TRANSNUCLEAR An AREVA Company TIMO PROCEDURE		TITLE: Reactivity Test for Shielding Components			
Procedure No.	**Rev.**	**APPROVALS**	**NAME**	**SIGNATURE**	**DATE**
96-T-001	**4**	**Author**	**W. Bracey**	W Bracey	1/27/05
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1.0 PURPOSE AND SCOPE

1.1 The purpose of this procedure is to test the reactivity of the resin, catalyst and accelerator supplied for cask manufacturing when any of the shelf lives has elapsed. Continued use of resin, accelerator or catalyst that is 18 months or more beyond the original manufacturer's stated shelf life is not recommended; however, its use may continue if acceptable testing is performed.

2.0 REQUIREMENTS

This test is to be performed whenever one or more of the following conditions occur:

2.1 If the resin, catalyst or accelerator reaches the manufacturer's recommended shelf life, the test shall be performed prior to continuing or starting resin pouring.

2.2 This test will be valid for one month, at which time it must be repeated if resin pouring is to continue with the expired component(s).

2.3 This shelf life extension is only valid for the combination of resin, catalyst and accelerator lots tested. Replacing any lot of component material with another lot of component material requires a test for the new combination.

2.4 The test may be performed on components that are not expired in order to determine the proper amount of accelerator prior to first use.

3.0 TEST DESCRIPTION

3.1 A mixture of resin, catalyst and accelerator is prepared in a suitable container under temperature controlled conditions, and the mixture temperature and viscosity is monitored as a function of time.

3.2 The time elapsed between adding the catalyst and reaching the maximum temperature is called the exothermal reaction time.

3.3 The time elapsed between adding the catalyst and when the resin will no longer drop or flow off the mixing tool is called the gel time

4.0 COMPONENTS FOR TESTING

4.1 The resin is a product of Cray Valley with a trademark Norsodyne M0070C - unsaturated polyester resin.

4.2 The catalyst is a cyclohexanone peroxide product of AKZO Nobel with the trademark of Cyclonox LR.

4.3 The accelerator is a solution of cobalt octoate with 1% of active cobalt content product of AKZO Nobel with the trademark of NL49P.

5.0 EQUIPMENT

- Polypropylene or equivalent cup with 50 ml to 100 ml capacity
- 2 eye droppers, one for the catalyst and one for the accelerator
- Digital thermometer in °C or °F, calibrated
- Mixing tool made of wood, polypropylene, or shielded thermocouple probe.
- Timer
- Scale with 0.01 gram or smaller graduations, calibrated.

6.0 TEST PROCEDURE

Safety Note: The catalyst and accelerator will react vigorously if mixed directly. Keep these components and their eye droppers apart.

6.1 Perform the following steps:

1. Record the test number, time, date, and air temperature. Record the batch numbers and manufacturer's recommended shelf life expiration date for the resin, catalyst and accelerator to be tested.

2. Put empty cup on scale and zero the scale.

3. Weigh 50 g $\pm$ 0.1 g of resin inside the cup. Record the weight of resin.

4. Add 0.5 $\pm$ 0.02 g (or other amount A_t based on experience) of accelerator (purple liquid) with the eye dropper and mix vigorously. Record the weight of accelerator added.

5. Measure the temperature of the mixture and adjust it if necessary until it is between 21 °C (70 °F) and 23 °C (73 °F). Record temperature on the data sheet.

(Practical note: temperature adjustment can be achieved by cooling in a refrigerator or in front of an air conditioner, or heating in an oven, on a warm surface, or in your hand. Stir while adjusting, then allow the temperature to equilibrate for about 30 seconds before verifying and recording the temperature.)

6. Insulate the cup from your hand and the table for the remainder of the procedure.

7. Add 1 g $\pm$ 0.02 g of catalyst with another eyedropper. Record the weight of catalyst added, and mix vigorously until the mixture becomes homogenized (approximately 15 seconds).

8. Start the timer, and continue mixing for an additional 30 seconds.

9. Record the temperature in at least one minute intervals until the temperature begins to fall or level out.

10. Stir the resin occasionally to test its viscosity.

11. Record the time when the resin mixture starts to thicken, when it will not drop off the mixing tool (gel time) and when it is completely set.

12. As the resin hardens, use the thermocouple probe to form a hole at the center of the resin.

13. Record the exothermic reaction time and maximum temperature on the data sheet. (Maximum temperature should be less than 280 °C (536 °F).

6.2 If the gel time is greater than 11 minutes 30 seconds, increase the amount of accelerator. Ratio the actual gel time to the average acceptable gel time (10.5 minutes). Increase the amount of accelerator by this ratio. For example, if the gel time is 21 minutes, double the amount of accelerator.

(Practical note: The relationship is not really linear, but it's a good first approximation.)

6.3 If the gel time is less than 9 minutes, reduce the amount of accelerator by ratioing the actual gel time to the average acceptable gel time (10.5 minutes). For example, if the gel time is 5.25 minutes, reduce the amount of accelerator by 50%.

6.4 Record the amount of accelerator used and repeat the test. Vary the amount of accelerator until the gel time is between 9 minutes and 11 minutes 30 seconds and the exothermic reaction time is between 17 and 30 minutes.

6.5 Continue the testing until two tests have been performed with

- the same amount of accelerator, within 0.02 g
- the gel time between 9 minutes and 11 minutes 30 seconds
- the exothermic reaction time between 17 and 30 minutes
- exotherm < 260 °C (536 °F).

(Practical note: It may be difficult to achieve two tests within the gel time limits if the accelerator amounts are 0.02 g apart. In practice, the test is very sensitive to the accelerator amount and the starting temperature. It is best to change these values as little as possible in order to achieve two passing tests.)

7.0 DETERMINATION OF PRODUCTION QUANTITY OF ACCELERATOR

7.1 To determine the amount of accelerator to be used in a production batch, record the amount of accelerator used for the acceptable reactivity test, A_t in grams.

7.2 If the two qualifying tests used the same weight of accelerator, the value of A_t may be the test value $\pm$ 0.01 g. If the two test values were 0.01 or 0.02 g apart, either test value or the value between them may be used. This may avoid making unnecessary changes in the production quantity.

7.3 The amount of accelerator to be used in the production batch is 4 cc/kg x A_t/0.5.

8.0 REFERENCES

8.1 ASTM D2471, Standard Test Method for Gel Time and Peak Exothermic Temperature of Reacting Thermosetting Resins

Note: This reference is for information only. It is not intended that this procedure conform to the reference.

TRANSNUCLEAR An AREVA Company	**Data Sheet** **Reactivity Test for Resin Components**	**TIMO: 96-T-001** **Page 1 of 2**

Date:	Test Number:	Time:

Room Temperature:_____(Must be between 18 °C and 25 °C (64 °F and 77 °F)

Component	Actual Weight grams	Req'd Weight grams	Lot Number	Manufacturer's Recommended Shelf Life Expiration Date
Resin		49.9 - 50.1		
Catalyst		0.98 – 1.02		
Accelerator		0.48-0.52 (See Note)		

Note: Alternate weight of accelerator may be used based on previous test_____

Temperature of mixture prior to adding Catalyst _____
(Must be between 21 and 23 °C (70 °F and 73 °F)

Time	Temperature	Comment
		Thickens
		9:00 ≤ Gels ≤ 11:30
		Sets
		17:00 ≤ Exotherm ≤ 30:00 min; < 260 °C (536 °F)

Instrument	Identification	Calibration	
		last	due
Temp. Meas.			
Scale			

Test operator:

TRANSNUCLEAR An AREVA Company	**Data Sheet** **Reactivity Test for Resin Components**	**TIMO: 96-T-001** **Page 2 of 2**

Date:	Test Numbers:	Times:

Determination of Production Accelerator Amount

A_t	(grams)
A_p = 4cc/kg x A_t / 0.5=	(ml/kg)

95 pound (43.1 kg) batch

Amount of Production Accelerator 43.1 x A_p =	(ml)

60 pound (27.2 kg) batch

Amount of Production Accelerator 27.2 x A_p =	(ml)

Test Operator:	Date:
Checked:	Date: