

PX 1103

MONSANTO INDUSTRIAL CHEMICALS COMPANY
Sauget, Illinois

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TENTATIVE AMENDMENT "E"

TITLE

Tentative Amendment "E" to the Standard Manufacturing Process Description for Parachlorophenol and Orthochlorophenol, dated February, 1974, Dept. 237.

OBJECTIVE

To minimize formation of dioxins by deleting the neutralization of crude monochlorophenol with 50% caustic.

PRESENT PRACTICE

Dept. 237 currently produces refined chlorophenols in a batchwise operation. Virgin and recycled phenol are chlorinated to a specific gravity of 1.212 to 1.215 to produce crude monochlorophenol. The crude chlorinated mass is then aerated for four hours to reduce dissolved HCl from 2000-3000 ppm to about 40 ppm. The material is then neutralized with approximately 5 gallons of 50% caustic for an 8000 gallon still charge. A still charge contains six chlorinator batches.

Once the still is charged, the material is heated and vacuum is applied. Water is first removed from the batch. Forward flow is diverted to the Wet Fractions Storage Tank until the water content is less than 0.25%. The reflux ratio is then varied to separate the various chlorophenol isomers. Residue is removed from the still once every 10-15 batches.

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SUGGESTED CHANGE

Neutralization of crude monochlorophenol with caustic will be eliminated. Since most of the water content of crude monochlorophenol previously originated from the caustic neutralization step, no wet fractions cut will be made. The dry fractions cut will be sampled for water and if the concentration is more than 0.2%, the fractions will be transferred to the Wet Fractions Storage Tank.

Due to increased levels of HCl in the crude monochlorophenol, it is important to eliminate water from the system. The most important source of moisture after elimination of caustic is the still pot washout. The normal procedure of charging the still with 2000 gallons of water will continue to be followed. However, after each washout, the still will be charged with dry fractions and dried in the normal manner. Wet fractions from this source will be accumulated and eventually redried.

JUSTIFICATION

Deletion of the caustic neutralization step is expected to reduce or eliminate dioxins and make a more environmentally acceptable product.

This process change is also expected to reduce batch cycle times due to elimination of the wet fractions cut. Residue formation and waste disposal costs will also be reduced.

DISCUSSION

Elimination of the caustic neutralization step will expose the column, still pot, and condensers to increased levels of HCl. In order to quantify the risk of corrosion, test coupons were placed in the system to determine corrosion rates before and after caustic elimination. The data obtained from these tests will be examined and action taken if necessary.

Other steps will be taken to minimize HCl and water content in crude monochlorophenol. Aeration time will be monitored to insure that the full four hour period is used. In addition, phenol shipments will be sampled routinely for water and rejected if water content is greater than 0.07%.

Additional data concerning this process change is provided in TSD Progress Report 91370:4507, Nos. 1 and 2, "Eliminate Neutralization of Crude Chlorophenols," dated 10/27/75 and 11/19/75, by G. E. Mertz (F. J. Helmer).

YIELDS

A yield improvement is expected as a result of reduced residue and fractions generation. This improvement will be quantified after demonstration of the process change.

SAFETY

No additional safety precautions will be taken as a result of this process change.

QUALITY

Samples taken before the process amendment indicated that small amounts of chlorinated dibenzodioxins were formed as a result of the neutralization step. Elimination of this step is expected to reduce dioxin concentrations to lower levels. Results of analyses will be studied to determine if additional process modifications are necessary.

EXTENT OF DEMONSTRATION

Start-up and finalization of operating procedures and conditions will require two months. If difficulties arise, due to increased corrosion or product quality, this period may be extended.

CRITERIA FOR ACCEPTANCE

1. No unexpected safety problems of unacceptable nature develop.
2. Formation of dioxins is reduced to an acceptable level.
3. No corrosion problems of an unacceptable nature develop.

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