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Environmentally Safe and Effective Method for treating Pyrophoric Iron*

Several technical papers, assorted patents (both current and expired), and current procedural cleaning / neutralization documents were read and compared for preparation of a suitable product (based upon these reports) for Iron Sulfide cleaning projects.

This product **PI-170** provides a method of treating a reactive compound which contains sulfur in a reactive form with a suitable stabilized oxidizing agent to produce a different compound which does not contain sulfur in a reactive form. The purpose of **PI-170** is to decontaminate and clean a vessel containing surfaces in contact with, or subject to being in contact with, sulfides, mercaptans, sulfites, reactive multi-sulfur compounds and/or mixtures thereof.

Insoluble iron sulfides are difficult to clean / treat as they are either not soluble or have limited solubility in typical cleaning solutions.

PI-170 provides a method of treating the surface of pyrophoric iron sulfide with a suitable oxidizing agent to form a film on the surface of the insoluble iron sulfide whereby the film shields the surface of the iron sulfide from an environment containing oxygen in the gas phase to thereby prevent spontaneous ignition of the pyrophoric iron sulfide.

The functionality of **PI-170** is based on documented studies that show the oxidizing agent employed will prevent the formation and/or continued existence of reactive multi-sulfur compounds by converting the sulfur content of water soluble precursor compounds, i.e., sulfides and mercaptans, the sulfur content of water soluble sulfites and the sulfur content of water soluble reactive multi-sulfur compounds from a reactive form to a non-reactive form.

The inorganic persulfates also react with insoluble iron sulfide as described above and watersoluble hazardous sulfur containing compounds to produce water soluble sulfates. There are no by-products of the reaction between inorganic persulfates and reactive sulfur which are hazardous or environmentally offensive.



Case histories indicate that the quantity of oxidizing agent useful herein sufficient to increase the valence of sulfur from some value in the range of from -2 to +5 to a value of +6 is an amount in the range of from about 3 moles to about 12 moles of oxidizer per mole of sulfur compound to be oxidized. However, in a typical industrial setting, the actual quantity of hazardous sulfur containing compound to be treated is not usually readily available; therefore, the quantity of oxidizer to be employed cannot be calculated with precision. Accordingly, the amount of oxidizer initially employed in an industrial application is an amount in the range of from about 500 to about 2000 parts oxidizer per million parts of aqueous solution. Thus, the hazardous sulfur containing compound, or compounds, to be treated can be initially contacted with an aqueous solution containing a minimum of about 2000 ppm oxidizer. The concentration of oxidizing agent can be continuously monitored by known analytical methods during performance of the process and oxidizing agent can be added to the solution to maintain an oxidizer concentration of at least about 500 ppm.

The vessel to be decontaminated should be isolated from other vessels by methods known to be effective by cleaning contractors. The vessel is then drained of its contents, preferably in a manner which will prevent the entry of oxygen into the interior of the vessel. Thereafter, the concentrate, which can be diluted in place, or the diluted concentrate, is introduced into the vessel to establish contact with the hazardous sulfur containing compounds therein.

The contact between the sulfur compound (or compounds) to be treated and the oxidizer can be effected by simple fill and soak techniques. Cascading should work as well. Contact is preferably established by dynamic means. In this regard, un-reacted oxidizing agent and untreated sulfur compound(s) can be continuously mechanically mixed together in the vessel in order to establish continuous contact between the sulfur compound and fresh oxidizing agent for a time sufficient to convert reactive sulfur to un-reactive sulfur. Establishing contact by dynamic means is especially useful in a situation where the sulfur compound adheres to a substrate and/or is shielded by a coating of some sort or enclosed in a film or varnish. **PI-170** contains an effective surfactant to help penetrate the coating to assist the oxidizing agent to establish contact with the sulfur compound.

The contact time is a function of the concentration of oxidizing agent in the aqueous solution. In this regard, the concentration of oxidizing agent in the solution will decrease as the valence of sulfur increases. Thus, the valence of all oxidizable sulfur available for oxidation will be considered to have been increased to a value of +6 when the concentration of oxidizing agent in the aqueous solution stabilizes at some constant value. As a matter of prudent practice, contact is maintained at a constant concentration of oxidizer for a time in the range of from about 1 to about 2 hours to help assure complete oxidation of all sulfur to a valence of +6.



Example of typical cleaning / treating procedure

100 gallons of the **PI-170** concentrate solution together with 2,150 gallons of de-ionized water is added to the vessel containing the mentioned residual crude oil / hazardous sulfur containing compounds. A buffering material (Soda Ash) is added to the vessel as necessary to adjust the pH of the solution in the vessel to a value in the range of from about 7 to about 8. (**PI-170** must be provided below pH 7 for stability purposes, typically between 6 - 6.5). At this point the concentration of **PI-170** in the vessel is 0.4% active oxidizer by weight of solution or about 4000 parts oxidizer per million parts of solution.

The vessel and its contents are heated to a temperature of 180F and the contents of the vessel are continuously circulated to promote contact between the **PI-170** solution and the hazardous sulfur containing compounds. The aqueous content of the vessel is sampled as soon as practicable and analyzed for active oxidizer. The analysis is conducted to enable maintenance of an active concentration of oxidizer at a value of at least 500 parts per million.

The treating solution is drained from the vessel after a period of contact of about 2 to 3 hours at 180F during which time the concentration of oxidizer is maintained at a value in the range of from about 500 parts per million to about 2000 parts per million. At this point all soluble, hazardous sulfur containing compounds will have been converted either to elemental sulfur or to the sulfate ion. In addition, all insoluble, hazardous sulfur containing compounds, such as FeS, will have been coated with a layer of iron oxide to shield and thereby inactivate the insoluble hazardous sulfur containing compounds.

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