4.1 Air

4.1.1 Experimental design

4.1.1.1 Sampling sites

The objective of the air sampling network is to obtain representative data for assessing time trends and the regional and global transport of POPs. We interpret 'representative' as being a sufficient number of sampling sites to make general conclusions about POPs trends and not to be representative of heterogeneity of that region. Ch. 3. shows that complete geographical coverage for a particular region or continent is not economically feasible and would require an extremely dense sampling network and considerable prior investigatory work to assess regional variability on air concentrations in POPs.

Initially, for addressing POPs trends, the GMP should strive for (in each region) at least:
i.) 3 to 5 stations with active high-volume sampling.
ii.) A larger network of 10 to 15 passive sampling stations arranged in a grid with spacing of approximately $20^{\circ} \times 20^{\circ}$. Passive samplers should be co-located at the high volume sites for comparison purposes.

These sites may be located centrally so as to obtain information on time trends of regional sources. The sites need to be sufficiently remote from urban centres and industrial and other sources of POPs as to reflect concentrations typical of a large area around the site (at least 100 km radius). Requirements for such a site include the availability of meteorological observations and station personnel who could be trained in the sampling techniques. Regional decision on site selection might also include geographic considerations. In North America, Europe and the Arctic, some stations already exist as part of the Integrated Atmospheric Deposition Network (IADN), Cooperative Programme for Monitoring and Evaluation of the Long-range Transmission of Air Pollutants in Europe (EMEP) and Arctic Monitoring and Assessment Programme (AMAP) programmes and would be used for the GMP. The East Asian POPs monitoring network, coordinated by Japan, is now producing measurements for 9 UNEP POPs from several East Asian countries. In other regions, use should be made of existing air quality monitoring sites that meet the appropriate site selection criteria, such as those operated by members of the World Meteorological Organization (WMO) under the Global Atmosphere Watch (GAW) programme.

This network may be supplemented by additional passive sampling sites situated on islands and on continental margins to yield information for addressing transcontinental transport between regions.

In summary, two types of measurements of a full range of POPs are envisioned in each region: (i) cumulative sampling (for 1 to 2 days every week or continuously over periods of 1 to 2 weeks) by active high volume sampling ($\sim$0.5-1 m^3/min flow rate) at a few sites in each region. These samples would be separated into particulate and gaseous fractions and (ii) continuous, cumulative passive (diffusive) sampling for integration periods of 3 months to 1 year using passive samplers deployed at a large number of sites including the high volume sampling sites.

4.1.1.2 Siting considerations

The combination of a number of long-term active sampling sites supplemented by a larger number of passive sampling sites will yield a cost-effective programme with flexibility to address a variety of issues. Regional availability of laboratories and consideration of sources and air transport pathways will influence the spatial configuration and density of the network.

It is important to encourage co-operation between countries within regions and consultation with POPs modellers to ensure that the best sites are selected and that observational practices are standardized. Available facilities at which other atmospheric composition measurements are made should be used whenever possible or feasible.

Positioning and installation of samplers should follow standard operating procedures for air sampling programs. A detailed description of all selected sites should be provided. More general criteria are given here:
> 1. *Regional representativity*: A location free of local influences of POPs and other pollution sources such that air sampled is representative of a much larger region around the site.
> 2. *Minimal meso-scale meteorological circulation influences*: Free of *strong* systematic diurnal variations in local circulation imposed by topography (e.g. upslope/ down slope mountain winds; coastal land breeze/lake breeze circulation).
> 3. *Long term stability*: In many aspects including infrastructure, institutional commitment, land development in the surrounding area.
> 4. *Ancillary measurements*: For the super-sites, other atmospheric composition measurements and meteorological wind speed, temperature and humidity and a measure of boundary layer stability. For the passive sites, meteorological wind speed, temperature and humidity.
> 5. Appropriate *infrastructure and utilities*: Electrical power, accessibility, buildings, platforms, towers and roads.

4.1.1.3 Characterization of transport to the sites

Measurements of POPs need to be understood in terms of the processes responsible for the observed air concentrations and trends at the site as well as their regional and global scale transport. To do this, an understanding of local (meso-scale) as well as large (synoptic) scale air transport pathways to the site is required. This is achieved through local meteorological measurements to characterize meso-scale influences as well as use of Lagrangian or Eulerian transport models to reconstruct the large scale transport pathways to the site. It is also important that for water-soluble POPs, oceanic and riverine transport and air-water exchange be considered, especially for sites located on coastlines.

As a first step, it may be useful and insightful to assess GMP data for a particular region using a measure of the long-range transport potential (LRTP) for the various POPs. The characteristic travel distance (CTD) – defined as the "half-distance" (analogous to a half-life) for a substance present in a mobile phase – is a useful parameter in this context. CTDs in air and water having been calculated using the TaPL3 model, which considers various degradation and transport pathways that chemicals may undergo based on their physical-

chemical properties (Beyer et al., 2000). CTDs for chemicals discharged into air and water are listed in Table 4.1. It is important to note that these distances should be compared in a relative manner and are dependent on model parameterizations (Stroebe et al., 2004).

Table 4.1.1 – Characteristic travel distances (CTDs, km) for air and water for UNEP POPs. (POPs are ranked highest to lowest in terms of the CTDs for air).

Chemical	CTD (air)	CTD (water)
Hexachlorobenzene	110 000	26 000
PCB (tetra homolog)	8900	2900
p,p'-DDE	2800	4300
Toxaphene	2500	9700
PCB (hepta homolog)	1900	2000
Dieldrin	1100	12 000
Chlordane	1000	4000
p,p'-DDT	830	3300
2378-TCDD	810	1300
OCDD	460	1900
Aldrin	100	1800

PCB-polychlorinated biphenyl; DDT – dichlorodiphenyltrichloroethane; DDE – dichlorodiphenyltrichloroethane; TCDD – tetrachlorodibenzodioxin; OCDD – octachlorodibenzodioxin.

The resulting CTDs suggest that of the POPs listed the ones with important air transport pathways (the "flyers") include: hexachlorobenzene, lower molecular weight PCBs, *p,p'*-DDE and toxaphene; while ones that have important water transport pathways (the "swimmers") include: hexachlorobenze, dieldrin and toxaphene.

A common transport pathway analysis tool that can facilitate the detection and interpretation of trends in POPs air concentrations is based on air-parcel back-trajectory analysis. In this approach, the transport path of air to a site during sampling is reconstructed from observed wind fields. There are various methodologies that have been applied to improve trend detection ranging from trajectory sector analysis to cluster analysis. In the latter, discriminate analysis is utilized to identify the main groups of trajectory pathways to a site (Moody *et al.*, 1998). This can also be done for samples that fall in various percentile ranges of the trajectory distribution. Another approach that utilizes trajectories to identify sources and "preferred transport pathways" is potential source contribution function analysis (PSCF) pioneered for POPs by Hsu *et al.* (2003a and b). In this approach, upwind areas in a grid placed over the map are identified that are most frequently occupied by points in a 3 to 5 days back trajectory for high concentration versus low concentration percentile trajectories. Insight into upwind sources and trends in air transported from those regions that is gained from the above analyses is much more effective in addressing policy questions than simple time-series analysis of observations. Gouin et al., (2005) demonstrated how probability density maps (a modification of the PSCF approach) could be used to interpret time-integrated, passive sampler-derived data (Fig. 4.1.1) by identifying an airshed associated with the history of the air mass transported to a particular site.
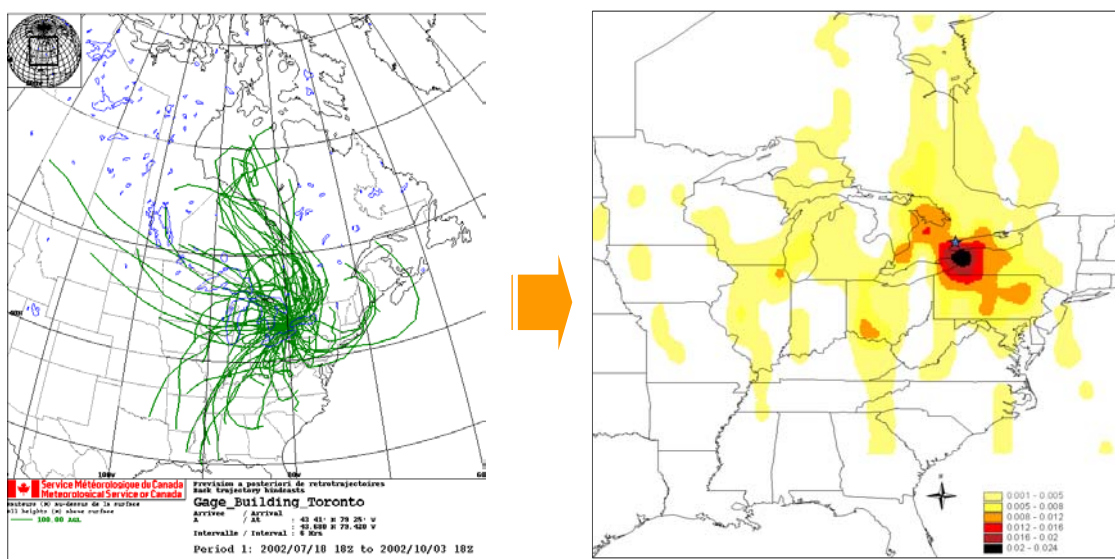
Figure 4.1.1. Example of probability density map (right panel) constructed from daily 3-day air parcel back trajectories for a time-integrated air sample.

Several models of regional and global scale POPs transport in the environment, including the atmosphere, exist (Chapter 4 of the RBA/PTS Global Report, UNEP, 2003). They simulate the large scale spatial and temporal distribution of a POP compound including the processes of direct emissions to the atmosphere, transport and dispersion on winds, chemical transformation in the atmosphere, and air-surface exchange. These models are either coarsely resolved box models (Breivik and Wania, 2002, MacLeod *et al*., 2001, Wania *et al*., 1999) or meteorology-based models with high spatial and temporal resolution (e.g. Koziol and Pudykiewicz, 2001, Semeena and Lammel, 2003, Hansen *et al.,* 2004). In either case the size of the model domain ranges from regional to global. These models can be useful in network design and can be evaluated using POPs observations. The data together with the models are used to support the "evaluation of the effectiveness of measures taken to fulfill the Stockholm Convention". This will likely be an iterative process where differences between model predictions and measurements are identified and used to improve model design and measurement strategy.

4.1.2 Sample matrices

Air is an important matrix because it has a very short response time to changes in atmospheric emission and is a relatively well-mixed environmental medium. It is also an entry point into food chains and a global transport medium. Air data are required to validate atmospheric POPs transport models. Some sampling networks exist. As mentioned above, active and passive samplers can be combined, offering an opportunity to create a cost-effective programme. In both active and passive sampling, POPs in particulate matter and/or the gas phase are filtered from air, separated, concentrated on a filter medium and extracted into a small amount of organic solvent for subsequent chemical analysis of POPs.

4.1.3 Sampling and sample handling

Air sampling requires the following capacities: (1) active and passive air samplers, (2) trained station personnel to operate and maintain the high-volume samplers, (3) meticulous preparation of clean sampling media in the laboratories performing the extraction procedures and chemical analysis. Sampling methods and QA/QC procedures should, as far as possible, be adopted from existing air monitoring programmes for POPs, but they will need to be adapted to and validated for the specific conditions, concentration levels and temperature at the sampling sites.

4.1.3.1 High volume sampling

High volume samplers should have a size-selective inlet for collecting only those particles smaller than 10 micrometers diameter. Sampling should take place using techniques practiced by routine long term monitoring networks in temperate areas (e.g. Fellin *et al.,* 1996; Environment Canada, 1994) and sub-tropical to tropical regions (e.g. Japanese Environment Ministry and National Institute of Environmental Studies). These groups recommend the technique of separating particles from gases using the combination of glass fibre filters in series with two gas absorbants. The nature of the absorbants used need to be matched to the needs of the regional monitoring programme. Several possibilities exist which are favoured for long term measurements and should be selected by experienced experts planning a regional study:
> • 2 PUF plugs recognizing that some volatile chemicals (e.g. chlorobenzenes) will not be trapped efficiently. In this case, keep sample times short (e.g. especially when it is warm).
> • PUF/XAD combination generally extracting and analyzing both media together.
> • PUF followed by active carbon fibre felt disks.

Two absorbants are necessary to check periodically for breakthrough losses and to avoid substantive losses for some relatively volatile compounds (e.g. HCB), especially in tropical areas.

Samples could be taken for 1 to 2 days once every week or two weeks. Every fourth sample should include a field blank. This is a set of filter and absorbants that is treated exactly as the samples including placement in the sampler except no air is drawn through them. The method detection limit (MDL) is often determined by the background amounts on these blanks rather than the instrumental detection limit.

Filters and absorbents are pre-treated prior to sampling according to a methodology similar to that described in Fellin *et al*. (1996). Samples should be put into the sampling head using environment and handling practices that are free of contamination and volatilization losses. Many POPs are semi-volatile and may evaporate from sampling media if they are warmed appreciably above ambient temperatures. After sampling, samples and field blanks are extracted in the appropriate solvent (e.g. hexane and dichloromethane are common) by placing them in a Soxhlet extractor with 450 ml solvent are reduced in volume down to approximately 20 ml (e.g. see Fellin *et al*., 1996). These extracts are then split into two, placed in pre-weighed pre-cleaned vials, and sealed. One half is shipped to the laboratory and the other half archived. This archive is extremely important to recover from accidental sample loss during shipping and laboratory analysis. It also allows samples to be reanalyzed

years later when analytical techniques may have improved and there is new information to be gained.

4.1.3.2 Passive sampling

Passive air sampling of POPs has undergone considerable technological development in the past decade. Early studies used semipermeable membrane devices (SPMDs) to survey POPs over large spatial scales (Ockenden et al., 1998). Now, samplers made of polyurethane foam (PUF) disks (Shoeib and Harner, 2002) and XAD-resin (Wania et al., 2003) have been developed and widely adopted. These samplers have been used to map the spatial variability of POPs in regional studies (Motelay-Massei et al., 2005; Gouin et al., 2005; Daly et al. 2007) and on a continental scale in North America (Shen *et al*., 2004, 2005, 2006) and Europe (Jaward *et al*., 2004 a, b). The first results from the Global Atmospheric Passive Sampling (GAPS) study have demonstrated the feasibility of these samplers for global spatial mapping at more than 60 sites around the world (Pozo et al., 2006). A key aspect of GAPS is technology transfer and capacity building – especially in regions lacking POPs air data. Numerous regional passive sampling efforts have been initiated recently in Europe and East Asia (Jaward et al., 2004a, 2004b, 2005 – coordinated through Lancaster University) and in central and eastern Europe (CEEPOPSCTR) – either as one-time surveys or on a continuous basis.

Passive air samplers for POPs typically rely on a sorbent with a high capacity for POPs, such as polyurethane foam (PUF) or styrene/divinylbenzene-co-polymer resin (e.g. XAD-2). For example, Shoeib and Harner (2002) use PUF disks (approximately 14 cm diameter, 1.35 cm thick), whereas Wania *et al.* (2003) employ XAD-2 resin filled into a stainless steel mesh cylinder (Fig. 4.1.2). The sorbent is typically housed in protective stainless steel chambers, which can either be shaped like a dome (Shoeib and Harner, 2002) or a cylinder (Wania *et al.,* 2003). Such shelters protect the sorbent from direct deposition of large particles, sunlight, and precipitation and help to diminish the wind speed effect on the sampling rate.
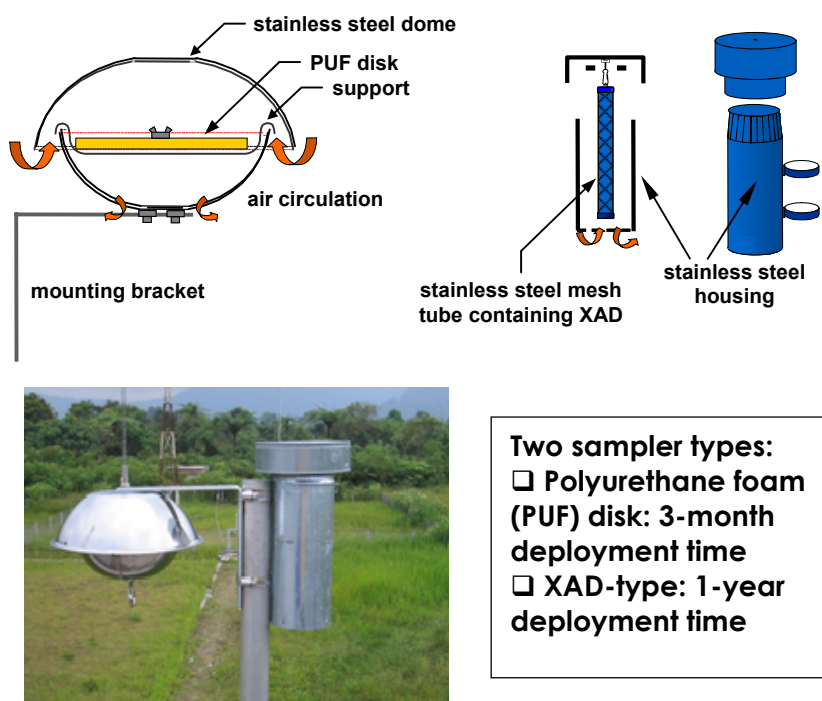
Fig. 4.1.2 Schematics and photograph of PUF-disk (left) and XAD-based passive air samplers.

Sampling rates for PUF-disk samplers are typically on the order of 3 to 4 m^3/day (Pozo et al., 2006) as so a 3-month deployment provides an equivalent sample air volume of approximately 270-360 m^3, which is sufficient for the detection of most POPs. Shorter integration periods of 1 month have also be incorporated successfully. The wind-effect on sampling rate for the domed chamber design has been evaluated under controlled conditions (Tuduri et al., 2006), from field study results (Pozo et al., 2004; Klanova et al., 2006) and using flow simulation models (Thomas et al., 2006). Generally, the chamber is capable of dampening the wind-effect on sampling rate (by maintaining the air flow within the chamber at less than ~1m/s. However, higher sampling rates have been observed at windy, coastal and mountain sites (Pozo et al., 2004, 2006).

A more precise measure of the air volume sampled may be achieved by spiking the sorbent prior to exposure with known quantities of "depuration compounds". These are isotope labeled chemicals or native compounds that do not exist in the atmosphere and cover a wide range of volatility (assessed based on their vapor pressure and/or octanol-air partition coefficient, K_{OA}). The loss of depuration compounds over the sampling period is used to calculate the effective air sample volume (Pozo *et al.*, 2004, 2006). The air concentration is then calculated based on this air volume and the amount of chemical collected over the sampling period. When depuration compounds are used it is possible to report semi-quantitative concentrations for PUF-disk sampler with an expected accuracy within about a factor of 2 (Gouin et al., 2005). Otherwise, results may be reported as amount/sampler or as concentrations using a previously derived sampling rate. In this case, it is important that the greater uncertainty in the data be acknowledged.

It is imperative to account for approach to equilibrium that may occur for the more volatile POPs (e.g. HCB) (Harner et al., 2004; Gouin et al., 2005; Pozo et al., 2006). This is mainly a consideration for PUF disk samplers that have lower capacities compared to XAD-samplers. The effect is larger at warmer temperatures at which the equilibrium is shifted to the atmospheric gas phase, and the capacity of the sampling sorbents is greatly lowered. It is important to note that this limited capacity of the PUF disk is required to allow depuration compounds (of similar volatility to POPs) to be used to establish site-specific sampling rates. Alternatively, PUF disk capacities can be increased by impregnating with sorptive polymers such as XAD powder. However, this would preclude the use of depuration compounds.

Sampling rates for XAD-samplers are somewhat lower at ~0.5 m^3/day (Wania et al., 2003). These samplers are designed to integrate over an entire year with an equivalent sample air volume of approximately 180 m^3. Wind tunnel experiments measuring the uptake rate over the wind speed range 5 to 15 m/s showed that the shelter employed in the XAD-based passive sampler dampens the movement of air close to the sorbent sufficiently, to assure that molecular diffusion is controlling the rate of uptake (Wania *et al.,* 2003). Approach to equilibrium is not a concern for XAD-samplers because of the relatively higher capacity of XAD (vs PUF) (Shen et al., 2002). However, this also prevents the use of depuration compounds for assessing site-specific sampling rates.

Prior to use, the sorbents such as the PUF disks and XAD-2 resin, are pre-cleaned by sequential soxhlet extraction using a combination of polar and non-polar solvents (e.g. acetone: hexane and/or acetone followed by hexane). Samples are stored in solvent-rinsed and gas-tight glass jars or metal or teflon containers prior to and after deployment. One field blank should be deployed at each site to assess potential contamination. These are typically inserted to the sampling chamber, removed immediately and then stored and treated as a sample. Samples are extracted using the same techniques as for active air samples described above. Similarly, analysis of extracts proceeds following procedures outlined in Chapter 5.

4.1.4 Considerations for time trend analysis

Chapter 3 (Statistical Power) outlines key considerations for conducting trend analysis on environmental data. Although, much of the analysis is presented with respect to biological data, many of the issues are applicable to air samples and should be considered when planning a sampling strategy.

Trend analysis for air data, particularly high volume data, has additional complexity. This is due to the responsive nature of air (air has a relatively low capacity for POPs) coupled with relatively short sampling durations for high volume air samples (typically days). Consequently, time series data for air typically demonstrated periodicity that may occur seasonally or over shorter time intervals. Further, these 'harmonics' are compound- and site-specific. Digital Filtration (DF) (Hung et al., 2002) and Dynamic Harmonic Regression (DHR) (see figure 4.1.3; Becker et al., 2006) are two techniques that have been used successfully to assess time trends.
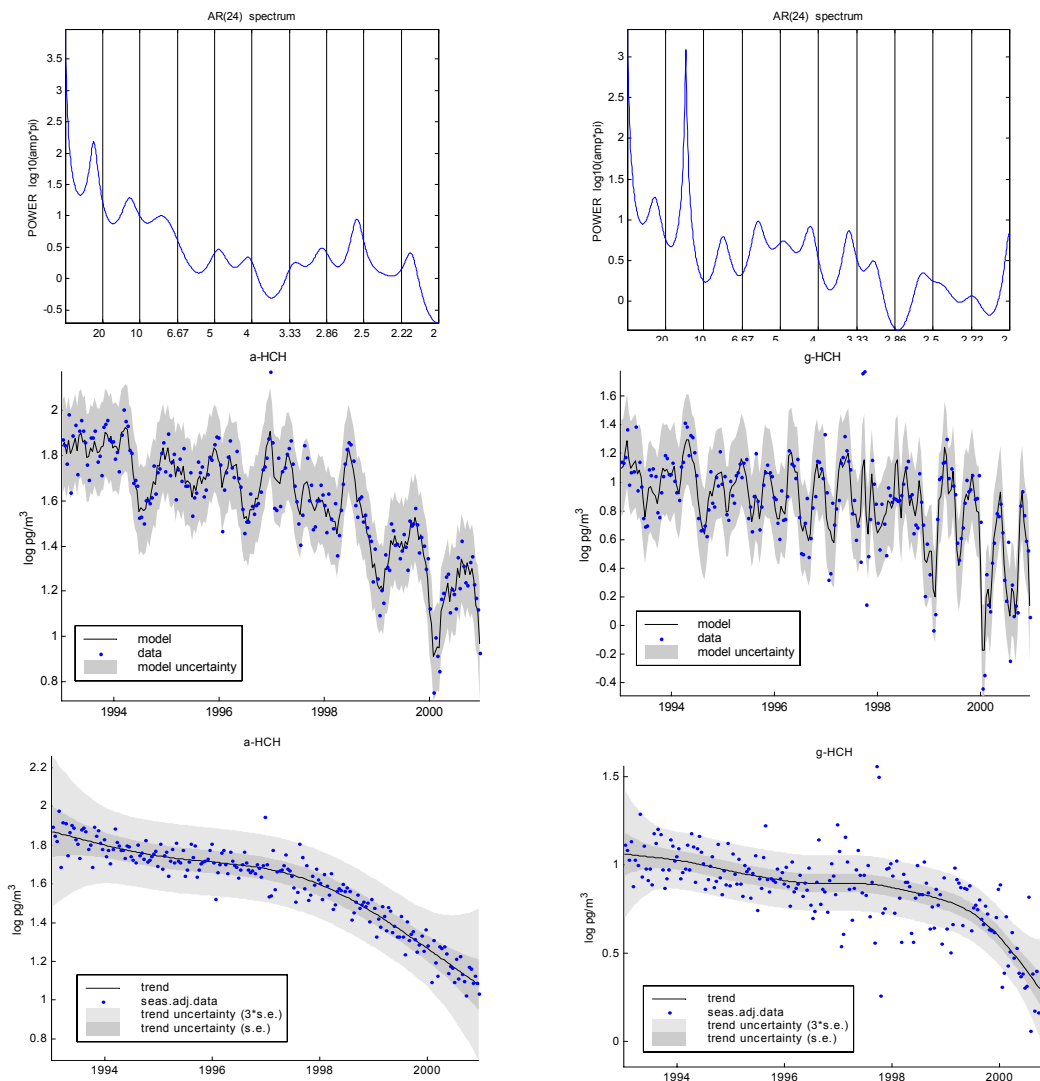
Fig 4.1.3 Example of Dynamic Harmonic Regression (DHR) for active high volume sampling data for a- and g-HCH (hexachlorocyclohexane) from 1993-2000 from Zeppelin Mountain in Svalbard, Norway. Harmonics (top panel) – harmonics are shown in biweekly periods and vary over time and with different chemicals and can provide information on chemical behaviour over short time periods (i.e. inter- or intra-seasonal patterns); Model fit (middle panel) – the measured concentrations are compared to the model fit with uncertainty to within 95% confidence levels; Trends (bottom panel) – seasonally adjusted data is used to evaluate long-term trends with uncertainty given for 95% and 90% confidence levels (Becker et al., unpublished results).

The topic of climate change and its impact on contaminant pathways introduces even more complexity for temporal trend data analysis (Macdonald et al., 2005). Correlations between air concentrations of POPs and low-frequency climate variations (e.g. North Atlantic Oscillation - NAO, El Nino-Southern Oscillation (ENSO) and the Pacific North American (PNA) pattern) have already been demonstrated (Ma et al., 2004). This is of special concern in regions such as the arctic where expected temperature increases and associated geophysical cycles are maximimized (Macdonald et al., 2005). In addition to temperature increases, other meteorological disruptions associated with climate change (e.g. increased floods, droughts) may affect POP mobility and air concentrations trends.

All of these topics should be considered when interpreting trends. Because of the site specific nature of these processes it is important that trends be considered on a site-by-site basis rather than implying regional coverage with the given number of sites. This strategy will also help to ensure comparability of data.

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AMAP	http://www.amap.no
CEEPOPSCTR	http://www.recetox.muni.cz/ceepopsctr/research-projects.html
East Asian POPs Network	http://www.env.go.jp/en/chemi/pops.html
EMEP	http://www.emep.int.html
GURME	http://www.wmo.ch/web/arep/gaw/gaw_home.html
IADN	http://www.msc-smc.ec.gc.ca/iadn/index_e.html
Lancaster Environment Centre	http://www.lec.lancs.ac.uk/
University of Toronto (Wania Group)	http://www.utsc.utoronto.ca/~wania/main.html
WMO/GAW	http://www.wmo.ch/web/arep/gaw/gaw_home.html