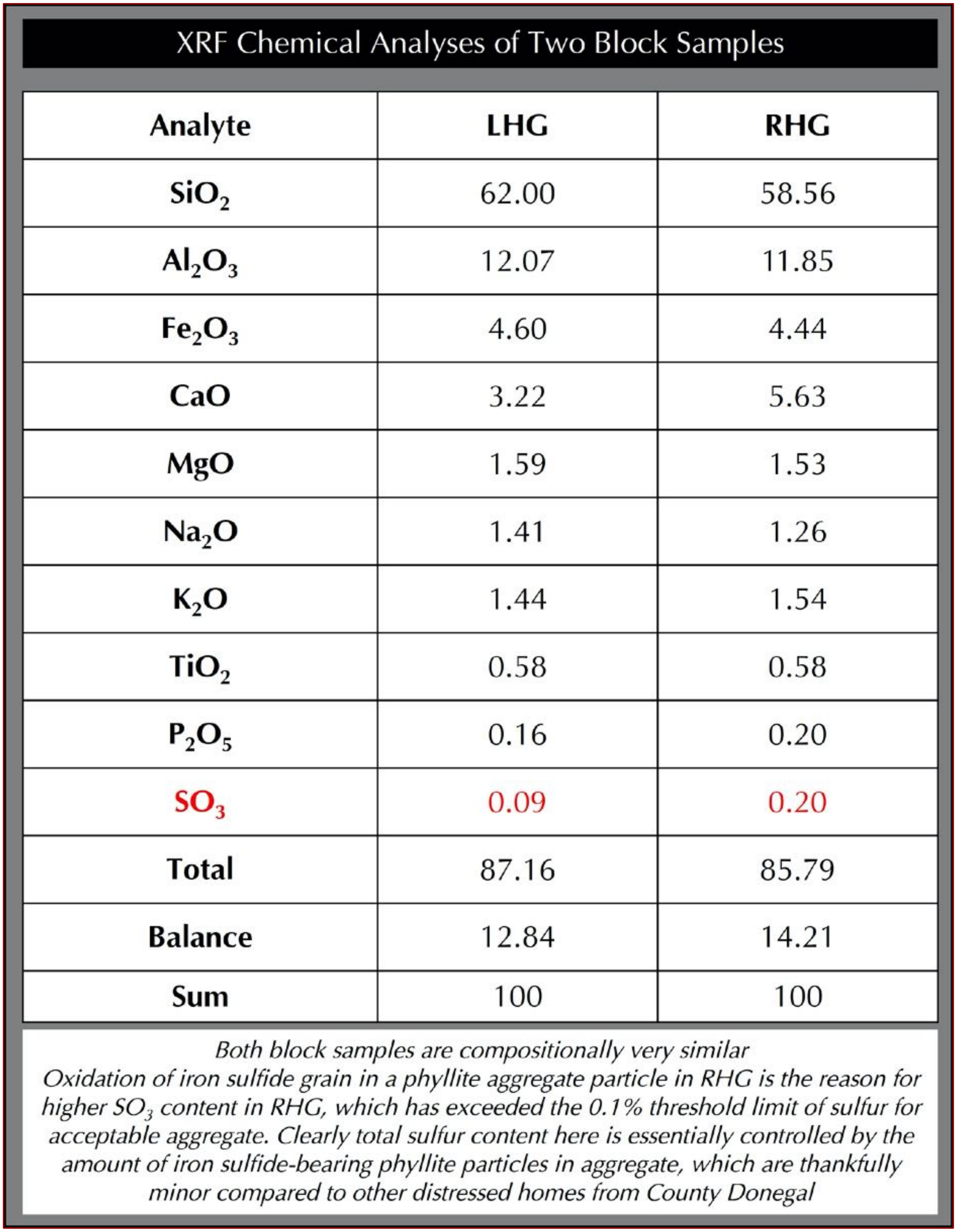


Figure 48: Overall mineralogical similartities of two block samples in XRD patterns showing muscovite, chlorite, quartz, albite, and calcite as the five main detectable peaks.

X-RAY FLUORESCENCE SPECTROSCOPY



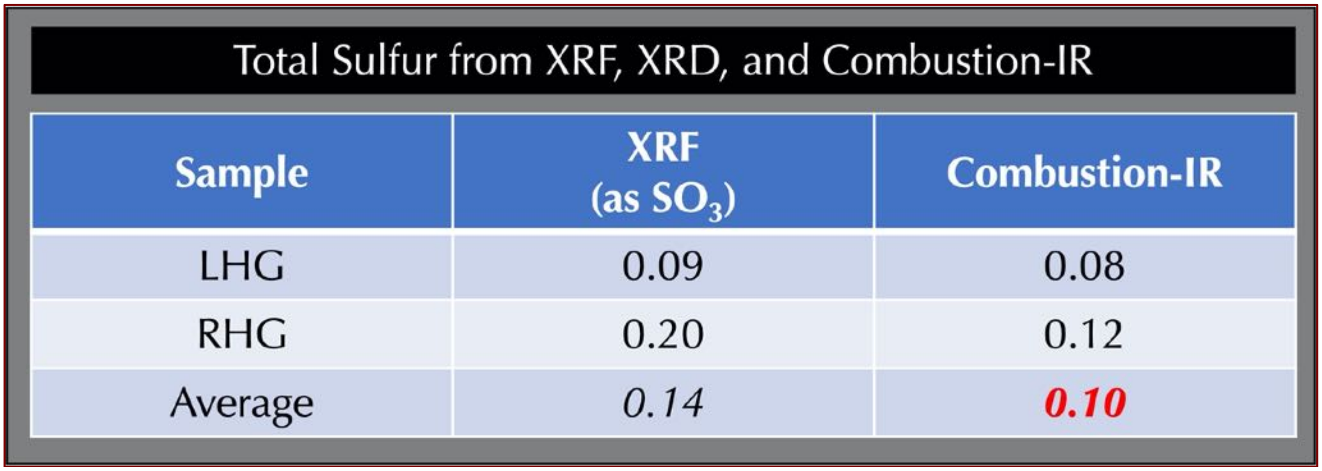
XRF Chemical Analyses of Two Block Samples

Analyte	LHG	RHG
SiO_2	62.00	58.56
Al_2O_3	12.07	11.85
Fe_2O_3	4.60	4.44
CaO	3.22	5.63
MgO	1.59	1.53
Na_2O	1.41	1.26
K_2O	1.44	1.54
TiO_2	0.58	0.58
P_2O_5	0.16	0.20
SO_3	0.09	0.20
Total	87.16	85.79
Balance	12.84	14.21
Sum	100	100

Both block samples are compositionally very similar
Oxidation of iron sulfide grain in a phyllite aggregate particle in RHG is the reason for higher SO_3 content in RHG, which has exceeded the 0.1% threshold limit of sulfur for acceptable aggregate. Clearly total sulfur content here is essentially controlled by the amount of iron sulfide-bearing phyllite particles in aggregate, which are thankfully minor compared to other distressed homes from County Donegal

Figure 49: Chemical comnpositions of two block samples determined from energy-dispersive X-ray fluorescence spectroscopy. Total sulfate (expressed as SO_3) is determined to be < 0.1% in LHG and 0.2% in RHG.

TOTAL SULFUR



Total Sulfur from XRF, XRD, and Combustion-IR

Sample	XRF (as SO_3)	Combustion-IR
LHG	0.09	0.08
RHG	0.20	0.12
Average	*0.14*	***0.10***

Figure 50: Total sulfur contents determined from XRF and combustion-IR (Leco) method.

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The above conclusions are based solely on the information and sample provided at the time of this investigation. The conclusion may expand or modify upon receipt of further information, field evidence, or samples. Sample will be discarded after submission of the report. All reports are the confidential property of clients, and information contained herein may not be published or reproduced pending our written approval. Neither CMC nor its employees assume any obligation or liability for damages, including, but not limited to, consequential damages arising out of, or, in conjunction with the use, or inability to use this resulting information.

APPENDIX 1 –

IRISH STANDARD

I.S.465:2018+A1:2020

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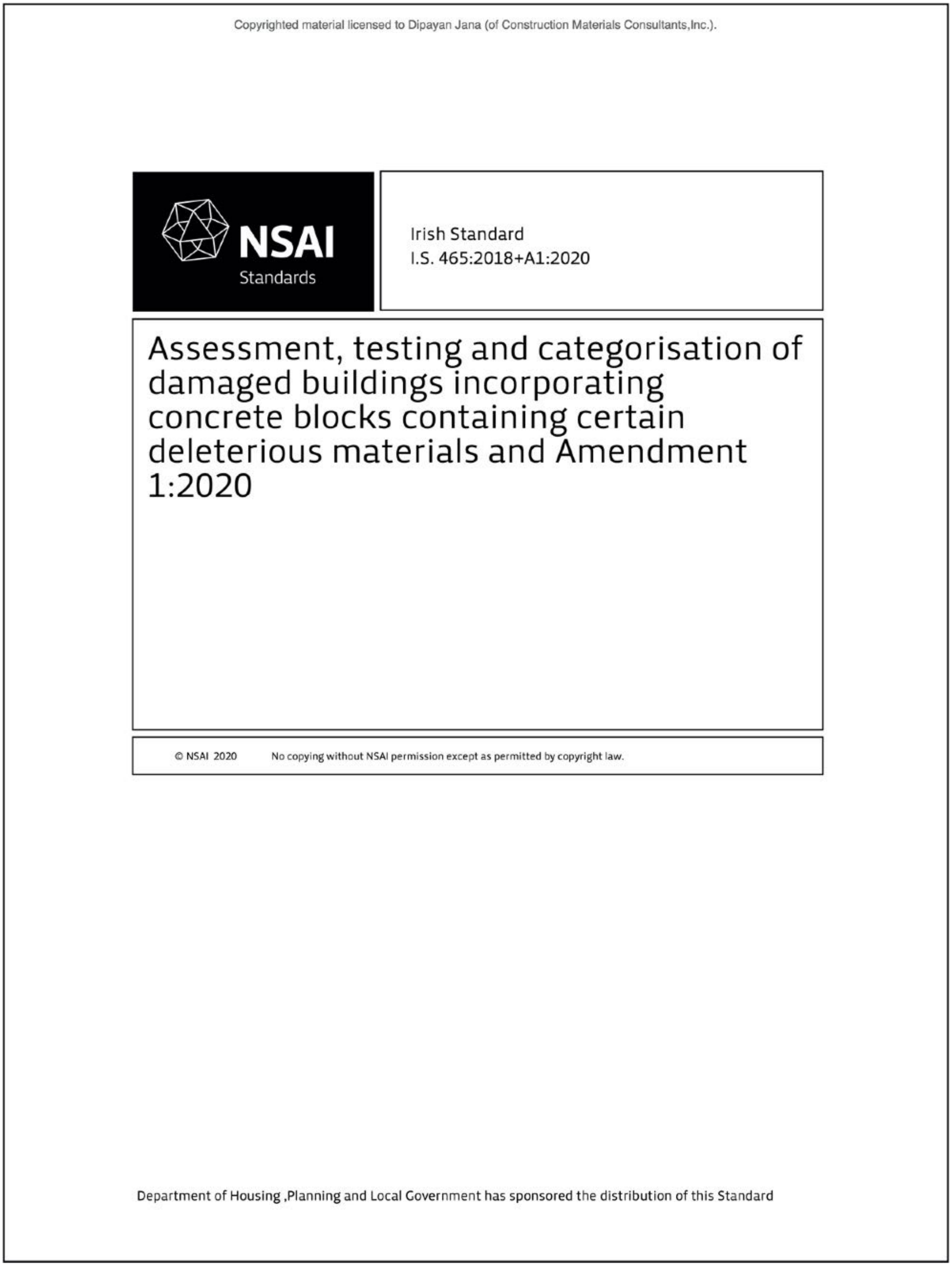

NSAI
Standards

Irish Standard
I.S. 465:2018+A1:2020

Assessment, testing and categorisation of damaged buildings incorporating concrete blocks containing certain deleterious materials and Amendment 1:2020

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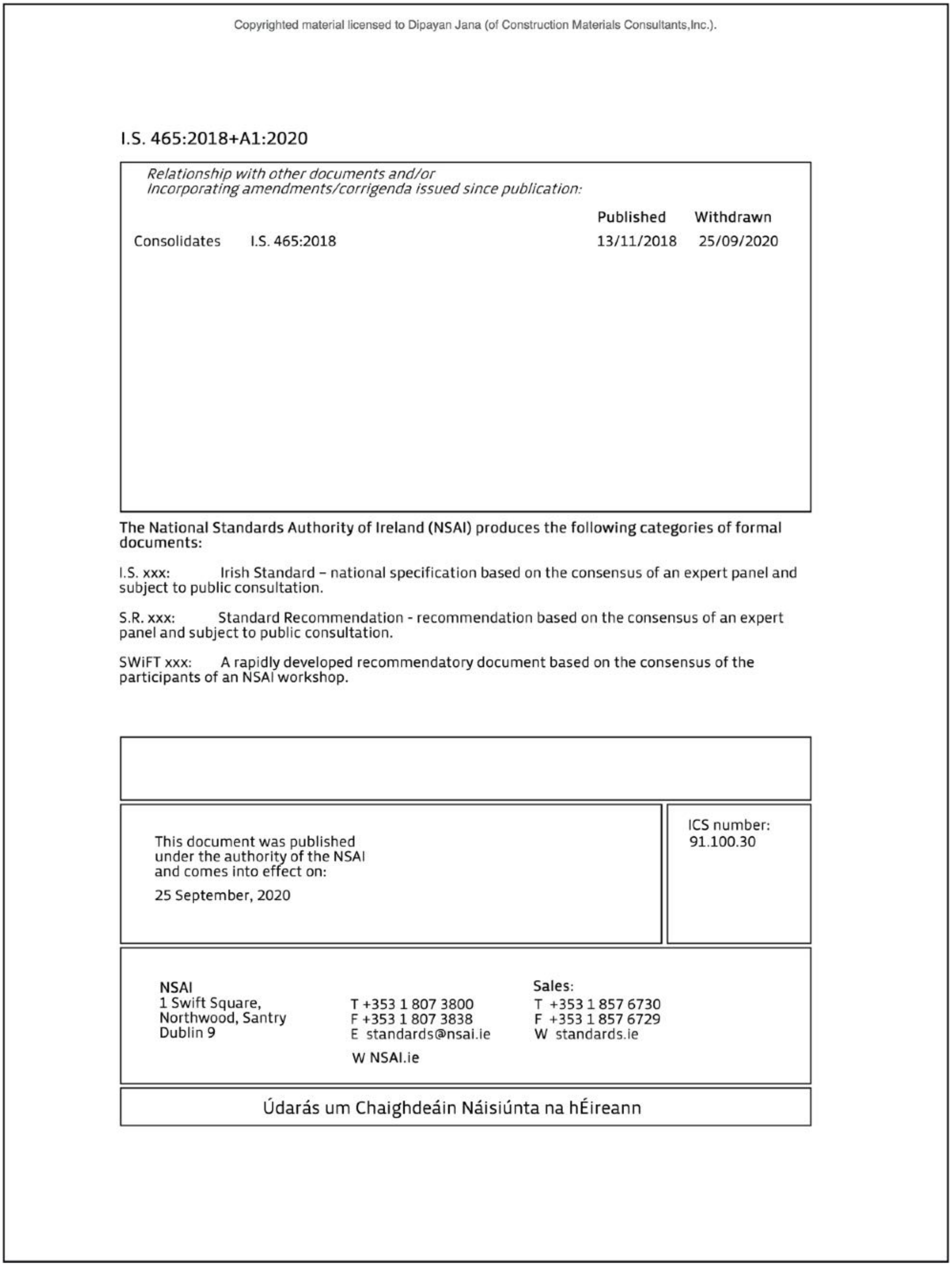

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Incorporating amendments/corrigenda issued since publication:

		Published	Withdrawn
Consolidates	I.S. 465:2018	13/11/2018	25/09/2020

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I.S. xxx: Irish Standard – national specification based on the consensus of an expert panel and subject to public consultation.

S.R. xxx: Standard Recommendation - recommendation based on the consensus of an expert panel and subject to public consultation.

SWiFT xxx: A rapidly developed recommendatory document based on the consensus of the participants of an NSAI workshop.

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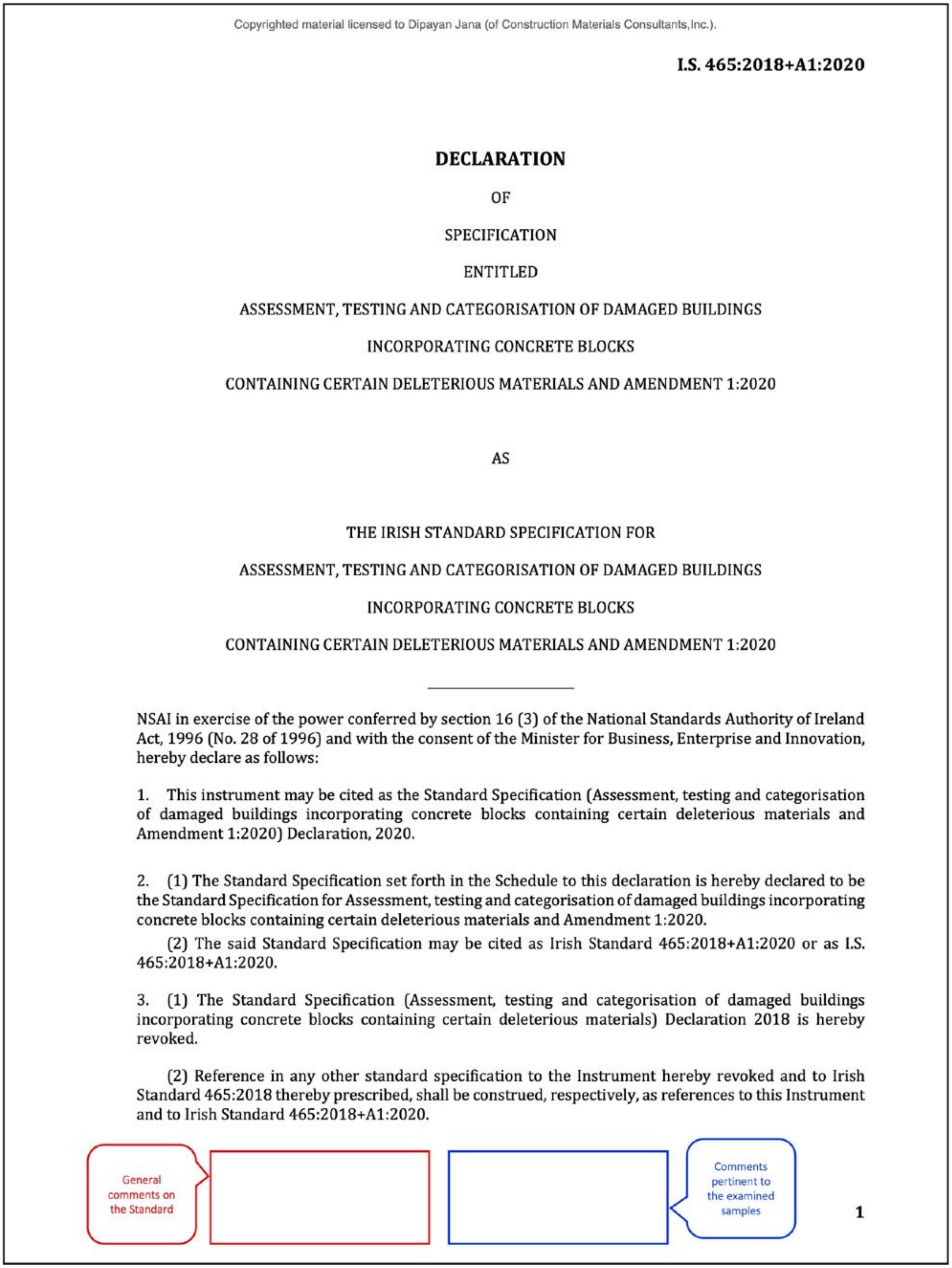


I.S. 465:2018+A1:2020

DECLARATION

OF

SPECIFICATION

ENTITLED

ASSESSMENT, TESTING AND CATEGORISATION OF DAMAGED BUILDINGS

INCORPORATING CONCRETE BLOCKS

CONTAINING CERTAIN DELETERIOUS MATERIALS AND AMENDMENT 1:2020

AS

THE IRISH STANDARD SPECIFICATION FOR

ASSESSMENT, TESTING AND CATEGORISATION OF DAMAGED BUILDINGS

INCORPORATING CONCRETE BLOCKS

CONTAINING CERTAIN DELETERIOUS MATERIALS AND AMENDMENT 1:2020

NSAI in exercise of the power conferred by section 16 (3) of the National Standards Authority of Ireland Act, 1996 (No. 28 of 1996) and with the consent of the Minister for Business, Enterprise and Innovation, hereby declare as follows:

1. This instrument may be cited as the Standard Specification (Assessment, testing and categorisation of damaged buildings incorporating concrete blocks containing certain deleterious materials and Amendment 1:2020) Declaration, 2020.

2. (1) The Standard Specification set forth in the Schedule to this declaration is hereby declared to be the Standard Specification for Assessment, testing and categorisation of damaged buildings incorporating concrete blocks containing certain deleterious materials and Amendment 1:2020.

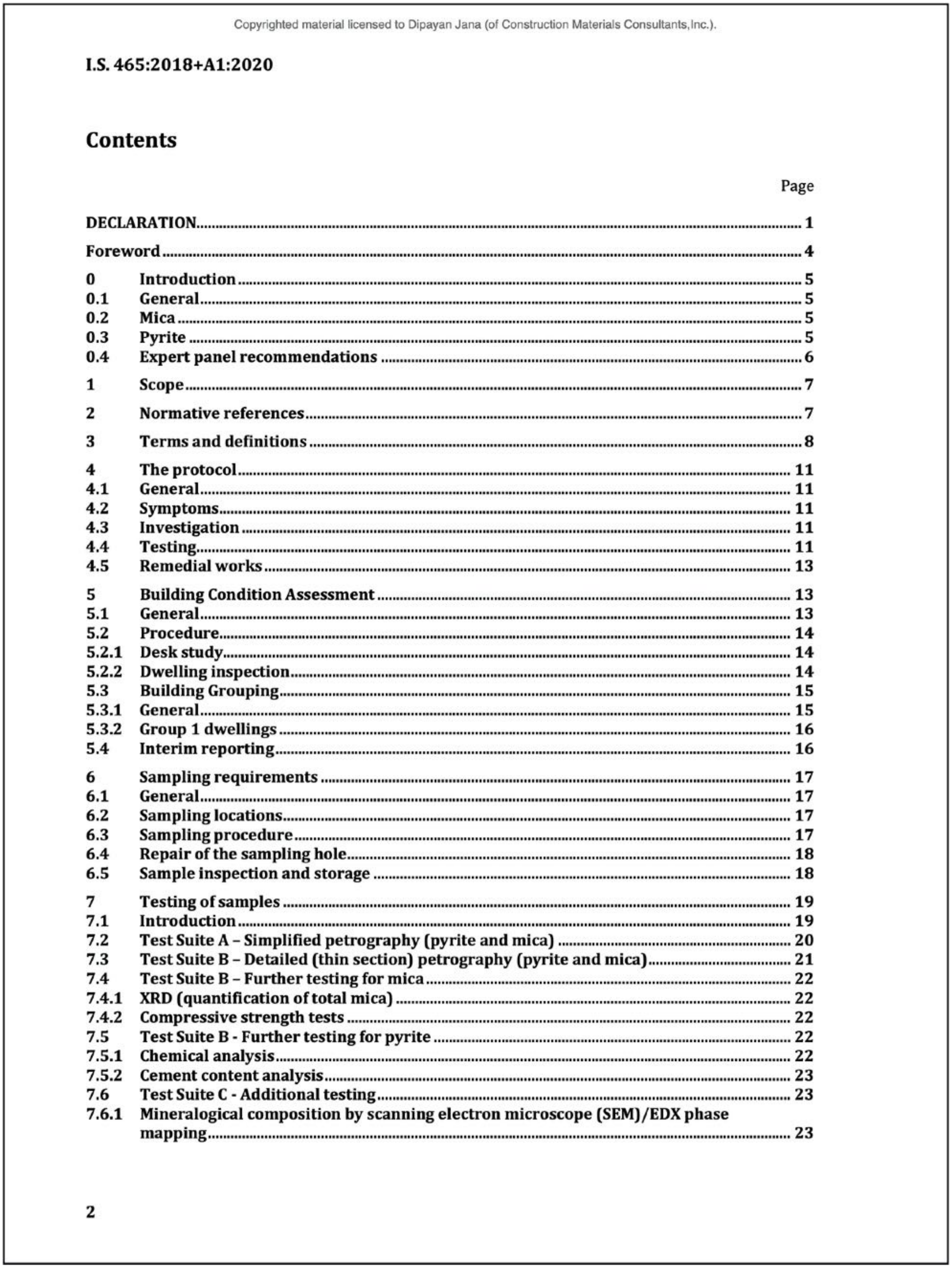
(2) The said Standard Specification may be cited as Irish Standard 465:2018+A1:2020 or as I.S. 465:2018+A1:2020.

3. (1) The Standard Specification (Assessment, testing and categorisation of damaged buildings incorporating concrete blocks containing certain deleterious materials) Declaration 2018 is hereby revoked.

(2) Reference in any other standard specification to the Instrument hereby revoked and to Irish Standard 465:2018 thereby prescribed, shall be construed, respectively, as references to this Instrument and to Irish Standard 465:2018+A1:2020.

General comments on the Standard

Comments pertinent to the examined samples



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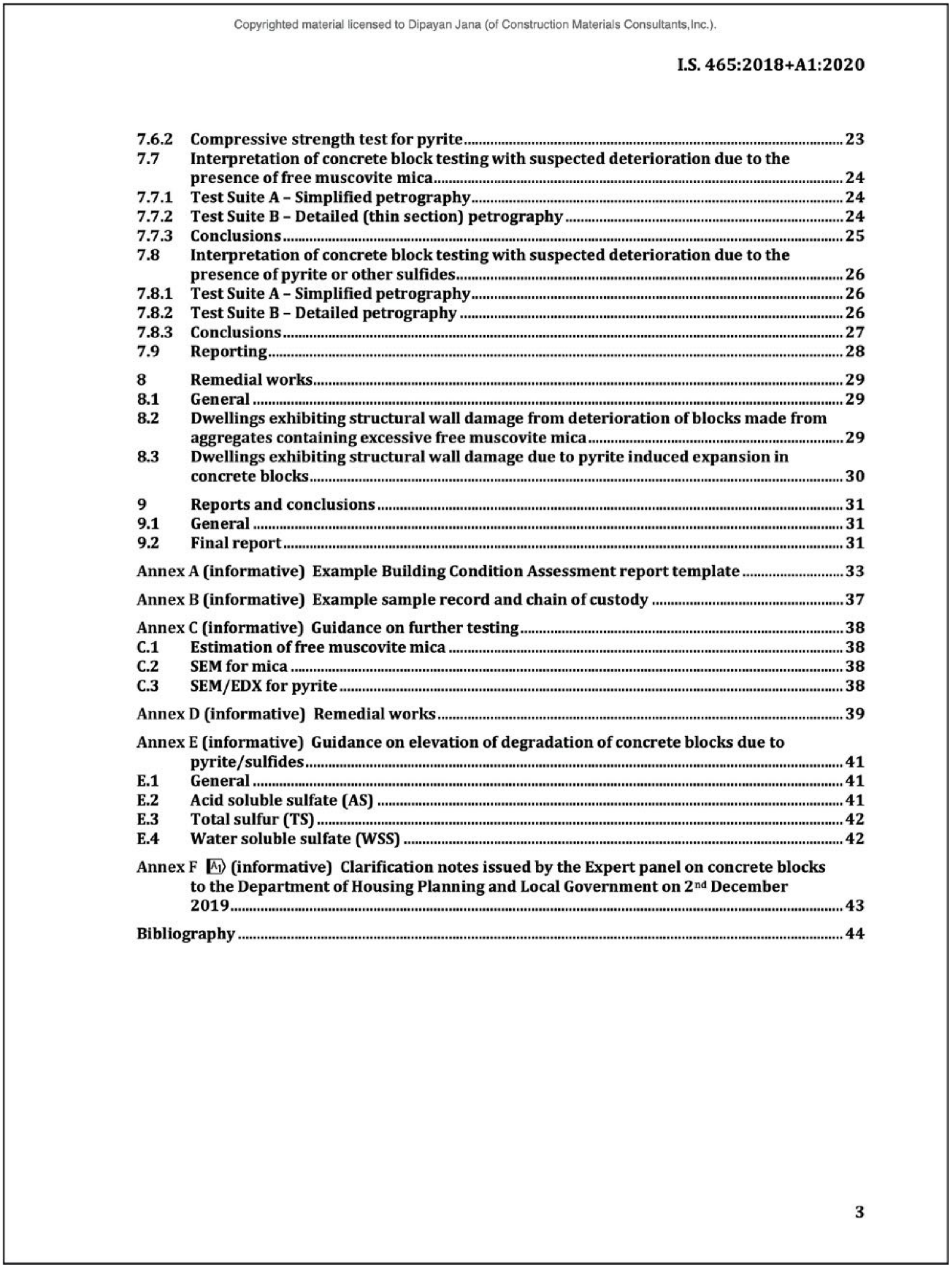
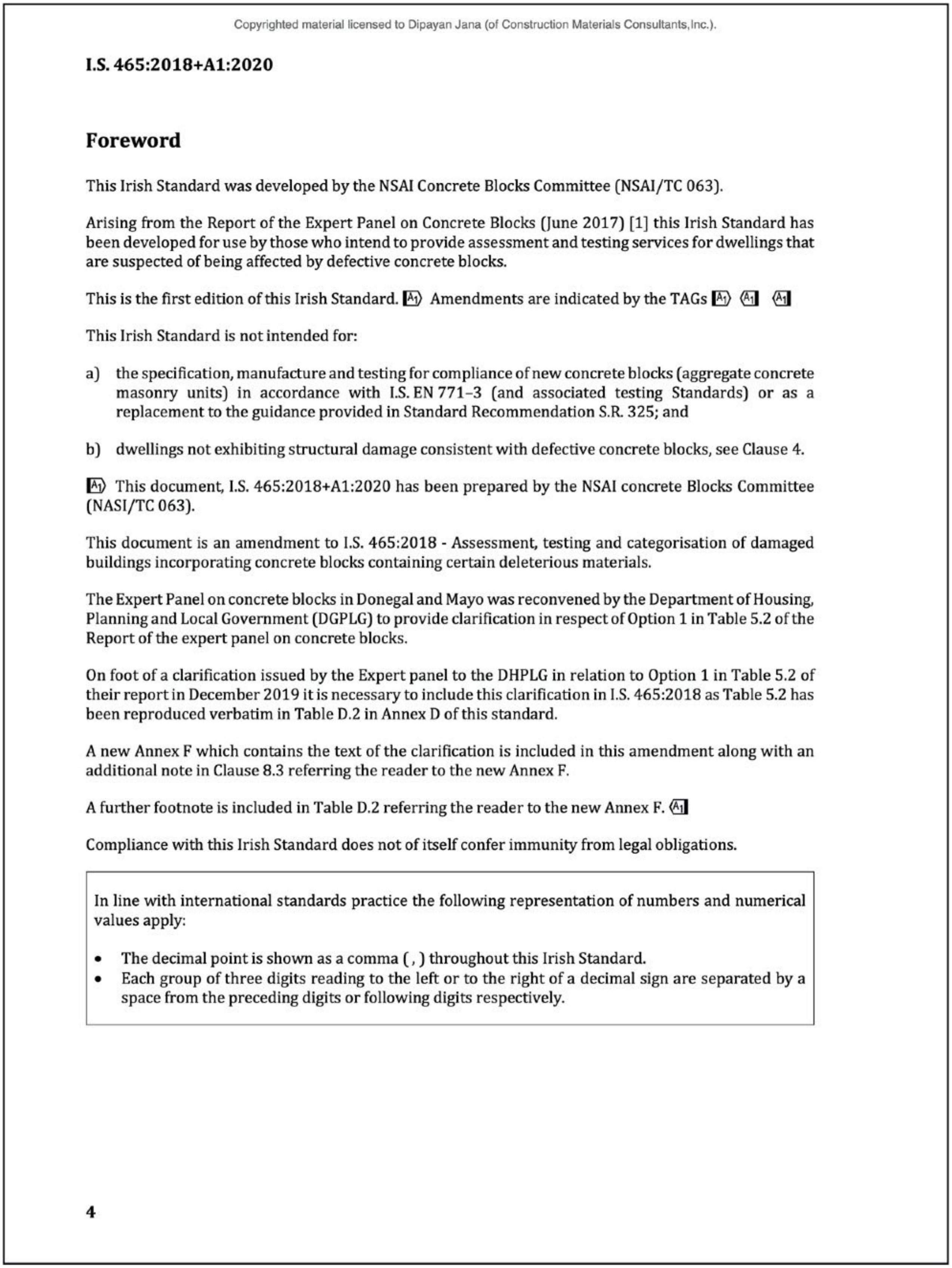




Foreword

This Irish Standard was developed by the NSAI Concrete Blocks Committee (NSAI/TC 063).

Arising from the Report of the Expert Panel on Concrete Blocks (June 2017) [1] this Irish Standard has been developed for use by those who intend to provide assessment and testing services for dwellings that are suspected of being affected by defective concrete blocks.

This is the first edition of this Irish Standard. A1⟩ Amendments are indicated by the TAGs A1⟩ ⟨A1 ⟨A1

This Irish Standard is not intended for:

a) the specification, manufacture and testing for compliance of new concrete blocks (aggregate concrete masonry units) in accordance with I.S. EN 771-3 (and associated testing Standards) or as a replacement to the guidance provided in Standard Recommendation S.R. 325; and

b) dwellings not exhibiting structural damage consistent with defective concrete blocks, see Clause 4.

A1⟩ This document, I.S. 465:2018+A1:2020 has been prepared by the NSAI concrete Blocks Committee (NASI/TC 063).

This document is an amendment to I.S. 465:2018 - Assessment, testing and categorisation of damaged buildings incorporating concrete blocks containing certain deleterious materials.

The Expert Panel on concrete blocks in Donegal and Mayo was reconvened by the Department of Housing, Planning and Local Government (DGPLG) to provide clarification in respect of Option 1 in Table 5.2 of the Report of the expert panel on concrete blocks.

On foot of a clarification issued by the Expert panel to the DHPLG in relation to Option 1 in Table 5.2 of their report in December 2019 it is necessary to include this clarification in I.S. 465:2018 as Table 5.2 has been reproduced verbatim in Table D.2 in Annex D of this standard.

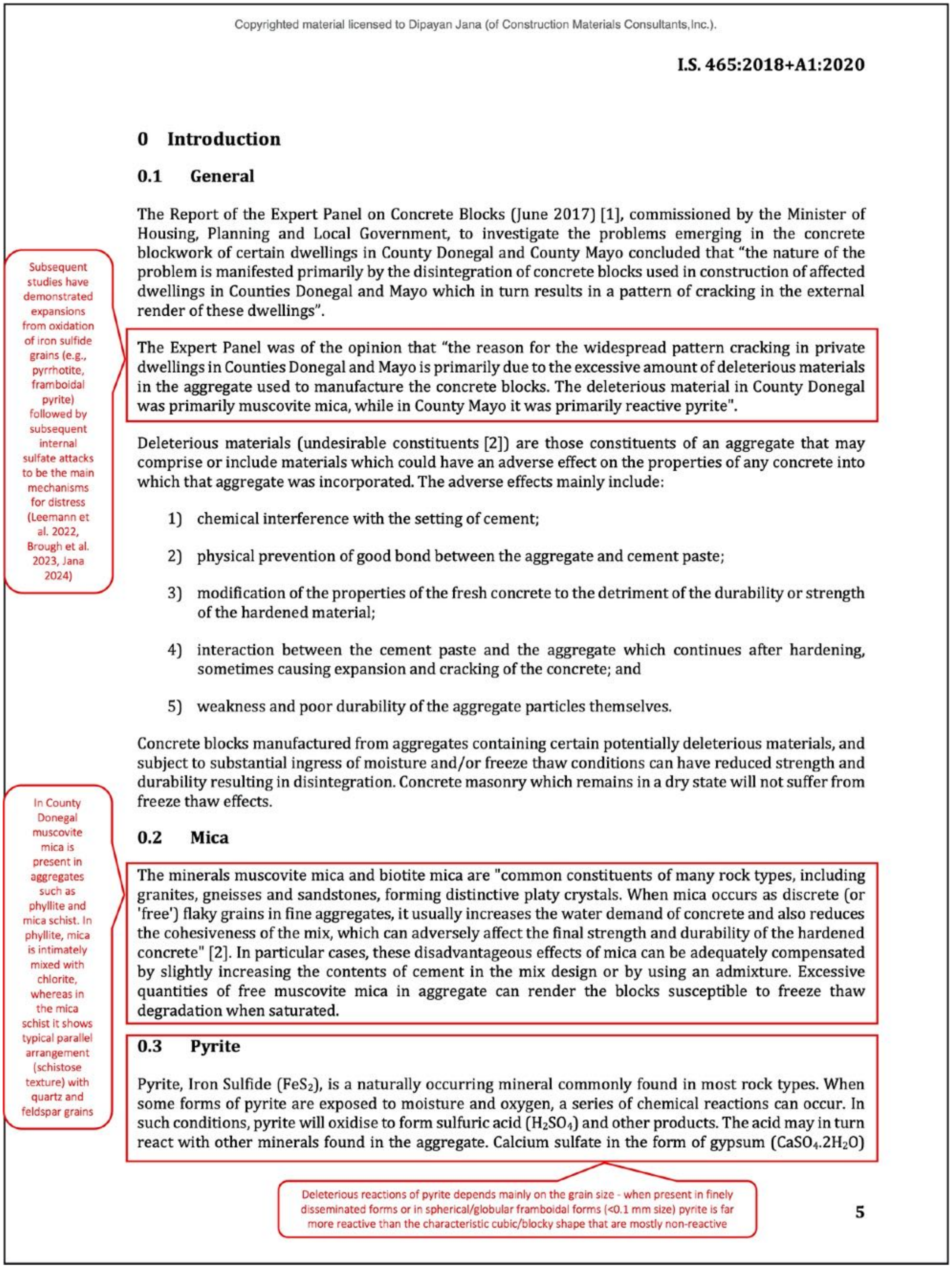
A new Annex F which contains the text of the clarification is included in this amendment along with an additional note in Clause 8.3 referring the reader to the new Annex F.

A further footnote is included in Table D.2 referring the reader to the new Annex F. ⟨A1

Compliance with this Irish Standard does not of itself confer immunity from legal obligations.

In line with international standards practice the following representation of numbers and numerical values apply:

- The decimal point is shown as a comma (,) throughout this Irish Standard.
- Each group of three digits reading to the left or to the right of a decimal sign are separated by a space from the preceding digits or following digits respectively.



0 Introduction

0.1 General

The Report of the Expert Panel on Concrete Blocks (June 2017) [1], commissioned by the Minister of Housing, Planning and Local Government, to investigate the problems emerging in the concrete blockwork of certain dwellings in County Donegal and County Mayo concluded that "the nature of the problem is manifested primarily by the disintegration of concrete blocks used in construction of affected dwellings in Counties Donegal and Mayo which in turn results in a pattern of cracking in the external render of these dwellings".

The Expert Panel was of the opinion that "the reason for the widespread pattern cracking in private dwellings in Counties Donegal and Mayo is primarily due to the excessive amount of deleterious materials in the aggregate used to manufacture the concrete blocks. The deleterious material in County Donegal was primarily muscovite mica, while in County Mayo it was primarily reactive pyrite".

Subsequent studies have demonstrated expansions from oxidation of iron sulfide grains (e.g., pyrrhotite, framboidal pyrite) followed by subsequent internal sulfate attacks to be the main mechanisms for distress (Leemann et al. 2022, Brough et al. 2023, Jana 2024)

Deleterious materials (undesirable constituents [2]) are those constituents of an aggregate that may comprise or include materials which could have an adverse effect on the properties of any concrete into which that aggregate was incorporated. The adverse effects mainly include:

1) chemical interference with the setting of cement;
2) physical prevention of good bond between the aggregate and cement paste;
3) modification of the properties of the fresh concrete to the detriment of the durability or strength of the hardened material;
4) interaction between the cement paste and the aggregate which continues after hardening, sometimes causing expansion and cracking of the concrete; and
5) weakness and poor durability of the aggregate particles themselves.

Concrete blocks manufactured from aggregates containing certain potentially deleterious materials, and subject to substantial ingress of moisture and/or freeze thaw conditions can have reduced strength and durability resulting in disintegration. Concrete masonry which remains in a dry state will not suffer from freeze thaw effects.

0.2 Mica

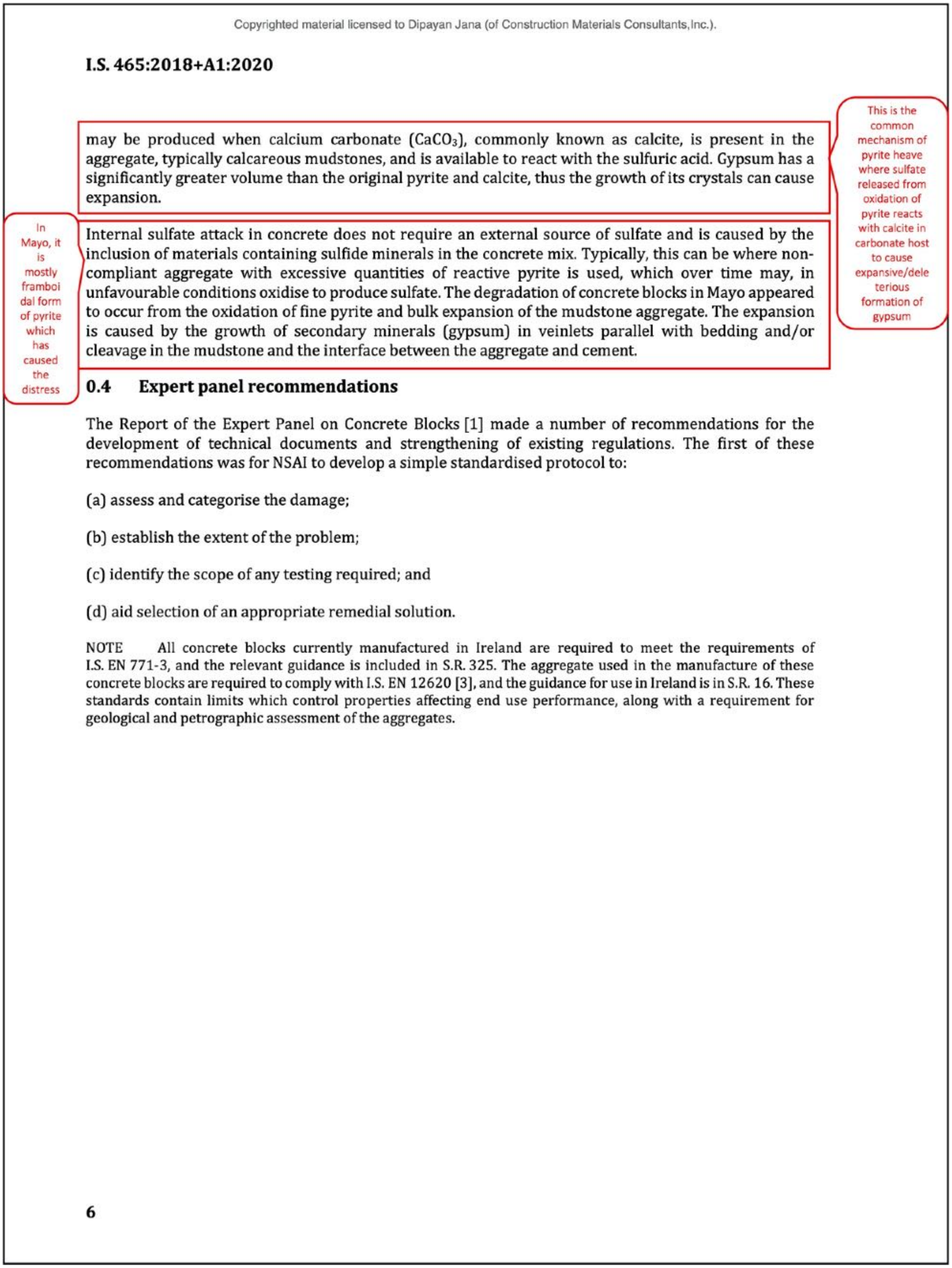
The minerals muscovite mica and biotite mica are "common constituents of many rock types, including granites, gneisses and sandstones, forming distinctive platy crystals. When mica occurs as discrete (or 'free') flaky grains in fine aggregates, it usually increases the water demand of concrete and also reduces the cohesiveness of the mix, which can adversely affect the final strength and durability of the hardened concrete" [2]. In particular cases, these disadvantageous effects of mica can be adequately compensated by slightly increasing the contents of cement in the mix design or by using an admixture. Excessive quantities of free muscovite mica in aggregate can render the blocks susceptible to freeze thaw degradation when saturated.

In County Donegal muscovite mica is present in aggregates such as phyllite and mica schist. In phyllite, mica is intimately mixed with chlorite, whereas in the mica schist it shows typical parallel arrangement (schistose texture) with quartz and feldspar grains

0.3 Pyrite

Pyrite, Iron Sulfide (FeS_2), is a naturally occurring mineral commonly found in most rock types. When some forms of pyrite are exposed to moisture and oxygen, a series of chemical reactions can occur. In such conditions, pyrite will oxidise to form sulfuric acid (H_2SO_4) and other products. The acid may in turn react with other minerals found in the aggregate. Calcium sulfate in the form of gypsum ($CaSO_4.2H_2O$)

Deleterious reactions of pyrite depends mainly on the grain size - when present in finely disseminated forms or in spherical/globular framboidal forms (<0.1 mm size) pyrite is far more reactive than the characteristic cubic/blocky shape that are mostly non-reactive



may be produced when calcium carbonate ($CaCO_3$), commonly known as calcite, is present in the aggregate, typically calcareous mudstones, and is available to react with the sulfuric acid. Gypsum has a significantly greater volume than the original pyrite and calcite, thus the growth of its crystals can cause expansion.

This is the common mechanism of pyrite heave where sulfate released from oxidation of pyrite reacts with calcite in carbonate host to cause expansive/deleterious formation of gypsum

Internal sulfate attack in concrete does not require an external source of sulfate and is caused by the inclusion of materials containing sulfide minerals in the concrete mix. Typically, this can be where non-compliant aggregate with excessive quantities of reactive pyrite is used, which over time may, in unfavourable conditions oxidise to produce sulfate. The degradation of concrete blocks in Mayo appeared to occur from the oxidation of fine pyrite and bulk expansion of the mudstone aggregate. The expansion is caused by the growth of secondary minerals (gypsum) in veinlets parallel with bedding and/or cleavage in the mudstone and the interface between the aggregate and cement.

In Mayo, it is mostly framboidal form of pyrite which has caused the distress

0.4 Expert panel recommendations

The Report of the Expert Panel on Concrete Blocks [1] made a number of recommendations for the development of technical documents and strengthening of existing regulations. The first of these recommendations was for NSAI to develop a simple standardised protocol to:

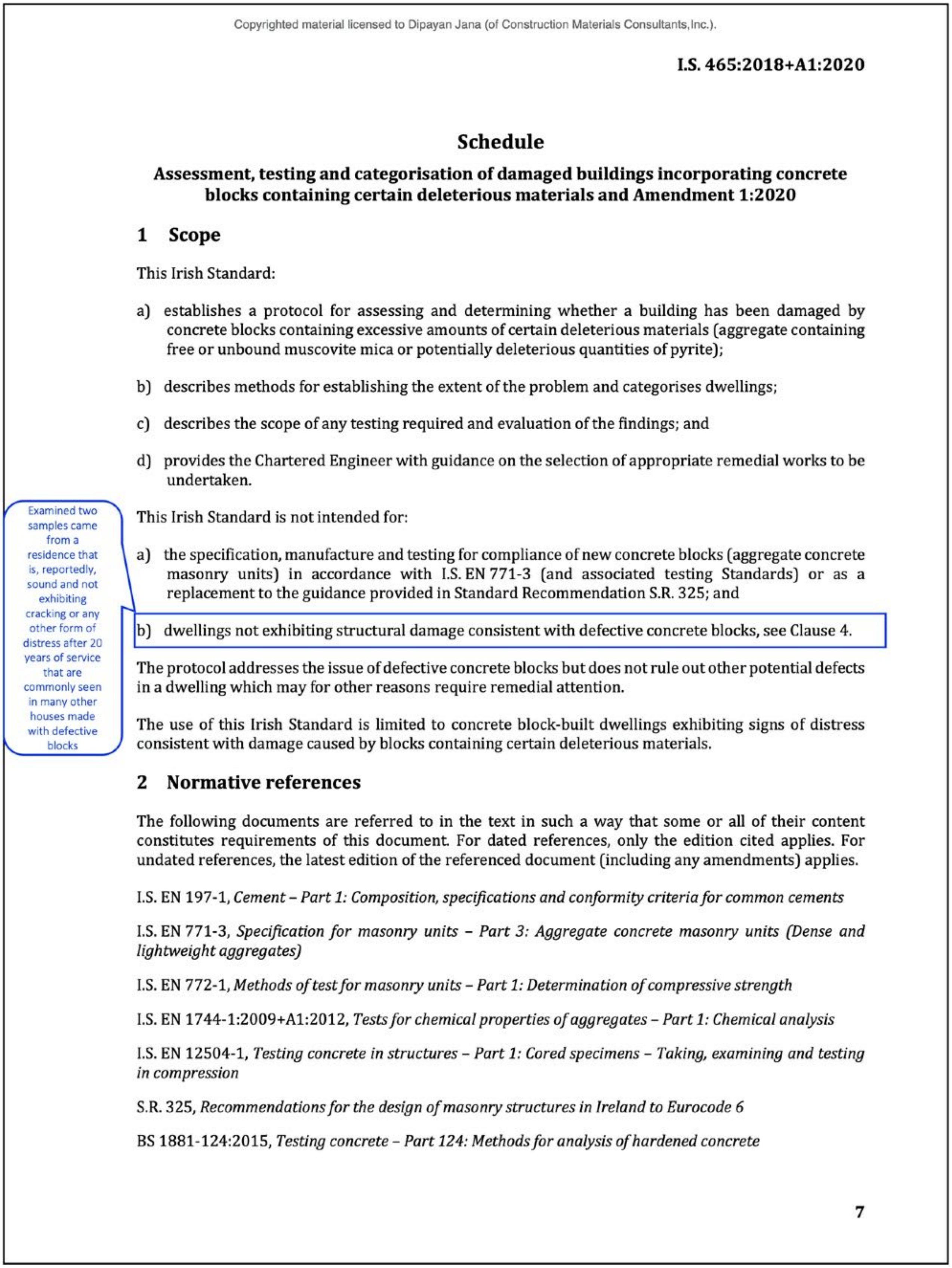
(a) assess and categorise the damage;

(b) establish the extent of the problem;

(c) identify the scope of any testing required; and

(d) aid selection of an appropriate remedial solution.

NOTE All concrete blocks currently manufactured in Ireland are required to meet the requirements of I.S. EN 771-3, and the relevant guidance is included in S.R. 325. The aggregate used in the manufacture of these concrete blocks are required to comply with I.S. EN 12620 [3], and the guidance for use in Ireland is in S.R. 16. These standards contain limits which control properties affecting end use performance, along with a requirement for geological and petrographic assessment of the aggregates.



Schedule

Assessment, testing and categorisation of damaged buildings incorporating concrete blocks containing certain deleterious materials and Amendment 1:2020

1 Scope

This Irish Standard:

a) establishes a protocol for assessing and determining whether a building has been damaged by concrete blocks containing excessive amounts of certain deleterious materials (aggregate containing free or unbound muscovite mica or potentially deleterious quantities of pyrite);

b) describes methods for establishing the extent of the problem and categorises dwellings;

c) describes the scope of any testing required and evaluation of the findings; and

d) provides the Chartered Engineer with guidance on the selection of appropriate remedial works to be undertaken.

This Irish Standard is not intended for:

a) the specification, manufacture and testing for compliance of new concrete blocks (aggregate concrete masonry units) in accordance with I.S. EN 771-3 (and associated testing Standards) or as a replacement to the guidance provided in Standard Recommendation S.R. 325; and

b) dwellings not exhibiting structural damage consistent with defective concrete blocks, see Clause 4.

Examined two samples came from a residence that is, reportedly, sound and not exhibiting cracking or any other form of distress after 20 years of service that are commonly seen in many other houses made with defective blocks

The protocol addresses the issue of defective concrete blocks but does not rule out other potential defects in a dwelling which may for other reasons require remedial attention.

The use of this Irish Standard is limited to concrete block-built dwellings exhibiting signs of distress consistent with damage caused by blocks containing certain deleterious materials.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

I.S. EN 197-1, *Cement – Part 1: Composition, specifications and conformity criteria for common cements*

I.S. EN 771-3, *Specification for masonry units – Part 3: Aggregate concrete masonry units (Dense and lightweight aggregates)*

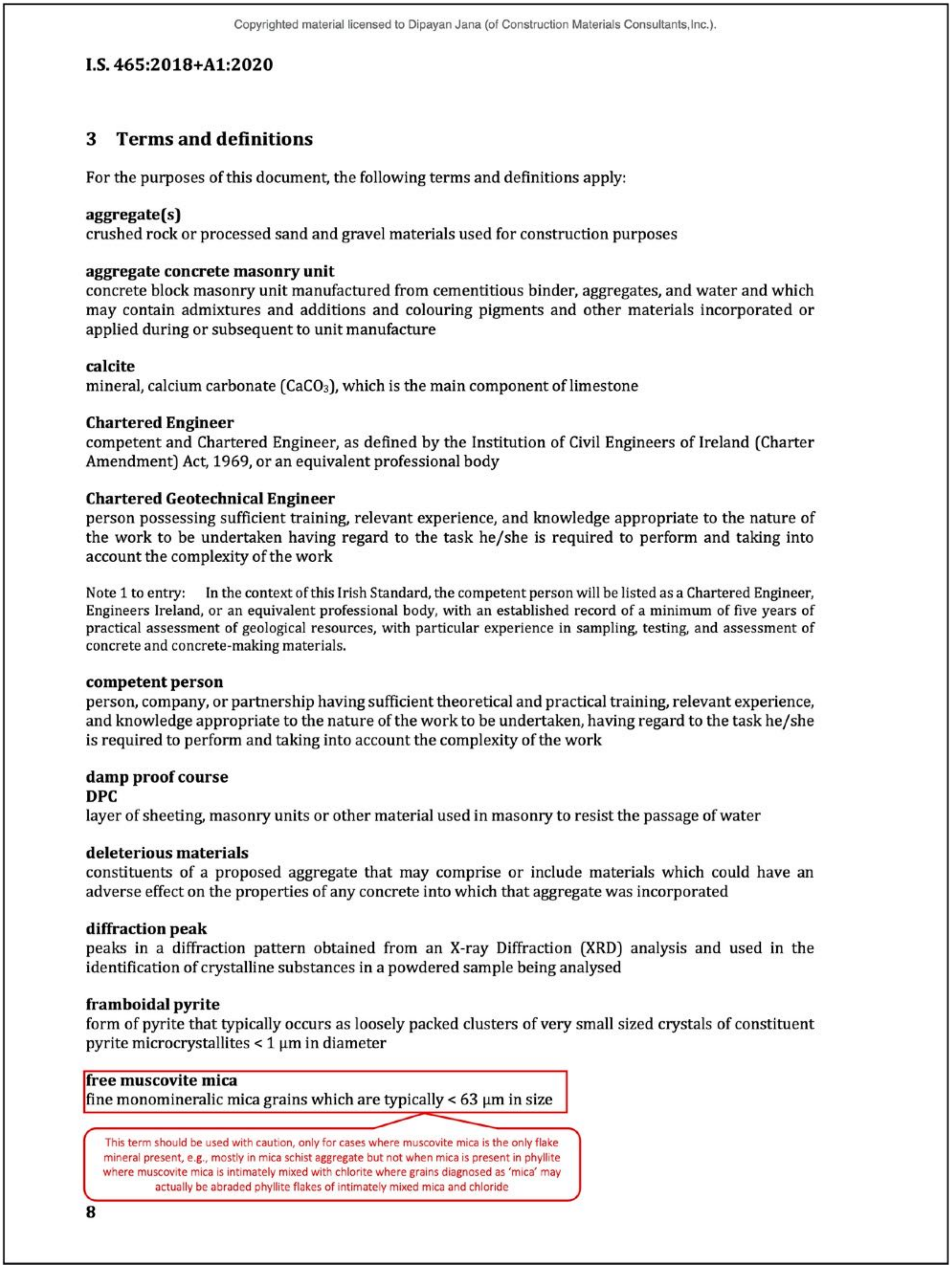
I.S. EN 772-1, *Methods of test for masonry units – Part 1: Determination of compressive strength*

I.S. EN 1744-1:2009+A1:2012, *Tests for chemical properties of aggregates – Part 1: Chemical analysis*

I.S. EN 12504-1, *Testing concrete in structures – Part 1: Cored specimens – Taking, examining and testing in compression*

S.R. 325, *Recommendations for the design of masonry structures in Ireland to Eurocode 6*

BS 1881-124:2015, *Testing concrete – Part 124: Methods for analysis of hardened concrete*



I.S. 465:2018+A1:2020

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply:

aggregate(s)
crushed rock or processed sand and gravel materials used for construction purposes

aggregate concrete masonry unit
concrete block masonry unit manufactured from cementitious binder, aggregates, and water and which may contain admixtures and additions and colouring pigments and other materials incorporated or applied during or subsequent to unit manufacture

calcite
mineral, calcium carbonate ($CaCO_3$), which is the main component of limestone

Chartered Engineer
competent and Chartered Engineer, as defined by the Institution of Civil Engineers of Ireland (Charter Amendment) Act, 1969, or an equivalent professional body

Chartered Geotechnical Engineer
person possessing sufficient training, relevant experience, and knowledge appropriate to the nature of the work to be undertaken having regard to the task he/she is required to perform and taking into account the complexity of the work

Note 1 to entry: In the context of this Irish Standard, the competent person will be listed as a Chartered Engineer, Engineers Ireland, or an equivalent professional body, with an established record of a minimum of five years of practical assessment of geological resources, with particular experience in sampling, testing, and assessment of concrete and concrete-making materials.

competent person
person, company, or partnership having sufficient theoretical and practical training, relevant experience, and knowledge appropriate to the nature of the work to be undertaken, having regard to the task he/she is required to perform and taking into account the complexity of the work

damp proof course
DPC
layer of sheeting, masonry units or other material used in masonry to resist the passage of water

deleterious materials
constituents of a proposed aggregate that may comprise or include materials which could have an adverse effect on the properties of any concrete into which that aggregate was incorporated

diffraction peak
peaks in a diffraction pattern obtained from an X-ray Diffraction (XRD) analysis and used in the identification of crystalline substances in a powdered sample being analysed

framboidal pyrite
form of pyrite that typically occurs as loosely packed clusters of very small sized crystals of constituent pyrite microcrystallites < 1 µm in diameter

free muscovite mica
fine monomineralic mica grains which are typically < 63 µm in size

This term should be used with caution, only for cases where muscovite mica is the only flake mineral present, e.g., mostly in mica schist aggregate but not when mica is present in phyllite where muscovite mica is intimately mixed with chlorite where grains diagnosed as 'mica' may actually be abraded phyllite flakes of intimately mixed mica and chloride

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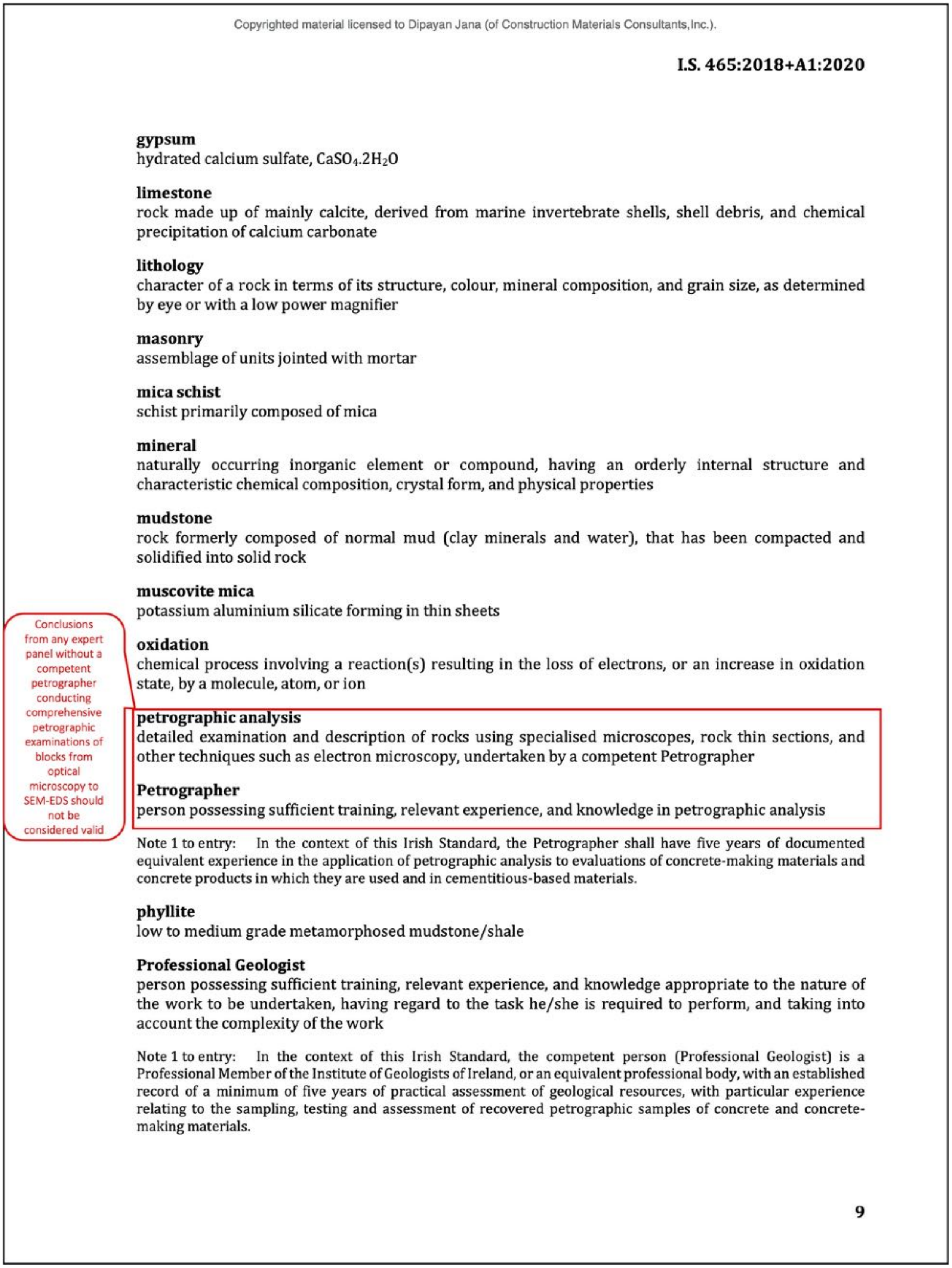

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gypsum
hydrated calcium sulfate, $CaSO_4.2H_2O$

limestone
rock made up of mainly calcite, derived from marine invertebrate shells, shell debris, and chemical precipitation of calcium carbonate

lithology
character of a rock in terms of its structure, colour, mineral composition, and grain size, as determined by eye or with a low power magnifier

masonry
assemblage of units jointed with mortar

mica schist
schist primarily composed of mica

mineral
naturally occurring inorganic element or compound, having an orderly internal structure and characteristic chemical composition, crystal form, and physical properties

mudstone
rock formerly composed of normal mud (clay minerals and water), that has been compacted and solidified into solid rock

muscovite mica
potassium aluminium silicate forming in thin sheets

oxidation
chemical process involving a reaction(s) resulting in the loss of electrons, or an increase in oxidation state, by a molecule, atom, or ion

petrographic analysis
detailed examination and description of rocks using specialised microscopes, rock thin sections, and other techniques such as electron microscopy, undertaken by a competent Petrographer

Petrographer
person possessing sufficient training, relevant experience, and knowledge in petrographic analysis

Conclusions from any expert panel without a competent petrographer conducting comprehensive petrographic examinations of blocks from optical microscopy to SEM-EDS should not be considered valid

Note 1 to entry: In the context of this Irish Standard, the Petrographer shall have five years of documented equivalent experience in the application of petrographic analysis to evaluations of concrete-making materials and concrete products in which they are used and in cementitious-based materials.

phyllite
low to medium grade metamorphosed mudstone/shale

Professional Geologist
person possessing sufficient training, relevant experience, and knowledge appropriate to the nature of the work to be undertaken, having regard to the task he/she is required to perform, and taking into account the complexity of the work

Note 1 to entry: In the context of this Irish Standard, the competent person (Professional Geologist) is a Professional Member of the Institute of Geologists of Ireland, or an equivalent professional body, with an established record of a minimum of five years of practical assessment of geological resources, with particular experience relating to the sampling, testing and assessment of recovered petrographic samples of concrete and concrete-making materials.

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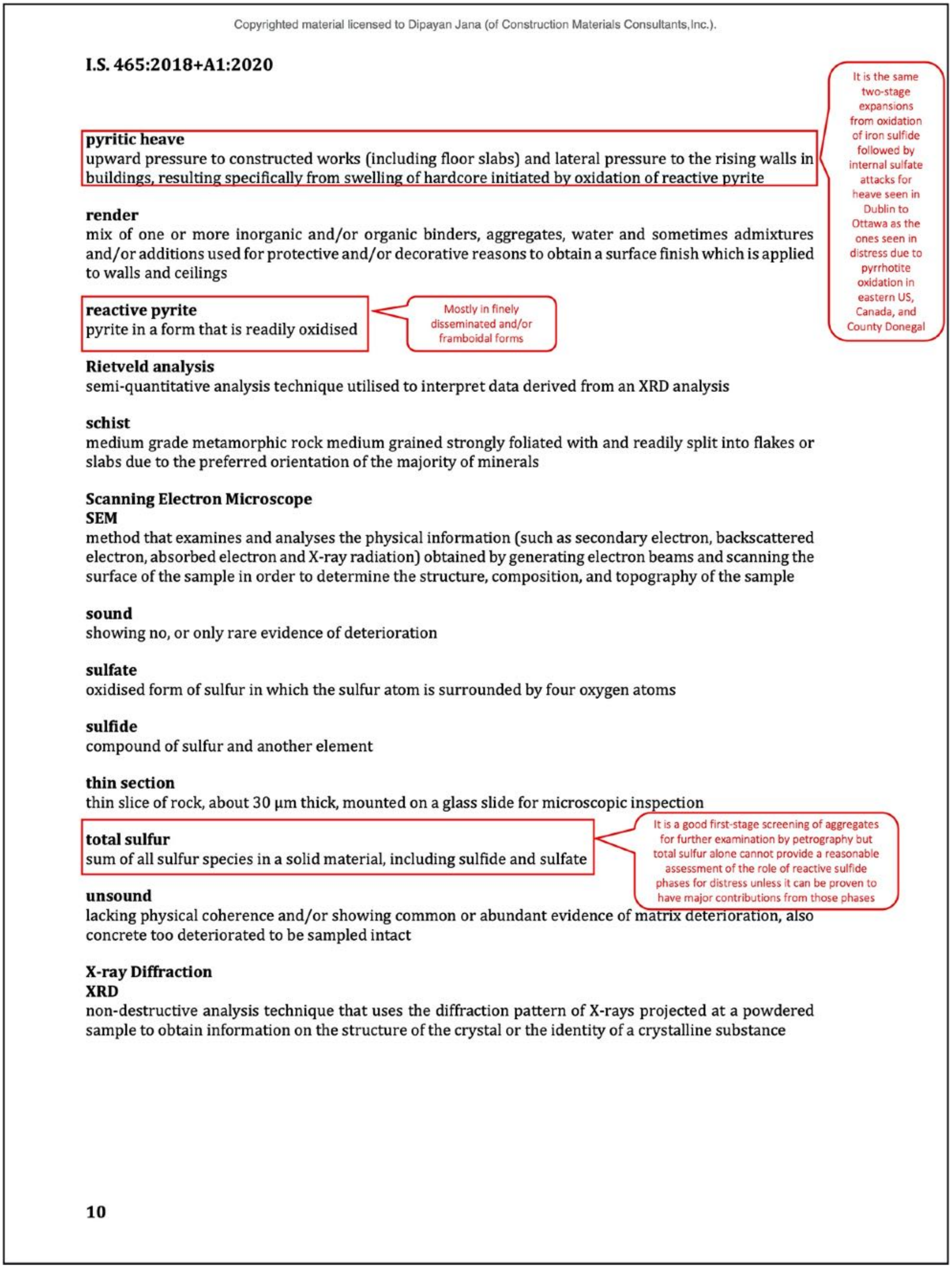

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pyritic heave
upward pressure to constructed works (including floor slabs) and lateral pressure to the rising walls in buildings, resulting specifically from swelling of hardcore initiated by oxidation of reactive pyrite

It is the same two-stage expansions from oxidation of iron sulfide followed by internal sulfate attacks for heave seen in Dublin to Ottawa as the ones seen in distress due to pyrrhotite oxidation in eastern US, Canada, and County Donegal

render
mix of one or more inorganic and/or organic binders, aggregates, water and sometimes admixtures and/or additions used for protective and/or decorative reasons to obtain a surface finish which is applied to walls and ceilings

reactive pyrite
pyrite in a form that is readily oxidised

Mostly in finely disseminated and/or framboidal forms

Rietveld analysis
semi-quantitative analysis technique utilised to interpret data derived from an XRD analysis

schist
medium grade metamorphic rock medium grained strongly foliated with and readily split into flakes or slabs due to the preferred orientation of the majority of minerals

Scanning Electron Microscope
SEM
method that examines and analyses the physical information (such as secondary electron, backscattered electron, absorbed electron and X-ray radiation) obtained by generating electron beams and scanning the surface of the sample in order to determine the structure, composition, and topography of the sample

sound
showing no, or only rare evidence of deterioration

sulfate
oxidised form of sulfur in which the sulfur atom is surrounded by four oxygen atoms

sulfide
compound of sulfur and another element

thin section
thin slice of rock, about 30 μm thick, mounted on a glass slide for microscopic inspection

total sulfur
sum of all sulfur species in a solid material, including sulfide and sulfate

It is a good first-stage screening of aggregates for further examination by petrography but total sulfur alone cannot provide a reasonable assessment of the role of reactive sulfide phases for distress unless it can be proven to have major contributions from those phases

unsound
lacking physical coherence and/or showing common or abundant evidence of matrix deterioration, also concrete too deteriorated to be sampled intact

X-ray Diffraction
XRD
non-destructive analysis technique that uses the diffraction pattern of X-rays projected at a powdered sample to obtain information on the structure of the crystal or the identity of a crystalline substance

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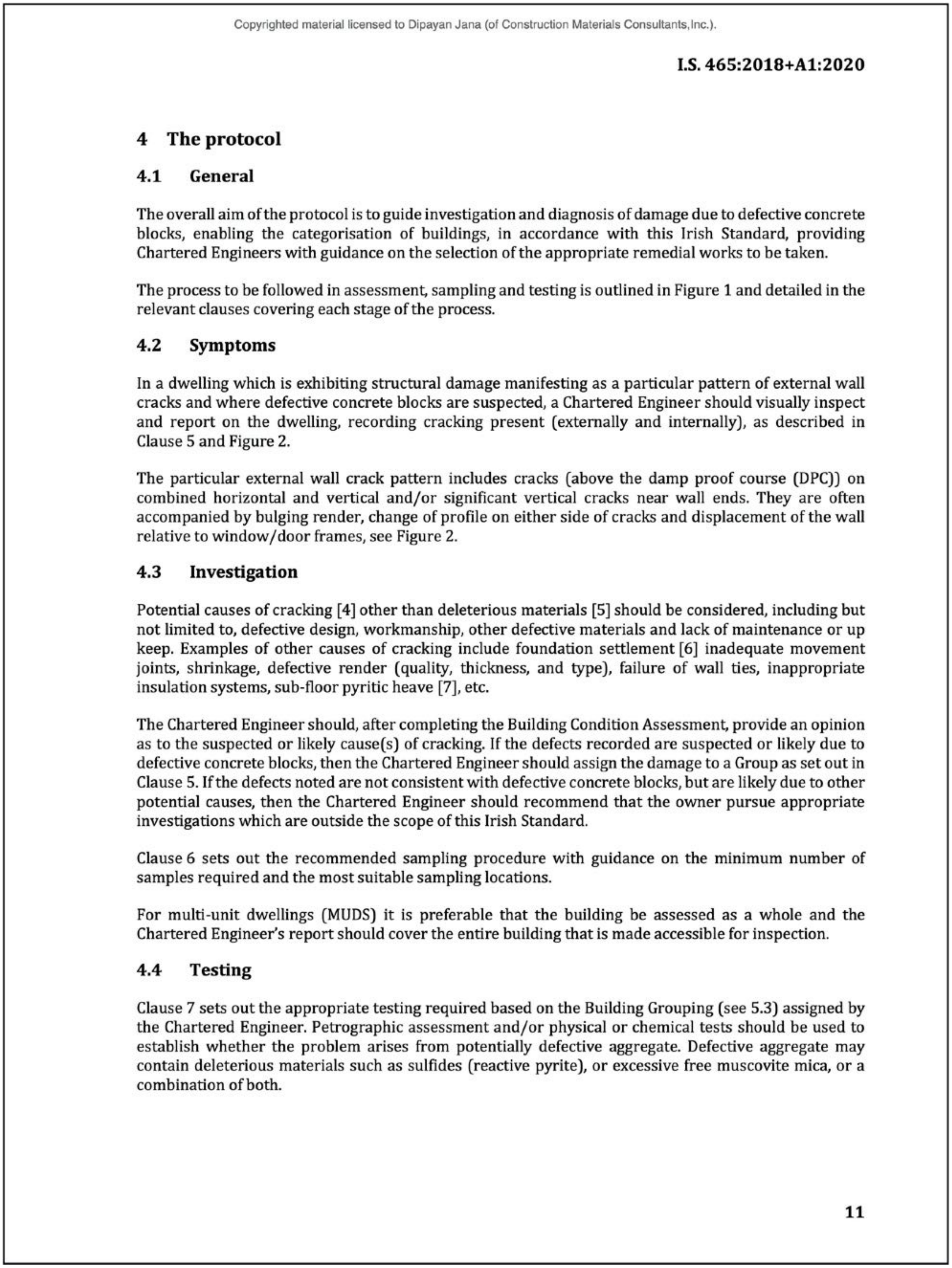


4 The protocol

4.1 General

The overall aim of the protocol is to guide investigation and diagnosis of damage due to defective concrete blocks, enabling the categorisation of buildings, in accordance with this Irish Standard, providing Chartered Engineers with guidance on the selection of the appropriate remedial works to be taken.

The process to be followed in assessment, sampling and testing is outlined in Figure 1 and detailed in the relevant clauses covering each stage of the process.

4.2 Symptoms

In a dwelling which is exhibiting structural damage manifesting as a particular pattern of external wall cracks and where defective concrete blocks are suspected, a Chartered Engineer should visually inspect and report on the dwelling, recording cracking present (externally and internally), as described in Clause 5 and Figure 2.

The particular external wall crack pattern includes cracks (above the damp proof course (DPC)) on combined horizontal and vertical and/or significant vertical cracks near wall ends. They are often accompanied by bulging render, change of profile on either side of cracks and displacement of the wall relative to window/door frames, see Figure 2.

4.3 Investigation

Potential causes of cracking [4] other than deleterious materials [5] should be considered, including but not limited to, defective design, workmanship, other defective materials and lack of maintenance or up keep. Examples of other causes of cracking include foundation settlement [6] inadequate movement joints, shrinkage, defective render (quality, thickness, and type), failure of wall ties, inappropriate insulation systems, sub-floor pyritic heave [7], etc.

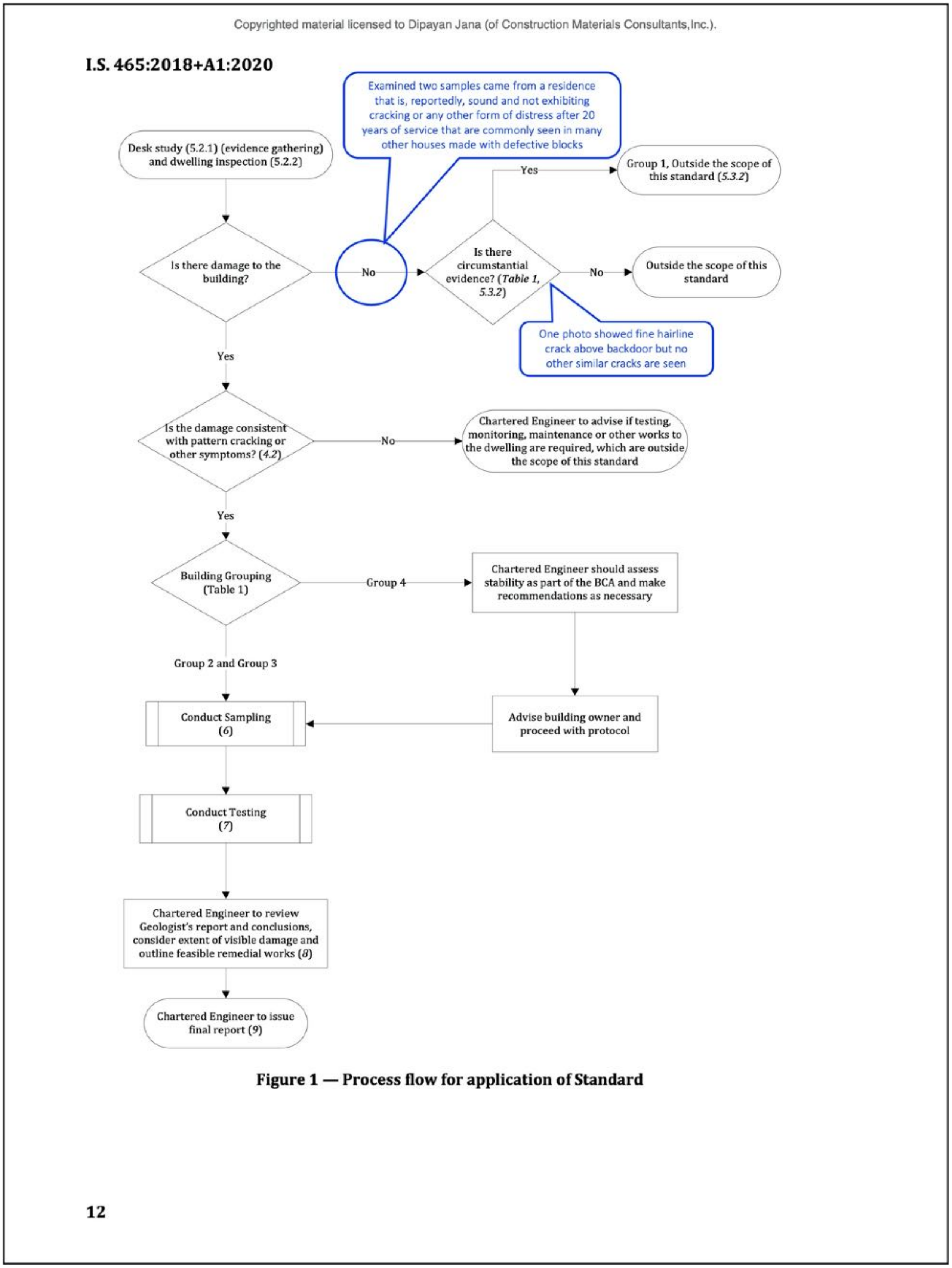
The Chartered Engineer should, after completing the Building Condition Assessment, provide an opinion as to the suspected or likely cause(s) of cracking. If the defects recorded are suspected or likely due to defective concrete blocks, then the Chartered Engineer should assign the damage to a Group as set out in Clause 5. If the defects noted are not consistent with defective concrete blocks, but are likely due to other potential causes, then the Chartered Engineer should recommend that the owner pursue appropriate investigations which are outside the scope of this Irish Standard.

Clause 6 sets out the recommended sampling procedure with guidance on the minimum number of samples required and the most suitable sampling locations.

For multi-unit dwellings (MUDS) it is preferable that the building be assessed as a whole and the Chartered Engineer's report should cover the entire building that is made accessible for inspection.

4.4 Testing

Clause 7 sets out the appropriate testing required based on the Building Grouping (see 5.3) assigned by the Chartered Engineer. Petrographic assessment and/or physical or chemical tests should be used to establish whether the problem arises from potentially defective aggregate. Defective aggregate may contain deleterious materials such as sulfides (reactive pyrite), or excessive free muscovite mica, or a combination of both.



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Examined two samples came from a residence that is, reportedly, sound and not exhibiting cracking or any other form of distress after 20 years of service that are commonly seen in many other houses made with defective blocks

Desk study (5.2.1) (evidence gathering) and dwelling inspection (5.2.2)

Is there damage to the building?

Is there circumstantial evidence? (*Table 1, 5.3.2*)

Group 1, Outside the scope of this standard (*5.3.2*)

Outside the scope of this standard

One photo showed fine hairline crack above backdoor but no other similar cracks are seen

Is the damage consistent with pattern cracking or other symptoms? (*4.2*)

Chartered Engineer to advise if testing, monitoring, maintenance or other works to the dwelling are required, which are outside the scope of this standard

Building Grouping (Table 1)

Chartered Engineer should assess stability as part of the BCA and make recommendations as necessary

Conduct Sampling (*6*)

Advise building owner and proceed with protocol

Conduct Testing (*7*)

Chartered Engineer to review Geologist's report and conclusions, consider extent of visible damage and outline feasible remedial works (*8*)

Chartered Engineer to issue final report (*9*)

Figure 1 — Process flow for application of Standard

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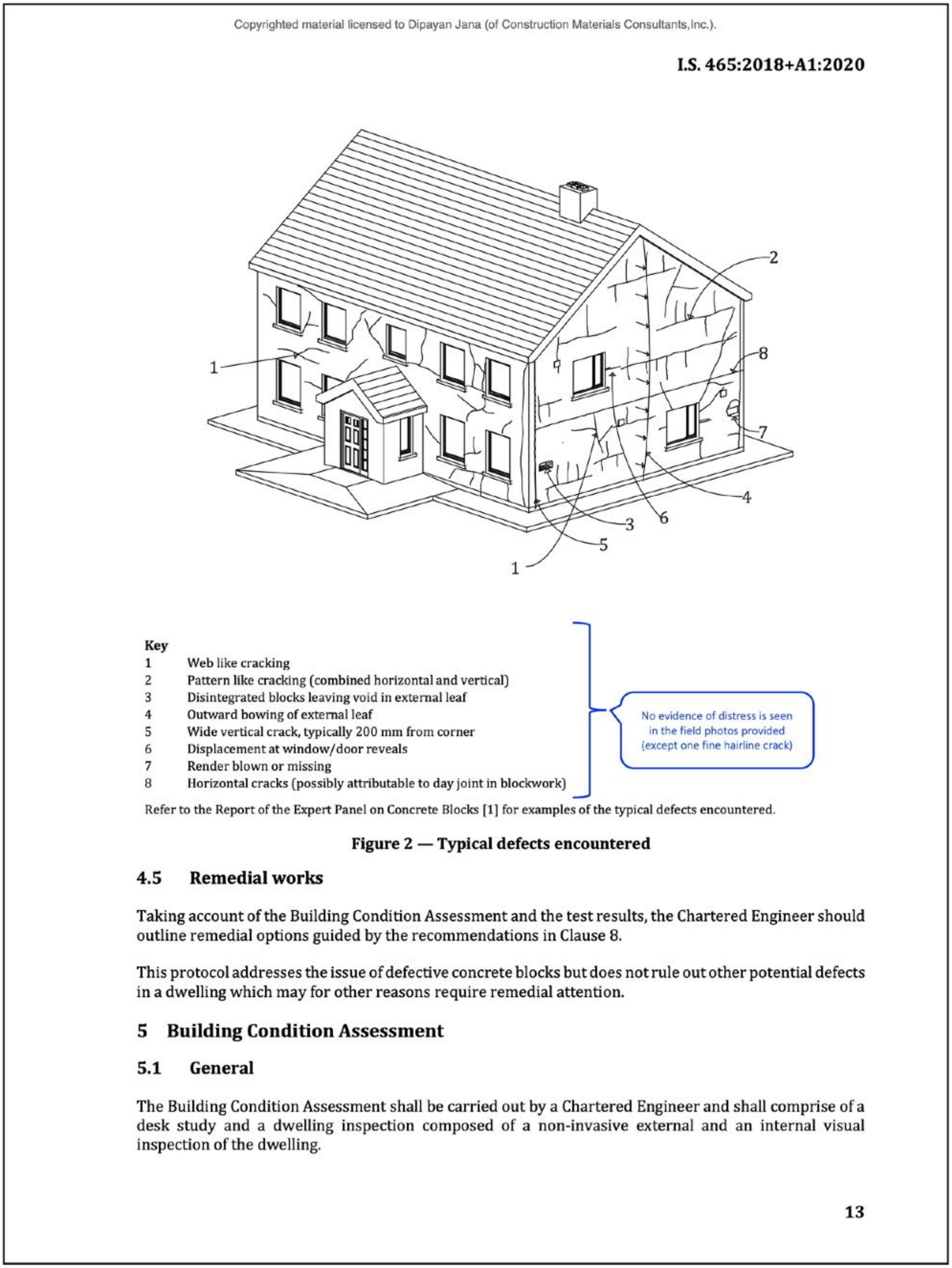


Key

1	Web like cracking
2	Pattern like cracking (combined horizontal and vertical)
3	Disintegrated blocks leaving void in external leaf
4	Outward bowing of external leaf
5	Wide vertical crack, typically 200 mm from corner
6	Displacement at window/door reveals
7	Render blown or missing
8	Horizontal cracks (possibly attributable to day joint in blockwork)

No evidence of distress is seen in the field photos provided (except one fine hairline crack)

Refer to the Report of the Expert Panel on Concrete Blocks [1] for examples of the typical defects encountered.

Figure 2 — Typical defects encountered

4.5 Remedial works

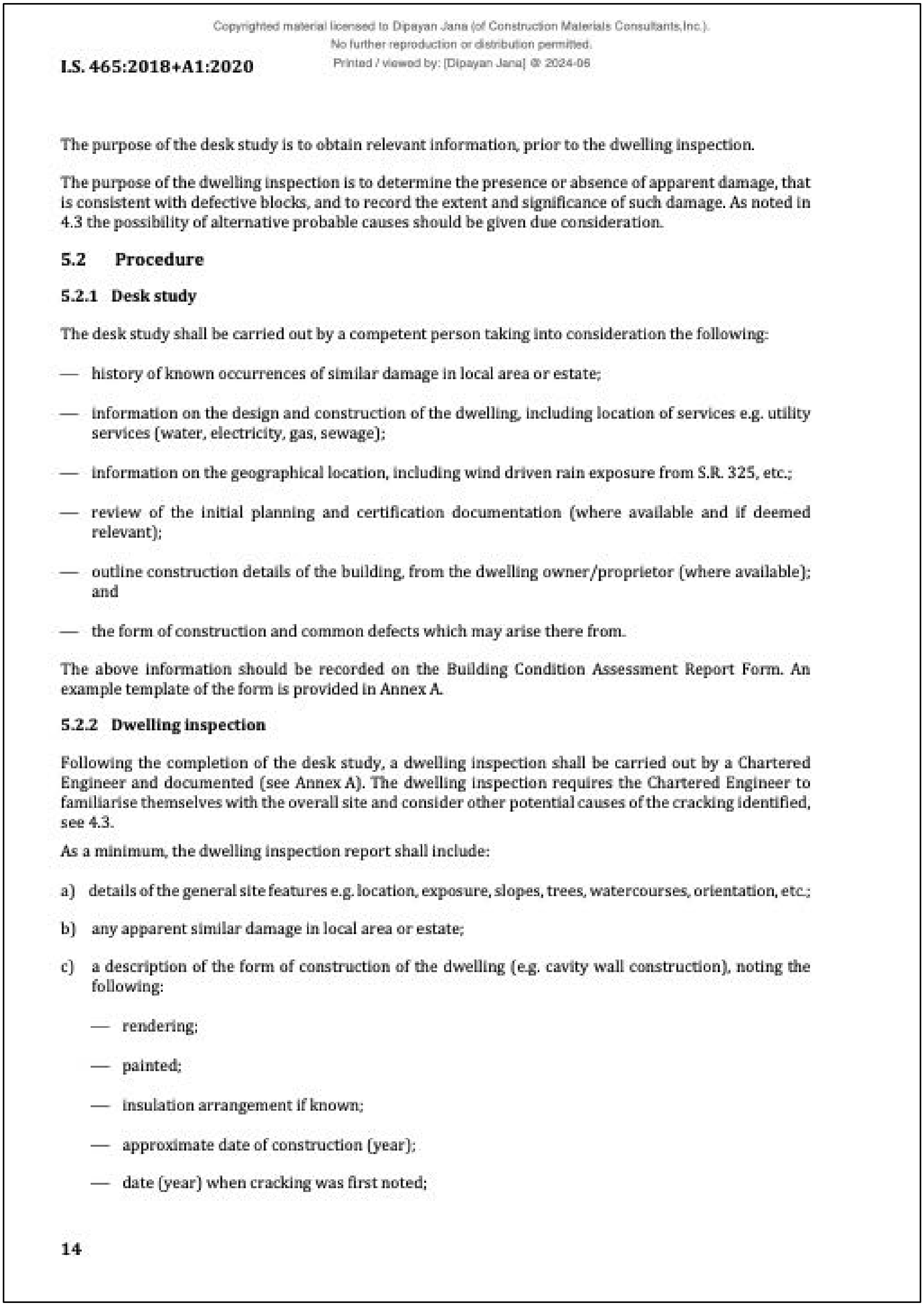
Taking account of the Building Condition Assessment and the test results, the Chartered Engineer should outline remedial options guided by the recommendations in Clause 8.

This protocol addresses the issue of defective concrete blocks but does not rule out other potential defects in a dwelling which may for other reasons require remedial attention.

5 Building Condition Assessment

5.1 General

The Building Condition Assessment shall be carried out by a Chartered Engineer and shall comprise of a desk study and a dwelling inspection composed of a non-invasive external and an internal visual inspection of the dwelling.



The purpose of the desk study is to obtain relevant information, prior to the dwelling inspection.

The purpose of the dwelling inspection is to determine the presence or absence of apparent damage, that is consistent with defective blocks, and to record the extent and significance of such damage. As noted in 4.3 the possibility of alternative probable causes should be given due consideration.

5.2 Procedure

5.2.1 Desk study

The desk study shall be carried out by a competent person taking into consideration the following:

- history of known occurrences of similar damage in local area or estate;
- information on the design and construction of the dwelling, including location of services e.g. utility services (water, electricity, gas, sewage);
- information on the geographical location, including wind driven rain exposure from S.R. 325, etc.;
- review of the initial planning and certification documentation (where available and if deemed relevant);
- outline construction details of the building, from the dwelling owner/proprietor (where available); and
- the form of construction and common defects which may arise there from.

The above information should be recorded on the Building Condition Assessment Report Form. An example template of the form is provided in Annex A.

5.2.2 Dwelling inspection

Following the completion of the desk study, a dwelling inspection shall be carried out by a Chartered Engineer and documented (see Annex A). The dwelling inspection requires the Chartered Engineer to familiarise themselves with the overall site and consider other potential causes of the cracking identified, see 4.3.

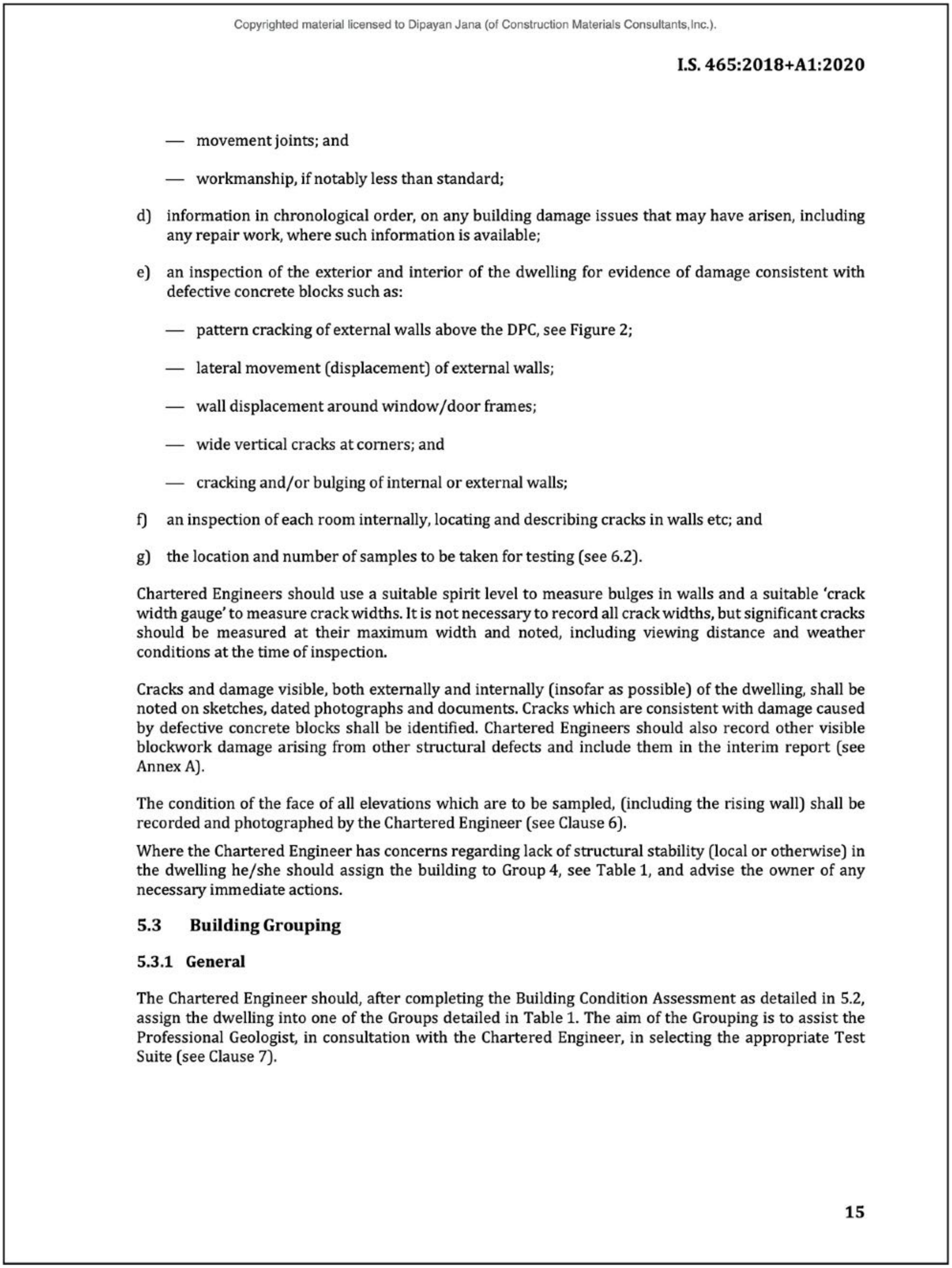
As a minimum, the dwelling inspection report shall include:

a) details of the general site features e.g. location, exposure, slopes, trees, watercourses, orientation, etc.;

b) any apparent similar damage in local area or estate;

c) a description of the form of construction of the dwelling (e.g. cavity wall construction), noting the following:

  - rendering;
  - painted;
  - insulation arrangement if known;
  - approximate date of construction (year);
  - date (year) when cracking was first noted;



— movement joints; and

— workmanship, if notably less than standard;

d) information in chronological order, on any building damage issues that may have arisen, including any repair work, where such information is available;

e) an inspection of the exterior and interior of the dwelling for evidence of damage consistent with defective concrete blocks such as:

— pattern cracking of external walls above the DPC, see Figure 2;

— lateral movement (displacement) of external walls;

— wall displacement around window/door frames;

— wide vertical cracks at corners; and

— cracking and/or bulging of internal or external walls;

f) an inspection of each room internally, locating and describing cracks in walls etc; and

g) the location and number of samples to be taken for testing (see 6.2).

Chartered Engineers should use a suitable spirit level to measure bulges in walls and a suitable 'crack width gauge' to measure crack widths. It is not necessary to record all crack widths, but significant cracks should be measured at their maximum width and noted, including viewing distance and weather conditions at the time of inspection.

Cracks and damage visible, both externally and internally (insofar as possible) of the dwelling, shall be noted on sketches, dated photographs and documents. Cracks which are consistent with damage caused by defective concrete blocks shall be identified. Chartered Engineers should also record other visible blockwork damage arising from other structural defects and include them in the interim report (see Annex A).

The condition of the face of all elevations which are to be sampled, (including the rising wall) shall be recorded and photographed by the Chartered Engineer (see Clause 6).

Where the Chartered Engineer has concerns regarding lack of structural stability (local or otherwise) in the dwelling he/she should assign the building to Group 4, see Table 1, and advise the owner of any necessary immediate actions.

5.3 Building Grouping

5.3.1 General

The Chartered Engineer should, after completing the Building Condition Assessment as detailed in 5.2, assign the dwelling into one of the Groups detailed in Table 1. The aim of the Grouping is to assist the Professional Geologist, in consultation with the Chartered Engineer, in selecting the appropriate Test Suite (see Clause 7).

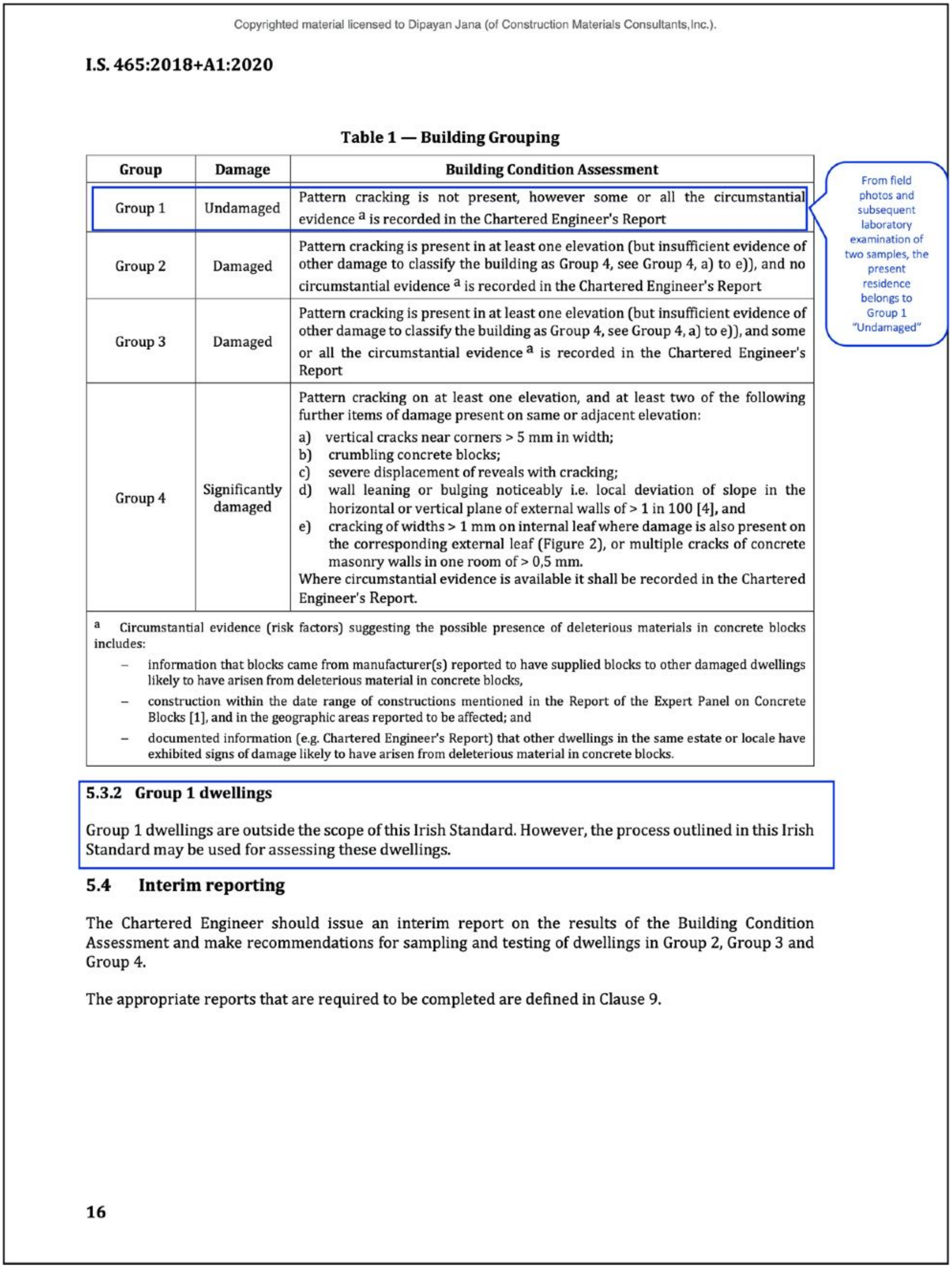


Table 1 — Building Grouping

Group	Damage	Building Condition Assessment
Group 1	Undamaged	Pattern cracking is not present, however some or all the circumstantial evidence [a] is recorded in the Chartered Engineer's Report
Group 2	Damaged	Pattern cracking is present in at least one elevation (but insufficient evidence of other damage to classify the building as Group 4, see Group 4, a) to e)), and no circumstantial evidence [a] is recorded in the Chartered Engineer's Report
Group 3	Damaged	Pattern cracking is present in at least one elevation (but insufficient evidence of other damage to classify the building as Group 4, see Group 4, a) to e)), and some or all the circumstantial evidence [a] is recorded in the Chartered Engineer's Report
Group 4	Significantly damaged	Pattern cracking on at least one elevation, and at least two of the following further items of damage present on same or adjacent elevation: a) vertical cracks near corners > 5 mm in width; b) crumbling concrete blocks; c) severe displacement of reveals with cracking; d) wall leaning or bulging noticeably i.e. local deviation of slope in the horizontal or vertical plane of external walls of > 1 in 100 [4], and e) cracking of widths > 1 mm on internal leaf where damage is also present on the corresponding external leaf (Figure 2), or multiple cracks of concrete masonry walls in one room of > 0,5 mm. Where circumstantial evidence is available it shall be recorded in the Chartered Engineer's Report.

[a] Circumstantial evidence (risk factors) suggesting the possible presence of deleterious materials in concrete blocks includes:

- information that blocks came from manufacturer(s) reported to have supplied blocks to other damaged dwellings likely to have arisen from deleterious material in concrete blocks,
- construction within the date range of constructions mentioned in the Report of the Expert Panel on Concrete Blocks [1], and in the geographic areas reported to be affected; and
- documented information (e.g. Chartered Engineer's Report) that other dwellings in the same estate or locale have exhibited signs of damage likely to have arisen from deleterious material in concrete blocks.

From field photos and subsequent laboratory examination of two samples, the present residence belongs to Group 1 "Undamaged"

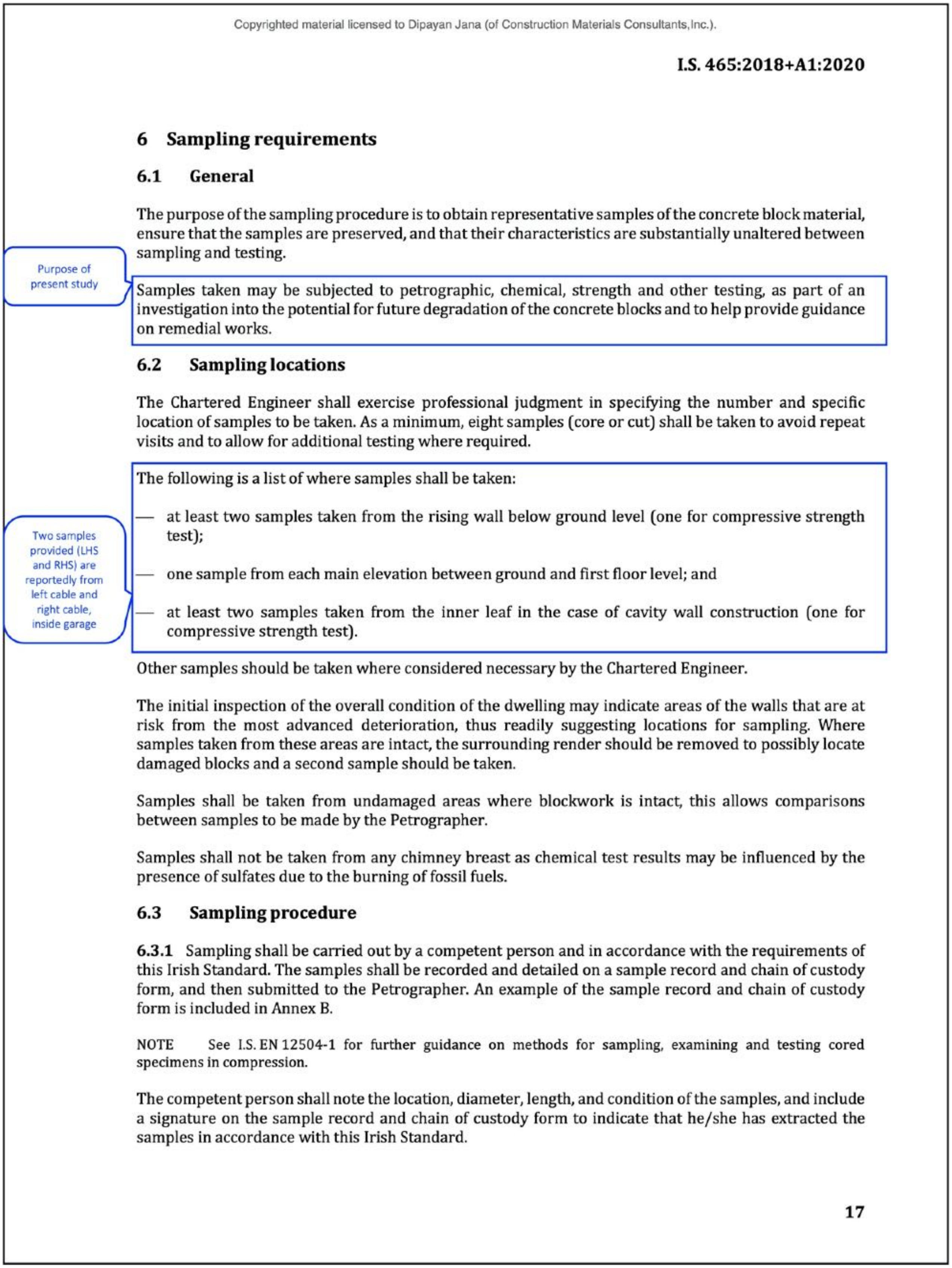
5.3.2 Group 1 dwellings

Group 1 dwellings are outside the scope of this Irish Standard. However, the process outlined in this Irish Standard may be used for assessing these dwellings.

5.4 Interim reporting

The Chartered Engineer should issue an interim report on the results of the Building Condition Assessment and make recommendations for sampling and testing of dwellings in Group 2, Group 3 and Group 4.

The appropriate reports that are required to be completed are defined in Clause 9.



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6 Sampling requirements

6.1 General

The purpose of the sampling procedure is to obtain representative samples of the concrete block material, ensure that the samples are preserved, and that their characteristics are substantially unaltered between sampling and testing.

Purpose of present study

Samples taken may be subjected to petrographic, chemical, strength and other testing, as part of an investigation into the potential for future degradation of the concrete blocks and to help provide guidance on remedial works.

6.2 Sampling locations

The Chartered Engineer shall exercise professional judgment in specifying the number and specific location of samples to be taken. As a minimum, eight samples (core or cut) shall be taken to avoid repeat visits and to allow for additional testing where required.

The following is a list of where samples shall be taken:

- at least two samples taken from the rising wall below ground level (one for compressive strength test);
- one sample from each main elevation between ground and first floor level; and
- at least two samples taken from the inner leaf in the case of cavity wall construction (one for compressive strength test).

Two samples provided (LHS and RHS) are reportedly from left cable and right cable, inside garage

Other samples should be taken where considered necessary by the Chartered Engineer.

The initial inspection of the overall condition of the dwelling may indicate areas of the walls that are at risk from the most advanced deterioration, thus readily suggesting locations for sampling. Where samples taken from these areas are intact, the surrounding render should be removed to possibly locate damaged blocks and a second sample should be taken.

Samples shall be taken from undamaged areas where blockwork is intact, this allows comparisons between samples to be made by the Petrographer.

Samples shall not be taken from any chimney breast as chemical test results may be influenced by the presence of sulfates due to the burning of fossil fuels.

6.3 Sampling procedure

6.3.1 Sampling shall be carried out by a competent person and in accordance with the requirements of this Irish Standard. The samples shall be recorded and detailed on a sample record and chain of custody form, and then submitted to the Petrographer. An example of the sample record and chain of custody form is included in Annex B.

NOTE See I.S. EN 12504-1 for further guidance on methods for sampling, examining and testing cored specimens in compression.

The competent person shall note the location, diameter, length, and condition of the samples, and include a signature on the sample record and chain of custody form to indicate that he/she has extracted the samples in accordance with this Irish Standard.

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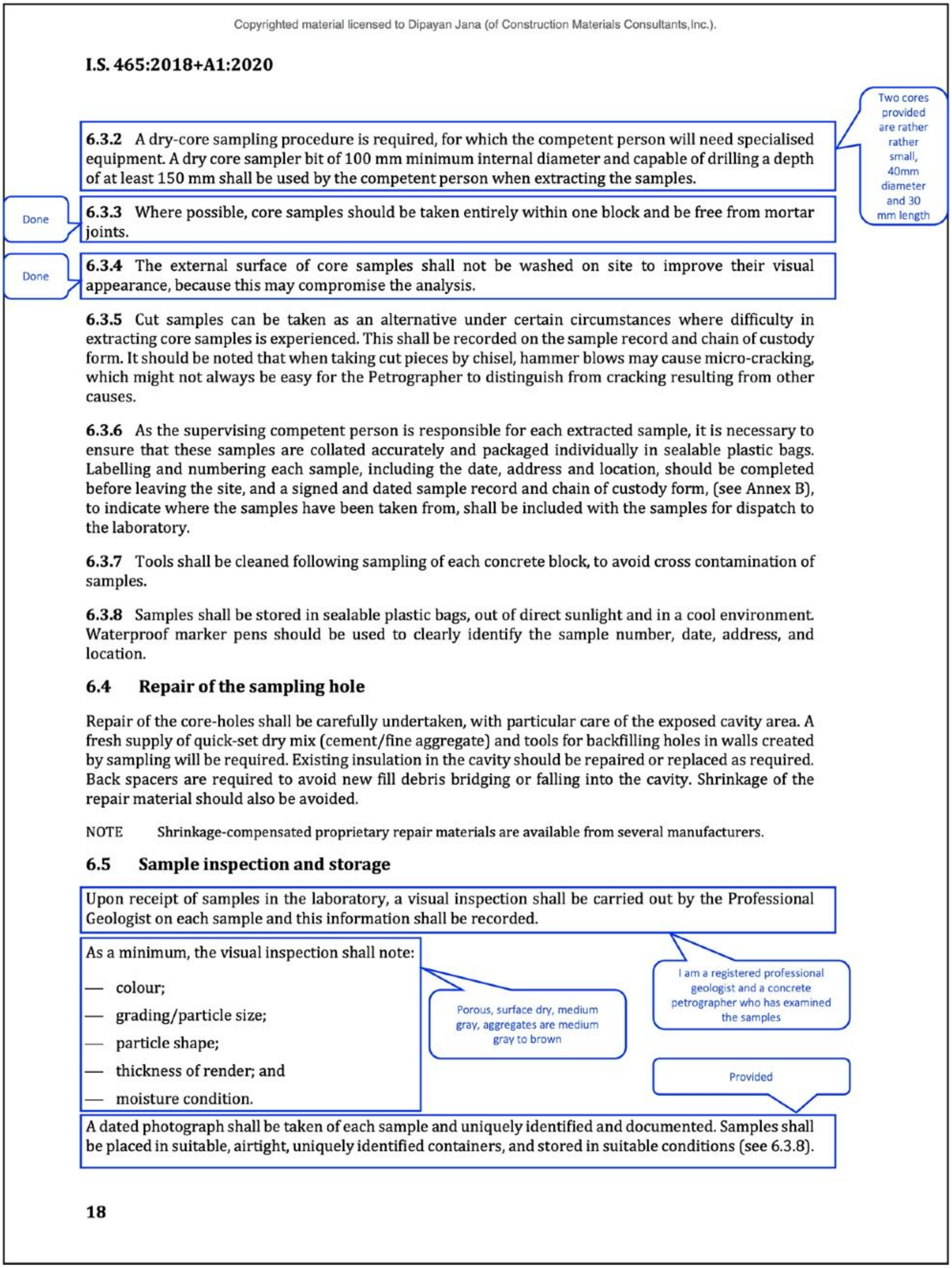

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6.3.2 A dry-core sampling procedure is required, for which the competent person will need specialised equipment. A dry core sampler bit of 100 mm minimum internal diameter and capable of drilling a depth of at least 150 mm shall be used by the competent person when extracting the samples.

Two cores provided are rather rather small, 40mm diameter and 30 mm length

6.3.3 Where possible, core samples should be taken entirely within one block and be free from mortar joints.

Done

6.3.4 The external surface of core samples shall not be washed on site to improve their visual appearance, because this may compromise the analysis.

Done

6.3.5 Cut samples can be taken as an alternative under certain circumstances where difficulty in extracting core samples is experienced. This shall be recorded on the sample record and chain of custody form. It should be noted that when taking cut pieces by chisel, hammer blows may cause micro-cracking, which might not always be easy for the Petrographer to distinguish from cracking resulting from other causes.

6.3.6 As the supervising competent person is responsible for each extracted sample, it is necessary to ensure that these samples are collated accurately and packaged individually in sealable plastic bags. Labelling and numbering each sample, including the date, address and location, should be completed before leaving the site, and a signed and dated sample record and chain of custody form, (see Annex B), to indicate where the samples have been taken from, shall be included with the samples for dispatch to the laboratory.

6.3.7 Tools shall be cleaned following sampling of each concrete block, to avoid cross contamination of samples.

6.3.8 Samples shall be stored in sealable plastic bags, out of direct sunlight and in a cool environment. Waterproof marker pens should be used to clearly identify the sample number, date, address, and location.

6.4 Repair of the sampling hole

Repair of the core-holes shall be carefully undertaken, with particular care of the exposed cavity area. A fresh supply of quick-set dry mix (cement/fine aggregate) and tools for backfilling holes in walls created by sampling will be required. Existing insulation in the cavity should be repaired or replaced as required. Back spacers are required to avoid new fill debris bridging or falling into the cavity. Shrinkage of the repair material should also be avoided.

NOTE Shrinkage-compensated proprietary repair materials are available from several manufacturers.

6.5 Sample inspection and storage

Upon receipt of samples in the laboratory, a visual inspection shall be carried out by the Professional Geologist on each sample and this information shall be recorded.

I am a registered professional geologist and a concrete petrographer who has examined the samples

As a minimum, the visual inspection shall note:

- colour;
- grading/particle size;
- particle shape;
- thickness of render; and
- moisture condition.

Porous, surface dry, medium gray, aggregates are medium gray to brown

A dated photograph shall be taken of each sample and uniquely identified and documented. Samples shall be placed in suitable, airtight, uniquely identified containers, and stored in suitable conditions (see 6.3.8).

Provided

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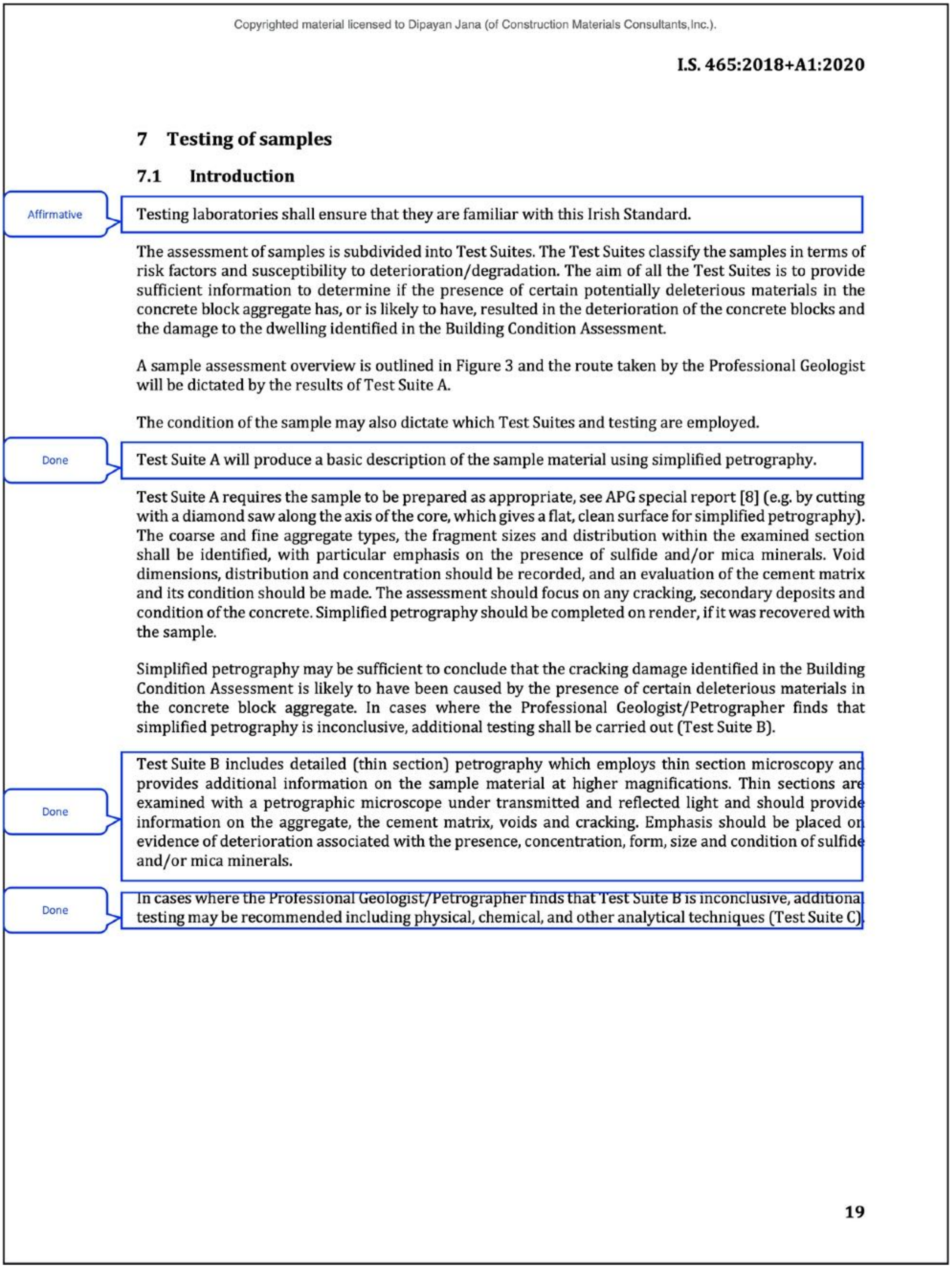


7 Testing of samples

7.1 Introduction

Affirmative

Testing laboratories shall ensure that they are familiar with this Irish Standard.

The assessment of samples is subdivided into Test Suites. The Test Suites classify the samples in terms of risk factors and susceptibility to deterioration/degradation. The aim of all the Test Suites is to provide sufficient information to determine if the presence of certain potentially deleterious materials in the concrete block aggregate has, or is likely to have, resulted in the deterioration of the concrete blocks and the damage to the dwelling identified in the Building Condition Assessment.

A sample assessment overview is outlined in Figure 3 and the route taken by the Professional Geologist will be dictated by the results of Test Suite A.

The condition of the sample may also dictate which Test Suites and testing are employed.

Done

Test Suite A will produce a basic description of the sample material using simplified petrography.

Test Suite A requires the sample to be prepared as appropriate, see APG special report [8] (e.g. by cutting with a diamond saw along the axis of the core, which gives a flat, clean surface for simplified petrography). The coarse and fine aggregate types, the fragment sizes and distribution within the examined section shall be identified, with particular emphasis on the presence of sulfide and/or mica minerals. Void dimensions, distribution and concentration should be recorded, and an evaluation of the cement matrix and its condition should be made. The assessment should focus on any cracking, secondary deposits and condition of the concrete. Simplified petrography should be completed on render, if it was recovered with the sample.

Simplified petrography may be sufficient to conclude that the cracking damage identified in the Building Condition Assessment is likely to have been caused by the presence of certain deleterious materials in the concrete block aggregate. In cases where the Professional Geologist/Petrographer finds that simplified petrography is inconclusive, additional testing shall be carried out (Test Suite B).

Done

Test Suite B includes detailed (thin section) petrography which employs thin section microscopy and provides additional information on the sample material at higher magnifications. Thin sections are examined with a petrographic microscope under transmitted and reflected light and should provide information on the aggregate, the cement matrix, voids and cracking. Emphasis should be placed on evidence of deterioration associated with the presence, concentration, form, size and condition of sulfide and/or mica minerals.

Done

In cases where the Professional Geologist/Petrographer finds that Test Suite B is inconclusive, additiona testing may be recommended including physical, chemical, and other analytical techniques (Test Suite C).

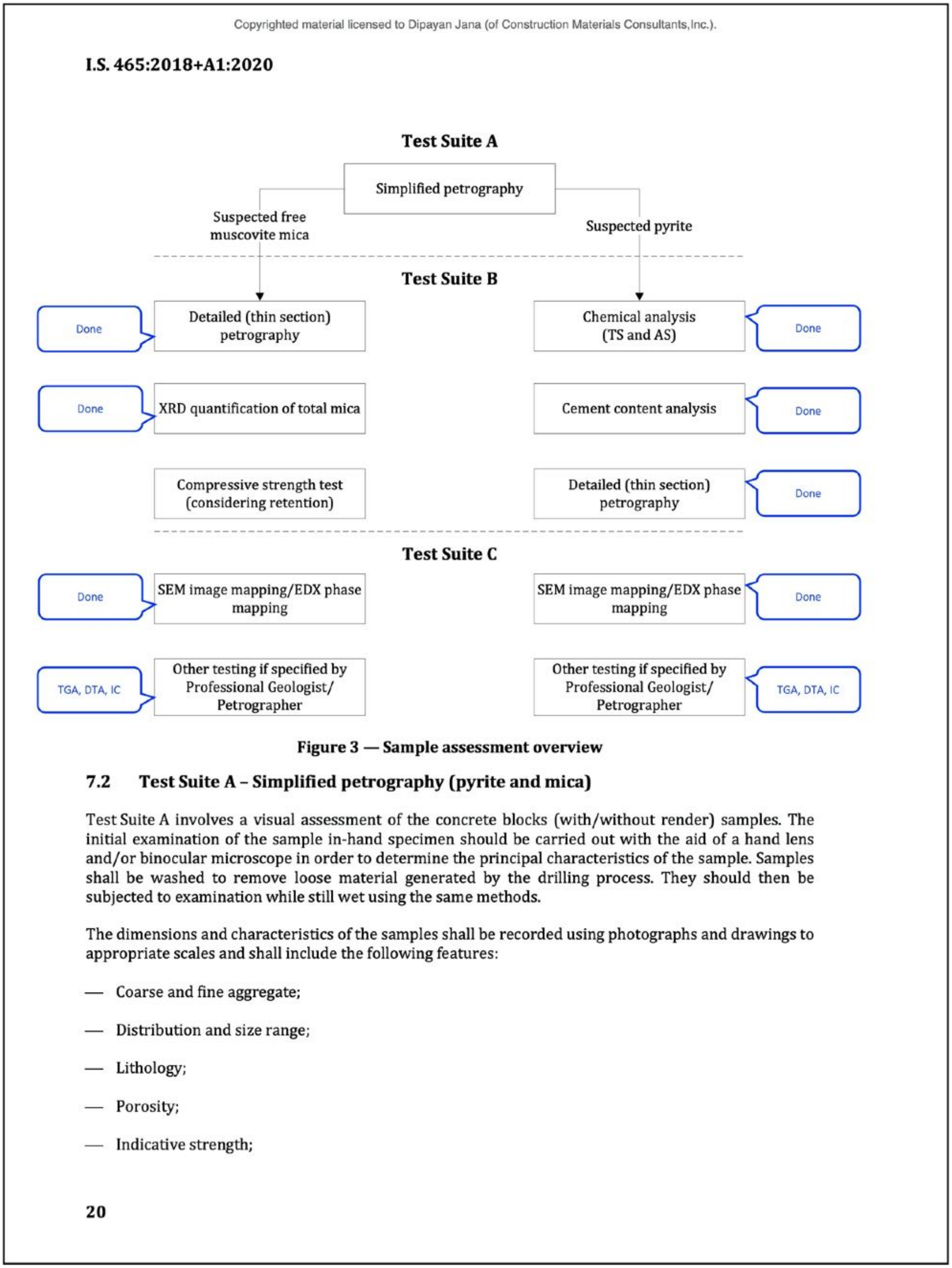


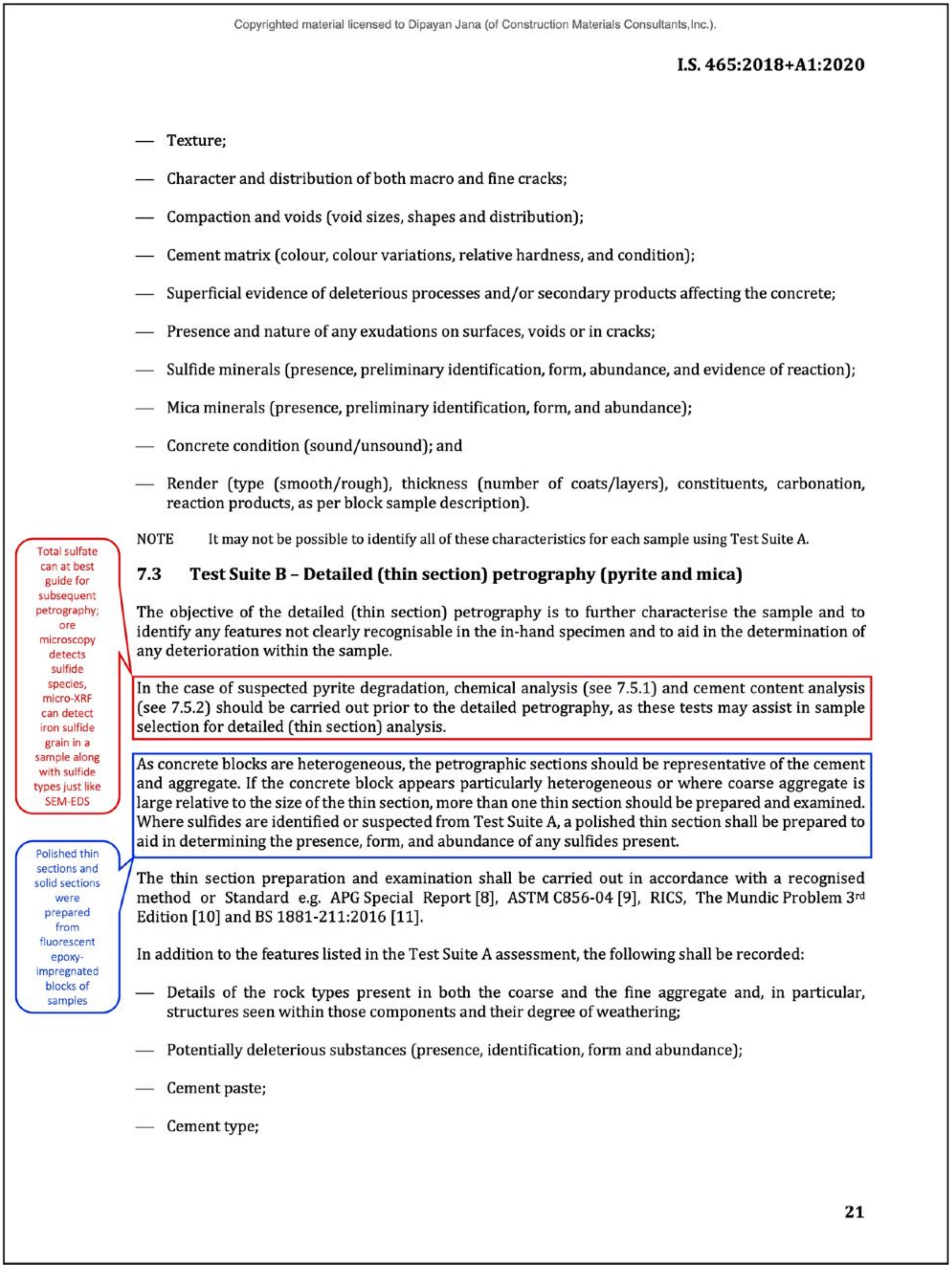
Figure 3 — Sample assessment overview

7.2 Test Suite A – Simplified petrography (pyrite and mica)

Test Suite A involves a visual assessment of the concrete blocks (with/without render) samples. The initial examination of the sample in-hand specimen should be carried out with the aid of a hand lens and/or binocular microscope in order to determine the principal characteristics of the sample. Samples shall be washed to remove loose material generated by the drilling process. They should then be subjected to examination while still wet using the same methods.

The dimensions and characteristics of the samples shall be recorded using photographs and drawings to appropriate scales and shall include the following features:

- Coarse and fine aggregate;
- Distribution and size range;
- Lithology;
- Porosity;
- Indicative strength;



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- Texture;
- Character and distribution of both macro and fine cracks;
- Compaction and voids (void sizes, shapes and distribution);
- Cement matrix (colour, colour variations, relative hardness, and condition);
- Superficial evidence of deleterious processes and/or secondary products affecting the concrete;
- Presence and nature of any exudations on surfaces, voids or in cracks;
- Sulfide minerals (presence, preliminary identification, form, abundance, and evidence of reaction);
- Mica minerals (presence, preliminary identification, form, and abundance);
- Concrete condition (sound/unsound); and
- Render (type (smooth/rough), thickness (number of coats/layers), constituents, carbonation, reaction products, as per block sample description).

NOTE It may not be possible to identify all of these characteristics for each sample using Test Suite A.

7.3 Test Suite B – Detailed (thin section) petrography (pyrite and mica)

The objective of the detailed (thin section) petrography is to further characterise the sample and to identify any features not clearly recognisable in the in-hand specimen and to aid in the determination of any deterioration within the sample.

In the case of suspected pyrite degradation, chemical analysis (see 7.5.1) and cement content analysis (see 7.5.2) should be carried out prior to the detailed petrography, as these tests may assist in sample selection for detailed (thin section) analysis.

Total sulfate can at best guide for subsequent petrography; ore microscopy detects sulfide species, micro-XRF can detect iron sulfide grain in a sample along with sulfide types just like SEM-EDS

As concrete blocks are heterogeneous, the petrographic sections should be representative of the cement and aggregate. If the concrete block appears particularly heterogeneous or where coarse aggregate is large relative to the size of the thin section, more than one thin section should be prepared and examined. Where sulfides are identified or suspected from Test Suite A, a polished thin section shall be prepared to aid in determining the presence, form, and abundance of any sulfides present.

Polished thin sections and solid sections were prepared from fluorescent epoxy-impregnated blocks of samples

The thin section preparation and examination shall be carried out in accordance with a recognised method or Standard e.g. APG Special Report [8], ASTM C856-04 [9], RICS, The Mundic Problem 3rd Edition [10] and BS 1881-211:2016 [11].

In addition to the features listed in the Test Suite A assessment, the following shall be recorded:

- Details of the rock types present in both the coarse and the fine aggregate and, in particular, structures seen within those components and their degree of weathering;
- Potentially deleterious substances (presence, identification, form and abundance);
- Cement paste;
- Cement type;

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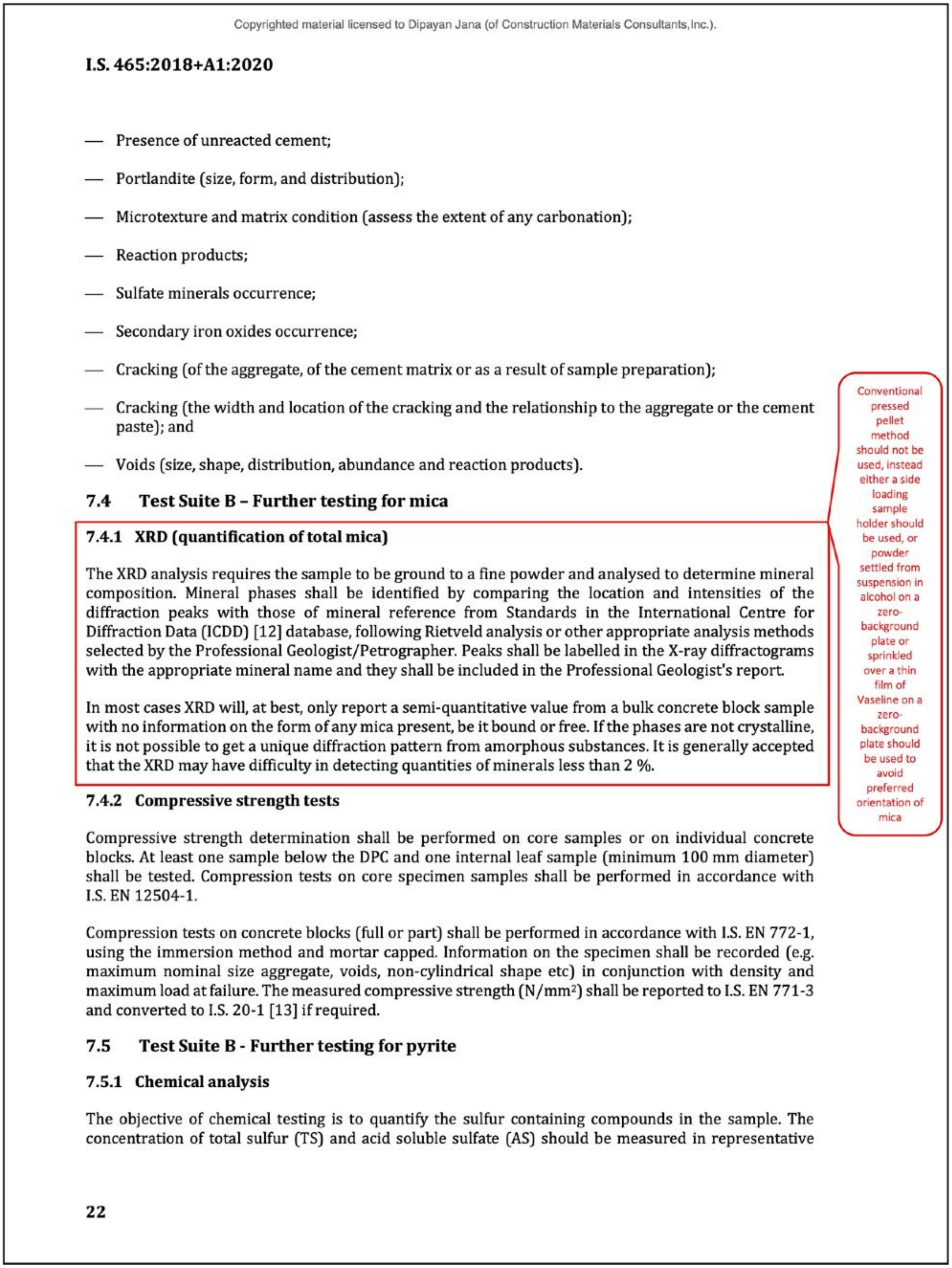


- Presence of unreacted cement;
- Portlandite (size, form, and distribution);
- Microtexture and matrix condition (assess the extent of any carbonation);
- Reaction products;
- Sulfate minerals occurrence;
- Secondary iron oxides occurrence;
- Cracking (of the aggregate, of the cement matrix or as a result of sample preparation);
- Cracking (the width and location of the cracking and the relationship to the aggregate or the cement paste); and
- Voids (size, shape, distribution, abundance and reaction products).

7.4 Test Suite B – Further testing for mica

7.4.1 XRD (quantification of total mica)

The XRD analysis requires the sample to be ground to a fine powder and analysed to determine mineral composition. Mineral phases shall be identified by comparing the location and intensities of the diffraction peaks with those of mineral reference from Standards in the International Centre for Diffraction Data (ICDD) [12] database, following Rietveld analysis or other appropriate analysis methods selected by the Professional Geologist/Petrographer. Peaks shall be labelled in the X-ray diffractograms with the appropriate mineral name and they shall be included in the Professional Geologist's report.

In most cases XRD will, at best, only report a semi-quantitative value from a bulk concrete block sample with no information on the form of any mica present, be it bound or free. If the phases are not crystalline, it is not possible to get a unique diffraction pattern from amorphous substances. It is generally accepted that the XRD may have difficulty in detecting quantities of minerals less than 2 %.

Conventional pressed pellet method should not be used, instead either a side loading sample holder should be used, or powder settled from suspension in alcohol on a zero-background plate or sprinkled over a thin film of Vaseline on a zero-background plate should be used to avoid preferred orientation of mica

7.4.2 Compressive strength tests

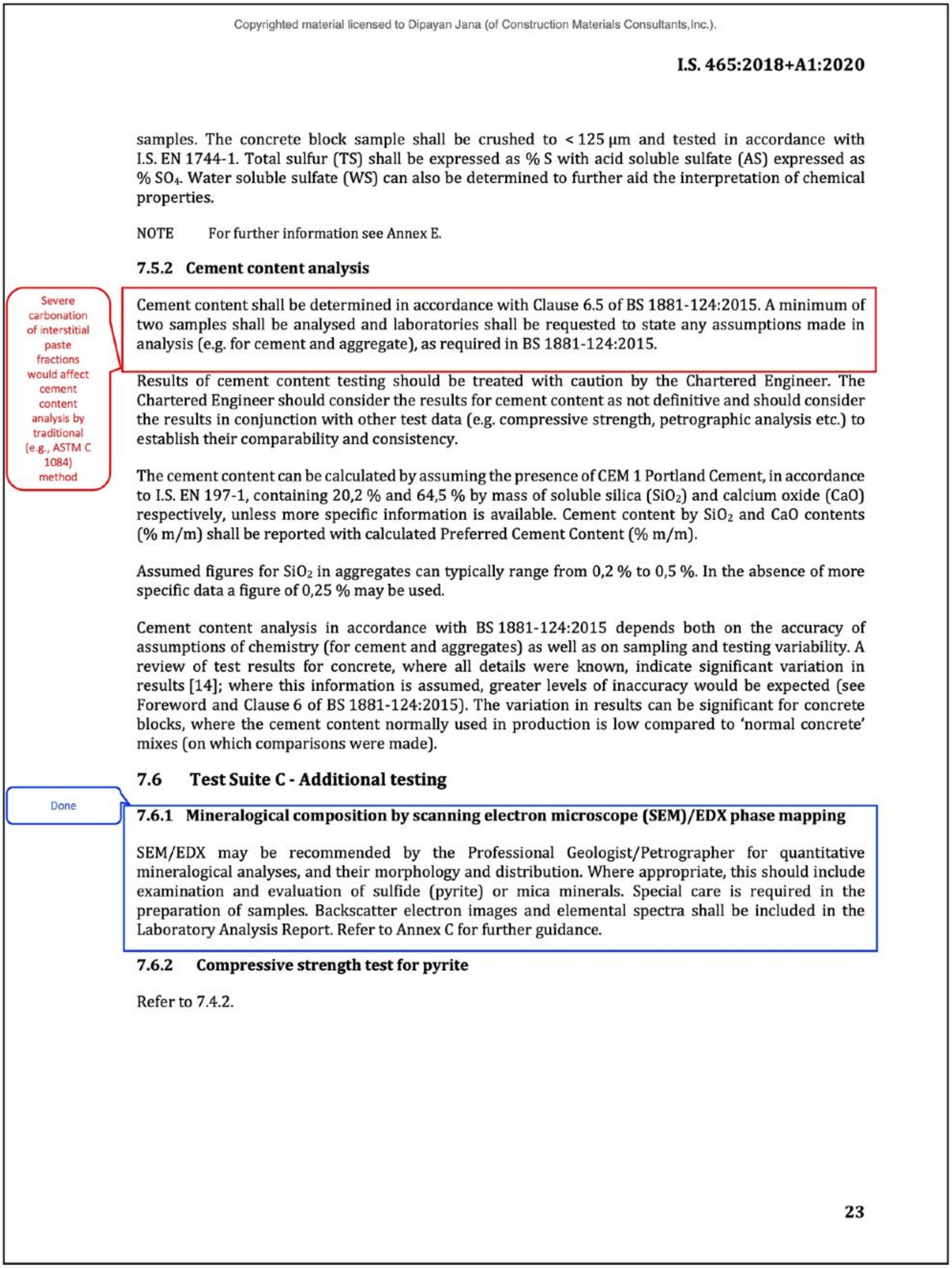
Compressive strength determination shall be performed on core samples or on individual concrete blocks. At least one sample below the DPC and one internal leaf sample (minimum 100 mm diameter) shall be tested. Compression tests on core specimen samples shall be performed in accordance with I.S. EN 12504-1.

Compression tests on concrete blocks (full or part) shall be performed in accordance with I.S. EN 772-1, using the immersion method and mortar capped. Information on the specimen shall be recorded (e.g. maximum nominal size aggregate, voids, non-cylindrical shape etc) in conjunction with density and maximum load at failure. The measured compressive strength (N/mm^2) shall be reported to I.S. EN 771-3 and converted to I.S. 20-1 [13] if required.

7.5 Test Suite B - Further testing for pyrite

7.5.1 Chemical analysis

The objective of chemical testing is to quantify the sulfur containing compounds in the sample. The concentration of total sulfur (TS) and acid soluble sulfate (AS) should be measured in representative



samples. The concrete block sample shall be crushed to < 125 µm and tested in accordance with I.S. EN 1744-1. Total sulfur (TS) shall be expressed as % S with acid soluble sulfate (AS) expressed as % SO_4. Water soluble sulfate (WS) can also be determined to further aid the interpretation of chemical properties.

NOTE For further information see Annex E.

7.5.2 Cement content analysis

Cement content shall be determined in accordance with Clause 6.5 of BS 1881-124:2015. A minimum of two samples shall be analysed and laboratories shall be requested to state any assumptions made in analysis (e.g. for cement and aggregate), as required in BS 1881-124:2015.

Severe carbonation of interstitial paste fractions would affect cement content analysis by traditional (e.g., ASTM C 1084) method

Results of cement content testing should be treated with caution by the Chartered Engineer. The Chartered Engineer should consider the results for cement content as not definitive and should consider the results in conjunction with other test data (e.g. compressive strength, petrographic analysis etc.) to establish their comparability and consistency.

The cement content can be calculated by assuming the presence of CEM 1 Portland Cement, in accordance to I.S. EN 197-1, containing 20,2 % and 64,5 % by mass of soluble silica (SiO_2) and calcium oxide (CaO) respectively, unless more specific information is available. Cement content by SiO_2 and CaO contents (% m/m) shall be reported with calculated Preferred Cement Content (% m/m).

Assumed figures for SiO_2 in aggregates can typically range from 0,2 % to 0,5 %. In the absence of more specific data a figure of 0,25 % may be used.

Cement content analysis in accordance with BS 1881-124:2015 depends both on the accuracy of assumptions of chemistry (for cement and aggregates) as well as on sampling and testing variability. A review of test results for concrete, where all details were known, indicate significant variation in results [14]; where this information is assumed, greater levels of inaccuracy would be expected (see Foreword and Clause 6 of BS 1881-124:2015). The variation in results can be significant for concrete blocks, where the cement content normally used in production is low compared to 'normal concrete' mixes (on which comparisons were made).

7.6 Test Suite C - Additional testing

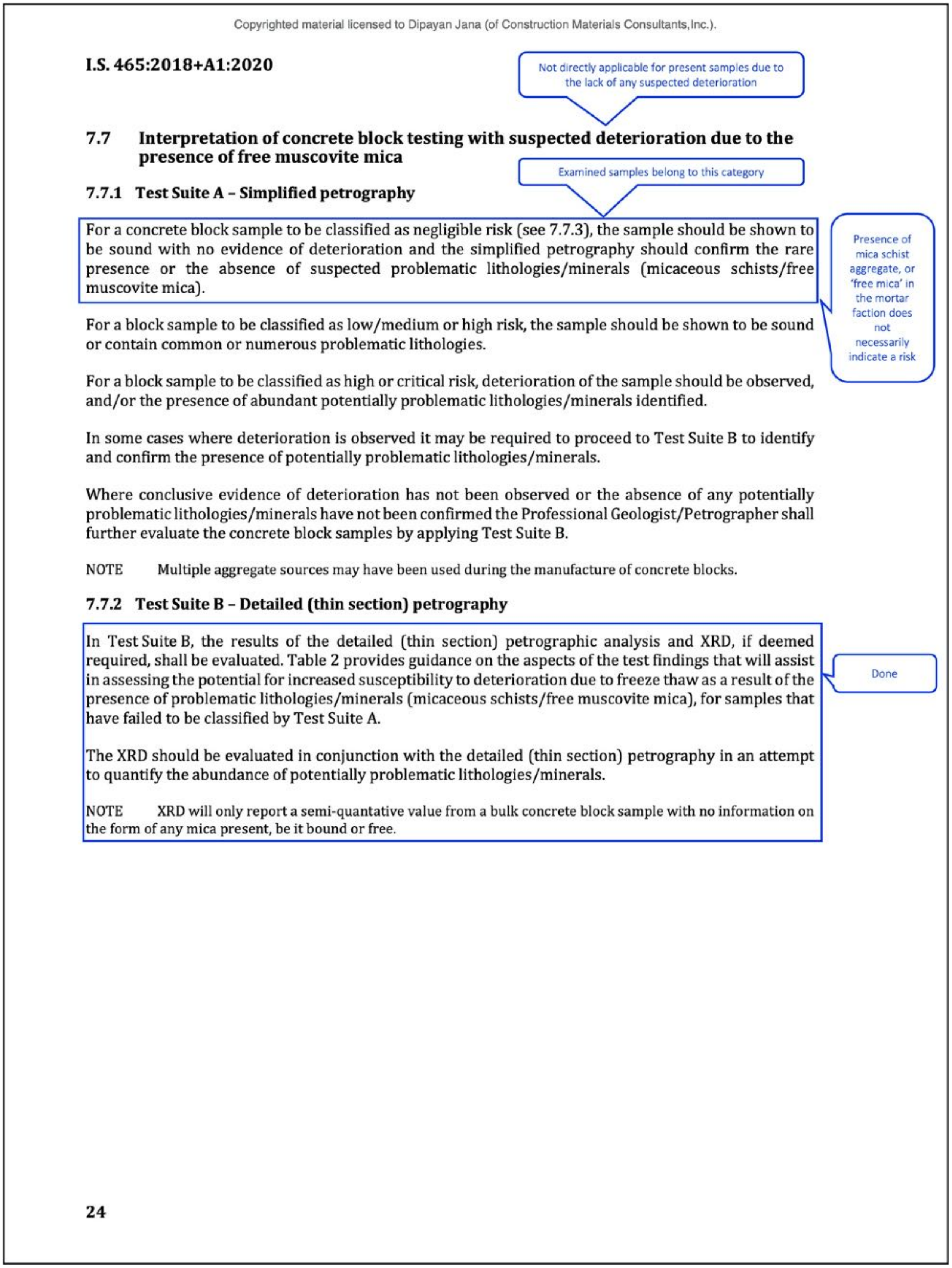
Done

7.6.1 Mineralogical composition by scanning electron microscope (SEM)/EDX phase mapping

SEM/EDX may be recommended by the Professional Geologist/Petrographer for quantitative mineralogical analyses, and their morphology and distribution. Where appropriate, this should include examination and evaluation of sulfide (pyrite) or mica minerals. Special care is required in the preparation of samples. Backscatter electron images and elemental spectra shall be included in the Laboratory Analysis Report. Refer to Annex C for further guidance.

7.6.2 Compressive strength test for pyrite

Refer to 7.4.2.



Not directly applicable for present samples due to the lack of any suspected deterioration

7.7 Interpretation of concrete block testing with suspected deterioration due to the presence of free muscovite mica

Examined samples belong to this category

7.7.1 Test Suite A – Simplified petrography

For a concrete block sample to be classified as negligible risk (see 7.7.3), the sample should be shown to be sound with no evidence of deterioration and the simplified petrography should confirm the rare presence or the absence of suspected problematic lithologies/minerals (micaceous schists/free muscovite mica).

Presence of mica schist aggregate, or 'free mica' in the mortar faction does not necessarily indicate a risk

For a block sample to be classified as low/medium or high risk, the sample should be shown to be sound or contain common or numerous problematic lithologies.

For a block sample to be classified as high or critical risk, deterioration of the sample should be observed, and/or the presence of abundant potentially problematic lithologies/minerals identified.

In some cases where deterioration is observed it may be required to proceed to Test Suite B to identify and confirm the presence of potentially problematic lithologies/minerals.

Where conclusive evidence of deterioration has not been observed or the absence of any potentially problematic lithologies/minerals have not been confirmed the Professional Geologist/Petrographer shall further evaluate the concrete block samples by applying Test Suite B.

NOTE Multiple aggregate sources may have been used during the manufacture of concrete blocks.

7.7.2 Test Suite B – Detailed (thin section) petrography

In Test Suite B, the results of the detailed (thin section) petrographic analysis and XRD, if deemed required, shall be evaluated. Table 2 provides guidance on the aspects of the test findings that will assist in assessing the potential for increased susceptibility to deterioration due to freeze thaw as a result of the presence of problematic lithologies/minerals (micaceous schists/free muscovite mica), for samples that have failed to be classified by Test Suite A.

Done

The XRD should be evaluated in conjunction with the detailed (thin section) petrography in an attempt to quantify the abundance of potentially problematic lithologies/minerals.

NOTE XRD will only report a semi-quantative value from a bulk concrete block sample with no information on the form of any mica present, be it bound or free.

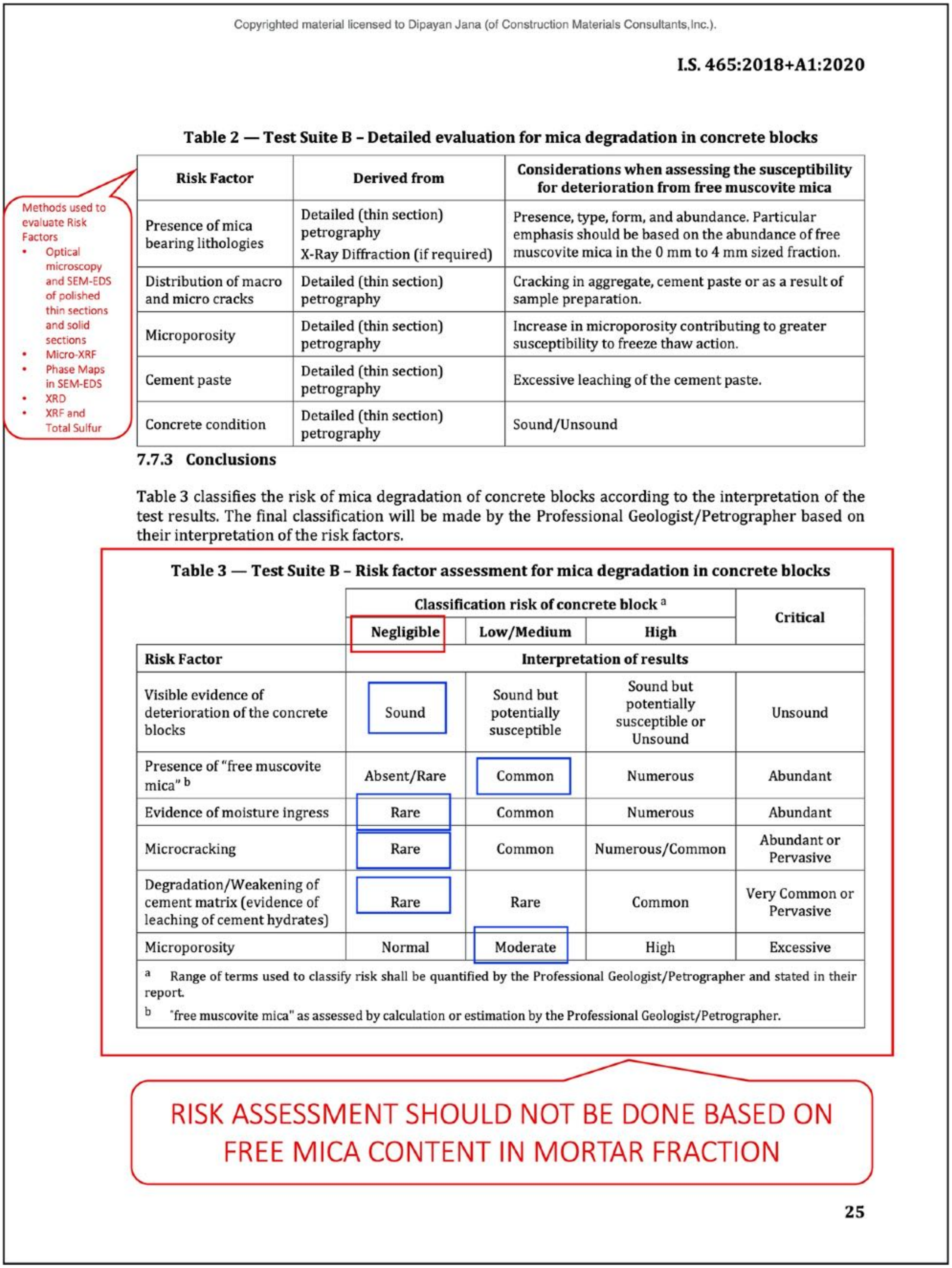


Table 2 — Test Suite B – Detailed evaluation for mica degradation in concrete blocks

Risk Factor	Derived from	Considerations when assessing the susceptibility for deterioration from free muscovite mica
Presence of mica bearing lithologies	Detailed (thin section) petrography X-Ray Diffraction (if required)	Presence, type, form, and abundance. Particular emphasis should be based on the abundance of free muscovite mica in the 0 mm to 4 mm sized fraction.
Distribution of macro and micro cracks	Detailed (thin section) petrography	Cracking in aggregate, cement paste or as a result of sample preparation.
Microporosity	Detailed (thin section) petrography	Increase in microporosity contributing to greater susceptibility to freeze thaw action.
Cement paste	Detailed (thin section) petrography	Excessive leaching of the cement paste.
Concrete condition	Detailed (thin section) petrography	Sound/Unsound

Methods used to evaluate Risk Factors
- Optical microscopy and SEM-EDS of polished thin sections and solid sections
- Micro-XRF
- Phase Maps in SEM-EDS
- XRD
- XRF and Total Sulfur

7.7.3 Conclusions

Table 3 classifies the risk of mica degradation of concrete blocks according to the interpretation of the test results. The final classification will be made by the Professional Geologist/Petrographer based on their interpretation of the risk factors.

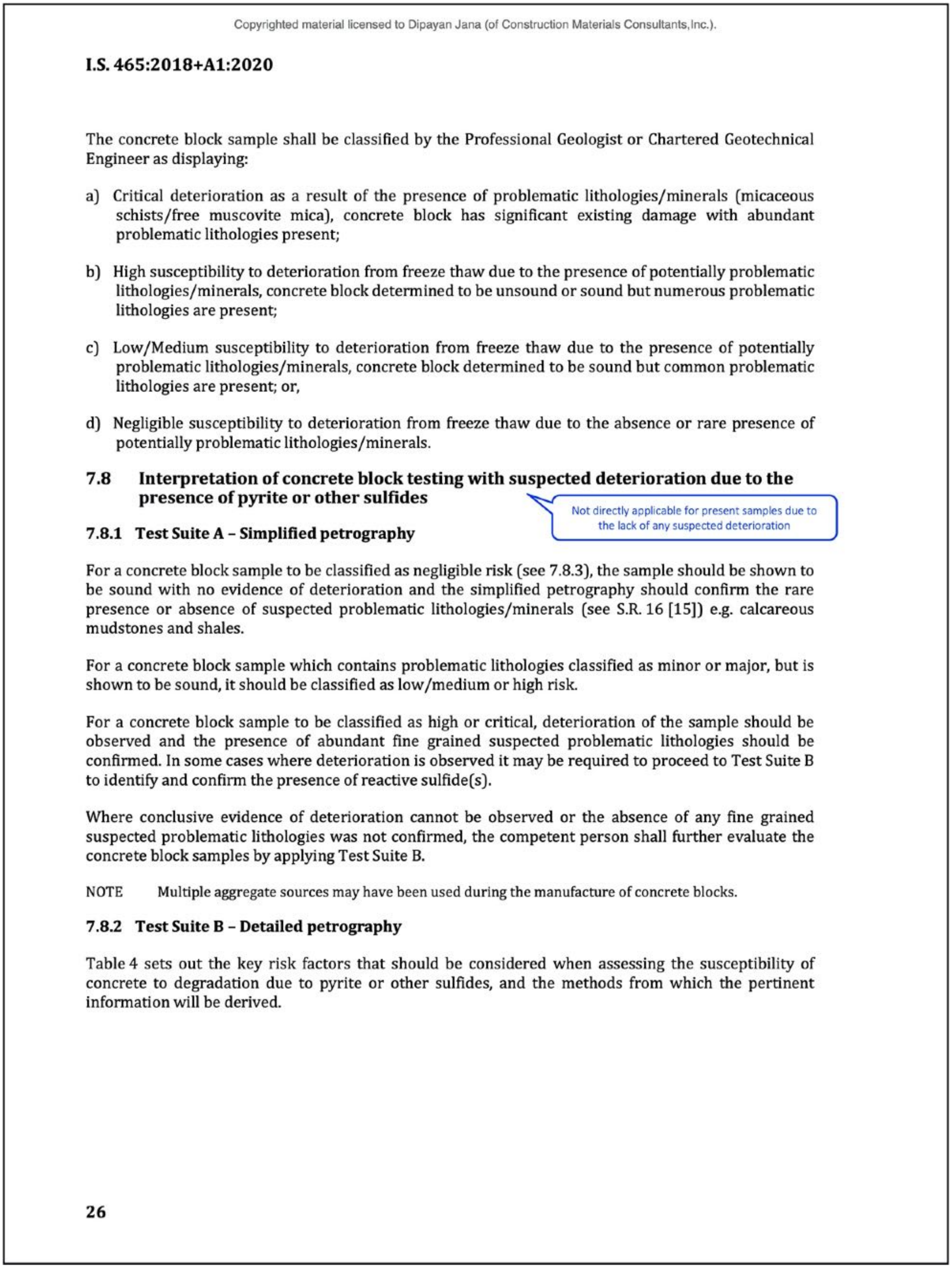
Table 3 — Test Suite B – Risk factor assessment for mica degradation in concrete blocks

	Classification risk of concrete block [a]			Critical
	Negligible	Low/Medium	High	
Risk Factor	**Interpretation of results**			
Visible evidence of deterioration of the concrete blocks	Sound	Sound but potentially susceptible	Sound but potentially susceptible or Unsound	Unsound
Presence of "free muscovite mica" [b]	Absent/Rare	Common	Numerous	Abundant
Evidence of moisture ingress	Rare	Common	Numerous	Abundant
Microcracking	Rare	Common	Numerous/Common	Abundant or Pervasive
Degradation/Weakening of cement matrix (evidence of leaching of cement hydrates)	Rare	Rare	Common	Very Common or Pervasive
Microporosity	Normal	Moderate	High	Excessive

[a] Range of terms used to classify risk shall be quantified by the Professional Geologist/Petrographer and stated in their report.

[b] "free muscovite mica" as assessed by calculation or estimation by the Professional Geologist/Petrographer.

RISK ASSESSMENT SHOULD NOT BE DONE BASED ON FREE MICA CONTENT IN MORTAR FRACTION



The concrete block sample shall be classified by the Professional Geologist or Chartered Geotechnical Engineer as displaying:

a) Critical deterioration as a result of the presence of problematic lithologies/minerals (micaceous schists/free muscovite mica), concrete block has significant existing damage with abundant problematic lithologies present;

b) High susceptibility to deterioration from freeze thaw due to the presence of potentially problematic lithologies/minerals, concrete block determined to be unsound or sound but numerous problematic lithologies are present;

c) Low/Medium susceptibility to deterioration from freeze thaw due to the presence of potentially problematic lithologies/minerals, concrete block determined to be sound but common problematic lithologies are present; or,

d) Negligible susceptibility to deterioration from freeze thaw due to the absence or rare presence of potentially problematic lithologies/minerals.

7.8 Interpretation of concrete block testing with suspected deterioration due to the presence of pyrite or other sulfides

Not directly applicable for present samples due to the lack of any suspected deterioration

7.8.1 Test Suite A – Simplified petrography

For a concrete block sample to be classified as negligible risk (see 7.8.3), the sample should be shown to be sound with no evidence of deterioration and the simplified petrography should confirm the rare presence or absence of suspected problematic lithologies/minerals (see S.R. 16 [15]) e.g. calcareous mudstones and shales.

For a concrete block sample which contains problematic lithologies classified as minor or major, but is shown to be sound, it should be classified as low/medium or high risk.

For a concrete block sample to be classified as high or critical, deterioration of the sample should be observed and the presence of abundant fine grained suspected problematic lithologies should be confirmed. In some cases where deterioration is observed it may be required to proceed to Test Suite B to identify and confirm the presence of reactive sulfide(s).

Where conclusive evidence of deterioration cannot be observed or the absence of any fine grained suspected problematic lithologies was not confirmed, the competent person shall further evaluate the concrete block samples by applying Test Suite B.

NOTE Multiple aggregate sources may have been used during the manufacture of concrete blocks.

7.8.2 Test Suite B – Detailed petrography

Table 4 sets out the key risk factors that should be considered when assessing the susceptibility of concrete to degradation due to pyrite or other sulfides, and the methods from which the pertinent information will be derived.

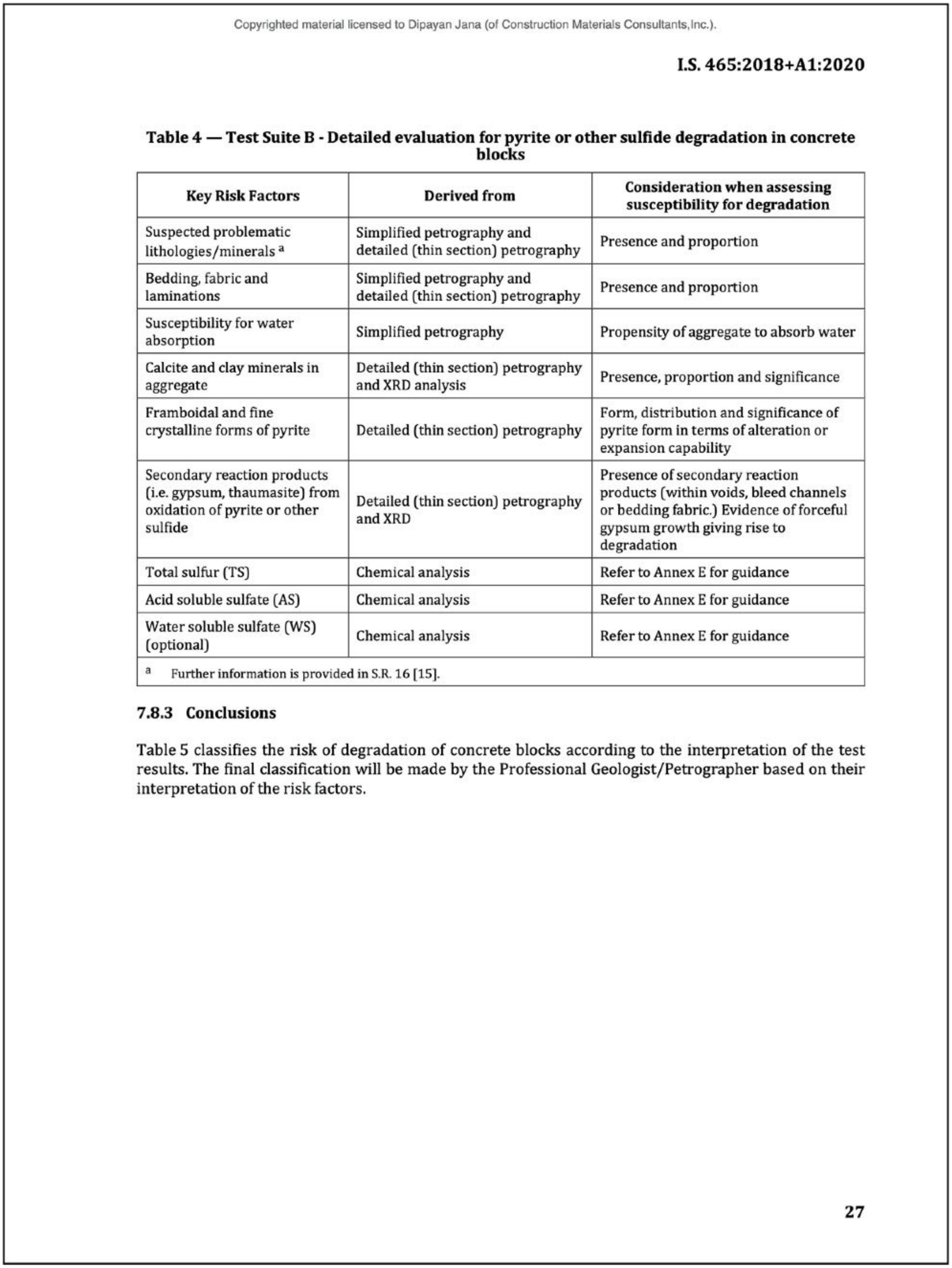


Table 4 — Test Suite B - Detailed evaluation for pyrite or other sulfide degradation in concrete blocks

Key Risk Factors	Derived from	Consideration when assessing susceptibility for degradation
Suspected problematic lithologies/minerals [a]	Simplified petrography and detailed (thin section) petrography	Presence and proportion
Bedding, fabric and laminations	Simplified petrography and detailed (thin section) petrography	Presence and proportion
Susceptibility for water absorption	Simplified petrography	Propensity of aggregate to absorb water
Calcite and clay minerals in aggregate	Detailed (thin section) petrography and XRD analysis	Presence, proportion and significance
Framboidal and fine crystalline forms of pyrite	Detailed (thin section) petrography	Form, distribution and significance of pyrite form in terms of alteration or expansion capability
Secondary reaction products (i.e. gypsum, thaumasite) from oxidation of pyrite or other sulfide	Detailed (thin section) petrography and XRD	Presence of secondary reaction products (within voids, bleed channels or bedding fabric.) Evidence of forceful gypsum growth giving rise to degradation
Total sulfur (TS)	Chemical analysis	Refer to Annex E for guidance
Acid soluble sulfate (AS)	Chemical analysis	Refer to Annex E for guidance
Water soluble sulfate (WS) (optional)	Chemical analysis	Refer to Annex E for guidance

[a] Further information is provided in S.R. 16 [15].

7.8.3 Conclusions

Table 5 classifies the risk of degradation of concrete blocks according to the interpretation of the test results. The final classification will be made by the Professional Geologist/Petrographer based on their interpretation of the risk factors.

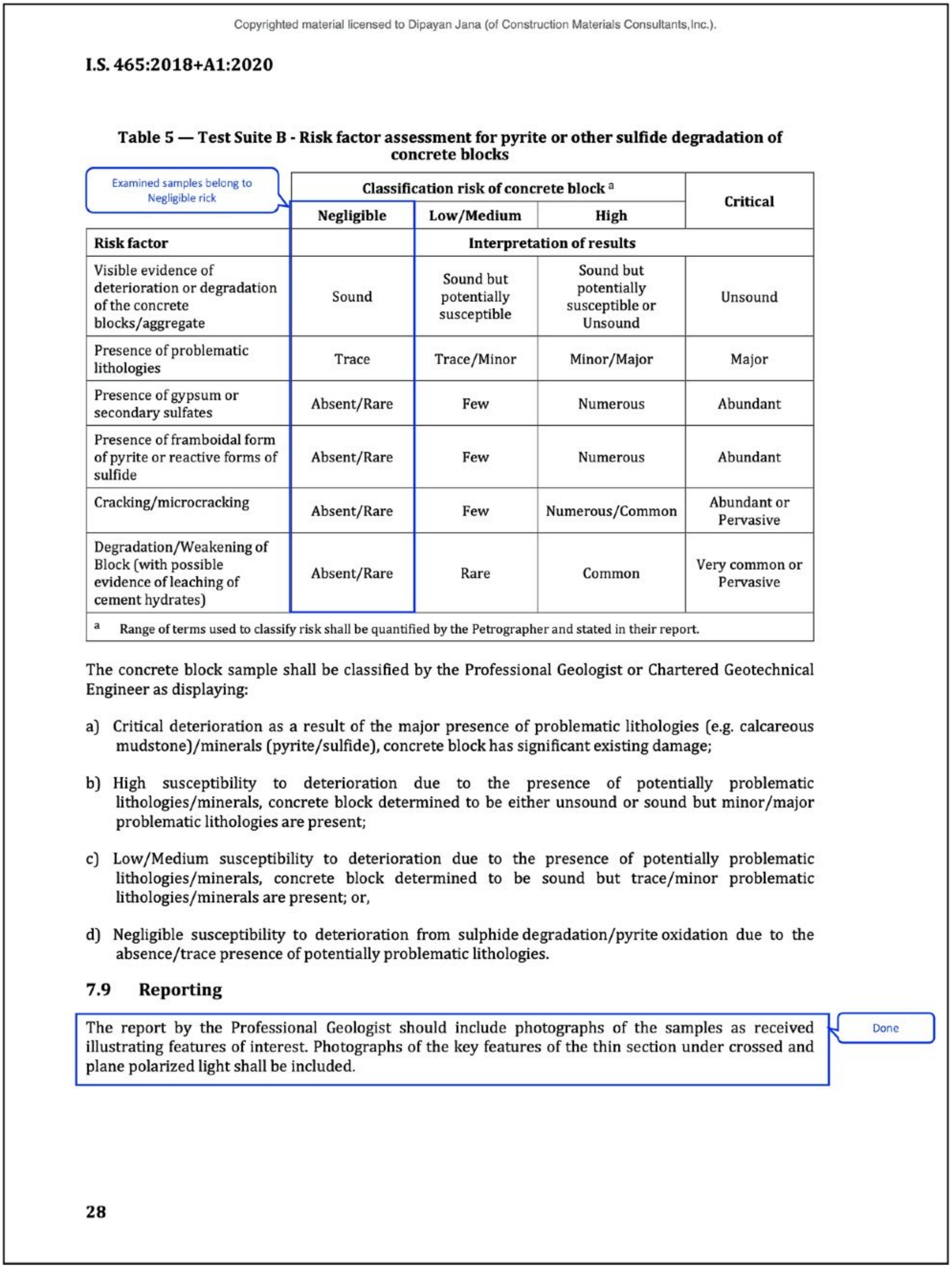


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Table 5 — Test Suite B - Risk factor assessment for pyrite or other sulfide degradation of concrete blocks

Examined samples belong to Negligible rick

	Classification risk of concrete block [a]			**Critical**
	Negligible	**Low/Medium**	**High**	
Risk factor		**Interpretation of results**		
Visible evidence of deterioration or degradation of the concrete blocks/aggregate	Sound	Sound but potentially susceptible	Sound but potentially susceptible or Unsound	Unsound
Presence of problematic lithologies	Trace	Trace/Minor	Minor/Major	Major
Presence of gypsum or secondary sulfates	Absent/Rare	Few	Numerous	Abundant
Presence of framboidal form of pyrite or reactive forms of sulfide	Absent/Rare	Few	Numerous	Abundant
Cracking/microcracking	Absent/Rare	Few	Numerous/Common	Abundant or Pervasive
Degradation/Weakening of Block (with possible evidence of leaching of cement hydrates)	Absent/Rare	Rare	Common	Very common or Pervasive

[a] Range of terms used to classify risk shall be quantified by the Petrographer and stated in their report.

The concrete block sample shall be classified by the Professional Geologist or Chartered Geotechnical Engineer as displaying:

a) Critical deterioration as a result of the major presence of problematic lithologies (e.g. calcareous mudstone)/minerals (pyrite/sulfide), concrete block has significant existing damage;

b) High susceptibility to deterioration due to the presence of potentially problematic lithologies/minerals, concrete block determined to be either unsound or sound but minor/major problematic lithologies are present;

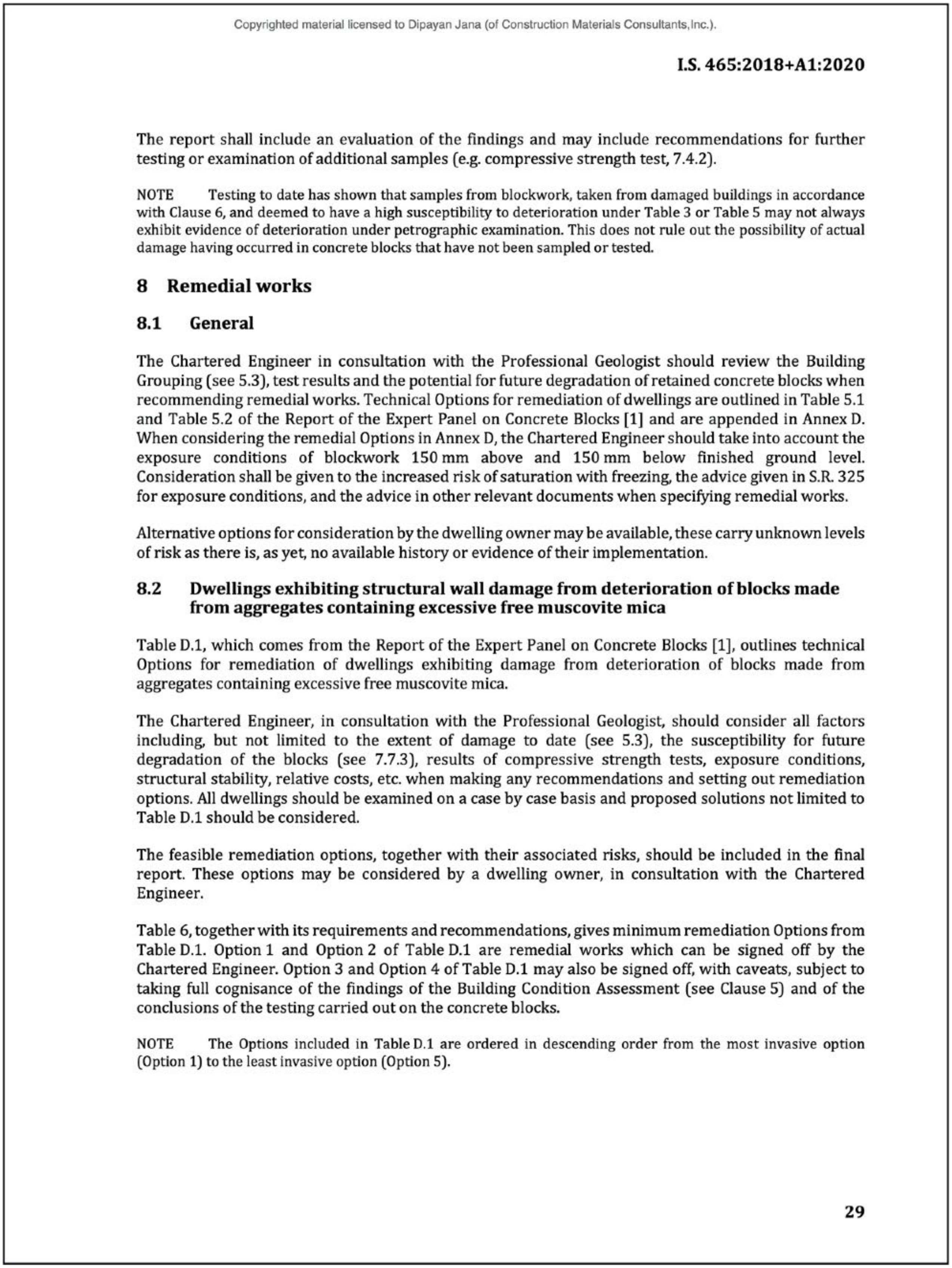
c) Low/Medium susceptibility to deterioration due to the presence of potentially problematic lithologies/minerals, concrete block determined to be sound but trace/minor problematic lithologies/minerals are present; or,

d) Negligible susceptibility to deterioration from sulphide degradation/pyrite oxidation due to the absence/trace presence of potentially problematic lithologies.

7.9 Reporting

The report by the Professional Geologist should include photographs of the samples as received illustrating features of interest. Photographs of the key features of the thin section under crossed and plane polarized light shall be included.

Done



The report shall include an evaluation of the findings and may include recommendations for further testing or examination of additional samples (e.g. compressive strength test, 7.4.2).

NOTE Testing to date has shown that samples from blockwork, taken from damaged buildings in accordance with Clause 6, and deemed to have a high susceptibility to deterioration under Table 3 or Table 5 may not always exhibit evidence of deterioration under petrographic examination. This does not rule out the possibility of actual damage having occurred in concrete blocks that have not been sampled or tested.

8 Remedial works

8.1 General

The Chartered Engineer in consultation with the Professional Geologist should review the Building Grouping (see 5.3), test results and the potential for future degradation of retained concrete blocks when recommending remedial works. Technical Options for remediation of dwellings are outlined in Table 5.1 and Table 5.2 of the Report of the Expert Panel on Concrete Blocks [1] and are appended in Annex D. When considering the remedial Options in Annex D, the Chartered Engineer should take into account the exposure conditions of blockwork 150 mm above and 150 mm below finished ground level. Consideration shall be given to the increased risk of saturation with freezing, the advice given in S.R. 325 for exposure conditions, and the advice in other relevant documents when specifying remedial works.

Alternative options for consideration by the dwelling owner may be available, these carry unknown levels of risk as there is, as yet, no available history or evidence of their implementation.

8.2 Dwellings exhibiting structural wall damage from deterioration of blocks made from aggregates containing excessive free muscovite mica

Table D.1, which comes from the Report of the Expert Panel on Concrete Blocks [1], outlines technical Options for remediation of dwellings exhibiting damage from deterioration of blocks made from aggregates containing excessive free muscovite mica.

The Chartered Engineer, in consultation with the Professional Geologist, should consider all factors including, but not limited to the extent of damage to date (see 5.3), the susceptibility for future degradation of the blocks (see 7.7.3), results of compressive strength tests, exposure conditions, structural stability, relative costs, etc. when making any recommendations and setting out remediation options. All dwellings should be examined on a case by case basis and proposed solutions not limited to Table D.1 should be considered.

The feasible remediation options, together with their associated risks, should be included in the final report. These options may be considered by a dwelling owner, in consultation with the Chartered Engineer.

Table 6, together with its requirements and recommendations, gives minimum remediation Options from Table D.1. Option 1 and Option 2 of Table D.1 are remedial works which can be signed off by the Chartered Engineer. Option 3 and Option 4 of Table D.1 may also be signed off, with caveats, subject to taking full cognisance of the findings of the Building Condition Assessment (see Clause 5) and of the conclusions of the testing carried out on the concrete blocks.

NOTE The Options included in Table D.1 are ordered in descending order from the most invasive option (Option 1) to the least invasive option (Option 5).

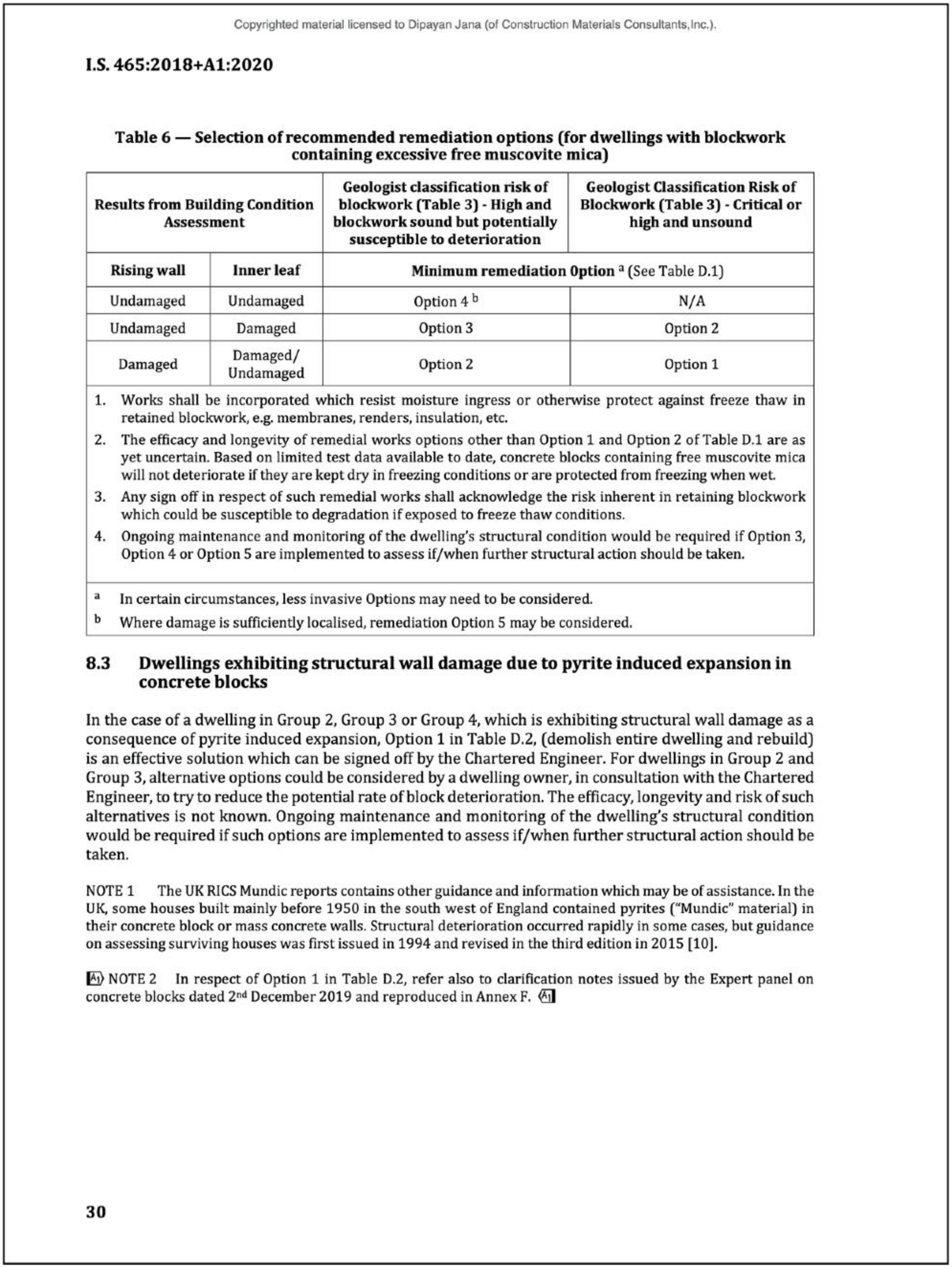


Table 6 — Selection of recommended remediation options (for dwellings with blockwork containing excessive free muscovite mica)

Results from Building Condition Assessment		**Geologist classification risk of blockwork (Table 3) - High and blockwork sound but potentially susceptible to deterioration**	**Geologist Classification Risk of Blockwork (Table 3) - Critical or high and unsound**
Rising wall	**Inner leaf**	**Minimum remediation Option** [a] (See Table D.1)	
Undamaged	Undamaged	Option 4 [b]	N/A
Undamaged	Damaged	Option 3	Option 2
Damaged	Damaged/ Undamaged	Option 2	Option 1

1. Works shall be incorporated which resist moisture ingress or otherwise protect against freeze thaw in retained blockwork, e.g. membranes, renders, insulation, etc.
2. The efficacy and longevity of remedial works options other than Option 1 and Option 2 of Table D.1 are as yet uncertain. Based on limited test data available to date, concrete blocks containing free muscovite mica will not deteriorate if they are kept dry in freezing conditions or are protected from freezing when wet.
3. Any sign off in respect of such remedial works shall acknowledge the risk inherent in retaining blockwork which could be susceptible to degradation if exposed to freeze thaw conditions.
4. Ongoing maintenance and monitoring of the dwelling's structural condition would be required if Option 3, Option 4 or Option 5 are implemented to assess if/when further structural action should be taken.

[a] In certain circumstances, less invasive Options may need to be considered.

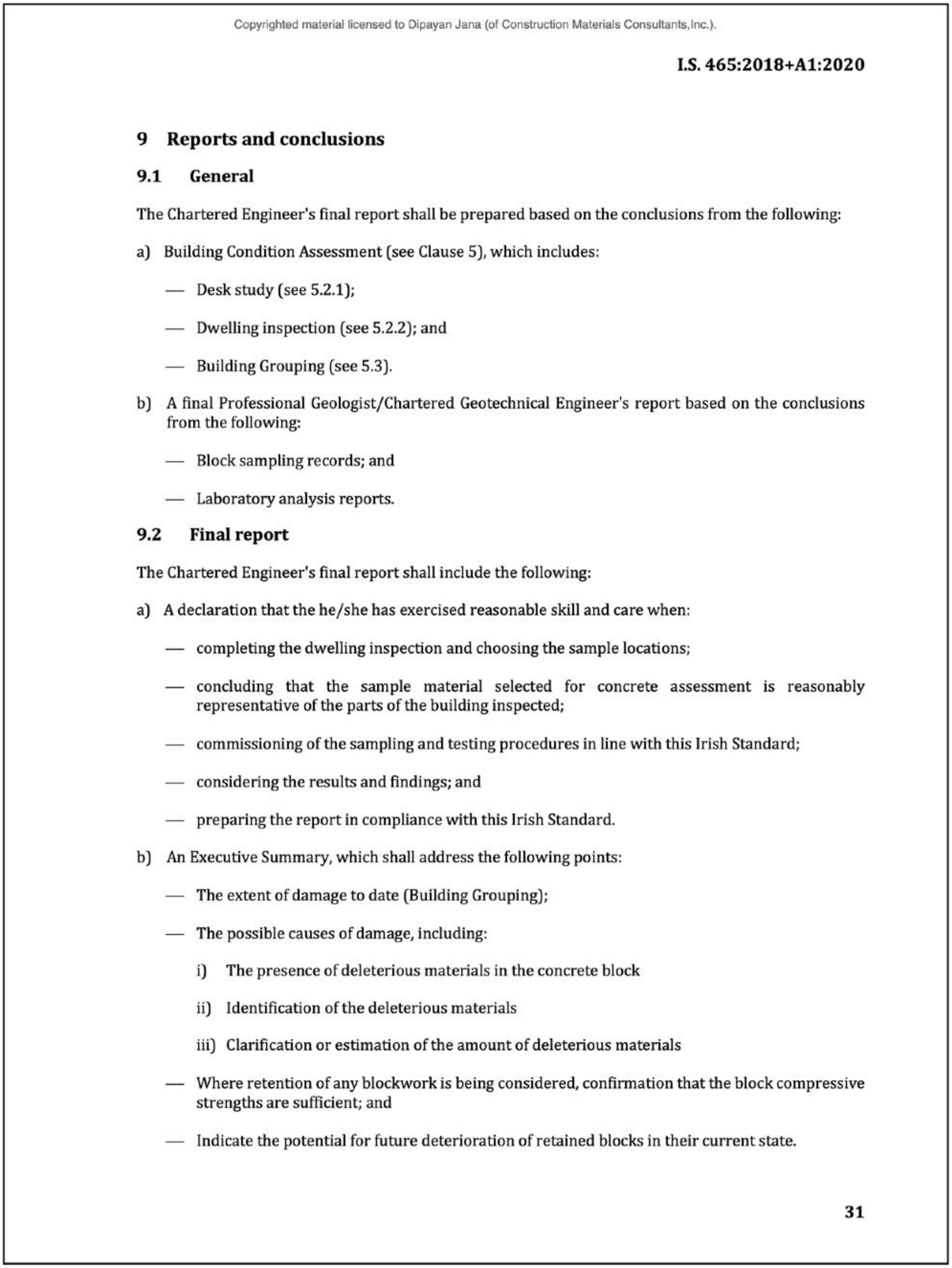
[b] Where damage is sufficiently localised, remediation Option 5 may be considered.

8.3 Dwellings exhibiting structural wall damage due to pyrite induced expansion in concrete blocks

In the case of a dwelling in Group 2, Group 3 or Group 4, which is exhibiting structural wall damage as a consequence of pyrite induced expansion, Option 1 in Table D.2, (demolish entire dwelling and rebuild) is an effective solution which can be signed off by the Chartered Engineer. For dwellings in Group 2 and Group 3, alternative options could be considered by a dwelling owner, in consultation with the Chartered Engineer, to try to reduce the potential rate of block deterioration. The efficacy, longevity and risk of such alternatives is not known. Ongoing maintenance and monitoring of the dwelling's structural condition would be required if such options are implemented to assess if/when further structural action should be taken.

NOTE 1 The UK RICS Mundic reports contains other guidance and information which may be of assistance. In the UK, some houses built mainly before 1950 in the south west of England contained pyrites ("Mundic" material) in their concrete block or mass concrete walls. Structural deterioration occurred rapidly in some cases, but guidance on assessing surviving houses was first issued in 1994 and revised in the third edition in 2015 [10].

A1 NOTE 2 In respect of Option 1 in Table D.2, refer also to clarification notes issued by the Expert panel on concrete blocks dated 2nd December 2019 and reproduced in Annex F. A1



9 Reports and conclusions

9.1 General

The Chartered Engineer's final report shall be prepared based on the conclusions from the following:

a) Building Condition Assessment (see Clause 5), which includes:

- Desk study (see 5.2.1);
- Dwelling inspection (see 5.2.2); and
- Building Grouping (see 5.3).

b) A final Professional Geologist/Chartered Geotechnical Engineer's report based on the conclusions from the following:

- Block sampling records; and
- Laboratory analysis reports.

9.2 Final report

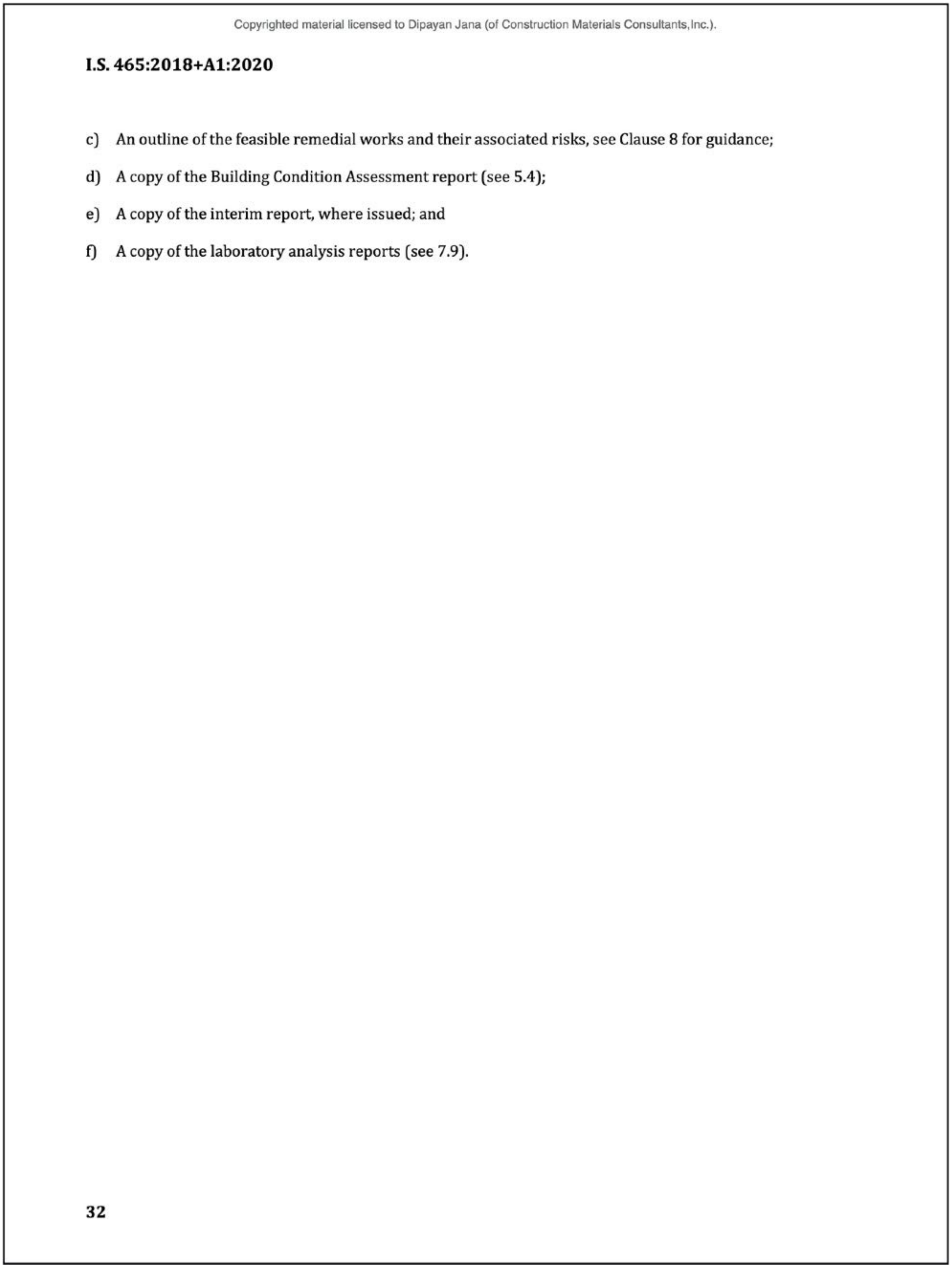
The Chartered Engineer's final report shall include the following:

a) A declaration that the he/she has exercised reasonable skill and care when:

- completing the dwelling inspection and choosing the sample locations;
- concluding that the sample material selected for concrete assessment is reasonably representative of the parts of the building inspected;
- commissioning of the sampling and testing procedures in line with this Irish Standard;
- considering the results and findings; and
- preparing the report in compliance with this Irish Standard.

b) An Executive Summary, which shall address the following points:

- The extent of damage to date (Building Grouping);
- The possible causes of damage, including:
    i) The presence of deleterious materials in the concrete block
    ii) Identification of the deleterious materials
    iii) Clarification or estimation of the amount of deleterious materials
- Where retention of any blockwork is being considered, confirmation that the block compressive strengths are sufficient; and
- Indicate the potential for future deterioration of retained blocks in their current state.

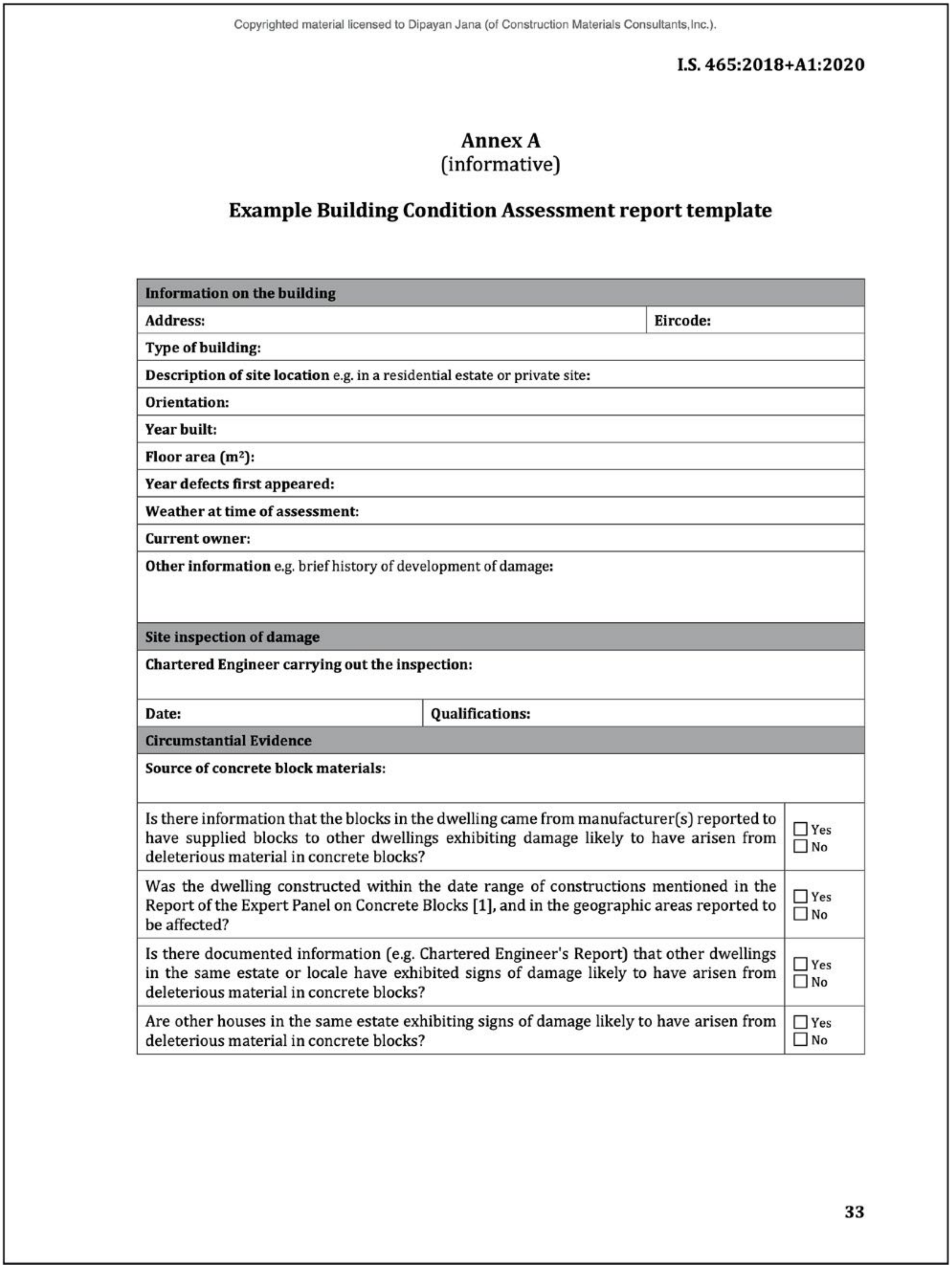


c) An outline of the feasible remedial works and their associated risks, see Clause 8 for guidance;

d) A copy of the Building Condition Assessment report (see 5.4);

e) A copy of the interim report, where issued; and

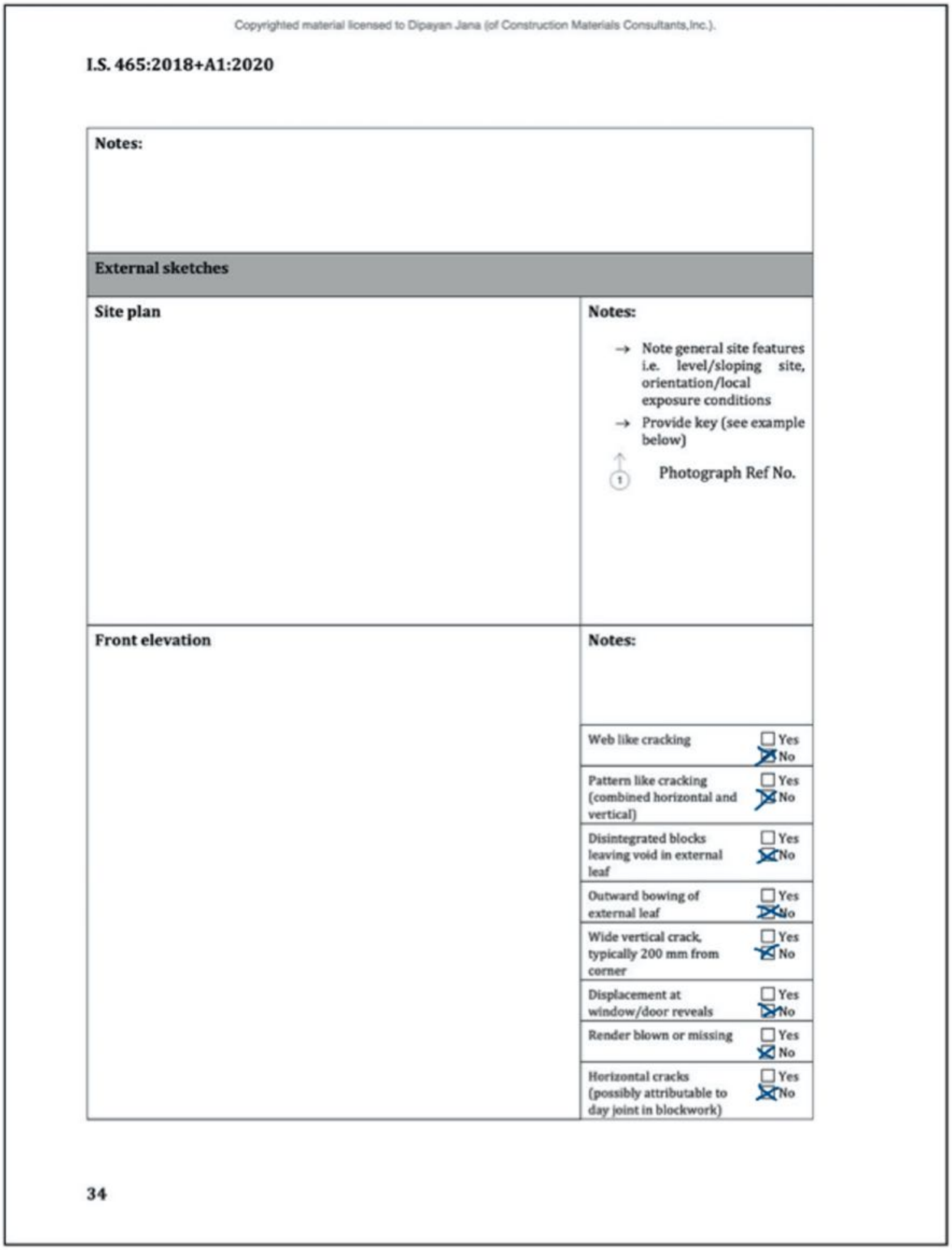
f) A copy of the laboratory analysis reports (see 7.9).



Annex A
(informative)

Example Building Condition Assessment report template

Information on the building	
Address:	**Eircode:**
Type of building:	
Description of site location e.g. in a residential estate or private site:	
Orientation:	
Year built:	
Floor area (m²):	
Year defects first appeared:	
Weather at time of assessment:	
Current owner:	
Other information e.g. brief history of development of damage:	
Site inspection of damage	
Chartered Engineer carrying out the inspection:	
Date:	**Qualifications:**
Circumstantial Evidence	
Source of concrete block materials:	
Is there information that the blocks in the dwelling came from manufacturer(s) reported to have supplied blocks to other dwellings exhibiting damage likely to have arisen from deleterious material in concrete blocks?	☐ Yes ☐ No
Was the dwelling constructed within the date range of constructions mentioned in the Report of the Expert Panel on Concrete Blocks [1], and in the geographic areas reported to be affected?	☐ Yes ☐ No
Is there documented information (e.g. Chartered Engineer's Report) that other dwellings in the same estate or locale have exhibited signs of damage likely to have arisen from deleterious material in concrete blocks?	☐ Yes ☐ No
Are other houses in the same estate exhibiting signs of damage likely to have arisen from deleterious material in concrete blocks?	☐ Yes ☐ No



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Notes:

External sketches

Site plan

Notes:

- → Note general site features i.e. level/sloping site, orientation/local exposure conditions
- → Provide key (see example below)

① Photograph Ref No.

Front elevation

Notes:

Web like cracking	☐ Yes ☒ No
Pattern like cracking (combined horizontal and vertical)	☐ Yes ☒ No
Disintegrated blocks leaving void in external leaf	☐ Yes ☒ No
Outward bowing of external leaf	☐ Yes ☒ No
Wide vertical crack, typically 200 mm from corner	☐ Yes ☒ No
Displacement at window/door reveals	☐ Yes ☒ No
Render blown or missing	☐ Yes ☒ No
Horizontal cracks (possibly attributable to day joint in blockwork)	☐ Yes ☒ No

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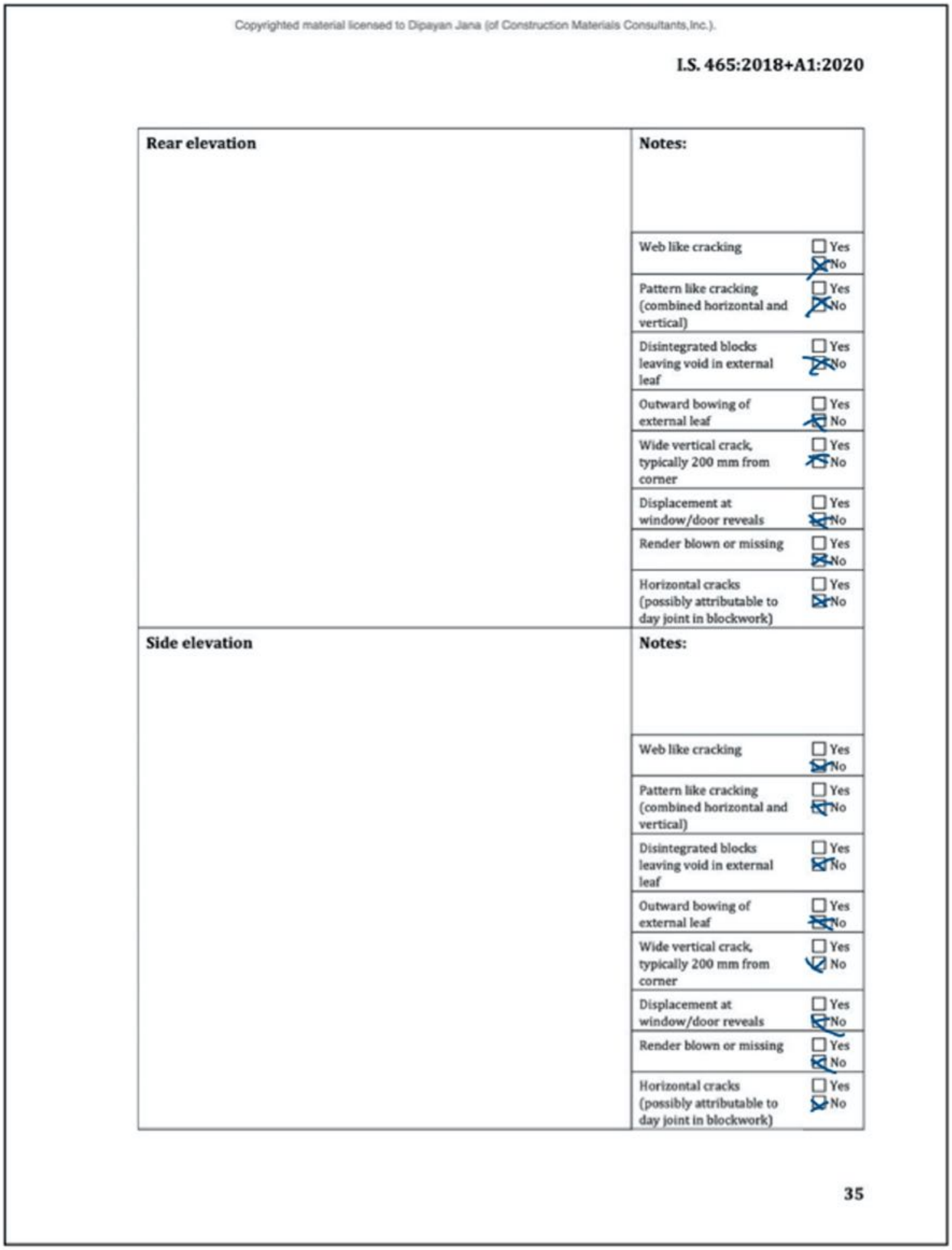
Homeowner's authorization



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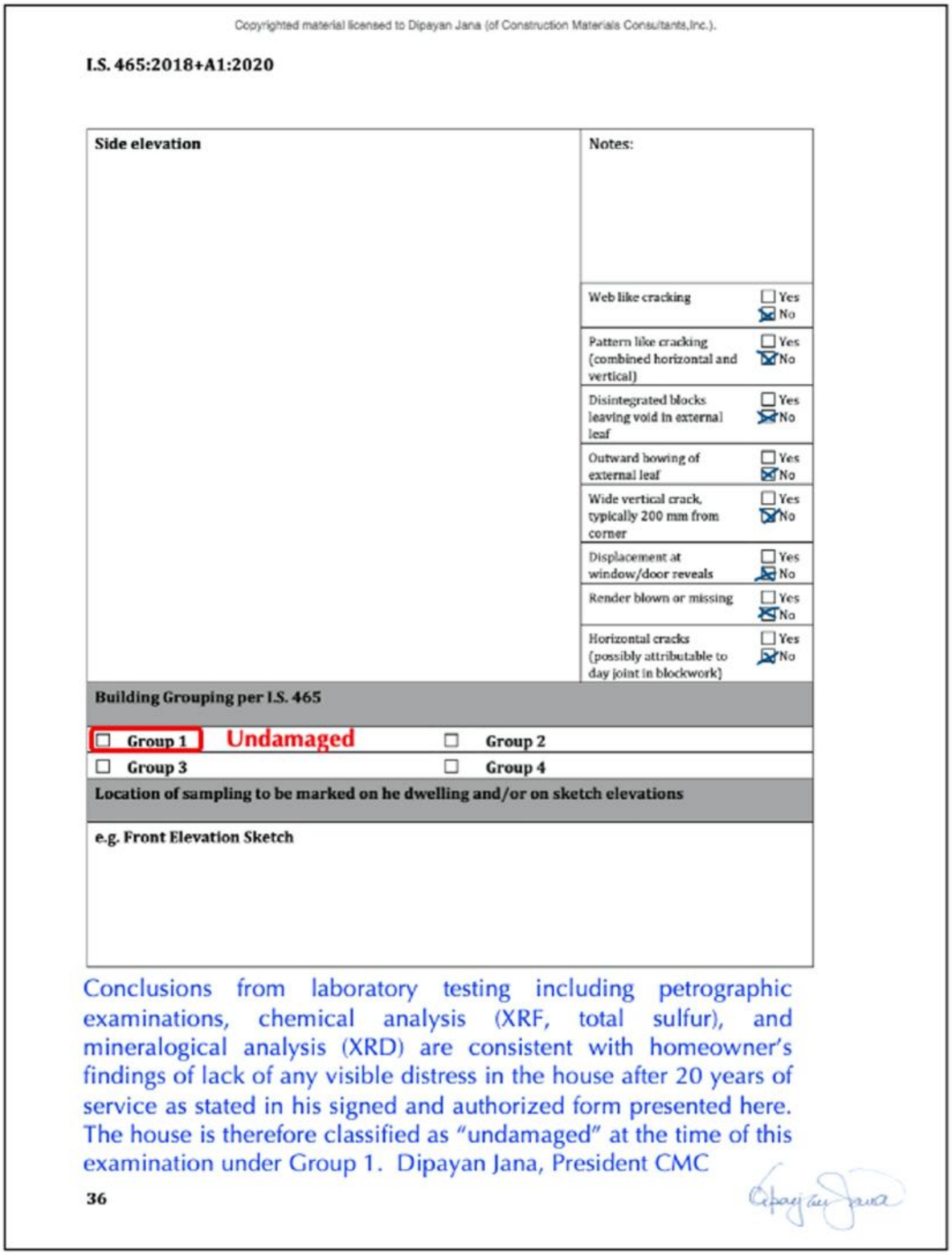
Rear elevation	**Notes:**	
	Web like cracking	☐ Yes ☒ No
	Pattern like cracking (combined horizontal and vertical)	☐ Yes ☒ No
	Disintegrated blocks leaving void in external leaf	☐ Yes ☒ No
	Outward bowing of external leaf	☐ Yes ☒ No
	Wide vertical crack, typically 200 mm from corner	☐ Yes ☒ No
	Displacement at window/door reveals	☐ Yes ☒ No
	Render blown or missing	☐ Yes ☒ No
	Horizontal cracks (possibly attributable to day joint in blockwork)	☐ Yes ☒ No
Side elevation	**Notes:**	
	Web like cracking	☐ Yes ☒ No
	Pattern like cracking (combined horizontal and vertical)	☐ Yes ☒ No
	Disintegrated blocks leaving void in external leaf	☐ Yes ☒ No
	Outward bowing of external leaf	☐ Yes ☒ No
	Wide vertical crack, typically 200 mm from corner	☐ Yes ☑ No
	Displacement at window/door reveals	☐ Yes ☒ No
	Render blown or missing	☐ Yes ☒ No
	Horizontal cracks (possibly attributable to day joint in blockwork)	☐ Yes ☒ No



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Side elevation	Notes:	
	Web like cracking	☐ Yes ☒ No
	Pattern like cracking (combined horizontal and vertical)	☐ Yes ☒ No
	Disintegrated blocks leaving void in external leaf	☐ Yes ☒ No
	Outward bowing of external leaf	☐ Yes ☒ No
	Wide vertical crack, typically 200 mm from corner	☐ Yes ☒ No
	Displacement at window/door reveals	☐ Yes ☒ No
	Render blown or missing	☐ Yes ☒ No
	Horizontal cracks (possibly attributable to day joint in blockwork)	☐ Yes ☒ No

Building Grouping per I.S. 465

☐ **Group 1** Undamaged	☐ **Group 2**
☐ **Group 3**	☐ **Group 4**

Location of sampling to be marked on he dwelling and/or on sketch elevations

e.g. Front Elevation Sketch

Conclusions from laboratory testing including petrographic examinations, chemical analysis (XRF, total sulfur), and mineralogical analysis (XRD) are consistent with homeowner's findings of lack of any visible distress in the house after 20 years of service as stated in his signed and authorized form presented here. The house is therefore classified as "undamaged" at the time of this examination under Group 1. Dipayan Jana, President CMC

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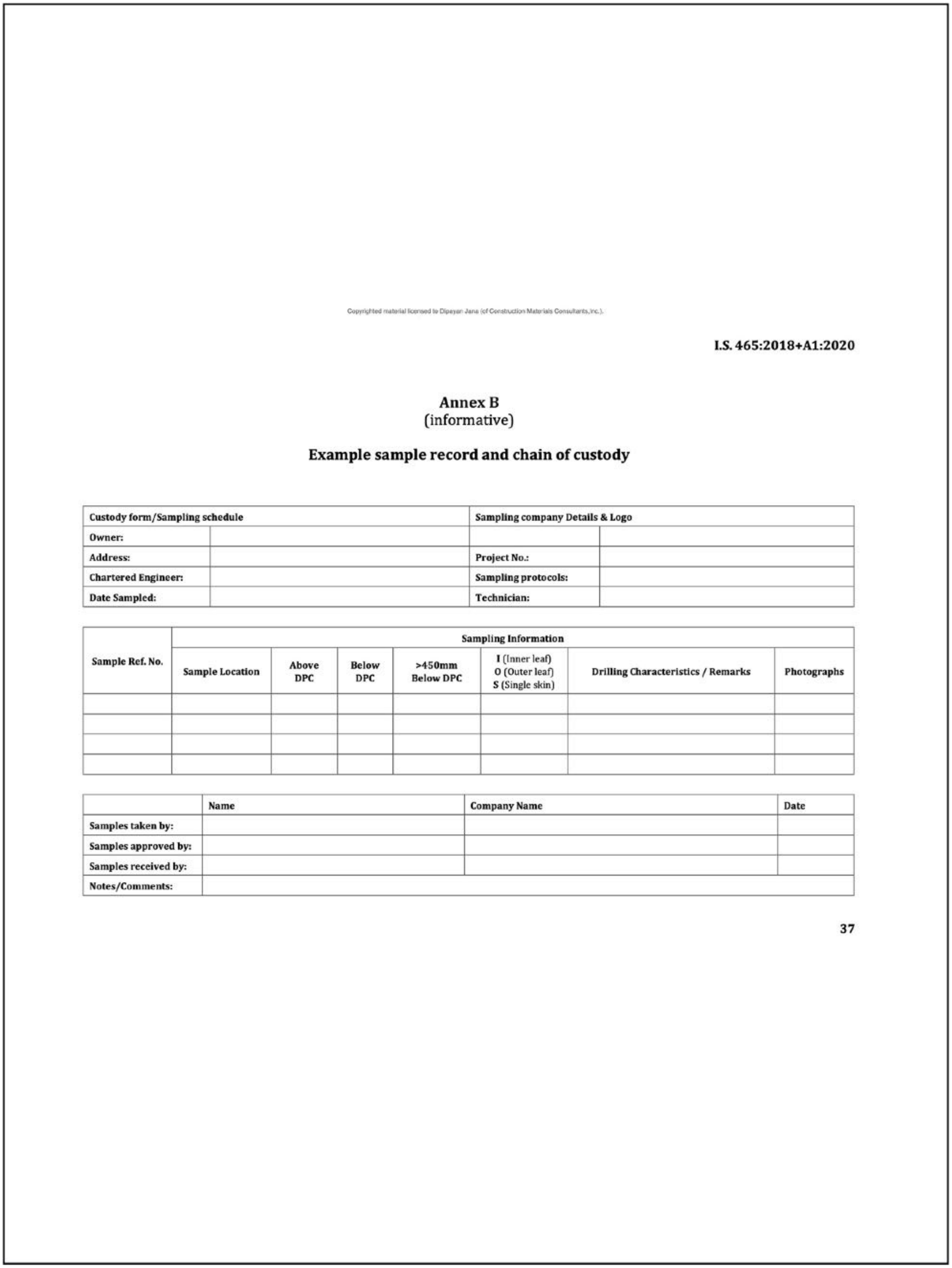
Dipayan Jana

STATE OF TEXAS
Dipayan Jana
Geology
2712
PROFESSIONAL LICENSED GEOSCIENTIST

State of Washington
1107
Licensed Geologist
Dipayan Jana



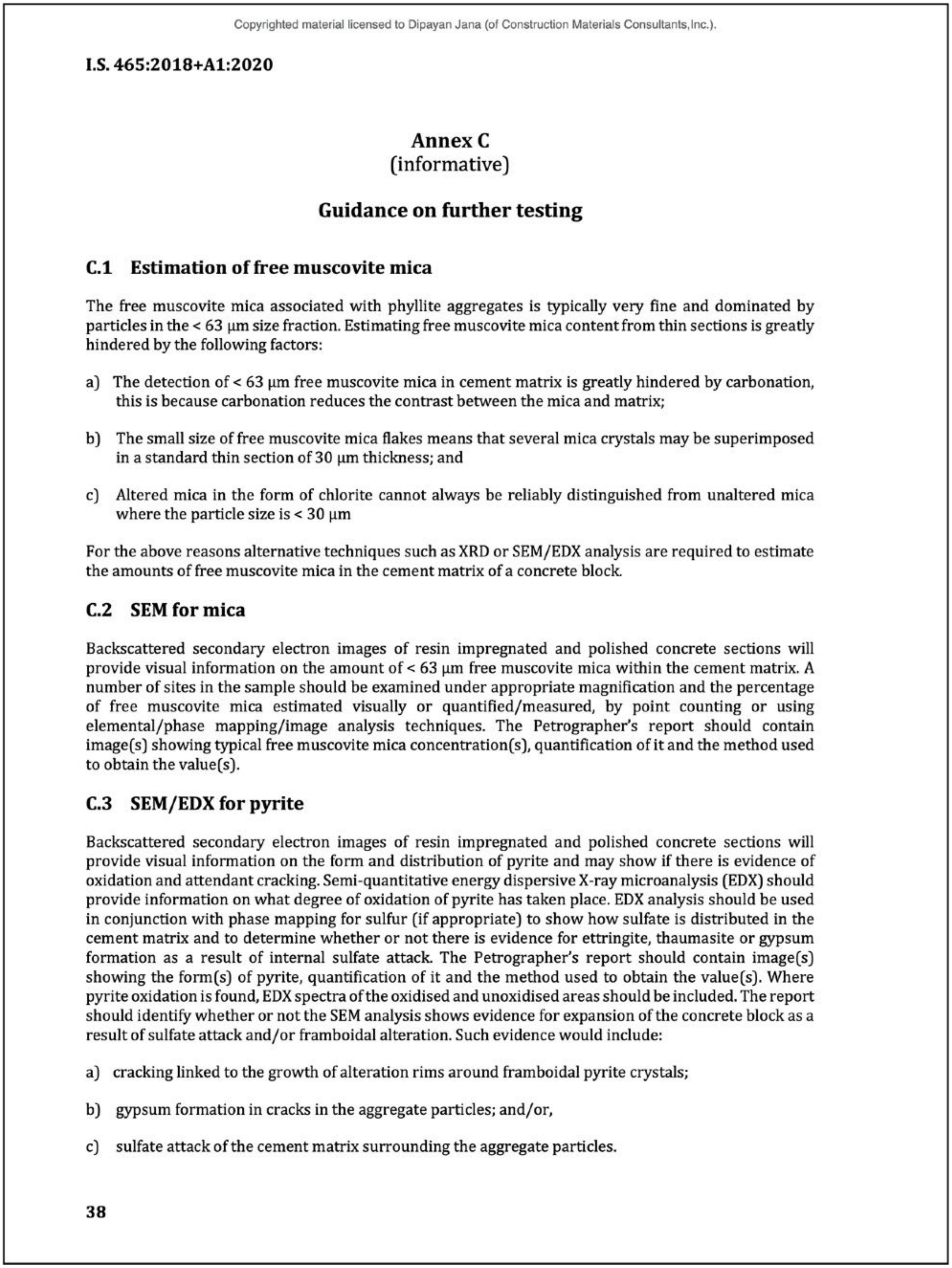
Annex B
(informative)

Example sample record and chain of custody

Custody form/Sampling schedule		**Sampling company Details & Logo**	
Owner:			
Address:		**Project No.:**	
Chartered Engineer:		**Sampling protocols:**	
Date Sampled:		**Technician:**	

Sample Ref. No.	**Sampling Information**						
	Sample Location	**Above DPC**	**Below DPC**	**>450mm Below DPC**	**I** (Inner leaf) **O** (Outer leaf) **S** (Single skin)	**Drilling Characteristics / Remarks**	**Photographs**

	Name	**Company Name**	**Date**
Samples taken by:			
Samples approved by:			
Samples received by:			
Notes/Comments:			



Annex C
(informative)

Guidance on further testing

C.1 Estimation of free muscovite mica

The free muscovite mica associated with phyllite aggregates is typically very fine and dominated by particles in the < 63 µm size fraction. Estimating free muscovite mica content from thin sections is greatly hindered by the following factors:

a) The detection of < 63 µm free muscovite mica in cement matrix is greatly hindered by carbonation, this is because carbonation reduces the contrast between the mica and matrix;

b) The small size of free muscovite mica flakes means that several mica crystals may be superimposed in a standard thin section of 30 µm thickness; and

c) Altered mica in the form of chlorite cannot always be reliably distinguished from unaltered mica where the particle size is < 30 µm

For the above reasons alternative techniques such as XRD or SEM/EDX analysis are required to estimate the amounts of free muscovite mica in the cement matrix of a concrete block.

C.2 SEM for mica

Backscattered secondary electron images of resin impregnated and polished concrete sections will provide visual information on the amount of < 63 µm free muscovite mica within the cement matrix. A number of sites in the sample should be examined under appropriate magnification and the percentage of free muscovite mica estimated visually or quantified/measured, by point counting or using elemental/phase mapping/image analysis techniques. The Petrographer's report should contain image(s) showing typical free muscovite mica concentration(s), quantification of it and the method used to obtain the value(s).

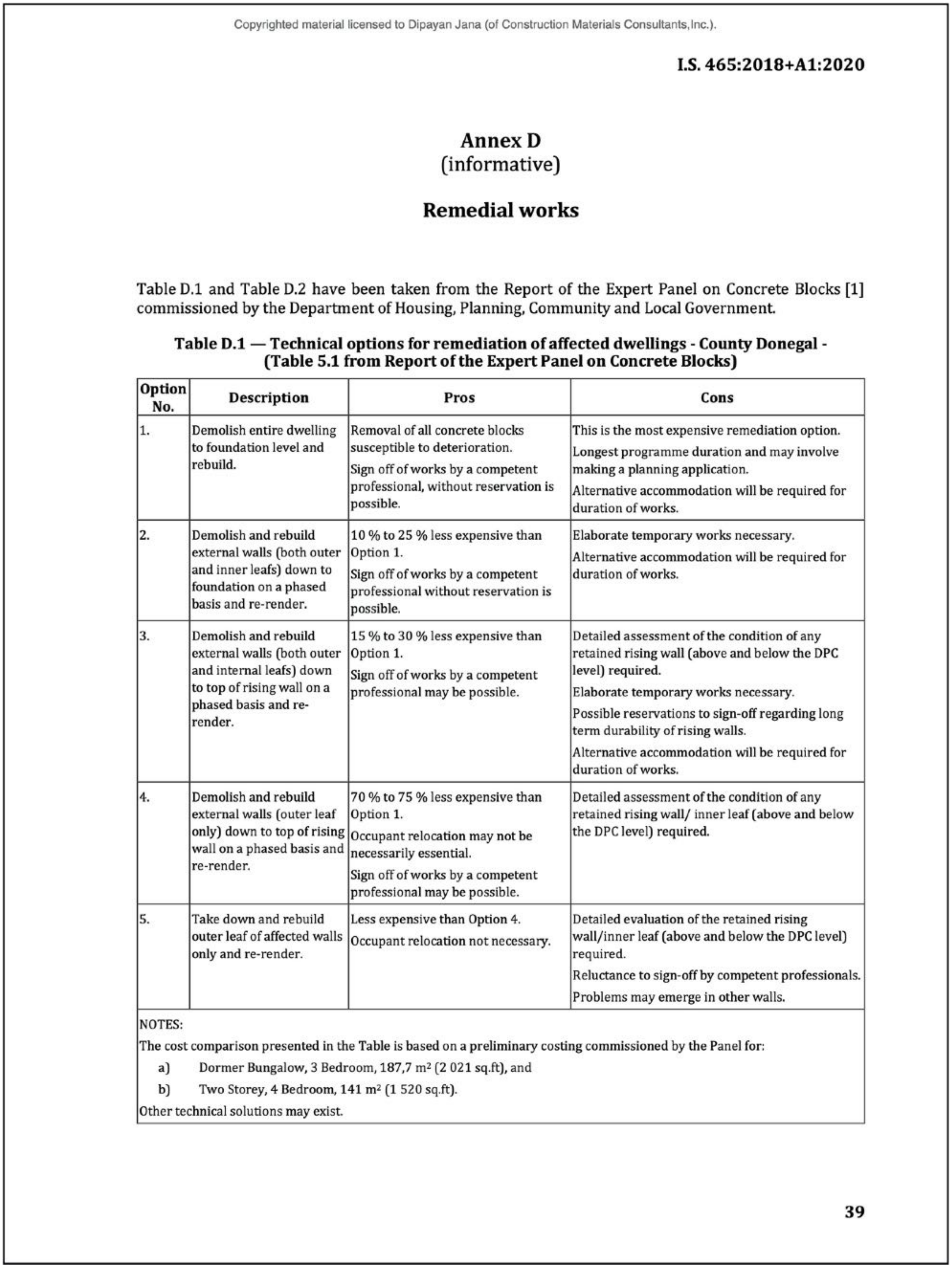
C.3 SEM/EDX for pyrite

Backscattered secondary electron images of resin impregnated and polished concrete sections will provide visual information on the form and distribution of pyrite and may show if there is evidence of oxidation and attendant cracking. Semi-quantitative energy dispersive X-ray microanalysis (EDX) should provide information on what degree of oxidation of pyrite has taken place. EDX analysis should be used in conjunction with phase mapping for sulfur (if appropriate) to show how sulfate is distributed in the cement matrix and to determine whether or not there is evidence for ettringite, thaumasite or gypsum formation as a result of internal sulfate attack. The Petrographer's report should contain image(s) showing the form(s) of pyrite, quantification of it and the method used to obtain the value(s). Where pyrite oxidation is found, EDX spectra of the oxidised and unoxidised areas should be included. The report should identify whether or not the SEM analysis shows evidence for expansion of the concrete block as a result of sulfate attack and/or framboidal alteration. Such evidence would include:

a) cracking linked to the growth of alteration rims around framboidal pyrite crystals;

b) gypsum formation in cracks in the aggregate particles; and/or,

c) sulfate attack of the cement matrix surrounding the aggregate particles.



Annex D
(informative)

Remedial works

Table D.1 and Table D.2 have been taken from the Report of the Expert Panel on Concrete Blocks [1] commissioned by the Department of Housing, Planning, Community and Local Government.

Table D.1 — Technical options for remediation of affected dwellings - County Donegal - (Table 5.1 from Report of the Expert Panel on Concrete Blocks)

Option No.	Description	Pros	Cons
1.	Demolish entire dwelling to foundation level and rebuild.	Removal of all concrete blocks susceptible to deterioration. Sign off of works by a competent professional, without reservation is possible.	This is the most expensive remediation option. Longest programme duration and may involve making a planning application. Alternative accommodation will be required for duration of works.
2.	Demolish and rebuild external walls (both outer and inner leafs) down to foundation on a phased basis and re-render.	10 % to 25 % less expensive than Option 1. Sign off of works by a competent professional without reservation is possible.	Elaborate temporary works necessary. Alternative accommodation will be required for duration of works.
3.	Demolish and rebuild external walls (both outer and internal leafs) down to top of rising wall on a phased basis and re-render.	15 % to 30 % less expensive than Option 1. Sign off of works by a competent professional may be possible.	Detailed assessment of the condition of any retained rising wall (above and below the DPC level) required. Elaborate temporary works necessary. Possible reservations to sign-off regarding long term durability of rising walls. Alternative accommodation will be required for duration of works.
4.	Demolish and rebuild external walls (outer leaf only) down to top of rising wall on a phased basis and re-render.	70 % to 75 % less expensive than Option 1. Occupant relocation may not be necessarily essential. Sign off of works by a competent professional may be possible.	Detailed assessment of the condition of any retained rising wall/ inner leaf (above and below the DPC level) required.
5.	Take down and rebuild outer leaf of affected walls only and re-render.	Less expensive than Option 4. Occupant relocation not necessary.	Detailed evaluation of the retained rising wall/inner leaf (above and below the DPC level) required. Reluctance to sign-off by competent professionals. Problems may emerge in other walls.

NOTES:

The cost comparison presented in the Table is based on a preliminary costing commissioned by the Panel for:

a) Dormer Bungalow, 3 Bedroom, 187,7 m^2 (2 021 sq.ft), and
b) Two Storey, 4 Bedroom, 141 m^2 (1 520 sq.ft).

Other technical solutions may exist.

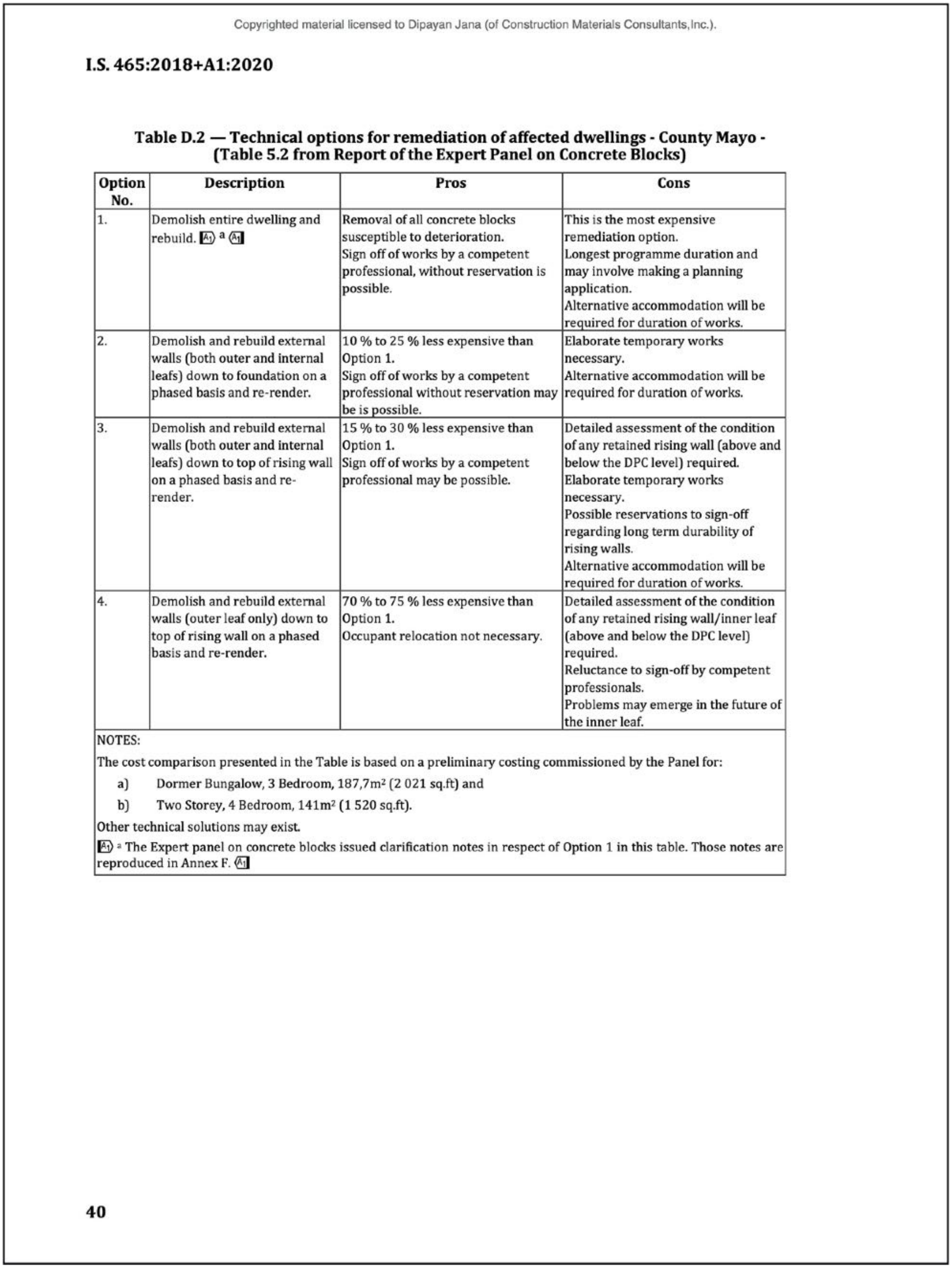


Table D.2 — Technical options for remediation of affected dwellings - County Mayo - (Table 5.2 from Report of the Expert Panel on Concrete Blocks)

Option No.	Description	Pros	Cons
1.	Demolish entire dwelling and rebuild. A1 [a] A1	Removal of all concrete blocks susceptible to deterioration. Sign off of works by a competent professional, without reservation is possible.	This is the most expensive remediation option. Longest programme duration and may involve making a planning application. Alternative accommodation will be required for duration of works.
2.	Demolish and rebuild external walls (both outer and internal leafs) down to foundation on a phased basis and re-render.	10 % to 25 % less expensive than Option 1. Sign off of works by a competent professional without reservation may be is possible.	Elaborate temporary works necessary. Alternative accommodation will be required for duration of works.
3.	Demolish and rebuild external walls (both outer and internal leafs) down to top of rising wall on a phased basis and re-render.	15 % to 30 % less expensive than Option 1. Sign off of works by a competent professional may be possible.	Detailed assessment of the condition of any retained rising wall (above and below the DPC level) required. Elaborate temporary works necessary. Possible reservations to sign-off regarding long term durability of rising walls. Alternative accommodation will be required for duration of works.
4.	Demolish and rebuild external walls (outer leaf only) down to top of rising wall on a phased basis and re-render.	70 % to 75 % less expensive than Option 1. Occupant relocation not necessary.	Detailed assessment of the condition of any retained rising wall/inner leaf (above and below the DPC level) required. Reluctance to sign-off by competent professionals. Problems may emerge in the future of the inner leaf.

NOTES:

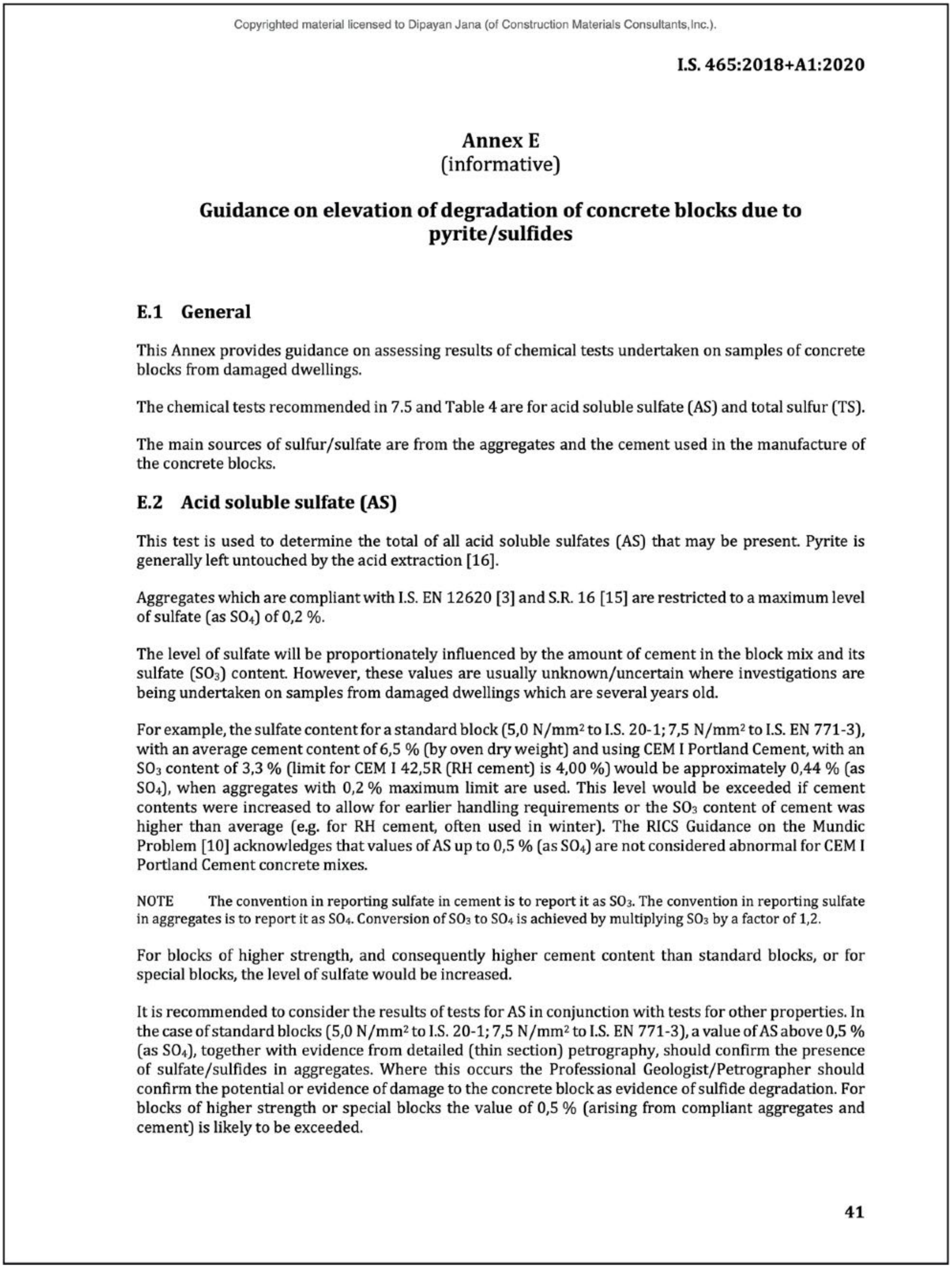
The cost comparison presented in the Table is based on a preliminary costing commissioned by the Panel for:

a) Dormer Bungalow, 3 Bedroom, 187,7m^2 (2 021 sq.ft) and

b) Two Storey, 4 Bedroom, 141m^2 (1 520 sq.ft).

Other technical solutions may exist.

A1 [a] The Expert panel on concrete blocks issued clarification notes in respect of Option 1 in this table. Those notes are reproduced in Annex F. A1



Annex E
(informative)

Guidance on elevation of degradation of concrete blocks due to pyrite/sulfides

E.1 General

This Annex provides guidance on assessing results of chemical tests undertaken on samples of concrete blocks from damaged dwellings.

The chemical tests recommended in 7.5 and Table 4 are for acid soluble sulfate (AS) and total sulfur (TS).

The main sources of sulfur/sulfate are from the aggregates and the cement used in the manufacture of the concrete blocks.

E.2 Acid soluble sulfate (AS)

This test is used to determine the total of all acid soluble sulfates (AS) that may be present. Pyrite is generally left untouched by the acid extraction [16].

Aggregates which are compliant with I.S. EN 12620 [3] and S.R. 16 [15] are restricted to a maximum level of sulfate (as SO_4) of 0,2 %.

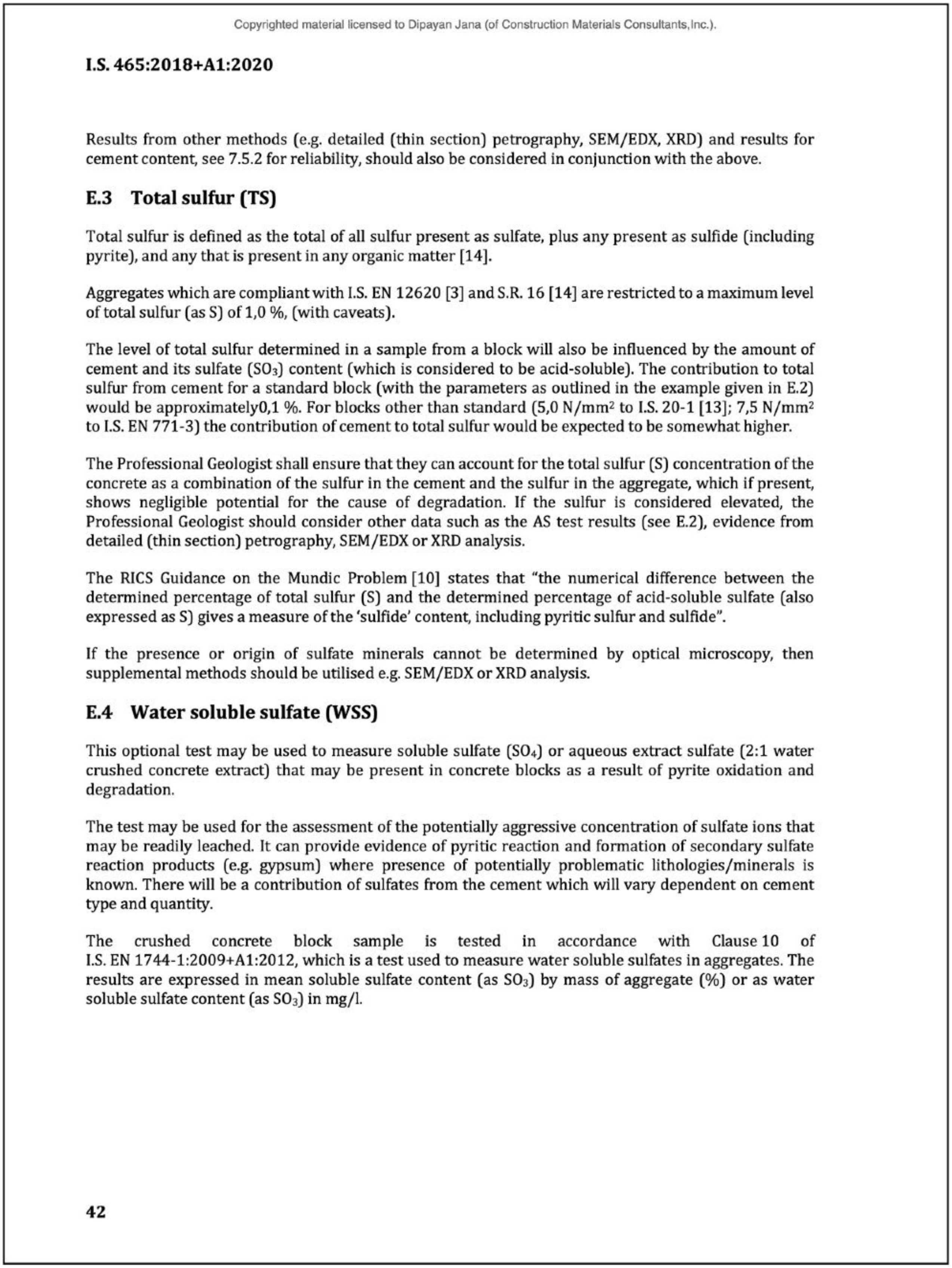
The level of sulfate will be proportionately influenced by the amount of cement in the block mix and its sulfate (SO_3) content. However, these values are usually unknown/uncertain where investigations are being undertaken on samples from damaged dwellings which are several years old.

For example, the sulfate content for a standard block (5,0 N/mm^2 to I.S. 20-1; 7,5 N/mm^2 to I.S. EN 771-3), with an average cement content of 6,5 % (by oven dry weight) and using CEM I Portland Cement, with an SO_3 content of 3,3 % (limit for CEM I 42,5R (RH cement) is 4,00 %) would be approximately 0,44 % (as SO_4), when aggregates with 0,2 % maximum limit are used. This level would be exceeded if cement contents were increased to allow for earlier handling requirements or the SO_3 content of cement was higher than average (e.g. for RH cement, often used in winter). The RICS Guidance on the Mundic Problem [10] acknowledges that values of AS up to 0,5 % (as SO_4) are not considered abnormal for CEM I Portland Cement concrete mixes.

NOTE The convention in reporting sulfate in cement is to report it as SO_3. The convention in reporting sulfate in aggregates is to report it as SO_4. Conversion of SO_3 to SO_4 is achieved by multiplying SO_3 by a factor of 1,2.

For blocks of higher strength, and consequently higher cement content than standard blocks, or for special blocks, the level of sulfate would be increased.

It is recommended to consider the results of tests for AS in conjunction with tests for other properties. In the case of standard blocks (5,0 N/mm^2 to I.S. 20-1; 7,5 N/mm^2 to I.S. EN 771-3), a value of AS above 0,5 % (as SO_4), together with evidence from detailed (thin section) petrography, should confirm the presence of sulfate/sulfides in aggregates. Where this occurs the Professional Geologist/Petrographer should confirm the potential or evidence of damage to the concrete block as evidence of sulfide degradation. For blocks of higher strength or special blocks the value of 0,5 % (arising from compliant aggregates and cement) is likely to be exceeded.



Results from other methods (e.g. detailed (thin section) petrography, SEM/EDX, XRD) and results for cement content, see 7.5.2 for reliability, should also be considered in conjunction with the above.

E.3 Total sulfur (TS)

Total sulfur is defined as the total of all sulfur present as sulfate, plus any present as sulfide (including pyrite), and any that is present in any organic matter [14].

Aggregates which are compliant with I.S. EN 12620 [3] and S.R. 16 [14] are restricted to a maximum level of total sulfur (as S) of 1,0 %, (with caveats).

The level of total sulfur determined in a sample from a block will also be influenced by the amount of cement and its sulfate (SO_3) content (which is considered to be acid-soluble). The contribution to total sulfur from cement for a standard block (with the parameters as outlined in the example given in E.2) would be approximately0,1 %. For blocks other than standard (5,0 N/mm^2 to I.S. 20-1 [13]; 7,5 N/mm^2 to I.S. EN 771-3) the contribution of cement to total sulfur would be expected to be somewhat higher.

The Professional Geologist shall ensure that they can account for the total sulfur (S) concentration of the concrete as a combination of the sulfur in the cement and the sulfur in the aggregate, which if present, shows negligible potential for the cause of degradation. If the sulfur is considered elevated, the Professional Geologist should consider other data such as the AS test results (see E.2), evidence from detailed (thin section) petrography, SEM/EDX or XRD analysis.

The RICS Guidance on the Mundic Problem [10] states that "the numerical difference between the determined percentage of total sulfur (S) and the determined percentage of acid-soluble sulfate (also expressed as S) gives a measure of the 'sulfide' content, including pyritic sulfur and sulfide".

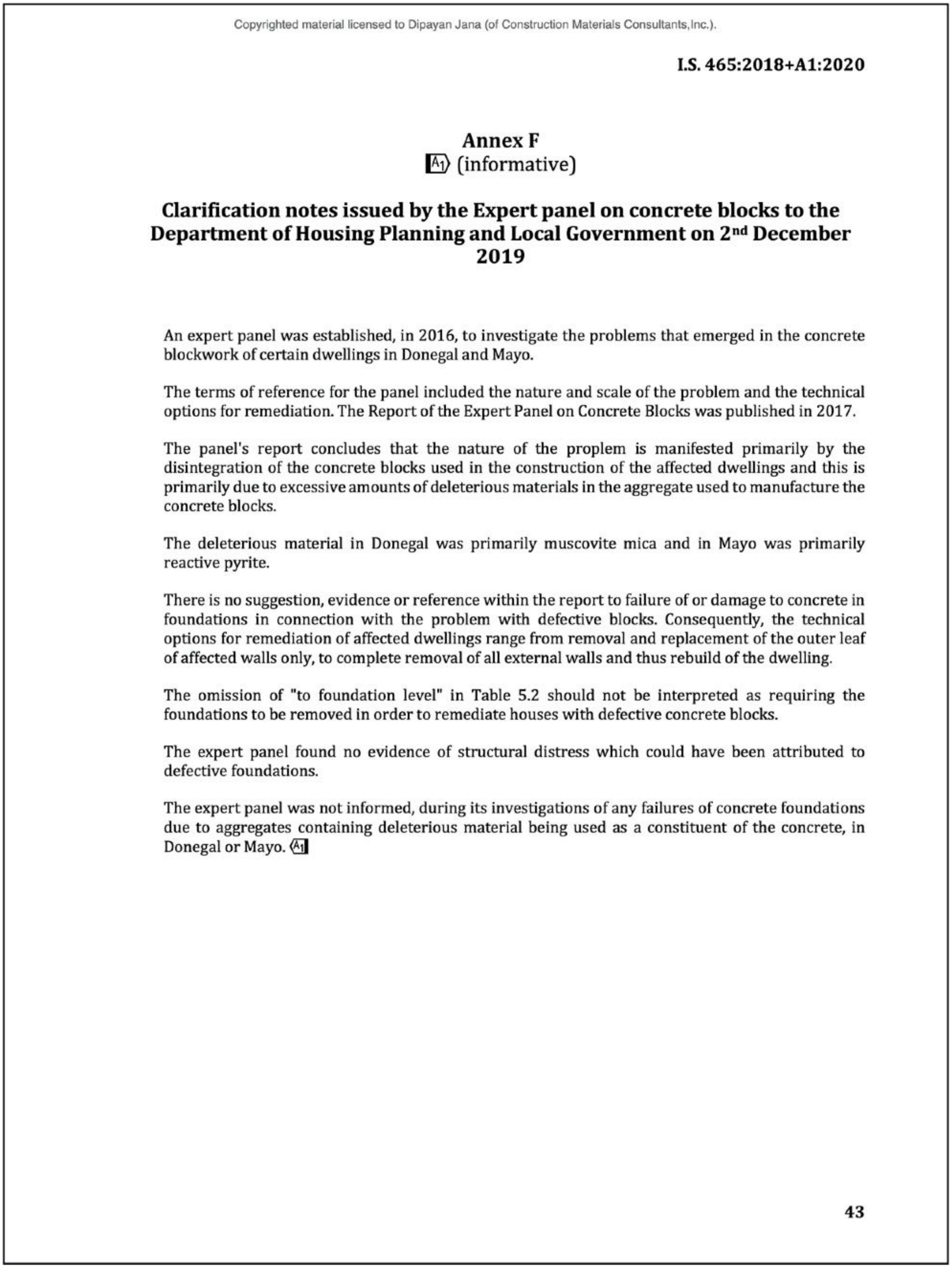
If the presence or origin of sulfate minerals cannot be determined by optical microscopy, then supplemental methods should be utilised e.g. SEM/EDX or XRD analysis.

E.4 Water soluble sulfate (WSS)

This optional test may be used to measure soluble sulfate (SO_4) or aqueous extract sulfate (2:1 water crushed concrete extract) that may be present in concrete blocks as a result of pyrite oxidation and degradation.

The test may be used for the assessment of the potentially aggressive concentration of sulfate ions that may be readily leached. It can provide evidence of pyritic reaction and formation of secondary sulfate reaction products (e.g. gypsum) where presence of potentially problematic lithologies/minerals is known. There will be a contribution of sulfates from the cement which will vary dependent on cement type and quantity.

The crushed concrete block sample is tested in accordance with Clause 10 of I.S. EN 1744-1:2009+A1:2012, which is a test used to measure water soluble sulfates in aggregates. The results are expressed in mean soluble sulfate content (as SO_3) by mass of aggregate (%) or as water soluble sulfate content (as SO_3) in mg/l.



Annex F
A1〉 (informative)

Clarification notes issued by the Expert panel on concrete blocks to the Department of Housing Planning and Local Government on 2nd December 2019

An expert panel was established, in 2016, to investigate the problems that emerged in the concrete blockwork of certain dwellings in Donegal and Mayo.

The terms of reference for the panel included the nature and scale of the problem and the technical options for remediation. The Report of the Expert Panel on Concrete Blocks was published in 2017.

The panel's report concludes that the nature of the proplem is manifested primarily by the disintegration of the concrete blocks used in the construction of the affected dwellings and this is primarily due to excessive amounts of deleterious materials in the aggregate used to manufacture the concrete blocks.

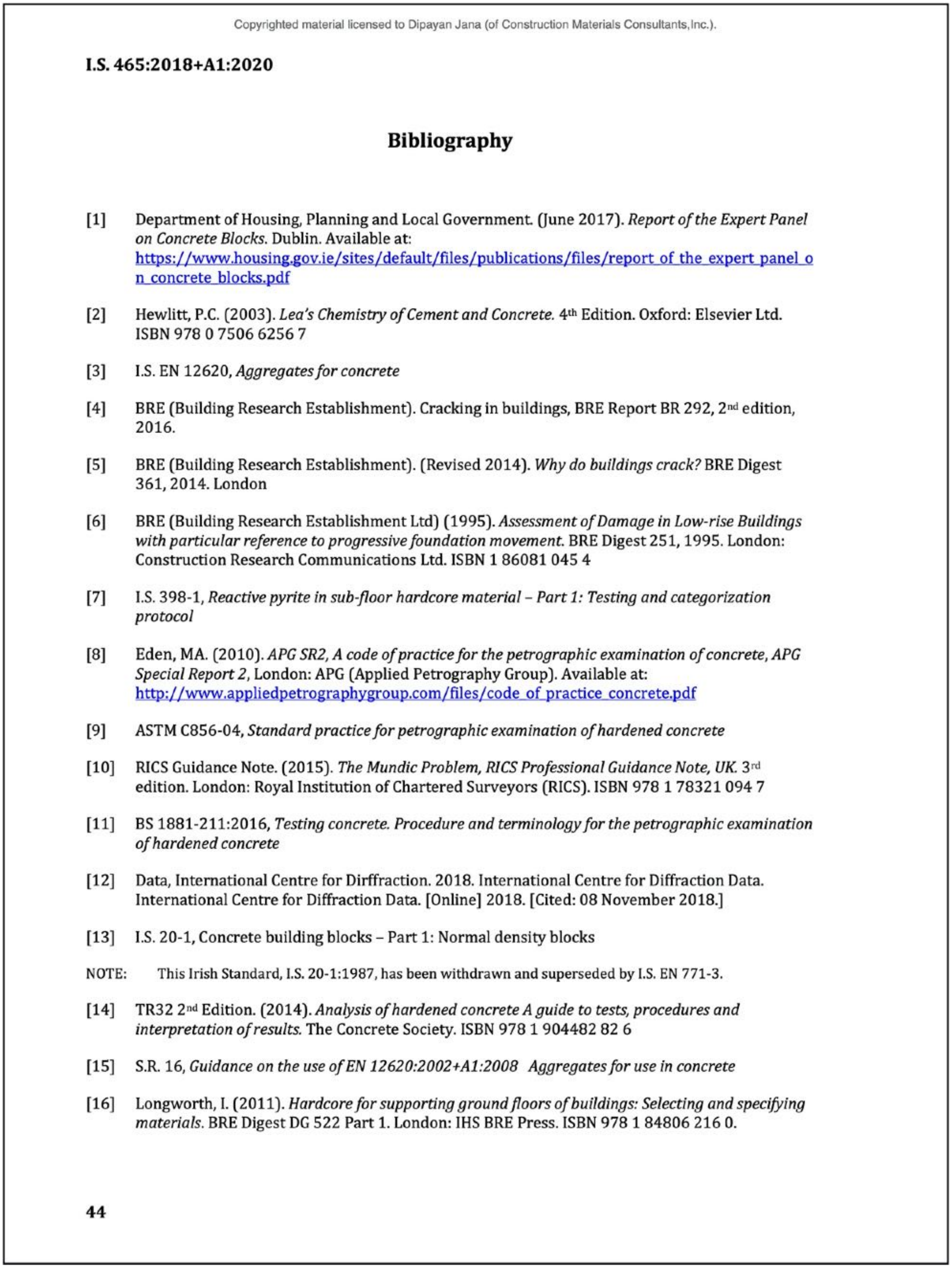
The deleterious material in Donegal was primarily muscovite mica and in Mayo was primarily reactive pyrite.

There is no suggestion, evidence or reference within the report to failure of or damage to concrete in foundations in connection with the problem with defective blocks. Consequently, the technical options for remediation of affected dwellings range from removal and replacement of the outer leaf of affected walls only, to complete removal of all external walls and thus rebuild of the dwelling.

The omission of "to foundation level" in Table 5.2 should not be interpreted as requiring the foundations to be removed in order to remediate houses with defective concrete blocks.

The expert panel found no evidence of structural distress which could have been attributed to defective foundations.

The expert panel was not informed, during its investigations of any failures of concrete foundations due to aggregates containing deleterious material being used as a constituent of the concrete, in Donegal or Mayo. 〈A1



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[13] I.S. 20-1, Concrete building blocks – Part 1: Normal density blocks

NOTE: This Irish Standard, I.S. 20-1:1987, has been withdrawn and superseded by I.S. EN 771-3.

[14] TR32 2nd Edition. (2014). *Analysis of hardened concrete A guide to tests, procedures and interpretation of results.* The Concrete Society. ISBN 978 1 904482 82 6

[15] S.R. 16, *Guidance on the use of EN 12620:2002+A1:2008 Aggregates for use in concrete*

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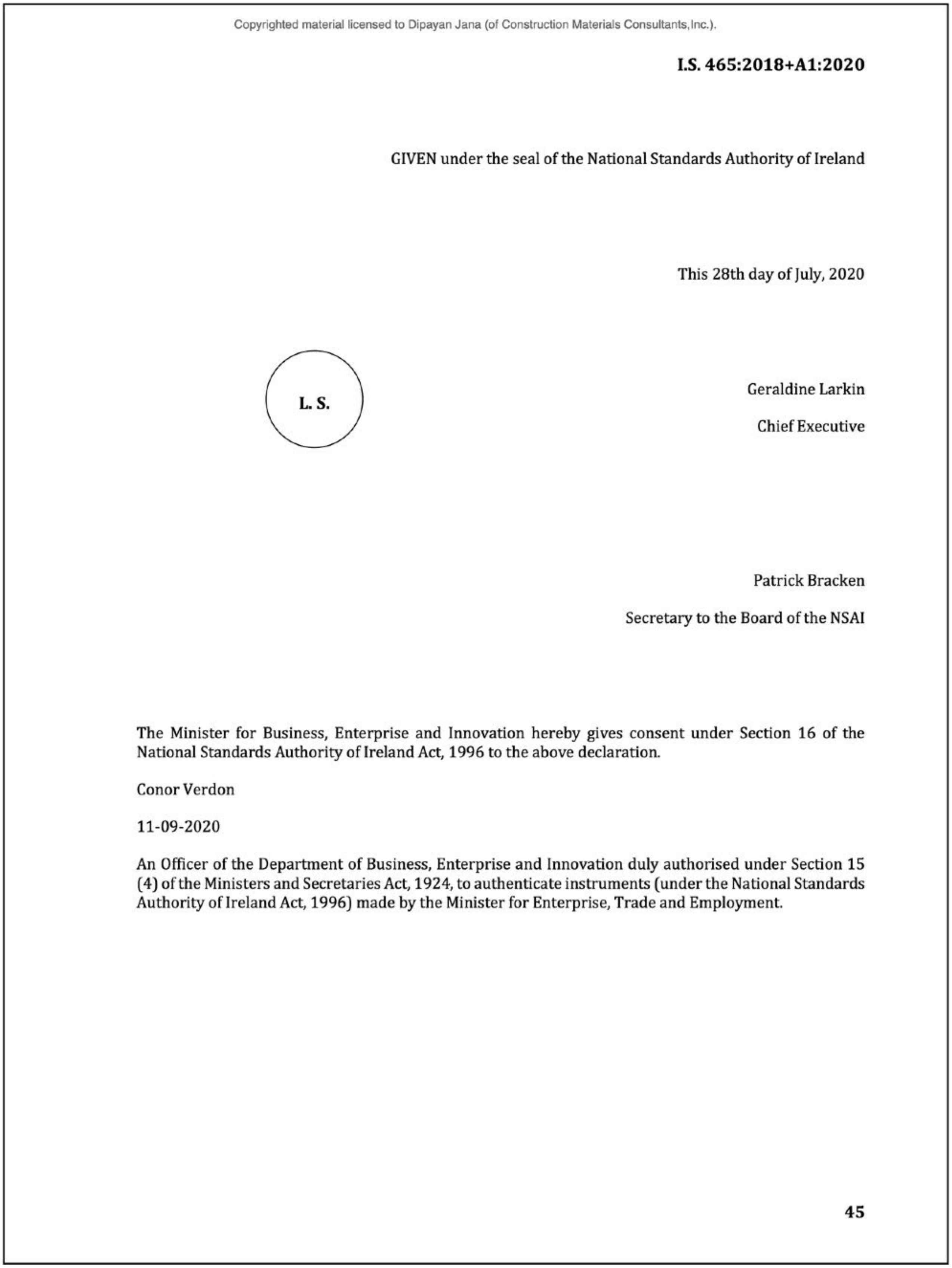


GIVEN under the seal of the National Standards Authority of Ireland

This 28th day of July, 2020

L. S.

Geraldine Larkin

Chief Executive

Patrick Bracken

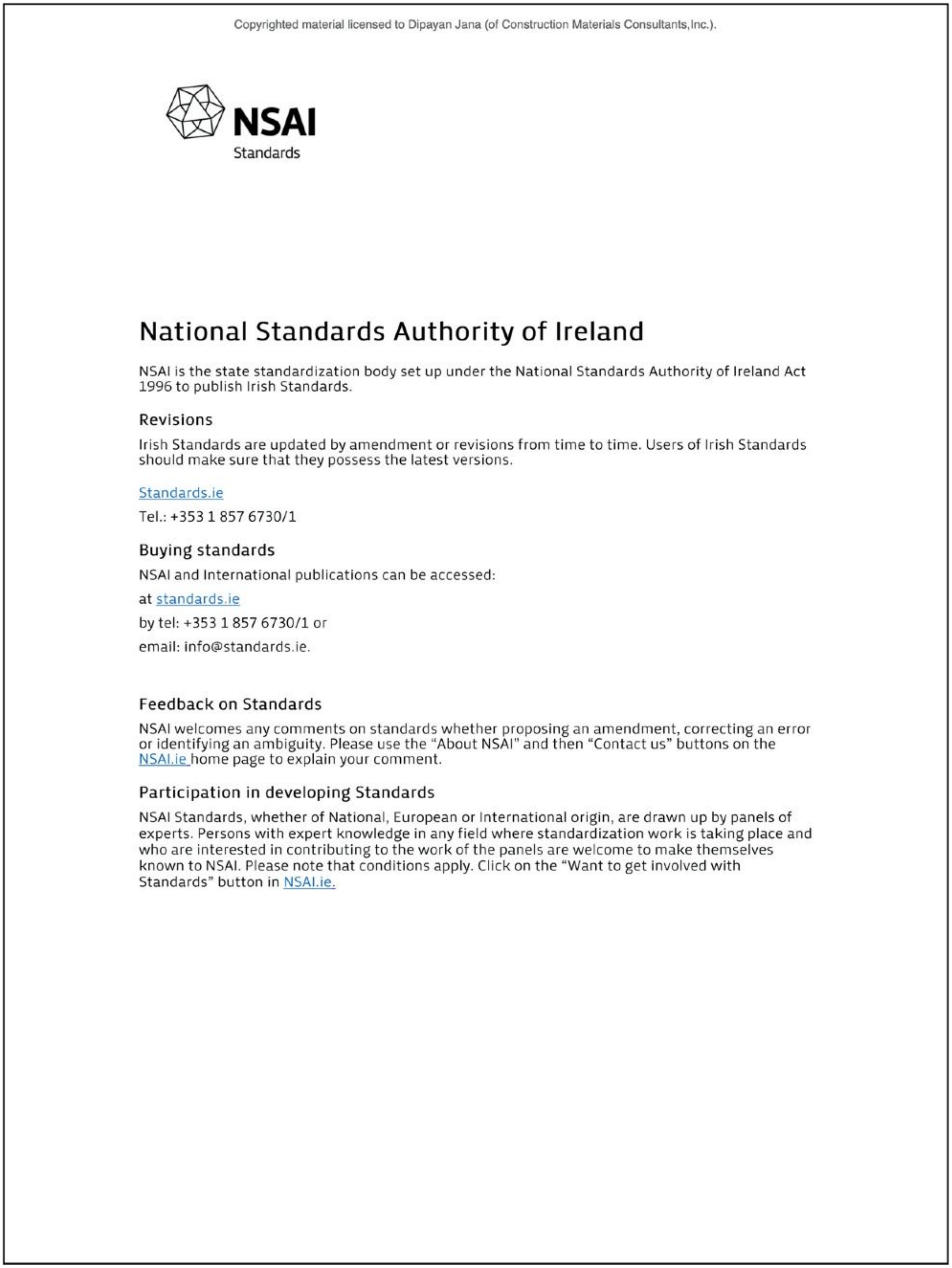
Secretary to the Board of the NSAI

The Minister for Business, Enterprise and Innovation hereby gives consent under Section 16 of the National Standards Authority of Ireland Act, 1996 to the above declaration.

Conor Verdon

11-09-2020

An Officer of the Department of Business, Enterprise and Innovation duly authorised under Section 15 (4) of the Ministers and Secretaries Act, 1924, to authenticate instruments (under the National Standards Authority of Ireland Act, 1996) made by the Minister for Enterprise, Trade and Employment.



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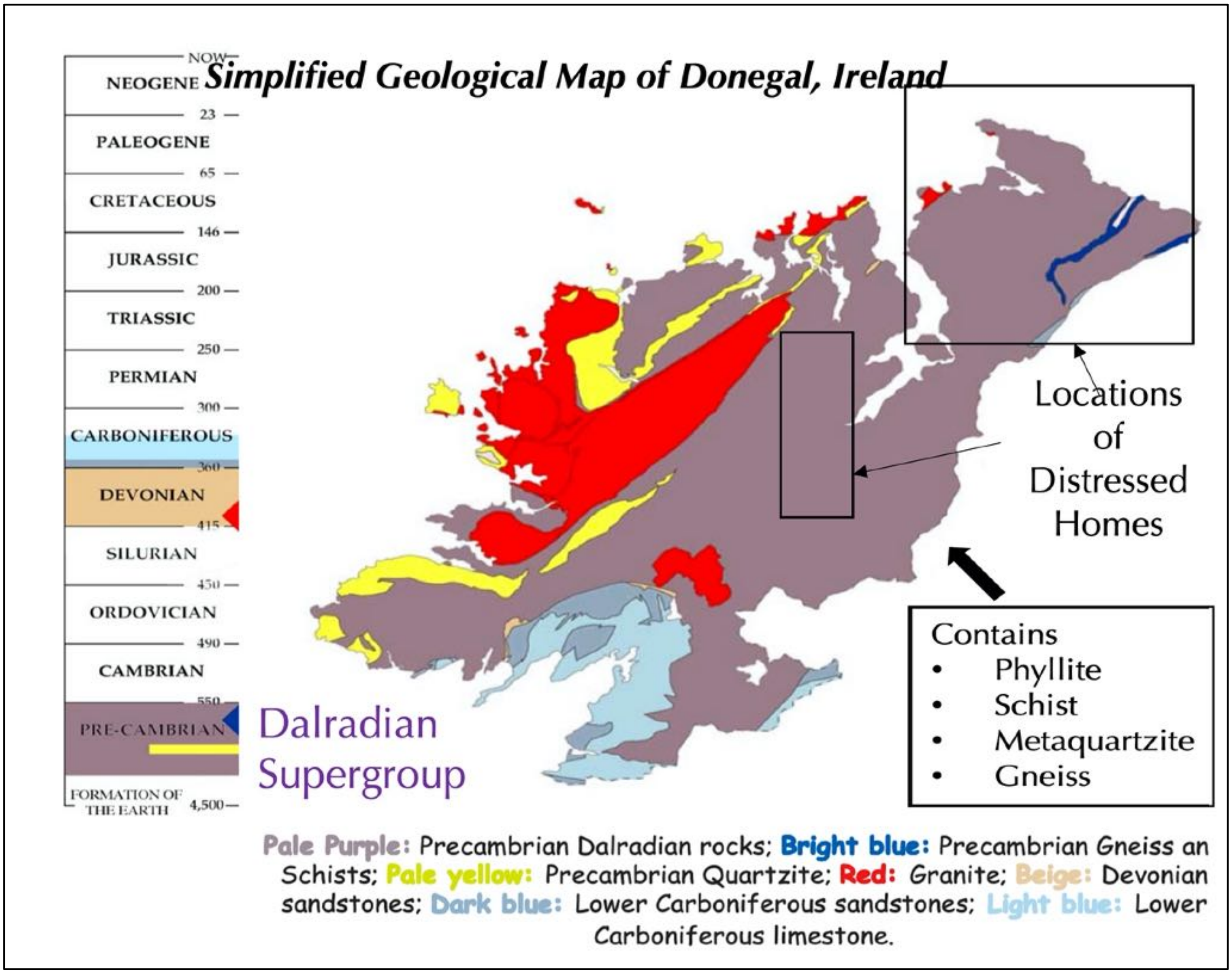
APPENDIX 2 –

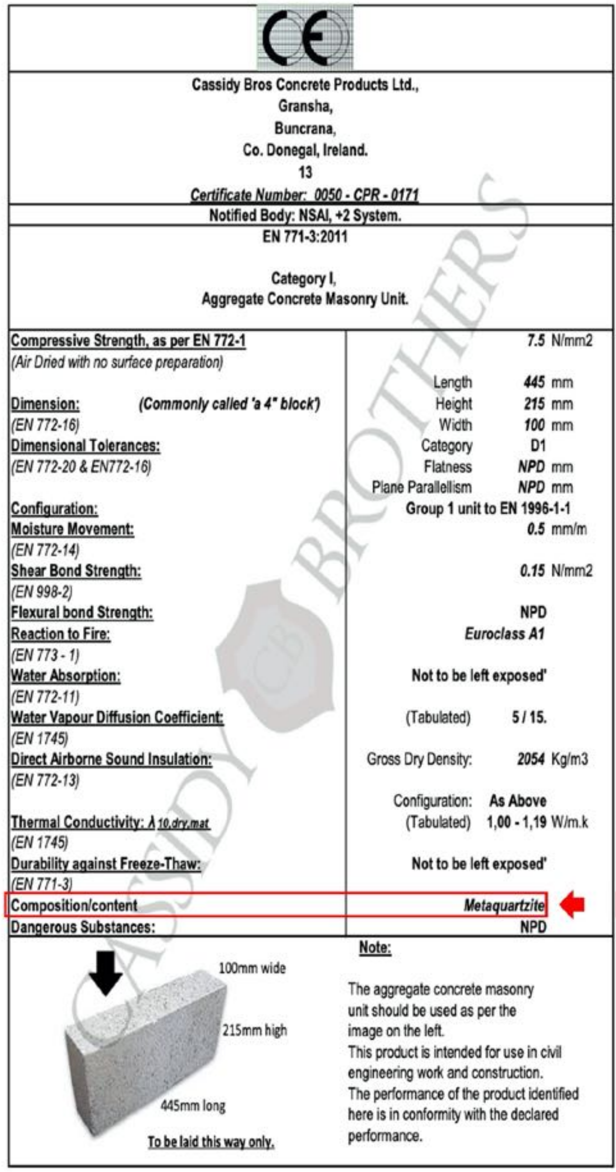
GEOLOGICAL MAP OF DONEGAL, SPECIFICATION OF BLOCKS



Pale Purple: Precambrian Dalradian rocks; **Bright blue:** Precambrian Gneiss an Schists; **Pale yellow:** Precambrian Quartzite; **Red:** Granite; **Beige:** Devonian sandstones; **Dark blue:** Lower Carboniferous sandstones; **Light blue:** Lower Carboniferous limestone.

Manufacturer's Specification of Concrete Blocks

(from https://www.cassidybros.ie)



Cassidy Bros Concrete Products Ltd.,
Gransha,
Buncrana,
Co. Donegal, Ireland.
13
Certificate Number: 0050 - CPR - 0171
Notified Body: NSAI, +2 System.
EN 771-3:2011

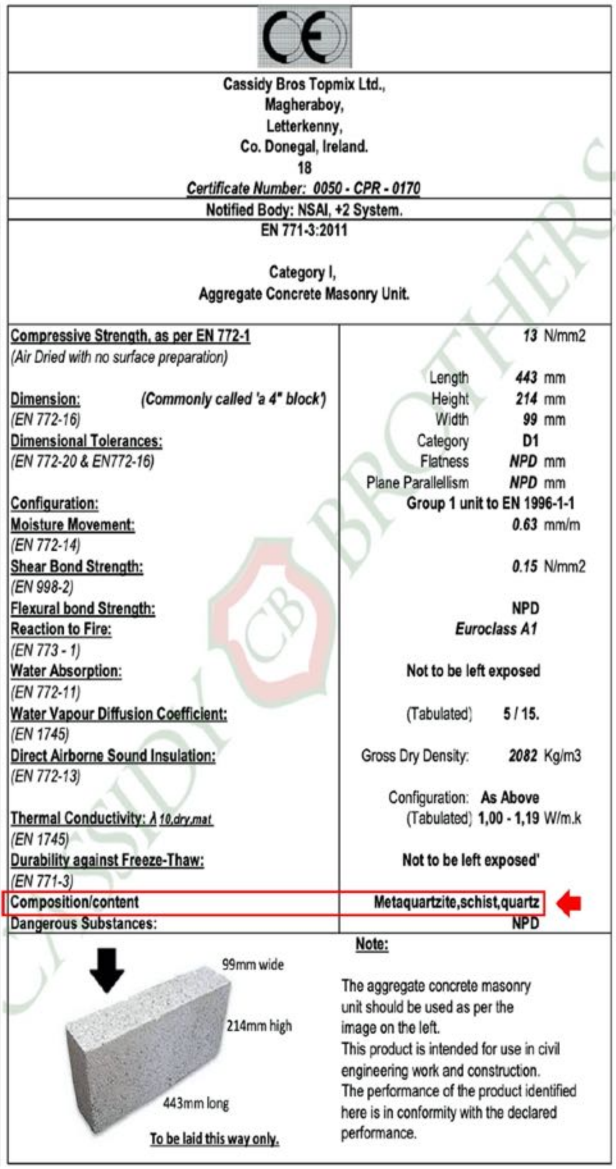
Category I,
Aggregate Concrete Masonry Unit.

Compressive Strength, as per EN 772-1 *(Air Dried with no surface preparation)*	*7.5* N/mm2
Dimension: *(Commonly called 'a 4" block')* *(EN 772-16)*	Length *445* mm Height *215* mm Width *100* mm
Dimensional Tolerances: *(EN 772-20 & EN772-16)*	Category D1 Flatness *NPD* mm Plane Parallelism *NPD* mm
Configuration:	Group 1 unit to EN 1996-1-1
Moisture Movement: *(EN 772-14)*	*0.5* mm/m
Shear Bond Strength: *(EN 998-2)*	*0.15* N/mm2
Flexural bond Strength:	NPD
Reaction to Fire: *(EN 773 - 1)*	*Euroclass A1*
Water Absorption: *(EN 772-11)*	Not to be left exposed'
Water Vapour Diffusion Coefficient: *(EN 1745)*	(Tabulated) 5 / 15.
Direct Airborne Sound Insulation: *(EN 772-13)*	Gross Dry Density: *2054* Kg/m3 Configuration: As Above
Thermal Conductivity: λ 10,dry,mat *(EN 1745)*	(Tabulated) 1,00 - 1,19 W/m.k
Durability against Freeze-Thaw: *(EN 771-3)*	Not to be left exposed'
Composition/content	***Metaquartzite***
Dangerous Substances:	NPD

100mm wide
215mm high
445mm long
To be laid this way only.

Note:

The aggregate concrete masonry unit should be used as per the image on the left.
This product is intended for use in civil engineering work and construction.
The performance of the product identified here is in conformity with the declared performance.



Cassidy Bros Topmix Ltd.,
Magheraboy,
Letterkenny,
Co. Donegal, Ireland.
18
Certificate Number: 0050 - CPR - 0170
Notified Body: NSAI, +2 System.
EN 771-3:2011

Category I,
Aggregate Concrete Masonry Unit.

Compressive Strength, as per EN 772-1 *(Air Dried with no surface preparation)*	*13* N/mm2
Dimension: *(Commonly called 'a 4" block')* *(EN 772-16)*	Length *443* mm Height *214* mm Width *99* mm
Dimensional Tolerances: *(EN 772-20 & EN772-16)*	Category D1 Flatness *NPD* mm Plane Parallelism *NPD* mm
Configuration:	Group 1 unit to EN 1996-1-1
Moisture Movement: *(EN 772-14)*	*0.63* mm/m
Shear Bond Strength: *(EN 998-2)*	*0.15* N/mm2
Flexural bond Strength:	NPD
Reaction to Fire: *(EN 773 - 1)*	*Euroclass A1*
Water Absorption: *(EN 772-11)*	Not to be left exposed
Water Vapour Diffusion Coefficient: *(EN 1745)*	(Tabulated) 5 / 15.
Direct Airborne Sound Insulation: *(EN 772-13)*	Gross Dry Density: *2082* Kg/m3 Configuration: As Above
Thermal Conductivity: λ 10,dry,mat *(EN 1745)*	(Tabulated) 1,00 - 1,19 W/m.k
Durability against Freeze-Thaw: *(EN 771-3)*	Not to be left exposed'
Composition/content	**Metaquartzite,schist,quartz**
Dangerous Substances:	NPD

99mm wide
214mm high
443mm long
To be laid this way only.

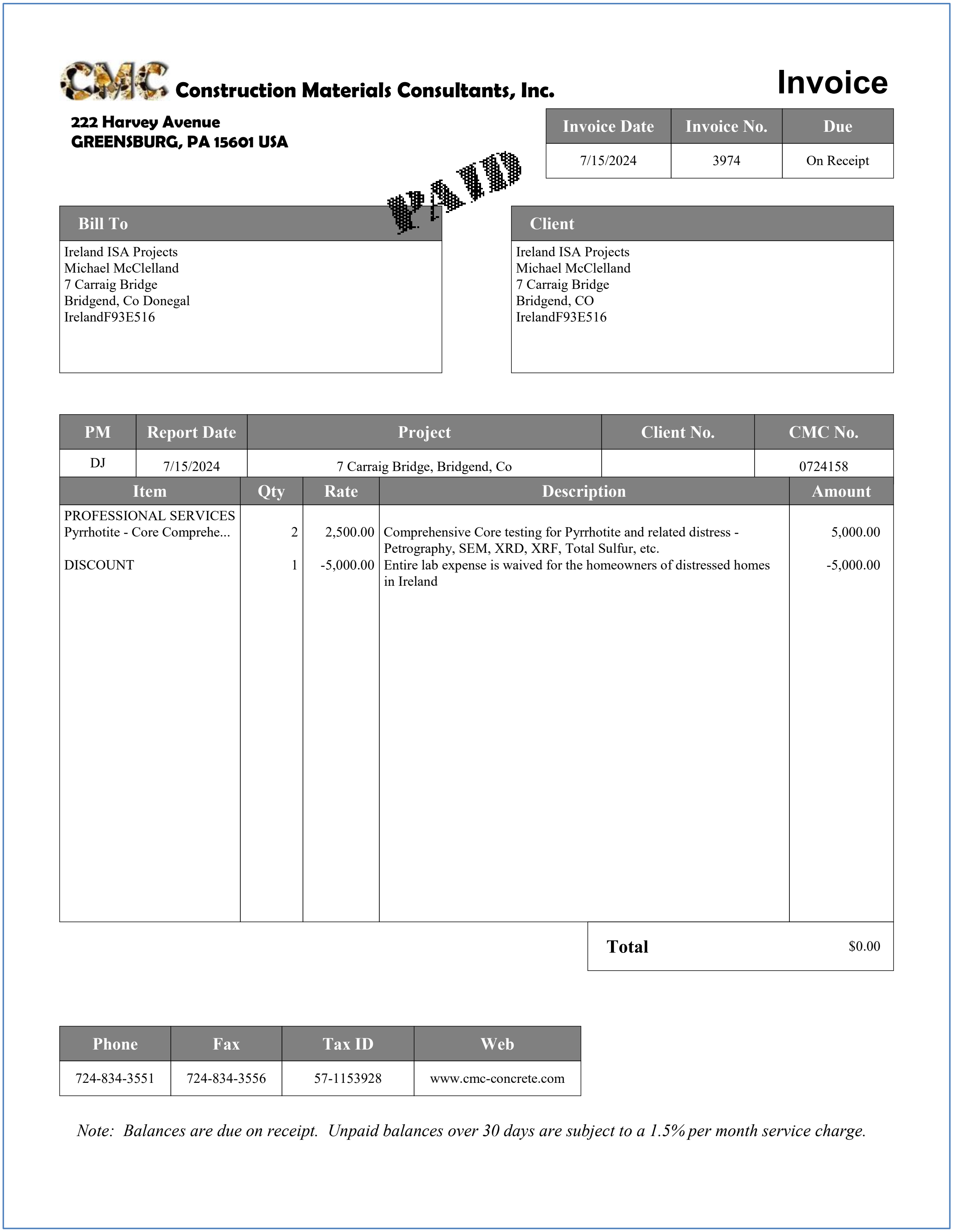
Note:

The aggregate concrete masonry unit should be used as per the image on the left.
This product is intended for use in civil engineering work and construction.
The performance of the product identified here is in conformity with the declared performance.

- Aggregates for the blocks are reportedly derived from the Buncrana Quarry.
- None of the reported specifications for concrete blocks mentions use of **phyllite,** which is the most abundant aggregate used in the blocks in most of the distressed homes in County Donegal.
- Blocks containing **metaquartzite** aggregates would perform better than the ones with phyllite

APPENDIX 3 –

WAIVED INVOICE FOR TESTING



Construction Materials Consultants, Inc.

222 Harvey Avenue
GREENSBURG, PA 15601 USA

Invoice

Invoice Date	Invoice No.	Due
7/15/2024	3974	On Receipt

PAID

Bill To	Client
Ireland ISA Projects Michael McClelland 7 Carraig Bridge Bridgend, Co Donegal IrelandF93E516	Ireland ISA Projects Michael McClelland 7 Carraig Bridge Bridgend, CO IrelandF93E516

PM	Report Date	Project	Client No.	CMC No.
DJ	7/15/2024	7 Carraig Bridge, Bridgend, Co		0724158

Item	Qty	Rate	Description	Amount
PROFESSIONAL SERVICES				
Pyrrhotite - Core Comprehe...	2	2,500.00	Comprehensive Core testing for Pyrrhotite and related distress - Petrography, SEM, XRD, XRF, Total Sulfur, etc.	5,000.00
DISCOUNT	1	-5,000.00	Entire lab expense is waived for the homeowners of distressed homes in Ireland	-5,000.00

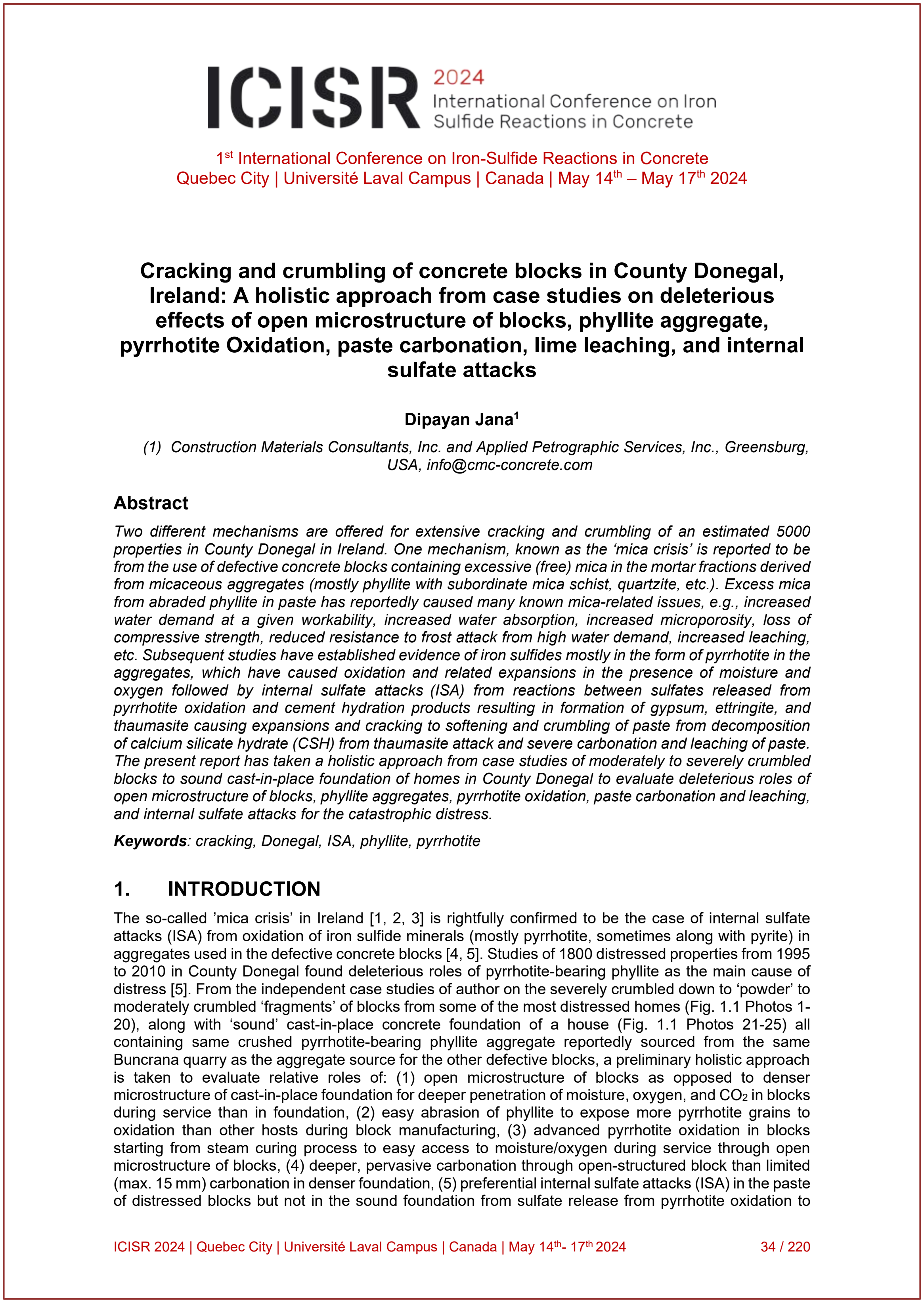
Total	$0.00

Phone	Fax	Tax ID	Web
724-834-3551	724-834-3556	57-1153928	www.cmc-concrete.com

Note: Balances are due on receipt. Unpaid balances over 30 days are subject to a 1.5% per month service charge.

APPENDIX 4 –

AUTHOR'S LATEST PUBLICATIONS



ICISR 2024
International Conference on Iron Sulfide Reactions in Concrete

1st International Conference on Iron-Sulfide Reactions in Concrete
Quebec City | Université Laval Campus | Canada | May 14th – May 17th 2024

Cracking and crumbling of concrete blocks in County Donegal, Ireland: A holistic approach from case studies on deleterious effects of open microstructure of blocks, phyllite aggregate, pyrrhotite Oxidation, paste carbonation, lime leaching, and internal sulfate attacks

Dipayan Jana[1]

(1) Construction Materials Consultants, Inc. and Applied Petrographic Services, Inc., Greensburg, USA, info@cmc-concrete.com

Abstract

Two different mechanisms are offered for extensive cracking and crumbling of an estimated 5000 properties in County Donegal in Ireland. One mechanism, known as the 'mica crisis' is reported to be from the use of defective concrete blocks containing excessive (free) mica in the mortar fractions derived from micaceous aggregates (mostly phyllite with subordinate mica schist, quartzite, etc.). Excess mica from abraded phyllite in paste has reportedly caused many known mica-related issues, e.g., increased water demand at a given workability, increased water absorption, increased microporosity, loss of compressive strength, reduced resistance to frost attack from high water demand, increased leaching, etc. Subsequent studies have established evidence of iron sulfides mostly in the form of pyrrhotite in the aggregates, which have caused oxidation and related expansions in the presence of moisture and oxygen followed by internal sulfate attacks (ISA) from reactions between sulfates released from pyrrhotite oxidation and cement hydration products resulting in formation of gypsum, ettringite, and thaumasite causing expansions and cracking to softening and crumbling of paste from decomposition of calcium silicate hydrate (CSH) from thaumasite attack and severe carbonation and leaching of paste. The present report has taken a holistic approach from case studies of moderately to severely crumbled blocks to sound cast-in-place foundation of homes in County Donegal to evaluate deleterious roles of open microstructure of blocks, phyllite aggregates, pyrrhotite oxidation, paste carbonation and leaching, and internal sulfate attacks for the catastrophic distress.

***Keywords**: cracking, Donegal, ISA, phyllite, pyrrhotite*

1. INTRODUCTION

The so-called 'mica crisis' in Ireland [1, 2, 3] is rightfully confirmed to be the case of internal sulfate attacks (ISA) from oxidation of iron sulfide minerals (mostly pyrrhotite, sometimes along with pyrite) in aggregates used in the defective concrete blocks [4, 5]. Studies of 1800 distressed properties from 1995 to 2010 in County Donegal found deleterious roles of pyrrhotite-bearing phyllite as the main cause of distress [5]. From the independent case studies of author on the severely crumbled down to 'powder' to moderately crumbled 'fragments' of blocks from some of the most distressed homes (Fig. 1.1 Photos 1-20), along with 'sound' cast-in-place concrete foundation of a house (Fig. 1.1 Photos 21-25) all containing same crushed pyrrhotite-bearing phyllite aggregate reportedly sourced from the same Buncrana quarry as the aggregate source for the other defective blocks, a preliminary holistic approach is taken to evaluate relative roles of: (1) open microstructure of blocks as opposed to denser microstructure of cast-in-place foundation for deeper penetration of moisture, oxygen, and CO_2 in blocks during service than in foundation, (2) easy abrasion of phyllite to expose more pyrrhotite grains to oxidation than other hosts during block manufacturing, (3) advanced pyrrhotite oxidation in blocks starting from steam curing process to easy access to moisture/oxygen during service through open microstructure of blocks, (4) deeper, pervasive carbonation through open-structured block than limited (max. 15 mm) carbonation in denser foundation, (5) preferential internal sulfate attacks (ISA) in the paste of distressed blocks but not in the sound foundation from sulfate release from pyrrhotite oxidation to

cause (5a) deleterious ettringite formation (E-ISA) in the denser confined spaces of less/non-carbonated regions to cause expansions and cracking to (5b) deleterious thaumasite formation (T-ISA) in the porous, moderately carbonated regions to cause decomposition of calcium silicate hydrate (CSH) with softening and disintegration to (5c) gypsum form of ISA (G-ISA) in severely carbonated paste by replacing calcium carbonate, and (6) leaching of lime and products of ISA from continued moisture circulation throughout the open-structured block during service to re-precipitate lime and ISA products in voids and turn the paste into a silica-alumina-magnesia-based gelatinous mass with little or no cementitious property, which was already started with T-ISA to cause catastrophic crumbling of blocks across County Donegal.

1: Cracking and crumbling of one of the most distressed homes in County Donegal from where fragment shown in '2' came.
2: Soft, porous, carbonated render on a defective block having abundant interstitial voids and white secondary deposits in voids.
3: Abundant phyllite flakes (marked red) in block.
4: Severely crumbled block from another house.
5: Oxidation products of pyrrhotite caused splitting of phyllite.
6: Fluorescent dye-mixed whole slide thin section of block in PPL/XPL showing abundant pyrrhotite-bearing phyllite in porous carbonated paste.
7: Microcracks in phyllite along foliation planes and crenulation cleavages for moisture access.
8: Radial cracking from pyrrhotite in phyllite.
9: Cracking in an oxidized pyrrhotite and associated cracking in host phyllite.
10: Abundant abraded phyllite in mortar fraction - the origin of 'excess mica' hypothesis.
11: Severe carbonation of paste.
12: Pyrrhotite mostly along foliation plane in phyllite.
13: Pyrrhotite along biotite flakes in mica schist.
14: Severe microcracking in mortar fraction of defective blocks.
15: Thaumasite along aggregate-paste interface.
16: Thaumasite filling an air void.
17: Characteristic iron oxide bands in oxidized pyrrhotite.
18: Abundant residual Portland cement particles in porous, carbonated, inadequately cured render in defective blocks.
19: SEM-EDS analyses of pyrrhotite, pyrite, and oxidized products in S/Fe vs O/Fe atomic ratio plots.
20: SEM-EDS analyses of compositional variations of paste and various secondary deposits in S/Ca vs. Al/Ca plots.
21: Sound cast-in-place concrete foundation containing abundant pyrite and pyrrhotite-bearing phyllite and mica schist.
22. Fine, disseminated iron sulfide grains in phyllite in foundation concrete.
23: Interlocked pyrrhotite and pyrite in phyllite in foundation concrete.
24: Oxidized pyrrhotite in mortar fraction.
25: Abundant phyllite aggregates in foundation concrete with iron sulfide grains. Interior paste is non-carbonated.

Figure 1.1: A microstructural collage of defective concrete blocks (Photos 1 to 20) and sound cast-in-place foundation (Photos 21 to 25) from County Donegal, Ireland.

2. METHODOLOGIES

All samples were examined by: (1) polarized-light microscopy (reflected-light microscopy of polished thin and solid sections, transmitted-light microscopy of polished thin sections) for iron sulfide minerals, oxidation products, aggregates' mineralogies/textures, paste compositions/microstructures/alterations, and secondary deposits, (2) SEM-EDS studies of polished thin/solid sections for further characterization of phases detected in optical microscopy, (3) XRD studies of bulk samples and extracted aggregates for detection and quantifications of iron sulfide minerals, aggregate mineralogies, and ISA products, (4) XRF and combustion IR for major element oxide compositions and total sulfur content (S_T), (5) thermal analysis for sulfate/carbonate phases, and (6) ion chromatography of filtrates of deionized water-digested samples for water-soluble sulfate contents. Details can be found in Jana [6].

3. RESULTS

Crumbling in one of the most distressed homes in County Donegal (Fig. 1.1 Photo 1) examined by author was the result of multiple episodes that started with the use of defective concrete blocks having: (1) abundant easily splitable phyllite aggregates (as high as 80% by volume of coarse aggregate, Fig.

1.1 Photos 2, 6), along with (2) overall porous, high water-cement ratio (*w/c*), and low cement content (4% [3]) nature of blocks leaving a lot of interstitial spaces for migration of moisture, oxygen and CO_2 through the renders to the blocks especially at the outer leaf to cause pyrrhotite oxidation, paste carbonation, ISA, and leaching/re-precipitation.

Phyllite occurred as low-grade, fine-grained metamorphic rock of Precambrian Dalradian Supergroup consisting of major amounts of interlayered muscovite, chlorite and subordinate amounts of very fine-grained silty quartz and alkali feldspar all in parallel layered arrangements to form the typical foliation or sheet-like flakes and characteristic crenulation cleavages of deformed flakes (Fig. 1.1 Photos 10-12). Weak zones along foliation planes and crenulation cleavages provided crisscross intersecting pathways for migration of moisture and oxygen to pyrrhotite (Fig. 1.1 Photo 7). Muscovite and chloride are non-swellable sheet silicates but their presence as easily breakable foliated sheets in phyllite along with their inherent finer grain size than schist have contaminated the mortar fractions of blocks with abraded phyllite flakes (>10% to as high as 50%, by paste volume) to increase the water demand at a given workability and reduce the compressive strength. After phyllite, coarser-grained mica schist is detected at subordinate (15-20%) amount consisting of parallel alignments of muscovite mica, biotite, chlorite, deformed quartz, and alkali feldspar grains (Fig. 1.1 Photo 13). Only a minor (1-5%) amount of noticeably denser and non-foliated coarse-grained metaquartzite aggregate is found, which, if were used as the primary aggregate would have drastically reduced the observed disintegration of blocks let alone to expose pyrrhotite grains to oxidize.

Abundant pyrrhotite grains in phyllite (sizes from < 1 mm at long to <0.5 mm at short directions, Fig. 1.1 Photos 6-9) have acted as the effective sites for expansion from oxidation during service especially at the moist and alkaline (pH >10, less to non-carbonated) conditions of blocks. Some prior oxidations, however, may have occurred in geologic formation and during storage in the aggregate stockpile. Pyrrhotite grains are detected mostly in phyllite as opposed to mica schist or metaquartzite as (a) fine-grained isolated disseminated irregular-shaped to subhedral equant grains (Fig. 1.1 Photo 7) to (b) mostly elongated grains aligned along the dominant foliation planes (Fig. 1.1 Photo 12), which shows extensive reddish-brown oxidation products (e.g., goethite, ferrihydrite, Fig. 1.1 Photo 5) causing further cracking, splitting, and disintegration of phyllite. In situ oxidation of pyrrhotite has caused further splitting and crumbling of phyllite (Fig. 1.1 Photos 8,9) especially since most pyrrhotite grains have crystallized along the prevailing foliation planes where the inherent phyllitic texture of foliation and deformed (crenulation) cleavage planes provided pathways for ready migration of moisture to facilitate oxidation. Additionally, spectacular radial cracks are often seen from oxidized pyrrhotite grains in phyllite (Fig. 1.1 Photos 8, 9) as a testament of *in situ* expansion from pyrrhotite oxidation. Reddish-brown oxidized pyrrhotite products are preferentially noticed between the greenish sheened (from chlorite) foliated sheets where expansive oxidation reactions have further disintegrated the phyllite. Pyrrhotite contents in phyllite aggregates varied from 0.75 to 1.5 percent, by volume. Pyrrhotite is associated with pyrite though no evidence of oxidation of pyrite, or its subsequent sulfate attack (as found in the neighboring County Mayo from framboidal pyrite) was found in the present study.

S_T varied from 0.5 to 1% by mass in distressed blocks and sound foundation depending on the pyrrhotite and pyrite contents, which after converting to aggregate sulfide contents for appropriate aggregate contents are a factor of 5 to 10 times higher than the maximum allowable sulfur content of 0.1% as per EN 12620 specification. At least for the foundation, a high S_T did not necessarily transform to high risk, which amongst various reasons is due to the abundance of non-reactive (blocky) form of pyrite, which has contributed to the lion's share of S_T, and non-reactive finely disseminated form of pyrrhotite.

Fig. 1.1 Photos 19 and 20 show SEM-EDS analysis of compositional variations of pyrrhotite with oxidation products, and paste with secondary deposits, respectively. XRD studies showed both pyrite and pyrrhotite in blocks and foundation but higher amounts of pyrrhotite in blocks leading to higher risk along with indication of potential variations in pyrrhotite contents across the quarry.

Along with aggregate disintegration, mortar fractions of blocks (Fig. 1.1 Photos 10, 11, 14) became the junkyard of (1) abundant abraded fractions of phyllite, (2) oxidized pyrrhotite grains, (3) products of carbonation, (4) lime-poor silica-alumina-magnesia gel from carbonation to decalcification (magnesia was from chlorite decomposition), (5) scattered products of E/T/G-forms of ISA either in least leached/carbonated paste, or as (6) innocuous secondary deposits lining air voids and pores from dissolution and reprecipitation in the decalcified regions of paste. All these products are initially characterized from optical microscopy but best diagnosed during subsequent SEM-EDS studies.

Since pyrrhotite-bearing phyllite occupied much larger volumes (80%) of blocks than the mortar fractions (<10%), distress from aggregate disintegration by pyrrhotite oxidation contributed to the larger part of distress at the initial stage to cause initial cracking, which was followed by subsequent disintegration of the lower volume mortar fraction mostly from carbonation, ISA, and leaching to cause eventual softening and crumbling of blocks.

For the sample from cast-in-place foundation, nonreactive pyrite is more abundant than pyrrhotite, where the former contributed to the lion's share of S_T and the latter mostly occurred as nonreactive fine disseminated grains in phyllite (Fig. 1.1 Photo 22) instead of along foliations, and lesser in the mortar fractions where it shows some oxidation (Fig. 1.1 Photo 24), which is consistent with lesser amount of abraded phyllite in the mortar fraction (Fig. 1.1 Photo 25) than found in defective blocks. Characteristic 'striated' appearance of oxidized pyrrhotite grains having iron oxide bands in iron sulfide body so common in the blocks (Fig. 1.1 Photos 9, 17) are virtually absent in the foundation (Fig. 1.1 Photo 23). Carbonation was restricted to the top 15 mm mostly in a porous cementitious coating on concrete, where secondary ettringite is found in voids. Evidence of E-ISA, T-ISA, or G-ISA are not found in the interior concrete, nor any cracking, which are due to the inherent denser microstructure of concrete along with the absence of moisture during service. Cases from Canada and US, however, showed that denser microstructure alone cannot prevent the distress if foundation were exposed to moisture during service.

4. DISCUSSIONS

Sulfates released from pyrrhotite oxidation as sulfuric acid create an ideal scenario for ISA from deleterious formation ettringite (E-ISA) in the confined spaces in paste from reactions with monosulfates and associated expansions and cracking. E-ISA, however, is more common in the denser and less carbonated concrete, e.g., as seen in numerous residential concrete foundations in the eastern Connecticut [6,7] than in the defective concrete blocks that are inherently porous with a high void content (for use of a low cement content mix), and a porous, high *w/c* paste (for the high-water demand of abraded phyllite). Extent of distress from pyrrhotite oxidation and subsequent forms of ISA are essentially controlled by the host rock for pyrrhotite and whether the distress is occurring in a dense concrete microstructure (e.g., cases in eastern Connecticut or Canada [6,7,9]), or in an inherently porous microstructure of concrete block as in the present case.

In the distressed blocks, sulfates released from pyrrhotite oxidation did cause E-ISA but mostly in the moist condition in initial non-carbonated low-volume porous paste, and in intergranular void spaces and air voids as the first form of ISA, whose remains became limited as the porous paste became dried and carbonated when thaumasite-form of ISA (T-ISA) became evident especially during the cold weather conditions (at 5 to 15°C). Localized occurrence of gypsum is reported in the severely carbonated paste by reaction with calcite as the final form of ISA (G-ISA) at the high sulfate areas [4]. Though not detected in the presently examined block fragments probably for advanced dissolution, but G-ISA is found in the study of other distressed blocks by Leemann et al. [4] and is, indeed, very common in many other cases in Ireland, e.g., causing cracking of defective blocks in County Mayo containing framboidal pyrite in limestone aggregates [1] or devastating building heaves in Dublin constructed on carbonate mudstone fill containing framboidal pyrite where reactions between sulfates released from pyrite oxidation and calcite in the host rock have caused expansive formation of gypsum [8].

High intergranular void contents and low volume, porous, high *w/c* paste of blocks have caused effective carbonation of paste especially when relative humidity was in the range of 60-80% to convert original calcium hydroxide and CSH components to fine-grained calcite and hydrated silica-alumina gel thereby providing the seeds for T-ISA. Carbonation occurred more effectively in the relatively drier inner leaf and during intermittent dry periods in the outer leaf, which itself at the advanced stage can cause severe loss of strength from decomposition of CSH. Deleterious effects of carbonation-induced T-ISA, however, are more pronounced at the early stages of carbonation and in the least decalcified regions. Severe carbonation (pH <10) and lime leaching to the point of silica-alumina-gel formation from original CSH either prevent T-ISA or restrict secondary thaumasite formation mostly in the porous regions and as linings in air voids and cracks simply from dissolution, redistribution, and reprecipitation of available dissolved sulfate and carbonate ions – as seen in the severely disintegrated blocks. Such three-stage alterations of CSH, i.e., first by carbonation, then by subsequent T-ISA, and finally by severe leaching/decalcification has caused severe disintegration and crumbling of blocks. T-ISA, however, is more pronounced in the distressed blocks than in the foundation sample (as also not seen in distressed foundations in eastern Connecticut, USA [6,7]), which is directly related to much denser microstructure of foundation concrete to prevent severe carbonation (or any other source of carbonates). Carbonates released from paste carbonation during drier periods (at 60-80% relative humidity) along with sulfates released from pyrrhotite oxidation during wet periods have caused T-ISA preferentially in the outer leaf to cause its higher extent of damage than the inner leaf.

Distresses from E/T-ISA, however, are less evident in the severely carbonated, porous, silica-alumina-rich gelatinous regions of paste where products of ISA are mostly found as secondary deposits in coarse voids (as interlayered secondary ettringite and thaumasite fibers, Fig. 1.1 Photo 16) from dissolution and re-participation, whereas in the least de-calcified regions E/T-ISA products are often found mixed within the paste to implant seeds for potential expansions. Distresses from E/T-ISA, however, are inherently restricted for the low (<10%) paste volumes of blocks and even more so only in the moderately carbonated and less decalcified paste regions. The outcome, however, is severe crumbling of blocks.

Abundant pyrrhotite provided the starting ingredients for ISA, foliated nature of phyllite provided ideal pathways for entry of moisture and oxygen to release sulfates for E/T-ISA, porous microstructure of blocks provided pervasive carbonation and released carbonates and silica gel for T- ISA, but the extent of distress from E/T-ISA are controlled by the abundance of pyrrhotite (for sulfates) in the phyllite host, paste volume, and the degree of leaching and carbonation (for carbonate and silica source for thaumasite). Moist conditions in the outer leaf promoted pyrrhotite oxidation and sulfate release to cause E-ISA distress from expansive ettringite formation in the noncarbonated paste leading to initial cracking, whereas subsequent T-ISA at moderately carbonated paste at colder and relatively drier conditions mostly contributed to crumbling. Relatively drier conditions in the inner leaf promoted advanced carbonation but intermittent drier conditions between moist seasons in the outer left promoted cyclic formation of E-ISA during moist conditions to T-ISA during intermitted carbonation at drier and colder conditions. The end results of carbonation and T-ISA are a paste of little or no cementitious property eventually causing mass-scale crumbling of blocks. Effects of pyrrhotite oxidation are more towards initial cracking and crumbling of blocks, whereas that of carbonation and T-ISA are towards eventual decomposition of paste to cause large-scale crumbling of blocks. Not one single factor alone has had the lion's share for catastrophic failure of houses – as many as six different factors are discussed here, which have worked together to cause the progressive damage. *Pyrrhotite was the undoubted 'cancer cell' in the blocks whose deleterious effect was initiated with moisture and spread rapidly through interactions of other five factors mentioned in the title and discussed here*. *It is time to take a holistic approach in Ireland instead of polarized views of wrongfully advertised mica-only to rightfully determined ISA-only theories*.

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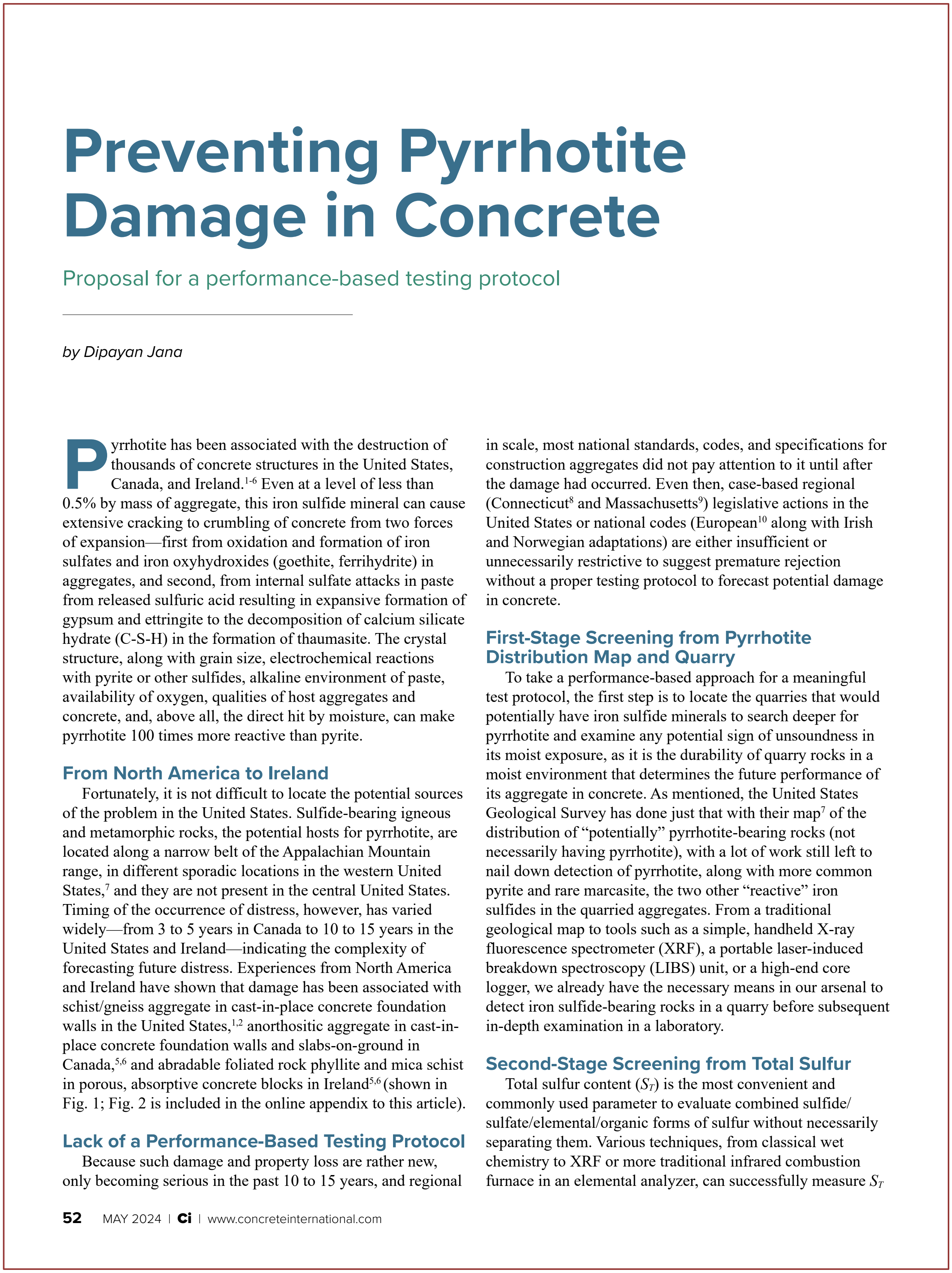


Preventing Pyrrhotite Damage in Concrete

Proposal for a performance-based testing protocol

by Dipayan Jana

Pyrrhotite has been associated with the destruction of thousands of concrete structures in the United States, Canada, and Ireland.[1-6] Even at a level of less than 0.5% by mass of aggregate, this iron sulfide mineral can cause extensive cracking to crumbling of concrete from two forces of expansion—first from oxidation and formation of iron sulfates and iron oxyhydroxides (goethite, ferrihydrite) in aggregates, and second, from internal sulfate attacks in paste from released sulfuric acid resulting in expansive formation of gypsum and ettringite to the decomposition of calcium silicate hydrate (C-S-H) in the formation of thaumasite. The crystal structure, along with grain size, electrochemical reactions with pyrite or other sulfides, alkaline environment of paste, availability of oxygen, qualities of host aggregates and concrete, and, above all, the direct hit by moisture, can make pyrrhotite 100 times more reactive than pyrite.

From North America to Ireland

Fortunately, it is not difficult to locate the potential sources of the problem in the United States. Sulfide-bearing igneous and metamorphic rocks, the potential hosts for pyrrhotite, are located along a narrow belt of the Appalachian Mountain range, in different sporadic locations in the western United States,[7] and they are not present in the central United States. Timing of the occurrence of distress, however, has varied widely—from 3 to 5 years in Canada to 10 to 15 years in the United States and Ireland—indicating the complexity of forecasting future distress. Experiences from North America and Ireland have shown that damage has been associated with schist/gneiss aggregate in cast-in-place concrete foundation walls in the United States,[1,2] anorthositic aggregate in cast-in-place concrete foundation walls and slabs-on-ground in Canada,[5,6] and abradable foliated rock phyllite and mica schist in porous, absorptive concrete blocks in Ireland[5,6] (shown in Fig. 1; Fig. 2 is included in the online appendix to this article).

Lack of a Performance-Based Testing Protocol

Because such damage and property loss are rather new, only becoming serious in the past 10 to 15 years, and regional in scale, most national standards, codes, and specifications for construction aggregates did not pay attention to it until after the damage had occurred. Even then, case-based regional (Connecticut[8] and Massachusetts[9]) legislative actions in the United States or national codes (European[10] along with Irish and Norwegian adaptations) are either insufficient or unnecessarily restrictive to suggest premature rejection without a proper testing protocol to forecast potential damage in concrete.

First-Stage Screening from Pyrrhotite Distribution Map and Quarry

To take a performance-based approach for a meaningful test protocol, the first step is to locate the quarries that would potentially have iron sulfide minerals to search deeper for pyrrhotite and examine any potential sign of unsoundness in its moist exposure, as it is the durability of quarry rocks in a moist environment that determines the future performance of its aggregate in concrete. As mentioned, the United States Geological Survey has done just that with their map[7] of the distribution of "potentially" pyrrhotite-bearing rocks (not necessarily having pyrrhotite), with a lot of work still left to nail down detection of pyrrhotite, along with more common pyrite and rare marcasite, the two other "reactive" iron sulfides in the quarried aggregates. From a traditional geological map to tools such as a simple, handheld X-ray fluorescence spectrometer (XRF), a portable laser-induced breakdown spectroscopy (LIBS) unit, or a high-end core logger, we already have the necessary means in our arsenal to detect iron sulfide-bearing rocks in a quarry before subsequent in-depth examination in a laboratory.

Second-Stage Screening from Total Sulfur

Total sulfur content (S_T) is the most convenient and commonly used parameter to evaluate combined sulfide/sulfate/elemental/organic forms of sulfur without necessarily separating them. Various techniques, from classical wet chemistry to XRF or more traditional infrared combustion furnace in an elemental analyzer, can successfully measure S_T

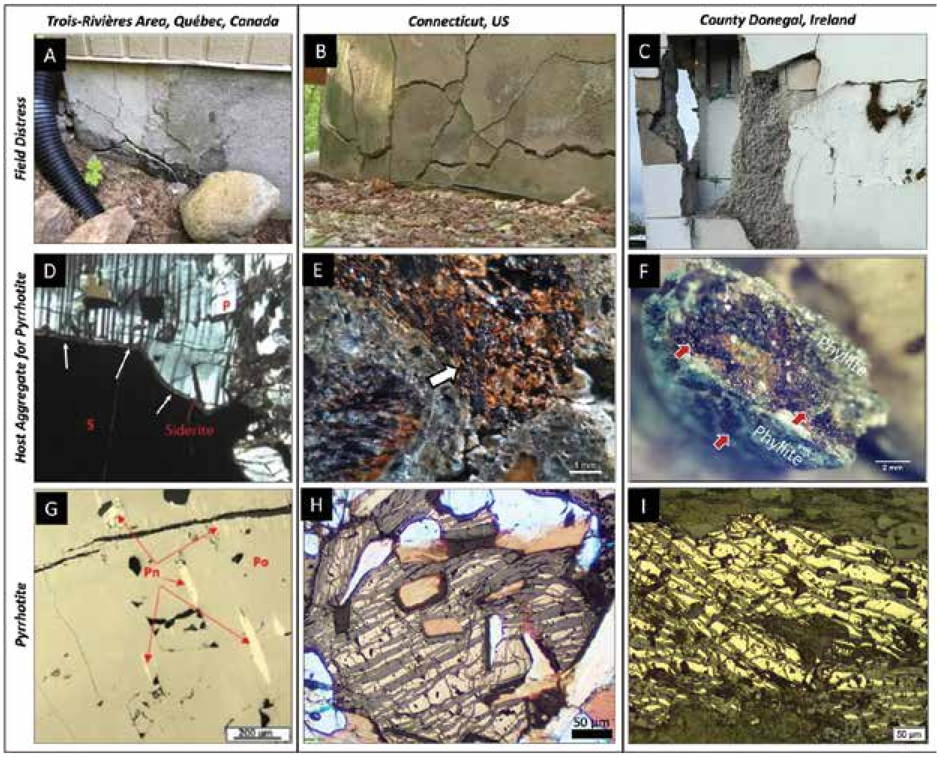


Fig. 1: Pyrrhotite distress (top row) in Canada (left), United States (center), and Ireland (right), host aggregates (middle row) and pyrrhotite (bottom row): (a) major cracks in a residential foundation wall are mostly at the corner next to rain downspouts, indicating role of water in distress; (b) extensive cracking and crumbling of a residential foundation wall in Connecticut; (c) an extreme case of crumbling of a house in Ireland; (d) anorthosite host for pyrrhotite, where an iron carbonate (siderite) rim (arrows) around sulfide promoted thaumasite attack in concrete; (e) rust stain from oxidized pyrrhotite and cracking of host garnetiferous quartzo-feldspathic schist and gneiss from Brimfield Schist formation in Connecticut; (f) similar rust stains and cracking/crumbling of foliated muscovite-chlorite-quartz-based phyllite aggregate in distressed concrete block in Ireland; (g) main pyrrhotite (Po) and other minor pentlandite (Pn) phases in Canada; and (h) and (i) characteristic bands of oxidized iron in pyrrhotite in Connecticut and Ireland. Left-column photos are reproduced with permission from Nova Science Publishers from Duchesne at al.[4]

for screening—depending on $S_T < 0.1\%$, between 0.1% and 1.0%, and > 1.0% by mass of aggregate traditionally used in most European,[10] Canadian,[11] Irish, Norwegian, and U.S. codes[8,9] as parameters for accepting, further testing, or rejecting aggregate, respectively, for potential pyrrhotite distress in a moist exposure condition. Stringent reliance on S_T alone, usually tested on less than a gram of sample, can unnecessarily reject aggregates that do not contain pyrrhotite, marcasite, or reactive forms of pyrite (for example, framboidal clusters of pyrite microcrystals) or accept ones that do have those perhaps at low amounts to be registered in a small mass, or missed for known heterogeneous distribution of pyrrhotite in a quarry.

Third-Stage Screening from Petrography, SEM-EDS, XRD, WDXRF, and XPS

This stage is most crucial because it nails down detecting pyrrhotite along with all other potentially unsound iron sulfides. Petrography is the most convenient method[12] because, after all, the issue with pyrrhotite is, first and foremost, an issue with rocks, and petrography can detect pyrrhotite, pyrite, their oxidation products, and resultant sulfate attacks in concrete (Fig. 2 to 4, shown in the online appendix to this article), and thus offer an assessment of the extent of damage in a structure, or in a laboratory sample undergoing performance-based testing.

To enhance petrography, X-ray diffraction (XRD) can

quantify pyrrhotite content down to 0.05% by mass level with the latest silicon drift detectors and high-resolution XRD units with synchrotron radiation. Wavelength dispersive XRF technique (WDXRF) can separate and quantify sulfide and sulfate phases, though it still cannot separate pyrrhotite per se from other sulfides. Micro-XRF technique can quickly detect iron sulfide phases in a cross section of a quarried core or epoxy-encapsulated slab of aggregates from iron and sulfur elemental maps. Due to pyrrhotite's weak magnetism (unlike pyrite), a thermomagnetic test can measure the drop in magnetic susceptibility during heating across its Curie temperature (at 325°C [617°F], where it loses its magnetism) and quantify that drop at different pyrrhotite (or S_T) contents in a calibrated set of standards to detect and quantify pyrrhotite in an unknown aggregate or concrete down to 0.1% by mass level simply from S_T and the susceptibility drop. X-ray photoelectron spectroscopy (XPS) not only detects sulfide and sulfate but actually separates pyrite and pyrrhotite contents. Raman spectroscopy, LIBS, Auger electron spectroscopy, thermal analysis, and nuclear magnetic resonance spectroscopy are now being tested at various research laboratories for pyrrhotite detection. We have more than enough tools to detect and quantify pyrrhotite, though not all are equally accessible or extensively researched to include in a standardized testing protocol.[1]

Assessing Oxidation and Sulfate Attack

Petrography[12] provides the first-hand detailed anatomy of an aggregate (mineralogy/texture) for concrete or of concrete in a distressed structure, including where the pyrrhotite grains are, in which forms and other sulfide associations, their products of oxidation, sulfate attacks, and the extent of damage. To evaluate performance of a pyrrhotite-bearing aggregate in field concrete, laboratory tests must be conducted. We have tests that measure oxygen consumption of aggregates from pyrrhotite oxidation either by directly measuring loss of O_2 in air above pulverized aggregates in a sealed container with an O_2 probe[4] or by indirectly detecting the water-soluble sulfate level in a filtrate by ion chromatography from a pulverized aggregate digested in a highly oxidizing solution like hydrogen peroxide.[2]

Expansion Tests

The last step is to measure potential expansion of some laboratory-made mortar bars or concrete prisms containing aggregates that are confirmed to have pyrrhotite, before rejecting them entirely from use in a moist environment. We have at least four different tests, all of which were originally devised for (or derived from) tests proposed for assessing expansion potential of alkali-silica reactive aggregates, which are not necessarily reactive to pyrrhotite. In the absence of a standardized expansion test for pyrrhotite, these are the best options available, at least to start with. Ranging from as fast as 16 days for the ASTM accelerated mortar bar test (AMBT),[13] to 180 days for the Canadian mortar bar test (CMBT),[14] to 8 to 12 weeks for the AASHTO miniature concrete prism test (MCPT),[15] to as long as 2 years for the ASTM concrete prism test (CPT),[16] potential expansion of pyrrhotite-bearing aggregates can be tested without necessarily paying attention to their imposed threshold expansion limits for acceptance, for example, < 0.10% for AMBT and CMBT and < 0.04% for MCPT and CPT, which were (except CMBT) proposed strictly for two-stage expansions from alkali-silica reaction (ASR) gel formation and moisture absorption of gel during ASR, and not for two-stage expansions from pyrrhotite oxidation and subsequent internal sulfate attack.

Field Lessons to Develop a Performance-Based Protocol

To assess expansion potential of pyrrhotite-bearing aggregates from oxidation and sulfate attack, we must first incorporate the lessons learned from North America and Ireland. There are too many variables and seemingly complex and rather confusing scenarios in field cases that need to be evaluated and implemented in a performance-based expansion test.

Aggregate: Whether the host aggregate is as dense as an igneous rock (anorthosite) in Canada, a deformed metamorphic rock (schist and gneiss) in the eastern United States, or a highly foliated and easily abradable phyllite and mica schist in Ireland, none provided the necessary protection to its pyrrhotite from oxidation. The texture of aggregate, however, did play a role, at least for metamorphic aggregates, such as phyllite in Ireland, in breaking down easily during mixing and preparation of concrete blocks to not only expose more pyrrhotite grains to O_2 and moisture but also enrich pyrrhotite in the mortar fraction for rapid oxidation in an alkaline medium. Foliations or weak planes in phyllite, schist, or gneiss provided easy access of moisture to pyrrhotite, not just in the United States or Ireland but also in Spain, where pyrrhotite preferentially aligned along schistosity planes defined by parallel arrangements of deformed minerals were preferentially oxidized. These aggregates and others similar to them, therefore, require special attention to the role of aggregate texture in a performance-based test.

Concrete mixture: Perhaps one reason for the relatively faster appearance of distress in Canada, despite having a dense anorthosite host for pyrrhotite, is the 0.7 water-cement ratio (*w/c*) of concrete, which is far higher than any industry specification for durability of concrete in a moist outdoor environment. Without bringing other variables for pyrrhotite oxidation, and just comparing cases between the United States and Canada, a *w/c* of 0.7 in Canada versus a *w/c* of 0.45 to 0.50 in the eastern United States can theoretically accelerate pyrrhotite oxidation in Canada. The real situation, however, is not that simple, as can be seen from very different host aggregates. Incorporation of supplementary cementitious materials (SCMs) should restrict moisture/O_2 ingress from pore size and grain size refinements and cement type. For example, ASTM C150 Type IV (low C_3A content and low heat of hydration) or Type V (high sulfate resistance) portland cement should provide additional protection from sulfate attacks. This has already been confirmed from laboratory tests by the Canadian group, where a 50 to 85% reduction in expansion was noted from these beneficial effects of SCMs and sulfate-resistant cement, a necessary preventive measure to be taken when pyrrhotite-bearing aggregate is detected.

Iron sulfide: Pyrrhotite is far more reactive than pyrite, but its reactivity increases when pyrite is also present to engage in electrochemical reactions. Cracked foundations from Canada showed a close association of pyrrhotite with pyrite, pentlandite, and chalcopyrite, which can potentially increase the rate of oxidation of pyrrhotite. Even without other sulfides, oxidation rate of pyrrhotite can vary considerably from crystal structures (% Fe vacancies and % Fe^{3+}), moisture conditions, pH, and oxygen contents. Pyrrhotite's distribution is also quite heterogeneous in a quarry. A thin rim of iron carbonate (siderite) around iron sulfide was enough to cause thaumasite attack in Canada, which is also seen at a larger scale in Ireland, though not from a separate carbonate source but from pervasive carbonation through an open structure of blocks to cause severe crumbling. Therefore, a carbonate source can potentially be detrimental to aggravating the damage from expansive gypsum/ettringite-based to corrosive thaumasite-based sulfate attacks by decomposing C-S-H. Similar to faster oxidation of clustered pyrite microcrystals in framboids than the cubic/blocky ones, pyrrhotites in elongated forms preferentially aligned along weak planes in the host or coarser grains in the mortar fraction are more susceptible to oxidation than scattered finely disseminated ones in aggregates.

Cast-in-place concrete versus manufactured blocks: Cases of cracked foundation walls and slabs in North America and crumbled walls of defective concrete blocks in Ireland are two end scenarios of pyrrhotite distress irrespective of how dense or porous the concrete or the host aggregate is. The inherent porous microstructure of defective blocks in Ireland (with high interstitial void contents along with a low-volume, high *w/c* paste) has undoubtedly caused deeper penetration of moisture, O_2, and CO_2 during service, leading to pervasive oxidation of pyrrhotite grains, gypsum/ettringite-based internal sulfate attack, carbonation of paste with subsequent thaumasite attack, and eventual leaching of lime—where the blocks worsened to the point of crumbling to fine powder and falling from the walls. The inherent denser microstructure of concrete foundations in Connecticut, however, could not combat the damage even though a denser microstructure with SCMs does have a positive influence. The author recently examined a foundation slab from a house located in County Donegal, where crumbled blocks in the walls were to be removed, and the slab was found sound despite finding the same pyrrhotite (plus pyrite)-bearing phyllite aggregate as in the block, reportedly supplied by the same contractor and installed at the same time. Only a few pyrrhotite grains, mostly in the mortar fraction, showed oxidation for exposure to a highly alkaline environment of paste during mixing and service, but pyrrhotite grains within phyllite were mostly unaffected. The mortar fraction of the slab was rich in abraded phyllite flakes from mixing and placement, as also seen, quite ubiquitously, in the defective blocks. The paste, however, was considerably denser in the well-consolidated slab, and it had a higher volume and lower *w/c* than the altered paste in the blocks. Hence, no carbonation of the interior slab was noticed, except at the top exposed 15 to 20 mm (0.6 to 0.8 in.) depth compared to severely carbonated and leached paste in the porous blocks. Clearly, the reported lack of a moist exposure condition of the slab has helped it survive the damage from pyrrhotite. Steam curing of blocks may have accelerated pyrrhotite oxidation at the early stage compared to oxidation in an alkaline medium in the slab.

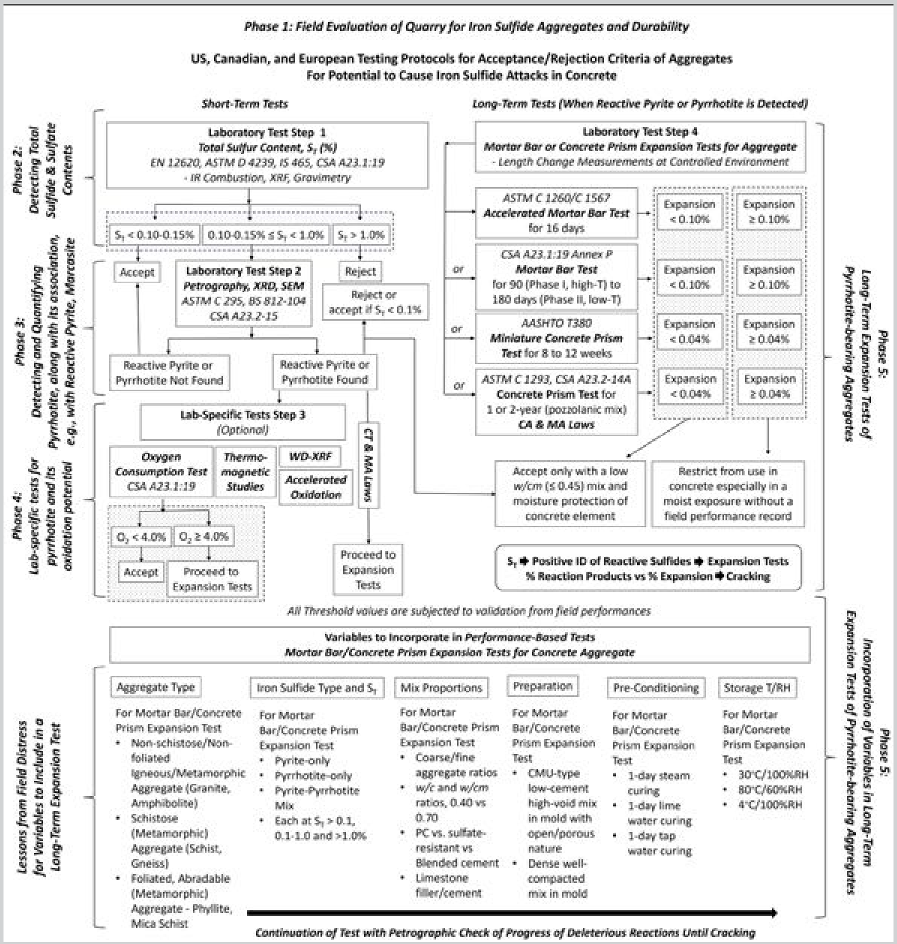
Conclusions

The following factors are important to incorporate in a performance-based test protocol:

- Evaluation of a quarry for its geology, structure, and field durability of potentially sulfide-bearing rock formations after initial screening of drilled cores or quarry beds with handheld XRF/LIBS units for collecting samples for laboratory testing of aggregates;
- Total sulfur contents (S_T) of field-screened sulfur-bearing aggregates can then be tested only for a rough screening of aggregates of low, intermediate, and high S_T, for example, at the conventional < 0.1%, 0.1 to 1.0%, and > 1.0% S_T by mass, respectively, but without necessarily imposing those limits as criteria for acceptance/rejection/further testing, only for the sole purpose of prioritizing the members to test for pyrrhotite;

Workflow of Test Protocols

This chart describes the assessment of aggregate from field evaluation of a quarry (Phase 1) through short-term (within a month) laboratory tests (Phases 2 to 4) to detect pyrrhotite/pyrite to, finally, a long-term (from 2 weeks to 2 years) expansion test (Phase 5) guided by lessons from field distress to mitigate pyrrhotite distress in concrete (modified from Jana[1]). Relevant standardized methods are mentioned. Threshold limits are subjected to validation from long-term tests and field performance. Total sulfur (S_T) is universally suggested by British, Irish, Canadian, and United States agencies to be followed by petrography[12] for aggregates having S_T values between 0.1 and 1.0% to evaluate the presence of reactive pyrite and pyrrhotite (which is best done by a combination of reflected-light optical microscopy of polished sections, often with ancillary XRD and scanning electron microscopy and energy dispersive X-ray spectroscopy [SEM-EDS]). Suggestions from United States and Canadian agencies varied when reactive sulfide was found, where the Canadian standard suggested an intermediate oxygen consumption test before the final long-term expansion tests of mortar bars or concrete prisms. Two long-term expansion tests suggested are CSA A23.1:19 for Canadian aggregates[11] and ASTM C1293/C1293M-20a [17] for U.S. aggregates. Based on field evidence, however, a performance-based expansion test should incorporate variables from aggregate types, individual sulfide types, mixture proportions, porous block versus dense concrete preparation, various pre-conditioning, and storage temperatures and relative humidity.



- Determine the presence of pyrrhotite, along with the detection of pyrite, marcasite, and other sulfide minerals, mostly by petrography, with the option to exercise other lab-specific tests,[1] such as thermomagnetic studies, WDXRF, or oxygen consumption;
- Irrespective of any testing route followed for detection of pyrrhotite, especially along with pyrite at any level, it is enough for a decision of whether to restrict aggregate use only in moderate/mild/dry exposure regimes with adequate moisture protection, entirely reject the aggregate for moist outdoor exposures, or engage in a long-term performance-based expansion test, with appropriate modifications after incorporating all the variables of pyrrhotite distress that are known to affect rate of oxidation of pyrrhotite and resultant distress. Instead of paying attention to the threshold limits for acceptance/rejection originally implemented for ASR-related expansions, emphasis should be given to the time of occurrences of the products of oxidation and internal sulfate attacks in relation to cracking of the tested bars/prisms by regular checks with petrography;
- Such a protocol, which was built upon the conventional scheme along with the incorporation of factors that have effects on pyrrhotite distress in concrete, is shown in the sidebar "Workflow of Test Protocols"; and
- Modification of the concrete mixture to densify the microstructure and make it sulfate resistant and adequate moisture protection of structural elements are two mandatory requirements to mention before the use of aggregates having pyrrhotite at any level.

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Selected for reader interest by the editors.

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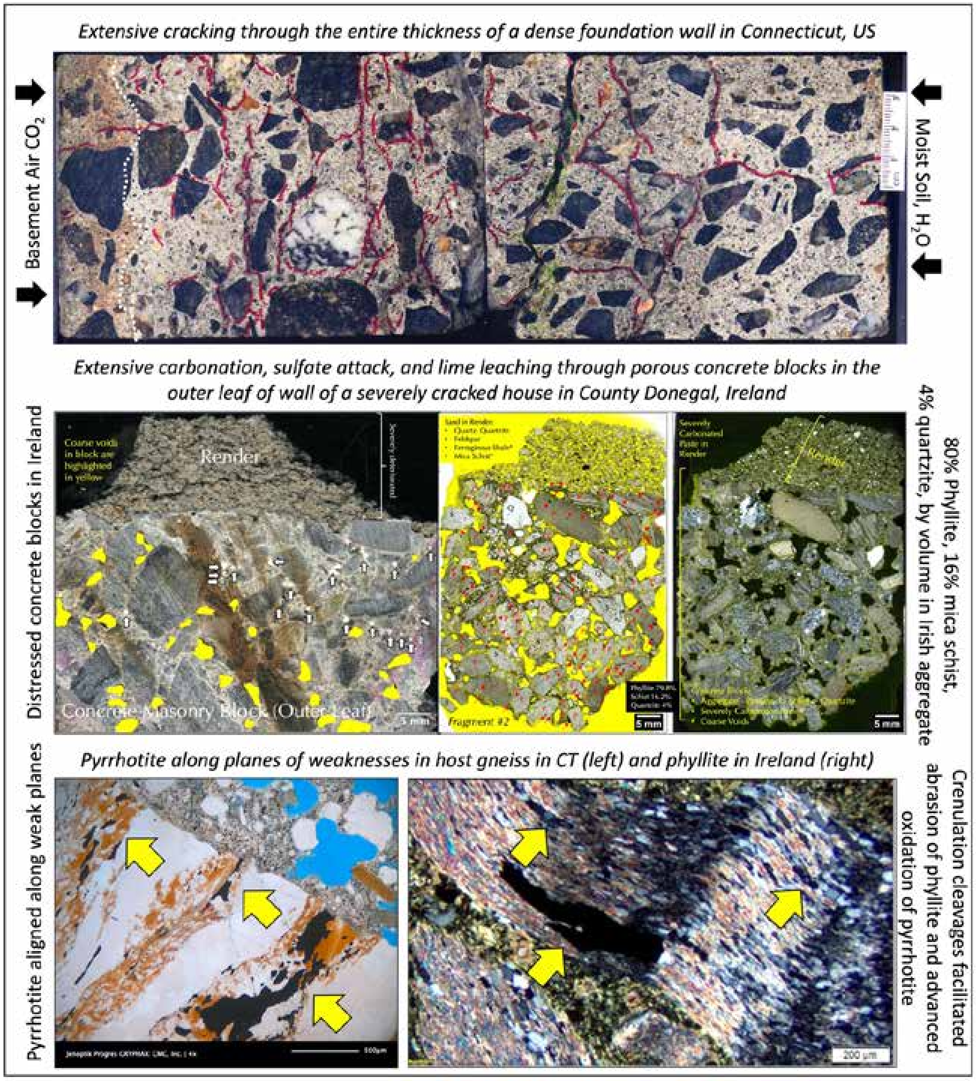

Fig. 2: At the top, extensive cracking through the entire foundation wall in a house in Connecticut shown on lapped cross section of a core, where the wall shows 15 to 20 mm carbonation from the interior surface in contact with basement air, whereas moisture entered from the outer surface from moist soil. In the middle, porous structure of low-volume paste and high interstitial void in defective concrete blocks of a wall in County Donegal, Ireland, that has facilitated migration of moisture and CO_2 for carbonation, pyrrhotite oxidation, and resultant internal sulfate attack. At the bottom, preferential alignment of pyrrhotite along the weak planes of host gneiss in association with biotite flakes in Connecticut (left) and foliation planes of phyllite in Ireland (right)

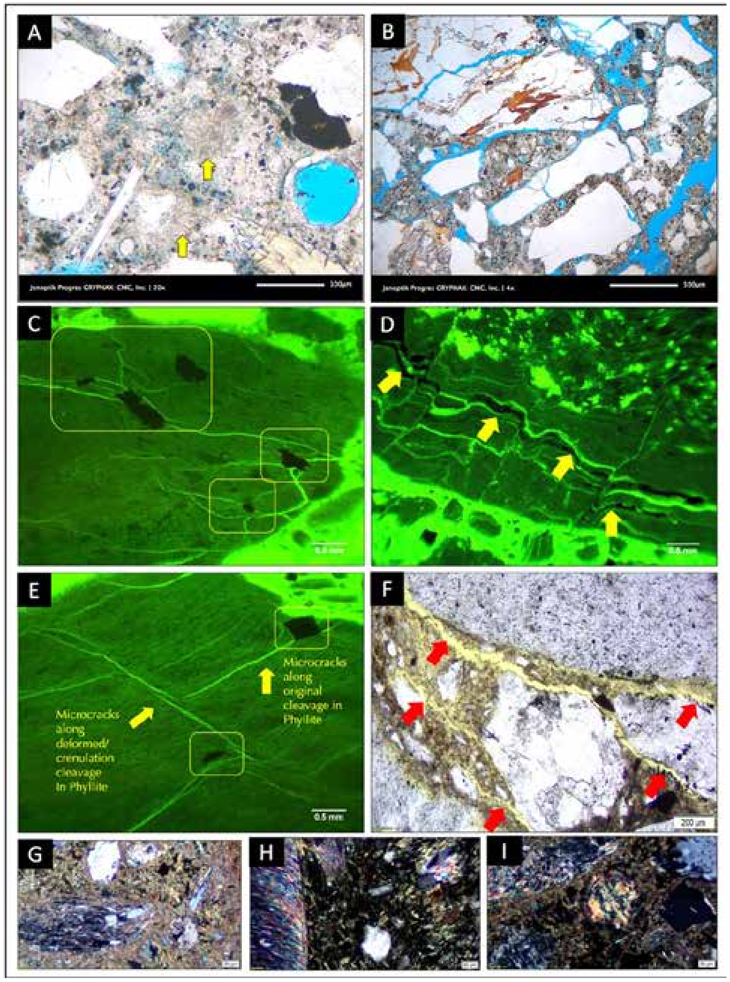

Fig. 3: Petrographic examination findings: (a) ettringite in dense, confined spaces in paste that has resulted in expansion and cracking in Connecticut,[1] whereas secondary ettringite precipitation in an air void at right is the result of dissolution and reprecipitation of ettringite from paste; (b) cracks traversing through pyrrhotite-bearing gneiss coarse aggregate, sand, and paste are highlighted by blue epoxy in a thin section micrograph of distressed concrete in Connecticut[2]; (c) fluorescent epoxy-mixed thin section micrograph of defective block from Donegal, Ireland, showing extensive cracking in phyllite aggregate with many oxidized pyrrhotite grains in phyllite showing radial cracks from expansion associated with oxidation; (d) deformation of pyrrhotite grains aligned along pre-existing foliation planes in phyllite during crenulation cleavage formation that had further enhanced its oxidation rate; (e) microcracks along two sets of cleavage planes in phyllite that has facilitated splitting and enrichment of abraded phyllite flakes in the mortar fraction as well as enhanced oxidation of pyrrhotite both within phyllite and after incorporation in highly alkaline mortar fraction in Ireland; (f) extensive cracking in the mortar fraction of defective blocks in Ireland highlighted by fluorescent epoxy; (g) severe carbonation; (h) lime-leached (Si-Al-Mg rich) portions of paste; and (i) thaumasite formation in air void and paste in thin section micrographs (XPL) of defective concrete blocks from Ireland where mortar fractions in (g) to (i) are enriched in abraded phyllite flakes

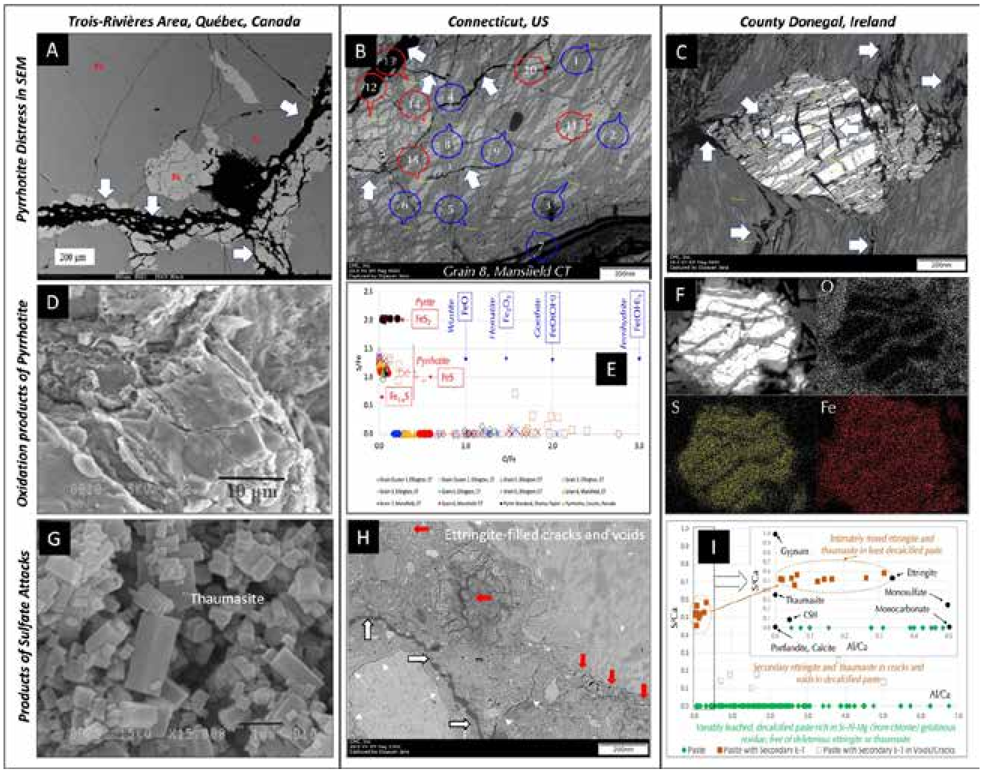

Fig. 4: In the top row, SEM-EDS studies showing cracking (arrows) from pyrrhotite oxidation in: (a) Canada, (b) United States; and (c) Ireland, forming characteristic darker gray iron oxide bands in brighter pyrrhotite in backscatter electron images Also in (c) crumbling of phyllite by pyrrhotite oxidation in Ireland. In the middle row, products of pyrrhotite oxidation: (d) iron oxy-hydroxide in secondary electron image; (e) atomic ratios of oxidized pyrrhotite from Connecticut plotting S/Fe versus O/Fe showing pyrrhotite grains at S/Fe around 1.0 and 1.5 whereas a range of oxidation products encompassing wustite to ferrihydrite; (f) X-ray elemental maps of an oxidized pyrrhotite from Connecticut showing O-rich oxidized bands in Fe,S-rich matrix. In the bottom row, products of internal sulfate attack: (g) euhedral, prismatic crystals of thaumasite from Canada; (h) ettringite formation in paste, along cracks, aggregate-paste interfaces, and within air void in Connecticut[1]; (i) atomic ratios S/Ca versus Al/Ca plots of altered paste from defective blocks in Ireland showing a linear trend of mixed secondary ettringite and thaumasite in the least decalcified but carbonated paste (solid orange squares), ettringite/thaumasite only in voids and cracks in decalcified paste (open orange squares), and Si-Al-Mg (from chlorite)-rich gelatinous residue from lime-leached areas of paste that are free of ettringite or thaumasite (green diamonds). Left column photos are reproduced with permission from Nova Science Publishers from Duchesne at al.[4]

END OF REPORT[1]

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[1] The CMC logo is made using a lapped polished section of a 1930's concrete from an underground tunnel in the U.S. Capitol.