W. K. Kessler
April, 1974

Chambers Works
234-448 300
I-1-1

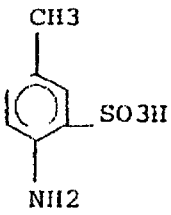
"LITHOSOL" ACID RBA
(4-Aminotoluene-3-sulfonic Acid Purified)

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 adsorb 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-sulfinic acid is isolated in a nutsch, dried and ground for sale as "Lithosol" Acid RBA.

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" Acid RBA)
M.W. 187.2

DUP050467889

OPERATING INSTRUCTIONS

W. K. Kessler
April, 1974

Chambers Works
234-448 300
III-1

SUBJECT: 4-AMINOTOLUENE-3-SULFONIC ACID PURIFIED "LITHOSOL" ACID RBA 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)

Materials and Orchem Product Code Numbers	Lb./ Chg.
4-Aminotoluene-3-sulfonic acid crude 049 335	2,600
Caustic Soda Liquid 30% 877 500	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 or "Lithosol" Acid RBA 448 300	2,400

Reference:

Chambers Works Process for the Manufacture of "Lithosol" Acid RBA, dated July 28, 1969.

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.

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N33036.01

OPERATING INSTRUCTIONS

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234-448 300
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SUBJECT: 4-AMINOTOLUENE-3-SULFONIC ACID PURIFIED "LITHOSOL" ACID RBA 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 Sutis" 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" Acid RBA (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 from beginning of charging in K-2 to shoveling off the nutsch is 24 hours.

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

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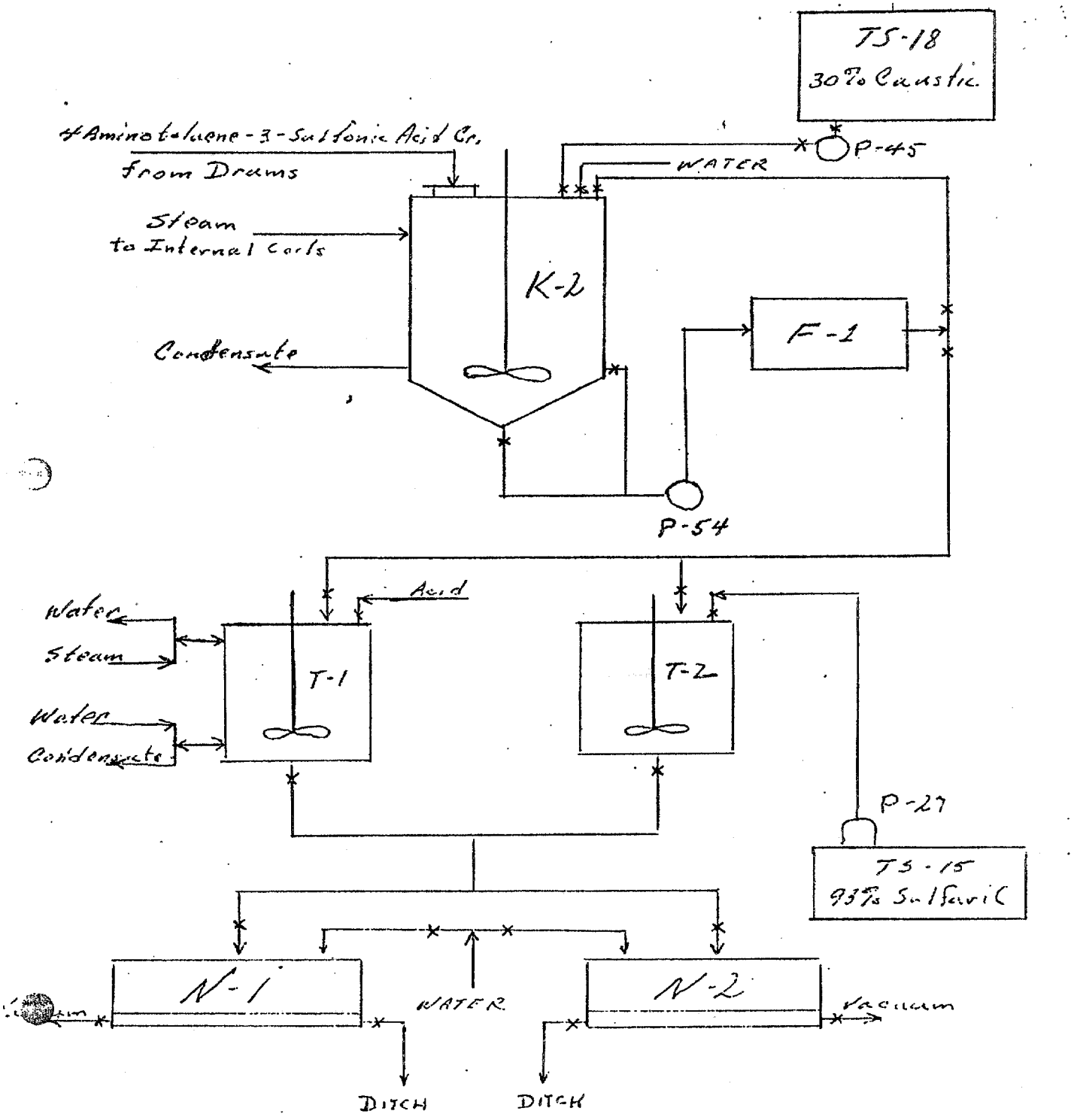
OPERATING INSTRUCTIONS

W. K. Kessler
April, 1974

Chambers Works
234-448 300
III-3

SUBJECT: 4-AMINOTOLUENE-3-SULFONIC ACID PURIFIED "LITHOSOL" ACID RBA

OPERATION: Equipment Flow Sheet



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OPERATING INSTRUCTIONS

W. K. Kessler
April, 1974

Chambers Works
234-448 300
III-4

SUBJECT: 4-AMINOTOLUENE-3-SULFONIC ACID PURIFIED "LITHOSOL" ACID RBA

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 <u>orange</u>, 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.

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OPERATING INSTRUCTIONS

SUBJECT: 4-AMINOTOLUENE-3-SULFONIC ACID PURIFIED "LITHOSOL" ACID RBA

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 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. Lable cans "Darco Sludge from Lithosol Acid RBA".

    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 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 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.

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SUBJECT: 4-AMINOTOLUENE-3-SULFONIC ACID PURIFIED "LITHOSOL" ACID RBA 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.

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

    Spray 2 to 3 minutes per wash.

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 to leverpaks 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" Acid RBA.

    10 to 12 packs per charge.

26. Containers must be wiped clean before shipping.

DUP050467895

OPERATING INSTRUCTIONS

W. K. Kessler
April, 1974

Chambers Works
234-448 300
III-7

SUBJECT: 4-AMINOTOLUENE-3-SULFONIC ACID PURIFIED "LITHOSOL" ACID RBA

OPERATION:

ANALYTICAL REPORT — AREA: INTERMEDIATES — BLDG. D M A

COST CODE 1351
PRODUCT CODE
SENT BY:
DATE: A. M. P. M.

SAMPLE OF: LITHOSOL ACID R B A

CHARGE NO.

LAB ONLY

ANALYZE FOR	✓	TECH. NO.	RESULTS			
PURITY						
SCREEN TEST						
40 MESH						
60 MESH						

MU-136-R6

SAMPLE OF: Lithosol Acid RBA

No. ✓ LOT ✓ CHARGE ✓ EQUIPMENT
✓ BUILDING ✓ AREA ✓ TAKEN BY
✓ DATE ✓ TIME
ADDN'L. INFO.

MU-712-A-NI

HAZARDS

TOXIC ☑
CORROSIVE ☐
FLAMMABLE ☐
NO UNUSUAL HAZARD ☐

MU 838-R8

DUP050467896

OPERATING INSTRUCTIONS

W. K. Kessler
April, 1974

Chambers Works
234-448 300
III-8

MU-1756-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

LOCATION (BLDG.) OR NAME) DMA Line

APPROX. QUANTITY
POUNDS-- ✓
GALLONS--

FORM—LIQUID [ ] POWDER [ ] GRANULAR [ ] PASTE [✓] SLURRY [ ] OTHER [ ]

CONTAINER	NO. OF CONTAINERS		NO. OF CONTAINERS
DRUM		DEMPSTER TANK	
OPEN HEAD		UNHEATED	
CLOSED HEAD	✓	HEATED	
LEVERPAK		DEMPSTER HOPPER	
N. G. TANK		OPEN	
UNHEATED		CLOSED	
HEATED		TRUCK TANK TRAILER	
N. G. SLUDGE CAR		UNHEATED	
		HEATED	

HAZARDS	
SKIN IRRITANT	
RESPIRATORY IRRITANT	
CORROSIVE	
FLAMMABLE	
CYANOTIC	✓
POISON	✓
EXPLOSIVE	
DUST CAN CAUSE SILICOSIS	
FORMS POISON GAS ON CONTACT WITH ACID	
EXPOSURE MAY RESULT IN BLADDER TUMOR	

TO BE HAULED BY TRUCK	
OPEN FLAT	
OPEN FLAT WITH TAIL GATE LIFT	
DUMP	
PICKUP	
PICKUP WITH TAIL GATE LIFT	
TRACTOR	
FORK LIFT WITH "LO-BOY"	
DEMPSTER TRACTOR	
N. G. ENGINE	✓

TO BE DELIVERED TO	
LIQUID WASTE INCINERATOR—TO BE BURNED	
OPEN PIT TRASH BURNER—TO BE BURNED	
SLUDGE DISPOSAL AREA--DUMP & BURY	✓
OPEN DITCH	
HYLENE SLURRY TANK	
TAR PIT	

DRIVER— DATE-- RECEIVED BY--

MU-1123

SLUDGE

Contains: 4 Aminotoluene 3 Sulfonic Acid

To: Dump

From: DMA Line

Shipped By ✓

HAZARDS:

		Other Hazard (Describe)
Corrosive	[✓]	
Cyanotic	[✓]	
Flammable	[ ]	
Toxic	[ ]	
No Unusual Hazard	[ ]	

DUP050467897

OPERATING INSTRUCTIONS

W. K. Kessler
April, 1973

Chambers Works
234-448 300
III-9

SUBJECT: 4-AMINOTOLUENE-3-SULFONIC ACID PURIFIED "LITHOSOL" ACID RBA

OPERATION:

"These Operating Instructions have been issued after a full review of current technology and building experience as of 1-15-74 ~~1-1-73~~ (date), and conform with the current process technology as recorded." (signed) W K Kessler (Supervisor's signature).

Building Supervisor Warren K. Kessler Date 1-17-74

Senior Supervisor W L Rewkirk Date 1/30/74

Chief Supervisor, Operations [illegible] Date 2/28/74

Chief Supervisor, RDG Tech. or Plant Assist. [illegible] Date [illegible]

/ehb
2604

NU-2331-82

DUP050467898