

#### Introduction

Medical device manufacturers currently require packaging materials to provide a microbial barrier and device protection that last for an extended period of time. For example, devices may need protection for up to five years. Package material aging information is needed to insure package integrity, satisfy current FDA validation requirements, and provide evidence of sterility and fitness-for-use over a product's life cycle. To decrease the time required for testing prior to device commercialization, manufacturers perform accelerated aging studies on the product/package combination. An important aspect of the aging of plastics is physical aging. The physical properties of plastics can improve or deteriorate over time as a plastic is held below its glass transition temperature. It is important that accelerated aging studies be performed properly since each plastic material ages differently. This technical tip will provide guidelines for the proper aging of Eastar Copolyester 6763.

#### General Accelerated Physical Aging Guidelines

- The ASTM F1980-99 standard guidelines or methodology for accelerated aging of sterile medical device packages is based on the following Arrhenius equation:

$$t_{23} = t_T * Q10^{((T - T_{23})/10)}$$

Where:

$t_{23}$  = Time at ambient temperature and 50% relative humidity

$t_T$  = Accelerated aging time duration

$T$  = Accelerated aging temperature, °C

$T_{23}$  = Ambient or room temperature, 23°C

$Q10^a$  = Accelerated aging factor

<sup>a</sup>The ASTM guideline suggests using a Q10 factor of 2 as a conservative estimate for aging the packaging and device. The guideline also states that each material has a unique accelerated aging factor (Q10). Other Q10 factors can be used if they are derived from experimentation. Eastman has found through extensive experimentation that the Q10 factor for copolyesters is 9.8.

- Recommended conditions for copolyesters: Perform aging at 50°C and 50% relative humidity. At these conditions, 18 hours is equivalent to 1 year of actual aging at room temperature (23°C). Therefore, 92 hours of aging at accelerated conditions is equivalent to 5 years of actual aging at room temperature (23°C).
- Plastic material aging is reset, or returns to a zero age, during thermoforming.

#### Detailed Description of Accelerated Physical Aging

Physical aging is a process of molecular relaxation that occurs in all amorphous polymers held at temperatures below their glass transition temperature. It has been observed in PVC, polystyrene, SAN, and polycarbonate, as well as in copolyesters. When a polymer is rapidly cooled to below its  $T_g$ , (as occurs with all commercial melt phase processing techniques) it freezes into a non-equilibrium conformation with excess free volume. In an attempt to attain thermodynamic equilibrium, the chains rearrange themselves into a more dense structure reducing the free volume of the system. Although this densification is difficult to detect, it directly affects other thermodynamic and mechanical properties easier to measure and can be used to track the extent of aging with time.

## Accelerated Aging of *Eastar* Copolyester 6763 in Medical Packaging (TT-71B)

In general, these properties increase in linear fashion with the log of aging time. Aging also proceeds more quickly at higher temperatures closer to the materials' glass transition temperature. These trends are consistent with other similar viscoelastic molecular relaxation processes, such as rheological behavior. As with other relaxation processes, time and temperature can relate through the principal of time-temperature superposition. According to this principal, molecular motions that occur over a given period of time at one temperature are equivalent to motions that occur over a longer time period at lower temperatures. In other words, an elevated temperature acts as a multiplier for the rate of motion. Because of time-temperature superposition, it is possible to generate data as a function of time at different temperatures, and then shift the data together on one common master curve.

Numerous experiments were performed at Eastman to determine the mechanical and thermal properties of our copolyesters as a function of aging time and temperature. Time-temperature superpositions were performed on the data generated from these experiments to create master curves for each material. Aging times at each aging temperature that provided equivalent material properties were recorded. These times and temperatures, shown in Table 1, can be used to perform "accelerated aging" experiments. A part is aged at an elevated temperature for a short period of time to simulate aging at a lower temperature for an extended period of time. For example, 1 hour at 60°C is equivalent to 96 hours at 40°C or 4,700 hours at 23°C. Likewise, 1 hour at 40°C equals 48 hours at 23°C. Therefore, if one wanted to simulate the performance of a part after 10 years of life (87,600 hours) at 23°C, one should age the part at either 50°C for 180 hours or 60°C for 19 hours. This aging protocol will reasonably represent the lifetime of a typical copolyester application. Note that aging copolyesters at higher temperatures for longer periods of times is not generally recommended. For example, aging for 250 hours (10 days) at 60°C is equivalent to aging for 1,200,000 hours (133 years) at room temperature (23°C). Aging for this length of time or longer at 60°C may over-age the material and provide an unrealistic expectation of what the properties would be after aging at room temperature.

**Table 1: Accelerated Physical Aging Data**

Aging Temperature	Simulated Age/ Accelerated Aging Time (Hours)						
	~165 years	~17 years	~20 months	~12 months	~7 months	~3 weeks	~2 days
23°C (74°F)	1,400,000	150,000	15,000	8,800	5,100	500	48
30°C (86°F)	290,000	30,000	3,000	1,800	1,000	100	10
40°C (104°F)	30,000	3,100	300	180	110	10	1.0
50°C (122°F)	3,000	310	31	18	11	1.0	—
60°C (140°F)	310	32	3.1	1.9	1.1	—	—
65°C (150°F)	99	10	1.0	0.6	—	—	—

### Accelerated Age Testing of *Eastar* Copolyester 6763

Eastman has performed accelerated aging on *Eastar* Copolyester 6763 using these guidelines and methodology. The data generated suggests that *Eastar* Copolyester 6763 provides the required package integrity for a minimum of five years if good manufacturing practices are followed during extrusion, forming, package design, and sterilization and the packaging is stored at room temperature under normal conditions.



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