ARIZ 509a
September 5, 1996
(6 Pages)

RAPID DETERMINATION OF ASPHALTENES AND CHEMICAL REACTIVITY OF ASPHALTS

(An Arizona Method)

SCOPE

1. (a) This method describes a procedure for the determination of Asphaltene, (A), content and Chemical Reactivity Ratio, (CRR), of asphalts. It is not intended to supplant the complete asphalt analytical methods, but should be used when only the A and CRR values are needed.

(b) This test method involves hazardous material, operations, or equipment. This test method does not purport to address all of the safety concerns associated with its use. It is the responsibility of the user to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

(c) See Appendix A1 of the Materials Testing Manual for information regarding the procedure to be used for rounding numbers to the required degree of accuracy.

APPARATUS

2. Requirements for the frequency of equipment calibration and verification are found in Appendix A3 of the Materials Testing Manual. Apparatus for this test procedure shall consist of the following:

(a) Erlenmeyer flasks with glass stoppers, 125 mL capacity.

(b) Analytical balance capable of measuring the maximum weight to be determined and conforming to the requirements of AASHTO M 231, except the readability and sensitivity of any balance or scale utilized shall be at least 0.0001 gram.

(c) Glass funnels, 75 mm top diameter.

(d) Hot plate, with variable heat control.

(e) Beakers, Griffin type, 250 mL capacity.

(f) Funnel support.

(g) Infra-red heat lamp, variable intensity.

MATERIALS

3. (a) Filter paper, Whatman Glass Microfibre, 12.5 cm, Grade GF/A.

(b) Normal pentane, (nC_5H_{12}), practical grade.

(c) Petroleum Ether, 30 to 60 °C, reagent grade

(d) Concentrated Sulfuric Acid, reagent grade, (H_2SO_4 Assay: 95-98%)

(e) Sodium Hydroxide, pellets, reagent grade.

CAUTION: Pentane and Petroleum Ether are extremely flammable solvents. Smoking or open flames should not be permitted in the area while they are in use. With the exception of weighing, all operations should be carried out in a functioning fume hood. Protective goggles, gloves and laboratory apron shall be worn.

PROCEDURE

4. (a) Weigh approximately 1 gram of the asphalt into a tared 125 mL Erlenmeyer flask. Weigh to the nearest 0.1 mg and record as S_f (weight of sample and flask).

(b) Add 75 mL of normal pentane to the flask and dissolve the asphalt completely. Frequent shaking during the first 15 minutes will aid in dissolving the asphalt. If it does not dissolve, warm cautiously until the asphalt is in solution.

(c) Allow the solution to settle overnight.

(d) Stir vigorously with a glass stirring rod to initiate precipitation, and allow to settle until clear.

(e) Carefully decant liquid through filter paper into a 250 mL beaker, using the Glass Microfibre filter paper. Wash the precipitate remaining in the flask 3 times with 15-20 mL portions of pentane, each time decanting liquid into filter paper. Thoroughly wash flask with pentane and add washings to filter paper. Thoroughly wash precipitate on filter paper with pentane until discoloration is removed from the filter paper. Discard paper containing the precipitate.

NOTE: It is very important that the filter paper be kept wet throughout the washing and filtering process in step (e).

(f) Thoroughly wash and dry the flask used in this operation. Methylene Chloride washing is recommended.

(g) Using the heat lamp, evaporate the solution in the beaker until approximately 25 mL remains. Pour solution into the 125 mL flask previously used. Wash the beaker with pentane until clean, adding the washings to the flask.

(h) Evaporate the contents of the flask with the heat lamp until all the solvent has been removed. Then place on the hot plate and heat cautiously until a white vapor is noticed at the top of the flask.

(i) Remove the flask from the hot plate. Cool and weigh to the nearest 0.1 mg. Record this weight as B_p (weight of flask and residue).

(j) Add 75 mL of Petroleum Ether to the flask containing the residue and dissolve ALL the residue.

(k) Pour the solution into a 250 mL beaker and thoroughly wash the flask with Petroleum Ether. Add the washings to the solution in the beaker. If necessary, reduce the volume of the solution to 100 - 125 mL by evaporation with the heat lamp.

(l) Thoroughly wash and dry the flask. Methylene Chloride washing is recommended.

(m) Slowly add 10 ± 0.5 mL concentrated sulfuric acid to the solution in the beaker while stirring vigorously. Continue stirring for at least 3 minutes.

(n) Allow the solution to settle until clear.

(o) Cover bottom of a 250 mL beaker with Sodium Hydroxide pellets, then immediately filter the liquid through a filter paper into the 250 mL beaker containing the Sodium Hydroxide pellets. Do not allow the sludge to leave the beaker. Wash the sludge in the beaker 3 times, using 15 - 20 mL portions of Petroleum Ether. Filter the washings into the pellets in the beaker, then discard filter paper.

CAUTION: Do not touch the pellets with bare hands.

CAUTION: Sludge contains large amounts of unreacted acid. Contact with Sodium Hydroxide pellets may cause explosion.

(p) Using the heat lamp, evaporate this solution to approximately 50 mL, then filter into the 125 mL Erlenmeyer flask previously used, thoroughly washing beaker and pellets into filter paper, using a 30-40 mL portion of Petroleum Ether.

(q) Discard the filter paper containing the Sodium Hydroxide pellets.

(r) Evaporate the contents of the flask with heat lamp until all the solvent has been removed. Then place on the hot plate and heat cautiously until a white vapor is noticed at the top of the flask.

(s) Remove the flask from the hot plate. Cool and weigh to the nearest 0.1 mg. Record this weight as "C" (weight of flask and residue).

CALCULATIONS AND REPORT

5. (a) Percent Asphaltenes:

$$A = \frac{S_f - B_p}{S} \times 100$$

Where:
S = Weight of sample, $S_f - T_f$
S_f = Weight of sample and flask, grams
T_f = Tare weight of flask, grams
A = Percent Asphaltenes.
B_p = Weight of flask and residue from Pentane washing, grams

(b) Percent Unreacteds:

$$U = \frac{C - T_f}{S} \times 100$$

Where: U = Percent Unreacteds.
C = Weight of flask and residue from Petroleum Ether washing, grams
S = Weight of Sample, grams
T_f = Tare weight of flask, grams

(c) CRR (Chemical Reactivity Ratio) Value:

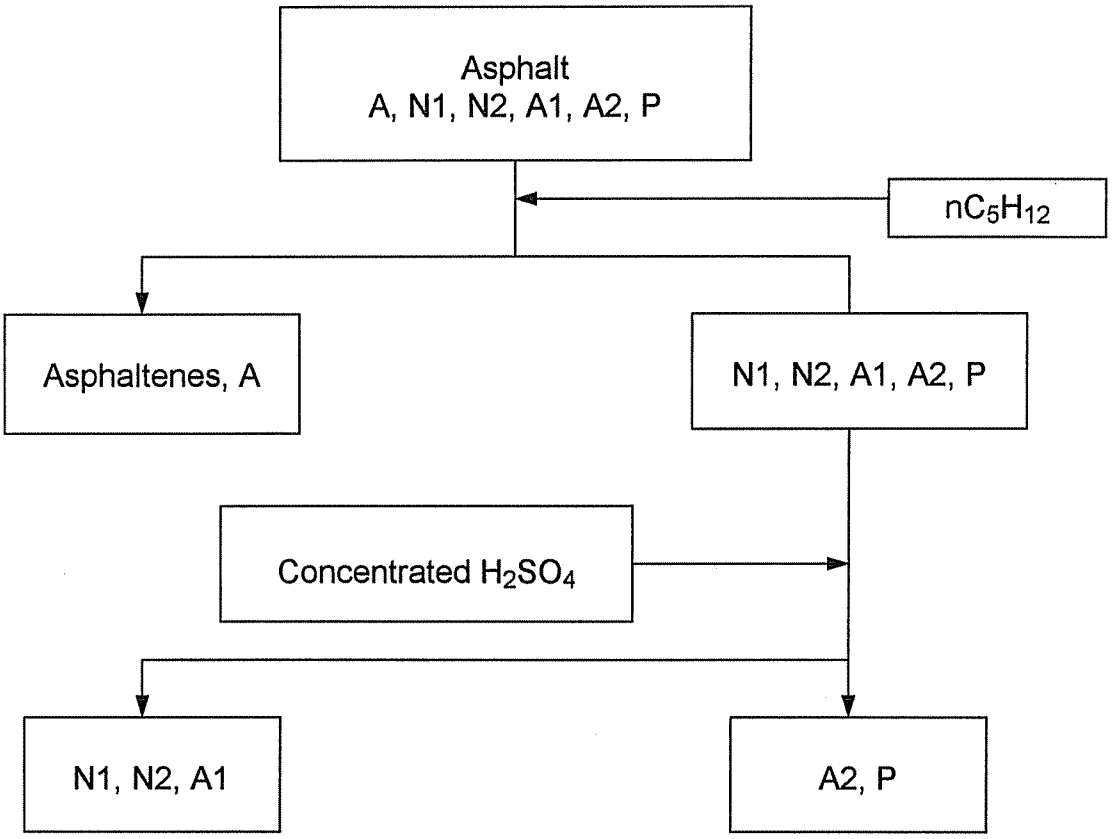
$$CRR = \frac{100 - (A + U)}{U}$$

Where: CRR = Chemical Reactivity Ratio
A = Percent Asphaltenes
U = Percent Unreacteds.

(d) Report Asphaltenes as a percentage of the asphalt sample, to the nearest 0.01%.

(e) Report CRR Value as a ratio of the Reacted and Unreacted asphalt components, to the nearest 0.01 unit, if this value is required.

FLOW SHEET - MODIFIED ROSTLER-STERNBERG ANALYSIS



NOTE:

A	=	Asphaltenes	Insoluable in nC_5H_{12}
*N1	=	1st Nitraffins	Precipitated by concentrated H_2SO_4
*N2	=	2nd Nitraffins	Precipitated by concentrated H_2SO_4
A1	=	1st Acidaffins	Precipitated by concentrated H_2SO_4
A2	=	2nd Acidaffins	Unreacted by concentrated H_2SO_4
P	=	Paraffins	Unreacted by concentrated H_2SO_4

* 1st and 2nd Nitraffins are Nitrogen Bases