



# Analysis of Residual Pesticides Veterinary Medicines Food Additives

**Shimadzu**

	Component	Molecular weight
1	Metamidophos	141
2	Dichlorvos	220
3	Propamocarb	188
	Acephate	183
	Isoprocarb	193
	Fenobucarb	207
	Ethoprophos	242
	Chlorproham	213
	Endaiaicarb	223
	methipin	210
4C		288
hoate		229
eton		246
		288
		288
		288
		301

	Component	Molecular weight
44	Chinomethionat	234
45	β-Chlorfenvinphos	358
46	Quinalphos	298
47	Phenthoate	320
48	Triadthione	266
	Vamidolthion	284
	Trichlamlide	359
	Methoprene	310
	Glutolani	
	aldrin	
	hiophos	
	butanil	

	Component	Leaver	Kidney	Meat (mustle)
1	Albendazole	5.0		
2	Isometamidium	0.50	5.0	0.10
3	Ivermectin	0.10	1.00	0.10
4	Carbadox			
5	Closantel	1.0	3.0	1.0
6	Diclazuril	0.6	0.3	0.2
7	Spira- mycin	0.10	0.10	0.10
8	Sulfadimidine	0.01		0.002
9	Zeranol	0.10	0.10	0.10
10	Thiabendazole	0.30	0.30	0.20
11	Triclabendazole	0.01		0.002
12	Trenbolone Acetate			
13	Nicarbazin	0.05	0.05	0.05
14	Flubendazole	0.10	0.05	0.02
15	Benzyl-penicillin	2.00	0.30	0.10
16	Moxidectin	2.0	0.0	0.0
17	Eprino- mectin	1.0	0.0	0.0
18	Ceftiofur	1.0	0.0	0.0
19	Tilmicosin			



## Increasing Food Imports to Japan

To ensure the safety of imported food, safety inspections before export and at time of import, as well as sampling inspections at the distribution stage are conducted.

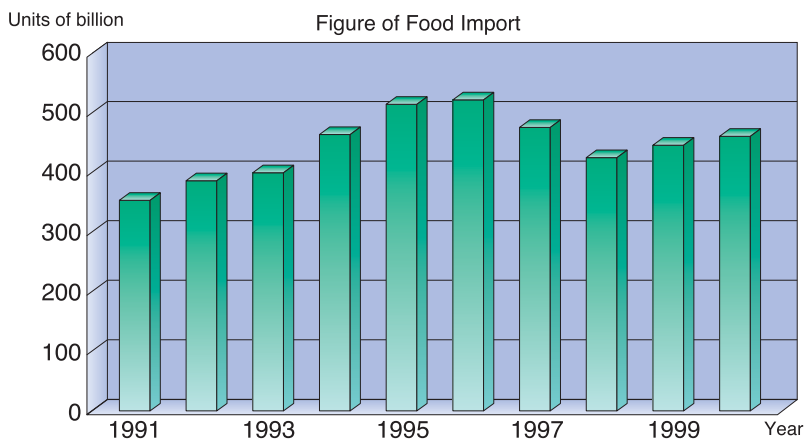
*(The graphs on the right show the transition in figures for Japanese food imports and import market shares held by individual countries.)*

Quickness is an important factor in food inspections to prevent deterioration in freshness and flavor. Especially at the time of import, accurate and prompt inspections are required to determine if the products can be accepted before they enter the domestic distribution network.

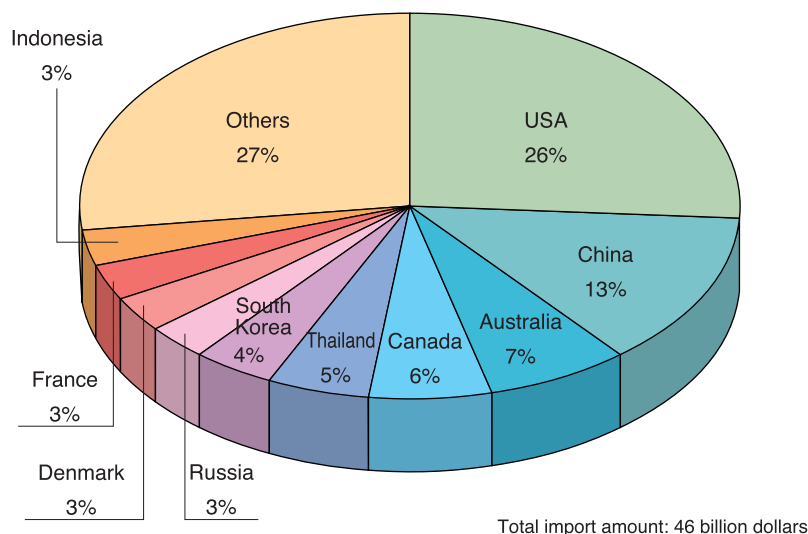
Shimadzu's wide range of analytical instruments including gas chromatographs, gas chromatograph mass spectrometers, high-performance liquid chromatographs and liquid chromatograph mass spectrometers are powerful tools for inspecting residual pesticides, veterinary medicines and additives in food products.

**The excellent performance and quality of Shimadzu analytical instruments respond to the requirements of food safety management.**

Japanese Food Import Transition



Import Market Share by Individual Country (2000)



**Shimadzu Supports  
Food Safety Management**



# Japanese Import Quarantine for Food and Related Products

After the submission of the Notification Form for Importation of Foods, the food sanitation inspector at the quarantine station inspects the product to examine whether the item meets the regulations under the Food Sanitation Law.

During the document examination, the food sanitation inspector validates the following items, based on the information reported in the Notification Form. The judgement will be made based on the information, such as the country of export, imported items, manufacturer, the place of manufacture, ingredients and materials, methods of manufacturing and use of additives.

- Whether the imported food, etc. complies with the manufacturing standards regulated under the Food Sanitation Law.
- Whether the use of additives complies with the standards.
- Whether poisonous or hazardous substance is contained.
- Whether the manufacturer or the place of manufacturing has a record of sanitation problem in the past.

Food products deemed to require a sanitation inspection from the results of the document checks are inspected within the bonded area to determine whether or not importation is permissible. In the case of fresh raw vegetables, tests include those for residual pesticides (including preservatives and coatings), additives (including coloring) and residual radioactivity.

(Taken from the Imported Foods Inspection Services Home Page of the Japanese Ministry of Health, Labour and Welfare)

## Residual Pesticides

As of 13 March 2002, 229 pesticides are regulated under the Japanese Food Sanitation Law, and 200 items will be added to the list in the next three years.

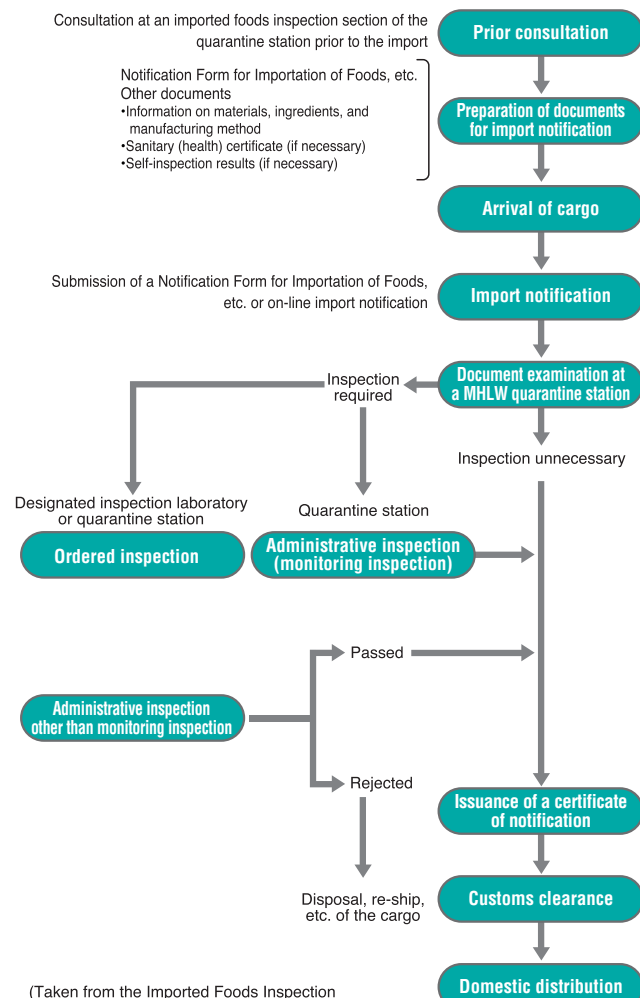
## Veterinary Medicines

In 1995 the Ministry of Health, Labour and Welfare started to establish standards for residual veterinary medicines in food products that have been scientifically proven to have a harmful effect on human health. Currently residue standards have been established for 22 items (6 antibiotics, 2 synthetic anti-bacterial agents, 12 parasite insecticides and 2 hormone agents).

## Food Additives

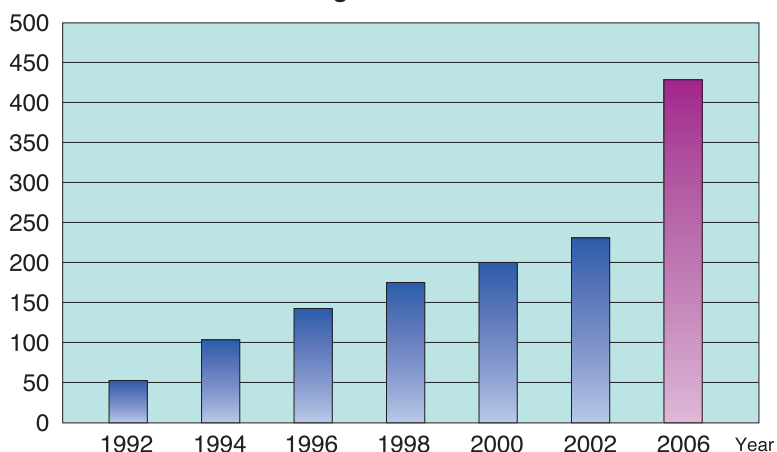
Food additives are items added, mixed, permeated or introduced in other ways to food in the manufacturing process or for the purpose of processing or storage (Clause 2, Article 2 of the Food Sanitation Law). The Ministry of Health, Labour and Welfare has established standard values for 339 items as specified food additives. Apart from specified additives, 489 existing additives, approximately 600 natural seasonings and 100 general beverage additives are allowed to be added to food products.

## Procedures of Import Notification of Foods and Related Products



(Taken from the Imported Foods Inspection Services Home Page of the Japanese Ministry of Health, Labour and Welfare)

## Transition of Regulated Pesticides Count



# Individual and Rapid (Simultaneous) Analyses of Residual Pesticides

The specifications and standards for foods and additives are laid down for each item based on scientific grounds and referring to the opinions of the Japanese Food Sanitation Investigation Council. Opinions from foreign nations are also considered.

For residual pesticides, inspection methods are laid down for individual agricultural and fishery products, as well as for individual pesticides. The Japanese Food Sanitation Law stipulates 125 different analysis methods for 229 pesticides and 262 agricultural products (as of 13 March 2002). On the other hand, with the increase of the number of items to be tested, methods of rapid analysis have been developed for screening.

## Individual analysis (adopted since mid 60s)

A single pesticide or a group of several pesticides with similar properties are handled together. They are analyzed by GC or HPLC after extraction from agricultural products, concentration and cleanup. If components suspected to be pesticides are detected, qualitative analysis using GC/MS or LC/MS is executed for verification. This method has an advantage that step-by-step testing is possible to gradually increase the reliability. However, this method is not suitable for handling the increasing number of pesticides to be analyzed due to the need of complex manual sample pretreatment and long time to obtain the results.

## Rapid analysis (adopted since 1997)

Screening analysis, which allows quick acquisition of test results, has been increasingly adopted to accommodate the increase of imported and processed foods and regulated agricultural chemicals. In rapid analyses, different types of agricultural products and chemicals are processed in the same pretreatment method (extraction, cleanup, etc.) and multiple components are simultaneously analyzed with GC, GC/MS or HPLC. The GPC cleanup method provides partial automation of the pretreatment process. Pesticides are measured by GC-ECD or GC-FPD, depending on their types such as chlorates and phosphates.

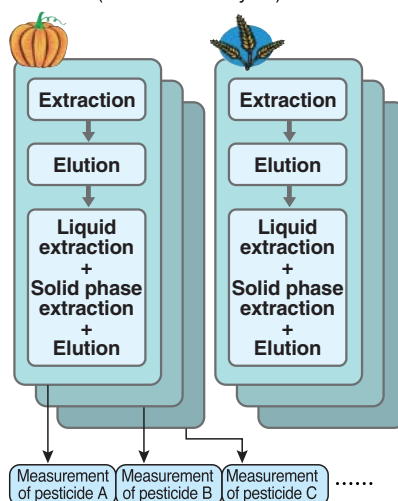
**Shimadzu is continuously working to develop the best method for all processes from sample pretreatment to instrument analysis to ensure rapid and accurate analysis of as many pesticides as possible.**

## (Analysis using Prep-Q)

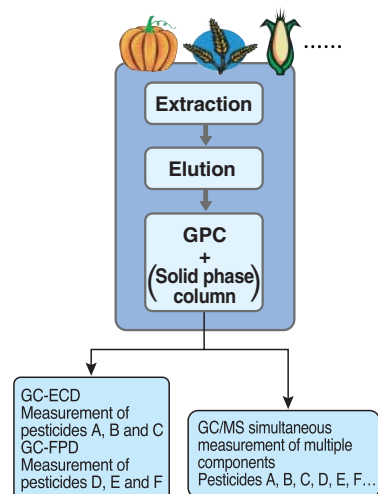
To further simplify the procedures of rapid analysis, Shimadzu has online connected the GPC cleanup system and GC/MS for a higher degree of automation. Faster and simpler screening analysis has been ensured by the automation of concentration and other operations.

## Procedures of Individual and Rapid Analysis Methods

### Conventional method (individual analysis)



### Rapid (simultaneous) analysis



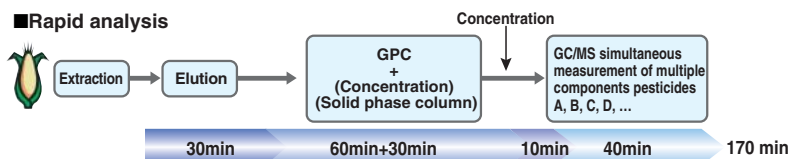
## Comparison of Individual and Rapid Analyses

	Individual analysis	Rapid (simultaneous) analysis
<b>1</b> Development of simple analysis method	<ul style="list-style-type: none"> <li>Difficult to accommodate all regulated pesticides because methods differ depending on pesticides and products.</li> </ul>	<ul style="list-style-type: none"> <li>Applicable to almost all pesticides</li> <li>Simple pretreatment</li> <li>Rapid screening using GC/MS</li> </ul>
<b>2</b> Reduction of toxic organic solvents used	<ul style="list-style-type: none"> <li>Toxic organic solvents used.</li> <li>Large amounts of solvents required for pretreatment and instrument cleaning.</li> </ul>	<ul style="list-style-type: none"> <li>Uses non-halogen solvents</li> </ul>
<b>3</b> Reliability improvement	<ul style="list-style-type: none"> <li>Accuracy control for all analysis methods is difficult due to time and labor required.</li> <li>Numerous manual operations affect accuracy.</li> </ul>	<ul style="list-style-type: none"> <li>Automation of pretreatment procedures reduces errors.</li> </ul>

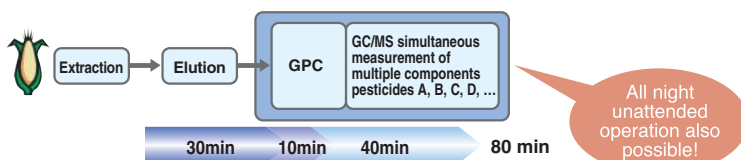
## Aiming for Even Faster Analysis

### Comparison of Online GPC-GC/MS and Rapid Analysis

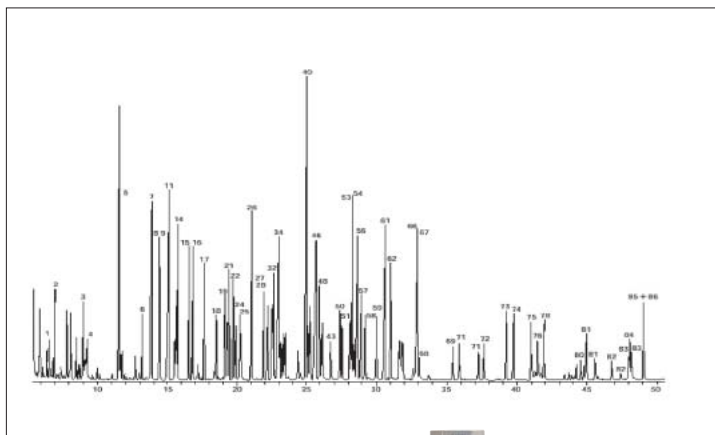
#### ■ Rapid analysis



#### ■ Online GPC-GC/MS method



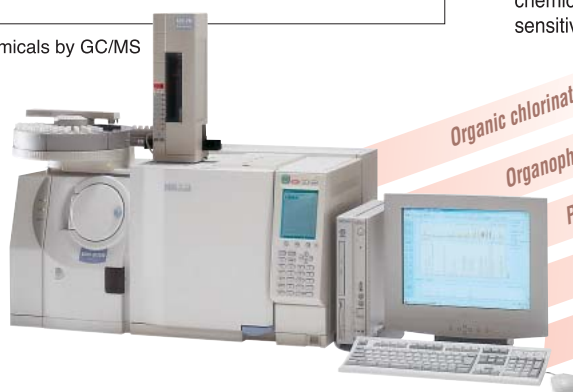
## Gas Chromatograph Mass Spectrometer GCMS-QP2010



Simultaneous analysis of 86 chemicals by GC/MS

GC/MS is an essential instrument for multi-component simultaneous analysis in the screening of residual pesticides and verification tests in the official method. Identification by mass spectrums remarkably improves the accuracy. When analyzing a large number of pesticide components that cannot be handled by the time-consuming individual analysis methods, GC/MS is the most effective approach because it allows simultaneous qualification and quantification of multiple components.

- The high-intensity ion source, low-noise detection unit and dual turbo system of the GCMS-QP2010 ensures high sensitivity.
- The GCMS-QP2010 achieves high reliability with software providing system check and quality control (QA/QC) functions.
- Besides the commonly used EI (electron ionization) method, the GCMS-QP2010 also supports the NCI (negative ion chemical ionization) method to achieve high selectivity and sensitivity for chlorinated and pyrethroid pesticides.

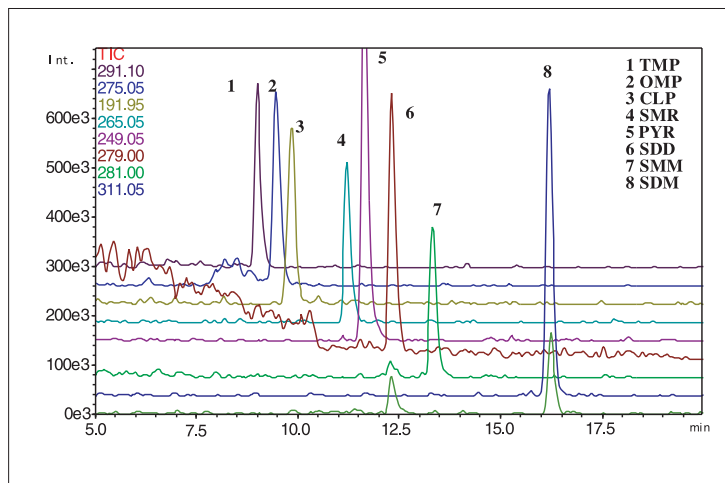


Organic chlorinated pesticides  
Organophosphorus pesticides  
Pyrethroid pesticides  
Nitrogen-containing pesticides  
Carbamate pesticides

## High-performance Liquid Chromatograph Mass Spectrometer LCMS-2010A

LC/MS is used for the verification tests on pesticides and veterinary medicines detected by LC. The highly selective and sensitive LC/MS is used for a wider range of applications in accordance with the increase of components to be analyzed by LC.

- The LCMS-2010A minimizes noise and ensures high stability with the employment of the Angle Spray method.
- The use of drying gas significantly improves sensitivity and stability.
- Maintenance is easy thanks to the use of the metal capillary ESI probe.
- The LCMS-2010A's multi-sequence mode allows LC/MS analyses under multiple conditions (up to 64 patterns) for a single sample injection.



Mass chromatogram of synthetic anti-bacterial agents



Antibiotics  
Parasite insecticides



# Lineup of Shimadzu Products to Support Food Safety Management

## Gas Chromatograph GC-2010



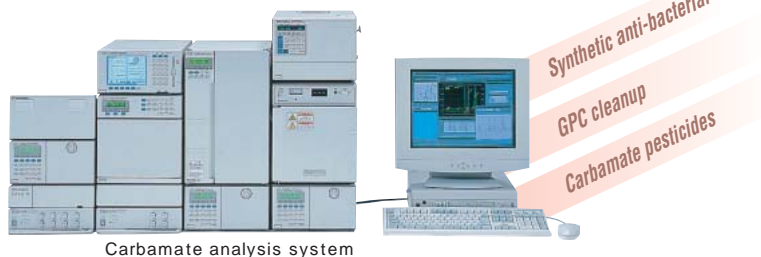
The capillary GC method is widely used in the Japanese official methods for analyzing residual pesticides in food products. This is because capillary GC's advantages such as simple configuration, easy operation, high resolution and the availability of various types of highly selective detectors. GC is also a powerful tool for analyzing organic acids, higher fatty acid compounds, aromatic compounds and fragrances in food additives.

- The GC-2010 is equipped with detectors with improved sensitivity.
- A wide range of detectors are available: ECD for chlorinated and pyrethroid pesticides, FPD for sulfur and phosphorus pesticides and FTD for nitrogen and phosphorus pesticides.
- ECD, FPD and FTD detectors can be attached to the GC-2010 at the same time.
- The GC-2010 is equipped with a third-generation Advanced Flow Controller (AFC) that allows constant linear velocity control at high pressure and flow rate.

## High-performance Liquid Chromatograph LC-VP Series

The Japanese official methods for the analysis of residual pesticides in food products stipulate that pesticides with high polarity and easily decomposed by heat shall be analyzed by LC, as they are difficult to analyze by GC or GC/MS. Most veterinary medicines are also analyzed by LC qualitatively and quantitatively. LC also is indispensable for the analyses of food additives such as vitamins, amino acids, nucleic acids and coloring compounds.

- The Shimadzu LC-VP series is provided with a wide range of highly sensitive detectors capable of detecting micro components.
- A host of peripheral instruments enable construction of various application systems to meet customer demands. Available systems include the carbamate pesticide analysis system, GPC cleanup system and synthetic anti-bacterial agent analysis system.



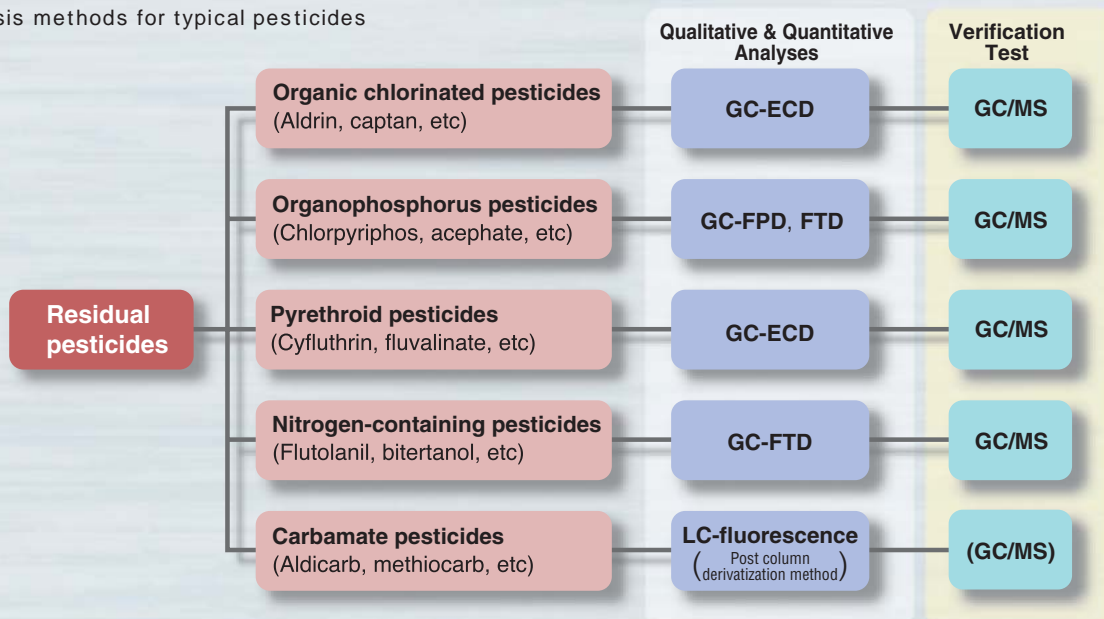
Carbamate analysis system

## Cleanup GPC-GC/MS Prep-Q for the Ana



## Instrument Matrix for the Residual Pesticides and Veterinary Medicines Analysis

\* Analysis methods for typical pesticides



## Atomic Absorption Spectrophotometer AA-6800



Toxic heavy metals

The atomic absorption spectrophotometer is used to measure inorganic compounds used as food additives and highly toxic elements such as As, Pb and Hg.

- Packed with an array of functions including flame/furnace auto switching and two-way background correction (D2 lamp and self-reversal methods).
- In furnace analysis, up to 20 elements can be automatically mixed, diluted and continuously measured by the use of an autosampler.
- The use of a hydrogen compound generator is effective for As analysis.

## Analysis of Residual Pesticides in Food Products

Japanese Ministry of Health and Welfare (now the Ministry of Health, Labour and Welfare) announced in 1997 the development of a rapid analysis method for residual pesticides in food products capable of simultaneous analysis of multiple components in order to improve the efficiency of analysis. This method employs the GPC cleanup method to automate parts of pretreatment procedures.

- The partial amendments to Japanese food standards in 1999 admitted test methods other than those stipulated in the Food Sanitation Law.
- The Prep-Q system combines the GPC cleanup system and GC/MS online to speed up and simplify the procedures of food analysis.
- The Prep-Q system can analyze chlorinated, pyrethroid, organophosphorus, nitrogen-containing and carbamate pesticides in food products (tea decoction not included).

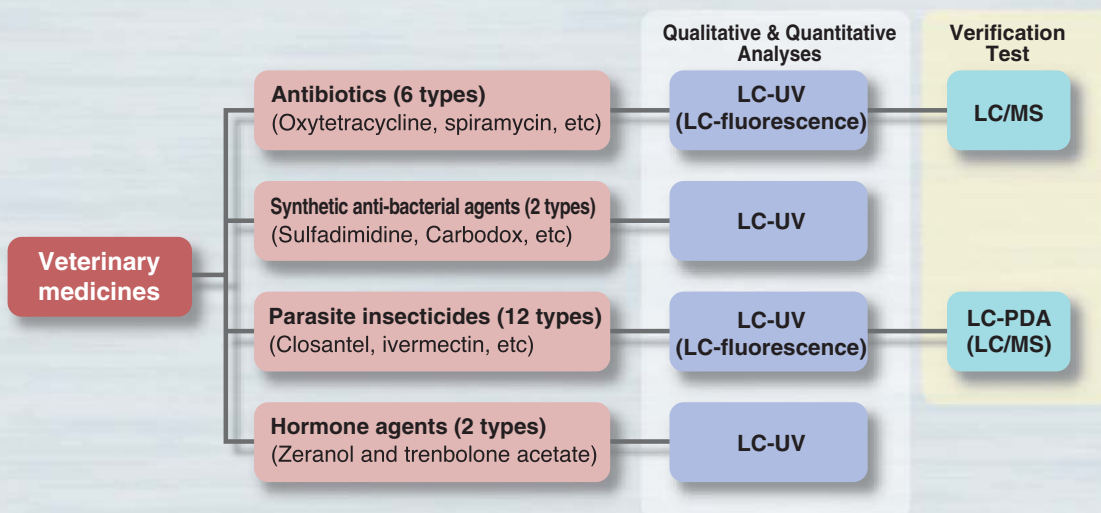
## Inductively Coupled Plasma Mass Spectrometer ICPM-8500



Toxic heavy metals

ICP/MS is a mass spectrometer that uses ICP (Inductively Coupled Plasma) as the ion source. It features high sensitivity, easy qualitative analysis and the possibility of simultaneous analysis of multiple elements and the analysis of isotopes.

- Extremely effective for analyzing highly toxic substances in food such as As, Pb and Cd
- Quantum leap in sensitivity achieved through the use of the mini-torch system
- Highly sensitive analysis of Fe and Se



# Shimadzu Overseas Customer Support

To support customers engaged in the management of food safety, Shimadzu has established a global service network incorporating customer support, training and service centers in the USA, Germany, China and Singapore, as well as in Japan. Shimadzu provides comprehensive support services including instrument maintenance, training workshops and the provision of relevant information to meet customer needs regarding both software and hardware.

- OVERSEAS SUBSIDIARIES
- OVERSEAS OFFICES
- JOINT VENTURES
- CUSTOMER SUPPORT CENTERS



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