

R. D. Alaburda
November 1975

Chambers Works
234-459 180
459 184
I-1-1

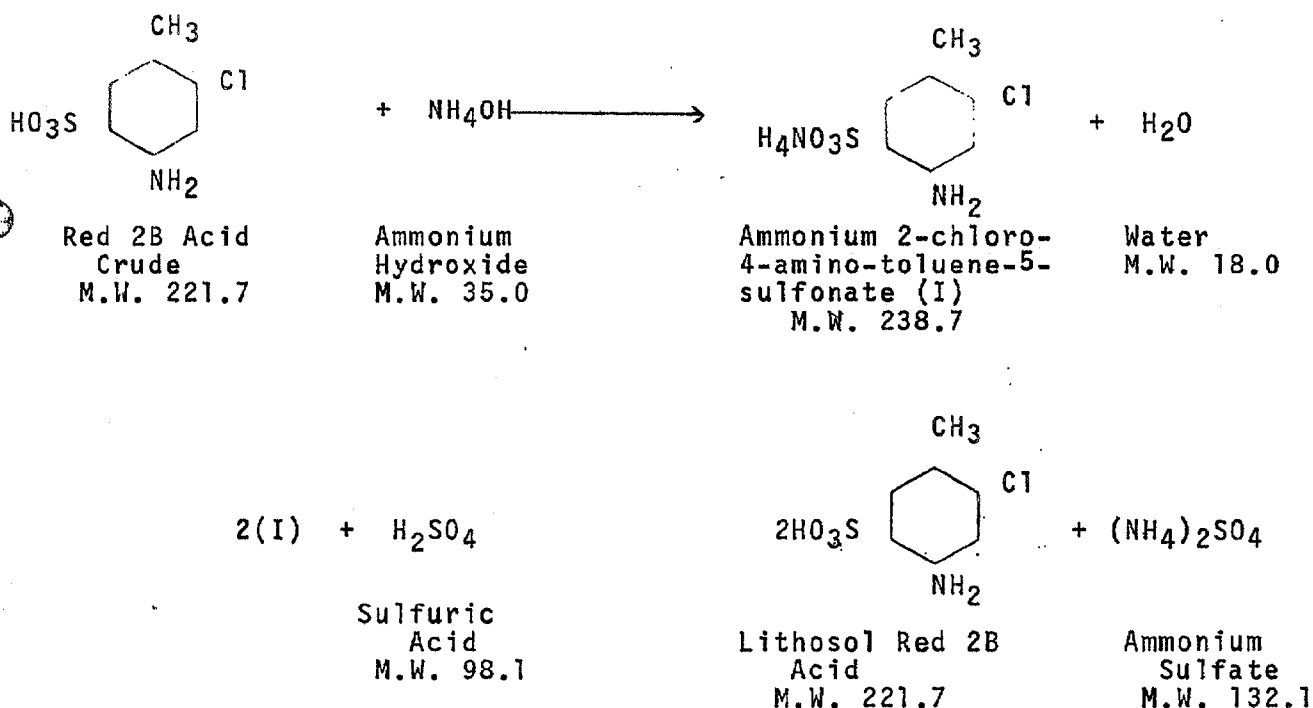
"LITHOSOL" RED 2B ACID PRESSCAKE

"LITHOSOL" RED 2B ACID

Principle of Process

Red 2B Acid Crude is dissolved in hot water with ammonia and clarified with Darco. The product is filtered then precipitated from the clarified solution with sulfuric acid, cooled, filtered and washed with water. The product is packed as wet cake from the nuts as "Lithosol" Red 2B Acid Presscake or it is dried and packed as "Lithosol" Red 2B Acid.

Reaction



Yield

The standard yield is 96.0% of theory.

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SUBJECT: "LITHOSOL" RED 2B ACID PRESSCAKE
"LITHOSOL" RED 2B ACID OPERATION: Materials and Equipment

Bill of Materials

<u>Code No.</u>	<u>Materials</u>	<u>Lbs./Chg.</u>	<u>Orchem Spec.</u>
848 200 2	Red 2B Acid Crude (2-Chloro-4-Toluidine- 5-Sulfonic Acid)	1,300	1. Purity 94.0% minimum 2. Unconv. 2.0% maximum
049 700	Ammonia 28%	294	31-A
190 000	Darco S-51	75	48-D
946 200	Super Cel	25	139-S
945 000	Sulfuric Acid	563	35-S

Product

459 180	"Lithosol" Red 2B Acid	1,250	59-RD
459 184	"Lithosol" Red 2B Acid Presscake	1,250	30-L-1

Equipment

DMA Bldg.

K-2	T-1	N-1
B-6	T-2	N-2

F-1 P-54

Technical Basis

Chambers Works Process 234-848 240, Red 2B Acid Purified by
R.D. Gano dated July 12, 1971.
TA-C-71-490 and conclusion dated August 19, 1971 "Purify Inferior
Red 2B Acid Crude".
TAC-72 835 and conclusion dated January 24, 1973 "Demonstrate
drying in ONA Graining Bowl".
TA-C-73-470 and conclusion dated August 6, 1973 "Purify Sub-
Normal Quality Crude".
TA-C-74-272 and conclusion dated November 4, 1974 "Purify Sub
Quality Crude".
TA-C-75-165 and conclusion dated March 5, 1975 "Purify Sub-
Normal Quality Crudes".

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Hazards

SUBJECT: "LITHOSOL" RED 2B ACID PRESSCAKE
"LITHOSOL" RED 2B ACID OPERATION:

Hazards

Red 2B Acid is not flammable or volatile. However, it may cause cyanosis or dermatitis. Wear rubber gloves when handling and a respirator if dusty.

Ammonia Aqua (ammonium hydroxide) is a colorless, alkaline liquid, m.p. -72°C . It is highly toxic by ingestion and moderately toxic by inhalation or contact. It can inflict serious burns. It can react with oxidizing materials. When heated it emits highly toxic fumes of ammonia. Wear rubber gloves, face shield, and acid suit when handling aqueous ammonia. Wash off affected areas with water should contact occur.

Filter Cake should be treated as a cyanogenic. Avoid skin contact by wearing rubber gloves and rubber boots when digging the nuts off. Wash off affected areas with water and report to Medical.

Sulfuric acid: A heavy corrosive, strong mineral acid varying from colorless to light brown in color, usually containing some white ferrous sulfate crystals. Will burn skin severely. Not flammable, although in contact with most metals hydrogen is released and can result in a pressure explosion. It is a dangerously strong oxidizing agent, liberates much heat when mixed with water (17.75 k-cal/mole), and spatters violently if water is poured into it. Wear rubber gloves, acid suit and face shield when handling sulfuric acid or where there is a possibility of leaks or spills. Wash off all affected areas with H_2O and report to Medical.

Super-Cel is a light tan to pink powder which has no known toxic properties. The effects of breathing the dusts are not known, but usual industrial care requires protection from dust inhalation.

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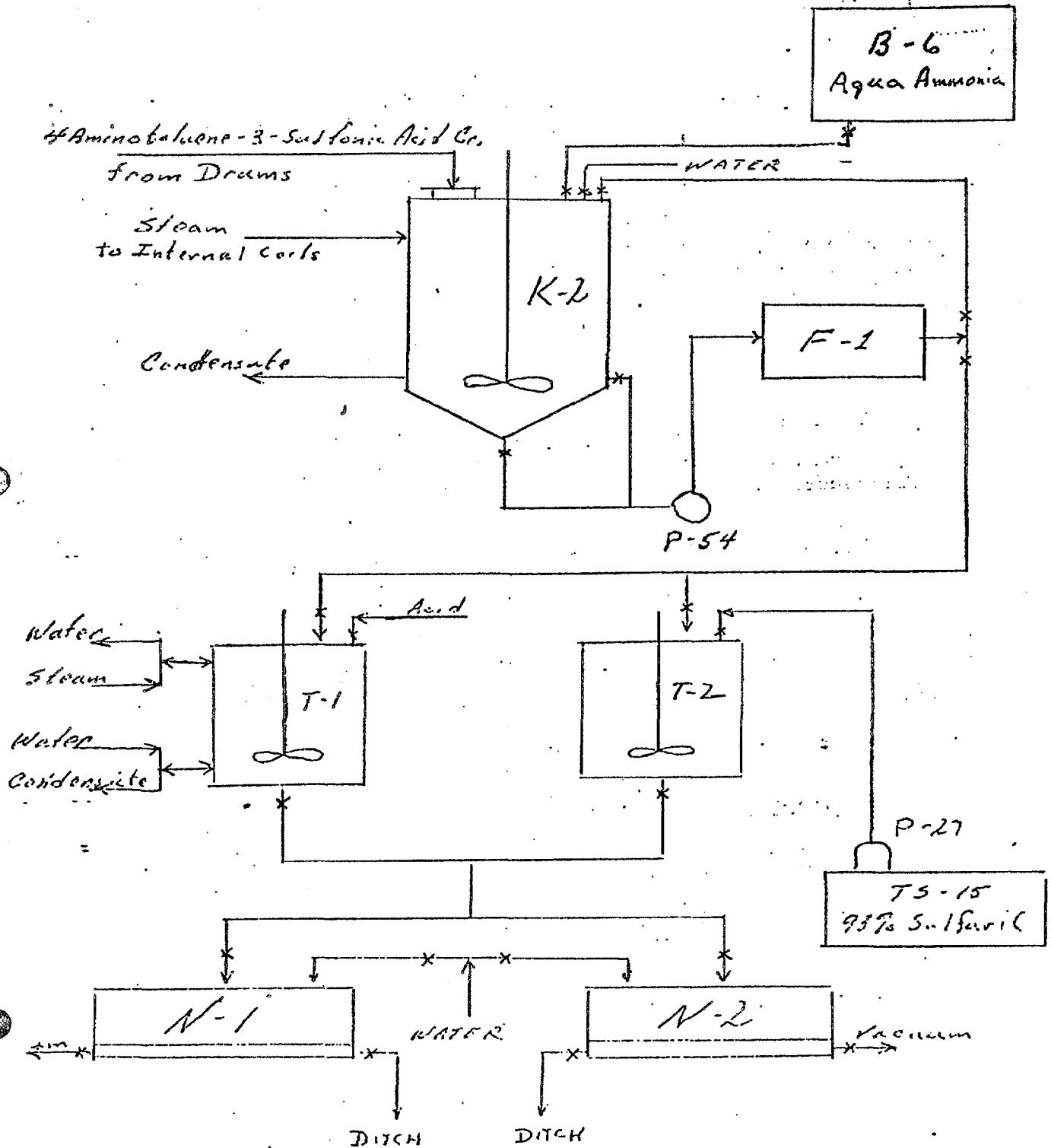
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Equipment Flow Sheet

SUBJECT: "LITHOSOL" RED 2B ACID PRESSCAKE
"LITHOSOL" RED 2B ACID OPERATION:



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SUBJECT: "LITHOSOL" RED 2B ACID PRESSCAKE
"LITHOSOL" RED 2B ACID OPERATION:

1. Charge kettle K-2 with 8,700 lbs. of water. Wet dip on side of K-2 should be 24 ± 2 ".

Check valves.

2. Put vacuum on blowcase B-6 and suck 1,050 lbs. ammonia 28% (or ammonia recovered 28%) into B-6. If spilled on clothing or skin, wash with cold water and be transported to Medical Building.

- a) Wear face shield.
- b) Check lines and valves.
- c) This is 10" in B-6.

3. Blow ammonia to kettle K-2 with the agitator on.

4. Charge K-2 with 1,300 lbs. Red 2B Acid Crude.

WEAR GLOVES AND IF PRODUCT IS DUSTY, WEAR
A RESPIRATOR.

5. Add 75 lbs. Darco and 25 lbs. Super Cel after all crude Red 2B Acid has been charged. Wear respirator if material is dusty.

6. Make total volume up with water to 70 ± 2 " wet dip on the side of K-2 (20,000 lbs. water volume).

Shut agitator off when rodding.

7. Mix and heat charge to boiling - $93-100^{\circ}\text{C}$ and boil one hour.

- a) Use automatic control on steam.
- b) Keep charge definitely alkaline (orange on Brilliant Yellow paper. Add more ammonia if necessary.
- c) Avoid fumes by using air on vent line.

8. After boiling vigorously for one hour, make up volume to 70 ± 2 " side wet dip again, and heat to 95°C .

Make up filter press - set lines to return to K-2.

9. Filter charge through press and back to K-2 until filtrate from all press spigots runs clear and free from Darco, etc.

Stand by during filtration. Pump from K-2 using pump P-54 and take off on side of cone at bottom of K-2.

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SUBJECT: "LITHOSOL" RED 2B ACID PRESSCAKE
"LITHOSOL" RED 2B ACID OPERATION:

10. When filtrate runs clear from all spigots, change lines and run filtrate to Haveg tubs T-1 and T-2.

Watch dip in T-1 and T-2, although charge should fit in with room to spare for the sulfuric acid. Divide charge evenly between T-1 and T-2.

11. After filtration is complete, fill K-2 cone with water (2,000 lbs.), heat to 90°C, and circulate through the press and back to K-2.

- a) Wash press for at least 5 minutes.
- b) Press will hold residue from 3 charges before cleaning is required. Blow dry before discarding sludge.
- c) Dump sludge into cans and label "Darco Sludge from Red 2B Acid".
- d) Start recharging K-2 as in Step No. 1 for next charge.

12. Heat charge in T-1 and T-2 to 95°C, agitation on.

13. Put steam on slowly to avoid rupturing lead coils. Always turn steam off coils before adding acid.

14. Add the 93% sulfuric acid slowly, splitting the 563 lbs. of acid equally between each tub until each tub is acid (blue) to Congo Red paper. Steam off during acid addition.

- a) Wear an acid suit; place warning sign on stairs.
- b) Acidification should take place over approximately one hour at a steady rate. Acid stream should be about size of a pencil. The product precipitates.

If spilled on clothing or skin, wash with cold water and be transported to Medical Building.

15. Test each tub for completeness of precipitation; add more sulfuric acid, if necessary, until a test shows the precipitation is complete.

Take sample, cool to 30°C, filter, and add a few drops of sulfuric acid to the clear filtrate. If no precipitate forms, the acidification is complete. If a precipitation occurs, add a few more pounds of sulfuric acid to the charge and request the test. Incomplete precipitation will result in yield loss. Wear an acid suit when performing precipitation test.

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SUBJECT: "LITHOSOL" RED 2B ACID PRESSCAKE
"LITHOSOL" RED 2B ACID OPERATION:

16. When charge is completely precipitated in each tub, cool to 25-30°C or colder.
17. At 25-30°C or colder, drop contents from both tubs to one nutsch, either N-1 or N-2.

This will require several loadings on the nutsch.

18. Wash cake, after sucking down, with 5 cold water washes for presscake and 10 water washes for the dried "Lithosol" Red 2B acid, sucking down after each wash. The water should be 3" above the cake level on each wash.

Excessive washes will result in yield loss.

19. Suck nutsch dry for at least 4 hours.

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SUBJECT: "LITHOSOL" RED 2B ACID PRESSCAKE
"LITHOSOL" RED 2B ACID OPERATION:

"Lithosol" Red 2B Acid Presscake

1. Pack the presscake into ten FDA 510 9043 fiber drums with polyethylene liners. Load 225 lbs. net into each drum.

WEAR RUBBER GLOVES AND RUBBER BOOTS WHEN DIGGING
THE NUTSCH.

2. Take at least four composite samples from the lot. Three samples are for the lab and the fourth is for the file.

The samples should be composite samples from the drums. Use the wet sand method. Take a composite sample from every three drums. The file sample can be a composite from all samples.

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 tube 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.

3. Label the samples:

"Lithosol" Red 2B Acid Presscake.

Request: % Solids (Oven dry) - All lab samples
% Purity (dry product) (one sample only)
% Unconverted and Solubility. (one sample only)

4. Label each drum: "Lithosol" Red 2B Acid Presscake.
5. Seal containers with a polyethylene disc and wipe clean for shipment.

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SUBJECT: "LITHOSOL" RED 2B ACID PRESSCAKE
"LITHOSOL" RED 2B ACID OPERATION:

ANALYTICAL REPORT		AREA INTERMEDIATES — WEST		BLDG. DMA		CHARGE NO.
COST CODE 1351		SAMPLE OF LITHOSOL RED 2B ACID Presscake				
PRODUCT CODE 459 184						
SENT BY: _____						
DATE: _____		A. M. P. M.				LAB ONLY
ANALYZE FOR	✓	TECH. NO.	RESULTS			
% PURITY (Dry Product)						
UNCON						
SOLUBILITY						
% Solids (oven dried)						

MU-156-R6

SAMPLE OF: LITHOSOL RED 2B ACID PRESSCAKE				HAZARDS	
No. ✓	LOT	CHARGE	EQUIPMENT	TOXIC	
✓	✓	✓	✓	☒	
ADMEY	DMA			CORROSIVE	
BUILDING	AREA	TAKEN BY		☐	
DATE	TIME			FLAMMABLE	
ADDN'L. INFO.				☐	
				NO UNUSUAL HAZARD	
				☐	

MU-712-A-R1

DUP050467869

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SUBJECT: "LITHOSOL" RED 2B ACID PRESSCAKE
"LITHOSOL" RED 2B ACID OPERATION:

"Lithosol" Red 2B Acid

1. Pack the presscake into 10 reclaimed drums with quick locks for drying in GB-3.

WEAR RUBBER GLOVES AND RUBBER BOOTS WHEN DIGGING THE NUTSCH.

2. Label the drum: "Lithosol" Red 2B Acid Presscake.
3. Follow the general operating procedure for operating GB-3.
4. Load the drum into GB-3 then seal GB-3. Accumulate 2 charges of 20 drums before drying in GB-3.
5. Use 50 psig steam on the jacket and dry at 100 to 110°C with full vacuum. The jacket temperature will reach 125-130°C.
6. Dry in GB-3 for 12 to 14 hours.

The vacuum should be down to 25 mm Hg for at least one hour for adequate drying.

7. Sample for moisture at the end of the drying period.
8. If the moisture is less than 0.5%, dump the charge into 11) FDA 510 9043 leverpaks at 225 lbs. net. If the moisture is too high, dry for an additional 2 hours and repeat Step 7.
9. Take three 8 oz. samples; from the drums; one for GAL, one for the file and the third for the Technical Laboratory.

Label: "Lithosol" Red 2B Acid

Request: Purity	99.0% min
Solubility	complete
and Unconverted	0.25% max.

10. Label the leverpaks - "Lithosol Red 2B Acid".
11. Seal and wipe clean each drum for shipment.

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SUBJECT: "LITHOSOL" RED 2B ACID PRESSCAKE
"LITHOSOL" RED 2B ACID OPERATION:

"These Operating Instructions have been issued after a full review of current technology and building experience as of Nov 13, 1975 (date), and conform with the current process technology as recorded."

R. D. Alaburda (Supervisor's signature)

Building Supervisor W. F. Zimmerman Date 11-14-75
Senior Supervisor George Rose Date 2/3/76
Chief Supervisor, Operations J. James E. Kinty Date 1/18/76
Chief Supervisor, Tech. or Plant Assist. R. G. McManus Date 1/6/76

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