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Alkyd Coatings Chemical Testing

NRC Presentation August 22, 2007



ALION
SCIENCE AND TECHNOLOGY

August 2007



Outline

- Background
- Testing Methodology
- Experimental details
- Test results
- Summary of key observations to date



Background

- NRC has expressed concerns that leaching from unqualified Alkyd coatings could contribute to the post-LOCA water chemistry.
- EPRI provided Alion samples of Alkyd OEM coatings from several plants.
- Alion performed a series of tests with the EPRI Alkyd OEM samples as well as a commercially available Alkyd.



Background (cont.)

Chemical & Physical Properties of Alkyds

- Alkyd coatings are manufactured from acid anhydrides and polyols.
- Alkyds are readily attacked by NaOH (alkaline hydrolysis, also known as “saponification”) which causes the polymer and cross-linking bonds to break.



Testing Methodology

- 1. Separate effects:** Immersion testing at various temperatures (140 – 280 °F), duration (30 min - 30 days), and buffer chemistries (TSP, STB, NaOH)
- 2. Integrated tests:** Combinatorial experiments with insulation and/or plant containment materials (e.g., Al, Zn, concrete, latent dirt/dust, etc.)



Testing Methodology (cont.)

1. **No buffer:** 2800 ppm Boron
2. **Sodium Hydroxide (NaOH):**
pH ≥ 9
3. **Trisodium Phosphate (TSP):**
pH 7.2 & 8.5
4. **Sodium tetraborate (NaTB):**
pH 8.4



Beaker/flask tests (<212 °F)



“Autoclave” (>212 °F)



Experimental Details – Test Matrix

Test	Material*	Test Duration	Buffer (g/L)	pH target	Temperature (°F)
209-9	Alkyd	30 min.	H ₃ BO ₃ only (16.0)	-	280
209-10	Alkyd	12 hr.	TSP (3.2)	7.1-7.3	250
210-1	Alkyd	24 hr.	TSP (24.2)	8.3-8.5	200 °F continuous
210-2	Epoxy	24 hr.	TSP (24.2)	8.3-8.5	200 °F continuous
210-3	Al, Zn, concrete, alkyd, dirt, Nukon	24hr.	TSP (3.2)	7.1–7.3	200 °F continuous
210-4	Al, Zn, concrete, alkyd, dirt, Mineral Wool	24 hr.	TSP (3.2)	7.1–7.3	200 °F continuous
211-2	Alkyd*	30-day	TSP (24.2)	8.3-8.5	200 °F continuous
211-3	Epoxy*	30-day	TSP (24.2)	8.3-8.5	200 °F continuous
211-4	Alkyd*, Nukon	30-day	TSP (24.2)	8.3-8.5	200 °F continuous
211-5	Epoxy*, Nukon	30-day	TSP (24.2)	8.3-8.5	200 °F continuous
211-6	Alkyd OEM	30-day	TSP (24.2)	8.3-8.5	200 °F continuous
211-7	Epoxy OEM	30-day	TSP (24.2)	8.3-8.5	200 °F continuous
220-1	Alkyd*	30-day	NaOH (4.6)	8.8-9.1	200 °F continuous
220-2	Alkyd*	30-day	NaTB (10.58)	8.0-8.5	200 °F continuous
220-3	Alkyd OEM	30-day	NaOH (4.6)	8.8-9.1	200 °F continuous
220-4	Alkyd OEM	30-day	NaTB (10.58)	8.0-8.5	200 °F continuous

* Commercially available paint



Experimental Details

- All immersion/exposures tests conducted in borated water (2800 ppm Boron)
- 0.7 ppm Lithium added in most instances (no overall effect observed on pH or chemistry with presence of Lithium)
- Unqualified coatings consisted of EPRI supplied OEM components and commercially available paint
 - Ameron Amercoat 5450 White Gloss Alkyd Enamel

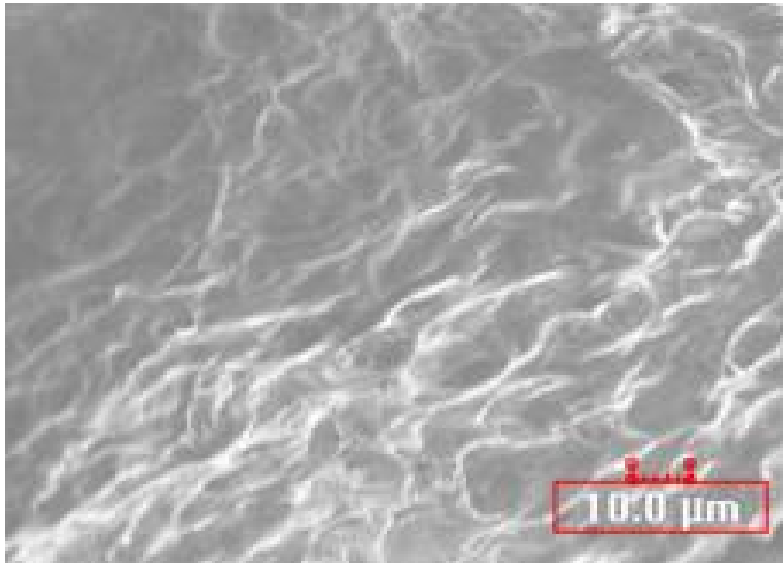


Experimental Details (cont.)

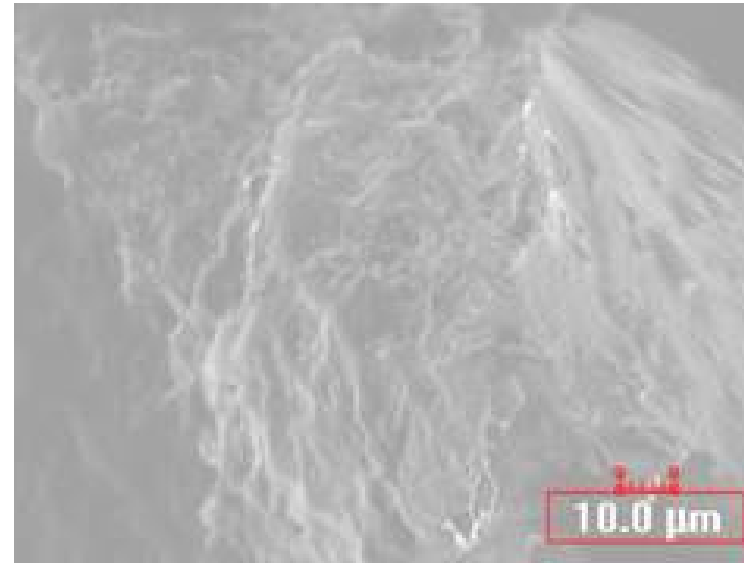
- The coatings were left intact or scraped off the OEM components while the commercially-available paints were applied according to manufacturer recommendations (single coat on non-stick surface and scraped; or Al strip dipped and cured for 24 hrs. at room temperature)
- Sample sizes ranged between 0.1 (autoclave) and 0.5 grams (beaker/flask experiments)
- Specimens for SEM-EDX were dried in the oven unless otherwise specified



Test results – OEM Alkyd at high temperature



Autoclave testing at 280 °F
for
30 min. in 2800 ppm Boron
(H_3BO_3)

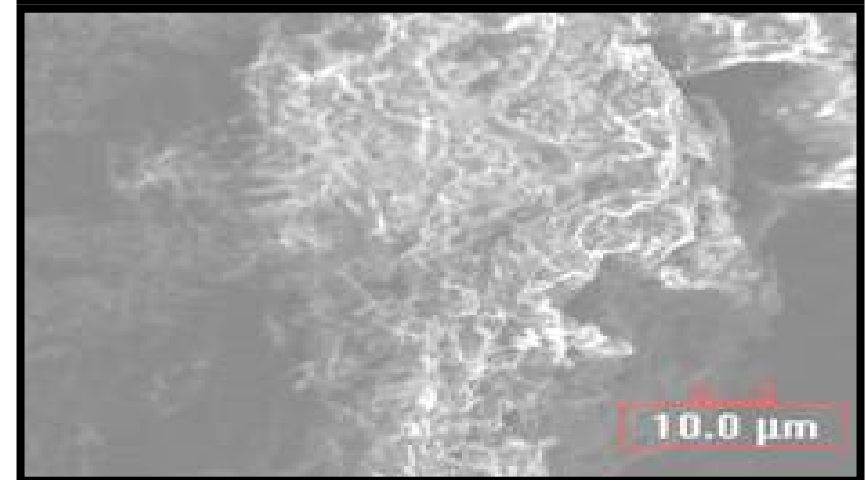
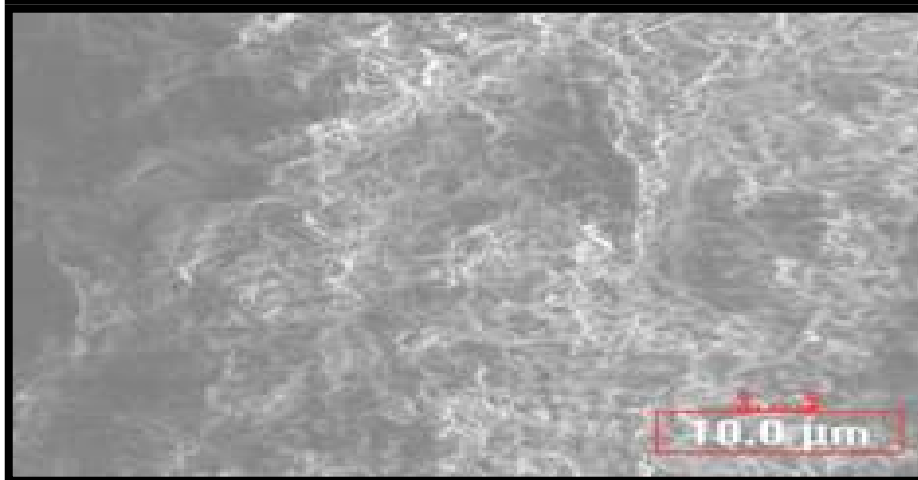
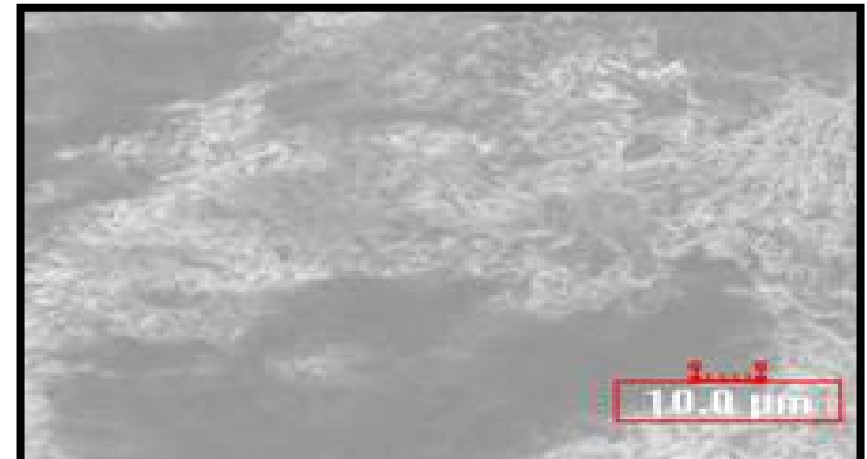
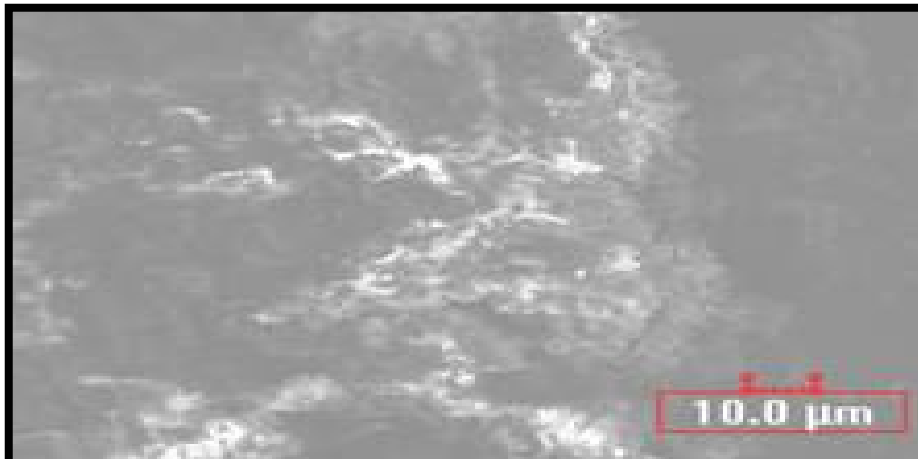


Autoclave testing at 250 °F
for 12 hrs. in TSP (w/ 2800
ppm B from H_3BO_3)

Minor morphological changes with temperature and buffers



Test results – Alkyd integrated tests w/ Al, Zn, concrete, latent dust and insulation in TSP at 200 F for 24 hrs.



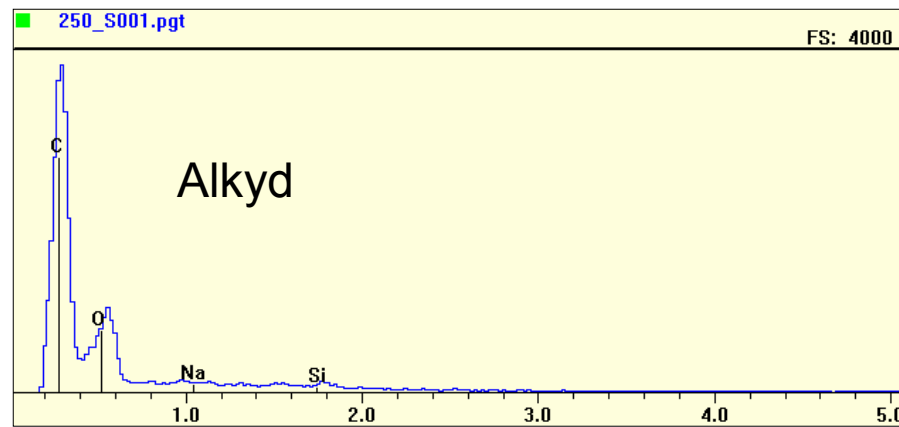
As-is (above) and post-testing (below)
w/ Nukon

As-is (above) and post-testing (below)
w/ Mineral wool

No significant changes to coatings in presence of other test materials



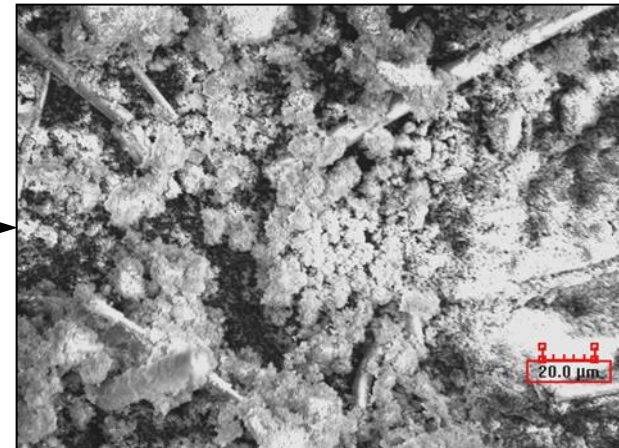
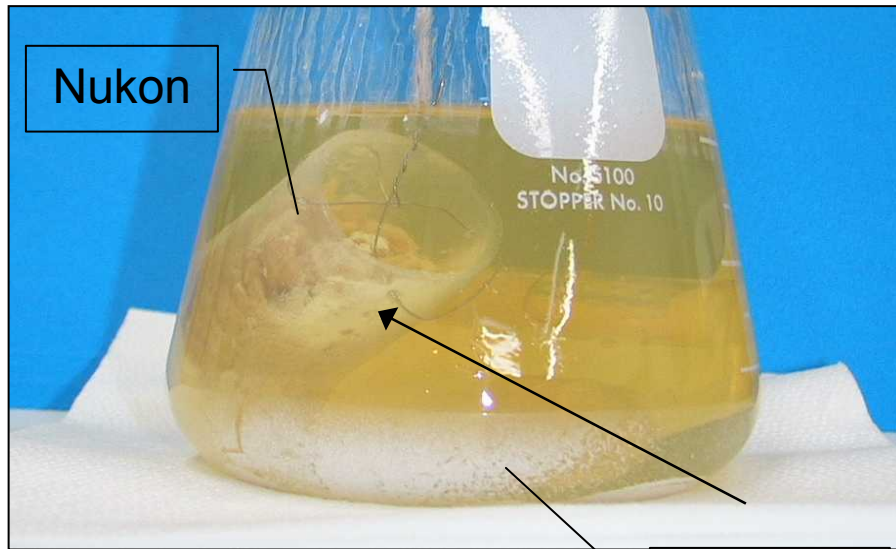
Test results – Initial Observations



- Both EPRI and commercial Alkyds readily degraded in the presence of post-LOCA fluid environment.
- SEM-EDX of Alkyds reveal primarily organic coatings with trace levels of Si, Na
- No significant differences between Commercial and EPRI Alkyds
- Both types of Alkyds contain pigments and other additives e.g. binders and driers.

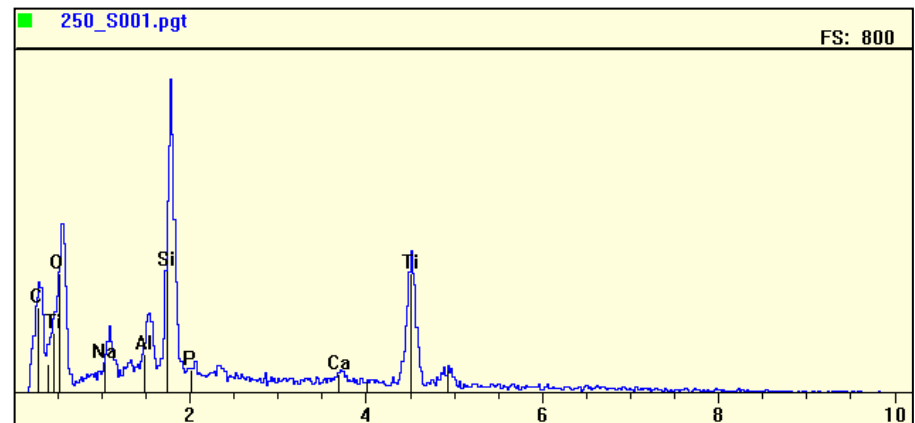


Test results – Commercial Alkyd & Nukon insulation in TSP (pH 8.3-8.5) at 200 °F for 30 days



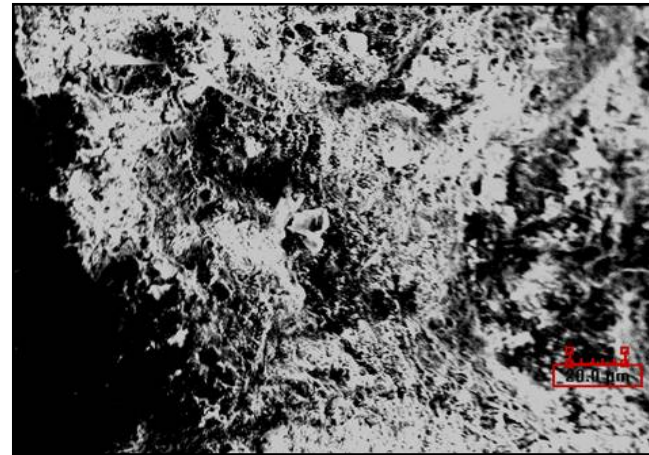
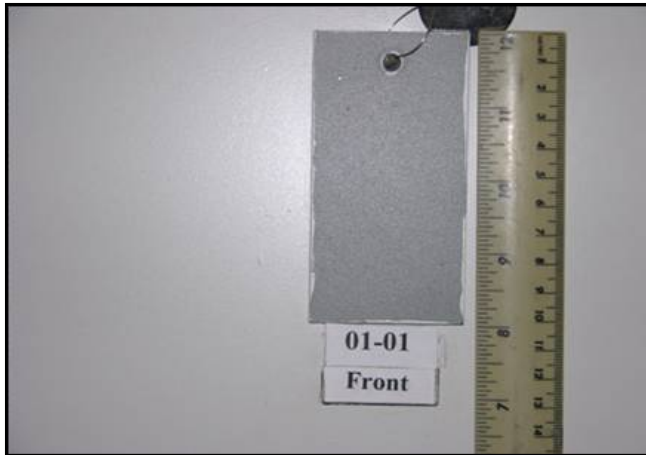
Alkyd
fine
sediment

- Paint chips immediately begin to swell & liquefy
- Solution discoloration; and release & dispersion of fine particles observed
- Analysis of fine sediments reveal primarily Ti, O, Si, Al, Na (pigment, fillers, driers)





Test results – EPRI alkyd OEM in TSP (pH 8.3-8.5) at 200 °F for 30 days

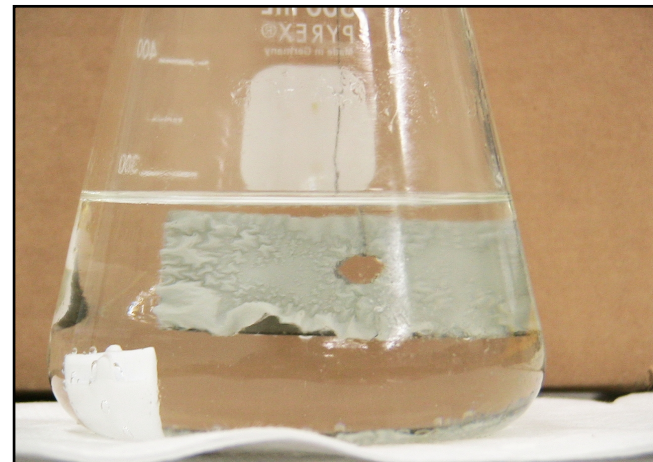


Alkyd OEM completely dissolved from coupon.



Test results – EPRI Alkyd OEM comparison between TSP (pH 8.3-8.5), NaOH (pH 8.8-9.1), & STB (pH 8.0-8.5) at 200 °F for 30 days

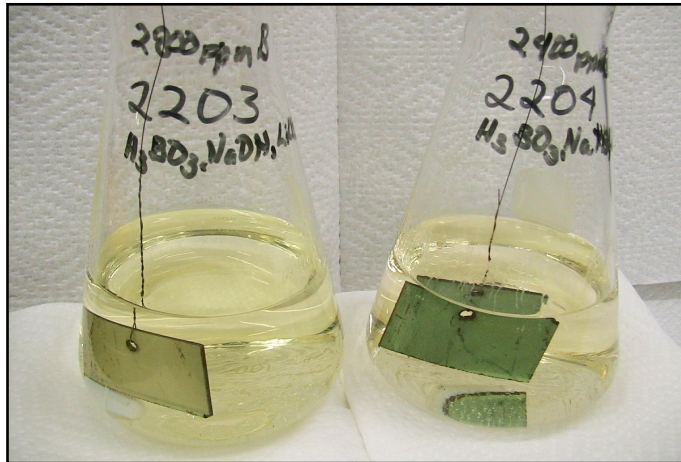
- **Bubbling and swelling on coatings observed**
- **Complete detachment after 30 days in TSP**
- **Degradation appears to be dependent on temperature effects and coating properties**



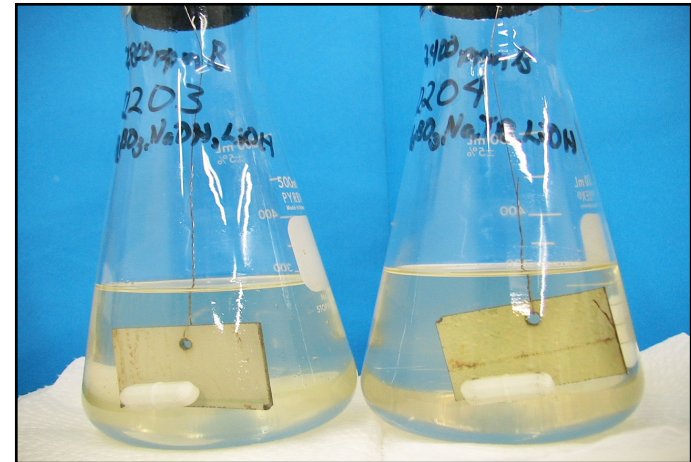
After 16 days in TSP



Test results – EPRI Alkyd OEM comparison between TSP (pH 8.3-8.5), NaOH (pH 8.8-9.1), & STB (pH 8.0-8.5) at 200 °F for 30 days



**After 5 days in NaOH
(left) & STB (right)**

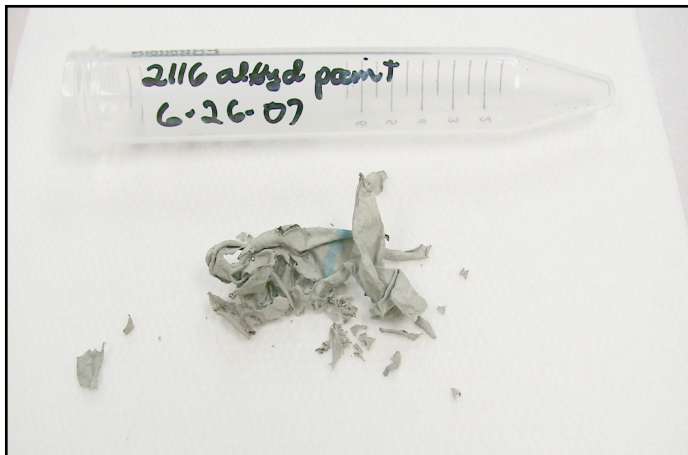


**After 19 days in NaOH
(left) & STB (right)**

- **Bubbling and swelling on coatings observed**
- **Some swelling/bubbling observed for both NaOH and TSP (previous slide) with solution and coating discoloration**
- **Degradation appears to be dependent on temperature effects and coating properties**



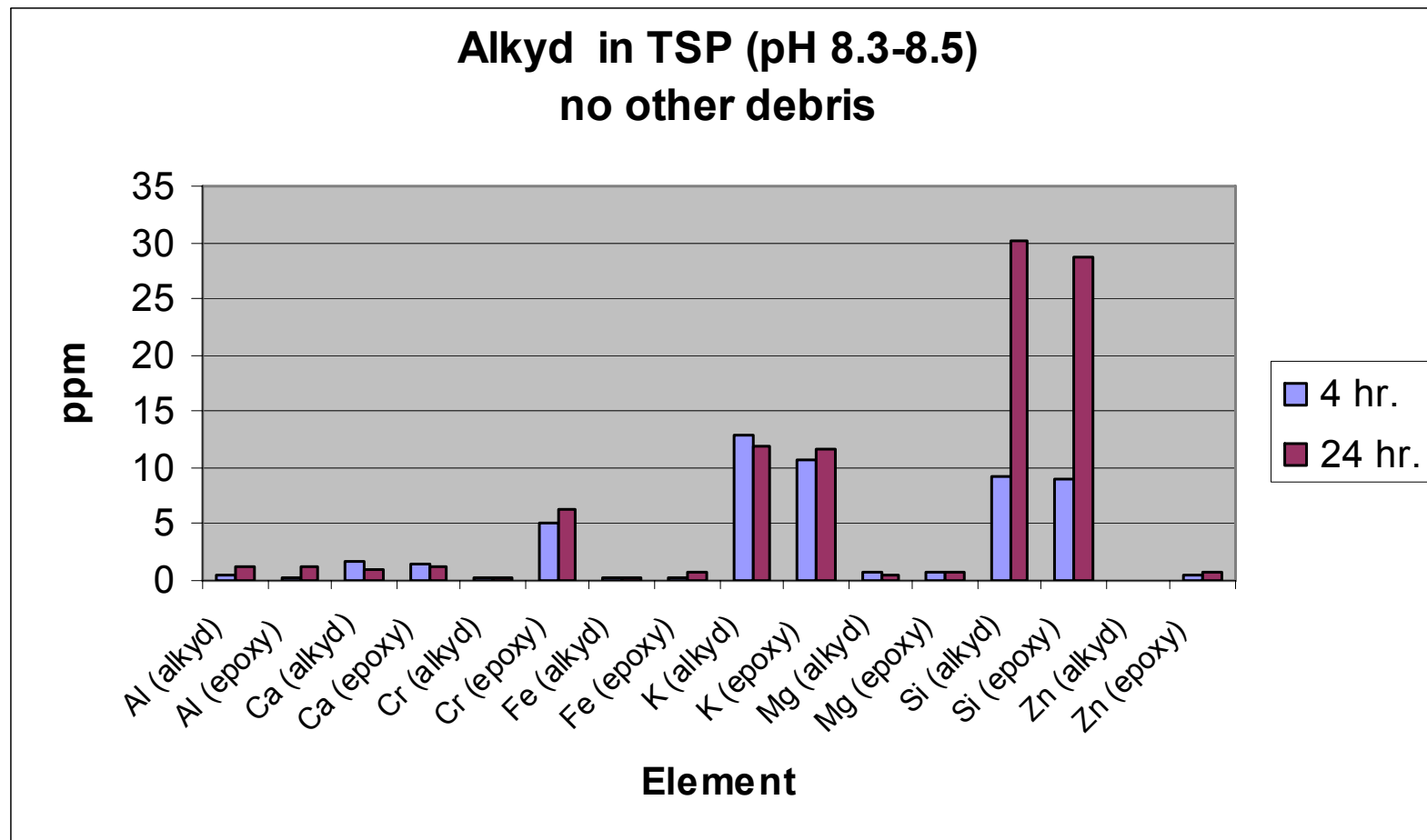
Test results: EPRI OEM alkyd in TSP (pH 8.5) at 200 °F for 30-days



Alkyd delaminated coating and clean underlying substrate after 30-day exposure.

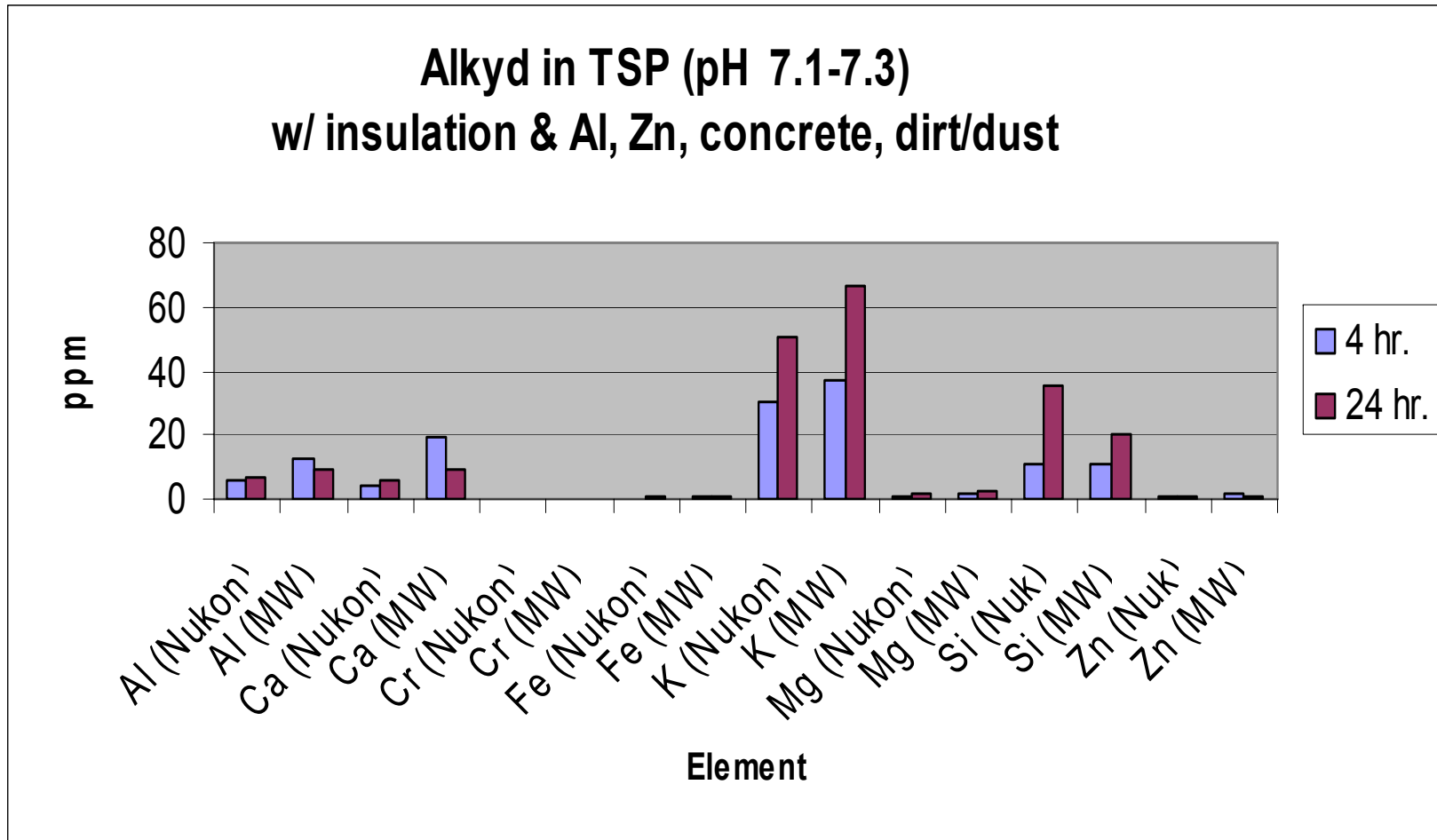


Test results – ICP data – No Other Debris





Test results – ICP data – With Other Debris





Key Observations to Date

- Alkyds start decomposing into oils and particulates after exposure to post-LOCA liquid environment and temperatures.
 - Time to complete decomposition varied from days to weeks
 - No significant variations due to different buffers
 - No significant variations due to presence of debris and other containment materials
- Oils migrated to the surface and formed a thin layer of “scum.”
- Particulates settled.
 - Particulate sizes varied from pigment particulates to “chips”
- ICP of fluid indicates minimal elemental contribution by decomposing Alkyds by-products.