

# High-Temperature Crude Oil Testing of Tank Linings

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Two novolac epoxy tank linings were exposed to acidic crude oil for 180 days at 160 °C according to NACE Standard TM0174. Both coatings passed the test without visible failure and with good adhesion (ISO 4624). Tests indicated that today's coating systems will be able to handle the ever increasing demand for high-temperature acidic crude oil exposure. We novolac epoxy tank linings, one solvent borne and one solvent free, were exposed to acidic crude oil for 180 days at 160 °C according to NACE Standard TM0174.<sup>1</sup> The solvent-borne tank lining was applied in three coats of 100  $\mu$ m dry film thickness (DFT) and the solvent-free tank lining was applied in one coat of 300  $\mu$ m DFT. Both coatings passed the test without visible failure and with good results.

# **Current Standards** and Test Methods

High-temperature crude oil testing can basically be done in two ways; static or cyclic. Static testing implies that the coated test panels are exposed to crude oil at a set temperature for a specific number of days. In cyclic testing, one usually varies the temperature during the test period, often from extreme cold to extreme hot.

Static testing in itself can be done in different ways. The simplest way is to immerse coated steel panels in jars containing crude oil and placing these jars in heated cabinets. The panels can be partially or fully immersed depending on whether one is interested in the gas phase exposure. When it comes to crude oil, care must be taken as the low boiling temperature components might cause a dangerous build-up of pressure; so from a safety viewpoint, this kind of testing cannot be done at the highest temperatures.

When using the jar method, the panels experience the same temperature from all sides; however, for life-size storage tanks, the outside of the tank is exposed to the surrounding cold air. This temperature difference causes a cold-wall effect that accelerates the permeation of the crude oil's chemical constituents into the coating.

The more realistic immersion test is based on NACE Standard TM0174. This standard describes the use of glass cells where two panels are fixed to a tubular glass cell (Figure 1). Figure 2 shows the test cell with its basic components. In this test, only one side of each steel panel is coated and exposed to the heated fluid. The uncoated side is exposed to air in ambient conditions. This will cause the cold-wall effect and temperature flux in the steel, which makes this the test of choice when it comes to accurately simulating actual conditions.

In this test, there will always be a portion of the panel that is exposed to the gas phase in the cell. In some cases, the exposure conditions in the gas phase are more aggressive than in the liquid phase, as the substances found here are comprised of smaller molecules, which migrate easily into the paint film. Also, this area is more exposed to water vapors and water condensation, especially when oil temperatures are ≤100 °C. Another factor the coating has to withstand is the continuous stream of distilled water coming down from the condenser. Experience has shown that problems (blistering) first occur at the transition between the gas and liquid phases, either just above or just below the liquid surface (or both).

# Suitable Paint Technology for High-Temperature Crude Oil Exposure

Crude oil is not among the most aggressive chemicals or mixtures, and most two-component epoxy coatings can easily handle normal crude oil exposure at ambient temperatures. Other requirements are needed for more acidic crude oils at higher temperatures. When both chemical resistance and heat resistance are required at once, such as is needed when handling an acidic crude oil at high temperatures, a novolac epoxybased coating is the number one choice. The word "phenolic" is often incorrectly used as a synonym for novolac. A novolac epoxy can be described as phenolic epoxy as it is derived from a phenolic-



(a) The NACE TM0174 test panel schematic. Square panels, 200 by 200 by 5 mm, are usually used. The holes in the panels are used to affix the two panels to the glass cell. (b) The circular area of the panel is exposed to the crude oil during the test period.



oxy as it is derived from a phenolic- A test cell is fitted with a coated steel panel. The glass cell is filled so that the formaldehyde resin, but a phenolic liquid level is just beneath the heater and thermometer intakes.

### COATINGS & LININGS



A solvent-borne novolac epoxy tank lining applied in three 100- $\mu$ m coats. The coating was exposed to acidic crude oil at 160 °C.

FIGURE 3(b)

A solvent-free novolac epoxy tank lining applied in one 300- $\mu$ m coat. The coating was exposed to acidic crude oil at 160 °C.

epoxy is not necessarily a novolac epoxy. Several different phenolic epoxy binders exist, which are derived from other sources. The main technology base for a tank lining is epoxy with various degrees of quality, ranging from "standard" solid bisphenol A-based epoxy to novolac epoxy resins with higher functionality. For crude oil exposure at lower temperatures, and for crudes with low acidity, solid epoxy-based tank linings might do the job. With higher temperatures (>90 °C), however, a novolac resin-based tank lining should be used. These epoxy resins are typically cured with some type of cycloaliphatic- or benzyl-amine, with at least two reactive amine hydrogens to get sufficient crosslinking.

Another important factor to define is the DFT. For a solvent-borne epoxy, it is important that the DFT is not too thick to avoid trapped solvents, which can cause problems later on. The recommended DFT for a solvent-borne epoxy tank lining would be two coats that are 125 µm thick (or three coats that are 100 µm thick if one prefers to have three coats). For a solvent-free epoxy tank lining, solvent entrapment is not a concern. One needs only to apply a sufficiently thick coat to give protection. A DFT of one coat that is 300 µm thick or two coats that are 200 µm thick should give adequate protection.

# Test Results and Discussion

Two coatings, with the technical composition described previously, were tested according to NACE Standard TM0174 with an acidic crude oil (harding: 20.7 API, 0.59 wt% S, 2.95 mg potassium hydroxide [KOH]/g) to which 2% water and 1 mL sulfuric acid ( $H_2SO_4$ )/1 L crude oil was added. The solvent-borne novolac epoxy tank lining was applied in three coats of 100 µm DFT. The first coat was light red, the second coat was yellow, and the third coat was light grey (Figure 3[a]). The solvent-free novolac epoxy tank lining was applied in one coat of 300 µm DFT in a grey color (Figure 3[b]).

The coated panels were cured for seven days at 23 °C and then exposed to the crude oil mixture for 180 days at 160 °C. The surrounding temperature varied between 18 and 22 °C. After 180 days, the cells were dismantled and pull-off adhesion was tested according to the ISO 4624 pull-off adhesion test.<sup>2</sup> Figure 3 shows both panels after exposure and pull-off testing.

For the solvent-borne tank lining, it clearly can be seen from the pull-off holidays that the break is cohesive and in the first coat (light red). For the solventfree tank lining, the adhesion breaks are mostly cohesive except for some adhesive failure between the coating and the epoxy adhesive in the gas phase.

### Conclusions

Both coatings show some discoloration, which was worse for the solventfree type. Color retention, however, is not considered a critical factor in this regard. Some discoloration is to be expected after half a year's exposure to crude oil at 160 °C.

The tests indicated that today's novolac coatings will be able to handle high-temperature acidic crude oil exposure.

### References

- NACE Standard TM0174-2002, "Laboratory Methods for the Evaluation of Protective Coatings and Lning Materials on Metallic Substrates in Immersion Service" (Houston, TX: NACE International, 2002).
- 2 ISO 4624:2002, "Paints and varnishes pull-off test for adhesion" (Geneva, Switzerland: ISO, 2007).

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