

Determination of herbicides atrazine and butachlor in soil by high performance liquid chromatography

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Abstract— A Liquid Chromatographic procedure was described for simultaneous determination atrazine and butachlor in soil with a diode-array-detector (DAD). Unequivocal identification by Rt alone, however, is sometimes difficult. The UV spectra offer a viable alternative to Rt. The distinct differences in Rt and UV of the peaks are noteworthy. UV spectra are extremely helpful and served to accurately identify the various component. Soil samples containing herbicide residues were extracted on a shaker using dichloromethane. The extractions were finally concentrated to 1 ml by a K-D concentrator then the residues were quantitated by peak height. Detection limits for atrazine and butachlor were 0.1 ppb and 0.8 ppb respectively. Fortified at 5.0 ng, 10.0 ng, and 50.0 ng levels, the recoveries of atrazine ($n=8$) were 88.4%, 95.1% and 91.6%. Fortified at 10.0 ng, 50.0 ng and 80.0 ng levels, the recoveries of butachlor ($n=8$) were 89.8%, 91.2% and 87.4%, respectively.

Keywords: RP-HPLC, atrazine, butachlor, herbicides, soil.

Summer corn and winter wheat rotation per year is the major crop system in North China. The S-triazines are among the most widely used pre-emergent herbicides in the world. Atrazine (2-chloro-4-ethylamino-6-isopropylamino-1,3,5-triazine) is a selective herbicide commonly used on many crops, including corn, sorghum and asparagus for the control of annual grasses and broad-leaf weeds. Residues cause problems in soil, subsequent rotational crops may be affected and atrazine residues can contaminate the water-table and the soil. This latter fact may pose problems for the health of animals or even humans. In order to improve the properties of herbicide, there are many methods, such as suspoemulsions of atrazine mixed with butachlor [N-(butoxymethyl)- α -chloro-N-(2,6-diethylphenyl) acetamide]. The experimental results indicated that a single application of atrazine gave poor control of *Digitaria sanguinalis* in summer corn field, and the residue effect was too long. The bioassay results showed that combination was safe on next crop-winter wheats. To measure atrazine and butachlor in environmental samples, a sensitive rapid method is required.

1 Chromatographic conditions

Separation was performed on a 200 mm × 4.6 mm I. D. stainless-steel Hypersil-ODS column. Gradient elution was carried out using 85% methanol for 1 min, then it was increased from 85% to 100% at 5%/min, finally it was maintained at 100% for 10 min. Raw data from each injection, using 210, 230 and 254 nm detection wavelengths are recorded. The max peak absorption is in 230 nm but the interruption is larger than it. So 254 nm was chosen and made sure that sensitivity is high enough. The DAD was used for confirmation of the identity of sample peaks by comparing their retention times and the UV spectra with those standards.

2 Quantitation and linearity

After a preliminary ratio measurement using a five-point calibration. The calibration standard curves cover the anticipated concentration range (0.5-10 ng/μl) in methanol. Peak height of atrazine and butachlor were measured and calibration was obtained from linear regression of the peak height against concentrations.

3 Recovery and accuracy ($n=8$; results are shown in Table 1 and Table 2)

Table 1 Value of measure atrazine

Spike level, ng	Mean, ng	Recovery, %	RSD, %
50.0	45.80	91.6	4.51
10.0	9.51	95.1	2.89
5.0	4.42	88.4	5.43

Table 2 Value of measure butachlor

Spike level, ng	Mean, ng	Recovery, %	RSD, %
80.0	69.92	87.4	6.88
50.0	45.60	91.2	4.18
10.0	8.98	89.8	6.33

4 Results of the analysis of soil samples (Table 3)

Table 3 Results of the analysis of soil samples

Unit, ng/g

Matrix	Atrazine (Rt*4.85)	Butachlor (Rt*9.52)
Surface layer	2.76	3.28

Table 3 (continued)

10 cm depth	6.02	3.59
40 cm depth	0.41	0.87
Wheat pole	4.35	1.53
Black earth soil (from another place)	< 0.1	10.62

* Retention time in minutes

In summary, this HPLC assay shows a good reproducibility, sensitivity and selectivity. It has the advantage of being a relatively convenient, rapid and simple method of determining atrazine and butachlor in the field of trace organic analysis in environmental samples specifically.

References

- Behki R, Eduard T, Warren D, Pierre. G. *Applied and Environmental*, 1993; 6,1955
Chen N, Zhang YK, Lu PZ. *Chinese Journal of Chromatography*, 1993; 11(1); 30
Chen ZX. *Environmental Chemistry (Chinese)*, 1986; 5(6);1
Edward T, Smith W. *J Environ Qual*, 1992; 21;316
Ferris IG, Haigh BM. *J Chromatogr Sci*, 1987; 25;170
Lu XY. *Pesticides (Chinese)*, 1993; 32(2);30
Qiao XW, Ma LP. *Chinese Journal of Chromatography*, 1995; 13(3);170

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