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INDUSTRY STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

SN/T 3149-2012

Determination of fentin and fenbutatin oxide residues and food for export. Gas chromatography-mass spectrometry method

出口食品中三苯锡、苯丁锡残留量检测方法 气相色谱-质谱法

(English Translation)

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Foreword

This standard was drafted in accordance with GB/T 1.1-2009 given rules.

Please note that some of the content of this document may involve patents. Distribution of this document Institutions do not assume the responsibility to identify these patents.

This standard is proposed and managed by the National Certification and Accreditation Administration Committee.

This standard was drafted. Tianjin People's Republic of China Exit Inspection and Quarantine.

1 Scope

This standard specifies the triphenyltin food for export and fenbutatin gas chromatography - mass spectrometry method.

This standard applies to export apples, cabbage, green onions, tea, rice, soybeans, fish, pork, kidney, milk, eggs and triphenyltin phenylbutyrate Determination of tin residues and confirmation.

2 Method summary

Sample remaining triphenyltin and tributyltin in benzene under acidic extraction with acetone, and then the n-hexane liquid - distribution, ethyl bromide derivative reagent, Generating ethyl triphenyl tin and tris (2-methyl-2-phenyl-propyl) ethyl tin, and then Florisil SPE column purification, eluent was concentrated After constant volume, gas chromatography - mass spectrometer and validation, external standard.

3 Reagents and materials

Unless otherwise specified, the reagents were of analytical grade, water is deionized water.

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3.1
hydrobromide.

3.2
acetone.

3.3
n-hexane.

Ethyl acetate 3.4. HPLC grade.

3.5
Cyclohexane. HPLC grade.

3.6
ethyl acetate - cyclohexane (11, volume ratio). exact amount of 500mL of ethyl acetate and 500mL of cyclohexane, after mixing for gel Chromatographic mobile phase.

3.7
anhydrous sodium sulfate. After burning 650 °C 4h, stored in a sealed container in the back.

3.8
sodium chloride.

3.9 3mol/L ethyl magnesium bromide solution in ether.

3.10 1mol/L hydrochloric acid solution. Take 20.8mL of concentrated hydrochloric acid, diluted with water to a 250mL volumetric flask.

3.11
acetone - n-hexane (12, volume ratio) solution.

3.12
hexane - diethyl ether (991 volume) was added.

3.13
triphenyltin standard (triphenyl tin, triphenyltinchloride, C18H15ClSn, CAS number. 639-58-7; triphenyl acetate Tin triphenyltinacetate C20H18O2Sn, CAS number. 900-95-8; triphenyltin hydroxide, C18H16OSn, CAS number. 76-87-9). purity of greater than or equal to 98%.

3.14
fenbutatin standards (fenbutatinoxide, C60H78OSn2, CAS Number. 13356-08-6). purity of greater than or equal to 98%.

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3.15

standard stock solution. accurately weigh the right amount of triphenyltin and fenbutatin standards were formulated with acetone to 100μg/mL standard Stock solution. Standard stock solution can be stored for 6 months at -18 °C condition.

3.16

standard working solution. Dilute into the corresponding standard working solution according to testing requirements with acetone. Standard liquid can be 0 °C ~ 4 °C 3 months under the conditions of storage.

3.17

Florisil SPE. 1g, 6mL or equivalent who filled in the column of about 1cm anhydrous sodium sulfate, previously living with 5mL n-hexane

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