

Pyrrhotite in Concrete Aggregates

Is legislation ahead of science?

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Concrete aggregates produced in two regions in North America have been identified as the cause of deterioration to tens of thousands of residential and light commercial concrete foundations. These concrete foundations have experienced extensive cracking and loss of structural integrity due to an oxidation reaction of an iron sulfide mineral, pyrrhotite (Fig. 1). No methods for mitigating

the reaction or repairing the compromised foundations have been successful. The only effective technique has been to lift the houses off their existing foundations and remove and replace the concrete (Fig. 2).

While pyrrhotite has the potential to cause concrete deterioration in some conditions, the amount of pyrrhotite needed to cause deterioration is not fully understood. Various research groups, trade associations, and standardization committees are working to develop techniques for identifying and quantifying potentially deleterious iron sulfides in concrete aggregates. Due to the small amount necessary to cause deterioration (potentially as low as 0.10% by weight of aggregate), the unique nature of the mineral, the difficulty with detection and quantification, and the complexity of the oxidation reaction, a standardized technique has not been validated. This article provides an overview of the deterioration mechanism, manifestations of the reaction in North America, current state of knowledge for identification and quantification, and current and proposed legislation.



Fig. 1: Typical indications of pyrrhotite deterioration in concrete foundations



Fig. 2: Replacement of residential foundation in Connecticut (photo courtesy of U.S. Government Accountability Office)

Legislation

Effective July 6, 2021, Connecticut enacted Public Act No. 21-120, “An Act Concerning Crumbling Concrete Foundations,”¹ and in Massachusetts, Bill S.548, “An Act Relative to Crumbling Concrete Foundations,”² has progressed through state senate and house committees. In both states, limits on sulfur contents of aggregates were established to prevent the use of aggregates containing pyrrhotite in concrete and minimize the potential for deterioration due to pyrrhotite oxidation.^{1,2}

Specifically in Section 9 of the Connecticut law (CT law), the operator of any quarry that intends to sell aggregates for use in concrete is required to report the total sulfur content in percent by mass (S_T) from a 10 lb (4.5 kg) sample of aggregate on a yearly basis. Tests must be conducted by X-ray fluorescence (XRF) analysis, purge and trap gas chromatography analysis, analysis by combustion furnace, or other technology “deemed at least as accurate by the State Geologist.” The sulfate content limits are:

- If $S_T < 0.1$, the aggregate is approved for use in concrete for a period of 4 years and shall not be required to test again during that period;
- If $S_T \geq 1.0$, the aggregate cannot be sold or provided for use in concrete; and
- If $0.1 \leq S_T < 1.0$, X-ray diffraction (XRD), magnetic susceptibility, or petrographic analysis are required to be completed to identify the presence of pyrrhotite:
 - If pyrrhotite is found, the operator of the quarry is

required “to conduct additional testing, including but not limited to a mortar bar expansion test pursuant to American Society for Testing and Materials standard C1293, Standard Test Method for Determination of Length Change of Concrete Due to Alkali-Silica Reaction, or C227, Standard Test Method for Potential Alkali Reactivity of Cement-Aggregate Combinations; and (2) implement restrictions on the sale or use of aggregate from such quarry in concrete.”

- If pyrrhotite is not found, the aggregate can be sold for use in concrete for a period of 1 year.

Pyrrhotite and Other Iron Sulfide Minerals

Pyrrhotite [$Fe_{1-x}S$] is one of a group of iron sulfide minerals, including pyrite [FeS_2], pentlandite [$(Fe,Ni)_9S_8$], and chalcocopyrite [$CuFeS_2$].³ Many readers of this article will be familiar with pyrite, commonly referred to as “fool’s gold,” which is the most abundant iron sulfide mineral. Pyrite has been known to cause near-surface deterioration in the form of staining and popouts due to the formation of corrosion products. Deterioration has been aesthetic in nature, typically observed in architectural precast concrete, stucco, and exterior concrete flatwork, such as sidewalks and driveways. In architectural precast concrete, surface deterioration has been minimized by recommending petrographic examination of concrete aggregates per ASTM C295/C295M⁴ to ensure that the selected aggregates are free from pyrite.⁵

Although much less common than pyrite, pyrrhotite is the second most abundant iron sulfide mineral. Pyrrhotite is often found associated with the other iron sulfide minerals (for example, pyrite, marcasite, magnetite, and chalcocopyrite). The mineral is a solid solution series that occurs in a range of compositions, different crystal structures, and varying magnetism.⁶ Because of these features and often being present in very small concentrations and combined with other minerals, the identification of the mineral is challenging (Fig. 3). Petrographic techniques, XRD, thermomagnetic analyses techniques, and scanning electron microscopy with energy dispersive X-ray spectroscopy (SEM-EDX) can all be used to identify the mineral, but quantification and locating the mineral still may be missed or challenged.

Pyrrhotite—Mechanism of Deterioration in Concrete

Iron sulfide minerals are unstable in the presence of oxygen and water and susceptible to oxidation corrosion reactions. The rate of oxidation of pyrrhotite can be 20 to 100 times faster than pyrite.⁷ When the oxidation reaction occurs, iron corrosion products form and sulfuric acid is released. Both products can impact the concrete, and the following reactions occur concurrently:

- Oxidation reaction—When iron sulfide minerals

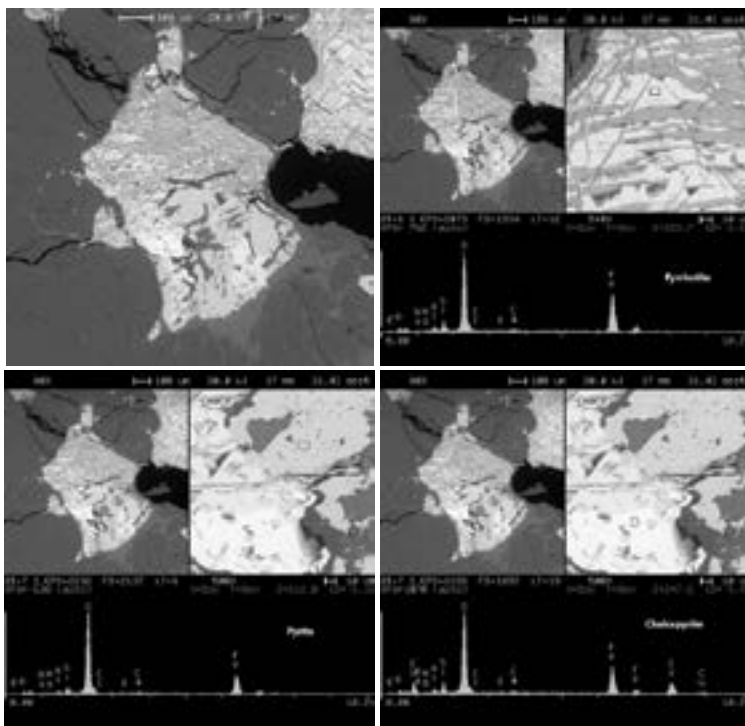


Fig. 3: Scanning electron microscope (SEM) backscattered electron (BSE) images with EDS spectra of occurrence of iron sulfides in concrete aggregate with pyrrhotite showing corrosion

oxidize in an alkaline environment, such as concrete, ferrihydrite ($\text{Fe}(\text{OH})_3$) and goethite ($\text{FeO}(\text{OH})$) are the predominant sulfide oxidation products. The initial reaction products provide the initial expansion and tensile strains on concrete; and

- Secondary mineralization—Sulfuric acid is produced as a by-product of the oxidation reaction. The sulfuric acid lowers the pH and reacts with a portland cement hydration product, portlandite ($\text{Ca}(\text{OH})_2$), to form gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$). Gypsum reacts with the anhydrous or hydrated aluminate phases in portland cement to form expansive ettringite ($\text{Ca}_6\text{Al}_2(\text{SO}_4)_3(\text{OH})_{12} \cdot 26\text{H}_2\text{O}$).^{8,9} If the concrete has a source of limestone (CaCO_3), either from aggregate, cement, or another source, thaumasite ($\text{Ca}_3\text{Si}(\text{OH})_6(\text{CO}_3)(\text{SO}_4) \cdot 12\text{H}_2\text{O}$) can be formed.³ Backscatter images of the expansive reaction products are shown in Fig. 4.

Degradation of the concrete due to pyrrhotite oxidation is initially caused by the expansive forces

created by the formation of corrosion products. Under certain conditions, there is a potential for subsequent secondary mineral formation leading to internal sulfate attack. A schematic of the reactions and chemical formulas is shown in Fig. 5.

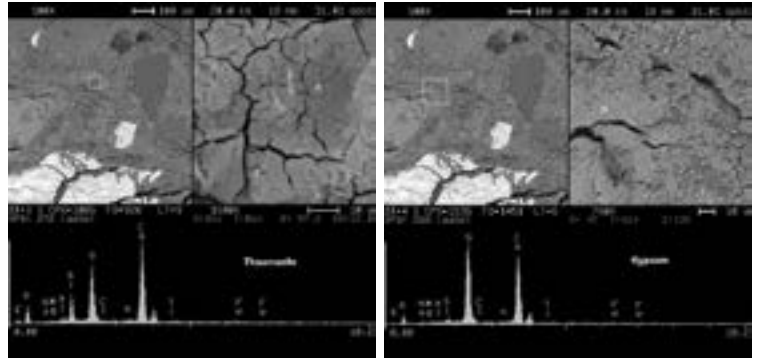


Fig. 4: BSE SEM images with EDS spectra of deleterious secondary minerals formed in the calcium silicate hydrate (paste) adjacent to corroded pyrrhotite-containing aggregate

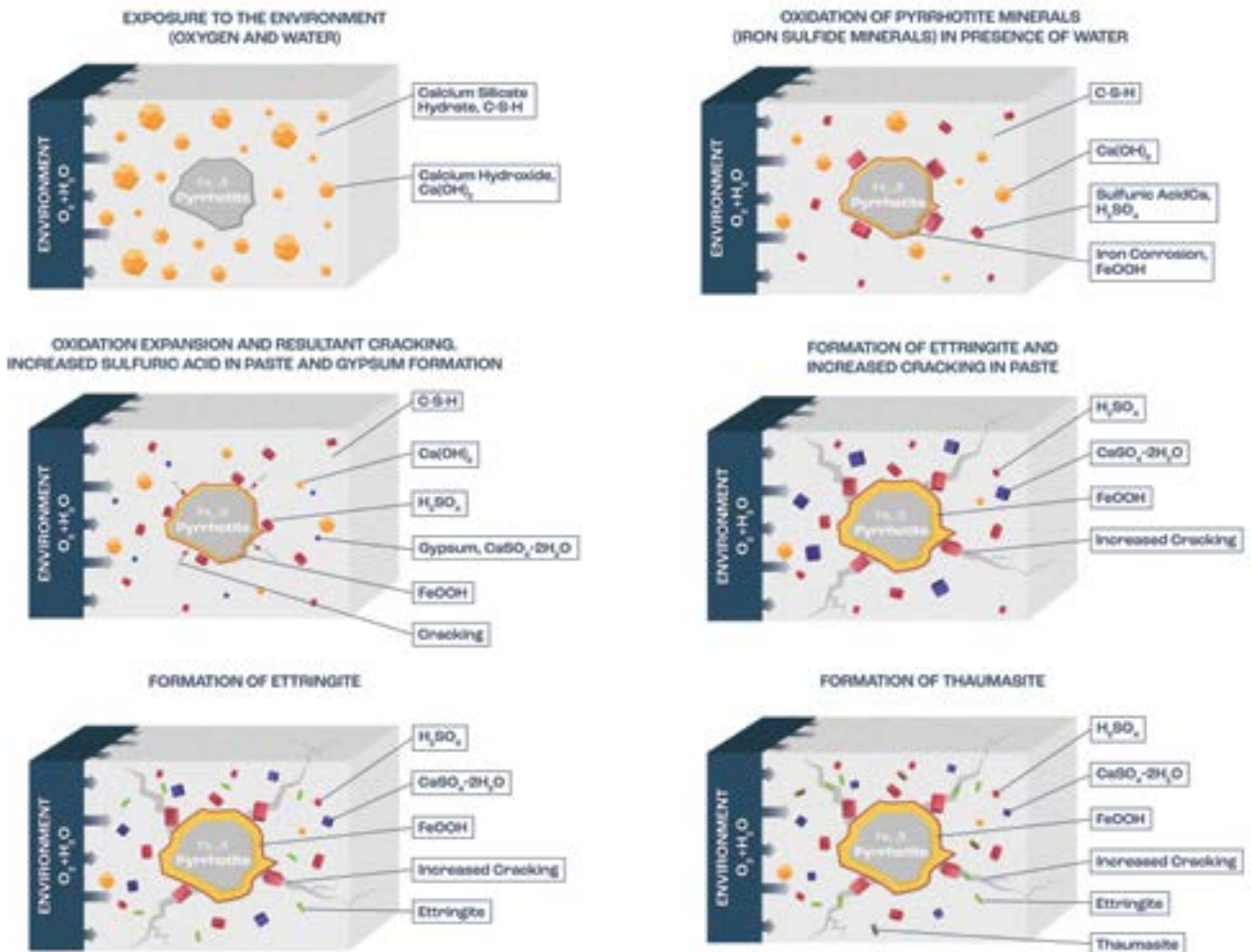


Fig. 5: Schematic of pyrrhotite oxidation and sulfate attack mechanism in concrete suffering deterioration from the presence of pyrrhotite mineral in concrete aggregates (read left to right)

These series of reactions are repeated; oxidation of the pyrrhotite minerals causes further staining, popouts, and expansive pressures while releasing more sulfuric acid, causing additional sulfate attack. Over time, cracking will develop in the form of map pattern, indicating uniform expansion with cracks bridging rust-stained aggregate particles. As the reaction furthers, in certain circumstances, the formation of thaumasite replaces the portland cement hydration product, calcium silicate hydrate (C-S-H), and causes a significant loss in strength. The extent of sulfate attack of the paste is controlled by the composition and size of the aggregate particles, the kinetics of sulfide oxidation, the composition of the cement, and proportioning of the concrete mixture.^{3,10}

The rate and severity of pyrrhotite oxidation and its deleterious effects within concrete depend on several factors. The primary factors are related to the mineral itself, the host rock, and its mineralization, and ultimately, the concrete quality and exposure conditions.

Manifestations in North America

In North America, there are two occurrences of widespread deterioration of concrete structures due to the pyrrhotite mineral. One is in the Trois Rivières region of Quebec, Canada, and the other occurred from aggregates produced in Willington, CT, USA.

In the Canada occurrence, concrete foundations constructed between 1996 and 2008 began experiencing symptoms of the reaction within 5 years of construction. These foundations were constructed with anorthositic gabbro with various metamorphic textures and containing pyrite and pyrrhotite with lesser amounts of other iron sulfide minerals (pentlandite and chalcopyrite). The National Research Council of Canada has indicated that over 1200 houses have had basement walls replaced, with thousands more to be evaluated. The financial impact in the region is over 400 million CAD.¹¹

In the United States, pyrrhotite-containing aggregates produced from Beckers Quarry in Willington, CT, from 1983 to 2015, were used to construct concrete foundations in Massachusetts and Connecticut. In this case, symptoms of the deterioration were not observed until 10 to 20 years after construction. These aggregates contained pyrrhotite in a metamorphic gneiss and schistose rock source. More than 35,000 homes in the surrounding area were constructed with aggregate from the quarry and the average cost to raise the homes and repair the foundations is 150,000 USD per home.^{12,13}

Due to these two occurrences, the U.S. Geological Survey's (USGS) Mineral Resources Program developed a map to show the distribution of pyrrhotite across the conterminous United States (Fig. 6). The map is a compilation of three different databases that uses rock formation type and reported occurrences of pyrrhotite to provide a reasonable

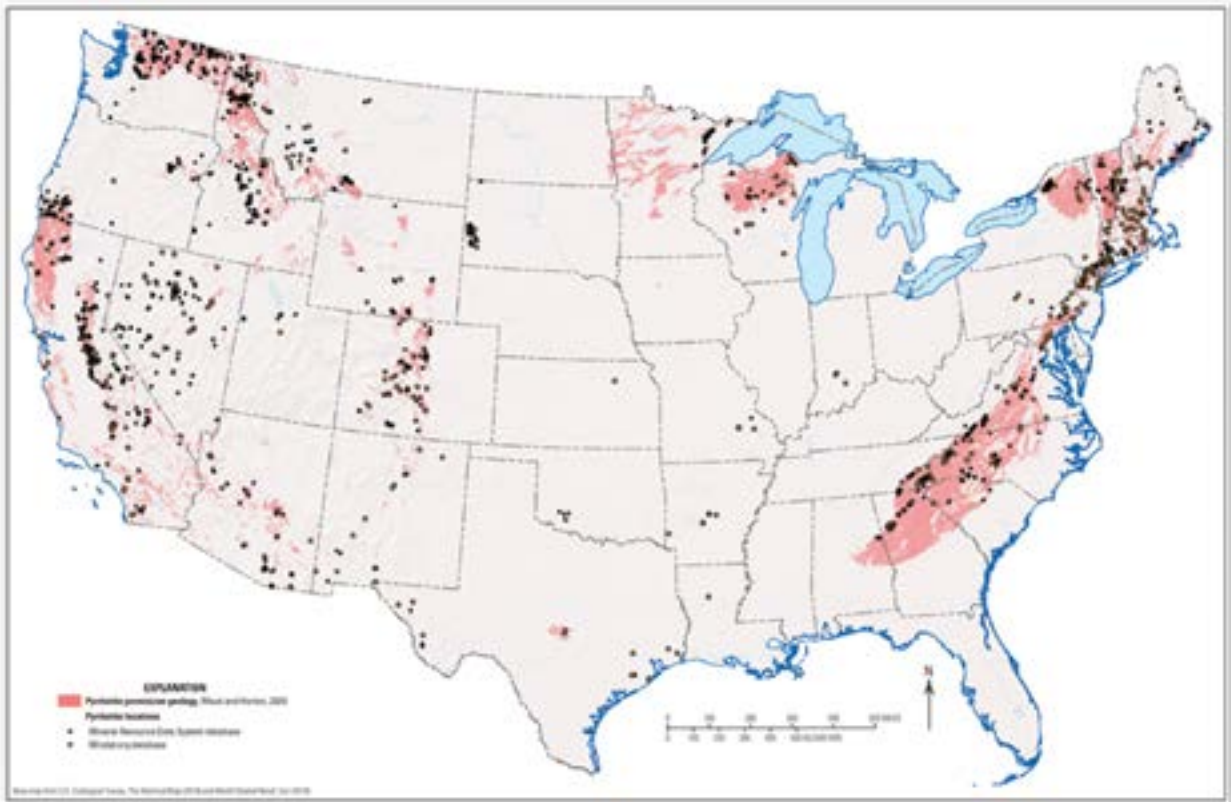


Fig. 6: USGS conterminous United States map showing the location of rock types that may contain pyrrhotite from three different sources (from Reference 14)

indication of where pyrrhotite may occur.¹⁴ Due to the frequency of potential occurrences of pyrrhotite on this map, the concrete aggregate and construction industries have become concerned about the potential for pyrrhotite-induced distress in other regions.

Standardized Procedures, Guides, Specifications, and Legislation

Due to the complex nature of the oxidation reaction and challenges with quantifying the amount of iron sulfides, the scientific community has yet to reach a consensus on an appropriate limit for use in concrete aggregates. To date, iron sulfide minerals are not excluded or limited for use in concrete aggregate standards in North America. Test methods for evaluation of the suitability of aggregates for use in concrete, such as ASTM C295/C295M⁴ or Canadian Standards Association (CSA) A23.1:19/A23.2:19,¹⁵ similarly do not exclude or limit suitable or acceptable amounts of these minerals in aggregate. An overview of the current procedures, guidance documents, specifications, and legislation is provided.

Standardized Procedures

In North America, there is no consensus on standardized test procedures for identifying and/or qualifying the iron sulfides or specifically the pyrrhotite mineral, or procedures for evaluating the potential for deterioration in concrete. ASTM C295/C295M⁴ recommends identifying iron sulfide minerals that may potentially oxidize in concrete but provides no guidance or limitations for use. Beyond this recommendation, we are aware of no other standardized testing procedures or guidance documents provided by ASTM International at this time.

In Canada, CSA A23.1:19/A23.2:19 includes an informative Appendix P—“Impact of sulphides in aggregate on concrete behavior and global approach to determine potential deleterious reactivity of sulphide-bearing aggregates.”¹⁵ This annex recommends using a three-step process for evaluating an aggregate: (1) quantifying the amount of sulfide sulfur content; (2) evaluating the oxidation potential; and (3) evaluating the potential reactivity in concrete (mortar bars). These test methods have proved successful in identifying the potential for deleterious reaction in foundations, but validation over a wide range of aggregate types is in progress. The oxidation and expansion test methods are onerous and preliminary evaluations of the methods indicate high variability of results. The CSA document provides guidance based on the tests, which is summarized in the flow chart (Fig. 7).¹⁶

Guides and Specifications

In the United States, aggregates for use in concrete generally must meet the requirements of ASTM C33/C33M,¹⁷ which defines the requirements for grading and quality of

coarse and fine aggregates. Currently, ASTM C33/C33M does not provide limits or requirements on iron sulfide minerals or total sulfur content. ACI Committee 201, Durability of Concrete, has recently approved changes to Chapter 5—Alkali-Aggregate Reactions of ACI 201.2R-16¹⁸ to include background and occurrences of the reaction. From the draft version of the chapter, limits and guidance on testing for the iron sulfide minerals and total sulfur content were not included.

In Canada, CSA A23.1-19/A23.2-19, in the materials section for aggregates, warns “of sulphides [sulfides], such as pyrite, pyrrhotite, and marcasite, in the aggregate that might oxidize and hydrate with volume increase or the release of sulphate that produces sulphate attack upon the cement paste, or both.”¹⁵ The specification does reference the informative testing procedure discussed previously, but no limits or requirements for testing are provided.

European standard EN 12620:2013 has established limits on total sulfur by mass of aggregate and fillers (excluding air-cooled blast-furnace slag) at 1%. If pyrrhotite is present in the aggregate, that limit is dropped to 0.1% total sulfur by mass.¹⁹ Some preliminary studies of degradation of these minerals have concluded that a level of 0.1% is exaggerated and is unnecessarily rejecting suitable aggregate.^{20,21}

Dilemma of Legislation Ahead of Science

Because the oxidation of pyrrhotite has caused deterioration to tens of thousands concrete foundations of residential and light commercial buildings in North America, USGS conducted a survey that identified the distribution of the potentially deleterious mineral along the Appalachian Mountains and the western United States. Due to these occurrences and concerns about future incidences, the concrete aggregate and construction industries are scrambling to provide guidance to prevent the recurrence of these events.

Ahead of the traditional specification, code, and consensus testing standardization organizations, as previously

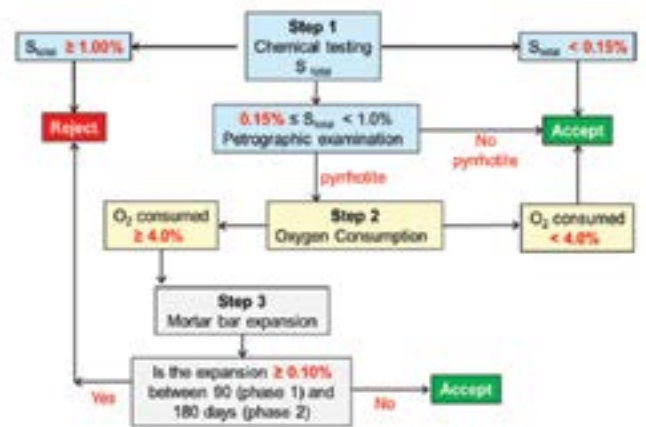


Fig. 7: CSA protocol for determining the potential reactivity of iron sulfide-bearing aggregates in Annex P of CSA A23.1:19/CSA A23.2:19^{15,16}

Table 1:
Summary of test methods for determining total sulfur content

Standard test procedure	Method for total sulfur analysis	Sample to pass	Sample size, g	Reproducibility limit (R)
ASTM D4239	Combustion method with infrared detection	250 µm (No. 60)	0.1 to 0.2	Varies dependent on the sulfur content from 0.05914 to 0.58563. For total sulfur contents from 0.38775 to 5.6125
ASTM E1915	Combustion method with infrared detection	150 µm* (No. 100)	0.2 [†]	Varies dependent on the sulfur content from 0.0133 to 0.648. For total sulfur contents from 0.004 to 4.70
CSA A23.1-19/ A23.2-19, Annex P	Combustion method with infrared detection or acid detection	160 µm	According to manufacturer —up to 5 g	Under development. Preliminary values vary dependent on the sulfur content from 0.08 to 0.28 for total sulfur contents 0.10 to 1.2
ASTM E1621	XRF	Varies [‡]	Varies 0.3 to 1.0	Function of sample preparation, calibration scheme, and sample homogeneity

*May be necessary to grind samples to pass 75 µm (No. 200) to improve precision of samples containing low contents of sulfur

[†]Section 10.5.1.1: Different instruments may require different sample masses for certain content ranges

[‡]Sample prep for powder XRF requires sample to pass 75 µm, with 50 µm ideal

Note: 1 g = 0.035 oz

mentioned, the state of Connecticut has passed a law (Public Act. No. 21-120¹) that requires quarry operators to test aggregates for the presence of iron sulfide minerals. While this law provides protocol for testing aggregates, the law is ahead of the industry and may be technically incorrect and may restrict the use of aggregates erroneously. From the authors’ experiences with the previously referenced testing procedures, knowledge of the occurrence of the pyrrhotite mineral, and experience with other aggregate testing procedures, the following concerns exist related to the CT law.

Sampling, Splitting, and Crushing/Pulverizing

It has been known that sampling, splitting, and crushing/pulverizing have introduced errors in testing concrete aggregates for alkali-silica reactivity.²² This processing of aggregates eliminates some of the reactive minerals and introduces variability and error to the tests. This occurs for reactive silica minerals, which are typically significantly more abundant in aggregates compared to pyrrhotite, where threshold values of 0.10% (4.5 g [0.16 oz]) by weight within a 10 lb aggregate sample are believed to have the potential to cause deterioration in concrete.

Nonstandardized Rapid Sulfur Test

The CT law requires aggregate samples to be evaluated using XRF, purge and trap gas chromatography, combustion furnace, or other technology “deemed at least as accurate.” While ASTM International test methods for XRF analysis (ASTM E1621²³), quantification of sulfur in coal (ASTM D4239²⁴), and metal-bearing ores (ASTM E1915²⁵) exist, no standardized test method has been established for detecting sulfur in concrete aggregates. Furthermore, the standard deviation, sample size, and crushing regime differ between methods. The sample sizes for these tests are approximately 0.1 to 10 g (0.0035 to 0.35 oz), and significant

errors can be introduced when a small sample is being prepared and used to represent a heterogeneous material, such as a 10 lb aggregate sample. In addition, the aforementioned techniques have their own inherent repeatability and interpretation challenges and the prescription of any of these techniques along with the other techniques, “deemed at least as accurate,” lacks robustness and creates challenges in repeatability and comparison of results. A summary of the rapid sulfur test procedures is provided in Table 1.

Identification of Pyrrhotite

Petrographic examination is a common and standardized protocol for evaluating concrete aggregates and identifying potentially deleterious minerals. However, the identification and quantification of pyrrhotite is complex. Due to the nature of the mineral (typically found associated with other iron sulfides, the varying degrees of magnetism, and varying crystal structure) and its presence in low concentrations, it can be difficult to locate. In addition, nontypical techniques may be needed to definitively identify and quantify the mineral, including metallographic microscopy, which is not commonly implemented in petrographic microscopy and requires a high degree of technical skill and experience. Standard XRD analyses may also be used to identify the mineral, but they have a low detection level (generally 1%, with samples containing multiple minerals up to 5%). Because of the sparseness of the mineral, different variations of the mineral, and the nontypical procedures, there is risk that the mineral may not be properly identified.

Incorrect Reference to Test Procedures

If pyrrhotite is identified, the aggregate is required to be tested in accordance with ASTM C1293²⁶ or ASTM C227 (Withdrawn 2018).²⁷ Both of these tests were developed for evaluating the alkali-silica reactivity of aggregates. While

the accelerated environmental conditions in the test methods may promote pyrrhotite oxidation, the interpretation of the results and delineation of the two causes of expansion (alkali-silica reaction and pyrrhotite oxidation) are not provided, nor understood. The CT law haphazardly references other aggregate testing standards which may produce false positive results.

Interpretation of Results

In the CT law, petrographic examination, total sulfur content, and linear expansion of mortar or concrete tests are all prescribed with established limits. These limits would indicate the threshold to where potentially deleterious oxidation of pyrrhotite mineral may cause deterioration in concrete. Currently, there is not enough established research or correlation of laboratory and field performance to correlate the prescribed limits to a potential for deleterious behavior in concrete.

Precision and Bias

Lastly, with any consensus-developed standardized test method, it is important to understand its precision and bias. The CT law provides no insight on the precision of the test method or the variability that can occur within the same laboratory or among different laboratories. Again, this information is important for the interpretation of test results, and it would have been provided if these procedures were developed by a consensus body.

For the reasons stated herein, the CT law may have been prematurely instituted and set a precedent for other states to follow incorrectly. While the authors agree that action is needed to limit the risk for potential deterioration to other concrete structures, implementing laws without scientific backing or understanding of the test procedures only further hurts the concrete and concrete aggregate industry.

Conclusions and Recommendations

This article provides an overview of the pyrrhotite deterioration mechanism, manifestations of the reaction in North America, current state of knowledge for identification and quantification, and current and proposed legislation. From this information, it is evident that our understanding of pyrrhotite mechanism and identification is evolving. However, due to the complex nature of the reaction and lack of correlation between laboratory testing and field performance of concrete, the active and proposed legislation to limit aggregate use in concrete may be premature. To accelerate our understanding, work toward development of standardized testing procedure, and limit the risk of the reaction, the following recommendations are provided:

- **Minimize contamination and variability in tests**—Many of the proposed aggregate test methods require excessive crushing, pulverizing, splitting, and testing of very small samples. These procedures have produced variability and inconsistency in laboratory results. Novel test methods

should be developed to minimize these risks and ensure representative aggregate samples are evaluated;

- **Influence of materials and permeability**—The influence of the quality of the concrete, presence of supplementary cementitious materials (slag cement, fly ash, and others), water-cementitious materials ratio (w/cm), and permeability of the concrete are not fully understood. Additional testing and evaluation of field structures with varying material compositions should be performed and studied in various regions;
- **Influence of exposure conditions**—The effect of the surrounding environment is suspected to have an influence on the reactivity of concrete containing aggregates with pyrrhotite minerals. Specifically, colder subgrade environments (basements) in northern climates are suspected to be worst case scenario for promoting pyrrhotite oxidation. Further investigation is needed to validate this theory and provide recommendations for construction in various climates; and
- **Correlate laboratory tests with field performance**—Accelerated tests with definitive limits (such as total sulfur content, oxygen consumption, and others) should be benchmarked to concrete cast and stored outdoors to determine the efficacy of the method to predict actual field performance.

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Selected for reader interest by the editors.



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