8 PLANT START-UP/SHUT-DOWN AND OPERATIONAL CONDITIONS
SULPHONATION/NEUTRALISATION

8.1 Plant start-up

Ensure availability at the various process units of the required utilities, set at the required process values and connected to the equipment items (example: cooling water flow, temperature and pressure to sulphonation reactor).

Ensure correct "routing" of the different raw materials from storage vessels and intermediate and final products to storage tanks (example: routing of the required type of organic feedstock to proportioning pump, from pump to reactor, from reactor discharge to start-up off-spec tank, etc., valve settings, level, temperature, pressure controls on).

Follow checklist of utilities and raw materials/products routing.

Switch on all panel instruments, alarms, etc.

Start all separate units which can be operated independently from the main process, i.e.:

* process air cooling system for air drying (freon compressor, glycol pumps, etc.);
* air-drier regeneration (stand-by);
* catalyst preheating;
* recycle pumps and cooling system for reactors (sulphonation, neutralisation) "ready for start-up";
* exhaust gas system: ESP on, H₂O/caustic feed to SO₂ scrubber, recycle pump on, etc.;
* start preheater burner, set relevant process temperatures;
* start process air blower, set relevant flows and pressures.

Ensure that the emergency system, dosing system, setting of flows, pressures and all other required functions/controls are ready for start-up.

When easy and logical procedures are defined there will be no need for special attention or parallel activities during the "critical" phase of production start-up. Therefore some actions are taken prematurely, in an order not exactly required at that time of the start-up procedures (example: caustic scrubber recycle and caustic feed, not necessary during plant preheating, will be started in advance to accumulate sufficient excess of caustic to absorb the initial extra amount of SO₂ produced during the initial start-up period after sulphur ignition).

Next start sulphur combustion and sulphonation reaction start-up.

Follow the step by step operating procedures. Then, once the required preheating temperatures are reached (normally within two hours), the plant will be ready for sulphur ignition: sulphur dosing and sulphur igniter will be started, the conversion of SO₂ → SO₃ will proceed from an initial 50% value to 98% and the sulphonation reaction with alkylbenzene will result in a partially-converted alkylbenzene sulphonic acid, to be returned to the off-spec circulation tank.
In this phase it is very important to follow a well-defined procedure and sequence of operation, in order to reduce the amount of off-spec material and start normal steady-state production leading to a product with the required specifications.
8.2  **Plant shut-down**

The philosophy of shut-down is to stop production in a way which minimises plant corrosion and product wastage. This is particularly true when the plant is to be shut down for more than 48 hours or when maintenance activities on the gas raising plant are envisaged. For short periods it is adequate simply to isolate the gas raising plant from the reactor. Therefore the following procedure is recommended.

A) Shut off the sulphur supply.
B) If any feedstock other than alkylate is currently being used, switch supply to alkylate and recirculate the alkylate continuously around the reactor until all the remaining SO$_3$ has been consumed.
C) Continue purging with dilution air to ensure the plant is free from residual SO$_3$ (30-60 minutes).

8.3  **Operating conditions**

8.3.1  **Sulph(on)ation**

The reaction of an organic feedstock with SO$_3$ in a falling-film reactor proceeds very quickly, with approximately 80% of the organic reacting in the first 1.5 to 2.0 metres of the 6 m-long reactor (see 5.5.3.). Associated with this quick reaction is a commensurate evolution of heat which must be dissipated equally as fast as it is produced to prevent hot spots leading to poor colour or even charring.

The film reactor design caters for this heat removal by utilising a split water cooling system to the reactor, allowing adjustment to water flow for the various types of feedstocks. LAB and lauryl alcohol require cooling water with typical temperatures between 20 and 30°C. Cooling water loops with high recirculation rates are used. Fresh cooling water is fed to the loop to keep the recirculating cooling water at the pre-set value; 90% of the fresh cooling water is used in the top section of the reactor, with the remaining 10% used for the lower part of the reactor cooling circuit.

The combination of high cooling water recirculation and high rates of fresh cooling water results in a high heat-transfer coefficient and a high driving force for heat exchange, as the cooling water temperature rise between inlet and outlet in the top cooling circuit is approximately 1°C. In the lower part, where far less heat is generated and where the viscosity of the organic becomes important, the cooling has to be tempered. This is accomplished by less water and allowing a rise of about 3°C between inlet and outlet cooling water temperatures.

The SO$_3$ concentration is equally important to ensure product of acceptable colour and low free oil levels. The concentration of SO$_3$ in the gas phase is used to temper the reaction rate (see also 5.5.3.). For LAB the SO$_3$ concentration should be between 5 and 6% in air. Lauryl alcohol requires an SO$_3$ concentration of 4 to 5%, lauryl alcohol ethers 3%, and alpha-olefins 2.5 to 3%.

The final element necessary for efficient operation of the sulphonation reactor is the temperature of the feedstock. Generally the organic feed temperature should be as low as possible, for example between 25 and 28°C for lauryl alcohol.