

APPROVED: OCTOBER 22, 1953

CHAMBERS WORKS PROCESS
FOR THE MANUFACTURE OF
"LITHOSOL" ACID RBA

(6-Amino-m-toluenesulfonic acid)

HISTORICAL

This process replaces the Dye Works Process for the Manufacture of Para Toluidine meta Sulfonic Acid Purified, approved June 21, 1938. There are no major alterations. The changes in the process cover the use of larger equipment and material of construction.

REFERENCES

Dye Works Process for the Manufacture of Para-Toluidine-Meta Sulfonic Acid, approved March 28, 1924.

Dye Works Process for the Manufacture of Para-Toluidine-Meta Sulphonic Acid, approved April 10, 1931.

Dye Works Process for the Manufacture of Para Toluidine-Meta Sulfonic Acid Purified, approved June 21, 1938.

PATENT STATUS

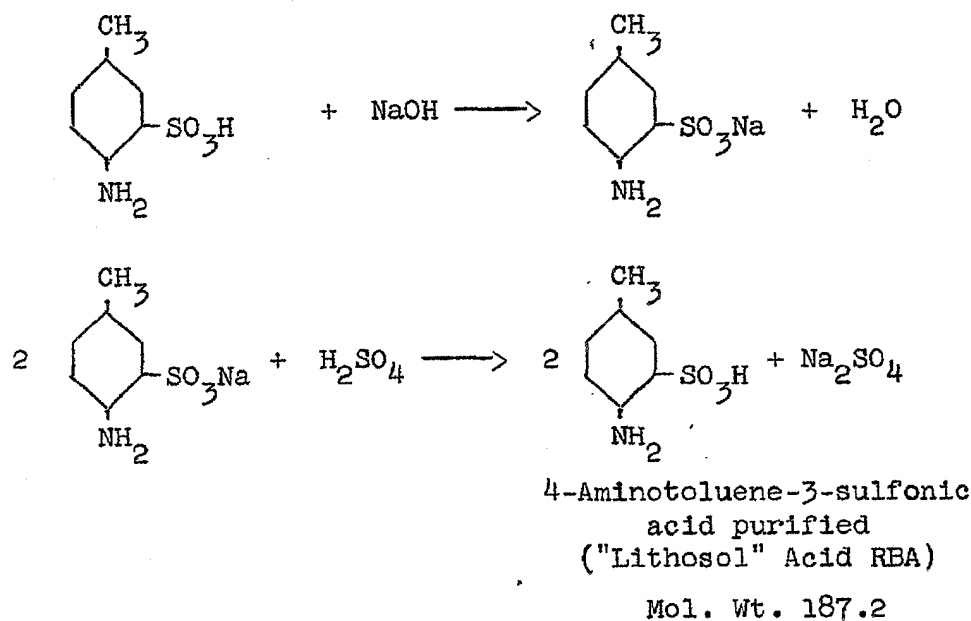
We are free to operate this process. See AN-4619, J. P. Phalan, October 22, 1953.

PRINCIPLE

4-Aminotoluene-3-sulfonic acid crude is dissolved in water as the sodium salt by the addition of soda caustic. Darco and Super Cel are added to absorb color and to aid filtration. The solution is heated to 95°C., filtered and the filtrate acidified with sulfuric acid to precipitate 4-aminotoluene-3-sulfonic acid

as the free acid. After cooling the 4-aminotoluene-3-sulfonic acid purified is filtered off on a nutsch, sucked as dry as possible, dried and ground as "Lithosol" Acid RBA.

Reactions



YIELD

The yield per charge is 2325 lbs. of 4-aminotoluene-3-sulfonic acid purified (Mol. Wt. 187.2) which is 93 per cent of theory based on 4-aminotoluene-3-sulfonic acid crude.

EQUIPMENT

Miscellaneous Intermediates Area - DMA Autoclave House No. 234

Kettle - K-2 - A vertical welded steel vessel with a cone shaped bottom 7-ft. 7-in. diameter by 8-ft. deep, capacity 2797 gals. Equipped with 2-in. iron coils connected to steam at 90 lbs. pressure. Also equipped with a gate-type agitator.

Tubs T-1 and T-2 - Each of these tubs is a Haveg 6-ft. x 6-ft. vertical tub of 1325 gals. capacity. Equipped with 2-in. lead coils wooden lead covered gate-type agitator, post and cover. The coil is connected to steam at 90 lbs. pressure and raw cooling water.

Filter Press - F-1 - A 24-in. Shriver press with 25-in. x 1-in. iron plates and frames. It is fitted with hydraulic jack.

Pump - P-24 - An American cast-iron centrifugal pump, size 1-1/4-in. direct driven by a 5-hp. motor.

Nutsch - N-1 and N-2 - Wooden 9-ft. diameter by 4-ft. deep fitted with Vinyon filter cloth.

Jet Exhauster - J-6 - A Schutte-Koerting single stage steam and water exhauster for high capacity, low vacuum.

Tank - TM-8 - A riveted steel tank, 4-ft. 6-in. diameter by 5-ft. high, capacity 480 gals. equipped with sight glass, vented to ditch and piping to miscellaneous equipment.

Tank - TS-15 - Riveted steel tank 4-ft. diameter by 10-ft. long, capacity 1000 gals., equipped with a submerged centrifugal pump.

PROCEDURE

	Lbs. Used	Mol. Wt.	Mols. Mols.	Mols./ Mol.	Parts/ Part
4-Aminotoluene-3-sulfonic acid	2500	187.2	13.35	1.0	1.0
Darco S-51	150	-	-	-	-
Super Cel	125	-	-	-	-
Sulfuric acid	663	98.0	6.77	0.507	0.265
Soda caustic liquid 30% (as 100%)	535	40.0	13.38	1.00	0.214

Product					
"Lithosol" Acid RBA	2325	187.2	12.42	0.93	0.93

Charge into kettle K-2, 72-in. of filtered water. Start agitation, shovel in 2500 lbs. of 4-aminotoluene-3-sulfonic acid crude. Add 150 lbs. of Darco, 125 lbs. of Super Cel. Then add enough 30% soda caustic liquid (approximately 535 lbs.-100%) to obtain complete solution and a slight pink coloration on Brilliant Yellow test papers. Heat to 95°C. and hold 1 hr. Filter through press F-1 using pump P-14. Return the filtrate to kettle K-2 until it is clear, then switch filtrate flow to tubs T-1 and T-2. After filtration is complete wash press with hot water for 10-min. Return washings to kettle K-2. Blow press dry with air, clean and discard press cake.

Acidify the filtered solution in tubs T-1 and T-2 while hot (95°C.) with 93% sulfuric acid. To obtain a cake that filters well it is necessary to precipitate hot and not too fast. Add acid until charge is distinctly acid to Congo Red test papers. When this point is reached cool a sample, filter, and add a few drops of acid to the filtrate. If no further precipitate appears, acidification is complete. If a precipitate appears, add a few pounds of acid to the charge and repeat the test.

Cool the slurries in tub T-1 and T-2 to 32°C. or below and drop to nutsches N-1 and N-2. Suck as dry as possible and then wash each nutsch with three displacement washes of water to remove the greater part of the sodium sulfate. Suck the nutsch cakes as dry as possible and shovel the product into wooden barrels. Sample, tag and store for drying. Dry in air at 100° $\pm$ 5°C. Grind on a limited mill and pack as "Lithosol" Acid RBA.

HAZARDS

Sulfuric acid and soda caustic liquid are both corrosive liquids. Wear goggles and rubber gloves when handling them.

4-Aminotoluene-3-sulfonic acid is an aromatic amino compound which may be hazardous. Avoid contact with this material.

WASTE DISPOSAL

The sludge from the clarification press is sent to the dump. The final filtrate, a dilute acid solution containing sodium sulfate, is run to the plant ditches.

"Lithosol" Acid RBA

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SPECIFICATIONS

	<u>Orchem Spec. No.</u>	<u>Orchem Code No.</u>
Sulfuric acid	35-S-2	945-000
Darco S-51	None	190-000
Soda caustic liquid 30%	53-S-2	877-500
Super Cel	None	946-200
4-Aminotoluene-3-sulfonic acid crude		049-335
4-Aminotoluene-3-sulfonic acid purif. dry	None	049-340
4-Aminotoluene-3-sulfonic acid purif. wet	None	049-345
"Lithosol" Acid RBA	Standard for dyetest is "Lithosol" Acid RBA Lot 122	448-300

USES

4-Aminotoluene-3-sulfonic acid purified wet is dried to produce 4-aminotoluene-3-sulfonic acid dry. The latter is ground and sold as "Lithosol" Acid RBA.