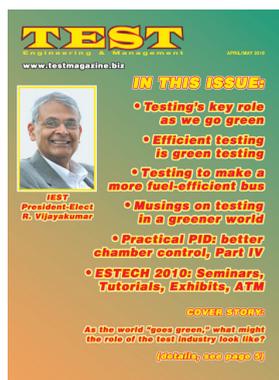


As we go green, testing will play a key role

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Testing always has been important in the engineering and technical realm. But as we move to new heights in our technically oriented society, increased testing—and precision—is expected to become more and more important. New needs will become necessary and new ventures are expected to evolve.

These testing needs will be centered on the quantification of our environment including the air we breathe, the water we use and the land upon which we live. Determining and quantifying air toxins is just one such area of concern. First, we must know what we now have and secondly, as improvements evolve, quantifying that improvement will become important. Improved instruments in the hands of trained and skilled technicians will be paramount to success.

Hydrogen sulfide in the air

This gas, H_2S , always has been in our air. Its effects upon humans can range from almost nothing, to an annoyance, to a health hazard, and to terrible effects which result in sudden and instant death depending upon concentration in the air and time of exposure.¹ So, knowing how much is present is paramount and the best way to know that quantity is to test. The focus of this article will be testing residential ambient air where concentrations of H_2S range from a fraction of a ppm (parts per million) to <100 ppm, at STP. Also, we must understand early-on that H_2S is highly soluble in water and water vapor in the air. This solubility is a factor regarding test instruments capabilities and accuracy.

Some history of testing for H_2S

Before the industrial revolution, people were exposed in early residential communities which included the gas in underground waste-disposing piping. Workers used their eyes and their noses to indicate the presence of this gas.² Those were the earliest “instruments” used to indicate the presence of H_2S and it was documented as early as the late 1800s. Moving forward in time to the present day, during that period

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a host of test instruments and gadgetry has evolved and been used with a range of accuracy and success.

State-of-the-art today

Measuring instruments and laboratory apparatus generally fall within one of the following several types. Instrument capabilities range widely regarding accuracy, capabilities, complexity, and cost. In nearly all cases only trained and skilled personnel can be expected to operate and achieve success with these presently available instruments. Let’s categorize these and look at their capabilities and shortcomings.

Hand-held instruments can range from the simple to the complex and can cost from a few hundred dollars to twenty thousand dollars or more. If one wants to know how much H_2S is in the air, the obvious answer is simply to go get a hand-held instrument and measure the H_2S . This is a proper solution, except when we learn that these test instruments, as a whole, will not measure the H_2S that is dissolved in water vapors, dew, or fog. Under these conditions, an operator can be standing in your yard with a \$20,000 meter in his hand and it is reading zero. Your body senses the presence of the H_2S via the nose and the eyes because you breathe in the fog-laden air where it is instantly raised to 98.6 degrees F, and you feel its effects. So, hand-held test instruments do have some shortcomings, particularly when measuring H_2S at the lower range of concentrations. At higher concentrations of H_2S —say >100 ppm—the solubility errors are smaller and less significant.

Colometric tubes. A host of these test instruments are commercially available. Although they are fairly economical, easy to operate, and require little or no calibration and maintenance work, they, like the hand-

held instruments, will not measure the H_2S that is dissolved in water vapor, dew, or fog. Well, where do we go from here?

Mobile and fixed station test instruments. There are a host of test instruments that fall into this category. A complete annotation is beyond the scope of this article, but certain representative types are shown and discussed here. They are typically more complex and require more skilled operators. Frequent calibration is required. As with the aforementioned test devices, there are many well-documented shortcomings of these instruments,^{3,4} including desensitization from the presence of other toxic gases, water vapor, effects of relative humidity, and oxygen-deficient air. Specific manufacturers’ literature and operating manuals are essential for review before purchase, installation, and operation.

One example type of H_2S instrument frequently used for fixed-point installation is a lead acetate-impregnated paper tape⁵ test meter. Here air is monitored for low concentrations of H_2S up to about three ppm. Here a water solution is used on the front end of the air intake—thus, casting doubt on accuracy due to H_2S being soluble in the water. The ASTM Standard⁵ also points out other errors that can result from poor selection of certain materials in the flow stream like O-ring material and lubricants.

Other instruments are used for testing ambient air at higher concentrations of H_2S (10-100 ppm) such as workplace environments.⁶ Similar errors as mentioned above are applicable to this group of testing devices.

Moving up the chain of complexity we come to a group of test devices generally called UV Fluorescence H_2S analyzers,⁷ which may be used for fixed stations (indoor or outdoor) or outdoor mobile monitoring vans. Usually this instrument is just one part of an assembly which consists of pumps, manifolds, tubing, calibration gases, converters, and scrubbers. The manual⁷ makes numerous

proper references to factors which can cause errors and desensitizing, including cautionary statements regarding tubing and metal selection in the flow stream and other gasses which fluoresce in a similar manner to the measured gas. In a nutshell, the test devices use a technique which takes in air containing H_2S , oxidizes that gas to SO_2 , then measures the total SO_2 and subtracts from that number the amount of SO_2 in the air sample. The Texas Commission on



FIG. 1—A gas processing plant in Texas which emits acid gas to the air, including up to four pounds per hour of H_2S .



FIG. 2—Low-to-the-ground emissions from an oil and gas plant, including H_2S , in the form of “Death Clouds.”

Environmental Quality (TCEQ) uses this instrument for both fixed station and mobile monitoring of H₂S in ambient air. As is common, the mobile monitoring vans are rigged with much other apparatus, including intake poles which collect the air sample some 20 feet above ground level. It is known that H₂S is heavier than air and gathers in low places. This is just the first problem contributing to inaccuracy. Then, the air sample goes through feet of tubing of unknown composition. It is known that certain flexible plastic tubes are better than others⁸ for H₂S testing, i.e., PTFE and FEP exhibit improved properties as compared to Tygon. All these tubes have an affinity for water in the air which accumulates on the inside surfaces and dissolves H₂S, contributing to a second source of test errors. The state agency arranges for the air sample to then enter a scrubber composed of four angstroms molecular sieve material to dry the air sample as required by the UV analyzer. This removal of water which collects in the scrubber dissolves more of the H₂S in the air sample, thus contributing to a third source of errors on the low side. It looks as if the more complex we get, the more errors we have—and that is true.

Oh, I've got the answer—let's just gather some air samples with the H₂S present and go to the lab and test using a Gas Chromatograph (GC). That's it, go get some canisters! Wait a minute, what kind do we use? We can't use steel because the iron reacts readily with the H₂S so as to lower its concentration in the sample waiting to be tested. About an hour of wait, will remove nearly all the H₂S in practical concentrations. Well, let's use stainless steel, say 316 or 304 grade. The iron alloyed in the SS (some 40 percent) will likewise react. What about internally coated SS cylinders? Although somewhat better, there is no practical way to check the internal coating for bonding, absence of cracks, and missing sections other than qualification using a span gas of known quality. Even glass with its known alloying components (used

to remove brittleness) will "eat up" a lot of H₂S in the air sample. Plastic canisters have problems as mentioned above with plastic tubing. Even the Railroad Commission of Texas (RRC) recognizes this known defect in the "Discussion of Rule 36 Requirements" document.⁹ Appropriately stated therein is a statement as follows: "All tests should be field tests, as Hydrogen Sulfide will impregnate all known sample containers: Glass, Plastic, Stainless Steel, etc." Statewide Rule 36 also addresses many important aspects of H₂S testing despite some deficiencies of the SWR itself. And then, there are other problems associated with canister use, such as water vapors being present in the air that can condense and collect in the container along with dissolved H₂S, which cannot practically be removed in the laboratory before the air goes to the GC.

What about the EPA?

The EPA has no recommended test instrument or device of any kind for measuring H₂S in residential air. In fact, the EPA does not even consider H₂S a HAP (Hazardous Air Pollutant) at this time. End of story here.

The Copper Rod Test

This is a new and improved test. Well—where do we go to get some simplicity along with decent accuracy while testing our air for the presence of H₂S? One known source of H₂S in the air is from the emissions of Amine Gas Processing Plants, which remove acid gas (H₂S and CO₂) from the raw natural gas stream and spew these gases into the air in Texas. One such plant (shown in Figure 1) is allowed to emit four pounds per hour of H₂S into the air. Characteristic emissions can gather in low places such as shown in Figure 2 and move like a "Death Cloud."^{10,11} In Denver City, Texas, in 1975 nine people died from H₂S exposure, so it was and still is a serious issue.

In the way of a little background, there was the "New Penny Test" where one could take a new penny and place it on a post or stump and wait for the H₂S gas to darken or blacken it. Then, you knew that H₂S was present. The U.S. penny changed in the early 1980s—no longer is it nearly pure copper. Pennies also became harder and harder to obtain. Handling the pennies could place oils from the fingers on the surface so as to desensitize the surfaces.

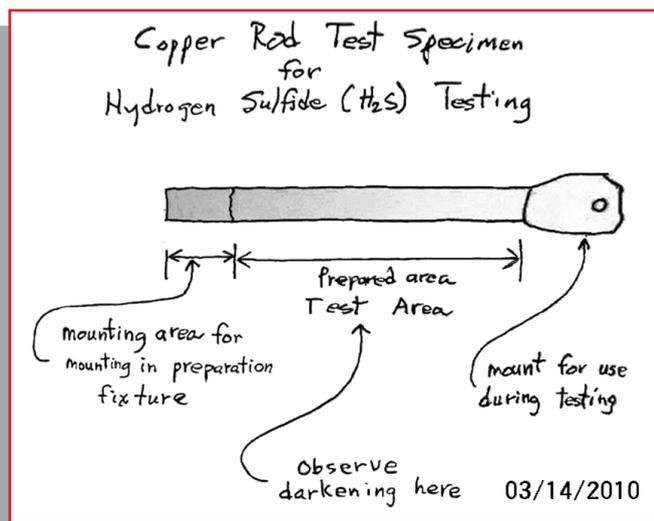


FIG. 3—Drawing of prepared test article of copper tubing.

So, what I developed is to simply take a six-inch long piece of L grade 3/8-inch copper tubing, and prepare the center section. See Figure 3 for a drawing. The prepared center section is sanded to the raw copper for exposure, while mounted in a simple drill press. Then, it is hung in the air about one to three feet from the ground and exposed to any H₂S than may be present. When exposure occurs, the center section of the rod turns to a characteristic blackish color. The visual color of copper that is exposed to H₂S is well-documented in the literature as this technique is used widely in various applications including testing for H₂S in propane,¹² in down-hole wellbores,¹³ and others.¹⁴ The prepared procedure, not included herein, prescribes that the prepared specimen not remain in the ambient air longer than three days so as to minimize oxidation by the oxygen in the air. Copper oxide has a reddish color, unlike the copper-sulfur compounds that can range from dark purple to dark bluish to black. Exposure is visible to the naked eye. Figure 4 shows a re-photographic example of an exposed copper rod. Observe the center section. This type of exposure is indicative of the presence of H₂S in the range of two to 20 ppm over some practical time period. This Copper Rod Test, although a positive indication of the presence of H₂S, will not tell the exact amount of H₂S, or the exact time of exposure; but a major advantage is that the rod is always "looking." During exposure, an operator is not required to be present, and the prepared section of

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search, engineering, and management positions in the areas of measurements and equipment design and testing, largely in the university environment and in the oil and gas

industry, over the last 35 years. Milberger now considers himself retired, but nevertheless undertakes special projects important to the advancement of the state of the art in selected areas of interest, and where financial gain is not an overriding consideration. Widely published, Milberger is an inventor on 60 U.S. patents, and holds BS and MS degrees from Texas A&M University.

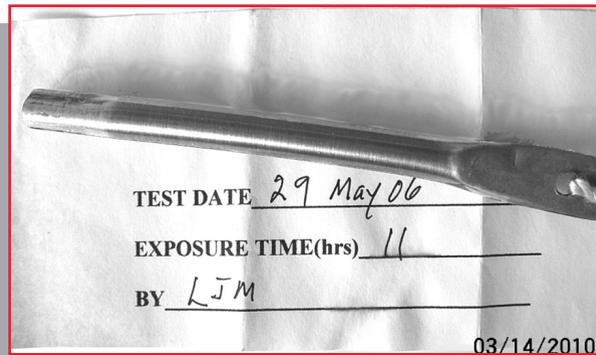


FIG. 4—Photograph of exposed copper rod. Note center section indicating the presence of H₂S.

As we go green, testing's key role (continued)

the rod will accumulate dew with dissolved H₂S. When the dew evaporates, the H₂S is instantaneously exposed to the copper and a positive indication is achieved. This ability is a major advantage of this test.

Future development of this test technique will include the following:

1. Identify and quantify the exact copper-sulfur compounds present on representative test specimens.

2. Correlate exposure patterns and amount of blackening to known concentrations of H₂S over practical time periods.

Summary and conclusions

H₂S exposure by residents who live on the land can be troubling, annoying, and even dangerous. Health effects are well-documented even for low-level exposure. So, testing for this dangerous gas in our air is very important—and this testing is difficult and time-consuming. State and local agencies are not always accommodating. On top of that, many problems and shortcomings are apparent with currently available test methods and instruments. As a result, residents are in the same position as before the advent of test instruments—dependent on the eyes and the nose for detection of H₂S.

It is clear that improved test instruments for measuring H₂S in our environment are sorely needed, and, it is expected that this evolution will take place.

In the meantime, an improved and simplified test method is documented herein, which includes the exposure of copper to ambient air that contains Hydrogen Sulfide (H₂S). Simple visual observing of the copper color and its magnitude proves the presence of this gas in the air. This color-change-in-copper technique is well-

documented and has already been used in a wide range of applications. Further development is expected to more precisely identify the amount of H₂S in the air and the time of exposure.

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