

# Studies Of Pesticidal Properties Of Tosyl- P – Amino Phenol And – P –Nitro Phenol

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## Abstract

The tosylated compounds of p-nitrophenol and p-aminophenol were characterized by NMR and IR spectroscopy and their physical properties determined. Both compounds are highly toxic with  $LC_{50}$  less than  $50.0 \mu\text{g/ml}$ . their insecticidal and herbicidal activities of the compounds are promising.

## INTRODUCTION

In the evolution of agriculture towards intensive monoculture, there has been a trend away from the traditional method of pest control such as crop rotation, stalk destruction, elimination of pest habitat and use of tolerant crop variety, to almost complete dependence on pesticides (1). In many part of the world, especially the third world countries, pest has become a major agricultural problem that threatens crops and farm animals. Although various techniques were employed on the combat against pest (2) The chemical method will continue to be the major method of pest and diseases control in agriculture world wide, in spite of its many disadvantages especially environmental pollution.

Most pesticides are broad band toxicants; any selectivity they may have is derived from the manner in which they are used than for inherent properties. Although it is conceivable that those highly selective poisons could be developed for pest and a few examples are known, e.g Antu for rat [2]. They can however be grouped into two main classes, the contact or non-systematic pesticides and systematic pesticides.

Agricultural products must be produced in such quantities, that will be enough for the food need of our ever increasing population, as well as, industrial raw material (3). For this reason there is the need for producing a chemical which is more effective on pest and less harmful to man, for preventing this undesirable elements, either by retarding their growth or eliminating them completely (3).

Many derivatives of toluene suphoxy compound have been synthesized by many workers in pyridine (4,7). Since most known pesticides contains such groups as  $\text{NO}_2$ , and  $\text{SO}_3$ . This necessitated the synthesis of these compounds, and their activity against shrimps, bean weevil, rat, maize and bean seeds as reported in this work.

## MATERIALS METHODS

The chemicals used are products of Andrich Chemical Company. The IR was run with peerkin-elmer 577 instrument, Nmr by nmr 317B. While the UV was run on sp 750 UV – Visible spectrometer. The  $\text{SO}_3$  were determined titrimetrically according to standard procedure (8).

## SYNTHESIS OF TOLUENE-P-SULPHONOXY-P-NITROPHENOL (A)

40 ml of pyridine was added to p-nitrophenol in a 500ml conical flask. The mixture was cooled to  $0^\circ\text{C}$  in an bath continuous swirling, and 40.60g of toluene-p-surphonic acid was added gently. The mixture was allowed to stand for six hours, with continuous swirling in the ice bath. 100ml of diethyl ether was added as solvent and the part which contained the product, was separated from the aqueous layer using a separating funnel. 50ml of dilute hydrochloric acid was added to the organic layer in the separating funnel to remove the pyridine. This was repeated thrice until the odour of pyridine was no longer observed.

Anhyfrous sodium sulphate was added as a drying agent to remove water molecules and the sulphate salt was filtered off and concentrated under vacuoo, leaving a brown solid 12.195% (mpt  $90^\circ\text{C}$ )  $\nu_{\text{max}}$  1580 ( $\text{C}=\text{O}$  aryl), 1360 ( $-\text{SO}_3-\text{C}$ ),

8500 (ph-NO<sub>2</sub>), H<sup>1</sup> nmr (CDCl<sub>3</sub> + Tms)  $\delta$ 7.6 (4H, S aryl) 2.2 (CH<sub>3</sub>),  $\lambda_{\text{max}}$  458nm, SO<sub>3</sub> 27.38% (27.30%).

### TOLUENE-P-SULPHONOXY-3-AMINOPHENOL (B)

40ml of pyridine was added to 40g of 4-Aminophenol in a 500ml conical flask. The mixture was cooled to 0 °C in an ice bath with continuous swirling, and 62.82g of toluene-p-sulphonic acid was added gently and treated as in the above procedure, resulting in a brown solid, 18.72% (m.p 82 °C) v max (Nujol), 1570 (C=C, aryl), 3150 (Ph-NH), 1365 (SO<sub>3</sub>-C) <sup>1</sup> Hnmr (CDCl<sub>3</sub> + TMS)  $\delta$ : 7.4 (4H, S, aromatic), 2.2 (CH<sub>3</sub>), 6.2 (NH – aryl)  $\lambda_{\text{max}}$  445nm, SO<sub>3</sub>, 30.82% (30.42%).

### LETHAL DOSE

20 brine shrimp eggs were added to the hatching chamber of a hatching vial and kept under fluorescent light for 24 hours to mature and were harvested. The two compounds were dissolved in different concentration in distilled water. The solutions were transferred into different vial. (3). Each dosage were tested in triplicate and their Lc50 recorded in table 1.

**Figure 1**

Table 1: Brine Shrimps Test

Sample	LC <sub>50</sub>
A	50.10µg/ml
B	50.10 µg/ml

### SOLUBILITY TEST

The compound synthesized were tested for their solubility in four different solvents water, ethanol, chloroform and ether are all soluble.

### INSECTICIDAL TEST

Bean weevil bioassay 20 unperforated bean selected from chemically untreated seed were obtained from a farmer in Gwagwalada FCT, are placed in three conical flasks. Five bean weevils were introduced into each of the flask and 0.1g of each compounds were added separately to two of the flask with the third as control. The flask were plugged with cotton wool and left on the bench at room temperature (3,7). They were monitored for two weeks and the results are presented in table 2.

**Figure 2**

Table 2: Bean weevil test with dry samples

Sample	Mortality rate					%cumulative mortality			
Days	1	2	3	4	5	1	2	3	4
A	2	2	1	-	-	40	80	100	-
B	2	1	2	-	-	40	60	100	-
Control	-	-	-	-	-	-	-	-	-

0.1g each of the two samples were dissolved separately in 10ml diethylether and sprayed to 20 beans infected with 5 weevil in each of the two conical flask with third flask been sprayed with diethyl ether without the compounds, which serve as the control (7). The results after one week is given in table 3.

**Figure 3**

Table 3: Bean weevil test with samples in diethyl ether

Sample	Mortality rate					%cumulative mortality			
Days	1	2	3	4	5	1	2	3	4
A	4	1	-	-	-	80	100	-	-
B	3	2	-	-	-	60	100	-	-
Control	-	-	-	-	-	-	-	-	-

### RODENTICIDAL TEST

1.0g of each sample were infected with bread feed with two rats and the third was feed with bread free of the compounds (3) and were observed for one week.

### HERBICIDAL TEST

Mono and dicot assay. Beans and maize seedlings were treated with 1.0g each of the sample in 10ml diluted water. The result is shown in table 4.

**Figure 4**

Table 4: Herbicidal Test

Sample	Mortality rate	%cumulative mortality
A	- - -	+ + +
B	- - -	+ + +

+ + + Highly effective, - - - Not effective

### RESULTS AND DISCUSSION

The reactions between toluene sulphonic acid with p-nitrophenol and 3-apinophenol in pyridine resulted in products with good yields.

The two compounds have low melting points of 90 °C and 82 °C for A and B respectively and are both soluble in aqueous and polar organic solvents.

These compounds are toxic as revealed from their  $LC_{50}$  which is less than 50 (table 1). The rodential tests are partially positive weakening the effected rodent and causing it to be less active but not active but not causing any fatality, thus implying that they are not liable to be toxic to man. The compounds show promising results as contact and systematic in action, in relation to their insecticidal properties (table 2 and 3). The herbal test indicates that the compound B is effective against monocot while compound A is positive against dicot plants (table 4).

The spectroscopy studies are in agreement with the expected features ( $\delta$ ) and are akin to that of a similar reported compound ( $\gamma$ ).

## CONCLUSION

The synthesis of toluene sulphonyl-3-amino and toluenesulphonyl-p-nitrophenol was successful and their pesticidal properties are highly encouraging.

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