Technical Bulletin RET2.1
Equipment and processing suggestions for manufacturing polymer-modified asphalt (PMA) for paving, using DuPont™ Elvaloy® RET reactive elastomeric terpolymer.
General Overview

DuPont™ Elvaloy® RET is a reactive elastomeric terpolymer that chemically bonds with asphalt (bitumen) and provides elastomeric characteristics to improve the hot-temperature performance of the road. The low-temperature properties, below approximately 4°C (40°F), of terpolymer-modified asphalt are determined mainly by the base asphalt.

Selection of the base asphalt and laboratory evaluations to determine optimum Elvaloy® RET levels should be completed prior to a production trial. There are two methods for completing the reaction of the Elvaloy® RET in asphalt:

1. The heat-reacted method to complete the chemical bond, time depending on the asphalt binder.

2. A catalyst technology method to speed up the reaction time to 1-4 hours.

If you have not completed the laboratory studies, please refer to the Technical Bulletin RET1.1-Laboratory Formulation of DuPont™ Elvaloy® RET in Asphalt Binder. Typically the Elvaloy® RET terpolymer concentration ranges from 0.8% to 2.0%, depending on the asphalt stiffness desired. Do not increase terpolymer above laboratory levels without first running a lab test to see if the asphalt will gel at the increased levels of terpolymer. Contract labs can perform initial screenings of this process on your asphalt, and will also provide SHRP data for the asphalt.

Equipment Overview

Elvaloy® RET is easily mixed into hot asphalt binder, typically using a vertical tank with a top-down rotating mixer assembly that has enough horsepower to create a vortex at the surface in the center of the tank. The system should be able to heat the asphalt to 185°C - 195°C (365°F - 383°F), depending on the reaction method.

Terpolymer pellet addition and catalyst addition systems are also part of the general equipment layout of a typical Elvaloy® RET plant operation. For more information and guidelines for setting up a commercial mix tank for processing Elvaloy® RET, please see Technical Bulletin RET2.2, DuPont™ Elvaloy® RET Mixing Design Guide.
**Blending Procedure**

Elvaloy® RET can be reacted with asphalt by one of two methods: heat reacting over a long period of time (up to 24 hours); or by the use of a patented catalyst process which will usually reduces the total mix time to less than 4 hours and the terpolymer concentration by some 10% - 20% (depending on the asphalt).

The patented catalyst technology uses polyphosphoric acid (PPA), also known as super phosphoric acid. The PPA is used in a concentration of 105%; however, concentrations greater than 105% can be used successfully. Typically, 0.2% to 0.4% by weight of the asphalt of 105% SPA is used as a catalyst. The optimum catalyst level should be determined in laboratory testing prior to commercial production.

*Note: It is absolutely critical that polyphosphoric acid be used as the catalyst and not standard aqueous phosphoric acid. Severe safety consequences can occur if standard aqueous acid is added, as the residual water will flash immediately, causing the asphalt to foam, potentially resulting in tank over-pressurization.*

The catalyst technology is the sole property of:

MTE Services Inc.  
915 Commercial Ct.  
P.O. Box 563  
Onalaska, Wisconsin 54650  
Phone number (608) 781-4683.

Specific questions regarding the Catalyst Technology should be directed to Mr. Gerald Reinke, President of MTE Services.

**Blend Preparation**

Blend preparation can be divided into five stages:

- Heating the asphalt
- Adding the Elvaloy® RET terpolymer
- Distributing and dispersing the terpolymer (see *Mixing Design Guide*).
- Adding polyphosphoric acid, if using catalyst technology
- Completing the reaction

The very first PMA production run should be a small-scale trial batch of production (~50 tons). This small run will be used to check the accuracy of the lab scale versus actual production scale.

**Heating the Asphalt**

1. Inspect the mix tank. Flush and empty if necessary before adding the base asphalt. The reaction tank should have less than 5% heel. There should be no residue of any other materials other than unmodified asphalt (or previous batch of Elvaloy® RET modified asphalt). Especially no catalysts, acids, caustics, etc. that may have been used to modify other materials. Any residues of these materials can have an adverse effect on the catalyst reaction of the Elvaloy® RET and the asphalt. The base asphalt must not be pre-acidified.

2. Check that the base asphalt meets the cold weather specifications. Remember that Elvaloy® RET modifies the hot temperature properties. The base stock must meet the cold temperature properties.

3. Begin adding the base asphalt to the tank and heat to 190°C (375°F) for the heat-reacted method or 185°C (365°F) for the catalyst technology method.

4. Sample the base asphalt in the blend tank. Measure the viscosity at 135°C (275°F). Check other necessary asphalt properties for baseline values.

5. Turn the mixer/agitator and re-circulation pump on. Look in at the top of the mix tank to be sure the tank is being vigorously agitated and the mixer is creating a vortex or rough churning to draw the pellets into the asphalt. Do not proceed if the tank is not properly agitated. Adjustments may need to be made to the mixer blade design, motor horsepower, or speed to achieve the desired agitation. Other addition methods should be discussed with your DuPont representative.

**Adding Elvaloy® RET terpolymer**

6. For the initial trial, begin adding the Elvaloy® RET at a rate of 10-15 kg (25 - 35 lb) per minute. Material should be added as close to the shaft as possible. Avoid pellets touching the shaft, blades or any other dry hot spot in the tank to prevent pellets from sticking. Look in at the top of the tank periodically to be sure that there is no terpolymer build-up or pellets fusing into clumps floating on the asphalt. This is a
sign of poor agitation and adjustments should be made before proceeding. If terpolymer build-up is seen, shut down the terpolymer addition and allow the residual terpolymer to mix into the asphalt. After the terpolymer is mixed in, start up the terpolymer addition again at a rate that is 2 kg/min (5 lb/min) less than the previous rate. Repeat this step until no terpolymer buildup is detected after 1 hr of continuous terpolymer addition.

7. If no terpolymer build-up is encountered at 15 kg/min (30 lb/min) for 15 minutes, increase terpolymer addition rate by 10 lb/min. Wait 15 minutes. If there is no terpolymer build-up, increase the rate by another 10 lbs. per minute. Repeat this step until either terpolymer build-up occurs, or maximum terpolymer feeder rates are achieved. If a terpolymer build-up is detected, reduce feed rates back to the previous level that did not produce terpolymer build-up. If the build-up continues, discontinue terpolymer addition until the build-up is gone.

8. Note: It is rare to be able to exceed 100 lbs. per minute without having terpolymer build-up

9. Continue adding Elvaloy® RET until the desired amount has been added.

(Note: After the optimum terpolymer addition rate is determined, continue to check this rate over the next few production runs to ensure no build-up forms on the surface of the asphalt blend.)

Distributing and Dispersing the Terpolymer

10. Once all the Elvaloy® RET has been added, look into the top of the tank to ensure that all the terpolymer has been dissolved (no floating pellets). The tank contents should then continue to be agitated, heated, and circulated for the remainder of the reaction time.

11. Sample the tank and run the absolute viscosity at 135°C (275°F). The viscosity measurement should be used to monitor the rate of reaction. Continue with the specific procedures below to complete the Elvaloy® RET reaction process.

Section A: Heat Reacted Method (steps 12-15)

Note: If using the catalyst method, skip to Section B (next page) to steps 16-25

Completing the Reaction

12. Continue monitoring the viscosity every 4 to 6 hours.

13. Once the viscosity reaches equilibrium, the reaction is complete. This usually will take 12 to 24 hours depending on the asphalt, but could be as soon as 4 hours. The terpolymer modified asphalt should be tested to ensure that it meets all the specifications.

14. The PMA can now be pumped to a finished product storage tank. The storage tank should have a gentle mixer to maintain even tank temperatures. The PMA will not separate or settle out of the asphalt since the Elvaloy® RET has been chemically reacted with the asphalt. If longer-term storage of the PMA is needed, the asphalt can be cooled, and re-heated.

15. Before starting another batch of Elvaloy®-modified asphalt, the reaction tank should have less than 5% heel in the tank to prevent gels during Elvaloy® addition. Residue of catalyst, acid, caustics, etc. can have an adverse effect on the catalyst reaction of the Elvaloy® RET and the asphalt.
Section B: Catalyst Technology Method

Note: When using polyphosphoric acid as a catalyst, it is important to keep the acid heated to 60°C (140°F) and to use appropriate materials of construction. Follow recommended safety/handling practices as specified by the polyphosphoric acid suppliers.

Complete steps 1 – 11 (above) in the common procedure.

16. Continue mixing and maintaining tank temperature at 185°C (365°F).

17. Once all the Elvaloy® RET has been added, look in the top of the tank to ensure that all of the terpolymer has been dissolved. (Note: Do not proceed with adding the catalyst until all of the terpolymer has been dispersed.) Dispersion usually takes approximately 1-2 hours after the addition of the terpolymer pellets.

18. Sample the asphalt and take to the lab. Test the sample by adding polyphosphoric acid IN THE LAB. Look for gel formation and compare viscosity or DSR results to previous lab formulation work, to verify the material is within specification. If no gel, then continue with step 19, the acid addition stage IN THE TANK.

Note: It is extremely difficult to disperse Elvaloy® RET in asphalt after the acid has been added due to the reaction that has occurred in the asphalt with the Elvaloy® RET and the polyphosphoric acid.

Adding Polyphosphoric Acid

19. Keep the recirculation flowing, the heat on and mixer running for at least one hour after completing Elvaloy® RET addition.

20. With the mixer running, begin injecting the polyphosphoric acid (PPA) into the top of the tank or into the recirculation line of the tank at the discharge of the recirculation pump. If 0.2% acid (by weight) is desired in the asphalt, inject PPA at a rate near the addition level into the recirculation line. (i.e. if re-circulation flow is 100,000 pph, and 0.2% acid is desired, inject acid at 200 pph).

Warning: Should recirculation flow be lost at any point during the catalyst injection, shut down the acid injection flow immediately. Injecting acid into a no flow asphalt line could cause the asphalt to gel, plugging the line. It is highly recommended that the acid pump be interlocked down on the loss of the recirculation pump.

21. Continue injecting the acid until desired amount has been added. During the acid addition and until the reaction is complete, keep the agitator and recirculation pump running and maintain the temperature at 185°C (365°F).

Completing the Catalyzed Reaction

22. Check the asphalt viscosity at 135°C (275°F) every 1/2 hour to track the reaction rate/time.

DO NOT add any more Elvaloy® to the tank after the PPA is added!

23. Once the viscosity reaches equilibrium, the reaction is complete. This usually takes 30 to 60 minutes after all of the acid has been injected. The PMA should be tested to ensure that it meets all the end use specifications.

24. The PMA can now be pumped to a finished product storage tank. The storage tank should have a gentle mixer to maintain even tank temperatures. The PMA will not separate or settle out of the asphalt since the Elvaloy® RET has been chemically reacted with the asphalt. If longer-term storage of the PMA is needed, the asphalt can be cooled, and reheated.

25. Before starting another batch of Elvaloy®-modified asphalt, the reaction tank should have less than 5% heel in the tank to prevent gels during Elvaloy® addition. Residue of catalyst, acid, caustics, etc., can have an adverse effect on the catalyst reaction of the Elvaloy® RET and the asphalt.

Disclaimer

While DuPont believes that the procedure set forth in this publication can be used to safely and effectively modify asphalt, DuPont does not warrant or guarantee the completeness, efficiency or safety of the process or its use. The information contained herein is intended for use only by competent personnel in designing and operating a facility to modify asphalt, and the user of this information accepts full responsibility for its use. Consistent with the foregoing, anyone using this information shall indemnify and hold DuPont harmless from any loss, claim, damage, or injury to persons or property whatever the cause may be arising out of or pertaining to the use of the information, unless the same is shown by the user to have been caused directly by bad faith or willful misconduct on the part of DuPont. Furthermore this publication is not to be taken as a license to operate under, or a recommendation to infringe, any patent.
For additional information on safety and handling of DuPont™ Elvaloy® RET, please read the Material Safety Data Sheet. If further assistance is needed, contact DuPont Packaging and Industrial Polymers at a regional office near you.

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