Ain Shams University

Faculty of Science

Determination Of Some Organic disinfectants In Some Agricultural Products

Thesis Submitted

By

Osama El-Sayed Hussein Ibrahim

M. Sc. Chemistry (2013)

For award the PhD. Degree in Science (Chemistry)

A Thesis

Submitted To

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Department of Chemistry, Faculty of Science,
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Supervised by

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Dr. Mohamed El Sayed Amer
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Ain Shams University **Faculty of Science**

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***Title*:** Determination Of Some Organic disinfectants In Some Agricultural Products.

Submitted in Fulfillment of the Requirements for the Degree of Doctor of Philosophy in chemistry (Ph.D.)

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2017

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ABSTRACT

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ABSTRACT

Name: Osama El-Sayed Hussein Ibrahim

Title of the thesis: Determination Of Some Organic disinfectants In Some Agricultural Products.

Position: Researcher Assistant.

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Some quaternary ammonium compounds (QACs) containing at least one long alkyl chain have biocidal properties and are widely used as general fabric softeners, disinfectants, preservatives, pest-control and anti-fouling products.

A Liquid chromatography tandem mass spectrometry (LC-MS/MS) method was developed and validated for determination of 5 quaternary ammonium compounds (QACs) in Green Beans, Orange and Olive using modified quick, easy, cheap, effective, rugged, and safe (QuEChERS) method for extraction.

QACs were separated on a LC-MS/MS HP (Agilent) 1100 series equipped with triple quadruple mass spectrometer (ABSCIEX), binary pump, Vacuum degasser and Auto sampler.

Gas Generator designed to provide nitrogen gas at least 5 L/min at 60 psi, source gas (zero air) at least 22 L/min at 100 psi and exhaust gas (air) at least 8 L/min at 60 psi.

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Separation was performed on a C18 column ZORBAX Eclipse XDB-C18 4.6 × 150 mm, 5 µm particle size using mobile phase 10 mM ammonium formate with methanol at pH 4.

Mean recovery ranged from 78 to 117%. No significant dispersion of results was observed for the QACs recovery at the lowest and the highest concentrations and RSD% showed that most of QACs were less than 10%.

The measurement uncertainty is expressed as expanded uncertainty and in terms of relative standard deviation (at 95% confidence level) is ±19%.

This method is suitable for laboratories engaged daily in routine analysis of a large number of samples.

Key words: QACs; QuEChERS; LC-MS/MS; C18 column and RSD.

Supervisors' approval:-

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The present work aim to develop and optimize an analytical method including separation and determination of some quaternary ammonium compounds (QACs) in food using Liquid chromatography tandem mass spectrometry (LC-MS/MS), followed by method validation for method used in analysis.

Method development and optimization

For this purpose, the work divided into the following parts:

1. Develop of suitable HPLC parameters.
2. Optimize suitable masses for QACs using the Multiple Reaction Monitoring (MRM) mode which is a way to reduce noise and to avoid matrix interference peaks.
3. Development and optimization of the analytical method for extraction then validation using the following criteria:-
   a) Recovery test
   b) Limit of detection (LOD)
   c) Limit of quantitation (LOQ)
   d) Linearity (including standard and method linearity)
   e) Accuracy (expressed as trueness)
   f) Precision (including repeatability and within-laboratory reproducibility)
   g) Measurement Uncertainty
4. The applicability of the developed method for the routine monitoring of locally produced or exported agricultural products will be investigated, as part of the National Monitoring Program for food contaminates in Egyptian food. This should help various Egyptian exporters to penetrate international markets.

ACN	Acetonitrile
ADBAC	Alkyldimethylbenzylammonium chloride
ADI	Acceptable Dietary Intake
AOAC	Association of Official Analytical Chemists
ARfD	Acute reference dose
BAC	Benzalkonium chloride
BfR	German Federal Institute for Risk Assessment
BKC	synonym for BAC
BPD	Biocidal Products Directive
bw	body weight
C18	Octadecyl carbon chain bonded silica
CAS	Chemical Abstracts Service
DAC	Didecyldimethylammonium chloride
DDAC	Didecyldimethylammonium chloride
DDDMAB	didecyl dimethyl ammonium bromide
dSPE	dispersive solid phase extraction
E.C.S	European Communities For Standarization
EDI	Estimated Daily Intake
EFSA	European Food Safety Authority
EPA	Environmental Protection Agency
ESI	Electrospray ionization
EU	European Union
FDA	Food and drug administration
HPLC	High Performance Liquid Chromatography
HSE	Health and Safety Executive
ISO	International Organization for Standardization
Kg	Kilo-gram