

FOR DU PONT USE ONLY

DATE December 30, 1975TEST AUTHORIZATION NO. TA-C- 75-1194 AND CONCLUSION

OFFICIAL PRODUCT NAME "Lithosol" Red 4B Acid Presscake
PRODUCT CODE NO. 459 205 POUNDS PRODUCT PROPOSED ---
AREA Intermediates West BUILDING NAME DMA
TITLE MANUFACTURING PROCEDURE FOR PRESSCAKE
PREPARED BY: R. D. Alaburda RESPONSIBLE FOR TEST AND TEST CONCLUSION: R. D. Alaburda *RDA*

PURPOSE OF TEST:

To document the manufacturing procedure for producing "Lithosol" Red 4B Acid Presscake specifically in regard to sampling drums to determining the per cent solids.

DURATION OF TEST:

STANDARD INVOLVED:

Chambers Works Process 459-200, dated August 6, 1974, R. D. Gano.

ANALYTICAL CONTROL OR SPECIFICATIONS: ☒ No change ☐ Change described under Test DetailsPROCESS HAZARDS REVIEW: ☐ Required before test ☒ Not requiredMATERIAL SAFETY DATA SHEET: ☐ Revision required ☒ No changePACKAGING AND SHIPPING CLASSIFICATIONS: ☒ Revision required ☐ No change

TECHNICAL BASIS FOR CHANGE:

See Attached

TEST DETAILS:

(Include Safety and Waste Disposal)

See Attached

DISTRIBUTION:

Jackson Lab. Files
J. V. Flynn, Adm. Bldg.
E. J. Fritz, Int. West (3)
C. H. Lake, Bldg. 588
T. E. Lewis, G.A.L.

J. J. Meyer, D & C Bldg.
M. S. Moores, Azo Lab.
D. Putnam, G.A.L. (3)
R. F. Stalzer, G.A.L.
P. B. Weinberg, Int. West

APPROVED BY:

8 PM 1/7/76
J. J. Meyer 1/7/76
SUPV. MANUFACTURING DATE

ROG *M. S. Moores* 1/2/76
SUPV. TECH. ULT. ASSIST. DATE
T. E. Lewis 1/15/76
QUALITY CONTROL OR ANALYTICAL DATE

NOT REQUIRED

MANUFACTURING MANAGER* DATE

NOT REQUIRED

TECHNICAL DIVISION* DATE

AUTHORIZED BY:

[Signature] 1/20/76
SUPV. MANUFACTURING DATE

NOT REQUIRED

GEN. SUPV. MANUFACTURING* DATE

NOT REQUIRED

PLANT MANAGER* DATE

*MARK "NOT REQUIRED" WHEN APPLICABLE
M-1752 (Rev. 6/75)

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TECHNICAL BASIS FOR CHANGE

The Chambers Works Process 459-200 already defines the manufacture of "Lithosol" Red 4B Acid Presscake. This product, 4-Aminotoluene-3-Sulfonic Acid Purified Wet, was sold in the past as a presscake, and Sales has requested its return. The purpose of this Test Authorization and Conclusion is to document a reliable sampling procedure so that accurate billings can be made.

TEST DETAILS

Follow the standard Operating Instructions, Steps 1 through 20, for making 4-Aminotoluene-3-Sulfonic Acid Purified Wet. After the cake has been washed on the nutsch, follow the following presscake instructions.

Presscake Instructions

1. Dry on the nutsch for at least four hours.
2. Transfer the nutsch cake to FDA-510-9043 fiber drums. Load 225 pounds of the presscake into each drum using a polyethylene liner and disc with each drum.
3. Take three, eight-ounce composite samples from the lot. Two samples are for the laboratory and the third is for the file. Use the wet sand method for sampling.

The first laboratory sample should be taken from drums One through Six. The second laboratory sample should be from Drums Seven through Twelve. The file sample should be a composite from all twelve drums.

Wet Sand Method

Force a thief tube through the entire depth of the product in the drum. Remove the thief tube and discard the sample, leaving the hole undisturbed. Reinsert the thief in the original hole and rotate it until it is filled with a representative sample taken from the walls of the original hole. (This procedure is known as the "Wet Sand" sampling technique.) Sample each drum of a lot in this manner, placing the samples in a single plastic bag. Seal the bag and mix its contents thoroughly by kneading.

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4. Label the samples:

"Lithosol" Red 4B Acid Presscake Drummed.

Request:

"Per Cent H₂O" (105°C. Oven Dry) and Purity
(Dry Product).

5. The total charge will take ten to twelve drums. Label
each drum:

"Lithosol" Red 4B Acid Presscake Drummed.

6. Seal the drums and wipe clean before shipment.

TEST RESULTS

Eight good quality charges of "Lithosol" Red 4B Acid Presscake
were made using the above procedure. On two charges (Lot 384)
samples were taken from each drum to determine the variance between
drums and compared with the composite samples for the lot.

LOT	% PERCENT	% DRY	DRY WT	% STD YIELD	No. SAMPLES	AVERAGE % SOLIDS ALL DRUMS	STANDARD DEVIATION	COMPOSITE ANALYSIS FROM ALL DRUMS
1	99.6	88.0	1003	—				
2	99.6	86	1355	56				
3	99.5	80.5	1976	82	11	79.8	1.1	80.5*
4	99.5	85.5	1931	80	10	86.0	1.0	85.5
5	99.8	82.2	2220	93				
6	99.6	82.6	2230	93				
7	99.7	84	1512	63				
8	99.6	89	1802	75				

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NOTE: Sample 3 was mixed on the nutsch before it was loaded into the presscake drums. Lot 4 was not. Note that there is no difference in drum variance of the per cent solids between Lots 3 and 4. The conclusion is that there is no need to premix on the nutsch.

There is a good agreement between the per cent solids composite sample of the lot and the individual analysis from each drum. The difference was less than the 1.0 per cent standard deviation. Based on these results, we can expect the presscake lots to vary from 80 per cent to 90 per cent solids and each individual drum in the lot to be within 2 per cent (± 2 standard deviation, 95 per cent confidence interval) of the true per cent solids.

The standard yield of all of the lots was very poor. This was due to incomplete precipitation of the acid in the precipitation tubs and has since been corrected.

SAFETY

No significant change.

WASTE DISPOSAL

No significant change.

RECOMMENDATIONS

Continue to manufacture "Lithosol" Red 4B Acid Presscake as outlined in the above procedure.

RDA:glm