
Standard Specification for

DETERMINATION OF PERCENT PHOSPHOROUS AS POLYPHOSPHORIC ACID IN ASPHALT BINDER USING XRF SPECTROPHOTOMETER

1. SCOPE

- 1.1 This test method describes the quantitative analysis of asphalt binder for % Phosphorus as Polyphosphoric Acid (PPA) using a wavelength dispersive x-ray fluorescence spectrometer equipped with a monochromator specific for detection of phosphorus.
- 1.2 The values stated in SI units are to be regarded as the standard.

2. REFERENCED DOCUMENTS

- 2.1 IDOT Chemistry Laboratory Test Method # XRAY 06-08
- 2.2 AASHTO T-40, Standard Method of Test for Sampling Bituminous Material

3. SUMMARY OF TEST METHOD

- 3.1 An 11-point calibration curve is prepared by mixing a known amount of PPA-free asphalt binder with a known amount of standard Polyphosphoric Acid (PPA with 105% equivalent H₃PO₄) standard PPA. Analyze the standards in the XRF spectrometer and plot intensity readings, cps (counts per second) vs concentration in %Phosphorus as PPA, to obtain a calibration curve. Determine % Phosphorus as PPA of the asphalt test specimen using the prepared calibration curve.

4. SIGNIFICANCE

- 4.1 Phosphoric acid may be added to asphalt binder to modify the physical properties of the binder. This test method determines the amount of phosphorus present in the asphalt binder.

5. APPARATUS

- 5.1 Wavelength dispersive x-ray fluorescence spectrometer (XRF).
- 5.2 Pipette—Disposable plastic transfer pipettes.
- 5.3 Containers—8 oz. cans or small beakers. Disposable cans or glassware recommended to avoid contamination. If glassware is reused, it must be washed with hot (1+3) hydrochloric acid (1 part hydrochloric acid to 3 parts distilled water) and rinsed with distilled water. Commercial detergents *should not be used* to clean the glassware as they may contain phosphates which will interfere with the results.

Note 1—One agency found the cans they were using for this test had been lubricated during the manufacturing process with a lubricant containing phosphates. This resulted in a positive test result when the can was empty. If there is doubt about the condition of the containers being used, then take the precaution of running a blank test.

- 5.4 Mixer—Mechanical, variable velocity laboratory stirrer/mixer

6. REAGENTS AND MATERIALS

- 6.1 *Polyphosphoric acid (PPA) 105% equivalents*
- 6.2 *Asphalt Binder*

7. SAMPLING

- 7.1 Sample the material in accordance with AASHTO T-40, Sampling Bituminous Materials

8. PROCEDURE

8.1 Preparation of Standards for Calibration Curve:

The following procedure is for the preparation of an 11-point calibration curve.

8.1.1 Choose a base asphalt binder that is known to be free of PPA.

8.1.2 Heat the quart can of base asphalt binder (AC) at 280°F (138°C) for several hours until soft.

8.1.3 Place the container of standard polyphosphoric acid (PPA with 105% equivalent H₃PO₄) in an oven or any warm chamber at 95°F (35°C) for several hours until it turns into a viscous liquid. PPA has a boiling point of 300°F (149°C) and a freezing point of 84°F (29°C)

8.1.3.1 Points for calibration curve:

<u>~ % PPA</u>	<u>W of AC, g</u>	<u>W of PPA, g</u>	<u>W total, g</u>
0.0	100.0	0.0	100
0.2	99.8	0.2	100
0.4	99.6	0.4	100
0.6	99.4	0.6	100
0.8	99.2	0.8	100
1.0	99.0	1.0	100
1.2	98.8	1.2	100
1.4	98.6	1.4	100
1.6	98.4	1.6	100
1.8	98.2	1.8	100
2.0	98.0	2.0	100

8.1.4 Tare an empty 8 oz metal cup. Using a syringe or a disposable pipet, add the required amount of PPA. Record the weight to the nearest 0.001 g.

8.1.5 Tare again and add the required amount of AC. Record the weight to the nearest 0.001 g. Based on these weights, calculate the exact % PPA (to the nearest 0.01%) in each of the standard samples.

8.1.6 While maintaining the temperature at 280°F (138°C), mix well until the solution is homogeneous or until the PPA is completely blended with the AC. It may be stirred with a mechanical stirrer for ~ 15 minutes.

8.1.7 Immediately pour the homogeneous solution into a 32-mm disposable aluminum sample cup. Allow to cool to room temperature. Then place the aluminum sample cup into a 42.7mm plastic Chemplex™ XRF sample holder and then cover the plastic holder with sample with a polypropylene pre-cut 63.5mm film..

8.1.8 Analyze the standards in the XRF spectrometer and plot intensity readings, cps vs concentration in %Phosphorus as PPA, to obtain a calibration curve using linear regression. Correlation coefficient should be 0.9950 or better.

8.2 Sample Analysis:

8.2.1 Heat the unknown asphalt binder at 280°F (138°C) for several hours until soft.

8.2.2 Immediately pour into a 31-mm disposable Aluminum Chemplex™ sample cup. Allow to cool to room temperature.

8.2.3 Place sample in 35mm plastic Chemplex™ sample cup and cover with polypropylene film. Analyze the sample in the x-ray spectrometer and determine %Phosphorus as PPA using the calibration curve prepared above.

9. REPORT

9.1 *The report shall include the following:*

9.1.1 Identification of sample

9.1.2 Percent Phosphorus as PPA detected by Xray diffraction

10. PRECISION AND BIAS

10.1 *Precision*—The research required to develop precision estimates has not been conducted.

10.1.2 *Bias*—This method has no bias since the values determined can only be defined in terms of this test method.

11. KEYWORDS

11.1 Asphalt; PPA; XRF