DUPONT™ ELVALOY® RET
LAB SCREENING GUIDE

Technical Bulletin RET1.1
Suggested Guidelines for Initial Screening of Elvaloy® RET in Asphalt for Paving Applications
Introduction

DuPont™ Elvaloy® reactive elastomeric terpolymer (RET) differs from asphalt modifiers such as rubber, SBS or SB. To obtain the full benefits of Elvaloy® RET, a reaction must take place between the terpolymer and the asphalt. Please read these instructions carefully and evaluate Elvaloy® RET in a laboratory prior to any plant trials, to determine the correct dosage and timing to complete the reaction.

There are two methods for completing the reaction of DuPont™ Elvaloy® RET in asphalt:

1. The *heat-reacted method* to complete chemical bond between the terpolymer and the asphalt over a 12- to 48-hr period (depending on the asphalt binder).

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

It is important to follow the guidelines carefully.

Select Proper Base Asphalt

The low-temperature properties (below approx. 4°C [40°F]) of a polymer-modified asphalt are determined mainly by the base asphalt. Select a base asphalt that meets the specified low-temperature requirements. The RET provides the high-temperature properties. Elvaloy® RET does not normally change the cold temperature PG specifications.

The base asphalt must not be pre-acidified. Acid will catalyze the RET reaction immediately. Elvaloy® added to acidified base will form gels as soon as it is added to the asphalt. Those gelled particles will not be melted or dispersed with the asphaltenes in the binder.

Elvaloy® RET Grade Selection and Amount

The appropriate RET addition level depends on the base asphalt, the desired specification, and the RET grade chosen (either Elvaloy® 4170 or Elvaloy® AM). Typical terpolymer levels are between 0.8% and 1.8% by weight, but can be as high as 3%. DuPont recommends initial evaluations at three terpolymer levels: 0.8%, 1.2%, and 1.8%. Results from these blend levels should provide information for fine-tuning the formulation.

Concentrated batches can be made; however the amount of terpolymer that can be added to a batch is asphalt-dependent. Terpolymer levels in excess of 2.5% to 3.0% by weight can gel the entire batch of asphalt binder. Laboratory testing of concentrate blends must be completed prior to plant trials. Some asphalt will gel with less than 2.5% Elvaloy® RET.

Typical Laboratory Equipment Needed

A standard bench-top mixer (does not have to be high shear) capable of creating a small vortex as well as a hot plate or heating mantle capable of maintaining a constant 370°F temperature will be necessary. See Figure 1 for an example of such equipment.

Using The Catalyst Technology (*)

The patented catalyst technology uses polyphosphoric acid, PPA (aka super phosphoric acid, SPA). Typically the PPA is used at a concentration of 105%, however, concentrations greater than 105% can be used successfully. Typically 0.2-0.4% PPA by weight of the asphalt is used as a catalyst. It is recommended that laboratory evaluations begin at 0.2% PPA and then ramp up to 0.4%. **Warning:** It is critical that polyphosphoric acid be used as the catalyst and not the more standard aqueous phosphoric acid (which is typically less than 85% in concentration). Aqueous phosphoric acid can flash off water causing the hot asphalt to spray out of the container. Please contact the catalyst supplier for specific safety and handling recommendations.

Figure 1 -- Lab Mixer and Heating Mantle

*Patented Catalyst Technology is the property of MTE Services Inc., Specific questions regarding the catalyst technology should be directed to Mr. Gerald Reinke, President of MTE Services, 915 Commercial Ct., P.O. Box 563, Onalaska, Wisconsin 54650 tel. (608) 781-4683.*
Experimental Blend Preparation

1. Carefully heat the base asphalt sample in its original container in an oven until it is fluid enough to pour. Usually, an oven set at 165°C (330°F) should be sufficient.
2. Stir the sample until it is homogeneous and pour into suitable blending containers. Quart or half gallon paint cans work well.
3. Place the container of asphalt in a heating mantle or on a hot plate and heat to the desired blending temperature (180-195°C, or 350-385°F). Once the sample is up to temperature, stir with a lab mixer set just fast enough to create a small vortex, without whipping excessive air into the sample (usually 200 rpm).

Note: Elvaloy® RET chemically reacts with asphalt binder and the reaction is dependent on time, temperature and chemical nature of the base asphalt. Some asphalts will oxidize or lose volatile components rapidly at high temperatures. The resulting increase in viscosity can overshadow the effects of the terpolymer and complicate the formulation process. Comparing formulation to control samples of unmodified asphalt is strongly recommended to determine the effects of the blending procedure on the asphalt.
4. Maintain the asphalt at the desired blending temperature for 10 minutes before adding the terpolymer.
5. Add the desired amount of Elvaloy® RET slowly (about 10 g/min for half-gallon of asphalt) allowing the vortex to pull the pellets into the asphalt.
6. Continue to stir the blend for 2 hours after the last of the Elvaloy® is added. Check the blend periodically; the speed of the blender may have to be adjusted as the viscosity increases. Be sure to maintain the desired mix temperature. After 2 hours, the terpolymer should be fully dispersed, and the heat reaction started; remove asphalt from the mixer.

Procedure for Heat-Reacted Method

7. Once the terpolymer has been mixed and agitated for 2 hours, take a sample of the asphalt, and record the results (i.e., G*/sin δ or viscosity) at “zero cure time”.
8. Pour the remaining blend into two containers and cover tightly with a lid. Place each container in an oven set at the desired curing temperature, normally around 165 to 190°C (330 to 375°F). Allow sufficient time for the reaction to take place. Cure times of 12 and 24 hours are recommended for initial testing. Record the G*/sin(δ) or viscosity every 2 hours until the stiffness has peaked and the reaction is complete.
9. After the reaction is complete, remove each container from the oven, allowing them to cool for 5 to 10 minutes and then open carefully. Run the desired tests and use the data obtained to find the best cure time for the particular asphalt being tested. Often this cure time will be less than 24 hours, depending on the asphalt.
10. Once a target formulation has been developed, run all of the tests required by the specification.

Procedure for Catalyst Technology

7a. Once the terpolymer has been mixed and agitated for 2 hours, the samples are ready for the catalyst. For initial screenings, check that there is no gel formation by adding 0.2 wt% (of the asphalt) PPA to the mixture, and stir for 30 more minutes. Take a sample and test the viscosity or DSR.

Warning: adding the acid too early in the process can prevent the Elvaloy® RET from fully dissolving properly; lumps may form. If this happens, the Elvaloy® must be mixed with the asphalt for a longer period of time prior to acid addition.
8a. Cover tightly with a lid and store in an oven at 165°C (330°F) overnight.
9a. After overnight storage, remove the sample from the oven, allow the container to cool for 5 to 10 minutes, and then open carefully. Check for gel, run the desired tests, and use the data obtained to find the best Elvaloy® level for the particular asphalt being tested.
10a. Once an initial target formulation has been developed, optimize reaction by running screening tests using 0.2-0.4% polyphosphoric acid. Additional acid usually increases the elastic recovery but doesn’t affect viscosity much.
11a. Once a target formulation has been developed, run all of the tests required by the specification.

Note: During initial formulation, it is important to track the time of the reaction Elvaloy® RET and the asphalt by tracking the increase in viscosity (either absolute or Brookfield) or the DSR. Once the value stops increasing, the reaction is complete.
Additional Testing
After the formulation is developed and the necessary cure time has been determined, additional testing is recommended to anticipate and prevent manufacturing problems. Tests may include:

- plant trials to decide how large scale batches correlate with lab results.
- stability tests to learn how the properties of the asphalt can change with time in storage;
- asphalt sensitivity studies to determine how variations in base asphalt properties, terpolymer level, storage temperatures, etc., affect the final product properties;
- asphalt compatibility studies to determine how to correct deficiencies in advance if off-spec product is made;
- over-treatment tests to find out the maximum amount of terpolymer that can be added without forming an undesirable gel.

**Warning:** Heating asphalt in a sealed container can lead to pressure buildup inside the container, due to volatile components in the asphalt. Use caution when opening the container, a lid with a built-in valve can be used to manually relieve the pressure before opening the container.

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.