TESTING OF THERMOPLASTIC PAVEMENT MARKING MATERIAL

(An Arizona Method)

SCOPE

1. (a) Thermoplastics to be tested according to the procedures given herein are mixtures of resins, mineral fillers, pigments, organic additives, and reflective glass spheres.

(b) All testing is done on hot melt test specimens made from the commercial products. These include field test specimens obtained from highway striping operations and specimens produced according to Part I of this method.

(c) This method is divided into two parts: Part I gives a procedure for producing hot melt test specimens. Part I also gives an alternate procedure for producing hot melt test specimens in accordance with AASHTO T 250. Part II gives the procedures for testing specimens.

(d) This test method involves hazardous material, operations, and 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.

(e) 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.

(f) Requirements for the frequency of equipment calibration and verification are found in Appendix A3 of the Materials Testing Manual.

PART I - PROCEDURE FOR PRODUCING HOT MELTS

APPARATUS

2. (a) Heating mantle - Glas-Col Model 620 or equivalent, capable of accepting a 4000 mL beaker.

(b) Stainless steel beaker - 4000 mL, Vollrath No. 84000 or equivalent, for use with heating mantle.

(c) Mixer - Variable-speed, air-driven, Cole-Parmer No. E-04685-00 or equivalent.

(d) Mixer stand - Cole-Parmer No. E-04546-00 or equivalent with 60 cm height and capability of holding mixer.

(e) Adjustable chuck - Cole-Parmer No. E-04423-00 or equivalent, capable of coupling mixer shaft to impeller assembly shaft.

(f) Impeller assembly - Any assembly with a 35-40 cm shaft of 0.5 cm minimum diameter, dual impellers of 8-10 cm diameter spaced 4-6 cm apart with one at the end of the shaft, capable of efficient mixing of 3000-3500 mL of fluid at 700-800 rpm. Alternatively, a heavy-duty "Jiffy" type impeller for one gallon paint containers is suitable.

(g) Temperature controller with thermocouple probe - Glas-Col Digitroll II, No. 104A PL612, with 6 in. type "J" iron-constantan thermocouple or equivalent, capable of controlling mixture temperature in conjunction with heating mantle at 218 °C.

(h) Ladles - Stainless steel, 30 mL and 60 mL capacity with long handles for sampling hot melts.

(i) Aluminum or stainless steel lid to cover beaker, with holes for impeller shaft and thermocouple, and turned-down edges for holding in place over beaker.

PROCEDURE

3. (a) Mount the mixer, with impeller assembly and lid, onto the mixer stand and note position of mixer which locates bottom of impeller assembly about 2-3 cm from bottom of beaker. Secure the mantle to the mixer stand in such a position as to permit the impeller assembly to be placed over the center of the beaker and to prevent relative movement between the impeller assembly and the beaker. Plug the mantle cord into the controller. Plug the controller into a wall outlet.

(b) Remove the mixer with impeller assembly and lid. Split out a 6000 gram sample of thermoplastic material and add enough to the beaker to come to within 5-6 cm of the top. Replace the mixer, impeller, and lid. (It may be necessary to first work the impeller assembly into the thermoplastic, and then replace the lid and mixer). Place the thermocouple in position through its hole in the lid, making sure it penetrates

into the thermoplastic. Set the controller parameters for automatic control of temperature at 218 °C, and start the heating cycle.

(c) When the thermoplastic has melted sufficiently, start the mixer at a low speed. As the temperature continues to rise, increase the mixer speed until it is evident that the entire mass of thermoplastic in the beaker is being turned over continuously. (Note this mixer speed for future use). Continue heating and mixing while periodically checking the level of thermoplastic in the beaker. If the level drops below 5-6 cm from the top of the beaker, add more thermoplastic as necessary. Continue this process for four hours after the material has reached the final temperature of 218 °C.

(d) At the end of the four hour period, turn the mixer off and remove it with the lid and impeller assembly. Immediately use the ladles to transfer enough of the melt to fill a set of suitable can lids, usually a one-gallon can lid and two one-quart can lids. Set the lids with the test specimens aside for cooling. These will be used for all of the tests of the thermoplastic material.

(e) Use heat resistant gloves to remove the beaker from the mantle and discard the remainder of the thermoplastic. Allow the beaker to drain completely while the material is hot.

Note: Alternatively, the procedure for producing hot melt test specimens for Reflectance, Color, and Yellowness Index as given in AASHTO T 250 may be used, if desired. If AASHTO T 250 is used, the amount of material specified may be increased to approximately 500 grams.

PART II - TESTS

REFLECTANCE AND YELLOWNESS INDEX

4. The apparatus and procedure for determining reflectance and yellowness is given in Sections 5 through 8.

APPARATUS

5. Reflectance and gloss meter - Photovolt 577 or equivalent, with "T" search head.

CALIBRATION OF METER

6. After an initial warmup time of a minimum of 30 minutes, calibrate the meter for use on the applicable filter channels (green "G", amber "A", and/or blue "B") as follows:

(a) Load the green filter in the filter rack of the "T" search unit and rotate the rack until the filter is in front of the lens.

(b) Select the green memory location by depressing the "G" button on the front face of the meter.

(c) Place the black cavity standard on the head of the search unit.

(d) Set the dark current (zero offset) by depressing first the "CHANGE" button and then the "ZERO" button on the face of the meter.

(e) Place the Photovolt ceramic reflectance standard plaque on the head of the search unit.

(f) Enter the standard reflectance value for the green filter on the meter display, by depressing first the "CHANGE" button, then the "STD" button, and finally, the "A", "B", and "G" buttons as necessary to increment the tens, units, and tenths digits respectively until the value is displayed. Then depress the "STD" button again. The standard reflectance value for the green filter is now stored in the memory, and the meter is now calibrated on the green filter channel.

(g) If reflectance data is to be obtained for a white test specimen, repeat steps 6(a) through 6(f) for the amber filter channel and again for the blue filter channel.

(h) For each channel used, immediately prior to obtaining reflectance data the calibration should be checked and, if necessary, corrected. First replace the black cavity standard on the head of the search unit and if drift has occurred, depress the "CHANGE" button and then the "ZERO" button. Next replace the standard plaque on the head of the search unit and if drift has occurred, depress the "CHANGE" button, and then depress the "STD" button twice.

MEASURING AND RECORDING REFLECTANCE DATA

7. (a) Obtain reflectance data for a white test specimen as follows:

(1) After calibration of the meter on the green filter channel, measure the reflectance of the specimen. Record the result as “Green.”

(2) Repeat step 7(a)(1) for the amber channel. Record the result as "Amber."

(3) Repeat step 7(a)(1) for the blue channel. Record the result as "Blue."

(b) Obtain reflectance data for a yellow test specimen as in step 7(a)(1) for the green channel only.

CALCULATING AND REPORTING REFLECTANCE PROPERTIES

8. (a) Report "Reflectance" of a white or yellow test specimen as the reflectance value "Green" obtained as in 7(a)(1) or 7(b) above, respectively.

(b) Calculate and report "Yellowness Index" of a white test specimen according to the following formula:

$$\text{Yellowness Index} = \frac{\text{Amber - Blue}}{\text{Green}} \times 100$$

BINDER AND GLASS BEAD CONTENT

9. The apparatus, reagents, and procedure for determining binder content and glass bead content is given in Sections 10 through 13.

APPARATUS

10. (a) A balance or scale 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 0.01 gram.

(b) Porcelain crucibles, 30 mL capacity.

(c) Muffle furnace, capable of holding a constant temperature of 538 °C and having a sufficiently large chamber for several crucibles.

(d) Drying oven, capable of holding a constant temperature of 100 °C.

(e) Mortar, 75-100 mL capacity, with pestle.

(f) Dessicator.

(g) Beakers, 400 mL capacity.

(h) Aluminum dishes, 57 mm diameter.

(i) Small spatula.

(j) Balance brush.

(k) Hot plate.

REAGENT

11. Hydrochloric acid solution, 50%, prepared by adding (cautiously) 500 mL concentrated hydrochloric acid to 500 mL demineralized water.

PROCEDURE

12. (a) Break up a test specimen into pieces sufficiently small to permit weighing of material into a crucible.

(b) Weigh a crucible to the nearest 0.01 gram. Record the weight as "C".

(c) Zero the balance and weigh 10.00 ± 0.10 gram of test specimen into the crucible. Record the weight as "S".

(d) Preheat the furnace to 538 °C.

(e) Place the crucible **(cautiously)** into the furnace.

(f) After one hour, remove the crucible from the furnace and place it into the dessicator for cooling.

(g) After cooling, remove the crucible and weigh it, with its contents, to the nearest 0.01 gram. Record the weight as "R".

(h) Use the spatula to scrape the inside of the crucible and quantitatively transfer all of the residue into the mortar.

(i) Using the pestle, gently grind the residue to a fine powder, taking care not to crush the glass beads.

(j) Using the brush, quantitatively transfer all of the powdered residue to a beaker.

(k) Slowly add 150 mL of acid solution to the beaker, taking care to minimize possible splattering due to generation of carbon dioxide.

(l) Stir the mixture by swirling, place the beaker on the hot plate, and heat to boiling with frequently stirring.

(m) Remove the beaker from the hot plate and add 150 mL of demineralized water.

(n) Let stand until the beads settle and decant the suspension, being careful not to lose any beads.

(o) Add about 100 ml of water and repeat step 12(n) until the decanted water is clear.

(p) Place the beaker into the drying oven.

(q) When completely dried, remove the beaker, with beads, from the oven.

(r) Allow the beaker and beads to cool. Weigh to the nearest 0.01 gram and record the weight as "B_1".

(s) Using the brush, quantitatively transfer the beads to a suitable receptacle for microscopic examination, if desired, or to a waste container.

(t) Reweigh the empty beaker to the nearest 0.01 gram. Record the weight as "B_2".

CALCULATIONS AND REPORT

13. (a) Calculate and report the binder content, to the nearest 0.1%, using the following formula:

$$\text{Binder Content, \%} = \frac{C + S - R}{S} \times 100$$

(b) Calculate and report the bead content, to the nearest 0.1%, using the following formula:

$$\text{Bead Content, \%} = \frac{B_1 - B_2}{S} \times 100$$