

# Analysis of the Distribution of DDT Residues in Soils of the Macintyre and Gwydir Valleys of New South Wales, Australia, Using ELISA

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## Abstract

DDT (1,1,1-trichloro-2,2-bis(4-chlorophenyl ethane)) is a persistent environmentally toxic organochlorine insecticide. It has been applied for many decades in countries around the world and its indiscriminate use and long persistence in soil have resulted in extensive soil contamination with DDT and its metabolites. Conventional gas chromatographic analytical techniques for the detection of these residues in soil are expensive and time consuming. An ELISA method was therefore developed and used to monitor DDT/DDE residues in soil samples collected from the Macintyre and Gwydir valleys of northern New South Wales, Australia. DDT was used extensively in this region particularly for protection of cotton crops, until its use was banned in 1982. The main DDT residue observed after several years in these soils when analysed by gas chromatography is DDE, in contrast to the soils at the more heavily contaminated dip sites used to control cattle tick, where DDT itself is the major residue.

Samples of soil predominantly grey-cracking clay (Vertisols) were collected from 120 sites in the Macintyre Valley and 150 sites in the Gwydir Valley. These sites were selected to cover a variety of past land uses for comparative purposes as part of a more extensive soil survey using geographical information system (GIS) techniques. Extraction with 90% methanol was found to be efficient and quantitative, using assay conditions specially developed to avoid interferences from the soil matrix. Of the samples collected in both the Macintyre and Gwydir valleys to 0–10 cm depth, more than half contained residues at a concentration in the range of 0.01–0.75 mg/kg (ppm). Soil from sites showing positive analysis in the 0–10 cm soil layer was also analysed at greater depth (10–20 cm). No positive analysis for DDE were observed with 10–20 cm samples from the Macintyre Valley whereas some contamination at 10–20 cm depth was observed in samples from the Gwydir Valley where cotton was grown more extensively before the 1982 ban.

This study indicates widespread persistence of DDT residues even though DDT has not been applied for over 15 years. Analysis of the distribution of DDE residues indicates that contamination is mainly in the surface layer where there may still be some potential for impacts on surface biota, crops or grazing stock. Surprisingly, soil from cotton farms in the Macintyre Valley was found by a statistical analysis to be less contaminated with DDE than was soil from nearby native pastures. However, analysis of soils in the Gwydir Valley indicated less difference between various usage patterns.

In many developed countries, the use of DDT has been banned for several years. Recognised as one of the more persistent organochlorines, there is never-

theless scant evidence about the extent of contamination that remains in soil more than 15 years after the previous application, although DDE in particular has been observed as a degradation product of DDT with particularly long persistence (Agarwal et al. 1994). This short paper describes a survey of the Macintyre and Gwydir valleys of New South Wales (NSW), Australia for DDE residues in a range of soil types and land uses including cotton growing with inten-

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sive DDT application in the past. Analyses for a third valley, the Namoi, are expected to be completed later. This study was conducted to determine the current extent of contamination with DDE, to examine whether there is a need for remediation and to suggest practical means of achieving it.

## Materials and Methods

### Soil sampling

In the river valleys studied (Fig. 1), the dominant soils are Vertisols (Soil Survey Staff 1992) characterised by a large proportion of clay. These Vertisols vary from grey to black and brown clays. Red and yellow brown earth areas are intersected with Vertisols in the valleys. Sample sites were randomly located based on a stratified simple random scheme, with 120 sites sampled in the Macintyre Valley (Fig. 2) and 153 in the Gwydir Valley (Fig. 3). The scheme allowed for preferential sampling in such a way that the sites cover a variety of land use types—ranging from cultivated to stock routes, native pastures and woodland (McGarry et al. 1989; Odeh and McBratney 1994). Samples were taken with cores divided into 10 cm depth sections. For the purpose of this study on DDE, only the 0–10 and 10–20 cm sections were analysed. Soils were air-dried, ground in a mill, and sieved of large particles before storage at room temperatures. For incurred residues of a highly immobile chemical of extremely low volatility and long half-life, such routine soil preparation would not be regarded as a potential source of loss of residues. Soil

samples for volatile chemicals such as endosulfan could not be treated in this fashion without losses.

### Materials for DDE analysis by ELISA

Bovine serum albumin (BSA) and horseradish peroxidase (HRP) were purchased from Boehringer-Mannheim, Germany. Fish skin gelatin (FG) and Tween 20 were obtained from Sigma Chemicals, St Louis, USA. Methanol (AR grade) was obtained from Ajax Chemicals, Clyde, NSW, Australia. Maxicorp polystyrene 96-microwell plates were purchased from Nunc, Roskilde, Denmark.

### Preparation of DDE standard

DDE (100 mg/L, ppm) was prepared in methanol as a stock solution. From this stock, a 1000 µg/L, ppb standard was prepared by dilution in 0.1% FG-phosphate buffered saline (PBS, 50 mM sodium phosphate/0.9% NaCl, pH 7.2) and then serially diluted to obtain 200, 40, 20, 4.2 and 0 ppb in borosilicate tubes for the standard curve. The standard curves for soil analysis were prepared in using an extract of soil diluted 1:20 in 0.1% FGPBS.

### Soil spiking

The Vertisol soil sample used for spiking was established as free from pesticide residue by solvent extraction followed by gas-liquid chromatographic (GLC) analysis. Ten gram sub-samples of this soil were distributed into glass jars (with aluminum lined caps) and spiked with 0.5, 1, 2, 5 and 10 mg/kg (ppm) concentrations of DDE.

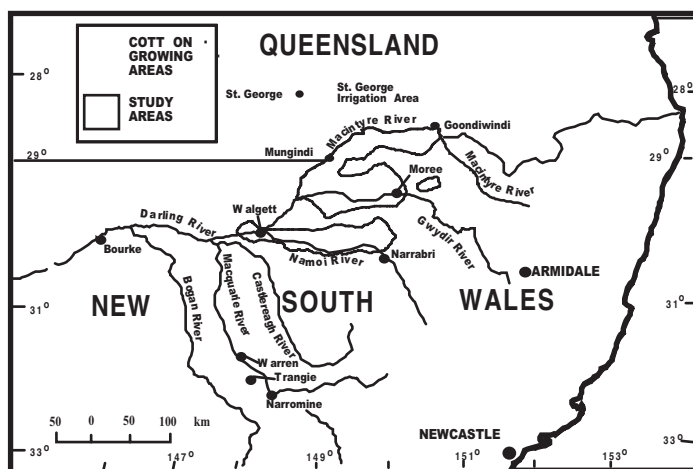
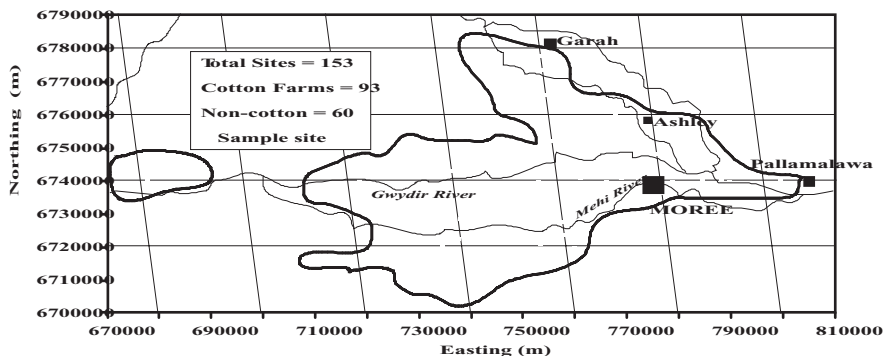
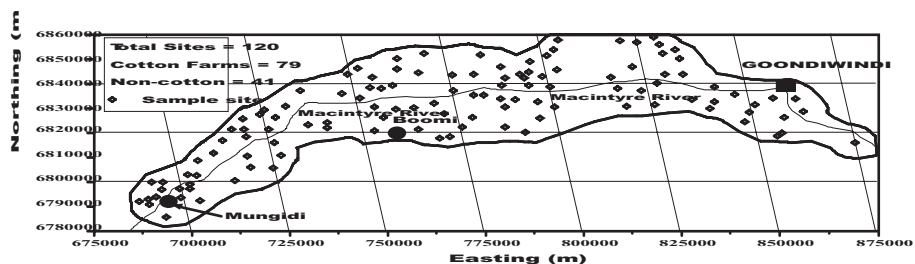


Figure 1. River systems of northern New South Wales, showing the Macintyre and Gwydir valleys



**Figure 2.** Gwydir Valley sampling sites. A large number of sites representing various land uses were selected and sampled to depths of 0–10 cm and 10–20 cm.



**Figure 3.** Macintyre Valley sampling sites. Numerous sites representing various land uses were selected and sampled to depths of 0–10 cm and 10–20 cm.

The soil was mixed thoroughly with a stainless steel spatula for 5 minutes and allowed to stand at room temperature for 3 days. The soil samples were extracted using 25 mL of 90% methanol by shaking for one hour in the original glass jars. Extraction of DDE from soil with 90% methanol was shown to be efficient by the use of spiked soil.

### ELISA–laboratory microwell assays

The assay for *p,p'*-DDE was obtained (Larkin et al. 1994; Beasley et al. 1998) using an immunogen which includes all elements of the DDE structure, except that one of the *p*-chloro groups was replaced by  $\beta$ -alanine carboxamide for coupling to carrier proteins. The same hapten was used for coupling to horseradish peroxidase (HRP). Antibodies to protein conjugates of this hapten exhibited specificity for *p,p'*-DDE (100%) over *p,p'*-DDT (7%). Cross-reactivity (%  $\times$ ) is calculated as the concentration of analyte that causes a reduction of 50% in the assay colour relative to a pesticide-free control ( $IC_{50}$ ), expressed as a percentage of the  $IC_{50}$  of the cross-reaction compound. The DDE

assay showed preferential recognition of the DDE and DDD compounds and were 10–80 times less sensitive for the *p,p'*-DDT parent compound. Thiobencarb, a herbicide used in rice cultivation, was detected only at very high levels (greater than 1000  $\mu\text{g/L}$ ) by the DDE assay (0.4% cross-reaction). Dicofol and methoxychlor were not cross-reactive (Beasley et al. 1998).

Antibodies were diluted in 50 mM carbonate buffer pH 0.6 to 10  $\mu\text{g/mL}$  and were coated at 100  $\mu\text{L}$  per well overnight at 20°C. The microwells were washed twice with PBS (50 mM sodium phosphate/0.9% NaCl, pH 7.2) containing 0.05% (v/v) Tween 20 (PBS/T) and then blocked with 150  $\mu\text{L}$  of BSA/PBS (1% bovine serum albumin) in PBS. One hundred  $\mu\text{L}$  of DDE standard or sample followed by 100  $\mu\text{L}$  of peroxide conjugate diluted in PBS containing 0.5% (w/v) fish skin gelatin (Sigma) was incubated for 1 hour at 20°C. Hydrogen peroxide substrate/ chromogen (3,3',5,5' tetra methyle benzidine/hydrogen peroxide in acetate buffer, pH 5.5) 150  $\mu\text{L}$  was added and incubated 30 minutes at 20°C. Colour development was stopped by adding 50  $\mu\text{L}$  of 1.25 M sulfuric acid and the colour

intensity was read at 450 nm. For control and blanks distilled water and solvent were used. The  $IC_{50}$ , minimum detectable limit, and also percent recovery were calculated from the standard graph.

### Soil extraction for GLC

For instrumental analysis, 50 g of soil was weighed into a stoppered conical flask and 150 mL of 90% methanol was added. The flasks were shaken for 12 hours and the solvent was filtered through paper containing 2 g of anhydrous sodium sulfate. The filtrate was concentrated to 5 mL using a Kuderna-danish apparatus and then chromatographed on a Florisil column. The column was eluted with 150 mL of hexane: diethyl ether 3:1. The first 10 mL of elute was discarded and remainder concentrated to 5 mL. GLC analysis was performed using a Hewlett Packard 5890 Series II gas chromatograph equipped with an electron capture detector.

### Immunoassay

Ten g soil of well-mixed soil was weighed into a 100 mL glass jar and 25 mL of 90% methanol was added. The jars were shaken for 1 hour and allowed to stand overnight until the particles settled. The supernatant was then collected and typically diluted 1:20 in water for ELISA.

### Statistical analysis

Statistical analysis of residue data was accomplished by analysis of variance (ANOVA) to establish

significant differences, using the JMP program to plot the range and mean values for different land uses.

## Results and Discussion

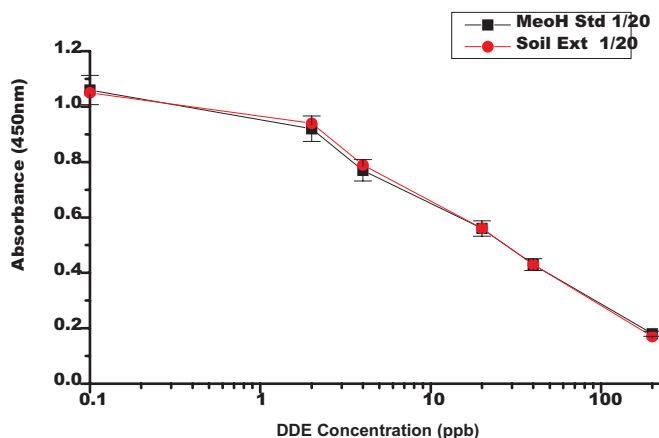
The standard graph for DDE analysis by ELISA is shown in Figure 4. Using standard concentrations of DDE dissolved in methanol, analyses were performed in the range 2 to 200 ppb ( $\mu\text{g/L}$ ) with an  $IC_{50}$  of 20 ppb. Since the DDE was to be detected in soil extract, a standard graph was also prepared using pesticide-free soil extract. Initial experiments indicated 1:10 dilution in 0.1% FG-PBS showed matrix interference in ELISA as indicated by elevated  $IC_{50}$  values. This could be overcome by diluting 1:20 in 0.1% FG-PBS.

### Spike and recovery test

DDE after extraction with methanol and dilution in 1:20 in FG-PBS was analysed by ELISA and the recovery data were comparable with values obtained from GLC analysis (Table 1). The data shown in Figure 5 indicate the recovery values calculated by the ELISA method were comparable to that of GLC values. For both methods, a recovery of more than 95% was achieved compared with the amount spiked.

### DDE residues in the Macintyre and Gwydir valley soil samples

When the top layer (0–10 cm) of the soil was analysed, DDE was detected in samples from 65 of 120 sites



**Figure 4.** Standard curve for DDE in methanol and in methanol soil extract using pesticide free soil. Standard errors are shown as bars; the value indicated as 0.1 ppb was the zero level absorbance.

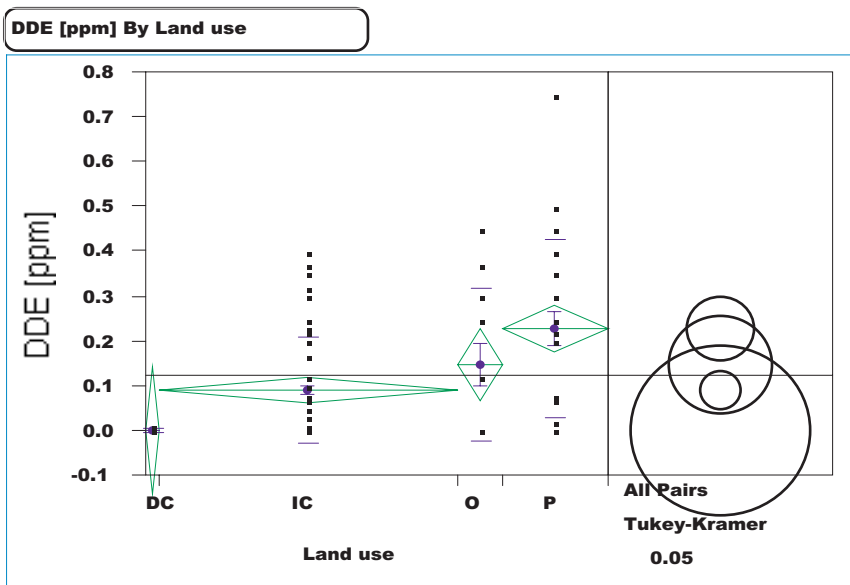
in the Macintyre Valley and samples from 97 of 151 sites from the Gwydir Valley (see Figs 2 and 3 for sampling sites). The concentration ranged from <0.01–0.40 ppm and <0.01–0.75 ppm in the Macintyre and Gwydir valleys soil samples, respectively. The soil samples analysed were classified as dryland (rainfed) cotton (DC),

irrigated cotton (IC), other crops (O), native pastures (P), stock routes (SR), and woodland (WL). The relative concentration in dryland cotton soil samples, on which DDT may never have been sprayed, was very low as compared with irrigated cotton, when compared using analysis of variance in the JMP program (Tables 2 and 3).

**Table 1.** Comparison of ELISA versus gas–liquid chromatographic analysis data of spike and recovery of DDT from soil

| Concentration spiked (ppb) | Mean ELISA recovery (ppb) | SD ELISA | Mean GC recovery (ppb) | SD GC | % ELISA recovery | %GC recovery |
|----------------------------|---------------------------|----------|------------------------|-------|------------------|--------------|
| 100                        | 92.5                      | 3.5      | 86.0                   | 1.4   | 92.5             | 86.0         |
| 200                        | 194.5                     | 7.8      | 210.5                  | 14.9  | 97.3             | 105.3        |
| 500                        | 425.0                     | 35.4     | 507.5                  | 10.6  | 85.0             | 101.5        |
| 1000                       | 992.5                     | 10.6     | 1004.0                 | 5.7   | 99.3             | 100.4        |
| 2000                       | 1570.0                    | 99.0     | 2002.5                 | 3.5   | 78.5             | 100.1        |
| 4000                       | 4025.0                    | 35.4     | 4001.0                 | 1.4   | 100.6            | 100.0        |
| 10000                      | 9982.0                    | 25.5     | 9795.0                 | 106.1 | 99.8             | 98.0         |

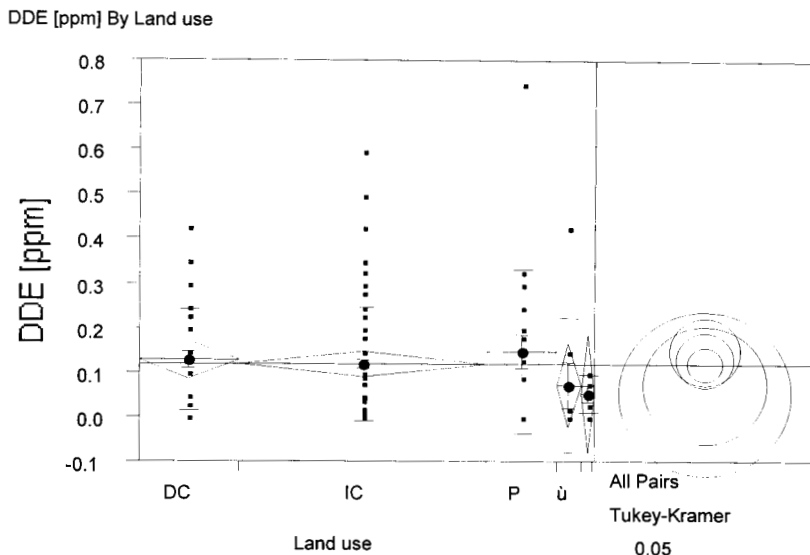
**Table 2.** Statistical analysis of Macintyre Valley soil samples



Land use key: DC = dryland cotton      O = other crops  
 IC = irrigated cotton                  P = pastures

ANOVA in the JMP program indicates a significant difference between residues in pastures and soils for cotton. Absence of overlap of circles indicates significant differences ( $P < 0.05$ , Tukey–Kramer method)

**Table 3.** Statistical analysis of Gwydir Valley soil samples



Land use key: DC = dryland cotton      SR = stock routes  
 IC = irrigated cotton                  WL = woodland  
 P = pastures

ANOVA in the JMP program indicates a significant difference between residues in pastures and soils for cotton. Absence of overlap of circles indicates significant differences ( $P < 0.05$ , Tukey–Kramer method)

However, soils with pasture contained higher levels of DDE residues (40 ppb) in the Macintyre Valley. The highest concentration was recorded in soil of other crops such as legumes and cereals. Similarly, the relative concentration in irrigated cotton soil samples was higher than in dryland cotton soil samples but the highest concentration was recorded in native pastures near cotton farms. Possibly, more extensive cultivation and consistent watering may result in a long-term remediating affect in cotton soils. This could result from accelerated biodegradation or by erosion in run-off. The analysis of the soil samples at lower depth in all the samples revealed absence of DDE residue in the Macintyre Valley. No relationship between the crop cover on the soil and DDE residue concentrations was observed.

In the Gwydir Valley, differences between soils with different land uses were less pronounced (Table 3). Possibly, this indicates that greater usage of DDT in the Gwydir Valley, where cotton was more exten-

sively grown when DDT was in use, may have resulted in a more even distribution and a greater amount of stored DDE. The concentration of residues ranged from 0.01–75 ppm. Whereas no soil samples in the Macintyre Valley below 10 cm depth contained residues, 12 samples of the Gwydir Valley at 10–20 cm depth were slightly contaminated.

The study indicates that the pesticide is still widely present in the soils of the study area even though DDT has not been in use since 1981, confirming the long persistence of its residues noted elsewhere (Samuel et al. 1988; Wan et al. 1989; Kausik 1991; Martijn et al. 1993; Agarwal et al. 1994; Boul 1995). Most of the residues were recovered in topsoil, indicating the absence of extensive leaching. Water movement in cotton soils is mainly lateral, during flood irrigation and in run-off from storms, these soil being regarded as having low hydraulic conductivity and low recharge of groundwater. Absence of DDT residues in the lower layers of soil indicated no downward movement of DDT in soil.

## Conclusion

The results of this study indicate widespread persistence of DDT residues as DDE in these cotton growing valleys in New South Wales even though DDT has not been applied for 15 years. Analysis of the distribution of DDE residues indicates that contamination is mainly in the surface layer where there may still be some potential for impacts on surface biota, crops or grazing stock. Management practices capable of reducing such impacts for such organochlorine residues have been discussed (McDougall et al. 1995). Surprisingly, soil from cotton farms in the Macintyre Valley was found by a statistical analysis to be less contaminated with DDE than soil from nearby native pastures. However, analysis of soils in the Gwydir Valley indicated less difference between various usage patterns.

## Acknowledgments

H.M. Shivaramaiah wishes to acknowledge the receipt of an AusAID PhD Fellowship from the Australian Government. The consistent support of Dr V. Prakash, Director of the CFTRI Mysore, India and of Dr N.G.K. Karanth is also gratefully acknowledged.

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# Concentrations of Pesticides and the $\alpha/\gamma$ HCH Ratio in Gas and Particle Phases in Air of Alsace, Eastern France)

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## Abstract

Eleven pesticides (HCB,  $\alpha$ -HCH,  $\gamma$ -HCH, trifluraline, mecoprop, phosalone, atrazine, carbofuran, carbaryl, diuron and isoproturon) were determined in atmospheric samples which were collected in the Alsace region of eastern France. Three stations were chosen: in an urban area (Strasbourg), in a rural area (Colmar), and in a remote area (Aubure).

Pesticides samples were collected during summers of 1993 and 1994, using Hi-Vol sampler fitted with Whatman filter paper and XAD-2 resin. The particle and gas phases were collected separately during 24 hours. After extraction, samples were fractionated using normal phase high performance liquid chromatography (HPLC), and three fractions were collected. Organochlorine and trifluraline pesticides (fraction 1) were analysed by gas chromatography-electron capture detection, and others pesticides (fraction 2 and 3) were analysed by reverse phase HPLC-UV.

The concentrations of pesticides in the particle phase were lower than those in the gas phase. For some organochlorines, concentrations were higher in Aubure samples than in Colmar and Strasbourg samples.

Only the  $\gamma$ -HCH isomer is still used in western Europe. It could be isomerised to  $\alpha$ -HCH by photochemical reaction in the atmosphere.

The  $\alpha$ -HCH/ $\gamma$ -HCH ratio varied from 0.06 to 2.8 and only 2 samples had a  $\alpha$ -HCH/ $\gamma$ -HCH ratio greater than 1. These values indicate the utilisation of pure  $\gamma$ -HCH, that HCH isomers generally came from countries around sampling stations and that therefore there may have been insufficient time for the  $\gamma$  form to isomerise to the  $\alpha$  form.

SYNTHETIC organic pesticides have been used since World War II and the introduction of DDT which was used to control of malaria and typhus. The use of such pesticides has become more and more common, especially in agriculture for crops protection. The intensive use of these compounds has resulted in widespread contamination of the terrestrial, aquatic, and atmospheric environments. Pesticides may enter the air 'directly' during agricultural spray

applications, by wind transport or by volatilisation of residues deposited on soils and plants (Spencer et al. 1973; Lewis and Lee 1976; Seiber et al. 1980). Depending on the type of formulation, physicochemical properties, and meteorological conditions, pesticides present in the atmosphere could be transported over long distances. Some authors have detected organochlorines such as polychlorinated biphenyl (PCBs), hexachlorocyclohexanes (HCHs), DDT, DDE in the air and surface water of the Arctic area, where pesticides were never been used (Pacyna and Oehme 1988; Hoff et al. 1992; Iwata et al. 1994; Hühnerfuss et al. 1992).

Most organochlorine pesticides have a high vapour pressure, so they could be readily transported easily

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even in light winds. The use of most organochlorine pesticides had been prohibited in North America and western Europe for some years, because of their persistence and toxicity.

$\gamma$ -HCH (lindane) is an organochlorine pesticide still in used in western Europe, and which can be photochemically transformed to  $\alpha$ -HCH by atmospheric UV-radiation (Newland et al. 1969; Steinwandter 1976). The  $\alpha/\gamma$ -HCH ratio can be used to estimate the age of the air masses (Oehme 1991). Air masses are considered as old (or pesticide transported over long distance) when the  $\alpha/\gamma$ -HCH ratio is greater than 3 (Lane et al. 1992) and as young air (or indicating fresh application of pesticide) when the  $\alpha/\gamma$ -HCH ratio is less than 1 (Hoff et al. 1992).

This paper presents the concentration levels of 11 pesticides (HCB,  $\alpha$ -HCH,  $\gamma$ -HCH, trifluraline, mecoprop, phosalone, atrazine, carbofuran, carbaryl, diuron and isoproturon) measured in urban, rural and remote atmospheres (gas + particle phases). These data are used to compare the atmospheric contamination of various ecosystems. The significance of the  $\alpha/\gamma$ -HCH ratio of each sampling areas is also discussed.

## Methods

### Sampling

Samples were collected at three sites using a Hi-Vol sampler through a 30 cm diameter glass-fibre filter (GF/A) and 20 g of XAD-2 resin. The procedure for sampling and pre-cleaning of traps has been described elsewhere (Millet et al. 1996; Sanusi et al. 1998). The duration of sampling was 24 hours which allowed sampling of 300–500 m<sup>3</sup> of air. The three sampling sites were situated in the east of France (Alsace). Their locations can be characterised as remote (Aubure), rural (Colmar), and urban (Strasbourg). For this study, three series of five sampling campaigns were performed simultaneously. During the campaigns, between spring 1993 and summer 1994, 27 atmospheric samples (gas and particles) were collected (Sanusi et al. 1998).

### Extraction and analysis

XAD-2 resins and glass fibre filters were separately Soxhlet extracted for 12 hours in *n*-hexane/CH<sub>2</sub>Cl<sub>2</sub> (85:15). Extracts were concentrated to about 1 mL on a rotary evaporator. Clean-up of these concentrated samples was carried out by fraction-

ation-based HPLC using a silica column and UV detection at 254 nm (Sanusi et al. 1998). With this clean-up procedure, three fractions were obtained: 1. HCB,  $\alpha$ -HCH,  $\gamma$ -HCH, trifluraline; 2. mecoprop, phosalone; and 3. atrazine, carbofuran, carbaryl, diuron and isoproturon.

Analyses of these fractions were done by several methods described in detail elsewhere (Sanusi 1996; Sanusi et al. 1998). Fraction 1 was analysed by GC-ECD (<sup>63</sup>Ni electron capture detector using a J&W DB-5 (30 m, i.d. 0.32 mm, film thickness 0.25  $\mu$ m) column. Quantifications were done using an internal standard: 0.05  $\mu$ g/mL  $\delta$ -HCH.

Fractions 2 and 3 were analysed by HPLC-UV (Waters) at 230 nm using a Nova-Pak<sup>TM</sup> C<sub>18</sub> reverse phase column (30 cm ; i.d. 3.9 mm; 4  $\mu$ m; 60  $\text{Å}$ ) at 1 mL/min. For fraction 2, a binary solvent (methanol:acetonitrile (80:20 v/v) mixture:water (0.17% TFA)) was used. A binary solvent was also used for the analysis of fraction 3 with methanol/acetonitrile (85:15 v/v) and water milli-Q. Quantification were done by using 2,3-dimethyl naphthalene at 0.5  $\mu$ g/mL as an internal standard.

## Results and Discussion

### Pesticide concentration

Dates, sites, duration of sampling, volume of air collected and concentrations of the 11 pesticides in the total (gas + particles) phase for the three series of five sampling campaigns are given in Table 1.

A comparative study of the two phases shows that the concentrations of pesticides measured in the gas phase are generally higher than those in the particulate phase (Sanusi 1996). The partition between gas and particulate phases is essentially the result of the combined influence of atmospheric temperature (the gas phase increases with the temperature), the vapour pressures of the pesticides (Haragushi et al. 1994), the concentration of particles in the atmosphere (the particulate phase increase with particle concentration) (Pankow and Bidleman 1991), and the sampling method used. Indeed, because of their high flow rate, Hi-Vol samplers under-estimate particulate-phase concentrations and over-estimate gas phase concentrations (Bidleman 1988; Haragushi et al. 1994). In our results here, we compare the contamination at the three sites by considering the total concentrations (gas + particulate) of pesticides measured.

**Table 1.** Date, sites, duration, volume of air sampled, and concentration (pg/m<sup>3</sup>) of pesticides in total (gas + particle) phases for three series of five sampling campaigns

| Date            | Station    | Duration | Volume of air (m <sup>3</sup> ) | trifluraline | α-HCH | HCB  | γ-HCH | meccoprop | phosalone | carbofuran | carbaryl | atrazine | isoproturon | diuron |
|-----------------|------------|----------|---------------------------------|--------------|-------|------|-------|-----------|-----------|------------|----------|----------|-------------|--------|
| <b>Series 1</b> |            |          |                                 |              |       |      |       |           |           |            |          |          |             |        |
| 19–20/04/93     | Aubure     | 24       | 162.52                          | 5650         | 423   | 1510 | 290   | 260       | 500       | <114       | –        | 254      | –           | –      |
|                 | Colmar     | 24       | 176.42                          | 3350         | 191   | 283  | 1090  | 310       | –         | 440        | 696      | 699      | 209         | 330    |
| 26–27/04/93     | Aubure     | 24h30'   | 284.16                          | 2520         | 163   | 572  | 228   | 5         | –         | <114       | –        | 75       | 100         | –      |
|                 | Colmar     | 22h30'   | 170.29                          | 4500         | 206   | 968  | 1564  | 99        | 76        | 4100       | 500      | 640      | 600         | –      |
| 04–05/05/93     | Aubure     | 24h      | 212.07                          | 5740         | 191   | 1620 | 316   | 360       | 830       | <114       | 130      | 420      | 900         | 1200   |
|                 | Colmar     | 24h      | 204.05                          | 5640         | 75    | 300  | 830   | 890       | 620       | 8100       | 670      | 970      | 1160        | 190    |
| 10–11/05/93     | Aubure     | 25h      | 324.97                          | 7980         | 142   | 900  | 542   | –         | 890       | <114       | –        | –        | –           | –      |
|                 | Colmar     | 30h      | 266.75                          | 3105         | 206   | 183  | 751   | 580       | –         | 4400       | 240      | 1200     | 670         | 980    |
| 17–18/05/93     | Aubure     | 26h      | 450.01                          | 6540         | 214   | 750  | 1630  | 2470      | –         | <114       | 1800     | 1400     | 720         | 1600   |
|                 | Colmar     | 24h      | 205.47                          | 2450         | 258   | 208  | 1364  | 2910      | –         | 4700       | 120      | 860      | –           | 358    |
| <b>Series 2</b> |            |          |                                 |              |       |      |       |           |           |            |          |          |             |        |
| 13–14/06/94     | Aubure     | 21h15'   | 523.86                          | 1315         | 76    | 107  | 80    | 117       | 460       | 3100       | 130      | 286      | 99          | 350    |
|                 | Strasbourg | 22h15'   | 386.15                          | 1624         | 190   | 470  | 624   | 69300     | 7040      | 14200      | 505      | 30000    | 450         | 1250   |
| 14–15/06/94     | Aubure     | 24h15'   | 620.04                          | 2210         | 167   | 176  | 1050  | 1820      | 3670      | 1710       | –        | 110      | –           | 390    |
|                 | Strasbourg | 23h45'   | 379.07                          | 3805         | 98    | 130  | 345   | 6200      | 11500     | 16330      | 767      | 30610    | 755         | 870    |

**Table 1.** Date, sites, duration, volume of air sampled, and concentration (pg/m<sup>3</sup>) of pesticides in total (gas + particle) phases for three series of five sampling campaigns

|                   |                      |                  |                  |              |            |            |              |               |              |               |             |               |             |               |
|-------------------|----------------------|------------------|------------------|--------------|------------|------------|--------------|---------------|--------------|---------------|-------------|---------------|-------------|---------------|
| Series 2 (cont'd) |                      |                  |                  |              |            |            |              |               |              |               |             |               |             |               |
| 22-23/06/94       | Aubure<br>Strasbourg | 24h45'<br>23h45' | 580.39<br>371.43 | 1300<br>3800 | 70<br>272  | 81<br>549  | 1122<br>1738 | 1720<br>18500 | 260<br>2144  | 140<br>12750  | 345<br>575  | 110<br>17440  | 112<br>620  | 646<br>1890   |
| 23-24/06/94       | Aubure<br>Strasbourg | 23h15'<br>24h15' | 568.33<br>366.50 | 2780<br>3320 | 149<br>307 | 101<br>685 | 697<br>1147  | 2400<br>5200  | -<br>8800    | 1075<br>28970 | 110<br>580  | 145<br>51260  | 49<br>800   | 634<br>1680   |
| 11-12/07/94       | Aubure<br>Strasbourg | 28h15'<br>28h45' | 742.75<br>458.51 | 300<br>940   | 98<br>200  | 126<br>508 | 528<br>1230  | 1680<br>3600  | 440<br>1000  | 230<br>14300  | 284<br>68   | 142<br>1630   | 166<br>120  | 430<br>3200   |
| Series 3          |                      |                  |                  |              |            |            |              |               |              |               |             |               |             |               |
| 12-13/07/94       | Colmar<br>Strasbourg | 24h15'<br>24h    | 609.17<br>367.36 | 465<br>743   | 163<br>404 | 145<br>688 | 767<br>1258  | 3890<br>5450  | 2533<br>1360 | 670<br>8150   | 166<br>1420 | 4550<br>690   | 950<br>700  | 480<br>870    |
| 21-22/07/94       | Colmar<br>Strasbourg | 23h30'<br>24h    | 578.24<br>348.99 | 1060<br>1510 | 279<br>313 | 240<br>268 | 807<br>1598  | 1265<br>3850  | 65<br>1050   | 790<br>15760  | 190<br>382  | 1700<br>20410 | 3300<br>660 | 7350<br>1730  |
| 25-26/07/94       | Colmar<br>Strasbourg | 7h45'<br>23h45'  | 140.64<br>355.42 | 1630<br>1950 | 415<br>481 | 211<br>675 | 1100<br>3940 | 2690<br>2500  | -<br>-       | <114<br>2050  | 130<br>125  | 1380<br>4340  | -<br>500    | 13800<br>5850 |
| 26-27/07/94       | Strasbourg           | 24h45'           | 375.46           | 1891         | 280        | 370        | 1520         | 8000          | 950          | 13570         | 770         | 23890         | 490         | 1100          |

Results obtained for Colmar, with the exception of isoproturon, are in agreement with those obtained by Millet et al. (1997) at the same site using similar analytical and sampling methods. Generally, pesticides used locally (trifluraline, mecoprop, atrazine, isoproturon, diuron) are present in higher concentrations. The difference for isoproturon might be the result of local spraying of this compound during the sampling campaigns in previous work (Millet et al. 1997). The abundance of atrazine in Colmar samples in the present work could be the result of the type of farming practiced in the Alsatian region.

In Strasbourg, pesticides used locally are also present at higher concentrations than other pesticides, even if Strasbourg is an urban site. The presence of many crops within about 5 km around Strasbourg could explain this.

Generally, lower concentrations of pesticides were found at Aubure. For example, concentrations of atrazine measured in Aubure are 10 to 100 times lower than those measured in Colmar and Strasbourg. This phenomenon could be explained by the geography of Aubure which is at an altitude of 1100 m and is far removed from crops.

HCb concentrations measured at Aubure were generally higher than those measured in Colmar. The use of HCB is forbidden in France for crop protection, but is still permitted for wood protection. That is probably the reason for the high level of HCB in Aubure. On the other hand, organochlorine pesticides concentrations are of the same order of magnitude at the three sites, which is surprising for Aubure because of its remote location. However, these pesticides are not intensively used in Europe, so none of our sites is closer than any other to an important source of organochlorines. Moreover, these compounds have a low

vapour pressure and consequently can be transported over long distances in the atmosphere.

Detailed analysis of the organochlorine pesticide concentrations shows that concentrations of  $\alpha$ -HCH at the three stations are very similar. This result is the consequence of the use of pure lindane ( $\gamma$ -HCH) in western Europe. Thus, any  $\alpha$ -HCH can come only from degradation of  $\gamma$ -HCH or by long range transport.

#### $\alpha/\gamma$ -HCH ratio

If we know the  $\alpha/\gamma$ -HCH ratio, we can estimate the age of the air masses passing the sites. A  $\alpha/\gamma$ -HCH ratio lower than 1 is characteristic of local emission of pesticide or of young air masses (Hoff et al. 1992). A  $\alpha/\gamma$ -HCH ratio higher than 3 indicates an old air mass or long-range transport (Lane et al. 1992). By photochemical reaction in the atmosphere,  $\gamma$ -HCH can be isomerised to  $\alpha$ -HCH which is more stable.

Table 2 presents the  $\alpha/\gamma$ -HCH ratio of the 27 atmospheric samples collected at the three sites. The ratio varied from 0.06 to 2.8. A ratio greater than 1 was obtained for Aubure samples. This result seems to show that technical HCH is not used in France and emissions of HCH are local. Technical HCH contains 50–80%  $\alpha$ -HCH which has no pesticidal activity. Only  $\gamma$ -HCH is pesticidal.

The  $\alpha/\gamma$ -HCH ratio greater than 1 found at the Aubure site could be explained by its geographic remoteness and high altitude. The  $\gamma$ -HCH arriving at Aubure comes not only from around the site but also via long-range transport.

The  $\alpha/\gamma$ -HCH ratios for Colmar and Strasbourg sites are low (<0.4), so we can consider local emission is predominant and the air masses young.

**Table 2.**  $\alpha/\gamma$ -HCH ratio for the 27 atmospheric samples analysed

| Aubure      | $\alpha/\gamma$ | Colmar      | $\alpha/\gamma$ | Strasbourg  | $\alpha/\gamma$ |
|-------------|-----------------|-------------|-----------------|-------------|-----------------|
| 19–20/04/93 | 1.43            | 19–20/04/93 | 0.17            | 13–14/06/94 | 0.34            |
| 26–27/04/93 | 0.72            | 26–27/04/93 | 0.14            | 14–15/06/94 | 0.28            |
| 04–05/05/93 | 0.60            | 04–05/05/93 | 0.09            | 22–23/06/94 | 0.16            |
| 10–11/05/93 | 2.80            | 10–11/05/93 | 0.28            | 23–24/06/94 | 0.27            |
| 17–18/05/93 | 0.13            | 17–18/05/93 | 0.18            | 11–12/07/94 | 0.15            |
| 13–14/06/94 | 0.99            | 12–13/07/94 | 0.22            | 12–13/07/94 | 0.32            |
| 14–15/06/94 | 0.15            | 21–22/07/94 | 0.28            | 21–22/07/94 | 0.20            |
| 22–23/06/94 | 0.06            | 25–26/07/94 | 0.37            | 25–26/07/94 | 0.12            |
| 23–24/06/94 | 0.21            |             |                 | 26–27/07/94 | 0.15            |
| 11–12/07/94 | 0.19            |             |                 |             |                 |

## Conclusion

Between spring 1993 and summer 1994, 27 atmospheric samples collected in remote, rural, and urban areas were analysed for 11 pesticides. The gas and particulate phases of each sample were analysed. Results shows that concentrations of pesticide in the gas phase are higher than those in the particulate matter.

Comparison of results from the three stations showed that pesticide concentrations in the remote area (Aubure) are, in general, lower than those in either the rural (Colmar) or urban area (Strasbourg) sampled.

The  $\alpha/\gamma$ -HCH ratio in samples varied between 0.06 and 2.8, with at higher ratio of  $\alpha/\gamma$ -HCH found at the Aubure site. These values indicated the presence of young air masses or local emission of HCH for rural and urban areas. For Aubure, air masses are sometimes older (ratio >1). This is the consequence of the geography of this station.

On the other hand, in Europe the use of pure lindane (99%  $\gamma$ -HCH) can explain the low  $\alpha/\gamma$ -HCH ratios found at Colmar and Strasbourg.

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