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OPERATING INSTRUCTIONS

FOR BEP-D AND BEP-T

Copies to:

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1) Operating instructions for BEP-D and BEP-T are the same only the charges are different.

2) CLEANLINESS - BEP batches must be uncontaminated with regular esters (Isopropyl or Butyl D or T). If the last batch run in the esterifier was a BEP-D, BEP-T, PEG 200-D, or PEG-300-T, then no further cleaning is necessary. If a regular ester (Butyl or Isopropyl D or T) was the last batch run, then the esterifier must be thoroughly cleaned in the following manner.

   a) Wash out the inside of the kettle with water, being careful to flush out all traces of ester that can be seen.

   b) Drain the separator and the reflux leg. Blow recovered alcohol out of receiver.

   c) Add 100 gallons of fresh benzol (or benzol that has been recovered from a BEP or PEG batch only!

      Fresh benzol must be obtained from tank 105. Do not use benzol obtained from tank 118.) to kettle and put on water distillation. Be sure drip leg is all the way up so as not to pass benzol out of drip leg. After refluxing starts continue for 2 hours, then put on benzol distillation. Benzol that is collected should be blown to a holder for use in Isopropyl-D, etc. When benzol stops coming off, cool down.

   d) Drain separator, reflux leg, and kettle of any benzol remaining.

3) CHARGES (For 1000 gallon Pfaudler)

BEP-D

B.E.P. - 2800 lbs. - 358 gallons (68" outage)

2,4-D - 3150 lbs. dry

Do not use crude 2,4-D

BEP-T

B.E.P. - 2400 lbs. - 317 gallons (71" outage)

2,4,5-T - 3110 lbs. - dry

NOTE that above acid is dry, laboratory will supply moisture to use for each batch.

DS 00013877

If laboratory does not supply moisture figure, use 10% for 2,4-D and 15% for 2,4,5-T.

4.) When charged, heat to 90° with agitation, take sample and have acid value run. Engineer will calculate adjustment (see calculation sheet); adjust loading of batch.

5.) When loading is adjusted cool to below 80°C and add 150 gallons of fresh benzol (from tank 105 only) or recovered benzol (from BEP or PEG batch only)

6.) Fill separator with water, raise drip leg, add 1/2 gallon of sulfuric acid and put on water distillation. Heat slowly so as not to boil over.

7.) When refluxing starts, lower drip leg slowly until water starts to come off. Every time water stops coming off lower drip leg some more. Check frequently to be sure no benzol is coming out of the drip leg. This may be done by catching several cc's from the drip leg in a test tube half full of water, benzol will float to the top forming two layers. If benzol is being passed out of the drip leg raise up drip leg until benzol stops coming off.

8.) The temperature should be kept below 135°C. If it gets this high, cool down and add 50 gallons benzol.

9.) When no more water comes off check to see that no water is going back to the esterifier. To do this drain off a quart of liquid from the reflux leg and discard, draw off a second quart and observe if two layers form. Water will form a greenish layer on the bottom. If much water is going back through the reflux leg, readjust drip leg.

10.) When water stops, sample and run A.V. If 12 or less the batch is ready for benzol distillation. If the A.V. is under 25 check again in (A.V. - 12) hours later (that is, let run for 1 hour for every A.V. unit between 12 and 25). If over 25 check again after refluxing for 4 hours, if the A.V. has not gone down by at least 3 units add more B.E.P. as follows:

For B.E.P.-D $\dfrac{(A.V.-10)}{(254)} \dfrac{(6000)}{(2)\ (7.82)}$ = gallons of B.E.P.

For B.E.P.-T $\dfrac{(A.V.-10)}{(219.5)} \dfrac{(6000)}{(2)\ (7.82)}$ = gallons of B.E.P.

Then put back on water distillation.

11.) When A.V. is 12 or less the batch is ready for benzol distillation. Cool down to about 70°C, turn vacuum on slowly and allow to build up (minimum vacuum is 20" but try to get as high a vacuum as possible). Heat up to 145°C and hold there for 1/2 hour. Then cool down to below 90°C. Sample and have A.V. and specific gravity taken. The batch is finished with an A.V. of 12 or less and a gravity of

BEP-D	1.185	or greater	(20°C)
BEP-T	1.225	or greater	(20°C)

12.) Be careful not to contaminate with water, alcohol, or other esters.

13.) Take and label sample for laboratory. Check valves for blowing to storage tank in warehouse.

Mark gage glass level (with masking tape) in storage tank before and after blowing. Record inches blown on esterifier sheet.

CALCULATION SHEET

FOR BEE-D AND T.

1.) Run all acid valves to first change of color with brom-thymol blue indicator.

2.) Adjusting loadings.

Stir sample well before putting in weighing beaker (any free water in batch will be on top). Weigh out a 1-2 gram sample and dissolve in about 100-150 cc of 150 propyl alcohol. Titrate using 0.5 N NAOH solution and brom-thymol blue indicator.

$$A.V. = \frac{(Factor)\ (c.c.\ s)}{\text{weight of sample in grams}}$$

To calculate theoretical A.V.

$$A.V.\ Theo. = \left(\frac{M_f}{\frac{M_f}{a} + M_2} \right) R$$

Where

M_f - Molecular weight of acid

M_f - 221 for 2,4-D

M_f - 255.5 for 2,4,5-T

M_2 - Molecular weight plus 5% of B.E.P. - 196

R - Factor

R - For 2,4-D - 254

R - For 2,4,5-T - 219.5

a - Fraction of real acid

(If laboratory does not give a to ester operator, use a - 0.9 for 2,4-D and a - 0.85 for 2,4,5-T)

3.) To adjust batch

Case I. A.V. Titrated is greater than A.V. theoretical. (over charged on acid)

Gallons of BEP to add = $\frac{(\text{A.V. Titr.} - \text{A.V. theo.})\ (6000)\ (196)}{(R)\ (\ M_f\ )\ (7.82)}$

Where R and M_f are defined above

Case II A.V. titrated is less than A.V. Theoretical (under charged on acid)

Lbs. of acid to add = $\frac{(\text{A.V. Theo.} - \text{A.V. Titr.})\ (6000)}{R}$

4.) Final A.V. is run with 0.1N NAOH solution.