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RESEARCH ARTICLE

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Determination of the residue of quizalofop-p-tefuryl in soybean by HPLC

Abstract A HPLC method was developed for the analysis of quizalofop-P-tefurylof in soybean .The samples were extracted with methanol-water（volume ratio）, The extracts were cleaned up with a column of silica gel. The final residue was detected by HPLC, using a UV detector. The recoveries from the analytical method for soybean were 84.32%-89.25%.Variable coefficients were 0.49%～1.51%.This method proved to be simple, reliable and accurate.

Keywords HPLC, quizalofop-p-tefuryl, residue, soybean

1 Introduction

Quizalofop-P-tefuryl is a new herbicide manufactured by Uniroyal Chemical in the USA, its chemical name is 2-[4-[(6-chloro-2-quinoxalinyl)-oxy]phenoxy]-,(tetrahydro-2-furanyl)methyl eater. It is used to control annual and perennial germanous weeds in soybean, peanut, rape, cotton and flax. It is a selective and post-emergence herbicide, which is absorbed by the leaves and transmitted to the entire plant to restrain the synthesis of normal esters and to retard burgeon and rootstock development.[1–5] At present, there are no reports about the analysis of Quizalofop-P-tefuryl in soybean in China and abroad. We derived our method of analysis from the methods used for the analysis of other propanoic acids [6,7] and developed a HPLC method for the determination of 4% of Quizalofop-P-tefuryl EC in soybean with precision..

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2 Experiment Part

2.1 Instruments and Reagents

LC-6A HPLC, with a UV detector and DP (Shimadzu Company, Japan)

Methanol(CR); chlorofonn, $CaCl_2$, Ethyl acetate, Petroleum ether, Sodium Sulfate anhydrous, Silica gel, active C (AR)

Standard quality Quizalofop-P-tefuryl (99.6% w/w) and 4% Quizalofop-P-tefuryl were obtained from Uniroyal Chemical in the USA.

2.2 The Condition of HPLC

Zorbax XDB-C_{18} Column (250mm×4.6mm i.d. 5 μm, Agilent)

Mobile phase: methanol-water (80:20 v/v) Flow rate: 0.7ml/min

Detector: UV at 240nm Injection: 5 μL

Oven temperature: 35℃ T_R (at above conditions):10.5min

2.3 The Predisposal of the Sample

(1) Extraction: A 20g sample of soybean was crushed and sieved to pass through a 0.9mm screen ultrasonicated with 50ml of 80:20 (v/v) methanol and water in a 250-ml conical flask for 10 min, and was filtered with a Buchner funnel with filter aid at reduced pressure. The conical flask and residue were rinsed with 20ml of 80:20 (v/v) methanol and water; the effluents were combined and transferred to a seperator funnel. After adding 80ml of 5% $CaCl_2$aq, the effluent was extracted with chlorofonn three times (50ml× 40ml × 40ml). After being passed through anhydrous Sodium Sulfate, the effluent was evaporated by rotary evaporation at 40℃ and at reduced pressure and transferred to a filled-column.

(2) Clean-up: The column was filled in by the

dry-method. 1g filter aid, 2g silica gel, 0.5g active C and a little anhydrous Sodium Sulfate were added in turn into the glass column (20cm×1.5cm i.d.), then the column was rinsed with 20ml of 95:5 (v/v) ethyl acetate and petroleum ether and the effluent was discarded. The concentration was dissolved with 3×1ml of rinsate and transferred into a column and eluted with 70ml of 95:5 (v/v) ethyl acetate and petroleum ether. The initial 5ml of eluate was discarded; the rest was collected and concentrated at 40℃ and less pressure, then dried under a stream of N_2. Samples were then diluted to the 1ml mark with methanol.

3 Result and Discussion

3.1 Extraction method

The method of extraction and conditions in soybean were tested, and surging and

ultrasonicating extraction were compared. The results showed that samples that were surged for 40min were similar to those ultrasonicated for 10min; in order to save time, we chose the second method.

3.2 Wave-length

A scan of the UV-spectrum showed that more is absorbed at 205nm than at 240nm.

But there is more interference at 205nm. So a wave-length of 240nm was chosen as there is less interference and the method is sensitive.

3.3 The rinsate used to column clean-up

When the rinsate is ethyl acetate, there is obvious interference at the peak of the standard. When it is changed to ethyl acetate –petroleum ether (95:5,v/v), there is high recovery and no interference.

3.4 Oven temperature

The influence of oven temperature (25-55℃) on t_R and the peak area were reviewed. The results showed that temperature has little influence, The column is proper under limited temperature, so 35℃ was chosen.

3.5 Linear Relation and Limit of Detect

Approximately 10.0mg of Quizalofop-P-tefuryl standard was accurately weighed and brought

to 100ml volume with a mobile phase to make a stock standard solution at approximately 100mg/ml. Immediate dilutions in the mobile phase at 0.025, 0.05, 0.1, 0.2, 0.5 mg/L were made, these were detected at the above conditions. The linear equation of the peak area (Y) to the relevant concentration (mg/L) is Y=34853x-139.89, r=0.9998, and the minimum of Limit of Detect (when S/N=3) is 0.01mg/kg.

3.6 The Recovery and Precision

0.01, 0.1, 1.0 mg/kg of Quizalofop-P-tefuryl standards were added to the blank soybean samples, and repeated three times, the recoveries by this method were detected at the above condition of predisposal and chromatography. The recovery's standard deviation is 0.58%～2.51% under the above additions, so this method is reliable.

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