National Agrifood Health and Quality Service

BIOLOGICAL AND CHEMICAL TESTING

Resolution 617/2002

Requirements, conditions and procedures for obtaining a technical operating licence for laboratories that have production animal facilities, maintenance and experimentation site. Test report of phytosanitary residues in plant matrices. Field report. Analytical report.

Buenos Aires, 18/7/2002

HAVING SEEN file No. 13.890/2001 issued by the NATIONAL AGRIFOOD HEALTH AND QUALITY SERVICE, Decree No. 1585 issued on December 19, 1996 and modified by its similar No. 394, issued on the 1st of April, 2001, article 2 of Resolution No. 230 issued on March 24, 2000 by the referred National Service, and

CONSIDERING:

That the Directorate of Agrichemicals, Pharmacological and Veterinary Products has summoned an Interdisciplinary Technical Forum of Experts for the exchange of concepts on the conditions that laboratories, in our country, that perform biological and chemical assays in order to produce toxicological, ecotoxicological data and data of pesticide residues in plant and environmental matrices should meet, for registration, renewal, reassessment or monitoring of phytosanitary products.

That the joint meeting of the above-mentioned Forum of Experts and data producing laboratories has concluded satisfactorily and has identified the need to harmonize criteria and work procedures related to toxicological and ecotoxicological tests and pesticides residues tests in plant and environmental matrices, as well as, the specific need to regulate the production, maintenance and use of laboratory animals for the purposes mentioned before.

That the humane and responsible treatment of animals intended to be used in tests to obtain toxicological and ecotoxicological data that are used to assess the safety of phytosanitary and veterinary products before, during and after any import procedure, production or utilization, should be ensured.

That one of the prerequisites for a responsible use of animals is the exhaustive knowledge of the biological characteristics of the species to be used in the studies, as well as the conditions needed for their housing, feeding and care.

That the welfare and health conditions of the animals should be observed by competent persons, understanding that a suitable preparation and training provide the basis needed to

adopt the correct attitude and to evaluate the ethical aspects of the management of the animals involved.

That housing and care standardization of experimental animals is essential for the reliability and reproducibility of the experimental results obtained in toxicological and ecotoxicological studies.

That Resolution No. 350, issued on August 30, 1999, by the ex-Secretariat of AGRICULTURE, LIVESTOCK, FISHING AND FOOD, establishes the requirements for the registration, renewal and reassessment of phytosanitary products, requiring the development of toxicological and ecotoxicological studies in order to quantify the risk of using these substances for human beings and non-target species.

That Resolution No. 230, issued on March 24, 2000 by the NATIONAL AGRIFOOD HEALTH AND QUALITY SERVICE, establishes the need to develop requirements and procedures for the registration of suitable toxicology and ecotoxicology laboratories, being the provision of experimentation animals essential for the good development of this type of studies.

That Law No. 3959, issued by the Animal Health Police, establishes the regulatory framework for health supervision and control of animals that are imported to the country.

That this supervision and control, carried out through technical inspections of laboratories and their animal facilities intended to the production and maintenance of animals for experimentation purposes, constitute a suitable mechanism that assures the results of the studies performed with them.

That the predictability and transparency of the control authority's operation requires establishing technical and procedural requirements for the installation and operation of animal facilities.

That the Directorate of Laboratory and Technical Control, the Directorate of Agrichemicals, Pharmacological and Veterinary Products and the Directorate of Legal Affairs have taken the intervention incumbent on them.

That the undersigned is competent to dictate the present act, by virtue of the provisions given in article 8, bullet m) of Decree No. 1585 issued on December 19, 1996 replaced by its similar Decree No. 394 issued on the 1st of April, 2001.

Therefore,

THE PRESIDENT OF THE NATIONAL AGRIFOOD HEALTH AND QUALITY SERVICE

RESOLVES:

Article 1st —Laboratories that carry out biological studies for the production of toxicological and ecotoxicological data and laboratories that perform analytical determinations of residues of chemical and biological active ingredients in plant and environmental matrices for registration, renewal, reassessment or monitoring purposes of phytosanitary products should register in the NATIONAL AGRIFOOD HEALTH AND QUALITY SERVICE, according to the provisions given in Resolution No. 279, issued on December 20, 1993 by the ex-ARGENTINE INSTITUTE OF PLANT HEALTH AND QUALITY, Resolution No.209, issued on March 8, 1994 by the ex-SECRETARIAT OF AGRICULTURE, LIVESTOCK AND FISHING, Resolution No. 301, issued on July 8, 1994 by the ex-ARGENTINE INSTITUTE OF PLANT HEALTH AND QUALITY, Resolution No. 230, issued on March 24, 2000 by the NATIONAL AGRIFOOD HEALTH AND QUALITY SERVICE and the present Resolution.

Art. 2nd — Biological and residues studies' protocols are those established in chapter 20, clause "List of Regulatory Bodies and Organizations that prepare Tests protocols and Laboratory procedures for Obtaining Data for Registration Purposes" of Resolution No. 350, issued on August 30, 1999 by the ex-SECRETARIAT OF AGRICULTURE, LIVESTOCK, FISHING AND FOOD.

Art. 3rd — The laboratories that perform the studies mentioned in the preceding article should, for their development, meet the following Good Laboratory Practices recommendations of the Organization for Economic Cooperation and Development (OECD):

— General Principles of Good Laboratory Practices (GLP): Nº 45, 1993.

— Quality Assurance and Good Laboratory Practices: Nº 48, 1993.

— Compliance of Laboratory providers with the GLP principles: Nº 49, 1993.

— The application of GLP to short-term studies: Nº 73, 1993.

— The application of GLP to field studies: Nº 50, 1993.

— The role and responsibility of the GLP Study Director: Nº 74, 1993.

Any subsequent amendments to the above-mentioned OECD guidelines and any new OECD guidelines that could be developed related to the pre-existent ones should be automatically applied.

Art. 4th — The final reports of the studies established in article 2 of this resolution should contain the totality of the clauses indicated in the protocols developed, following the relevant guidelines established by the OECD.

Art. 5th — Laboratory animals used in the toxicological and ecotoxicological studies mentioned in article 2 should be bred, maintained and used in accordance with the conditions established in Annex I which is part of this resolution.

The conditions specified in this Annex are applicable to all the species being imported, bred and used in our country in the biological trials mentioned in article 1 of this resolution.

Art. 6th — The import of animals for biological tests remains subject to the provisions established in Law No. 3959, issued by the Sanitary Animal Police, and to the conditions established in this resolution.

Art. 7th — In order to be authorized to operate, applicant laboratories should meet the following requirements:

— The rules mentioned in article 1.

— To have a Chief Technical Director, a duly registered professional linked to the thematic areas of the laboratory.

— To have building and environmental authorizations at municipal, provincial and national level, as appropriate.

— The applicant laboratories will be inspected prior to the issue of the registry by TWO (2) professionals from the NATIONAL AGRIFOOD HEALTH AND QUALITY SERVICE, who may be accompanied by a third professional, external expert.

— The applicant laboratories will be subjected to an interlaboratory trial.

— The expenses demanded by the inspection and remittance of samples will be charged to the laboratory requesting the registration.

— Once the above-mentioned requirements are satisfactorily fulfilled the registration of the applicant laboratory takes place.

— The authorized laboratories will be inspected periodically by professionals of the NATIONAL AGRIFOOD HEALTH AND QUALITY SERVICE under the terms given in Resolution No. 279, issued on March 20, 1993 by the ex- ARGENTINE INSTITUTE OF PLANT HEALTH AND QUALITY.

Art. 8th —The laboratories that carry out analytical determinations of experimental chemical and biological active ingredients' residues or the laboratories already registered that seek an extension of the permit, should previously participate in the interlaboratory trials organized by the General Coordination of Plant Laboratory that reports to the Directorate of Laboratories and Technical Control of this Body.

Art. 9th — The assays of chemical and biological active ingredients' residues mentioned in article 7th, should be framed within the OECD Guideline on the application of GLP to field studies, while the protocol enclosed in Annex II, which is part of this resolution, should be included in the final report.

Art. 10. — The laboratories that carry out studies of acute toxicity by inhalation in rats will need to carry out the validation of the methods used, depending on the equipments they have.

Art. 11. — Data produced by foreign laboratories should fulfill the requirements specified in Resolution No. 239, issued on March 24, 2000 by the NATIONAL AGRIFOOD HEALTH AND QUALITY SERVICE.

Art. 12. — Violators of the rules established in this resolution will be prosecuted as detailed in article 18 of Decree No. 1585, issued on December 19, 1996.

Art. 13. — Be it thus notified, published, forwarded to the National Directorate of the Official Register and filed. — Bernardo G. Cané.

ANNEX I

REQUIREMENTS, CONDITIONS AND PROCEDURES FOR OBTAINING THE TECHNICAL OPERATING LICENCE OF LABORATORIES HAVING PRODUCTION ANIMAL FACILITIES, MAINTENANCE AND EXPERIMENTATION SITE

1 — LOCATION AND GENERAL CHARACTERISTICS OF THE BUILDING

1.1 —The locations for animal production, maintenance and / or experimentation should be separated from the administrative, production and analytical areas.

1.2 —The air conditioning and / or ventilation systems cannot be shared with other areas. They should be exclusive for the animal facility sector.

1.3 — In the construction of locations within the animal facility's zone all those physical factors that could affect the health and quality of the animals should be taken into account.

The locations should be proofed against the entry of wild rodents and insects.

The interior surfaces (walls, soils and ceiling) should be smooth and without cracks, no particles should come off and they should be easy to clean and disinfect.

The outlet grids inside the locations should have safety lids in order to avoid the entry of rodents and insects.

1.4 — The enclosures should be sufficiently spacious so as to enable the workers to perform their tasks comfortably and to avoid animal overload.

1.5 — The location and detailed plan of the premises should be presented along with the general documentation requested to the applicant laboratory.

A — LABORATORIES HAVING PRODUCTION ANIMAL FACILITY, MAINTENANCE AND EXPERIMENTATION SITE

A.1 — PRODUCTION AND MAINTENANCE ZONE:

A.1.1 — Breeding or production location

This location should only be destined to animals in mating or reproduction and their young. The breeding animals should be perfectly identified (tattoo / ring / other according to species); records should be maintained specifying at least the mating system used; date of birth / birth / period of incubation; dates of weaning; number of weaned / separated young and their destination.

A.1.2 — Maintenance or stock location

This location should be destined exclusively to animals kept in maintenance while waiting to be used later. The animals should be perfectly identified, recording at least the spontaneous or by euthanasia deaths, result of the necropsies and probable reason of death, destination of the animals.

When there are insoluble reasons of space or when the amount of animal production does not justify having independent locations, the breeding and maintenance of a species can be done in the same location, providing appropriate separations.

A.1.3 — Animal experimentation location

This location should be destined to house experimentation animals during the course of an experience.

Once the experience is over these animals should be rejected and should not be returned to maintenance rooms in order to be re-used, unless allowed by the characteristics of the study.

A record should be prepared indicating at least the date of initiation and ending of the test, the animals duly identified destined to the test and the final destination of the above mentioned animals.

Different species for reproductive purposes should not be housed in the breeding locations or in the maintenance locations, unless they share similar biological characteristics that do not affect their biological response, but segregation of species should be provided.

A.1.4 — Quarantine Location

This location should be destined to the maintenance of animals housed during a prudential time until possible pathologies are ruled out. A record should be kept of the animals' entry date, diseases, treatments and check-ups performed and their release.

A.2 — STORAGE AREA

A.2.1 — Storage of clean material

Cages, drinking-troughs, aquariums and other implements intended for animal use should be stored.

A.2.2 — Food storage

Food received from the supplier should be adequately stocked up. The warehouse should have an air renovation and conditioning system that ensures that the temperature and humidity are appropriate for the preservation of food.

A.2.3 — Storage of sterile material

Autoclave treated material for animals should be stored.

A.3 — WASHING AREA

This area should be kept apart from the breeding, maintenance and experimentation locations.

B — LABORATORIES THAT DO NOT PRODUCE THEIR OWN ANIMALS

These laboratories should have the same maintenance and experimentation facilities as the above-mentioned laboratories, except for the breeding sector.

2 — GENERAL CONDITIONS RELATED TO THE CLEANING AND MAINTENANCE OF ANIMALS

A daily, general cleaning of locations, corridors, warehouses and other areas related to the animal facility should be done using the detergents and disinfectants registered in SENASA.

No air freshener or other chemical agents should be used to eliminate the smells produced by the animals.

The cleaning of cages and aquariums, the renovation of bedding / water and the removal of excreta should be carried out frequently, so that the accumulation of ammonia and other harmful substances or elements is prevented, maintaining the animals clean and in good sanitary conditions.

The bedding for terrestrial animals should be made of absorbent materials, free from toxic chemical substances that may be harmful for animals and/or interfere with the biological responses. Bedding material that can be easily ingested should be avoided. When wood shavings are used, these should be of white non-resinous wood.

All bedding should be sterilized since it can be contaminated with pathogen germs.

Dirty bedding should be taken out of the cages/aquarium outside the locations containing animals in order to avoid contamination of the environment.

Cages/aquarium should be washed and disinfected before placing clean bedding material.

Water supply for terrestrial animals should be provided daily and feeding should not be restricted unless the assay requires otherwise.

The specific feeding conditions for each species should be known and records of these conditions should be maintained.

3 - FEEDING

Food supplied to animals should meet specific requirements for the correct rearing and maintenance of the species in question, and should be registered in SENASA as food for laboratory animals.

If it is a magistral formula it should meet the requirements established by SENASA for this topic.

The storage of food should be arranged in such a way that food kept in storage for a longer time is consumed first. A record of the entry date and location of the batch should be prepared.

Food should not be older than THREE (3) months.

4 —DISPOSAL OF EXCRETA AND OTHER CONTAMINANTS

The disposal of animal excreta, dead animals, rests of substances used in experiments, bedding and other contaminants that could be generated, should comply with the national, provincial and municipal regulations in force for the disposition of relevant pathological and hazardous waste.

5 — GENETIC QUALITY

The quality and genetic definition of the animal strains used should be accredited. This accreditation should be endorsed by an institution or professional recognized by SENASA.

If the animal facility carries out its own breeding activities, a periodical genetic control that ensures purity is needed.

If the animals are acquired, such accreditation should be required from the vendor.

For toxicological or ecotoxicological routine assays animals captured in nature or stray animals should not be used. If a particular circumstance requires using them, authorization should be requested to the Wildlife Directorate of the Environment and Sustainable Development Secretariat and its rules as well as those established by SENASA should be followed.

6 — HEALTH QUALITY

The health quality of the animals, whether produced or acquired should be accredited through adequate studies that ensure the absence of bacterial, viral or parasitic diseases, clinical or sub-clinical, which could interfere with the experimental results. Such an accreditation should be endorsed by a responsible institution or professional according to the methodology established by SENASA that will define the list of diseases by species that should be controlled and the health control procedure.

7 — PERSONNEL DESTINED TO THE ANIMAL FACILITY

The animal facility should seek the advice of a medical veterinary professional who meets the requirements established by the Department of Education and is duly registered. Likewise, it should have personnel that can demonstrate reliable training in the specific managing of laboratory animals.

8 — GENERAL ENVIRONMENTAL CONDITIONS FOR BREEDING AND MAINTENANCE OF RODENTS AND FISHES

8.1 — AIR

8.1.1 — The injection of air in the rooms should be done at the top angles level of the room and the extraction at the low angles level.

8.1.2 - The interior of the locations should have positive air pressure relative to the corridors and / or exterior areas, which is achieved by an appropriate regulation of the injected and extracted flow.

If the animal facility has a double corridor with central locations (clean and dirty traffic) the gradient of pressure should be from the clean corridor to the soil.

8.1.3 — The animal facility should have air pressure meters inside the locations that enable positive pressure monitoring.

8.1.4 - The air in the animal rooms should not be re-circulated unless harmful or contaminant particles and toxic gases have been eliminated.

8.1.5 - If re-circulating systems are in use, a careful maintenance procedure should be in place involving cleanliness and / or change of filters; the refill or cleanliness dates should be recorded.

8.2 — TEMPERATURE AND HUMIDITY

8.2.1 — The animal facility should have control systems for temperature and relative humidity, using maximum/minimum thermometers and hygrometers in each room. A continuous recording of the variations should be carried out daily.

8.3 — PHOTOPERIOD

8.3.1 —The light should be artificial, provided by fluorescent tubes, type sunlight, with oblique light incidence so that all the cages / aquariums independently of their location, receive similar light intensities. Control of photoperiod should be carried out.

8.3.2 — No external sources of light or noises should exist.

8.4 — WATER

8.4.1 — For the production of fish, in addition, a water temperature monitoring equipment with an automatic alarm system variation of TWO (2 ° C) Celsius degrees should be available.

8.4.2 – At least, twice a year, a quality control of the water used for dilution should be carried out.

9 — GENERAL ENVIRONMENTAL CONDITIONS FOR BREEDING AND MAINTENANCE OF BIRDS AND RABBITS

9.1 — TEMPERATURE AND HUMIDITY

9.1.1 — Control systems of temperature and relative humidity using maximum/minimum thermometers and hygrometers in each sector of the breeding site, should be available

Continuous recording of the variations should be carried out daily.

9.1.2 — During the breeding and maintenance of rabbits no draughts or temperatures above THIRTY DEGREES CELSIUS (30 ° C) should occur. If the rabbits should be transported, the boxes should have sufficient space and good openings for ventilation.

9.1.3 — During the breeding and maintenance of chicken the ventilation needed depends on the number and size of the animals and the room temperature. The ideal temperature for

chicks between ONE (1) AND THREE (3) days is THIRTY FIVE DEGREES CELSIUS (35 ° C), which is reached using heat lamps. Thereafter the temperature should be lowered gradually: during the following FOUR (4) days it should be lowered in ZERO POINT FIVE DEGREES CELSIUS (0,5 ° C) per day and later in ONE DEGREE CELSIUS (1 ° C) per day until EIGHTEEN-TWENTY ONE DEGREES CELSIUS (18-21 ° C) are reached.

10 — ENVIRONMENTAL CONDITIONS FOR EACH SPECIES

SPECIES	Room Temperature (°C)	Relative Humidity (%)	Ventilation (changes/hour)	Max. air velocity (m/s)	Light/darkness cycles (hours)	Light intensity (lux)	MIN. DIMENSIONS FROM GROUND TRAY (cm2)			Tray min. height (cm2)
							Indivually lodged adult (cm2)	Breeder with youngs (cm2)	Group (cm2/adult)	
Rat	20-24	30-70	15-20		12/12	300	350	800	250	14
Mouse	20-24	30-70	15-20		12/12	300	180	200	80	12
Guinea pig	20-24	30-70	15-20		12/12	300	600	1200	1000	18
Rabbit	17-23	30-70	15-20		12/12	300	1kg: 1400 2kg: 2000 3 kg: 2500	1 kg: 3000 3kg: 4000		1-2kg: 30 3kg: 35
Chicken	15-21	40-80	7 kg	20-40	14/10		25-1400		150-650	25-40
Dove	18-20	60-80			14/10		1600			40
Quail	18-23	40-60	34/animal		14/10		350		200	15
Dog	15-21	40-60	20-80				0.75-1.75		1-4	60-180
Cat	15-21	40-60	20-50				0.2-0.6	2	0.2-0.6	50
Pig	17-24	40-60	100-180				0.35-0.8		0.2-2.5	50-80
Sheep Goat	10-24	40-60	100-150				1.4/1.6		0.7/0.8	1200/2000
Squirrel monkey	22-28	40-60			13/11		0.25	0.25	0.25	60
Cynomolgus monkey	20-24	50-70	9-12		13/11		0.7-0.9	0.9	0.7	90
Rhesus monkey	20-24	50-70	9-12		13/11		0.9-1.1	1.1	0.9	90-120
Chimpanzee	20-24	50-70	9-12		13/11		2.5			200

11 — BASIC CONDITIONS TO BE MET BY COLONIES OF BEES DESTINED TO BIOLOGICAL ASSAYS

11.1 — COLONY COMPOSITION

Apis mellifera bees should be used. The colony of bees to be used for a biological assay should be made up of all the members of a colony: working adult bees and nurse bees; ONE queen and depending on the time of the year the presence of drones will or will not be required. The presence of young in all their stages will also depend on the time of the year and the use to which they are destined.

Where a group of bees is used, it should be obtained from colonies with the same characteristics. Bees destined to toxicity studies should be sisters of mother and between TEN (10) AND TWENTY (20) days old. This condition should be certified for each study.

11.2 — ENVIRONMENTAL CONDITIONS AND FEEDING

Free bee collection of pollen is allowed provided that the non-application of agrochemical products in the surrounding zone is ensured.

The beehives should be located in a zone in which the sector intended for the collection of pollen by bees does not have waste materials of any type, whether landfills or industrial premises. The zone should not be an urban zone, except when the beehives are kept under controlled conditions in tents or closed environment of any type.

The colony can be artificially fed with honey of known origin or glucose of maize or syrup of high fructose content, ultimately, with sugar syrup. The protein contribution can be provided by correctly preserved natural pollen or by the administration of pollen supplements whose use and commercialization is authorized by SENASA. The colony should not suffer food shortage ever.

11.3 — COLONY HEALTH

The bees used should come from healthy colonies, with no clinical signs of the bacterial diseases American foulbrood (AFB) and European foulbrood (EFB). With regard to Nosema disease a sample of between THIRTY FIVE (35) and SIXTY (60) adult bees should be examined, preferably taken from the beehive opening and sending it to a laboratory for the diagnosis of apicultural diseases in order to undergo a Nosema disease quantitative test. Irrespective of the time in which the diagnosis is carried out, the level of infection should be equal or a minor to ONE (1) (Rossi-Cornejo quantitative method). If the use of the whole colony should be required, inclusive the young, no mummies should be present, product of the contamination with Ascophaera apis, or clinical signs of any viral disease.

Regarding Varroa disease, the colony must have been treated with a product authorized by SENASA and a period of THIRTY (30) days must have passed from the date the action period of the authorized product is over. In case the colony has not been treated, it should neither present clinical signs of this ectoparasitic disease nor should individuals of Varroa Jacobsoni be visualized. Those beehives that get infected should receive the corresponding treatment and will automatically be rejected as providers of bees intended for tests. Records of detected diseases, quantity of affected beehives and treatments carried out should be maintained.

11.4 — LABORATORIES THAT BREED OR REBREED BEEHIVES FOR THEIR SELF-SUPPLY OF BEES. CONDITIONS APPLICABLE TO PROVIDERS' FACILITIES.

In addition to the environmental conditions inherent to the colony and the sanitary conditions, the laboratories should fulfill the following requirements:

11.4.1 — GENERAL CONDITIONS OF THE BUILDING

Storage room of inert material:

Wood materials, wires, tools for assembling material, wax and any other type of inert material should be retained.

Storage room of contaminant products:

Cleaning products and products used for the sanitary treatment of colonies should be duly identified and stored.

11.4.2 — PRODUCTION AND MAINTENANCE AREA

This area should be divided into a breeding, production and maintenance area that meets the environmental conditions previously specified and an experimentation site that meets the general characteristics established for other species and those applicable to each test according to the international standard used as a reference.

The laboratories that incorporate living apicultural material in order to assign it directly to biological assays should follow the guidelines established in the international standards in accordance with the assay to be performed. They should also require from the supplier a CERTIFICATION stating:

• Number of the provider facility operating licence issued by SENASA

• That the bees come from colonies of Apis mellifera bees.

• That the bees share the same mother ancestry.

• That the colonies that donate individuals or its own colony, when a complete colony is requested, is healthy, without clinical signs of bacterial diseases, with levels equal or less than ONE (1) as results of the quantitative diagnosis of NOSEMA, that they were treated against Varroa mites with a product authorized by SENASA, that they do no show clinical signs of any viral disease.

• That the bees are between TEN (10) and TWENTY (20) days of age.

12 — ENVIRONMENTAL CONDITIONS FOR OTHER SPECIES NOT CONSIDERED IN THESE RULES

Environmental, feeding and health conditions for other species may be established by SENASA at the request of the applicant laboratory, for the experimental purposes specified in Resolution ex-SAGPyA No. 350/99.

ANNEX II

TEST REPORT OF PHYTOSANITARY PRODUCT RESIDUES IN PLANT MATRICES

FIELD REPORT: PART A

RESPONSIBILITY

1 — Year

2 — Test identification or number.

3 — Company or organization. Name and address.

4 — Person (s) in charge (study director, coordinators according to responsibilities such as persons in charge of the study plan, implementation, sampling and remittance of the material, analysis).

IDENTIFICATION AND GENERAL INFORMATION RELATED TO THE ASSAY

1 —Active ingredient(s).

2 — Pesticide class or agricultural use.

3 — Commercial brand or code number.

4 — Formulation (type, concentration in international units).

5 — Crop/agricultural product (type, variety/to cultivate).

6 — Locality (country/region, place or map reference).

7 — Plagues/diseases.

8 — General information concerning the assay.

9 —Plot data (dimension, number of plots per treatment, number of control plots, crop spacing, number of plants per plot, number of rows per plot).

10 — Treatment with pesticides during the year before.

11 — Other pesticides applied to the plot (dose, moment, place).

12 — Cultural treatments (risk/ fertilizers).

13 — Summary of meteorological conditions (before, during and after the application).

APPLICATION DATA

1 — Application method/ equipment.

2 — Dose.

3 — Dilution or concentration of the sprayed product in international system units.

4 — Number of applications.

5 — Date of application.

6 — Development phase when the last treatment was performed (internationally recognized scales).

SAMPLING

1 — Part of the crop where sampling has been performed.

2 — Development phase when sampling.

3 — Sampling method.

4 — Number of samples per plot.

5 — Number of units of the primary sample.

6 — Sample weight.

7 — Dates (sampling, freezing, laboratory reception).

8 — Intervals (last treatment/harvest, sampling/freezing, sampling/laboratory reception).

SAMPLE REMITTANCE AND CONSERVATION

Route map of the samples, indicating cold chain and conservation (temperature, characteristics of the packages), person(s) in charge and dates.

ANALYTICAL REPORT, PART B

1 — Person(s) in charge of the analysis.

SAMPLE IDENTIFICATION

1 — Crop (agricultural product).

2 — Sample identification or number.

3 — Pesticide used.

SAMPLE CONDITION AND TREATMENT

1 — Date of reception by the laboratory.

2 —Date of analysis.

3 — Sample portion to be analyzed.

ANALYSIS – ANALYTICAL REQUIREMENTS FOR THE DETERMINATION OF RESIDUES

Presentation of the analytical method used, including extraction, clean-up and the determination itself.

The methods used should be validated by the laboratory carrying out the work.

The parameters to be included in the validation protocols are: Accuracy, Precision, and Linearity,

Detection limit (LOD) and Quantification limit (LOQ).

The procedure to be used is as follows:

A matrix blank and fortified samples are processed at THREE (3) concentration levels: 0,5 MRL, MRL and 2 MRL each in triplicate (MRL = Maximum Residue Limit, if this limit does not exist, it should be replaced by the expected value; the important thing is that the reported results lay within the validated range).

The following should be determined:

ACCURACY: percentage recuperation for each fortified R%.

PRECISION: Relative Standard deviation for the blank and for each fortified DSR%.

LINEARITY: The correlation coefficient is determined by plotting “Verified concentration vs. Nominal concentration”. (The average percentage recuperation can be determined from the graph slope).

Estimation of LD and LC: To determine these estimates, a graph Response vs. Concentration should be created for each fortified sample; using lineal regression analysis, the ordinate at the origin and its standard deviation can be calculated.

To estimate the LD a value of Response equal to the ordinate at the origin should be considered plus THREE (3) times its standard deviation. The concentration is obtained by interpolating with the straight line. For the LC the same procedure should be followed but adding TEN (10) times the standard deviation.

Once the LD and LC have been estimated they should be confirmed experimentally by fortifying a blank at the LD concentration and verifying that a differing signal from the bottom is obtained and by fortifying blanks in triplicate at the LC concentration and verifying that the accuracy and precision are acceptable.

RECOMMENDED VALUES

The quantification limit of a method should be less than or equal to 50% of the MRL.

A good accuracy and precision are those that generate recuperations in the range of 70 to 110% (with an average recuperation in the range or 80 to 100%) with a Relative Standard Deviation of ± 10%.

Higher Relative Standard Deviations can be accepted as the concentrations decrease.

In trace analysis average recuperation ranges outside the 80 and 100% values are frequently found. Methods that present average recuperations <50% and DSR >30% should not be used.

The correlation coefficient should not be less than 0,975.

RESULTS

1 — Dose

2 —Interval (treatment up to sampling).

3 — Residue and control (including the standard deviation).

4 —Stability of the residues in storage conditions.