International Journal of Science and Research (IJSR)
ISSN (Online): 2319-7064
Index Copernicus Value (2015): 78.96 | Impact Factor (2015): 6.391

Detection of Pesticide Residues and Other Additives on Some Imported Fruits and Vegetables using Gas Chromatography - Mass Spectrometry

Eman M. Rasheed[1], Qais A. Ibrahim[2]

[1]Ministry of Agriculture, Department Plant Protection, Iraq

[2]Division of Primaries Sciences, Collage of Agriculture, University of Baghdad, Iraq

Abstract: ***This paper presents a detection of pesticide residues and additives on imported fruits(Strawberry,Grapes,peaches and apples)using acetonitrile extraction method to determine the residual concentration during seasonal growth until it reaches the consumer. Extraction and purification processes conducted for crops in postgraduate Labs Faculty of Agriculture University of Baghdad. Different fresh fruits and vegetables includes; Egyptian strawberries, African grapes, peaches, American Apple, potato, Iranian tomato, cucumber, Iranian red pepper and Turkish potatoes and cucumberwere tested and analyzed using Gas Chromatography/Mass Spectroscopy(GC/M.S). QuEchERS method has been applied for extraction pesticide residues from targeted Vegetable and fruits. The(GC/M.S) has been carried out for distinguish the Vegetable and fruits. Different factors were studied; retention time, elution time, relative abundance and molecular weight. The results showed that the pesticides with low molecular weight were eluted before the high molecular weight pesticides residues of pesticides. Different types of Pesticides were identified; pheromones, organic acids and solvents and anti-fungal to develop a comprehensive intervention measures and to reduce the potential health risk to consumers.***

Keywords: pesticide residues, fruits, vegetables, acetonitrile, GC/MS, QUECHERS

1. Introduction

Pesticide is defined any substances or mixtures of substances intended to prevent, destroy, combat, repel, or mitigate the impact of a pest [1]. Therefore, a pesticide may be a chemical, biological agent or agent (such as a virus or bacteria), antimicrobial, disinfectant, bactericidal agent or even a tool used against any pest. Here the pest may be an insect.pesticide residues define To pesticides that may remain on or after agricultural products. Regulatory bodies in many countries usually determine the maximum allowable percentage of those residues [2].

Food is the mainstay of life, and it is the key to the continuation of human life and safety. In order to achieve this, not only food is needed, but the most important thing is to provide balanced and fresh food free from all pollutants that harm human health, including pesticides residues [3].

Pesticide residues of fruits and vegetables after being treated with pesticides, which reach the surface of the plant or nearby water ways[4].The residues are quantitatively estimated at one milligram per kilogram of food. Residues are shown in the exposed parts of the sprayed plant such as fruits, leaves, It can be said that pesticide residues are only a substance or a mixture of active ingredient and its decomposition products and other materials that help to be present in or on the crop used by humans as food or used as animal feed added to control the pests The crop is attacked[5]. Pesticide residues are classified according to the purpose of their use to their residues in vegetables and fruits. Studies have confirmed the presence of residues of the malathion herbicide Cucumber, cucumbers, tomatoes, potatoes, apples, French and American apples in a number of Arab countries, from which samples were collected, as well as chlorophosphate, spinach, cucumber, cucumber and tomatoes, as well as the detection of residues of dimethite on cucumbers in these countries. The remaining residues ranged between 0.001 - 0.015 ppm [6]. Other studies have identified the rate of disappearance and destruction of insecticides (delta-methine and pyrimethrin) and four types of fungicides: minremol, tri-dimphon, quinoithione, pyrazophos, and acaros dicofol and the remaining pesticides after repeated spraying on tomato fruits in the protected house. These fungicides were completely destroyed within three weeks and others showed an accumulation of residues with sprays [7]. In the results of other studies conducted in the Kingdom of Saudi Arabia, the presence of organic parathyroid insecticide (Permethrin) on green onions0.207 ppm, while it is allowed to be present from this pesticide by 0.1 (ppm). In order to determine the permissible limits of pesticide residues on vegetables, studies have been conducted in a number of Arab countries where organic phosphorus residues, phosphamidone, methadethione, On potato plants in open fields or on cucumber plants in greenhouses where it was observed that pesticide residues were less than allowed after 16 days of pesticide spraying. [8]. Researchers have shown that increasing the concentration of the pesticide in the human body causes bad changes in humans Through TGA B was conducted on laboratory animals. The pesticide dichloro-diphenyl-trichloroethane (DDT)The human body is concentrated in the fatty layers of the human body. The risk of the pesticide is concentrated in its survival in agricultural soil for a long period without decomposition. The continuous intake of grains and the contaminated fruits and its consumption of meat. The birds that feed on the insects that are contaminated with these pesticides accumulate inside them. Eating these birds for insects and when eaten by humans cause slow poisoning leads to death, the greater the

accumulation of quantity or the human being poisoned by eating the meat of animals that feed on contaminated plants [9]. The Daily Acceptable Daily Intake Limit (ADI)where the maximum residue of pesticide residues allowed for agricultural goods is found in marketing and in food, which can be identified as the amount of residual residues that humans consume without affecting their health. Eat daily with food and do no harm to human health. Most food products contain different concentrations of pesticides and these concentrations may be within the permissible limits or higher[10].This is called the maximum permissible limit (MRL)

These limits are determined by special organizations and laboratories that depend on the determination of these values based on, among other things, the type of pesticide, its method of effect and the way it is transmitted in the plant as well as the nutrition habits of the peoples which differ from one country to another[11]. Iraq is a member of the Codex Committee, IssuedThe countries of the world differ in determining the value of MRL and ADI according to the system adopted in each country. For example, the law in Scandinavia is the highest in terms of the permitted values for each pesticide, followed by Australia, New Zealand [12]. The Preharvest Interval (PHI) safety period, which is the period between the spraying of the plant with the pesticide until the crop is harvested, and the PHI, varies from one country to another. Crop to another that the residues of primary and final residues are dependent on the dynamic influencing factors. The initial residues on the leaves of treated plants are subject to dynamic processes due to climatic conditions such as heat, light and humidity. The pesticide is based on the chemical and physiological properties of the pesticide such as water solubility, vapor pressure, treatment method (air, ground spray) [13].

That there are many methods used to ensure the safety of food pesticides residues where they took wide space in different countries of the world for the preservation of human health and the fact that the principle of chemical control is the most used method. Food preparation, such as washing, peeling, cooking, canning and cooking, leads to a significant reduction in pesticide residues on these foods. Washing with water only, washing with soap and water, using saline solutions and manufacturing processes such as peeling and pickling have a significant role in removing or reducing pesticide residues in used plant parts as food helps these Processes in reducing pesticide residue in samples taken immediately after treatment from samples taken with a period of treatment for the probability of the pesticide entering the plant parts so that the pesticide or its metabolic product becomes part of the vegetable tissue[14]. The GC/M.S separation method is an effective method for separating mixtures up to several micrometers by passing the sample in a vapor state through a column that contains a liquid or solid medium and moves its components at varying speeds depending on the degree of boiling or solubility. Yassin mentioned that the sample to be separated must be constant under pressure conditions and the temperature used. The sample is usually inserted by injecting it directly at the top of the column where it is carried by the gas moving medium to separate the column and its separated components at the end[15].

2. Materials and Methods

Apparatus:
The (Table 1) Blew shows apparatus that used for detection process of pesticide residues in targeted vegetablesandfruits**.**

No	Instrument	Model	Company
1.	Pipes	China	China
2.	Pickat with different size	China	China
3.	Rotory Evaporator RE-300	Stuart	USA
4.	GC/M.S-QP2010 Plus	SHIMAOZU	Japan
5.	Blander	National	Japan
6.	Knife	Tramonina	Brazil
7.	Shaded glass boxes for sample preservation	Samarra	Iraq
8.	Filtration & Microfil Tration	Chmlab group	EEC (SPAN)
9.	Buechner funnel	China	China
10.	Plastic cans	Shiny Face L.L.C	Malaysia
11.	Camshafts	Shiny Face L.L.C	Malaysia
12.	Sensitive balance	Sartorius	Germany
13.	milipore filter	China	China
14.	Magnetic stirrer	Roche	Germany

Chemicals and Solvents:
The Chemicals and Solvents brought company from From multiple companies by OMA International office in Baghdad. Most used materials that are used described in the(Table2)

No	Chemicals and Solvents	Model	Company
1.	Acetiniteol	Sigma- Aldrich	Germany
2.	Mgso4 Ang.	HIMEDIA	India
3.	NaCl	BAKER J.T.	Denmark

GC/M.S optinal Condition Used for samples Extracted by QUECHERS method.
Were Conducted using Column of type DB-5MS, column length (30m), thickness 0.25 um, diameter of column 0.25mm(Stationary phase).Helium was used as the carrier gas (mobile phase) at flow rate 10 ºC/ min and temperatures between (280-60) ºC 0 and the time needed to separate each sample is (30 min).[16].

Determination of pesticide residues
Determination of pesticide residues have been carried out by using gas chromatography(GC/M.S).[16].

Sample Collection
The experiment was conducted for a group of fruits and vegetables imported from different countries as shown in Table (3). Method [16] and [17] was used after some modifications were made according to the possibilities available for the purpose of extracting the samples and then purifying them and finally detecting them using GC / M.S.

No	Imported Fruit	Origin	No	Imported Vegetables	Origin
1	Strawberry	Egypt	1	Tomatoes	Iran
2	Grapes	African	2	Potatoes	Turkish
3	Peaches	African	3	Cucumbers	Turkish
4	apples	American	4	Red pepper	Iran

Extraction Method

QuEchERS method used for extract pesticide residue from Fruit&Vegetablestissue. The name represent brief for those word,quick,easy,cheap,effective,rugged,and safe.(50 g) of the samples (50g) on vegetables and fruits under study washed and cut into small parts and placed each sample separately in the blender and add 100 ml of acetonitrile (ACN) and mix the mixture for 5 minutes at 3000 cycles / minute, and then extract the minute (D11) for the purpose of disposing of impurities and substances in the extract, take the resulting odor from the filtration process for each sample and add a chloride substance to each sample in a glass flask using Buchner funnel. Sodium NaCl by 1.5 g and $Mgso_4$ Anhy. placed on a magnetic stirrer (Hot Plate& Strrer) with no heat to be applied when the sample was placed. The voltages and frequency were 230 V 50 H2 and the flask was left on the machine for 10 minutes. The filter was then reintroduced in a glass flask Using the Buchner funnel funnel to remove all additives and impurities in the extract, take 100 ml of extract for each sample and put in a round glass flask (evaporator) diameter (32/29) and use the rotary Evaporator RE-300(300 ° C) and the water bath (40-35 ° C). The evaporation was delayed until the acetonitrile droplets stopped falling into the recovery flask. The remaining liquid in the glass beaker of each sample was less than 20 ml. Filter the remaining extract in the round glass beaker for each sample using Milli Pore filters. Chm SHIFT - filtration with pore size 0.45 and diameter 25 mm to ensure a very pure extract and thus obtain accurate results when conducting GC/M.S.[16][17]

3. Results and Discussion

GC/M.S Results

Using GC/M.S analyzed sample instrument after extraction process had been done. (Table 4)Show The results of GC/M.S.

Sample		Compound	Formula	Retention time	Peak area
American Apple	1	Propen	C_4H_60	5.394	25.87
American Apple	2	Allyl Carbonate	$C_7H_{10}O_3$	6.872	31.59
Egyptian Strawberry	1	Isopropyl ether	$C_6H_{14}O$	7.986	11.24
Egyptian Strawberry	2	Hydracrylonitrile	C_3H_5NO	2.014	1.49
African grapes	1	Acetonitrile	C_2H_3N	3.690	8.77
African grapes	2	2,3 Dihdrofuran	C_4H_6O	20.546	1.62
African Pears	1	2Propoxy ethanamine	$C_5H_{13}NO$	2.141	6.31
African Pears	2	Eoct	$C_8H_{16}O_4$	2.070	9.62
Iranian Red Pepper	1	Trimethly hexane	C_9H_2O	2.023	31.54
Iranian Red Pepper	2	Trimethly pentane	$C_{11}H_{24}$	2.066	34.24
Iranian Tomato	1	Allyl carbonate	$C_7H_{10}O_3$	2.341	0.35
Iranian Tomato	2	Butanesulfonyl chloride	$C_4H_9C_1O_{25}$	2.135	43.72
Turkish cucumber	1	5-Methoxazolidine	C_4H_9NO	2.632	2.45
Turkish cucumber	2	Amyl hydroperoxide	$C_5H_{12}O_2$	2.111	3.54
Turkish cucumber	3	Nitrobutane	$C_4H_9NO_2$	9.341	0.16
Turkish cucumber	4	2,4-dimethylpentane	C_7H_{16}	2.282	0.98

The results of the gas chromatography, where the x-axis represents the time required for the appearance of the sample. The yaw axis represents the height of the curved tops of the extract.

Figure 1:

Of the American apple extract is the presence of Propene, which is usually added to improve the effectiveness of pesticides through the work of solid solutions for pesticides used to control by spraying Spraying [18].

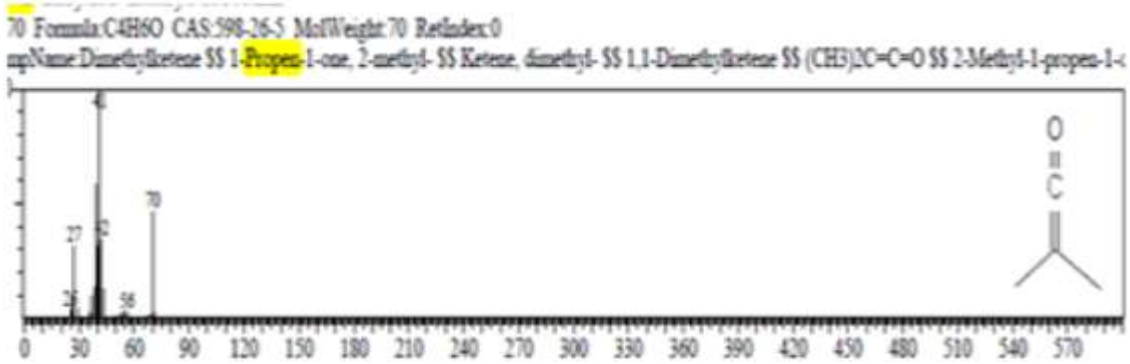

Figure1: Examination of the gas chromatography of the American apple extract

Figure 2

Shows the presence of another substance called Allyl Carbonate Fermium is added to the pesticide for the purpose of strengthening it and increasing its susceptibility to killing by attracting insects to the active ingredient of the pesticide.

The rest of the results were not mentioned because they are only natural compounds within the composition of the crop[19].

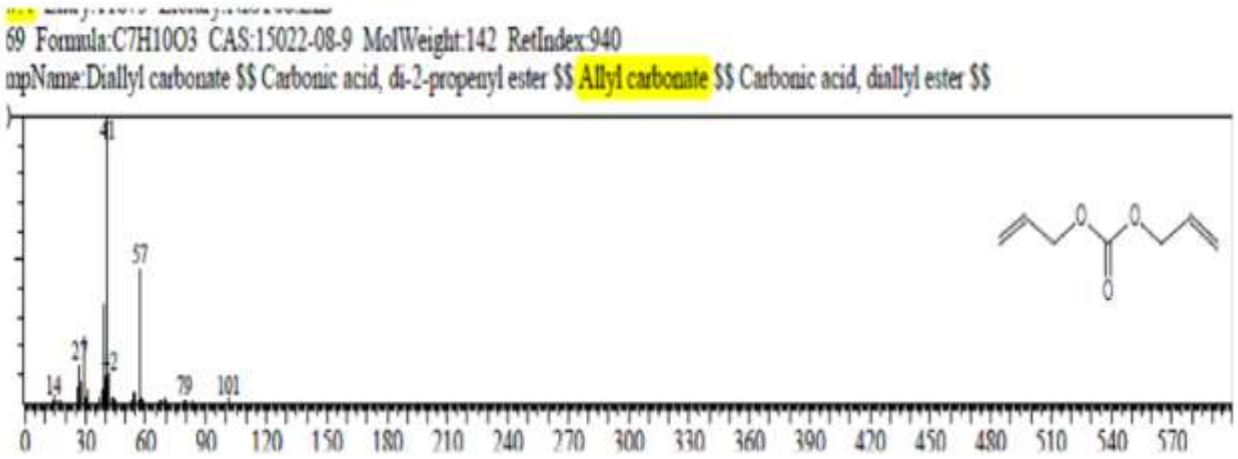

Figure 2: Examination of the gas chromatography of the American apple extract

Figure 3

Shows the compounds in the strawberry extract from the presence of Iso propyl ether, a substance added to the acetonitrile solvent used to dissolve the active ingredient of the pesticide as the pesticide appeared in the form of fumigant residues where these evaporators As new ways to control agricultural pests if they are better in terms of effectiveness as well as lower production cost compared to the usual pesticides used. The acetonitrile solvent or other solvent can be introduced as a developer of the pesticide and may also be incorporated into the pesticide formulation [20].

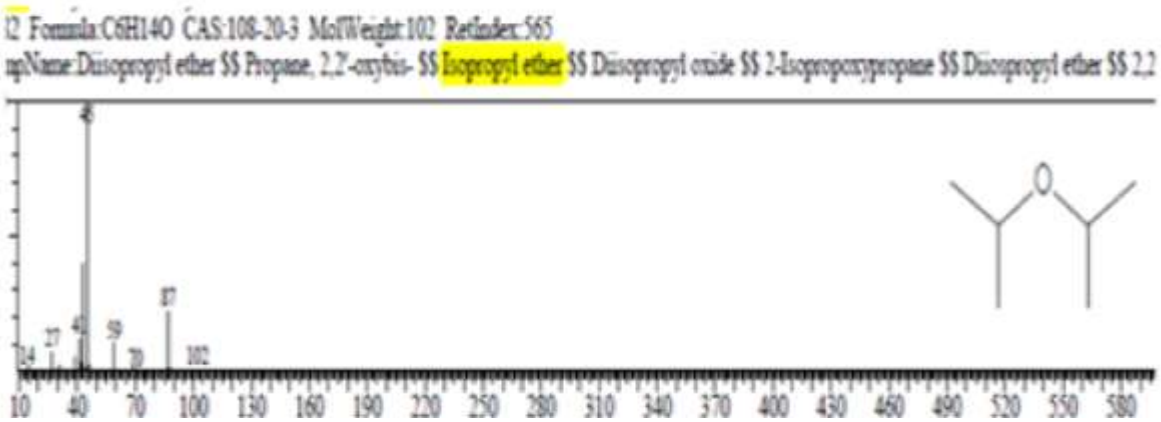

Figure 3: Examination of the gas chromatography of the Egyptian strawberry extract

Figure 4

Shows the compound Hydracrylonitrile, a compound in the synthesis of unsaturated organic acid (Cyanoalkyl propiolates) and the latter has the potential to inhibit the growth of micro-organisms (especially microorganisms), so it is used as a fungicide [21].

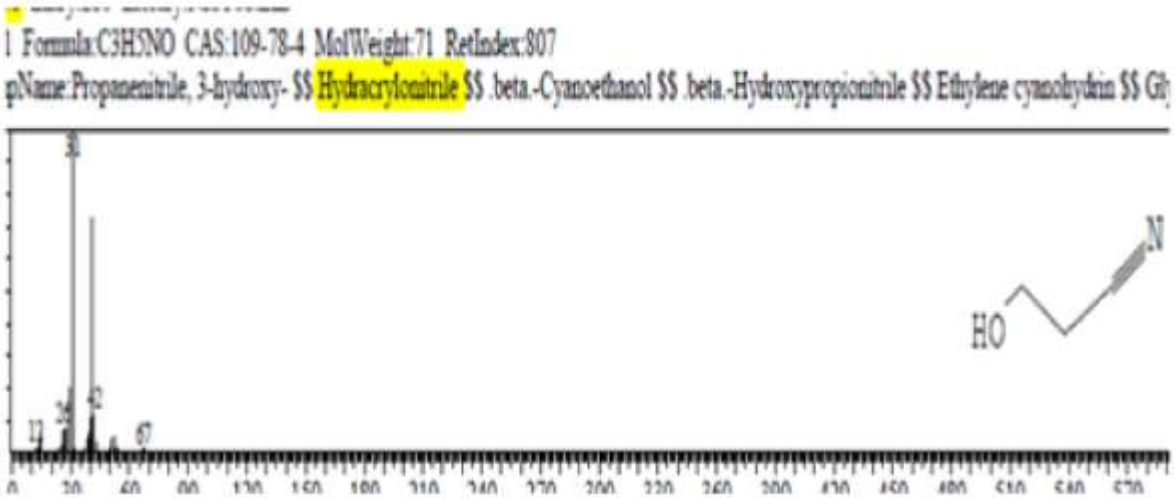

Figure 3: Examination of the gas chromatography of the Egyptian strawberry extract

Figure (5):
Shows the compounds obtained from the gas chromatography examination of African grapes if the acetonitrile solvent is present. It is used here to dissolve the pesticides and to prove that the acetonitrile solvent is used in the pesticide industry by efficiently removing the pesticide from the plant tissue because it is dissolved The plant cells are confined to the plant and are used on a concentration basis where it works more than the dilution and selects itsappropriate [22].

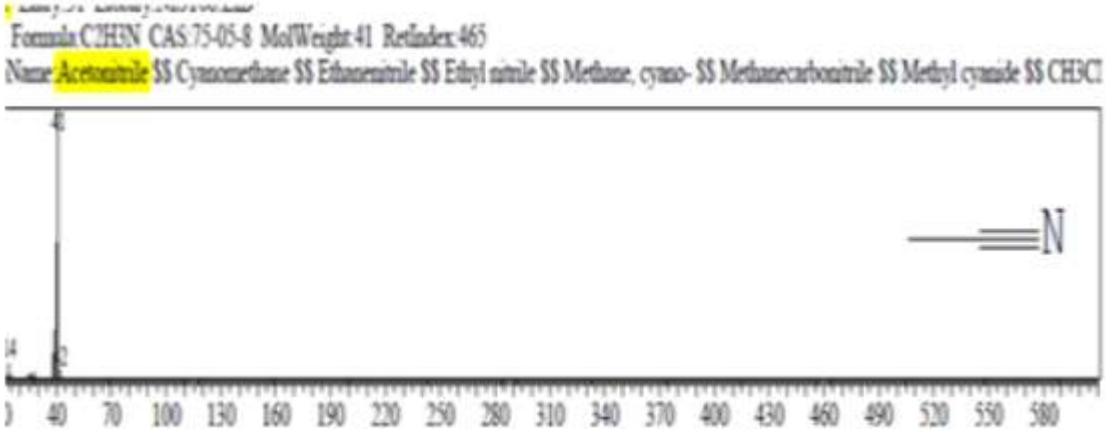

Figure 5: Examination of the gas chromatography of the African grape extract

Figure (6):
shows the presence of Article 2,3-Dihydrofuran and this article has the ability to stop feeding the insect where it is added to the pesticide as an antifungal agent to feed the insect and therefore when the feeding of the insect gets regression in the insect society,meaning that the number of insects decreases and therefore when reducing the number of insects that strike Plant less control processes and this has a good economic return [23].

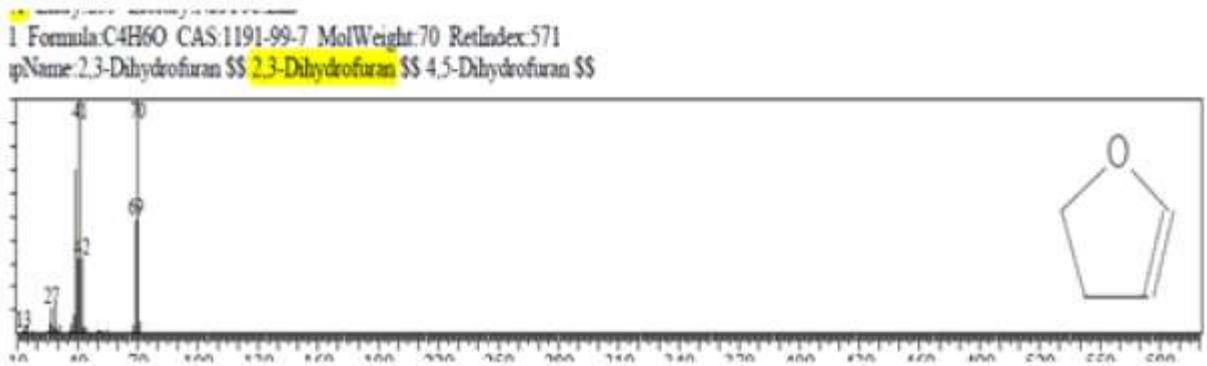

Figure 6: Examination of the gas chromatography of the African grape extract

Figure 7 :
Shows the compounds obtained from the gas chromatography of the African pea extract, the presence of the substance (2-Propoxy ethanamine). This material is produced by Triflumizole and the latter is a fungicide[24].

Figure 7: Examination of the gas chromatography of the African Pea Extract

Figure (8):
Shows the presence of a compound (Eoct), a pesticide registered by the National Commission for Pesticides accredited in India [25].The rest of the results were not mentioned because they are only natural compounds within the composition of the crop.

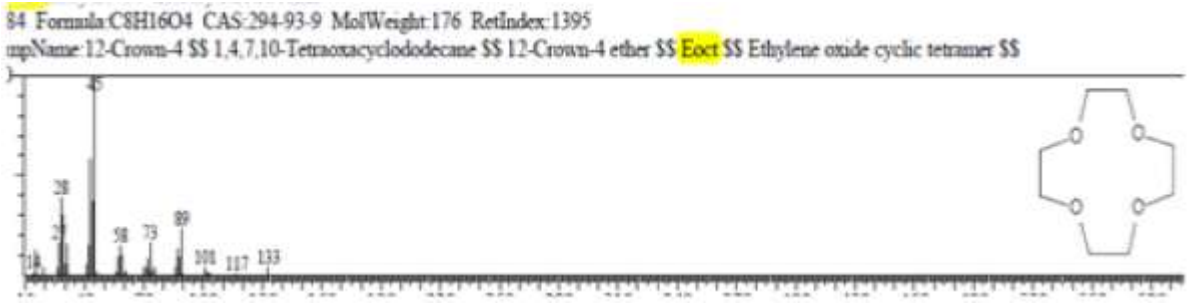

Figure 8: Examination of the gas chromatography of African pears

Figure (8):
The presence of the compound (Eoct) in the extract isolated from the potato crop, which is a pesticide registered by the National Committee for pesticides approved in India [26]. while the rest of the results did not mention Because they are only natural compounds within the composition of the crop

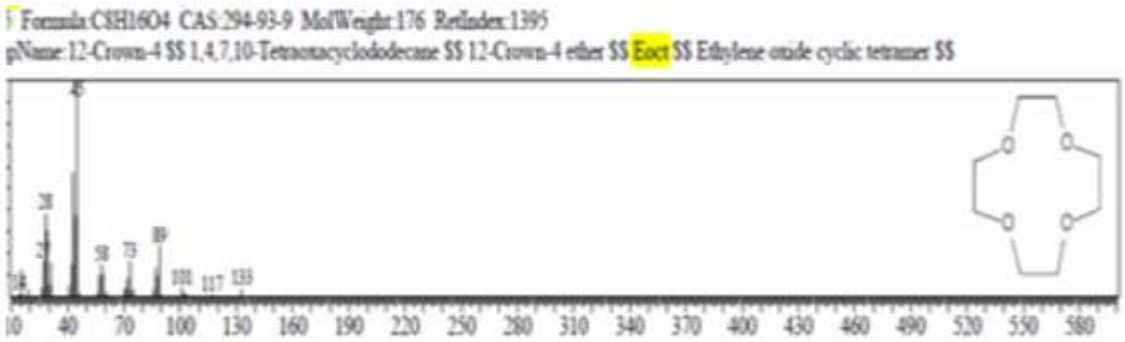

Figure 9: Examination of the gas chromatography of Turkish potato

Figure 10:
Shows the presence of the Trimethylhexane compound in the Iranian Red Pepper crop, which is a compound that increases the affinity between the pesticide and the water. In increasing intimacy, the solubility will increase and thus increase the plant's absorption of the active ingredient of the pesticide [27].

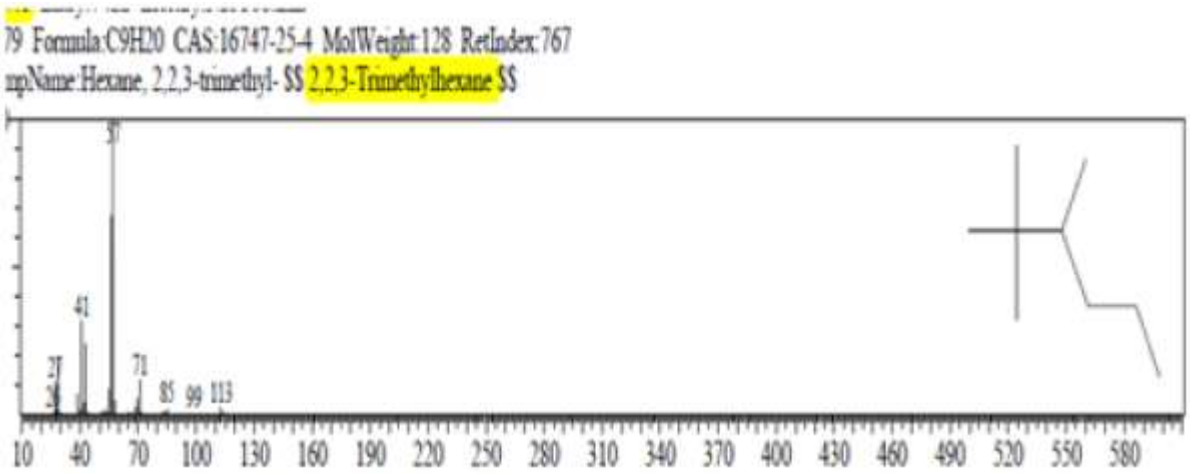

Figure 10: Examination of the gas chromatography of the extract of the Iranian red pepper

Figure (11):
Shows the presence of the compound (Trimethyl pentane), which is also a compound that increases the familiarity between the pesticide and water and when increasing the affinity will increase the solubility and thus increase the absorption of the plant to the active substance of the pesticide [28]. The rest of the results were not mentioned because it is only a Natural compounds within the composition of the crop.

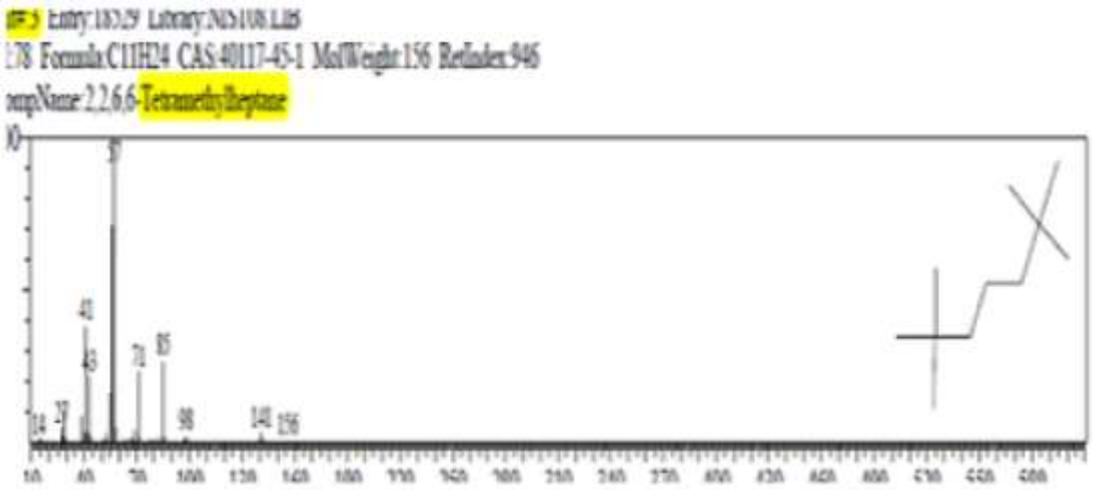

Figure 11: Examination of gas chromatography of the extract of Iranian red pepper

Figure 12:
Shows the compounds obtained from the gas chromatography examination of the Iranian tomato extract. The presence of Allyl carbonate, a substance used in the manufacture of the early stages of the cyclization precursor. This initiator is used in the manufacture of fermons and pheromones added to the pesticides to increase the efficiency of the pesticide because it works to attract the insect For the active ingredient of the pesticide [29].

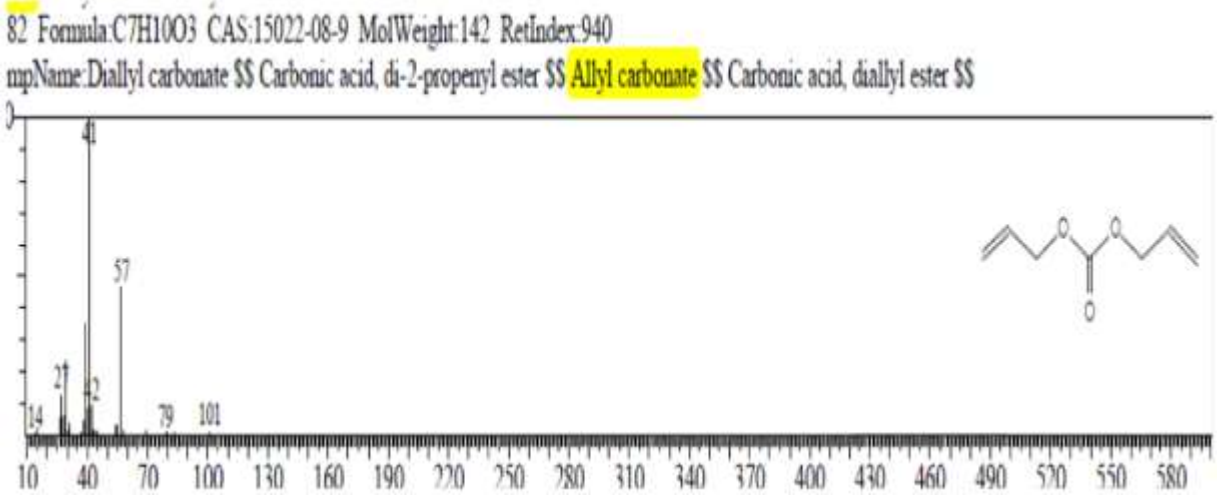

Figure 12: Examination of gas chromatography of the extract of the Iranian tomato

Figure 13:
Shows the presence of Butanesulfonyl chloride, a herbicide used to prevent bush growth in the irrigation channels of Paddy rice and bush growing on the edges of irrigation channels. Butanesulfonyl chloride is used in the manufacture of Pyrozole and the latter It is used in paddy rice[30]. The rest of the results were not mentioned because they are only natural compounds within the composition of the crop.

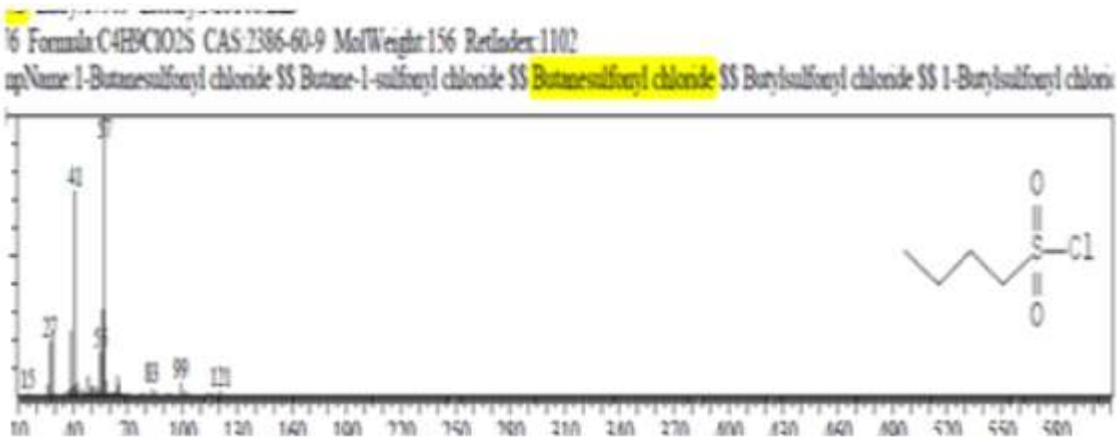

Figure 13: Examination of the gas chromatography of the extract of the Iranian tomato.

Figure 14:
Shows the compounds obtained from the gas chromatography examination of the cucumber extract of the Turkish cucumber (5-methloxazolidine) if they are used as fungicides, which are used as insecticides. Tetrahydrooxazine is added to it and works together as an insulating layer and the layer has an insect repellent activity[31].

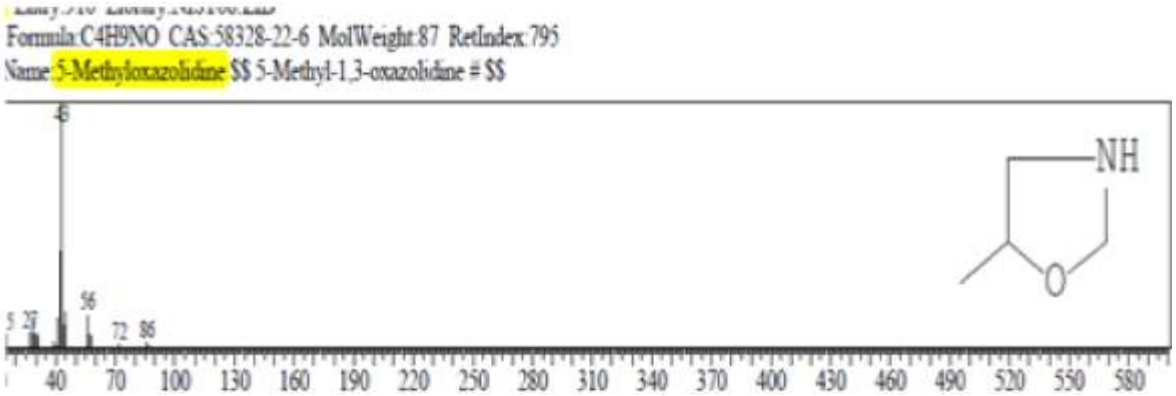

Figure 14: Examination of the gas chromatography of the cucumber extract of the Turkish cucumber

Figure (15):
Shows the presence of the substance (Amyl hydroperoxide) in the Turkish cucumber crop and this article works to inhibit phosphorus pesticides[32].

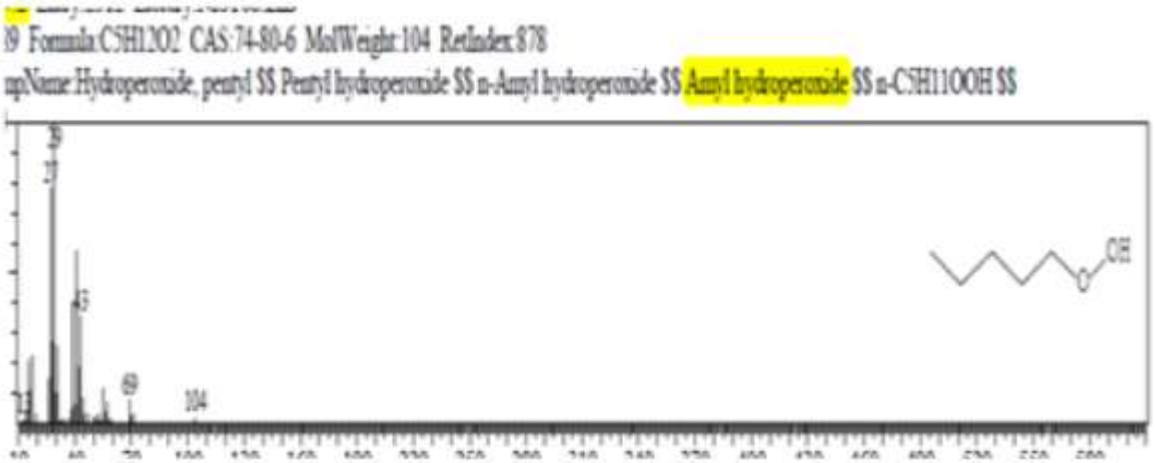

Figure 15: Examination of the gas chromatography of the cucumber extract of the Turkish cucumber

Figure (16): shows the presence of nitrobutane in the Turkish cucumber crop. This substance is mixed with bordex and works together to break down the insect wings and thus lead to its death and reduce its damage to the crop[33].

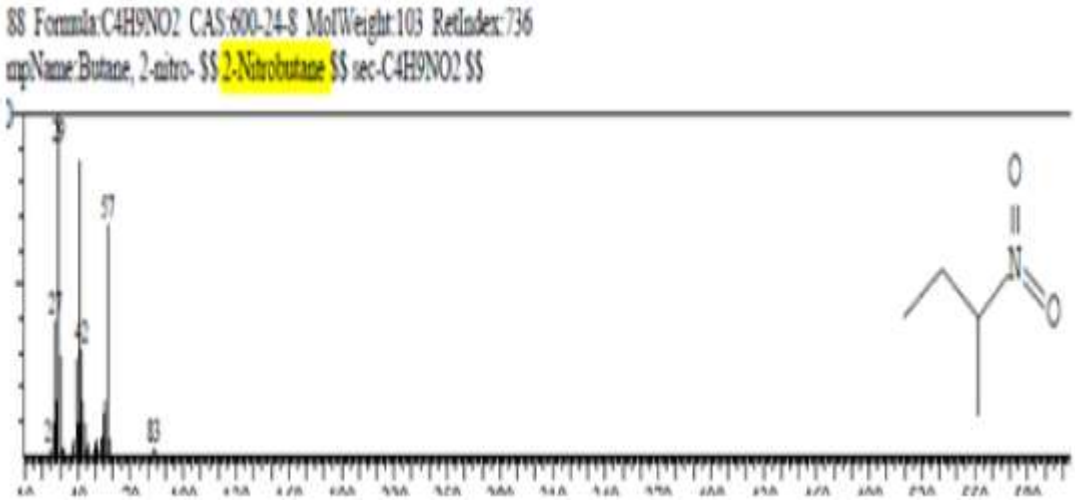

Figure 16: Examination of gas chromatography of the cucumber extract of the Turkish cucumber

Figure (17)

Shows the presence of a substance (2,4-dimethyl pentane) in the Turkish cucumber crop This article contains the cyclic boric acid (boric acid) and this acid value as starting materials or intermediates in the production of chemical derivatives can be used as additives in Production of pesticides [34].

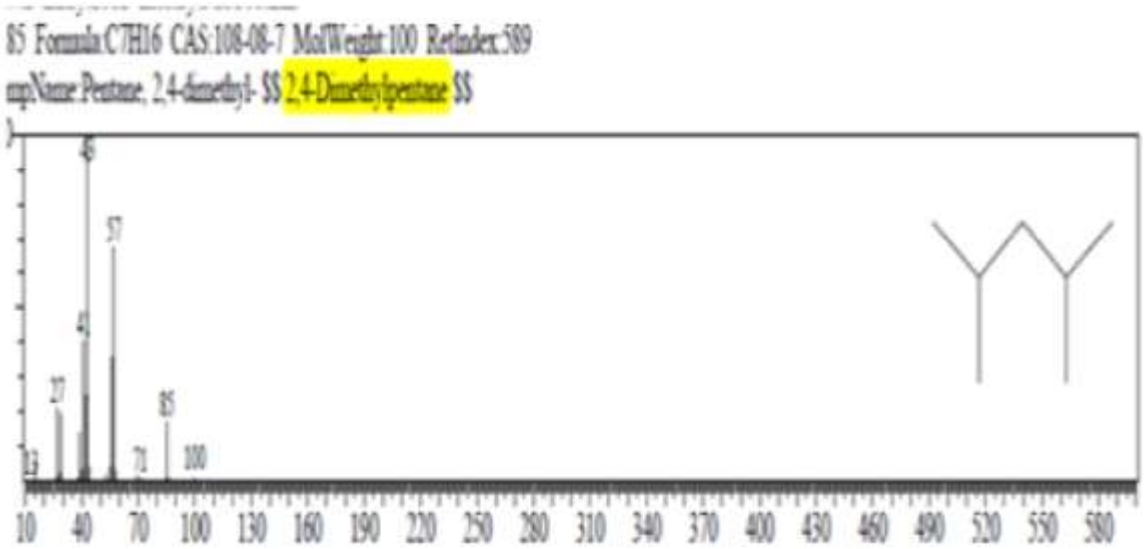

Figure 17: Examination of gas chromatography of the cucumber extract of the Turkish cucumber

4. Conclusions

We conclude from this study that imported fruits and vegetables carry many toxic and harmful substances to the health of the consumer as a result of the transactions carried out to ensure their access to the consumer of good quality. Therefore, it is preferable to encourage local agriculture on a regular and good basis and to protect the crops from the pests that they destroy through the optimum use of the pesticide within the permissible limits or the use of less toxic pesticides instead of importing crops subject to different treatments to ensure that they reach the consumer of high quality. Protection of consumer health the second is to encourage domestic product to compete importer.

References

[1] Thermo Instrument Systems Inc. History".(2015).International Directory of company Histories. Funding universe.com/company–histories/thermo–instrumensystem –inc – history.volume11ed. st.James Press.1995.pp.513-514.

[2] Alex,W.leonard,;RossV.hyne,*;andFleurpablo.(2002).Trimeihylpentane- Containing passive sample ERS for predicting time – in tegrated concentrations of pesticides in water .Department of Environmental Sciences,Universityoftechnology – Sydney, center for ecotoxicology, Westbourne street, Gore Hill, New South Wales, Centre for Ecotoxicology, Westbourne Street, Gore Hill, New South Wales 2065, Australia.

[3] Alon,t.; Amirav,A.(2006).Isotope Abundance Analysis Method and Software for Improved Sample Identification with the Supersonic GC-MS. Rapid Communications in Mass

[4] AmiravA,GordinA,Poliak.M,Fialkov.AB.(2008).Gas chromatography – mass spectrometry with supersonic molecular beams. Mass Spectrom.43(2):141-63.Doi:10.1002/jims.1380.PMID18225851.

[5] Azam.(2015).Competitive protein – binding assay – based Enzyme immunoassay Methods, Compared to High – pressure liquid chromatography immunoassay Methods, Compared to High pressure liquid chromotgraphy,Has a very lower Diagnostic Value to Detect Vitamin Deficiencyin.Deficiency in 9-12 years children Interactional journal of preventive medicine.

[6] Baldil et al .(2010). Pesticide residues in food .Nik Ronaid.6

[7] RobertP.,DrAdams.(2007).IdentificationofEssentialOilComponents By Gas Chromatography/ Mass Spectrometry . Allured Pub Crop . ISBN 1-932633-21-9.Rochester Medical Center Rochester.

[8] Smith,P.A.;Lepage,C.J.;Lukacs,M.;Martin,N.;Shufutinsky,ASavage,P.B.(2010)."Field-portablegas chromatography with transmission quadrupole and cylindrical ion trap mass spectrometric detection Chromatographic retention index data and ion / molecule interaction for chemical warfare agent identification". International JournalofMass Spectrometry. 295(3):113-118, doi: 10.1016/j.ims . http://doi .org/10.1016%2Fj.ijms.

[9] Brock,DavidC.(2014)."AMeasureofSuccess"(http://www.Chemheritage.org/distillations/article/measure–success). Chemical HeritageMagazine.29(1).

[10] Hites,Ronald A.(2016). Development of Gas chromatography and Mass spectrometry .Analytical Chmestry.88(14) : 6955- 6961doi : 10.1021 / acs. analchem 6b01628.

[11] Jane.(2015). How to Sook Vegetables& Fruit in Sea salt wate to Remove Pesticides.11.

[12] JeromeJeyakumar,J.;etal.(2015)."AstudyofPhytochemicalConstituents in Caralluma Umbellata By GC-MS Anaylsis [sic] " http://www.ijpsi.org/Papers/Vol2%/284%29%/version1/

E243741.pdf (PDF).International Journal of Pharmaceutical Science Invention: 37-4112.
[13] Webb-Halpern, Leah.(2008). "Detecting Success" Chemical Heritage Magazine. 26(2):31.
[14] Pitarch,E.,Serrano,R.Lopez,F.J. and Hernandez, F.(2003). Rapid multiresidue determination of Organochlorine and extraction and gas chromatography coupled to tandem mass spectrometry . Journal of Analytical Bio analytical chemistry. 376(2):189-197
[15] MinminLi,ChaoDai,Fengzhongwang,ZhiqiangKong,YanHe,YaTao,Huang&BeiFan.(2017).Chemometric–assistedQuEChRSE xtraction method for Posta harvest pesticide determination in fruits and vegetables. Scientific Reports 7,Article,number:42489.doi:1038/srep 42489.
[16] Alyaseri, Ismael Ibrahim ,Mukhtar Abdul Satter Ali ,Abdul Karim Jawad Ali and NadirKamil Bahi .2012.Determination of apaerticides Residues in some Fruits and Vegetables Imported to Iraq. Journal of Agricultural Science and Technology , ISSN 1939-1250
[17] Mitsuru shibata, Sode gaura .,Ichiro Nasuno. Ichihara.,Kazyoshi , Koike,.Masashi Sakamato.1995. Pyrazole compounds having athiochroman ringand pesticides.ContaininG.S.UCH Compounds .both of sodegaura all of Japan.
[18] A.Lajiness, Thomas; Racine , Wis.1972.Oxazo idine andtetrahydrooxazingcompounds having insect repellent activity . assignor to no drawing. Filed mar,ser. No.20,097 Int.cl.co7 d 87/ 06, 85/26,3,707,541.
[19] Philip James Garner , Hooton , England. 1953.Cyclic ester of boric acid ,Development company , Emeryville, calif ., corpora – tion of delaware no drawing .Application serial no . 337 , 427 . claims priority , application Great Britain 14 claims c1.260-462.
[20] Blair R.Brilett. 1964.Toxicity of some pesticide to eggs , Larvae and Adults of the green laceming , chrysopa ,carnea .University of California Citrus Research center and Agricultural experiment station , Riverside.
[21] Debra Edwards , ph. . D. Director . 2007.Triflumizole Summary Document Registration Review Docket . Registration on Triflumizole , CASE 7003. Special Review and Reregistration Division .
[22] Edwin A.kleinGebbinc ,GerritA.Stork, Ben J.M.Jansen andAededeGroot1999.Synthesis and Insect antifeedant Activity of 2- substituted 2,3-Dihydrofuran-3-ols and Butenolides, (part 11) Laboratory of organic chemistry. University Research center , Dreijenplein 8,6703 Wageningen. Environment, Elsevier. 450-451,pp.31-38/22.1.
[23] Hits , Ronald A. 2016. Development of Gas chromatography and Mass spectrometry Analytical Chemistry. 88(14) : 6955- 6961doi : 10.1021 / acs .analchem 6b01628.
[24] Karl,Kolter,6 more . 2016 Production of solid solutions of pesticides by short – term super heating and rapid drying US 9480250 B2. Germany.
[25] Keithd .Wing 2 ,Andrewh. Glickman 3 , and Johne . Casida 4.1983. Phosphorothiolate pesticides and related compounds oxidative bioactivation and aging of the inhibited acetyl cholin esterase. Department of entomological Sciences , University of calisfornia , Berkeley California 94 – 720.
[26] Lee A , Miller , Dayton , Ohio . 1961.Nitrile – ester pesticide . Assignor to Monsanto chem-ical company , st. Louis , mo., acorporation of Dela- ware No Drawing.,
[27] Mitsuru shibata, Sode gaura .,Ichiro Nasuno. Ichihara.,Kazyoshi , Koike,. Masashi Sakamato. 1995.Pyrazole compounds havin gathio chroman ringand pesticides .Containin G.S. UCH Compounds .both of sodegaura all of Japan,Ser.No. 80,027 13 cIaims.(CI. 167-22). United states patent office 3,078 , 210.
[28] R.M.Sacher * and J.F.Olin . 1972. The Insecticidal and Anticholin ester ase Activity of meta- Acylamidophenyl and meta – Thioureido phenyl N- Methyl carbamates .Agricultural Division , Monsanto company , st .Louis , Missouri 63166 – 354,J.agrfod chem.
[29] U.K. Srivaslava , N.T. patel.2012. Pesticides industry in India . Journal of india N- school of poetical economy.
[30] Young TaekHan, Nam-jung kim , jong – Whajung , Hwauoung Yun , sujinlee , and Young – Gersuh . 2011. AVersatile synthetic Approach to Grand is ol Monoter pene pheromone.college of pharmacy seoul National pheromone University ,SEOUL 151-742, Korea
[31] Patterson,G.E.;Guymon,A.J.;Riter,L.S.;Everly,M.;Griep Raming,J.; Laughlin,B.C.; Ouyang,Z.;Cooks,R.G.2002. "Miniature Cylindrical lon Trap Mass spectrometry" Analytical chemistry.74(24) 6145-6153.doi: 10.1021/ ac020494d
[32] Pitarch,E., Lopez, F.J., Serrano, R. and Hernandez, F.(2001). Multiresidue determination of organophosphorus and organochlorine pesticide in human biological fluids by capillary gas chromatography Journal of Analytica; chemistry .369(6):502
[33] S.G.HeuserandK.A.Scudamore.(1970).Selectivedeter mination of ionize bromides in food ,stuffs by gas-liquid chromatography withspecial reference to to fumigant residues . Fumigant residues of bromides in food stuffs33.
[34] John,W.andSons,L.(2003).Determination of residues of Endosulfan in human blood by a negative ion chemical ionization gas chromatographic/mass spectrometric method: Impact of long Term Aerial Spray Exposure.Pest Management Science. 59(3):252-258.