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Evaluation of Fentrazamide for Weed Control and Estimation of its Residues in Rice

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The active ingredient fentrazamide [4-(2-chloro-phenyl)-5-dihydro-tetrezole—1-carboxylic acid cyclohexyl ethyl amide (Figure 1,I), belongs to a new chemical class tetrazolinones. It is a cell division inhibitior (Minegishi *et al.*, 1999) and has efficacy against grasses, sedges and broadleaved weeds (Yasui et al, 1997). It was synthesized at Nihon Bayer Agrochem Yaki Research Center, Japan. In Japan and Korea, this compound is applied at transplanting time of rice due to its long residual activity.

In India, rice is grown in about 42.6 m ha representing 27.7% of world rice acreage and 13% of world's production. The gap between potential highest yield and national average yield is attributed to several production constraints of which weeds are one among the major ones limiting production and productivity of rice. As most of weed floras are of C4 type, they compete with rice for nutrients, space light and water. The controlled weed growth in rice grown during *Kharif* (rainy) season causes yield losses up to 64%. Some grass weed like *Echinoclova colonum* or *E.crus galli* resembles rice plant during initial stages and escapes manual weeding. Moreover manual weeding is costly and laborious. Due to this, herbicide usage is gaining momentum in India. So far butachlor and anilophos are being used for weed control in rice. Past experience has shown that most of the developing countries are facing the problem of herbicide resistance and shifting of weed flora due to continuous use of one herbicide.

When using herbicides in production of rice, the paddy soil is major sink for the applied active ingredient, except for the portion remaining on or in the crop or being washed off the rice field. Therefore, evaluation of new herbicides to decrease the selection pressure against resistant biotypes/tolerant weed species is required. Before introduction in India, the herbicide should be evaluated from residues point of view also to ensure that its residues do not accumulate in edible portion of this crop. The presence of excessive residues above the tolerance level poses health hazard to the consumers. The host country rejects the export consignment if the residues are above the prescribed limit. These experiments were conducted to evaluate this newly introduced herbicide for studying its effectiveness for weed control and to estimate its residues in rice after field trial in India.

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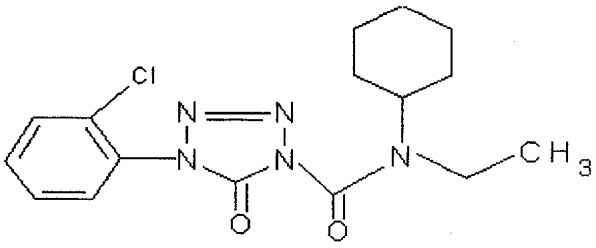

Figure 1. Fentrazamide (I)

MATERIALS AND METHODS

The study was conducted at IARI, New Delhi in 1999 and then at Resource Management Block, DWR, Karnal, during *Kharif* season of 2000-2001, in randomized block to see the impact of different dosage of YRC 2388 on the yield of rice (Malik, 2002). The soil of the experimental field was clay loam with pH 7.8 and organic matter content 0.4%.

Rice variety Pusa-44 was transplanted in the first week of June during all three years. The herbicide treatment comprised YRC 2388 (50 WP) at different dosages e.g. 90, 120, 150, 240 and 420 g a.i./ha 4 (four) days after transplantation (DAS). It was applied at highest dose of 420 g a.i./ha for phyto-compatibility studies in 2000 only (Chauhan and Chhonkar, 2000). The formulation of the herbicide was weighed and dissolved in 500 ml water and then mixed with 50 kg sand before application by hand in three replications.

Weedy and weed free checks were also maintained. Weed free plots were maintained by hand weeding as and when weed appeared. Fertilizer application and irrigation were done according to recommended package of practice for paddy in Northern India. Observations on weed population per square meter were taken at 60 DAS by placing a quadrant of 50 x 50 cm randomly at three places in each plot. The biomass of the weeds was recorded at maturing, when the crop was harvested in second week of October every year i.e. 125 to 130 day after transplanting. After harvesting paddy straw, pod cover and seed/grain were separated.

25 mg analytical sample of fentrazamide was dissolved in 25 ml HPLC grade methanol to obtain 1000-ppm stock solution of fentrazamide. Working solutions upto 1- μg/mL levels were made by its serial dilution. Recovery experiments were carried out to determine the efficiency of the method. Soil (25g), grain (10g) and paddy husk (10g) were fortified at 0.5 and 1 mg level. The limit of detection was 0.001 mg/kg, and the limit of determination was 0.01 mg/kg. Representative samples of soil (50 g), hulled rice grains (10 g) and rice husks (10 g) were extracted in a Soxhlet extractor with 1:1 hexane-acetone mixture (350 ml) for 6 hrs. The extract from the soil and husk samples were filtered and concentrated to 10 mL. The concentrate was then subjected to column clean up.

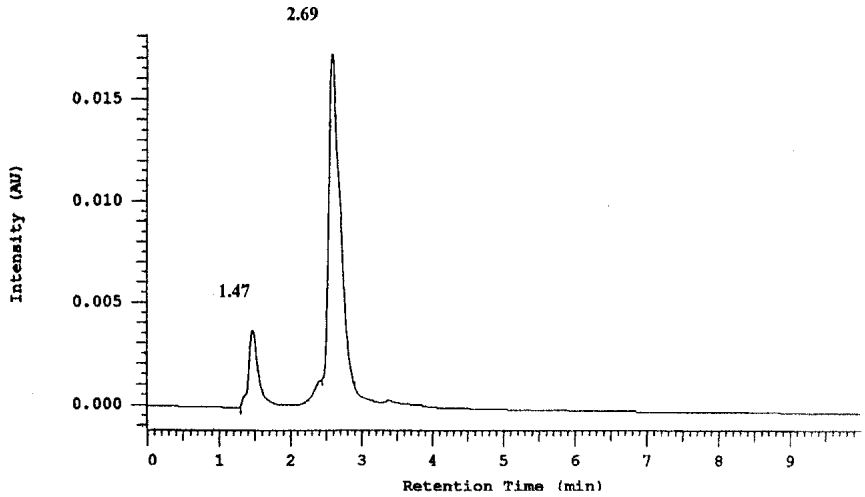

Figure 2. HPLC profile of analytical standard of fentrazamide

The clean up carried out using glass column (1.5 cm i.d x 30 cm) packed with sodium sulfate (2 g) + neutral alumina (2 g) + Florisil (1g)+ sodium sulfate (2g). The column was pre-washed with hexane 30 ml. The extract obtained from the above process was adsorbed on the column and the column was eluted with hexane-acetone (1:1, 100 ml). The eluted solvent was evaporated completely in order to remove any trace of the solvent and the residue dissolved in HPLC grade methanol.

The extract of rice grains was concentrated to 30 ml under vacuum. The extract was transferred to a separately funnel and saline water (2%, 150 ml) was added to it. The contents were subjected to liquid-liquid partitioning with acetonitrile saturated with hexane (50 ml) and was separated out from the hexane layer. The hexane layer was discarded and the acetonitrile phase was diluted with saline water (2 %, 300mL) and further subjected to partitioning with dichloromethane (3 x 50 ml). The dichloromethane was evaporated to dryness under rotary vacuum evaporator and finally made up in HPLC grade methanol before analysis by HPLC-UV.

The HPLC was carried out on Merck LC 7000, fitted with a pump L-7100 an auto sampler, L 7200 and UV detector, L 7400. The wavelength was set at λ_{max} 240 nm, and the mobile phase was methanol at a flow rate of 1 mL/min. The retention time of the fentrazamide was 2.69 min (Figure 2).

RESULTS AND DISCUSSIONS

The experimental field was dominated by *Echinocloa crusgalli* and *Cyprus iria* during all the three years (1999-2001). The treatments of YRC at various dosages decreased the dry weight of weeds as compared to weed check plots during all the three years of the experiment (Table 1). When the dose of the herbicide was

Table 1. Effectiveness of YRC 2388 against weeds in transplanted rice for 3 years

Treatments	Dose (gai/ha)	Dry wt/g/m^2			Grain Yield		
		1999	2000	2001	1999	2000	2001
YRC 50 WP	90	60	33	40.8	75.17	74.4	59.45
	105	-	26.3	28.6		76.8	63.56
	120	42	20.7	10.8	76.89	77.9	73.43
	150	55	-	4.5	74.70	-	75.22
	220	-	20.7	-	-	77.5	-
	240	-	22.7	1.7	-	73.1	73.67
Weed check		199	181	219.9	62.24	67.6	-
Weed free	-	-	-	-	78.6	77.5	76.11

increased from 90 to 240 g a.i./ha, the dry weight of weed was reduced significantly.

The yield and effective fillers of rice followed the same trend as in case of dry weight of weeds. The data for 1999 trial shows that the dry weight of weeds in weeded check was 199 g/m^2 and paddy yield was 62.24 Q/ha. Thus, the yield was poor in the control plots than in plots treated with YRC 2388.

The maximum grain yield was recorded under weed free check (78.65 Q/ha), while all the weed control treatments provided significantly higher grain yield as compared to the treated. While recommended rate of application is 120 g a.i./ha (x), the application of 4x (480 g a.i./ha) was done for phytoxicity studies in 2000. Visual phytotoxicity was 6.6 (less than 10); hence the compound is not phytotoxic to rice. However, the grain yield was less than maximum when the herbicide was applied over 150 g a.i./ha or less than 90 g a.i./ha.

The HPLC method used after column cleanup with neutral alumina–Florisil was effective for estimation of the residues of herbicide, fentrazamide from rice, grains, paddy straw, paddy husk and soil (Figure 3). The recovery of the method was over 80% hence no correction factor was applied.

Fentrazamide, when applied at double the recommended rate i.e. @ 240 g a.i./ha, 4 days after transplanting, its residues in rice grains at harvest time was 0.04 mg/kg in 2001, 0.03 mg/kg in 2000 and 0.03 mg/kg in 1999 (Table 2). The residues were below detectable limit, <0.01mg/kg in rice husk and rice straw even the herbicide was applied at higher the rate application at 240 and 420 g a.i./ha.

The average half-life of fentrazamide in soil is reported less than 20 days, and 2-chlorophenyltetrazoline and cyclohexyl ethylamine have been identified as the major and minor metabolites in soil (Hellpointer, 2001). In the present study, the metabolites formed, may be present in trace amounts and could not detected by HPLC.

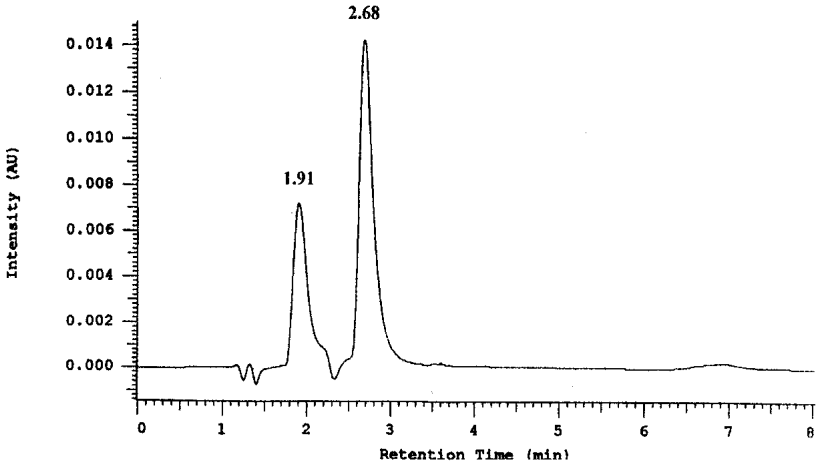

Figure 3. HPLC profile of treated grain sample

Table 2. Residues of YRC in different parts of paddy

Treatment - (Year 2001)			Average Residues (mg/kg)	
g a.i./ha	Rice	Husk	Straw	Soil
120	BDL	BDL	BDL	BDL
210	BDL	BDL	BDL	BDL
240	0.04	BDL	BDL	0.02
Treatment - (Year 2000)			Average Residues (mg/kg)	
g a.i./ha	Rice	Husk	Straw	Soil
120	BDL	BDL	BDL	BDL
240	0.03	BDL	BDL	0.01
420	0.05	BDL	BDL	0.03
Treatment - (Year 1999)			Average Residues (mg/kg)	
g a.i./ha	Rice	Husk	Straw	Soil
120	BDL	BDL	BDL	BDL
240	0.03	BDL	BDL	0.02

* Average of three replicates, BDL = below limit of detection (<0.01mg/kg)

The results of the study by Zepp and Cline 1977 indicate that the herbicide, fentrazamide does not persist long enough to cause environmental hazard to the aquatic animals as it undergoes photolysis in the aqueous medium to innocuous products.

Based on the persistence data of fentrazamide on paddy, it can be concluded that the herbicide can be recommended for use @ 105 – 150 g a.i./ha four day after transplantation In rice grains the residues of fentrazamide were below the detectable limit (<0.01 mg/kg) when the herbicide was applied @ 120 and 210

g a.i./ha and therefore safe for consumption and poses no threat to human health.

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