#### 60 X Recipe Summary

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| Introduction | This chapter describes product development if any, the reaction profile, standard operating conditions, autoclave batch handling and troubleshooting guide. |

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| Safety | The main safety hazard potential is exposure to surfactant GX902 and GX905D, other than normal FP polymerization and TFE hazards. This hazard is mitigated by the use of proper PPE. (**Refer to FP/Disp PPE matrix for proper protective equipment**). |

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| Standard Operating Conditions | The embedded file below describes the aim, INO, SOC and QSOC for the ingredient, process and process performance parameters. |

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| DCS Recipe | The DCS recipe index is 111. |

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| Application | T-60 X is polymerized to 37% solids. This polymer is used in the tape and gasketing markets. |

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| Product Development | 60 X is the GenX equivalent to 60 that was produced in autoclave #6 as TE-3981 during research GenX development. The initial offering had an SSG goal of 2.200. In July of 2014, under TA 6066 the product was redefined to have an SSG of 2.185 which more closely matched the original 60 and moved to autoclave #8.  60 X is qualified in autoclave #7 and #8. |

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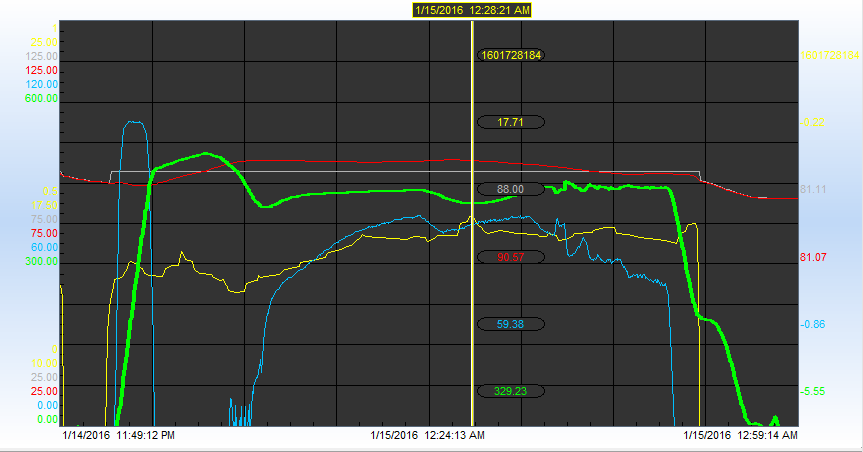
60 X Recipe Summary, Continued

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| **Key Features** | |
| Solids | 38% solids polymerization recipe, |
| Initiator | Two initiator ingredients are added, APS and DSP. The two initiators are added after the clave is pressured up with TFE |
| Modifier | Methanol is utilized as a chain transfer agent. The methanol is added as a precharge. Methanol is found to narrow the molecular weight distribution. |
| GX905D/FTD902 System | The nucleation and surfactant system for 60 X is GX905D and GX902   * Particle size control: In addition to PFBE, the FRD901 in GX905D is the main control for particle size (nucleation). * Stabilization: FRD903 in GX905D and GX902 is the stabilizer system for 60 X and is all injected after the aqueous charge with the agitator on. |
| Triton® X-45 | Triton® X-45 is added to the precharge for RDPS and molecular weight control. Increased Triton® X-45 will reduce RDPS. |
| FeCl2 Solution | FeCl2 is added as a promoter for DSP and is very important for polymerization "kickoff" and reaction rate. It also impacts RDPS (increased FeCl2 loading increases RDPS). |
| Tomadol 23-1 | Tomadol 23-1 is used to control coagulum. It will also impact SSG (higher). |
| Reactdown | Normally no react down, in order to reduce molecular weight distribution and control rheometer |
| Wax | Wax reduces coagulum formation, also found to narrow molecular weight distribution (higher levels). |

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60 X Recipe Summary, Continued

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| Reaction Profile | Polymerization pressure - Initial polymerization pressure is 350 psig, but the pressure is not always kept constant. Sometimes, within 20 or 30 minutes after kickoff, the heat being generated by the reaction exceeds the cooling capability of the jacket and the clave becomes "heat transfer limited" - meaning that the clave's jacket water system, even at full cooling, is not capable of removing all the heat being generated by the polymerization reaction. At this point, the clave's temperature begins to rise above setpoint and the clave's pressure is automatically reduced in order to reduce the TFE reaction rate (which in turn reduces the heat generation rate) and to bring temperature back under control. Clave pressure is then used to control clave temperature for the remainder of the batch. The TFE reaction rate tends to drop off towards the end of the batch and the clave pressure is increased to offset this until the pressure returns to 350 psig and then the jacket temperature is used to control the clave temperature again.  Figure I illustrates key features of the polymerization. A typical 60 X profile is shown in Fig 1. Figure 1 is a typical pressure and temperature profile for polymerization. 60 X is variable and the profiles can vary substantially.   * Green line is autoclave pressure in psig * Blue line is TFE flow by Monomer Flow Meter * Red/grey line is autoclave temperature in °C and * Yellow is the amps |



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60 X Recipe Summary, Continued

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| **Start of Campaign** | * Ensure that the HFP, PPVE PFBE manual valves on the autoclave feed manifold are closed. These modifiers can enter the clave if the associated automatic valves are leaking through and impact product properties * On the DCS, go to the Ingredients Addition Summary Page and select the appropriate “Next Product” * The DCS will request the appropriate boilouts * Complete transition and boilout requirements. You are ready to start the first batch. |

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| Starting of Batch | * Ingredients are made up at the beginning of each batch, after rinse drop and porch lights are activated * A campaign is started by:   + The DCS automatically posts the recipe on the screen next to the ingredients hood.   + On the Autoclave screen on the DCS, select “Start Batch” on the “Pic Path”   + Identifying the batch with the correct batch number. |

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| Batch Segregation Requirements | A 60 X batch must be segregated and processed as 2nd quality if actual operating conditions are outside the QSOC limits or clave properties are out of process specifications  Except for an aborted batch, take the following steps to segregate and separately process the batch:   * If possible lower wax decanter heel before dropping 2nd quality batch * Drop the batch to the decanter, Decant off the wax from the decanter * Work down a blend tank to minimum possible level * Transfer from the decanter to the blend tank, dropping the decanter level to the minimum possible level * Process the material from the blend tank through finishing and packout as second quality * Drain any residual material in the decanter and in the blend tanks into 55- gallon drums or totes and dispose by incineration |

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60 X Recipe Summary, Continued

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| Other Special Instructions | * Follow the instructions in 33P6A for a QSOC violation or an aborted batch. * All drains from the wax decanter are to be packed out for incineration off plant (WCR902-432TP, label 71154-60). * Zinc to pressure-up and reaction time will be also monitored for coagulum formation. * Batch should also be aborted if a rapid increase in amps is noticed during the batch, or if the clave temperature indicators start to deviate by more than 3 deg C (normally 1-2 deg C). The last 30% of the batch is the most critical for coagulum formation. * If the “C” injection tank is not used due to an early abort after the GX902/GX905D are added, the DCS will allow to re-use the “C” tank. * Aborted batches not processed as 2nd quality through the finishing end can be transferred into drums /totes in the decanter * Do not drain the “C” tank to the supernate sump. If the “C” containing GX905D/GX902 needs to be empty, transfer into 5-gallon containers using special packout line installed at the “C” tank discharge in the red tile floor. Any FRD903 sent to W-9 will quickly consume a carbon bed and should be avoided. * All drains from the wax decanter are to be packed out for incineration off plant (WCF 902-432TP label 71154-60). Do not ask for drums to be vacuumed to remove liquid. All drums collected are to be recorded in shift log. |

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| Pre-charge and Injection Tank Utilization | "A" Injection tank: 20 gallons of water added to "A" injection tank (recipe). No ingredients are added to this tank, but tank is used to chase lines during batch drop.  "B" Injection tank: 6 gallons of water added to "B" injection tank (recipe). Necessary ingredients added to tank (See SOC's). Note tank added to autoclave after clave is pressured-up with TFE.  "E" injection tank: Not used  "C" precharge tank: <25 % diluted solution of GX902/5 mixture. Necessary ingredients (GX905D/GX902 solution added to tanks either manually or using the GX905D and GX902 feed facilities. (See SOC's).  “D” precharge tank: 10 gallons of water added (recipe). Necessary ingredients added to tank referred to SOC's.  Note that the diluted surfactant in tank “C” will be added to the autoclave after the aqueous charge has been added to the clave with autoclave agitating  Boilout the autoclave per instructions given in the “Autoclave Boil-out” section (below). |

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60 X Recipe Summary, Continued

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| Batch Polymerization Steps | The steps below describe the batch polymerization steps. |

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| Pre Reaction Steps | Description |
| Rinse Drop | The water in the AC is dropped out to the RWDC to complete the rinse of the AC. |
| Precharge/ Ingredient Solutions Preparation Addition | After the start of rinse drop, the precharge tanks and injection tanks are prepared while the clave continues to the evacuation and pressure test.  Precharge and injection tank preparation   * DCS will add Demin water to the correct total dilution water amount to the tanks (will drain Demin water tanks if it is high or add Demin water if low) * Porch lights will turn “on” for precharge tanks and injection tanks * Manual addition of ingredients is added to “C”, “B” * After manual additions, operator clears porch lights for the precharge and injection tank * “C” tank sequence   + Tank agitator starts   + Agitates for 1 minute   + Agitator shutdown and level in the “C” tank recorded   + DCS adds GX905D to tank as per product recipe   + DCS checks and records GX905D addition level and “C” final level   + DCS adds GX902B to the “C” tank as per product recipe   + DCS checks and records GX902 addition level and “C” final level   + Agitator started, agitates for 4 minutes   + “C” Tank is now ready for addition * “D”, “A”, ”B” tank preparation sequence. * Tank agitator starts * Agitates for 2 minutes * Tanks ready to be used |
| Evacuation and Pressure Test | * After rinse drop the autoclave is evacuated to -10 psig with jacket temperature set at 95 deg C to remove any remaining water that might have been left in the autoclave * Autoclave is leak checked under vacuum for 1.0 minute |
| Precharge Additions Prior to Aqueous Addition | Precharge tank D is added to the autoclave depending on recipe precharge flags. For 60 X the precharge flags are:  DCS: Type 1 (“D” tank added before aqueous charge) |
| Aqueous Charge | Deaerated, demineralized water at 90-95 deg C is pumped to the AC from the aqueous charge tank (still under vacuum, with agitator down). Note aqueous addition is added before the “C” tank precharge addition after the “D” tank addition or at same time as “D” tank. |

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60 X Recipe Summary, Continued

**Batch Polymerization Steps (Continued)**

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| Pre Reaction Steps | Description |
| Precharge “C” Tank Addition After Aqueous Charge | If “C” tank is ready, it is added to the autoclave after the aqueous charge, while clave is still under vacuum, with agitator running and vent manual valves to the jet closed. |
| Wax Addition | Wax is added to the clave |
| Pressure Up MWT | ZnCl2 solution is pumped into the bottom of the MWT to start pressurizing the MWT to 250 psig in parallel to next steps |
| Deareation step | A one (1) minute dearation or ten (10) minutes if no dearated Aqueous charge was used. |
| MWT Pressure/  AC Pressure Up | Monomer weigh tank pressurized start to 460 psig  TFE added to autoclave if temperature in clave is above pressure up temperature setpoint. |
| Autoclave Heated to TFE Addition Temperature | As soon as the TFE starts going to the autoclave the Autoclave temperature is set to kickoff temperature. |
| Reaction;  Start B Injection | Starts adding contents of "B" Injection Tank into autoclave (see SOC’s) |
| Reaction: Kickoff | Kickoff detected (15 psig pressure drop) |
| Reaction | TFE feed is restarted and continues throughout the batch at a rate that maintains AC pressure at setpoint.   * After 150 lbs. of ZnCl2 have been transferred or 15 minutes since autoclave pressure-up, "A" Tank injection begins. The "A" Tank continues to inject through approximately 80% of the batch. When "A" Tank reaches low level, it is chased, injected, and then stopped. “A” Tank (which contains only water) is injected to help keep the injection ports unplugged during the polymerization. |
| End of Reaction | The TFE feed and block valves are closed when the specified amount of ZnCl2 solution, as measured by ZnCl2 tank level, has been pumped into the MWT. |
| Reactdown | Autoclave reactdown to target pressure as per SOCs.  Note: Current 60X does not react down. |
| Depressurize | * All injections stop. Injection tanks are drained and rinsed * Autoclave temperature controller set to local to reaction temperature setpoint * The agitator is stopped and the reactor is vented to the Mixed Gas Holder (MGH) to less than 2 psig. * The reactor is evacuated with the vacuum jet to a pressure that is 2 psi above the vapor pressure of water based on the higher of the two autoclave temperature transmitters. * The reactor is then pressured with N2 to 14 psig. * The reactor is evacuated with the vacuum jet to a pressure that is 2 psi above the vapor pressure of water based on the higher of the autoclave temperature transmitters again. |

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60 X Recipe Summary, Continued

**Batch Polymerization Steps (Continued)**

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| Reaction Steps | Description |
| Drain Back | During AC react down, the MWT pressure is decreased by drain back of the ZnCl2 solution to the ZnCl2 tank when pressure in the clave is less than 40 psig |
| Batch Drop | This reactor proceeds to transfer the batch to a Decanter.   * “A” injection pump injects water during batch drop. * “Prod Rinse” rinses the autoclave with 50 gallons of water to the Decanter after batch drop |
| RAM Clean and Steam Out | Autoclave is ram cleaned and after steam out with 250 gallons of water in the clave |

Boilout Requirements

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| Do a 425 grams DSP boilout  • At the start of a campaign  • After 12 hours of downtime |

Transition Matrix

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| Transition going into 60 X:   * B transition. Second quality or WTI first 4 drums   Transition going out 60 X   * B transition Second quality or WTI first 4 drums for non PSR products * If going into a PSR followed PSSR transition matrix |

End of topic

Troubleshooting Guide [Recipe adjustments at the direction of Technical/Supervision]

##### RDPS

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| Property | Action |
| **HIGH** | 1. Verify Triton® X-45 addition 2. If kickoff times are low, Triton® X-45 charge was probably low 3. Verify that the “C” tank has been properly added to the clave, and there are no leaks in piping 4. Verify for proper addition of GX905D to “C” tank. 5. Check to make sure wax quality is in control 6. Verify actual kickoff temperature and kickoff pressure are as per SOC. 7. Verify “B” injection addition 8. Verify FeCl2 solution level is not too high. 9. Increase FRD901 in increments of 10 grams. |
| **LOW** | 1. Verify Triton® X-45 addition 2. If kickoff times are high, Triton® X-45 charge was probably high 3. Check to see if wax quality has changed. 4. Verify for proper addition and amount of GX905D to “C” tank 5. Reduce FRD901 in 10 grams increments until the desired RDPS is obtained. |

**Reaction Time**

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| Property | Action |
| **HIGH** | 1. See OD 33P section 6B “Autoclave Batch Running Slow” – pay close attention to checking the injection system and lines 2. Check to see that RDPS is on aim and adjust if off aim (a decrease in particle size will often decrease reaction time). 3. Adjust DSP – increase DSP in 35 gram increments |
| **LOW** | 1. Check to see that RDPS is on aim and adjust if off aim (an increase in particle size will often increase reaction time). 2. Adjust DSP– decrease DSP 35 gram increments |

**SSG**

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| Product Property | Action |
| **VERY HIGH**  **> 2.195** | 1. Decrease DSP by 70 grams increments (a 70 grams will result in 0.005 decrease of SSG  2. APS can also be used to adjust SSG |
| **HIGH** | 1. Decrease DSP by 35 grams increments (a 35 grams will result in 0.0025 decreased of SSG)  2. APS can also be used to adjust SSG |
| **LOW** | 1. Increase DSP by 50 grams increments (a 35 grams will result in 0.0025 increase in SSG) 2. APS can also be used to adjust SSG |

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Troubleshooting Guide, Continued

**Rheometer**

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| Product Property | Action |
| **HIGH** | 1. High rheometer can be caused by operating the dryer at high temperatures. Verify that the dryer is not (and was not) being operated above maximum SOC temperatures. 2. If dryer temperatures were OK, check RDPS. Low RDPS will cause high Rheometer (referred to RDPS above). 3. If increasing the RDPS to aim will not bring about a sufficient decrease in rheometer (or if RDPS is already at or above aim), then increase both the amounts of APS and methanol. |
| **LOW** | 1. High RDPS will cause low rheometer. See High RDPS, Items above. Increasing the RDPS by 0.005 microns should reduce the rheometer by 150 psi. based on 60 experience. 2. If decreasing the RDPS to aim will not bring about a sufficient increase in rheometer (or if RDPS is already at or below aim), then decrease both the amounts of APS and methanol. |

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| Effects of Adjusted Ingredients | | The table below describes the effects of some of the added ingredients | |
|  | Ingredient | | **Effect** |
|  | FRD901 | | 1. Added to “C” Injection Tank 2. Increasing FRD901 decreases RDPS (which may increase extrusion pressure also). 3. Decreasing the FRD901 increases RDPS (which may decrease extrusion pressure also). |
|  | FRD903 | | 1. Added in two parts: Some in GX905 and the bulk in the GX902. This is the main stabilizer 2. Low level increased coagulum 3. High level decreases coagulum, but after a point is a cost issue. High level may also reduce rheometer. |
|  | APS/DSP | | 1. Added to “B” Injection Tank. 2. Lower level will reduce SSG 3. Higher level will increase SSG and forms more coagulum (especially APS) |
|  | FeCl2 solution | | Added to “D” tank. Higher levels of FeCl2 reduces reaction time but will result in higher coagulum and may increase RDPS. |
|  | Triton® X-45 | | 1. Added to “D” tank 2. Used for RDPS control 3. Triton 45 also stabilizes the dispersion and reduces the molecular weight (increases SSG) |
|  | Methanol | | 1. Added to “D” tank 2. Methanol increases SSG and also narrows molecular weight distribution |

End of topic