

07/2008:20813 during cultivation and after the harvest) of the treatment of the batch is known and can be checked precisely according to good agricultural and collection practice (GACP).

2.8.13. PESTICIDE RESIDUES

Definition. For the purposes of the Pharmacopoeia, a pesticide is any substance or mixture of substances intended for preventing, destroying or controlling any pest, unwanted species of plants or animals causing harm during or otherwise interfering with the production, processing, storage, transport or marketing of herbal drugs. The item includes substances intended for use as growth-regulators, defoliant or desiccants and any substance applied to crops, either before or after harvest, to protect the commodity from deterioration during storage and transport. Pesticide residues can be present and are controlled in herbal drugs and herbal drug preparations.

Limits. Unless otherwise indicated in the monograph, the herbal drug to be examined at least complies with the limits indicated in Table 2.8.13.-1. The limits applying to pesticides that are not listed in Table 2.8.13.-1 and whose presence is suspected for any reason comply with the limits (levels) cross referred to by Regulation (EC) No. 396/2005, including annexes and successive updates. Limits for pesticides that are not listed in Table 2.8.13.-1 nor in European Union texts are calculated using the following expression:

$$\frac{ADI \times M}{MDD_{HD} \times 100}$$

ADI = acceptable daily intake, as published by FAO-WHO, in milligrams per kilogram of body mass,

M = body mass in kilograms (60 kg),

MDD_{HD} = daily dose of the herbal drug, in kilograms.

The limits for pesticides in herbal drug preparations are calculated using the following expressions:

If *DER* ≤ 10:

$$MRL_{HD} \times DER$$

If *DER* > 10:

$$\frac{ADI \times M}{MDD_{HP} \times 100}$$

MRL_{HD} = maximum residue limit of the pesticide in the herbal drug as given in Table 2.8.13.-1 or in EU texts or calculated using the expression mentioned above;

DER = drug/extract ratio, i.e. the ratio between the quantity of herbal drug used in the manufacture of a herbal drug preparation and the quantity of herbal drug preparation obtained;

MDD_{HP} = daily dose of the herbal drug preparation, in kilograms.

The competent authority may grant total or partial exemption from the test when the complete history (nature and quantity of the pesticides used, date of each treatment

Table 2.8.13.-1

Substance	Limit (mg/kg)
Acephate	0.1
Alachlor	0.05
Aldrin and dieldrin (sum of)	0.05
Azinphos-ethyl	0.1
Azinphos-methyl	1
Bromide, inorganic (calculated as bromide ion)	50
Bromophos-ethyl	0.05
Bromophos-methyl	0.05
Brompropylate	3
Chlordane (sum of <i>cis</i> -, <i>trans</i> - and oxychlordane)	0.05
Chlorfenvinphos	0.5
Chlorpyrifos-ethyl	0.2
Chlorpyrifos-methyl	0.1
Chlorthal-dimethyl	0.01
Cyfluthrin (sum of)	0.1
λ-Cyhalothrin	1
Cypermethrin and isomers (sum of)	1
DDT (sum of <i>o,p'</i> -DDE, <i>p,p'</i> -DDE, <i>o,p'</i> -DDT, <i>p,p'</i> -DDT, <i>o,p'</i> -TDE and <i>p,p'</i> -TDE)	1
Deltamethrin	0.5
Diazinon	0.5
Dichlofluanid	0.1
Dichlorvos	1
Dicofol	0.5
Dimethoate and omethoate (sum of)	0.1
Dithiocarbamates (expressed as CS ₂)	2
Endosulfan (sum of isomers and endosulfan sulphate)	3
Endrin	0.05
Ethion	2
Etrimphos	0.05
Fenchlorophos (sum of fenchlorophos and fenchlorophos-oxon)	0.1
Fenitrothion	0.5
Fenpropathrin	0.03
Fensulfothion (sum of fensulfothion, fensulfothion-oxon, fensulfothion-oxonsulfon and fensulfothion-sulfon)	0.05
Fenthion (sum of fenthion, fenthion-oxon, fenthion-oxon-sulfon, fenthion-oxon-sulfoxid, fenthion-sulfon and fenthion-sulfoxid)	0.05
Fenvalerate	1.5
Flucytrinate	0.05
τ-Fluvalinate	0.05
Fonophos	0.05
Heptachlor (sum of heptachlor, <i>cis</i> -heptachlorepoxyde and <i>trans</i> -heptachlorepoxyde)	0.05
Hexachlorbenzene	0.1
Hexachlorocyclohexane (sum of isomers α-, β-, δ- and ε)	0.3

Substance	Limit (mg/kg)
Lindan (γ -hexachlorocyclohexane)	0.6
Malathion and malaoxon (sum of)	1
Mecarbam	0.05
Methacriphos	0.05
Methamidophos	0.05
Methidathion	0.2
Methoxychlor	0.05
Mirex	0.01
Monocrotophos	0.1
Parathion-ethyl and Paraoxon-ethyl (sum of)	0.5
Parathion-methyl and Paraoxon-methyl (sum of)	0.2
Pendimethalin	0.1
Pentachloranisol	0.01
Permethrin and isomers (sum of)	1
Phosalone	0.1
Phosmet	0.05
Piperonyl butoxide	3
Pirimiphos-ethyl	0.05
Pirimiphos-methyl (sum of pirimiphos-methyl and <i>N</i> -desethyl-pirimiphos-methyl)	4
Procymidone	0.1
Profenophos	0.1
Prothiophos	0.05
Pyrethrum (sum of cinerin I, cinerin II, jasmolin I, jasmolin II, pyrethrin I and pyrethrin II)	3
Quinalphos	0.05
Quintozene (sum of quintozene, pentachloraniline and methyl pentachlorophenyl sulfide)	1
S-421	0.02
Tecnazene	0.05
Tetradifon	0.3
Vinclozolin	0.4

Sampling of herbal drugs. Sampling is done according to the general chapter 2.8.20.

Herbal drugs: sampling and sample preparation.

Qualitative and quantitative analysis of pesticide residues.

The analytical procedures used are validated (e.g. according to Document N° SANCO/10232/2006). In particular, they satisfy the following criteria:

- the chosen method, especially the purification steps, is suitable for the combination pesticide residue/substance to be examined, and not susceptible to interference from co-extractives;
- natural occurrence of some constituents is considered in the interpretation of results (e.g. disulfide from Cruciferaeae);
- the concentration of test and reference solutions and the setting of the apparatus are such that the responses used for quantification of the pesticide residues are within the dynamic range of the detector. Test solutions containing pesticide residues at a level outside the dynamic range, may be diluted within the calibration range, provided that the concentration of the matrix in the solution is adjusted in the case where the calibration solutions must be matrix-matched;
- between 70 per cent to 110 per cent of each pesticide is recovered;
- repeatability of the method: RSD is not greater than the values indicated in Table 2.8.13.-2;
- reproducibility of the method: RSD is not greater than the values indicated in Table 2.8.13.-2.

Table 2.8.13.-2

Concentration range of the pesticide (mg/kg)	Repeatability (RSD) (per cent)	Reproducibility (RSD) (per cent)
0.001 - 0.01	30	60
> 0.01 - 0.1	20	40
> 0.1 - 1	15	30
> 1	10	20