Bull Environ Contam Toxicol (2011) 86:571–575
DOI 10.1007/s00128-011-0284-1

# Parameter Validation of Analytical Methods of Insecticide Residue Analyses in Foods of Animal Origin, Feed and Water

**Amsalu Debebe · S. Kuttalam**

Received: 1 November 2010 / Accepted: 15 April 2011 / Published online: 29 April 2011


**Abstract** The study was conducted to examine the interrelationship and coherence of analytical parameters in method validation. Recovery, sensitivity, linearity, precision and limits of detection (LOD) were tested in six methods for organochlorine and organophosphate insecticides. Compounds that fell out of the stipulated recovery, 70–120%, in a matrix have concurrently failed to meet the requirements for sensitivity ($\geq$0.7), linearity ($R^2 > 0.99$) and precision (<0.2) in the same matrix. Highest LOD was recorded in those compounds and matrices. Different from the conventional point estimate, a new approach was introduced for setting upper and lower confidence limits of the LOD in quantitative analyses.

**Keywords** Analytical methods · Parameters · LOD

Public concern for food safety increases as the quality of life improves. Foods of animal origin are prone to contaminants, including insecticides (Kan and Meijer 2007). Insecticide residues in food can be extracted and analyzed for their true identity and quantity. The reliability of analytical methods to provide accuracy and precision has to be demonstrated (Hill and Reynolds 1999). Parameters of validation and their coherence are subjects of controversy. However, accuracy, sensitivity, precision and detection limits are considered essential (Hill and Reynolds 1999; MacNeil et al. 2000; SANCO 2007). The purpose of this study was to investigate the interrelationship and coherence among these parameters and to provide a simplified approach for determining detection limits.

A. Debebe (✉)
Department of Biology, Faculty of Science, University of Gondar, PO Box 196, Gondar, Ethiopia
e-mail: amsaludebebe@yahoo.com

S. Kuttalam
Department of Agricultural Entomology, Tamil Nadu Agricultural University, Coimbatore, TN 641003, India

## Materials and Methods

Seventeen organochlorines and fifteen organophosphates were studied in food matrices of animal origin: egg, milk, fish, mutton, feed and water. An inventory of the analytical procedures was carried out, and for each matrix six methods (Sharma 2007) have been chosen for their comprehensiveness, commonness, and capacity for eluting the studied insecticides. Certified reference materials with 95 to 100 percent purity were used from 'AccuStandard' for each group of insecticides. The primary stock, intermediate working and working solutions were prepared in 1,000, 100 and 10 mg $kg^{-1}$, respectively, in 9:1 ratio (v/v) of hexane and acetone. All have been kept in a refrigerator at 4°C and brought to room temperature before use. A concentration of 0.01 mg $kg^{-1}$ was injected to gas chromatography for retention time and sensitivity.

An auto-system gas chromatography equipped with $^{63}Ni$ electron capture detector (ECD), flame thermo ionization detector (FTD), split injector and DB-5 capillary column with a 30 m length was used. The inner column diameter and film thickness were 0.25 mm and 0.25 μm, respectively, at a maximum usable temperature of 325°C. The standards were injected and similar conditions were opted for spiked matrices. The identity was compared with the retention time of standard peaks of every batch in order to meet the same conditioning of the instrument. Identity was confirmed in GCMS, and quantity was calculated (Sharma 2007).

Sampling was made per the recommendation of Codex Alimentary Commission (CAC 1999). The protocols of extraction were followed (Sharma 2007) and florosil clean up was done except for water. Recovery, sensitivity, precision, linearity, limits of detection and quantitation were studied in all matrices in organochlorines. Fish and mutton were excluded in organophosphates due to their improbable residues.

Recovery as a measure of accuracy for trueness (Barwick and Ellison 1999) was determined from the spiking of four levels of concentration: 0.01, 0.05, 0.1 and 0.5 mg $kg^{-1}$ to the matrices before extraction, except for water which was fortified with 0.1, 0.5, 1 and 5 μg $L^{-1}$. Blank matrices were simultaneously extracted and analyzed as a control, while each set of the experiment was replicated four times.

The calibration curve concept was adopted to determine the limits of detection (LOD) and quantitation (LOQ). For a given spiked level (x), a corresponding concentration (y) was obtained. The slope (b) and the intercept (a) were computed. The paired values in the plane were represented by a regression line; $Y = a + bx$. The LOD and LOQ values were computed (DFG 1992).

Sensitivity was measured by the slope of the regression line at ≥0.7. Precision or reproducibility was estimated from the coefficient of variation (CV) of the variances in the signal values from the replications of a series of concentrations. Linearity, estimated by the coefficient of determination ($R^2$), of signal values (x) and the corresponding changes in concentration (y) were used to prove the capacity to produce test results that were proportional to changing concentration.

## Results and Discussion

The recovery of studied compounds fell in the stipulated range, 70–120 percent (AOAC/FAO/IAEA/IUPAC 1999; Quevauviller and Morabito 2000; SANCO 2007), except: endrin aldehyde (69.4), endosulfan sulphate (68.1), methoxychlor (68.6), dichlorovos (59) and dimethoate (66.5) in egg; dichlorovos (60) in water; and dichlorovos (69.4) and aldrin (121.8) in CSK, cotton seed cake (Table 1).

Most of the compounds that fell out of the recovery range also failed to meet the required sensitivity or b-values ≥0.7. Endrin aldehyde, endosulfan sulphate, methoxychlor, dichlorovos and dimethoate in egg had 0.55, 0.46, 0.49, 0.44 and 0.43 sensitivities, respectively (Table 2). Likewise, a lower sensitivity was recorded from a water sample in dichlorovos (Table 2). However, the low sensitivity (69) which was very close to the stipulated range was recorded in water and cotton seed cake samples from delta HCH and endrin aldehyde, respectively. Neither of these compounds showed a low recovery in these samples. The slope of the calibration curve proved to serve as an estimate of sensitivity (DFG 1992; Quevauviller and Morabito, 2000). The coherence of sensitivity and recovery was reported (DFG 1992; Quevauviller and Morabito 2000).

**Table 1** Mean recovery of organochlorines and organophosphates in studied matrices (%)

| Compound | Egg | Milk | Water | Fish | Meat | CSK |
|---|---|---|---|---|---|---|
| Alpha HCH | 97.7 | 89.9 | 76.5 | 75.5 | 83.8 | 89.0 |
| Gamma HCH | 94.4 | 76.9 | 82.8 | 79.1 | 75.8 | 91.1 |
| Beta HCH | 94.6 | 100.6 | 93.0 | 82.4 | 90.3 | 88.4 |
| Delta HCH | 86.7 | 74.6 | 80.2 | 77.0 | 73.2 | 91.5 |
| Heptachlor | 78.2 | 75.9 | 89.9 | 88.9 | 74.3 | 81.6 |
| Aldrin | 76.9 | 91.9 | 91.9 | 76.5 | 77.0 | 121.8 |
| Heptachlor epoxide | 79.5 | 82.0 | 98.2 | 91.1 | 83.3 | 89.9 |
| Alpha endosulfan | 79.3 | 86.8 | 99.4 | 83.6 | 88.0 | 86.6 |
| *p,p′*-DDE | 76.3 | 82.6 | 100.5 | 80.7 | 86.9 | 82.7 |
| Dieldrin | 92.6 | 93.3 | 91.4 | 87.0 | 78.8 | 99.4 |
| Endrin | 85.2 | 92.5 | 86.3 | 80.4 | 82.3 | 89.3 |
| Beta endosulfan | 79.1 | 76.9 | 88.5 | 75.5 | 80.8 | 84.4 |
| *p,p′*-DDD | 89.6 | 76.6 | 77.9 | 86.8 | 78.0 | 85.7 |
| Endrin aldehyde | 69.4 | 86.8 | 80.1 | 85.2 | 79.3 | 84.9 |
| Endosulfan sulphate | 68.1 | 81.3 | 93.7 | 80.7 | 73.5 | 88.2 |
| *p,p′*-DDT | 93.4 | 73.5 | 79.2 | 74.8 | 83.8 | 79.6 |
| Methoxychlor | 68.6 | 79.1 | 70.5 | 78.1 | 72.3 | 86.9 |
| Dichlorvos | 59.0 | 81.8 | 60.0 | – | – | 69.4 |
| Acephate | 81.6 | 83.2 | 82.8 | – | – | 80.7 |
| Phorate | 82.5 | 81.9 | 78.3 | – | – | 81.0 |
| Monocrotophos | 80.1 | 84.3 | 84.2 | – | – | 82.9 |
| Dimethoate | 66.5 | 76.1 | 84.7 | – | – | 78.7 |
| Phosalone | 81.0 | 85.7 | 77.5 | – | – | 82.2 |
| Phosphamidon | 86.9 | 85.9 | 82.9 | – | – | 80.2 |
| Chlorpyrifos-methyl | 85.5 | 85.2 | 80.4 | – | – | 83.2 |
| Malathion | 82.6 | 80.7 | 91.7 | – | – | 75.7 |
| Phorate sulfone | 80.8 | 76.1 | 81.9 | – | – | 79.5 |
| Chlorpyrifos | 81.7 | 83.4 | 82.0 | – | – | 80.1 |
| Quinalphos | 77.8 | 83.2 | 78.0 | – | – | 76.4 |
| Profenofos | 78.6 | 84.0 | 84.5 | – | – | 80.9 |
| Ethion | 79.1 | 90.0 | 75.3 | – | – | 76.8 |
| Triazophos | 88.6 | 81.6 | 86.4 | – | – | 79.3 |

The coefficient of determination, $R^2 > 0.99$ serves as a proof of linearity (Rodrigues et al. 2007; Caldas et al. 2009). In this study, $R^2$ was almost one except in those compounds that failed to meet the recovery and sensitivity ranges. Endosulfan sulphate, monocrotophos and dimethoate in egg had 0.988, 0.988 and 0.955 $R^2$ values, respectively and 0.981 $R^2$ for aldrin in a cotton seed cake (Table 2). Linearity is supposed to fit the working range of

**Table 2** Sensitivity (b), linearity ($R^2$) and precision (CV) of organochlorine and organophosphate mixes in studied matrices

| Compound | Egg | | | Milk | | | Water | | | Fish | | | Mutton | | | CSK | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | b | $R^2$ | CV | b | $R^2$ | CV | b | $R^2$ | CV | b | $R^2$ | CV | b | $R^2$ | CV | b | $R^2$ | CV |
| Alpha HCH | .74 | .998 | .09 | .89 | 1 | .17 | .77 | 1 | .2 | .82 | 1 | .13 | .72 | .998 | .16 | .86 | 1 | .16 |
| Gamma HCH | .74 | .997 | .12 | .77 | 1 | .12 | .72 | .999 | .18 | .78 | 1 | .11 | .73 | 1 | .18 | .81 | 1 | .13 |
| Beta HCH | .98 | 1 | .19 | .93 | .999 | .1 | .84 | 1 | .25 | .93 | .999 | .2 | .82 | .999 | .2 | .89 | 1 | .18 |
| Delta HCH | .7 | 1 | .17 | .71 | 1 | .18 | .69 | 1 | .16 | .82 | 1 | .15 | .7 | .999 | .15 | .74 | .997 | .19 |
| Heptachlor | .74 | 1 | .2 | .74 | 1 | .19 | .79 | .999 | .21 | .76 | .996 | .16 | .72 | .999 | .18 | .76 | .999 | .2 |
| Aldrin | .75 | 1 | .16 | .81 | .999 | .13 | .99 | .997 | .34 | .71 | .999 | .07 | .7 | .996 | .17 | .84 | .981 | .15 |
| Heptachlor epoxide | .7 | .999 | .16 | .76 | .999 | .2 | .74 | .991 | .19 | .99 | 1 | .2 | .72 | 1 | .18 | .84 | .999 | .15 |
| Alpha endosulfan | .72 | 1 | .18 | .91 | .999 | .09 | .84 | .999 | .25 | .7 | .993 | .11 | .86 | .999 | .2 | .91 | 1 | .19 |
| *P,P′*-DDE | .77 | 1 | .10 | .85 | 1 | .15 | .96 | .998 | .32 | .71 | 1 | .09 | .75 | .992 | .19 | .78 | 1 | .17 |
| Dieldrin | .79 | .999 | .20 | .79 | .997 | .2 | .89 | .999 | .27 | .92 | 1 | .14 | .82 | .999 | .2 | .99 | 1 | .14 |
| Endrin | .75 | .999 | .19 | .84 | .996 | .09 | .86 | .999 | .25 | .84 | 1 | .15 | .71 | .998 | .17 | .95 | 1 | .11 |
| Beta endosulfan | .72 | .998 | .18 | .73 | 1 | .19 | .93 | 1 | .3 | .74 | 1 | .19 | .7 | .998 | .17 | .77 | 1 | .12 |
| *P,P′*-DDD | .7 | .994 | .16 | .75 | 1 | .2 | .71 | .999 | .17 | .87 | 1 | .12 | .71 | .998 | .17 | .75 | .999 | .2 |
| Endrin aldehyde | .55 | .994 | .2 | .77 | .999 | .11 | .71 | .998 | .17 | .79 | 1 | .12 | .75 | 1 | .19 | .69 | .998 | .2 |
| Endosulfan sulphate | .46 | .988 | .19 | .8 | 1 | .2 | .86 | .993 | .25 | .85 | 1 | .2 | .73 | 1 | .18 | .81 | 1 | .13 |
| *P,P′*-DDT | .93 | 1 | .13 | .72 | 1 | .16 | .84 | .999 | .25 | .74 | 1 | .19 | .77 | 1 | .2 | .82 | 1 | .14 |
| Methoxychlor | .49 | .99 | .19 | .83 | 1 | .14 | .72 | .999 | .18 | .76 | 1 | .1 | .7 | 1 | .17 | .82 | .999 | .2 |
| Dichlorovos | .44 | .998 | .17 | .76 | 1 | .12 | .46 | .999 | .07 | | – | – | | – | – | .7 | 1 | .17 |
| Acephate | .71 | 1 | .18 | .75 | 1 | .18 | .75 | .999 | .19 | | – | – | | – | – | .72 | .999 | .18 |
| Phorate | .75 | .999 | .20 | .86 | .999 | .2 | .72 | 1 | .18 | | – | – | | – | – | .74 | 1 | .19 |
| Monocrotophos | .7 | .988 | .10 | .73 | .998 | .18 | .86 | 1 | .25 | | – | – | | – | – | .74 | .999 | .19 |
| Dimethoate | .43 | .985 | .17 | .74 | .999 | .12 | .88 | 1 | .27 | | – | – | | – | – | .74 | 1 | .19 |
| Phosalone | .83 | 1 | .02 | .84 | 1 | .13 | .73 | 1 | .18 | | – | – | | – | – | .73 | 1 | .18 |
| Phosphamidon | .78 | .999 | .02 | .81 | .999 | .11 | .78 | 1 | .21 | | – | – | | – | – | .75 | .999 | .16 |
| Chlorpyrifos-methyl | .73 | .997 | .09 | .78 | .995 | .17 | .72 | .999 | .18 | | – | – | | – | – | .72 | 1 | .18 |
| Malathion | .71 | .997 | .02 | .77 | 1 | .07 | .94 | .999 | .31 | | – | – | | – | – | .76 | 1 | .2 |
| Phorate sulfone | .74 | 1 | .19 | .74 | 1 | .19 | .78 | 1 | .21 | | – | – | | – | – | .72 | 1 | .18 |
| Chlorpyrifos | .71 | .997 | .18 | .8 | .999 | .2 | .81 | 1 | .23 | | – | – | | – | – | .78 | 1 | .2 |
| Quinalphos | .72 | 1 | .18 | .76 | .999 | .15 | .71 | .999 | .17 | | – | – | | – | – | .72 | 1 | .18 |
| Profenofos | .75 | 1 | .02 | .86 | 1 | .09 | .8 | 1 | .22 | | – | – | | – | – | .75 | .999 | .17 |
| Ethion | .74 | 1 | .19 | .76 | .997 | .12 | .75 | 1 | .19 | | – | – | | – | – | .73 | 1 | .18 |
| Triazophos | .77 | .999 | .02 | .8 | 1 | .2 | .8 | 1 | .22 | | – | – | | – | – | .75 | 1 | .2 |

recovery (SANCO 2007). The linearity of a series of concentrations to the corresponding signal helped to show the lowest recovered concentration (Barwick and Ellison 1999; SANCO 2007).

Precision can be checked from the replicates of the recovery experiment (SANCO 2007) to indicate the agreement among sets of replicates and it is estimated by the coefficient of variation (DFG 1992; SANCO 2007). Its acceptable limit which is <0.2 can rise up to 0.35 in very low concentrations (AOAC/FAO/IAEA/IUPAC 1999). In this study, the coefficient of variation was consistently less than 0.2 in all the compounds and the matrices other than water in which the coefficient of variation was recorded as high as 0.34 (Table 2). Hence, the precision was good enough to witness the minimum variation of the variance of the signal values.

In this study, a new approach was introduced for establishing the upper and lower confidence limits for LOD. Unlike qualitative methods in which binary responses are anticipated and are valuable, the probabilities of committing $\alpha$-type and $\beta$-type errors for detection limit can be applicable to quantitative determinations (Trullols et al. 2004). The LOD value is numerical in quantitative analysis and remains to be the reference value for subsequent studies. So, there could be freedom of determination from intervals within which the population would lie with a

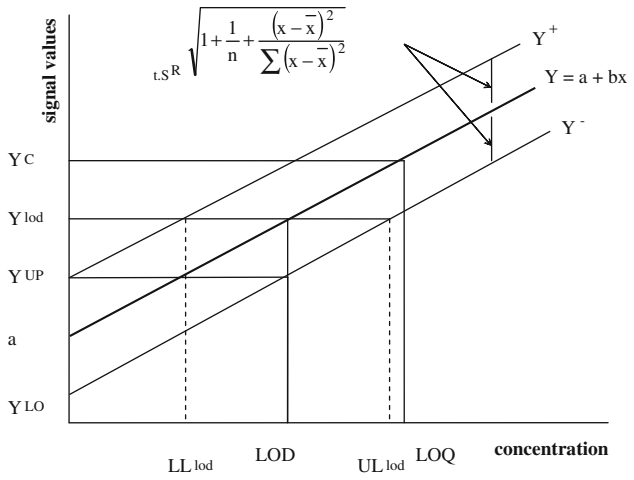


**Fig. 1** Graphic determination of LOD, LOQ, $LL_{LOD}$ and $UL_{LOD}$ (DFG 1992)

specified degree of confidence (IUPAC 1995; MacNeil et al. 2000). In other words, the LOD value in a study cannot always be maintained as a fixed value or point estimate for subsequent studies as there will be from sample-to-sample variations due to errors.

The calibration curve (Fig. 1) was designated by $Y = a + bx$. The upper and lower limit curves, in which the signal values at any concentration of X are presumed to fall at the level of significance chosen, were designated as $Y^+$ and $Y^-$, respectively. The points $Y_{UP}$ and $Y_{LO}$, where the curves for $Y^+$ and $Y^-$ intersect, have indicated the confidence interval for yielded signal values by samples with nil content (DFG 1992). The intercepts can be calculated from $Y^+$ and $Y^-$ curves. The limit of detection can be derived from the calibration curve and is "represented by the smallest value $X_{LDC}$ for which the confidence intervals of the corresponding signal value $Y_{LDC}$ and of the signal value for a 'nil' content do not overlap" (DFG 1992). After the LOD was established at the abscissa, the corresponding signal value was located at the ordinate. This $Y_{LOD}$ value which was greater than $Y_{UP}$ yielded three possible intersection points on the calibration curve, $Y^+$ and $Y^-$ curves. The corresponding values of these intersection points in the abscissa were found to be the upper limit ($UL_{LOD}$) and lower limit ($LL_{LOD}$) when they intersected at $Y^+$ and $Y^-$ curves, respectively. The third intersection, at the $Y = a + bx$, was the LOD (Fig. 1).

The LOD gave rise to the $Y_{lod}$ estimate of the line at $Y = a + bx$. The upper and lower limits of signal values ($Y^+$and $Y^-$) were designated by the equation. The upper and lower limits ($UL_{lod}$ and $LL_{lod}$) on the abscissa, shown by the broken lines, did not exceed the LOQ. The exact values were computed mathematically;

$$\bar{x} - \frac{b}{c}(\bar{y} - y_{LOD}) \pm \frac{t \cdot S_R}{C}\sqrt{(1+\frac{1}{n})C + \frac{(y_{LOD} - \bar{y})^2}{(K)}} \quad (1)$$

The LOQ at the line can be found from $Y_c$, which is a calculated one (DFG 1992);

$$LOQ = \frac{(y_c - a)}{b},$$
$$\text{where:} y_c = \bar{y} + b(LOD - \bar{x}) \pm t \cdot S_R \cdot \sqrt{1 + \frac{1}{n} + \frac{(LOD - \bar{x})^2}{(K)}} \quad (2)$$

In this study, the LOD and LOQ were mathematically computed (DFG 1992), and the lower and upper limits of LOD were computed as in equation1 (Table 3). Very low LOD values were recorded except in those compounds and matrices that failed to meet the required range of recovery, sensitivity, and linearity. Endosulfan sulfate, methoxychlor, monocrotophos and dimethoate in egg had LOD values of 0.095, 0.085, 0.095 and 0.104 mg $kg^{-1}$, respectively. A high LOD, 0.118 mg $kg^{-1}$, was recorded in aldrin from a cotton seed cake (Table 3). The upper limit of the LOD might have approached the LOQ values but never crossed the latter (Table 3).

**Table 3** LOD, lower and upper limits of LOD and LOQ values of organochlorine and organophosphate mixes in studied matrices*

| Compound | Egg (mg $kg^{-1}$) | | Milk (mg $kg^{-1}$) | | Water (μg $L^{-1}$) | | Fish (mg $kg^{-1}$) | | Mutton (mg $kg^{-1}$) | | CSK (mg $kg^{-1}$) | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | LOD± | LOQ | LOD± | LOQ | LOD± | LOQ | LOD± | LOQ | LOD± | LOQ | LOD± | LOQ |
| Alpha HCH | .042±.021 | .064 | .005±.002 | .008 | .088±.044 | .133 | .016±.008 | .025 | .036±.018 | .055 | .017±.008 | .026 |
| Gamma HCH | .048±.024 | .072 | .002±.001 | .003 | .194±.097 | .292 | .009±.004 | .014 | .008±.004 | .013 | .009±.004 | .014 |
| Beta HCH | .008±.004 | .013 | .028±.014 | .042 | .154±.077 | .232 | .021±.011 | .032 | .024±.012 | .037 | .014±.007 | .022 |
| Delta HCH | .008±.004 | .012 | .016±.008 | .024 | .091±.045 | .137 | .019±.009 | .029 | .02±.010 | .030 | .046±.023 | .07 |
| Heptachlor | .015±.008 | .023 | .004±.002 | .007 | .271±.135 | .406 | .054±.027 | .081 | .02±.010 | .031 | .024±.012 | .037 |
| Aldrin | .018±.009 | .027 | .025±.012 | .038 | .433±.216 | .649 | .023±.011 | .035 | .053±.026 | .079 | .118±.059 | .178 |
| Heptachlor epoxide | .023±.011 | .035 | .032±.016 | .048 | .822±.411 | 1.23 | .009±.004 | .014 | .005±.002 | .009 | .032±.016 | .049 |
| Alpha endosulfan | .006±.003 | .009 | .023±.011 | .035 | .297±.148 | .445 | .073±.036 | .11 | .021±.01 | .032 | .012±.006 | .019 |
| *P,P′*-DDE | .016±.008 | .024 | .009±.004 | .014 | .368±.184 | .552 | .004±.002 | .007 | .076±.038 | .115 | .013±.006 | .02 |
| Dieldrin | .022±.011 | .033 | .045±.022 | .068 | .279±.139 | .418 | .014±,007 | .022 | .024±.012 | .037 | .014±.007 | .022 |

**Table 3** continued

| Compound | Egg (mg kg$^{-1}$) | | Milk (mg kg$^{-1}$) | | Water (μg L$^{-1}$) | | Fish (mg kg$^{-1}$) | | Mutton (mg kg$^{-1}$) | | CSK (mg kg$^{-1}$) | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | LOD± | LOQ | LOD± | LOQ | LOD± | LOQ | LOD± | LOQ | LOD± | LOQ | LOD± | LOQ |
| Endrin | .031±.016 | .047 | .05±.025 | .076 | .243±.121 | .364 | .003±.001 | .005 | .036±.018 | .055 | .003±.002 | .006 |
| Beta endosulfan | .042±.021 | .064 | .008±.004 | .013 | .034±.017 | .052 | .005±.002 | .008 | .037±.018 | .056 | .016±.008 | .025 |
| *P,P′*-DDD | .064±.032 | .097 | .003±.002 | .006 | .213±.106 | .32 | .01±.005 | .015 | .037±.018 | .056 | .021±.01 | .032 |
| Endrin aldehyde | .064±.032 | .097 | .024±.012 | .037 | .343±.171 | .514 | .017±.008 | .026 | .017±.009 | .027 | .04±.02 | .06 |
| Endosulfan sulphate | .095±.047 | .143 | .009±.005 | .015 | .696±.348 | 1.04 | .012±.006 | .018 | .008±.004 | .012 | .007±.003 | .012 |
| *P,P′*-DDT | .009±.005 | .014 | .003±.002 | .005 | .311±.155 | .467 | .006±.003 | .01 | .017±.009 | .027 | .017±.008 | .026 |
| Methoxychlor | .085±.043 | .128 | .013±.006 | .02 | .193±.096 | .29 | .009±.004 | .014 | .007±.003 | .011 | .026±.013 | .04 |
| Dichlorovos | .04±.02 | .06 | .011±.006 | .017 | .222±.111 | .333 | – | – | – | – | .005±.002 | .008 |
| Acephate | .018±.009 | .027 | .013±.006 | .02 | .293±.146 | .44 | – | – | – | – | .019±.01 | .029 |
| Phorate | .019±.01 | .029 | .02±.01 | .031 | .161±.081 | .243 | – | – | – | – | .011±.005 | .017 |
| Monocrotophos | .095±.047 | .142 | .035±.017 | .053 | .072±.036 | .109 | – | – | – | – | .02±.01 | .031 |
| Dimethoate | .104±.052 | .156 | .023±.011 | .035 | .176±.088 | .265 | – | – | – | – | .016±.008 | .025 |
| Phosalone | .012±.006 | .019 | .014±.007 | .022 | .170±.085 | .255 | – | – | – | – | .004±.002 | .007 |
| Phosphamidon | .024±.012 | .037 | .03±.015 | .046 | .125±.062 | .188 | – | – | – | – | .021±.01 | .032 |
| Chlorpyrifos–methyl | .045±.023 | .068 | .06±.03 | .09 | .234±.117 | .351 | – | – | – | – | .01±.005 | .015 |
| Malathion | .049±.024 | .074 | .009±.005 | .015 | .199±.099 | .299 | – | – | – | – | .01±.005 | .015 |
| Phorate sulfone | .006±.003 | .009 | .008±.004 | .012 | .136±.068 | .205 | – | – | – | – | .014±.007 | .021 |
| Chlorpyrifos | .049±.025 | .074 | .02±.01 | .03 | .126±.063 | .190 | – | – | – | – | .014±.007 | .022 |
| Quinalphos | .008±.004 | .012 | .031±.016 | .048 | .196±.098 | .295 | – | – | – | – | .004±.002 | .007 |
| Profenofos | .005±.002 | .008 | .008±.004 | .012 | .081±.04 | .122 | – | – | – | – | .025±.013 | .039 |
| Ethion | .007±.004 | .012 | .05±.025 | .076 | .115±.058 | .174 | – | – | – | – | .012±.006 | .018 |
| Triazophos | .024±.012 | .037 | .012±.01 | .019 | .099±.049 | .149 | – | – | – | – | .007±.003 | .011 |

* The values have been shrank to 3 digits

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