

10/22 (1) RSG  
(2) File

~~M.S. Moore~~

R. D. Alaburda  
October 1975

Chambers Works  
234-049 340/459 200  
I-1-1

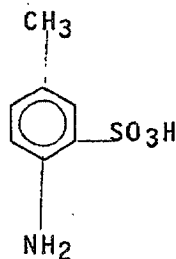
"LITHOSOL" RED 4B ACID  
(4-Aminotoluene-3-sulfonic Acid Purified)

Principle

4-Aminotoluene-3-sulfonic acid crude is dissolved in water as the sodium salt by the addition of soda caustic liquid. Darco and Super Cel are added to absorb the colored impurities and to aid filtration. The solution is heated to 95°C, filtered, and the filtrate acidified with sulfuric acid to precipitate 4-amino-toluene-3-sulfonic acid as the free acid. After cooling, the 4-aminotoluene-3-sulfonic acid is isolated in a nutsch, dried and ground for sale as "Lithosol" Red 4B Acid.

Reaction

The only chemical reaction involved is the conversion of the acid to the soluble sodium salt and, after clarification, the reconversion to the free acid by the addition of sulfuric acid. The product structure is:



4-Aminotoluene-3-sulfonic acid  
("Lithosol" Red 4B Acid)  
M.W. 187.2

# OPERATING INSTRUCTIONS

R. D. Alaburda  
October 1975

Chambers Works  
234-049 340/459 200  
III-1

**SUBJECT:** "LITHOSOL" RED 4B ACID  
(4-AMINOTOLUENE-3-SULFONIC  
ACID PURIFIED)

**OPERATION:**

## Equipment

K-2	T-1	TS-18	N-1	GB-3
F-1	T-2	P-54	N-2	ML-1
TS-15	P-27	P-45		

## Bill of Materials (100% Basis)

<u>Materials and Orchem</u> <u>Product Code Numbers</u>	<u>Lb./</u> <u>Chg.</u>
4-Aminotoluene-3- sulfonic acid crude 049 335	2,600
Caustic Soda Liquid 30% 887 500	535.5
Sulfuric Acid 945 000	665
Darco S-51 190 000	100
Super Cel 946 200	50

## Product

4-Aminotoluene-3- sulfonic acid purified 049 340	2,424
"Lithosol" Red 4B Acid 459 200	2,400

## Reference:

Chambers Works Process, 049 340/459 200 for the manufacture of "Lithosol" Red 4B Acid/4-Aminotoluene-3-sulfonic Acid Purified, R.D. Gano, dated August 6, 1974.

## Batch Size and Yield

The Bill of Materials is written for a standard 2,400-lb. charge in a 2,800-gal. solution kettle. The charge size may be varied proportionately within the working limits of the available equipment.

No goal yield has been established.

## OPERATING INSTRUCTIONS

R. D. Alaburda  
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Chambers Works  
234-049 340/459 200  
III-2

SUBJECT: "LITHOSOL" RED 4B ACID  
(4-AMINOTOLUENE-3-SULFONIC  
ACID PURIFIED)

OPERATION:

### Hazards

Sulfuric acid is a highly corrosive, dangerous liquid. It generates heat on dilution with water. The weak acid attacks most metals with the liberation of flammable hydrogen. The acid attacks and destroys skin tissue. Wear rubber gloves and face shield when handling this material as the minimum protection. "Chem Suits" should be worn where splashes or leaks are possible. If splashed, wash off with copious amounts of water and report to the hospital.

Soda caustic liquid is extremely corrosive in contact with all body tissue, whether as liquid or vapor, and even in dilute solutions. It is nonflammable, but generates heat when mixed with acids or water. If splashed, wash off with copious amounts of water and report to the hospital.

"Lithosol" Red 4B Acid (4-Aminotoluene-3-sulfonic acid) is an aromatic amine compound which may be hazardous. Avoid contact with this material. Wear respirator for dust exposure.

### Waste Disposal

The sludge from the clarification press is sent to the dump. The final filtrate is a dilute solution containing sodium sulfate and traces of 4-aminotoluene-3-sulfonic acid. This waste is run to the plant ditch.

### Cycle Time

The cycle time for beginning of charging in K-2 to shoveling off the nuts is 24 hours.

Drying time is 24 hours.  
Milling time is 8 hours.

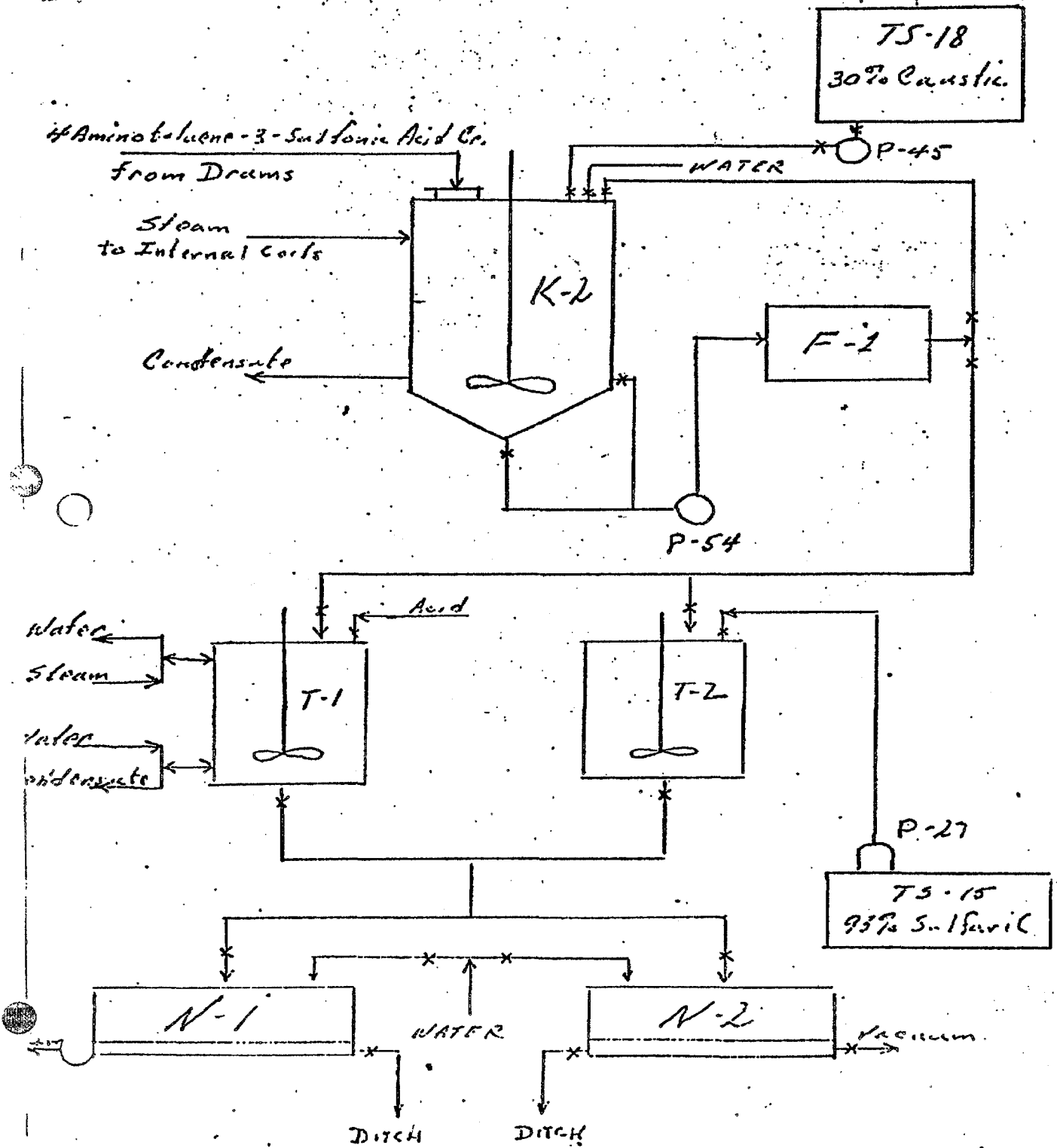
R. D. Alaburda  
October 1975

# OPERATING INSTRUCTIONS

Chambers Works  
234-049 340/459 200  
III-3

SUBJECT: "LITHOSOL" RED 4B ACID  
(4-AMINOTOLUENE-3-SULFONIC  
ACID PURIFIED)

OPERATION:



## OPERATING INSTRUCTIONS

R. D. Alaburda  
October 1975

Chambers Works  
234-049 340/459 200  
III-4

**SUBJECT:** "LITHOSOL" RED 4B ACID  
(4-AMINOTOLUENE-3-SULFONIC  
ACID PURIFIED)

**OPERATION:**

1. Charge K-2 kettle with 60 inches of water. Turn agitation on.

Measure dip on straight side of kettle.

2. Add 166 gallons of Caustic soda to K-2 through meter. (535.5 lbs. 100%).

WEAR RUBBER GLOVES AND FACE SHIELD. CAUSTIC SODA IS CORROSIVE AND WILL CAUSE SEVERE BURNS ON THE SKIN. WASH ANY FROM HANDS WITH WATER. REPORT TO MEDICAL DEPT.

3. Start heating K-2 to 90-95°C and turn on agitator. Charge in about 2400 lbs. of 4-Aminotoluene-3-Sulfonic Acid Crude.

Agitation must be on. 200 lbs. of crude is held out to adjust to test. Use gloves, if dusty use respirator.

4. Check alkalinity on Brilliant Yellow papers.

Test must be orange, not red color.

5. Adjust K-2 with balance of crude (200 lbs. held out) and with caustic soda as needed.

Use 2600 lbs. crude total. Final alkalinity on Brilliant Yellow Paper must be a light orange, not red. Add crude if test is red. Add caustic if test is yellow.

6. Add 100 lbs. Darco.

Add slowly--sprinkle. Otherwise foam-over may occur.

7. Add more water to K-2 to bring volume up to 80 inches total.

Measure on straight side of K-2. Stop agitator before measuring.

8. Heat to 95°C and hold 1/2 hour.

9. Test for color by filtering a sample. The filtrate should not be darker than a light amber color.

Add 50 lbs. more Darco and repeat test if too dark. Check supervision if color remains dark after addition.

## OPERATING INSTRUCTIONS

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October 1975

Chambers Works  
234-049 340/459 200  
III-5

**SUBJECT:** "LITHOSOL" RED 4B ACID  
(4-AMINOTOLUENE-3-SULFONIC  
ACID PURIFIED)

**OPERATION:**

10. Steam press and lines to be sure they are clear and hot.  

Lines and press must be hot or plugging will develop.
11. Start pump P-54 and circulate solution to F-1 press dressed with cotton duck #10 and back to K-2 until all spigots turn clean and clear.  

All Darco must be removed; otherwise, quality will be impaired.
12. Switch lines to run filtrate to T-1 & T-2 dividing the charge evenly.  

Both tubs will hold the charge from K-2.
13. When K-2 is empty, wash press with hot water back to K-2.  

Fill K-2 cone.
14. Blow press dry to K-2 and dump to cans. Label cans "Darco Sludge from 4-Aminotoluene-3-sulfonic Acid".  

It is sometimes necessary to rework Darco sludge to avoid yield loss if the press does not wash properly. Supervision will advise if this is necessary.
15. Turn on agitation. Heat T-1 & T-2 with coil steam to 90°C to 95°C.  

Be sure agitation is on.
16. Shut steam off tub coils and start slow addition of sulfuric acid to both T-1 & T-2. Acid is added until both tubs are definitely acid (blue) on Congo Red paper.  

The acid addition should take about one hour. (Stream the size of a pencil.) Be sure steam is off coils or tubs tend to boil over. Temperature must be at 90 to 95°C during addition.

Shut acid off either tub should it become acid before the other.

Do not add an unnecessary excess of acid.

WEAR RUBBER GLOVES AND FACE SHIELD.

## OPERATING INSTRUCTIONS

R. D. Alaburda  
October 1975

Chambers Works  
234-049 340/459 200  
III-6

**SUBJECT:** "LITHOSOL" RED 4B ACID  
(4-AMINOTOLUENE-3-SULFONIC  
ACID PURIFIED)

**OPERATION:**

17. Cool charge to 25-30°C and test for complete precipitation.

Additional sulfuric acid added to a sample of filtrate should show no precipitate.

18. Drop charge to either N-1 or N-2.

Both tubs go on 1 nutsch.

19. Rinse out tubs to nutsch.

Avoid using a large quantity of water, no more than 100 gallons.

20. Put 2 spray washes on the cake on the nutsch and suck dry.

Spray 2 to 3 minutes per wash (approximately 250 gallons).

21. Transfer to clean cans.

Label "4-Aminotoluene-3-Sulfonic Acid Purified Wet".

22. Dry in GB-3 at 100 ± 5°C.

One charge will usually be dried at a time. Follow instructions for graining bowl operation.

23. Dump dry product into clean steel can and allow to cool for milling.

Use a respirator and gloves. Dump at 0.5% water or less. Dryer is usually dry in morning after loading on day shift.

24. Grind on ML-1. Pack product at 225 lbs. net.

Sample each charge for: % Purity at lab. One sample for file. Follow separate instructions for milling.

25. Label: "Lithosol" Red 4B Acid.

10 to 12 packs per charge.

26. Containers must be wiped clean before shipping.

# OPERATING INSTRUCTIONS

R. D. Alaburda  
October 1975

Chambers Works  
234-049 340/459 200  
III-7

**SUBJECT:** "LITHOSOL" RED 4B ACID  
(4-AMINOTOLUENE-3-SULFONIC  
ACID PURIFIED)

**OPERATION:**

ANALYTICAL REPORT		AREA	INTERMEDIATES			BLOG.	D M A				
COST CODE <u>1351</u>		SAMPLE OF  <b>LITHOSOL RED 4B ACID</b>					CHARGE NO.				
PRODUCT CODE _____											
SENT BY: _____											
DATE: _____		A. M.									
		P. M.									
ANALYZE FOR	✓	TECH. NO.	RESULTS			LAB ONLY					
PURITY											
SCREEN TEST											
40 MESH											
60 MESH											

MU-156-86

SAMPLE OF: <u>Lithosol Red 4B Acid</u>			HAZARDS	
No. <input checked="" type="checkbox"/>	LOT <input checked="" type="checkbox"/>	CHARGE <input checked="" type="checkbox"/>	EQUIPMENT <input checked="" type="checkbox"/>	TOXIC <input checked="" type="checkbox"/>
BUILDING <input checked="" type="checkbox"/>	AREA <input checked="" type="checkbox"/>	DATE <input checked="" type="checkbox"/>	TAKEN BY <input checked="" type="checkbox"/>	CORROSIVE <input type="checkbox"/>
ADDN'L. INFO.			TIME <input checked="" type="checkbox"/>	FLAMMABLE <input type="checkbox"/>
				NO. UNUSUAL HAZARD <input type="checkbox"/>

MU-714A-81

MU-2341 R2

DUP050467880

# OPERATING INSTRUCTIONS

R. D. Alaburda  
October 1975

Chambers Works  
234-049 340/459 200  
III-8

MU-1766-R-1

## WASTE CHEMICAL TRANSPORTATION ORDER

ORDER NO.  
**79453**

REQUESTED BY--  AREA Intermediates PHONE 472 DATE--

**MATERIAL**

NAME Darco Sludge From 4 Aminotoluene 3 Sulfonic Acid APPROX. QUANTITY  
LOCATION (IN DG. OR NAME) DMA Line POUNDS--  
GALLONS--

FORM-- LIQUID  POWDER  GRANULAR  PASTE  SLURRY  OTHER

CONTAINER	NO. OF CONTAINERS	NO. OF CONTAINERS	HAZARDS
DRUM		DUMPSTER TANK	IRRITANT RESPIRATORY IRRITANT CORROSIVE FLAMMABLE CYANOTIC POISONOUS EXPLOSIVE DUST CAN CAUSE BRONCHITIS FORMS POISONOUS GAS ON CONTACT WITH ACID EXPOSURE MAY RESULT IN HEAD & THROAT CANCER
OPEN HEAD		UNHEATED	
CLOSED HEAD	<input checked="" type="checkbox"/>	HEATED	
LEVERPAK		DUMPSTER HOPPER	
N. G. TANK		OPEN	
UNHEATED		CLOSED	
HEATED		TRUCK TANK TRAILER	
N. G. SLUDGE CAR		UNHEATED	
		HEATED	

TO BE HAULED BY	TO BE DELIVERED TO
TRUCK	LIQUID WASTE INCINERATOR--TO BE BURNED
OPEN FLAT	OPEN PIT TRASH BURNER--TO BE BURNED
OPEN FLAT WITH TAN. GATE LIFT	SLUDGE DISPOSAL AREA--DUMP & BURY
DUMP	OPEN DITCH
PICKUP	HYDRO SLURRY TANK
PICKUP WITH TAN. GATE LIFT	TANK PIT
TRACTOR	
FORK LIFT WITH "LO-BOY"	
DUMPSTER TRACTOR	
N. G. ENGINE	

DRIVER-- DATE-- RECEIVED BY--

MU-1123

# SLUDGE

Contains: 4 Aminotoluene 3 Sulfonic Acid

To: Dump

From: DMA Line

Shipped By

**HAZARDS:**

- Corrosive
- Cyanotic
- Flammable
- Toxic
- No Unusual Hazard

**Other Hazard (Describe)**

MU-2361 R2

OPERATING INSTRUCTIONS

R. D. Alaburda  
October 1975

Chambers Works  
234-049 340/459 200  
III-9

SUBJECT: "LITHOSOL" RED 4B ACID  
(4-AMINOTOLUENE-3-SULFONIC  
ACID PURIFIED)

OPERATION:

"These Operating Instructions have been issued after a full review of current technology and building experience as of Oct 8, 1975 (date), and conform with the current process technology as recorded."

R. D. Alaburda (Supervisor's signature)

Building Supervisor Raymond R. Alaburda Date Oct 8, 1975

Senior Supervisor James S. Chorley, Jr Date 10/8/75

Chief Supervisor, Operations [Signature] Date 10/16/75

Chief Supervisor, R.D.A. Tech. or Plant Assist. [Signature] Date 10/15/75

/lec/paw  
#4328