

## A qualitative espial of distinct generation herbicide Sulfosulfuron adopting digital Synthetic tests

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### **Abstract**

*A novel strategic and cost-effective method for the detection of sulfosulfuron which is a distinct generation herbicide, commonly used as weed killers in different crop cultivations in the fields. Although the toxicity studies among mammals have shown a less toxic profile for the same compound, the potential toxicity of the sulfosulfuron on human beings cannot be estimated. As per survey, no study has been reported yet which showing the micro chemical tests which are economical and cost effective, when compared to the existing advanced costly instrumental techniques like HPLC, routinely used in the laboratories for the detection of Sulfosulfuron. Hence, in the present study, Combinations of different chemicals and reagents will be used for the detection of sulfosulfuron, through multiple trials in this study. An attempt would be done to analyze Sulfosuluron using micro chemical color tests for the qualitative detection. Presently, as such there is no relevant literature available to help the forensic scientists and analytical toxicologists to detect the same compound using micro chemical tests; it is believed that the results of the study could be useful for the scientific community including forensic scientists, clinical toxicologists, environmental toxicologists, food toxicologists and researchers.*

**Keywords:** Toxicity; human; detection; analysis; tests

### **Introduction**

An herbicide is a chemical compound used to kill unwanted plants. Herbicides have been alleged to cause a variety of health effects ranging from skin rashes to death. The pathway of attack can arise from improper application resulting in direct contact with field workers, inhalation of aerial sprays, food consumption and from contact with residual soil contamination. Selective herbicides kill certain targets while leaving the desired crop relatively unharmed. Some of these act by interfering with the growth of the weed and are often based on plant hormones. Herbicides used to clear waste ground are nonselective and kill all plant material with which they come into contact. Some plants produce natural herbicides, such as the genus *Juglans* (walnuts). Herbicides are widely used in agriculture and in landscape turf management. They are applied in total vegetation control (TVC) programs for maintenance of highways and railroads. Smaller quantities are used in forestry, pasture systems, and management of areas set aside as wildlife habitat. Herbicides can also be transported via surface runoff to contaminate distant surface waters and hence another pathway of ingestion through extraction of those surface waters for drinking. Some herbicides decompose rapidly in soils and other types have more persistent characteristics with longer environmental half-lives.



Sulfosulfuron is a member of the sulfonylurea family of herbicides used widely throughout the world for control of broadleaf and grass weeds in a range of crops including cereals and corn. The fundamental mode of action for sulfosulfuron and indeed all sulfonylurea herbicides entails inhibition of acetolactate synthase (ALS) an essential enzyme in aliphatic amino acid synthesis.

Sulfonylureas are a family of environmentally compatible herbicides that were discovered by DuPont Crop Protection in 1975 and first commercialized for wheat and barley crops in 1982. They have now been developed and commercialized worldwide in all major agronomic crops and for specialty uses (eg. Rangeland pasture, forestry, vegetation management). Sulfonylureas represent a major advance in global crop protection technology and have revolutionized weed control by introducing a unique mode of action. Specifically, these compounds interfere with a key enzyme required for weed cell growth-acetolactate synthase. Furthermore, sulfonylureas are compatible with the global trend towards post emergence weed control and integrated pest management.

The invention of sulfonylurea herbicides, in June 1975, by George Levin of DuPont revolutionized the use of herbicides and facilitated a change in direction in DuPont's corporate strategy from chemicals to life sciences. The company had been in agrichemicals since 1928, but the business had remained a small part of its portfolio. In the early 1970s, agrichemicals provided DuPont a strong rate of return, but its potential had yet to be fully exploited. Levitt's pioneering invention, made in this small business, came from his insight and creativity, and his supervisor's encouragement to explore. But DuPont would have profited only modestly from sulfonylurea herbicides were it not for a new research manager willing to take a risk and ramp up an innovation program that was being curtailed. Soon, the program grew further as

DuPont's corporate strategy changed and sulfonylureas presented an immediate opportunity for the company to expand its presence in life sciences. The growing agrichemicals business also adopted a new research strategy to improve productivity. DuPont thus built a sustained program of innovation to realize a pioneering invention's scientific and commercial promise.

There was no precedence for the high potency- and thus low application rates – of sulfonylurea herbicides when they went into field tests in the mid-1970s. A field agent thought the manager prescribing the low rates was “nuts”. Others conducting early field tests at a university concluded that there had to be an error in the instructions. Herbicides were normally applied at rates of a few kilograms/hectare, not the few grams/hectare suggested. That was unheard of. So, they moved the decimal point by two places, thereby increasing the application rate 100-fold. Two years later, weeds would still not grow in the test plot, despite the herbicide's having a half-life of about 6 to 8 weeks.

Sulfonylurea herbicides kill weeds by inhibiting the enzyme acetolactate synthase, which is essential to their growth. They work on a broad range of grasses and broadleaf weeds, but not on crops they are designed to protect. Crops like rice, wheat, barley, soybean, maize, and many others are able to metabolize sulfonylurease safely. And the herbicides are safe for humans and animals because they do not have the enzyme sulfonylurea molecules target. Owing to their low application rates, it is estimated that farmers now use around 1 to 5% of the quantity of herbicides used before sulfonylurease, and overall herbicide usage is estimated to be less by about 200 million pounds per year. Sulfonylureas replaced herbicides that were less safe and effective, and were used in larger quantities. Combined with the low application rates, selectivity for a variety of crops, and short half-life, sulfonylurea herbicides were a revolutionary advance in agrichemicals and an important

cornerstone of DuPont's change in corporate strategy in the 1980s. Sulfosulfuron is selective Post Emergence Herbicide and is used to controls Phalaris minor and broad leaf weeds, which are resistant to other generic herbicides. Sulfosulfuran is a selective, systemic sulfonyl urea herbicide, absorbed through both roots and leaves. It translocates throughout the plant and acts as an inhibitor of amino acid biosynthesis, hence stopping cell division and plant growth. It is effective against grasses and broad-leaved weeds in wheat. Barley and oats are sensitive.

Sulfonylurea generally enhances the level of insulin in blood and glucose can be utilized. However, if one takes incorrect dosage of it he may have abdominal disorders, headache, nausea, and other hypersensitivity reactions.

One has to take care and should not consume sulfonylurea during pregnancy or during expected pregnancy. Sulfonylurea may be fatal sometimes because it may increase the risk of cardio vascular deaths. Sulfonylurea herbicides are some of the environmentally friendly medicines. Overdose of this drug is very harmful as it can induce hypoglycemia. Therefore, emergency therapy by sulfonylurea overdoses is never recommended by the doctors. It is extremely harmful because glucose stimulates the secretion of insulin and it also initiates an urge for enhanced quantities for hypertonic glucose so that normal glycemia could be maintained in the system. However, there is one drug called octreotide that could reverse the adverse effect of hyperinsulinemia which is caused by normal glycemia. Sulfonylurea is one of the most commonly used anti diabetic drug used for treating diabetes mellitus type 2. However, an overdose of this anti diabetic medicine is very harmful. Primarily, the side effect reported by the overdose of sulfonylurea is hyperinsulinemic state and a subsequent hypoglycemia. Sulfonylurea binds to sulfonyl urea receptors which are present on the membrane of beta cells of islet of langerhans. Serious concerns

have been raised about the inhibition of ATP sensitive potassium channel by sulfonylurea which may even result in cardiovascular deaths.

### **Materials**

Sulfosulfuron (75%) of analytical grade was purchased by VIKRANT chemicals ltd, Haryana and were of analytical grade for experiment.

### **Sampling**

The present study involves 7 different combinations of reagents which were considered for the detection of the known sulfosulfuron samples. 7 individual trials of each reagent were considered for tests with extracted sulfosulfuron. The reagent combination, 1ml of 10% aqueous sodium hydroxide, 1ml of 10% sodium nitroprusside, 3ml of 10% potassium ferrocyanate in test sample was giving a result that was found to be replicative when it was performed for 5 times. Hence experimentation was planned for conducting multiple trials with the same reagent combinations and extract to check the reproducibility for validating the test. In total 50 sample extracts of sulfosulfuron were considered for the trials with 75% sulfosulfuron.

### **Methods for the sample preparation**

1. Sulfosulfuron (75%) pure sample was taken and dissolved in naphthalene and distilled water and used for testing with different combinations of chemicals and reagents in the pilot study.
2. The criteria for the selection of reagents were done based on their proven chemical reactions of the chosen chemicals with sulfonyl functional group and urea.

**The discrete reagent combinations used during the pilot testing are given below:-**

#### **REAGENT 1:-**

- a) 5 ml of 5gm ammonium molybdate in 35ml conc. Nitric acid in 65ml of distilled water.

- b) 5ml of 5gms perchloric acid in 50ml of distilled water.
- c) 5ml of 0.5gms stannous chloride in 100ml conc. HCL.

**REAGENT 2:-**

- a) 2ml of 5% sodium nitroprusside.
- b) 1ml of 10% aqueous sodium hydroxide.
- c) 5ml of 3% aqueous hydrogen peroxide.

**REAGENT 3:-**

- a) 1ml of 10% aqueous sodium hydroxide.
- b) 1ml of 10% sodium nitroprusside.
- c) 3ml of 10% potassium ferrocyanate.

**REAGENT 4:-**

- a) 2ml of 2% sodium hydroxide
- b) 2ml of pyridine

**RESULTS**

**REAGENT 5:-**

- a) A drop of 200mg rhodamine in 50ml ethanol.

**REAGENT 6:-**

- a) 1 drop of 1gm ninhydrin in 10ml glacial acetic acid and 150ml ethanol.

**REAGENT 7:-**

- a) Sodium nitrate-0.2gms in 100ml distilled water.
- b) Concentrated HCl.
- c) 20mg EDTA in 20ml distilled water with 2-3 drops of HCl.

Few drops of the test sample with few drops each of the reagents were tested in the test tube during the pilot testing.

S.NO	REAGENT NAME	RESULTS
1.	a) 5ml of 5gm ammonium molybdate in 35ml conc. Nitric acid in 65ml of distilled water. b) 5ml of 5gms perchloric acid in 50ml of distilled water. c) 5ml of 0.5gms stannous chloride in 100ml conc. HCL in test sample.	<b>White deposit at the bottom of the test tube.</b>
2.	a) 2ml of 5% sodium nitroprusside. b) 1ml of 10% aqueous sodium hydroxide. c) 5ml of 3% aqueous hydrogen peroxide in test sample.	<b>Color changed from red to brown and a yellow effervescence was observed.</b>
3.	1ml of 10% aqueous sodium hydroxide 1ml of 10% sodium nitroprusside 3ml of 10% potassium ferrocyanate in test sample	<b>Dark red color which on setting for 5 minutes turned ORANGE.</b>
4.	2ml of 2% sodium hydroxide. 2ml of pyridine. 2ml of test sample.	<b>White deposit at the base of spot tile.</b>
5.	A drop of 200mg rhodamine in 50ml ethanol in 2-3 drops of test sample.	<b>No significant color change.</b>
6.	1 drop of 1gm ninhydrin in 10ml glacial acetic acid and 150ml ethanol.	<b>No significant color change.</b>

7.	Reagent1:-sodium nitrite-0.2gms in 100ml distilled water. Reagent 2:- Concentrated HCl. Reagent 3:- 20mg EDTA in 20ml distilled water with 2-3 drops of Hcl. To 1ml of test sample add 1 drop of reagent 1,2 drops of reagent 2 and 3 drops of reagent 3.	<b>No significant color change.</b>
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Table No.1 Showing results of tests using different chemical combinations with Sulfosulfuron extract.

As part of the pilot testing 7 different reagent combinations were used for reacting with the Sulfosulfuron extract. Out of the seven individual trials, 1ml of 10% aqueous sodium hydroxide, 1ml of 10% sodium nitroprusside, 3ml of 10% potassium ferrocyanate in test sample (reagent 3) showed a result through a color change transition from dark red to a permanent orange color which did not change on standing.

The trials with rest of the reagents did not show any significant color change with the test sample SULFOSULFURON.

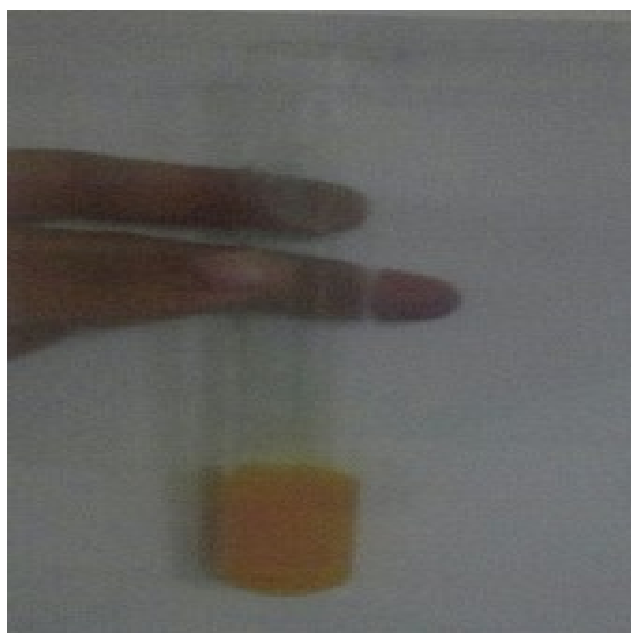


Image1. Showing colour reactions after reacting with reagent no:3 with sulfosulfuron extract in the pilot test.

After the performance of test with different chemical reagent combinations, it was found that 1ml of 10% aqueous sodium hydroxide, 1ml of 10% sodium nitroprusside, 1ml of 10% potassium ferrocyanate in test sample. (reagent 3) was giving a permanent color change, 50 trials of the same test were repeated to check for the replicability and for validating the obtained results. The results of 50 trials are shown below in the table.

COLORS OBSERVED	TOTAL NUMBER
YELLOW	22
ORANGE	15
GREENISH YELLOW	13

Table 3. Showing positive and negative results after 50 trials with 1ml 10% aqueous sodium hydroxide, 1ml 10% sodium nitroprusside, 1ml of 10% potassium ferrocyanate

Out of 50 trials done with reagent; 13 times the greenish yellow color while 15 time orange and 22 times 22 were successfully obtained. The trials with rest of the reagents did not show any significant color change with the test sample SULFOSULFURON.

The statistical analysis of the results was done using Chi square analysis. The Chi square value obtained was 39.69 and the standard probability value was determined to be;  $p < 0.995$

### STATISTICAL ANALYSIS USING CHI SQUARE

OBSERVERED VALUE ( $F_o$ )	EXPECTED VALUES ( $F_e$ )
15	50

$$\chi^2 = P \text{ value} < 0.995$$

### DISCUSSION & CONCLUSION

It was found that the results were statistically significant with the standard probability value obtained being,  $p < 0.995$  as the test results showed a high probability of replication of the results. It is giving a scope for further exploration in the similar lines to get better results for those who are concerned with the analysis of Sulfosulfuron. For the qualitative detection of sulfosulfuron for toxicological analysis, different analytical instruments and methods have been in use like HPLC etc. But as per date, no standardized method is detailed in literature pertaining to screening & detection of sulfosulfuron using simple micro chemical tests. In this present study, after trying with different chemical combinations, results were obtained with one of the chemical combination (1ml of 10% aqueous sodium hydroxide, 1ml of 10% sodium nitroprusside, 3ml of 10% potassium ferrocyanate in test sample) with a persistent orange color. This result was replicative in its nature when the same test was repeated 50 times. The positive result was found to be a promising color test for the

screening of the compound sulfosulfuron in forensic cases. This method would be beneficial for forensic science laboratories which may not be having state of the art advanced instrumentations like HPLC, GC, and HPTLC. Moreover, this test being economical, cost effective and involving common cheap chemicals available in the laboratories, it is advantageous over other techniques for the rapid screening of Sulfosulfuron. Since, this technique is a simple method, easy to perform, cost-effective, and also takes less time to analyse the sample, the results of the present study is giving a scope for the Forensic Toxicologists, Chemists, Scientists involved in Pesticide Analysis in Forensic Laboratory settings, Environmental Laboratory settings, Food Quality check Laboratories to use the presently developed color test useful for their routine casework. Apart from this, Sulfosulfuron being a potential chemical as a poison the cases involving accidents, murders, suicides, cannot be underestimated in forensic context.

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