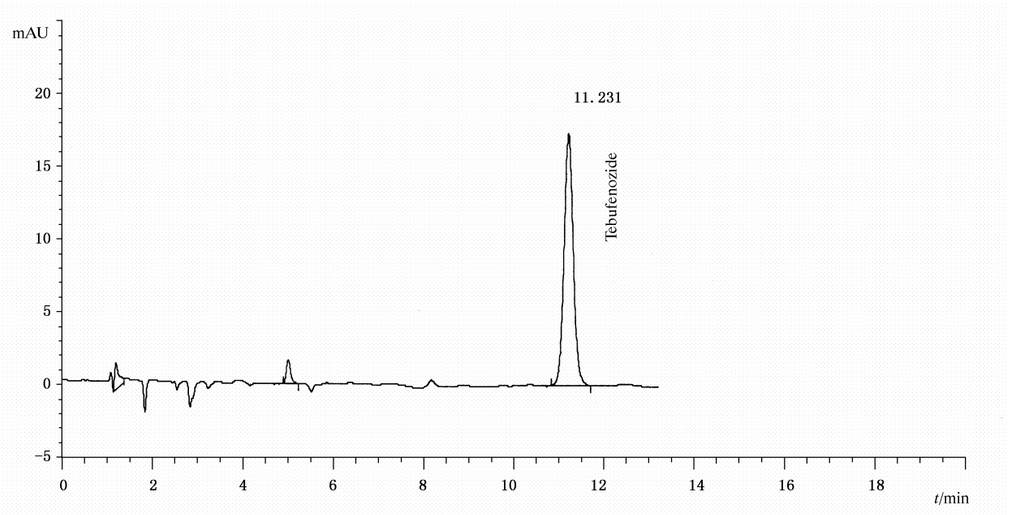
Annex A
(informative)
Chromatogram of the standard



Figwre A.1 Liquid chromatogram of the tebufenozide standard

SN/T 1770—2006

SN/T 1770-2006

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书号:155066·2-17034
定价: 10.00 元

SN

中华人民共和国出入境检验检疫行业标准

SN/T 1770—2006

进出口粮谷中抑虫肼残留量测定方法 液相色谱法

Determination of tebufenozide residues in cereals for import and export—Liquid chromatographic method

2006-04-25 发布 2006-11-15 实施

中华人民共和国
国家质量监督检验检疫总局 发布

4 Limit of determination and recovery

4.1 Limit of determination

The limit of determination of this method is 0.025 mg/kg.

4.2 Recovery

According to the experimental data, the fortifying concentration of tebufenozide in the sample and its corresponding recoveries are:

——0.500 mg/kg, the recovery is 94.8%;

——0.050 mg/kg, the recovery is 87.0%;

——0.025 mg/kg, the recovery is 85.6%.

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行 业 标 准
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SN/T 1770—2006
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中国标准出版社出版
北京复兴门外三里河北街16号
邮政编码:100045
网址 www.bzcbs.com
电话:68523946 68517548
中国标准出版社秦皇岛印刷厂印刷
*
开本 880×1230 1/16 印张 1 字数 22 千字
2006年8月第一版 2006年8月第一次印刷
印数 1—2 000
*
书号:155066·2-17034 定价 10.00 元

tary evaporator in a water-bath below 45 ℃, then blow to dryness under a nitrogen flow. the residue is dissolved accurately with 1 mL mobile phase(3.4.3.1), filtered through a 0.45 μm membrane and ready for LC determination.

3.4.3 Determination

3.4.3.1 LC operating condition

a) LC column: Nucleosil 100-5 C_{18}, 250 mm × 4.0 mm(i.d.), 5 μm or equivalent;
b) Mobile phase: acetonitrile-water (55+45, *V*/*V*);
c) Flow rate: 1.0 mL/min;
d) Column temperature: 40 ℃;
e) Detector wavelength: 234 nm;
f) Injector volume: 20 μL.

3.4.3.2 LC determination

According to the estimated approximate concentration of tebufenozide in the sample solution, select the standard working solution of similar concentration to that of sample solution. The responses of tebufenozide in the standard working solution and the sample solution should be in the linear range of the instrumental detection. The standard working solution should be injected randomly in-between the injections of the sample solution of equal volume. Under the above chromatographic condition, the retention time of tebufenozide is ca. 11.2 min, For the chromatogram of tebufenozide standard, see annex A.

3.4.4 Blank test

The operation of the blank test is the same as that described in the method of determination, but without addition of sample.

3.4.5 Calculation and expression of result

The calculate the content of tebufenozide in the test sample by LC data processor or according to the formula (2), the blank value should be subtracted from the result of calculation.

$$X = \frac{h \times c \times V}{h_s \times m} \quad \cdots\cdots (2)$$

where:

X——the residue content of tebufenozide the test samples, mg/kg;
h——the peak height of tebufenozide in the test sample solution, mm;
h_s——the peak height of tebufenozide in the standard working solution, mm;
c——the concentration of tebufenozide in the standard working solution, μg/mL;
V——the final volume of sample solution, mL;
m——the corresponding mass of test sample in the final sample solution, g.

前　言

本标准的附录 A 为资料性附录。
本标准由国家认证认可监督管理委员会提出并归口。
本标准由中华人民共和国黑龙江出入境检验检疫局负责起草。
本标准主要起草人：杨长志、康庆贺、田丰、韩广源、高勇。
本标准系首次发布的出入境检验检疫行业标准。