

TURNER-FAIRBANK HIGHWAY RESEARCH CENTER



## Detection of Phosphoric Acid And Trace Metals in Asphalt Binders

Federal Highway Administration  
Turner-Fairbank Highway Research Center (TFHRC)  
McLean, VA

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## Susan P. Needham Test for Phosphate in Asphalt


- No special equipment or expertise needed
- Requires a few inexpensive chemicals
- Test is rapid and simple
- A blue color is developed after 5 minutes
- Test can detect 0.1% PPA in asphalt
- Details available on TFHRC Website:  
<http://www.tfhrc.gov/>



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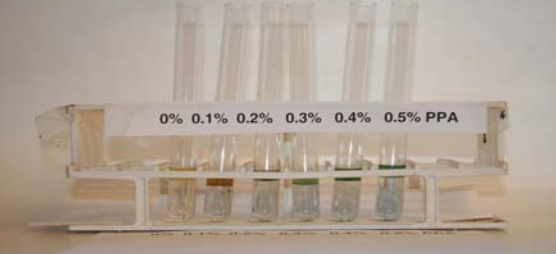

## Test for Phosphate in Asphalt

- Quantities are not critical
- Put 1 gram of hot asphalt into a 1oz can
- Add 1ml of n-butanol and swirl/stir in the can
- Add 2mls water
- Add 1 ml of ascorbic acid/ammonium molybdate solution
- A blue color is developed in the water phase within a few minutes.
- **A positive result does not necessarily mean phosphoric acid is present**



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## Test for Phosphate in Asphalt


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## XRF-Spectrometer




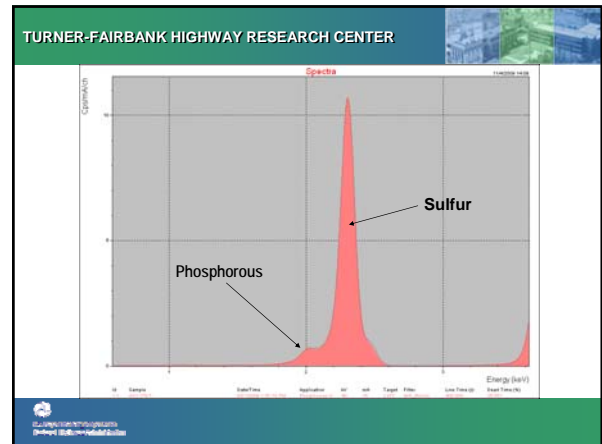

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## THE PERIODIC TABLE OF THE ELEMENTS



The Analytical X-ray C

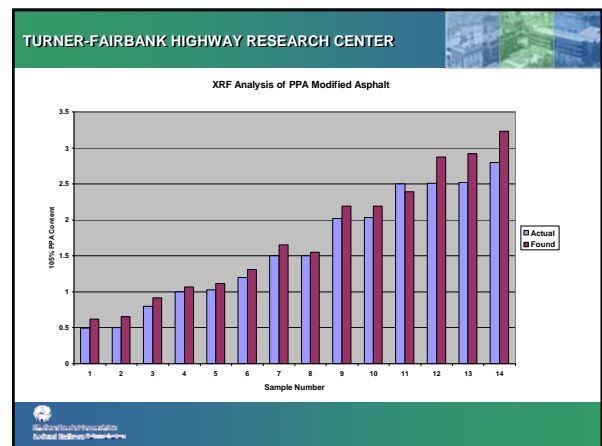
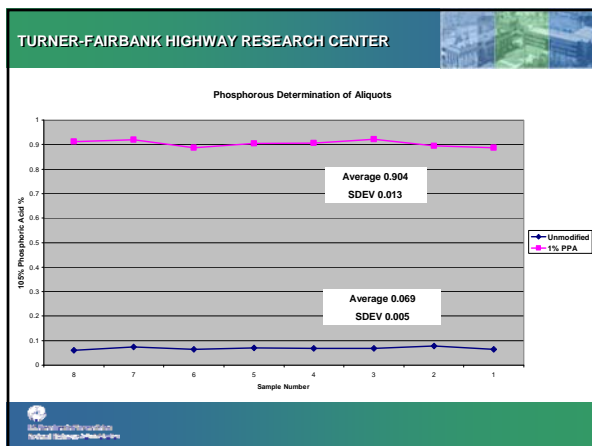
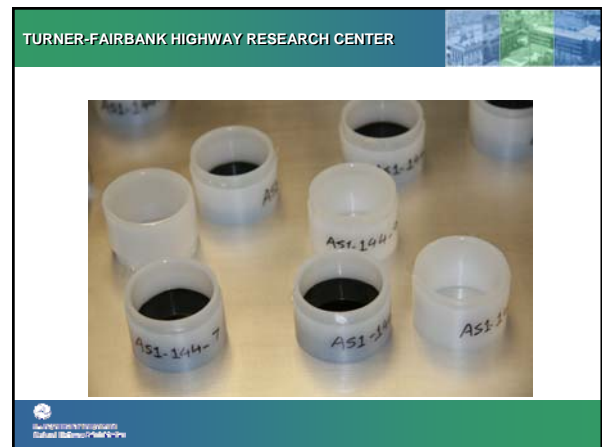


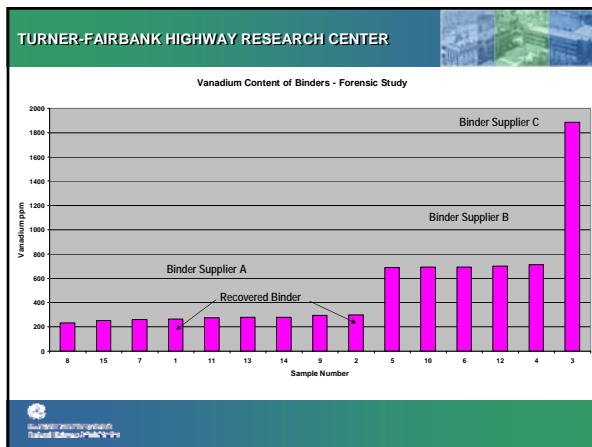
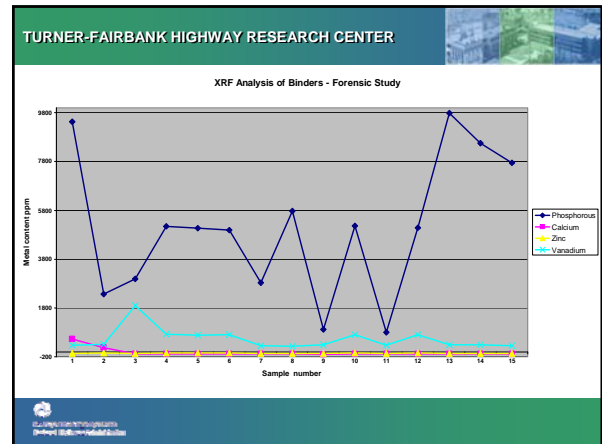
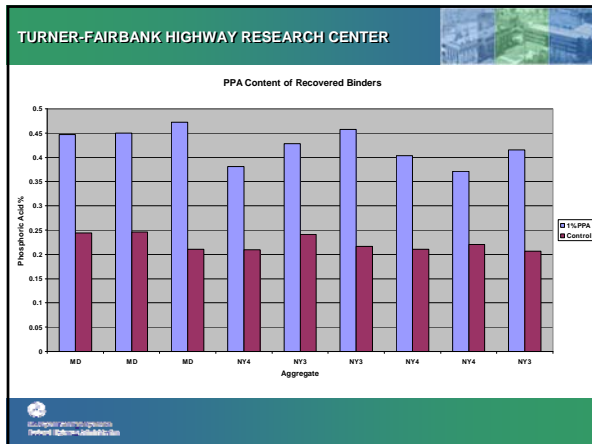


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### Sulfur Range Determined by Elemental Analysis

1.1% to 6.4%





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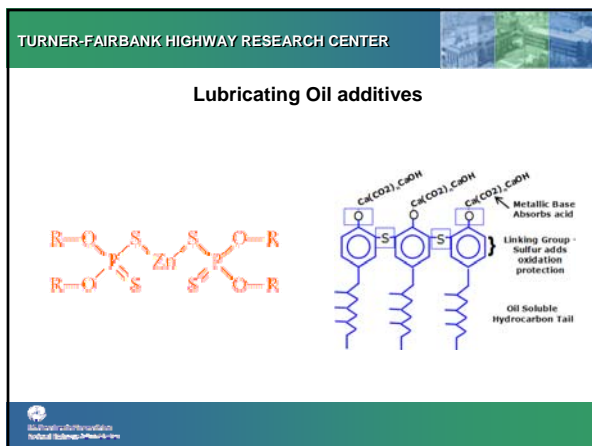
#### We need your Help!

We do not have enough samples to verify the fingerprint test.

We would like to receive samples of binders that have been exposed to aggregate (recovered binders, cores or gyratory specimens) along with samples of the original binder (that has NOT been exposed to aggregate).

Please send samples to :

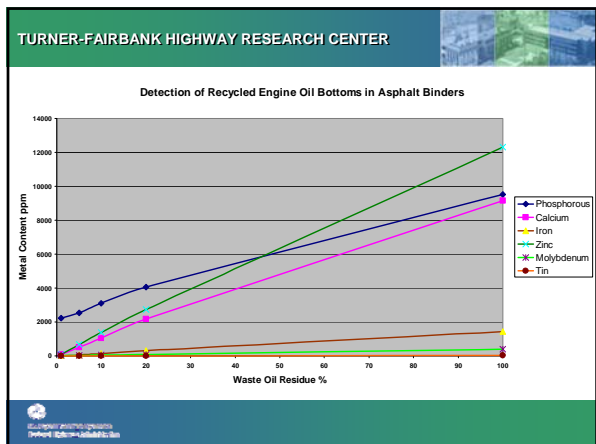
Terry Arnold  
Federal Highway Administration  
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HRDI-11  
6300 Georgetown Pike  
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#### Metal content of Recycled Engine Oil bottoms (REOB) ppm

	Evergreen Oil	Safety-Kleen
Organics	862390	884260
Sulfur	54490	43280
Calcium	33180	28790
Phosphorous	22940	17870
Zinc	12990	10730
Aluminum	4300	0
Silicon	3410	6100
Chlorine	1782	1925
Iron	1342	3150
Molybdenum	981	549
Potassium	948	2020
Copper	591	626
Lead	307	229
Tungsten	259	0
Titanium	51	111
Bromine	45	16
Barium	39	43
Chromium	33	100
Manganese	29	81
Nickel	22	41
Tin	13	19
Scandium	11	9
Strontium	10	14
Zirconium	0	23
Antimony	0	16



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### Forensic Analysis

Sample	PPA %	Ca ppm	Zn ppm	REOB %	Confirmed by	Vanadium ppm
1	0.303	96	94	Trace	Mo Sn	176
2	0.338	564	737	10	Cu Mo Sn Ba Pb	178
3	0.337	241	65	1-2	Cu Mo Sn Ba	180
4	0.616	14	12	Trace	Mo Sn	184
5	0.61	0	0	0		192
6	0.296	270	390	5	Cu Mo Sn Ba Pb	199
7	0.605	0	0	0		205
8	0.303	356	471	3	Cu Mo Ba Pb Sn	223
9	0.415	1182	1604	10-12	Cu Mo Sn Ba	236
10	0.292	364	490	3	Cu Mo Ba	248
11	0.351	669	1107	7-8	Cu Mo Sn Ba Pb	250
12	0.338	771	1191	8	Cu Mo Sn Ba Pb	262
13	1.138	0	0	0		301
14	1.067	0	57	07	Cu Mo Sn	301
15	0.247	0	0	0		1498

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- ### Conclusions
1. Phosphoric Acid content of asphalt binders can be readily measured using XRF Spectroscopy.
  2. The method is well established, developed by Puzic, Reinke and Glidden
  3. Not all the PPA is recovered from mixes
  4. Presence of recycled Engine Oil Bottoms can be detected by measuring Calcium and Zinc levels.
  5. Confirmation of REOB by trace metals Cu, Mo, Sn, Ba and Pb
  6. Measurement of Vanadium levels shows promise as an asphalt fingerprint
  7. More Research is needed

