Testing Permeation Resistance in Coatings for Wastewater Structures

Here's a look at ongoing research on the effect of permeation resistance on coatings in headspaces

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Biogenic sulfide corrosion of wastewater conveyance structures can sometimes seem as unstoppable as it is destructive and costly. With elevated levels of H_2S gas and the subsequent formation of sulfuric acid in domestic wastewater collection systems, it's critical to protect valuable infrastructure from sewer corrosion. A protective coatings system is a viable protector of these surfaces only as long as it can withstand the permeation of the sewer gases and acid attack, arguably the most important property of a severe wastewater protective coatings system.

Sewer Bugs: Tiny, Yet Destructive

Biogenic corrosion has been studied since its discovery in the mid-1940s. The basic biogenic processes that result in corrosion, which can be very severe, involve sequential steps and at least two sorts of microorganisms.[1] More than 60 types of microorganisms might be involved. The exact sequence of events varies widely, and depends on the conditions at a given site. However, a general process can be described.[2]

Domestic sewage entering wastewater collection systems contains large amounts of sulfate ions ($SO_4^=$), which are reduced by sulfate reducing bacteria (SRBs, e.g. *Desulfovibrio* sp.) under anaerobic conditions to form hydrogen sulfide gas (H_2S). Under turbulent and decreasing pH conditions, especially, H_2S escapes from the aqueous phase to the sewer atmosphere where it can react with oxygen to form elemental sulfur, which is deposited on the sewer wall. The sulfur then becomes a substrate for oxidizing bacteria (SOBs, e.g, *Thiobacilli* sp.) that convert the sulfur into a dilute sulfuric acid (H_2SO_4), theorized at a concentration no greater than 5–7%.[3] The sulfuric acid attacks the cement binder of the concrete, exposing aggregate, and thereby weakening the structure. This biogenic sulfuric acid corrosion process is a widely known culprit of corrosion in wastewater systems (Fig. 1).

Sewer Gases: An Unpleasant Concoction

Little has been reported about the gases and vapors that commonly emanate from septic sewages flowing in normal domes-

tic sewerage systems. In addition to hydrogen sulfide gas, concentrations of carbon dioxide (CO_2) and methane (CH_4) gases are thought to exist in the headspaces of wastewater conveyance and treatment structures as a result of the decomposition of waste.[3] (Gases such as ammonia [NH_3], sulfur dioxide, and nitrous oxide are also theorized to be present but at much lower levels.) As a group, all of the above gases are referred to as "sewer gases." Moreover, hydrogen sulfide and carbon dioxide are both considered "acid gases"[4] and known to be corrosive to steel, ductile/cast iron,[5,6] and some grades of stainless steel and aluminum.

Hydrogen sulfide gas has always been present in sewerage systems, but in the past, its average levels were thought to be less than 10 ppm. Its levels began to rise after federal regulations mandated the removal of heavy metals (e.g., mercury, cadmium, zinc, lead, etc.) from industrial waste discharges and the use of odor control to contain the noxious odors within these environments.

Although the direct H_2SO_4 attack on protective coatings for wastewater environments has been studied throughout the past several decades, an emerging view is that the sewer gases may play a dominant role in the permeation resistance of protective coatings. While the effects of sewer gases on protective coatings are poorly understood, new research suggests that sewer gases, especially in combination with H_2SO_4, may be the predominant destructive agent affecting the permeation resistance of coatings used to protect the wastewater infrastructure.

In the past, protective coatings such as 65–75% volume solids coal tar epoxies, 55–60% polyamide epoxies, and, less commonly, 90–100% volume solids novolac epoxies have been used with some success in moderately aggressive sewer environments (less than 10 ppm H_2S). However, as hydrogen sulfide levels increased, these types of protective coatings showed blistering and delamination in sewer environments (Fig. 2). It was thought that these coatings failed from direct sulfuric acid exposure generated through biogenic sulfide corrosion. But on many occasions, the authors observed failures of the protective coatings on surfaces with a pH above 4.0-5.0 and very little corrosion on unprotected, adjacent concrete, suggesting that the H_2SO_4 secretion was extremely dilute. Moreover, the authors saw that high-build protective coatings emerging onto the marketplace for these environments were failing, despite purportedly possessing resistance to dilute sulfuric acid exposure. These observations suggest the coating film degradation (permeation) was not necessarily from direct sulfuric acid alone but from a combination of sulfuric acid and the sewer gases in the headspace.

Fig. 1 (Facing page): Biogenic sulfide corrosion to the coal tar epoxy protective coating, exposed concrete substrate and exposed ductile iron piping in less than 5 years.
Fig. 2: (Above): Blistering of a coal tar epoxy coating in the headworks of a wastewater treatment plant. All photos courtesy of the authors.

These observations led the authors to suppose that the sewer gases (i.e., H_2S, CO_2, CH_4, and NH_3), having smaller molecular sizes and linear dimensions than sulfuric acid, could penetrate the matrix of the protective coatings to cause blistering and cracking, and to eventually reach the substrate thereby negating any barrier protection. This hypothesis was supported by accelerated laboratory testing

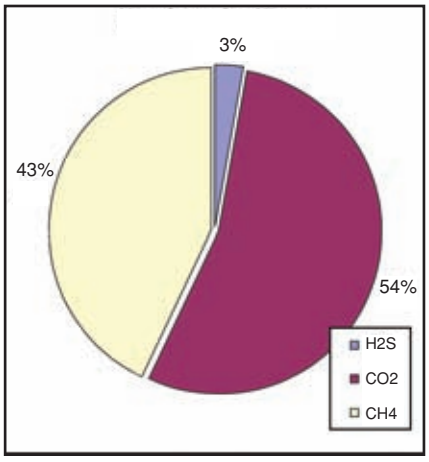

Fig. 3: Mean sewer gas mixture

in which steel panels coated with various high-performance systems commonly specified for severe wastewater environments, were exposed to chamber tests simulating gas/acid conditions of a sewer headspace environment. Using the Severe Wastewater Analysis Test (S.W.A.T.) chamber[7,8] with a vapor phase containing H_2S, CO_2, and CH_4 gases, and an immersion phase containing dilute H_2SO_4 and sodium chloride solution, researchers exposed panels to the sewer gases with periodic immersion in the solution (3 times daily, 15 minutes each) for 28 days.

The coated panels were measured for permeation resistance through electrochemical impedance spectroscopy (EIS) analysis after the 28-day exposure. EIS measures the electrical resistance (impedance) of a protective coating, considered related to its permeability property. Experimentally, impedance is determined as a function of the frequency of an applied AC voltage.[7,9] The data consist of a Bode plot of Log Z versus Log f, where Z is impedance in ohms•cm^2 and f is frequency in Hertz (0.05 Hz to 100 kHz). From the Bode plot, Log $Z_{0.1\ Hz}$ is determined by interpolation. The Log Z value at 0.1 Hz is tabulated and used as the basis of comparison between coatings and for monitoring the change of a coating as a function of exposure time to the test environment. Selection of Log $Z_{0.1\ Hz}$ represents a compromise between speed of

Table 1: Coating Types Common in Wastewater Protection, Subjected to the S.W.A.T

Test Conditions: Sewer Gases: H_2S, CO_2, CH_4; Solution: H_2SO_4, NaCl; Temperature: 65° C

System	Volume Solids	DFT	EIS Impedance Analysis (Log Z) at 0.1 Hz (Ohms•cm²)		Retained Impedance (permeation resistance)
			Pre-test	Post-test (28 days)	
Polyamide Epoxy	55%	19	10.2	0	0%
Polyamidoamine Epoxy	70%	13	9.4	0	0%
Coal Tar Epoxy	75%	33	10.8	0	0%
Cycloaliphatic Amine Epoxy	80%	21	9.5	0	0%
Cycloaliphatic Amine Epoxy	80%	17	10.2	0	0%
Novolac Epoxy	100%	12	10.9	0	0%
Amine Epoxy Mortar	100%	141	11.3	8.1	72%
Amine Epoxy Mortar	100%	119	11.4	7.3	64%
Amine Epoxy Mortar	100%	128	11.3	9.9	88%
Aromatic Polyurethane Fast-Set	100%	42	11.6	7.6	66%

Table 2: Sewer Gas Levels by Site (ppm)

Sewer Gas Levels by Site (ppm)			
	H_2S	CO_2	CH_4
Central Florida	78	11,700	6,000
Northwestern US	6	545	1,500
New England	14	1,550	2,000
Rocky Mountain US	328	1,910	2,500
Midwestern US	256	1,178	1,625
South Texas	590	17,520	4,000
Coastal Virginia	660	3,000	12,000
Average	**276**	**5,343**	**4,232**
Median	*256*	*1,910*	*2,500*

analysis and the selection of a frequency at which differences in coating performance can be reliably determined. Any reduction of a coating's impedance is related to the nature of the polymer, its density and fillers. Although dry film thickness (dft) can also influence impedance, the authors feel that dft is secondary, as evidenced by comparing the coal tar epoxy with the aromatic polyurethane and amine epoxy mortars, all having comparable film thicknesses yet different post-test impedance results, as seen in Table 1.

Because so little was known about the actual levels of sewer gases in typical U.S. wastewater collection systems across the U.S. and the gases' effect on high-performance coatings, the authors began a study in 2007 to measure sewer gas concentrations and study their effects on various traditional and emerging protective coatings technologies. The field study began with four testing sites but now has seven testing sites. The sites were chosen because they all had a history of severe biogenic sulfide corrosion and they represented different geographic locations and climates in the country. The current testing sites are in North Central Florida, Northwestern U.S., New England, Rocky Mountain U.S., coastal Virginia, Midwestern U.S., and South Texas.

The purpose of the investigation is two-fold: to measure the gas levels in the severe wastewater headspace environments and to study the effects of these sewer environments on typical protective coatings systems. The expectation is to gain a better understanding of the sewer gases present in sewerage structures and connect them to in situ biogenic corrosion and sewer gas attack. Although testing has yet to be concluded for any of the sites, enough usable data has been collected from them to obtain a general picture of the sewer gas concentrations in a "typical" severe wastewater headspace environment.

To date, a total of 17 sets of sewer air measurements have been taken at the seven testing sites. Researchers gathered grab samples of the sewer atmospheres with a remote multi-gas detector and a methane meter. Additional measurements have been taken by municipal wastewater treatment plant staff and others at some of the sites as needed, using a gas sampling pump or an H_2S logger. The three main sewer gases detected are hydrogen sulfide, carbon dioxide, and methane. Although attempts have also been made to measure concentrations of ammonia (NH_3), sulfur dioxide (SO_2), nitrous oxide (N_2O), and other sewer gases thought to be present, no significant levels have been detected in this study. The main gas composition by testing site, to date, is presented in Table 2, and the mean sewer gas mixture is given in Fig. 3.

The average gas concentrations are noteworthy because they provide a model for a sewer gas mixture in typical domestic wastewater conveyance head-

Fig. 4: Typical testing site with carbon steel and concrete samples suspended in severe wastewater headspace environment.

space environments across the U.S. Although $H_2S/CO_2/CH_4$ gas compositions varied within the testing sites, the general findings reveal that carbon dioxide comprises the overall majority of the sewer gas followed closely by methane gas (although the median values were reversed, with methane comprising the majority). Moreover, H_2S levels consistently composed only a small portion (less than 10 percent) of the overall sewer gas mixture found at each site. Added research found many factors influencing the varying sewer gas levels, such as wastewater detention times, waste temperatures, BOD, and industrial effluents. Nevertheless, the average gas mixture has proven to be representative of all testing sites in terms of the gases' relationship to one another. Once more is learned about the gas mixture, the data can be used for accelerated wastewater laboratory testing for coatings, such as the S.W.A.T.

Field Study: Proof Is in the Sewers

To test the authors' hypothesis that corrosion protection of protective coatings is altered by exposure to sewer gases and by the composition of the corrosive reagents in domestic wastewater conveyance and treatment structures, coated steel and concrete panels were suspended from stainless steel racks into the headspaces of each site (Fig. 4). Six steel coupons (4 in. x 12 in. x 1/8 in.) and five concrete cylinder coupons (3 in. x 8 in.) were prepared and coated with various protective coatings systems commonly used for wastewater corrosion protection. The generic types and target thicknesses are presented in Table 3; actual thicknesses may vary slightly on in situ panels. As a control, one uncoated (blank) concrete cylinder panel was simultaneously exposed to the environment. The panels were removed after 12 months' constant exposure and evaluated for permeation resistance and visible degradation.

The performance measures of the candidate protective coating system for this in situ study are based on the retained properties of permeability as well as a visual inspection of the film. The importance of these performance properties are explained below.

• *Permeability Analyses*—Protective coatings act as a barrier material separating the corrosive service environment from the substrate. Coatings which allow low permeation are assumed to offer better substrate protection within severe wastewater headspace environments. For the coated steel panels, permeability is measured using EIS analysis techniques to quantitatively measure the

Table 3: Field Exposure Panels, Typical

	Description	DFT (mils)*
Steel	Polyamide Epoxy, 2 coats	12
	Polyamide Epoxy Coal-Tar, 2 coats	18
	Fiber-reinforced Polyamine Epoxy	75
	Aromatic Polyurethane Hybrid	75
	Novolac Epoxy, 2 coats	12
	Polyamine Epoxy Mortar	125
Concrete**	Polyamide Epoxy, 2 coats	12
	Fiber-reinforced Polyamine Epoxy	75
	Novolac Epoxy, 2 coats	12
	Aromatic Polyurethane Hybrid	75
	Polyamine Epoxy Mortar	125
	Concrete Control (uncoated)	n/a

* Target dry film thickness (DFT) for each panel.

** All panels received parge coat of an epoxy cementitious resurfacer to fill bugholes and level surface prior to topcoating.

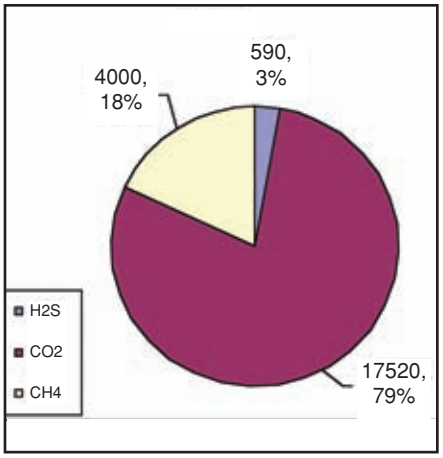

Fig. 5: Mean gas mixture South TX (ppm)

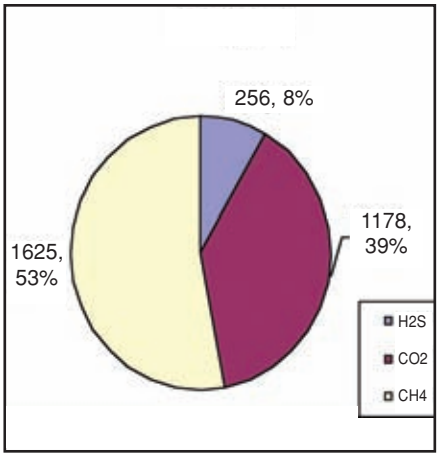

Fig. 6: Mean gas mixture, Midwestern U.S. (ppm)

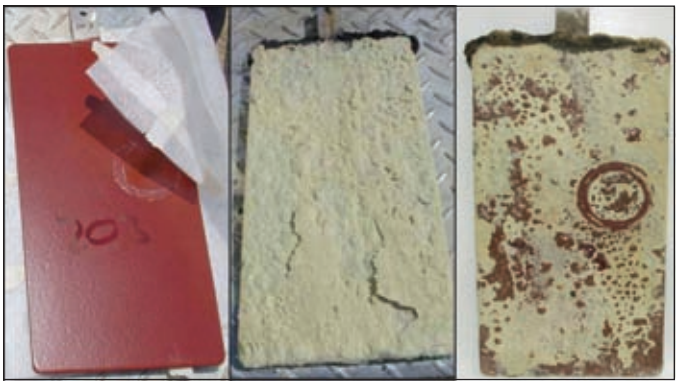

Fig. 7: South TX: 55% solids by volume polyamide epoxy applied at 11 mils DFT to carbon steel panel before 12-month exposure (left), following 12-month exposure (middle), following 12-month exposure, cleaned (right). Note heavy sulfur crust (middle) and ubiquitous blistering on cleaned panel (right).

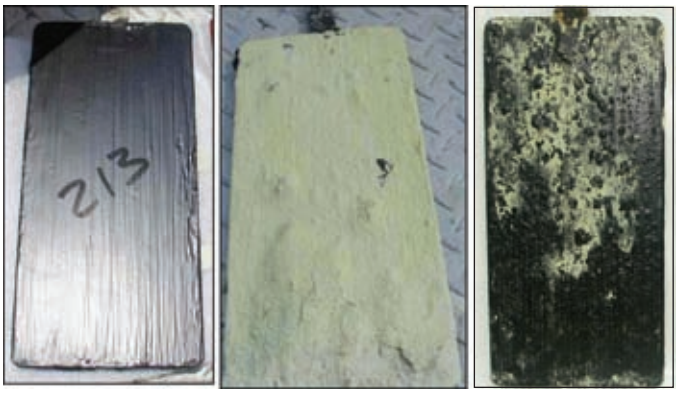

Fig. 8: South TX: A 75% solids by volume coal tar epoxy applied to carbon steel panel at 22 mils DFT before 12-month exposure (left), following 12-month exposure (middle), following cleaning (right). Note heavy sulfur crust (middle) and extensive blistering on cleaned panel (right).

polymer's barrier protection determined by the polymer's electrical resistance. EIS measurements are taken before and after approximately 365 days field exposure. A reduction in the EIS impedance measurement suggests the polymer is negatively affected, via permeation, by the severe wastewater headspace environments.

• Permeation was also measured on the cross sections of the coated concrete cylinder specimens using digitally enhanced optical microscopy. When the cross section of the coated concrete cylinder is viewed through a 100X stereo microscope with digital imaging, permeation is observed as discoloration of the film.

• *Visual Inspection*—Protective coatings should not blister, check, crack, or allow corrosion of the substrate when exposed to severe wastewater environments. Polymers that retain film quality are assumed to offer better substrate protection. Visual inspection of the panels is conducted using ASTM methods for rating blistering, rusting, checking and cracking.

A brief description of the one year exposure data from the South TX and Midwestern U.S. field sites is presented below. Due to limited space, specific data from the other sites is being withheld from this article but influence the authors' overall conclusion.

The South TX in situ testing site is the most severe testing site in terms of abnormally concentrated sewer air and accelerated pace of corrosion that was and continues to take place there. Although it was known previously that the H_2S levels in the headspace were elevated, municipal employees had not attempted to detect other sewer gases. When the authors began testing in 2007 one of the top priorities was to gain a better picture of the sewer gas mixture that was responsible for attacking their particular manhole structure.

Fig. 9: Midwestern US: Polyamide epoxy applied to carbon steel panel at 11 mils DFT before 12-month exposure (left) and following 12-month exposure, cleaned (right). Note moderate blistering following exposure.

Fig. 10: Midwestern US: A 75% volume solids coal tar epoxy applied to carbon steel panel at 15 mils DFT before 12-month exposure (left) and following 12-month exposure, cleaned (right).

The average of five H_2S readings in South TX was 590 ppm, which is considered by the authors as extremely elevated, even for severe wastewater headspaces (Fig. 5). In addition, the averages of CO_2 and CH_4 were also extremely concentrated registering an average of 17,520 ppm and 4,000 ppm, respectively. This particular mixture of sewer gases has proven to be highly corrosive with the three key gases ostensibly working together to penetrate protective coatings and deteriorate both concrete and steel panels.

In Midwestern U.S., the site chosen was an influent channel at the city's main wastewater treatment plant. The influent structure is typical of similar treatment facilities and was chosen to represent a typical sewer gas mixture. Unlike the South TX testing site, the Midwestern U.S.

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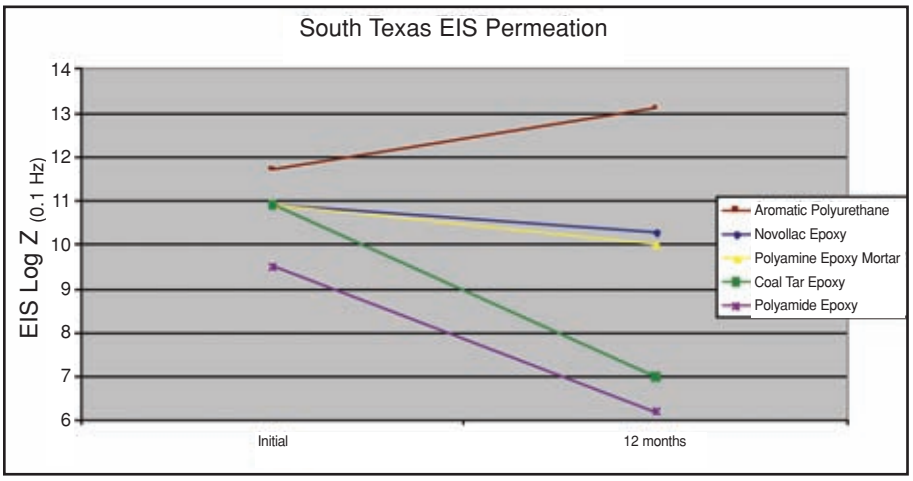

Fig. 11: Initial and 12-month EIS Analysis on steel panels exposed in situ.

site only appeared to experience moderate corrosion to uncoated concrete and cast iron surfaces. But septic wastewater entering the wastewater treatment plant influent channel contributed to consistently high concentrations of H_2S gas, regularly ranging from 200–250 ppm.

The average of four H_2S readings in Midwestern U.S. was 256 ppm (Fig. 6). Although considerably lower than South TX this is still high enough to be considered by the authors as a severe level. It is slightly higher (8%) than the average H_2S level from the entire test samples (3%). The other gases measured at considerably lower concentrations than South Texas and slightly lower than the averages. CO_2 registered at 1,176 ppm and CH_4 at 1,625 ppm.

The panels were removed from their respective sites for evaluation following twelve months exposure (Figs. 7 and 8). One notable difference between the panels was the heavy yellow insoluble sulfur precipitate (crust) on the South TX panels (Figs. 9 and 10). The surface pH was measured above 4.0 on these panels, similar to Midwestern US panels not exhibiting sulfur crust, again, suggesting that sulfuric acid formation is extremely dilute.

The steel panels coated with the polyamide epoxy were observed blistering from both sites following 12 months in situ exposures. This corresponds to the substantial drop in impedance reflected in Figs. 11 and 12. The coal tar epoxy panel exhibited blistering and a

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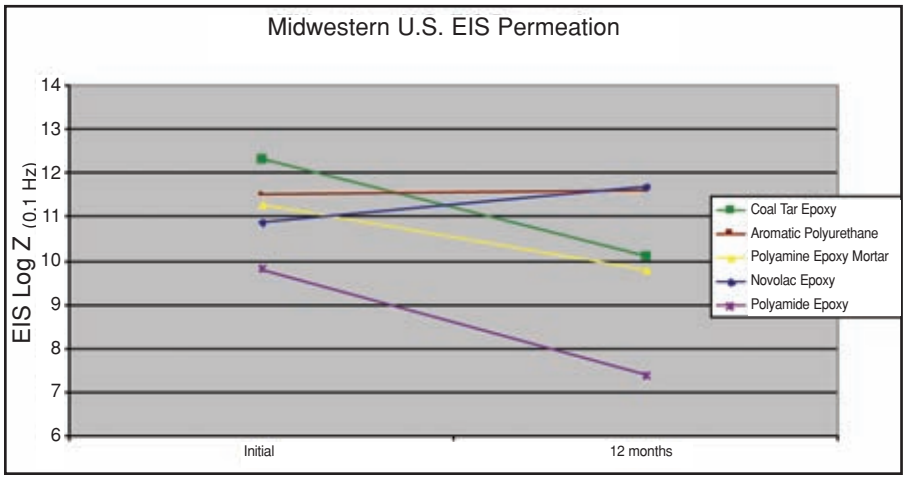

Fig. 12: Initial and 12-month EIS Analysis on steel panels exposed in situ.

Fig. 13: South TX: 55% volume solids polyamide epoxy applied at 17 mils DFT to concrete cylinder panel before 12-month exposure (left) and following 12-month exposure (right).

sharp drop in EIS from the South TX site, but only discoloration and a flatter drop in impedance were observed from the Midwestern US site. The other products, from both sites, showed much higher retained impedance and no signs of visual degradation following 12 months. The panels will be evaluated following an additional 12 months' exposure.

Permeation was also conducted on the coated concrete specimens using optical microscopy analysis of the coated cross section of the panel. Measurement points were taken at four, evenly divided locations (e.g., 3, 6, 9, 12 o'clock positions) circumferentially along cross section (Fig. 13). A 100X stereo microscope with digital imaging measured permeation of the film, via discoloration, at each of the four measurement points (Fig. 14 and Table 4).

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Table 4: Polyamide EP Concrete Panel: South TX

Position (o'clock)	Permeation (mils)	Total DFT (mils)
3	4.69	18.3
6	3.38	20.5
9	4.04	16.3
12	3.04	15.7
AVG.	3.79	17.3

known about how other sewer gases work together to permeate a coating and reduce its ability to protect the substrate. It is the authors' belief that hydrogen sulfide, carbon dioxide and methane act synergistically with dilute sulfuric acid to permeate protective coatings as well as the underlying substrates they protect. Field studies in various wastewater collection structures have demonstrated that higher concentrations of sewer gases are linked to increased rates of failure of organic coatings and subsequent corrosion of the substrate. Therefore, based on the authors' research, the most important factor in the success of a protective coating is its ability to resist permeation of gases, primarily. The authors' research continues and will be reported later. For more details on the research to date, contact the authors.

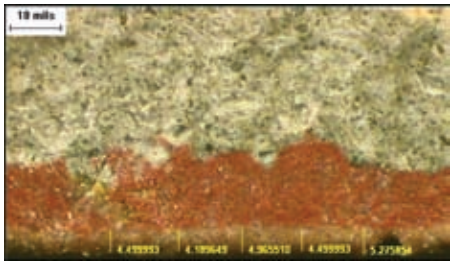

Fig. 14: South TX: Optical microscopy measurements of permeation at the 3 o'clock position of the polyamide epoxy cross section.

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