POPS ANALYSIS AND MONITORING IN THE ASIAN COASTAL HYDROSPHERE

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Introduction

The UNU's capacity development project on environmental pollutant analysis using a quadruple type gas chromatograph mass spectrometer (GC/MS, Shimadzu-GCMS QP5050A and Shimadzu-GCMS QP2010) has been implemented since 1996 with support from Shimadzu Corporation. Trained on sample pretreatment and data analysis using GC/MS for a wide variety of samples (water, biota, sediment, and food, fish scale and air), the project partners have monitored various target environmental pollutant chemicals from Volatile Organic Compounds (VOCs) to Persistent Organic Compounds (POPs) as shown in Table 1 (see the back of this flyer). The project's monitoring results as well as the quality assurance and quality control aspects of the project activities are discussed here.

Quality Assurance

Data quality was assured routinely by conducting blank tests, injection repeatability tests and standard addition recovery tests. A single surrogate of DDT-$^{13}C_{12}$ for all POPs (excluding PCDD/Fs and PCBs) was chosen to check the sample recoveries with the recovery ratio range of 70-130% as an acceptable range. No decomposition of the DDT in the GC was confirmed with the chromatogram peaks of DDD and DDE. Two internal standards, phenanthrene-d_{10} and chrysene-d_{12}, were injected as syringe-spike. An inter-laboratory calibration was carried out in 2002 using two reference water samples prepared by Shimadzu Corporation. Table 2 (see the back of the flyer) shows the statistical analysis of the reported results. All z-scores were lower than 2 indicating all data were within the acceptable range of inter-laboratory variability. However, a closer look at the inter-lab data and NIQR values reveals that some countries faced difficulties getting accurate concentrations of Aldrin, Endrin, and p,p'-DDT. Please refer to the Quality assurance document on blank tests, injection repeatability tests and standard addition recovery tests.

Monitoring data in the third phase (2002-2005)

The third phase of this project monitored POPs in water, sediment, and soil. Over the period of 4 years, 57 different compounds were measured in the 8 participating Asian countries.

The samples were collected at approximately 670 sites in rivers, lakes, costal waters and sediments throughout the 8 participating Asian countries and provide a general overview of POP levels in these countries. During the 4 consecutive years some countries conducted the sampling fully or largely at the same sites (e.g. Thailand, Philippines, Singapore, Indonesia) allowing inter-annual comparisons whereas other countries changed the sites partially (e.g. Vietnam, Korea and Malaysia) or even every year (China).

Among all samples the most commonly detected compounds were *p,p*'-DDT and its metabolites *p,p'*-DDD and *p,p'*-DDE, followed by HCB, Lindane and *cis*- and *trans*-Chlordane. Specifically, in limnic sediment samples *p,p'*-DDD, *p,p'*-DDE and HCB have exceeded 70% of detection frequency. In marine sediment Lindane, HCB and *p,p'*-DDD were recorded in more than 70% of the samples. In seawater, detection frequencies for *cis*-Chlordane, Heptachlor, Lindane, Dieldrin and *p,p'*-DDT ranged above 50% whereas in freshwater samples, the most common compounds *p,p'*-DDT, Lindane, *trans*-Chlordane, and HCB exceeded only the detection frequency of 30%. Figures 2-4 show temporal trend data in Thailand between 2002-2005, sea water (2002), and fresh water (2003), respectively.

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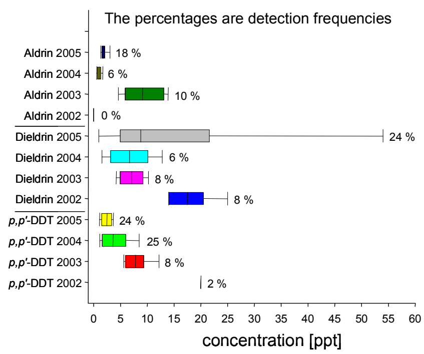
UNU Partnership Initiative: http://www.unu.edu/esd/pi

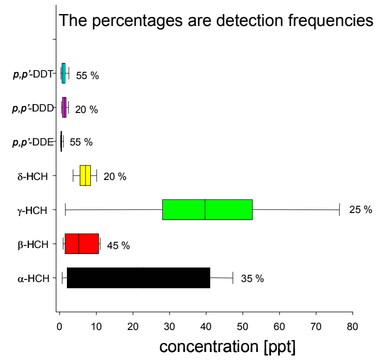
UNU POPs Monitoring Project

Fig. 1 Project partners countries



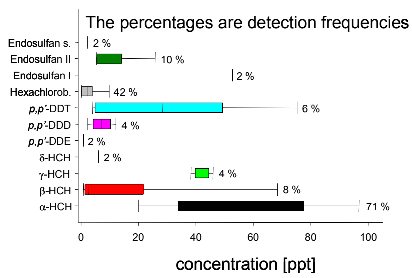
- Only substances mentioned which were measured in all years
- Sample sites were largely the same
- Compound concentrations are in the same magnitude in all years
- The percentage of sample sites, where the substances have been detected is comparatively low and do not vary remarkably between years

Fig. 2 Water samples, Thailand 2002-2005



- *p,p'*-DDT and *p,p'*-DDE were common among sample sites
- concentrations mainly up to 10 ppt,
- α-HCH up to 47 ppt, γ-HCH up to 76 ppt
- Pollution statistically dependent on water body (1-way-ANOVA, p<0.005, based on scores), Bohai Sea showing higher pollution than South, Yellow, East Sea or Dalian Bay

Fig. 3 Sea water samples, China 2002



- Only 11 out of 21 pesticides were detectable among sample sites
- only α-HCH was common among sites
- highest concentration was measured for α-HCH with 98 ppt
- Pollution statistically not dependent on season but on water body (1-way-ANOVA, p<0.05, based on scores), sites in Haihe River showing higher concentration than in Chaobai River and Miyun Reservoir

Fig. 4 River water samples, China 2003

Table 1. Target chemicals and media examined in this project during the last three phases (1996 - 2004)

	First Phase (1996 - 1998)					Second Phase (1999 - 2001)			Third Phase (2002 - 2004)		
	1996	1997		1998		1999	2000	2001	2002	2003	2004
	Pesticides	VOCs	TBTs	VOCs	Aldehydes	EDC-like Pesticides	EDC-like Phenols	EDC-like Phthalates	Pesticides POPs	Pesticides POPs	Pesticides POPs
Target Media	Rice	Tap/River Water	Fish Scales	Indoor/Ambient Air	Indoor/Ambient Air	River Water	River Water	River Water	River Water	River Water/Sediment	River Water/Sediment
Target Chemicals	Fenitrothion(MEP	1,1-Dichloroethylene	Tri Buthyl Tin	Trichloromethane	Formaldehyde	α-BHC	Bisphenol-A	Di methyl phthalate	Hexachlorobenzene	Hexachlorobenzene	Hexachlorobenzene
	Malathion	Dichloroethane	Tri Phenyl Tin	1,1,1-Trichloroethane	Acetaldehyde	β-BHC	4-t-Butylphenol	Di ethyl phthalate	Heptachor	Heptachor	Heptachor
	Chlorpyrifos	*t*-,1,2-Dichloroethene		Terachloromethane		γ-BHC	4-n-Butylphenol	Di i-propyl phthalate	Ardrin	Ardrin	Ardrin
	p,p′-DDT	*c*-1,2-Dichloroethene		Benzene		δ-BHC	4-n-Pentylphenol	Di allyl phthalate	*t*-Chlordane	*t*-Chlordane	*t*-Chlordane
		Trichloromethane		1,2-Dichloroethane		Aldrin	4-n-Hexylphenol	Di n-propyl phthalate	*c*-Chlordane	*c*-Chlordane	*c*-Chlordane
		1,1,1-Trichloroethane		Trichloroethene		Dieldrin	4-n-Heptylphnol	Di i-butyl phthalate	Dieldrin	Dieldrin	Dieldrin
		Terachloromethane		1,2-Dichloropropane		*p,p′*-DDE	4-t-Octylphenol	Di n-butyl phthalate	Endrin	Endrin	Endrin
		Benzene		Bromodichloromethane		Endrin	4-n-Octylphenol	Di n-pentyl phthalate	*p,p′*-DDT	*p,p′*-DDT	*p,p′*-DDT
		1,2-Dichloroethane		*c*-1,3-Dichloropropene		*p,p′*-DDD	4-Nonylphenol	Butyl benzyl phthalate			
		Trichloroethene		Toluene		*p,p′*-DDT	2,4-Dichloropheno	Di n-hexyl phthalate			
		1,2-Dichloropropane		*t*-1,3-Dichloropropene			Pentachlorophenol	Di butoxy ethyl phthalate			
		Bromodichloromethane		1,1,2-Trichloroethane				Di cycrohexyl phthalate			
		c-1,3-Dichloropropene		Tetrachloroethene				Di phenyl phthalate			
		Toluene		Dibromochloromethane				Di n-heptyl phthalate			
		t-1,3-Dichloropropene		*m,p*-Xylene				Di 2-ethyl hexyl phthalate			
		1,1,2-Trichloroethane		*o*-Xylene				Di n-octyl phthalate			
		Tetrachloroethene		Tribromomethane				Di 2-ethyl hexyl adipate			
		Dibromochloromethane		*p*-Dichlorobenzene							
		m,p-Xylene									
		o-Xylene									
		Tribromomethane									
		p-Dichlorobenzene									
Surrogate			Tri Pentyl Tin				Bisphenol-A d_{16}	Di n-pentyl phthalate-d_4	*p,p′*-DDT-$^{13}C_{12}$	*p,p′*-DDT-$^{13}C_{12}$	*p,p′*-DDT-$^{13}C_{12}$
Internal Standards		*p*-Bromofluorobenzene	Tetra Butyl Tin		Diphenylamine	Phenanthrene-d_{10}	Naphthalene-d_8	Di n-butyl phthalate-d_4	Pyrene-d_{10}	Pyrene-d_{10}	Pyrene-d_{10}
						Pyrene-d_{10}	Phenanthrene-d_{10}	Di 2-ethyl hexyl phthalate-d_4			
							Pyrene-d_{10}				
Instruments	GCMS-QP5050A					GCMS-QP5050A			GCMS-QP2010		

Table 2. Data from inter-laboratory calibration exercise conducted in 2002

Countries (A-H)	A	B	C	D	E	F	G	H				
	Z-scores								Average	Median	SD	NIQR
Hexachlorobenzene	1.77	1.01	0.29	1.15	0.52	0.32	0.19	0.14	8.0	7.6	2.1	2.3
Heptachlor	0.10	0.51	0.86	0.27	1.10	1.04	0.05	0.46	18.2	18.3	3.1	3.4
cis-Chlordane	0.67	0.27	1.46	0.03	0.16	0.30	0.48	0.78	19.5	19.2	2.0	1.8
Dieldrin	0.49	1.50	0.60	0.01	0.87	0.57	0.58	0.17	29.1	30.2	3.7	4.0
Aldrin	0.83	0.35	0.71	0.66	0.43	0.36	0.95	0.03	53.1	52.1	15.8	23.9
Dieldrin	0.20	1.15	0.70	0.10	0.56	0.26	1.09	0.25	54.8	55.5	6.9	7.9
Endrin	1.05	0.19	1.06	0.19	0.87	1.61	0.03	0.08	82.6	79.9	45.4	50.6
p,p′-DDT	0.42	0.04	0.66	0.77	0.68	1.24	0.03	0.84	54.1	51.4	13.7	18.2

|z|=<2: Satisfiable, 2<|z|<3: Doubtful, |z| >=3: Unsatisfiable, SD: Standard Deviations, NIQR: Normalized InterQuartile Range

Some of Scientific Journal Papers Produced using the Project Results

Korea

Donghao Li, Minseon Kim, Won Joon Shim, Un Hyuk Yim, Jae-Ryoung Oh, Young-Jin Kwon, 2004, “Seasonal flux of nonylphenol in Han River, Korea”, Chemosphere 56, 1–6.
Donghao Li, Minseon Kim, Jae-Ryoung Oh, Jongman Park, 2004, “Distribution characteristics of nonylphenols in the artificial Lake Shihwa, and surrounding creeks in Korea” Chemosphere 56, 783–790.
Donghao Li , Jae-Ryoung Oh , Jongman Park, 2003, “D irect extraction of alkylphenols, chlorophenols and bisphenol A from acid-digested sediment suspension for simultaneous gas chromatographic–mass spectrometric analysis” Journal of Chromatography A, 1012, 207–214.
Donghao Li, Meihua Dong, Won Joon Shim, Sang Hee Hong, Jae-Ryoung Oh, Un Hyuk Yim, Jee Hyun Jeung, Narayanan Kanan, Eun Soo Kim, Sung Rok Cho, 2004, “Seasonal and spatial distribution of nonylphenol and IBP in Saemangeum Bay, Korea” Marine Pollution Bulletin, available online.
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Donghao Li, Jongman Park, Jae-Ryoung Oh, 2001, “Silyl Derivatization of Alkylphenols, Chlorophenols, and Bisphenol A for Simultaneous GC/MS Determination” Anal. Chem.73,3089-3095.

Malaysia

Benjamin L. L. Tan, Mustafa Ali Mohd, 2003, “Analysis of selected pesticides and alkylphenols in human cord blood by gas chromatograph-mass spectrometer” Talanta, 00, 1-7.
Benjamin L. L. Tan, Normadiah M. Kassim, Mustafa Ali Mohd, 2003, “Assessment of pubertal development in juvenile male rats after sub-acute exposure to bisphenol A and nonylphenol”, Toxicology Letters 00, 1-10.

Philippines

Endocrine-disrupting phenols in selected rivers and bays in the Philippines, Evangeline C. Santiago, Charita S. Kwan, Baseline / Marine Pollution Bulletin 54 (2007) 1031–1071.

Singapore

Stéphane Bayen, Yinhan Gong, Hong Soon Chin, Hian Kee Lee, Yong Eu Leong, and Jeffrey Philip Obbard
Environmental Health Perspectives Volume 112, Number 15, November 2004
Androgenic and Estrogenic Response of Green Mussel Extracts from Singapore's Coastal Environment Using a Human Cell-Based Bioassay
Bayen, S., Wurl, O., Sivasothi, N., Subramaniam, K., Lee, H.K. & Obbard, J.P. Persistent Organic Pollutants in Mangrove Food Webs in Singapore. Chemosphere (2005) 61, 303-313.
Basheer, C., Jayaraman, A., Kee, M.K., Valiyaveettil, S. & Lee, H.K. Polymer-Coated Hollow-Fiber Microextraction of Estrogens in Water Samples with Analysis by Gas Chromatography-Mass Spectrometry. J. Chromatogr. A (2005) 1100, 137-143.
Basheer, C., Alnedhary, A.A., Rao, B.S.M., Valiyaveettil, S. & Lee, H.K. Development and Application of Porous Membrane-Protected Carbon Nanotube Micro-Solid-Phase Extraction Combined with Gas Chromatography/Mass Spectrometry. Anal. Chem. (2006) 78, 2853-2858.
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Zhang, J., Su, T. & Lee, H.K. Development and Application of Microporous Hollow Fiber Protected Liquid-Phase Microextraction via Gaseous Diffusion to the Determination of Phenols in Water. J. Chromatogr. A (2006) 1121, 10-15.
He, Y & Lee, H.K. Continuous Flow Microextraction Combined with High Performance Liquid Chromatography for the Analysis of Pesticides in Natural Waters. J. Chromatogr. A (2006) 1122. 7-12.
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Viet Nam

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Pham Hung Viet, Nguyen Thi Hanh, Nguyen Thuy Ngoc, Vo Thanh Le, Luong Manh Tuan, 2002, “Phan Tich Danh Gia Luong Vet Cua Cac Hop Chat Hydrocacbon Thom Da Vong Trong Khong Khi Tai Noi”, Hoi nghi Khoa hoc lien nganh ve “Khoa hoc va Cong nghe Moi truong”, 924-937.
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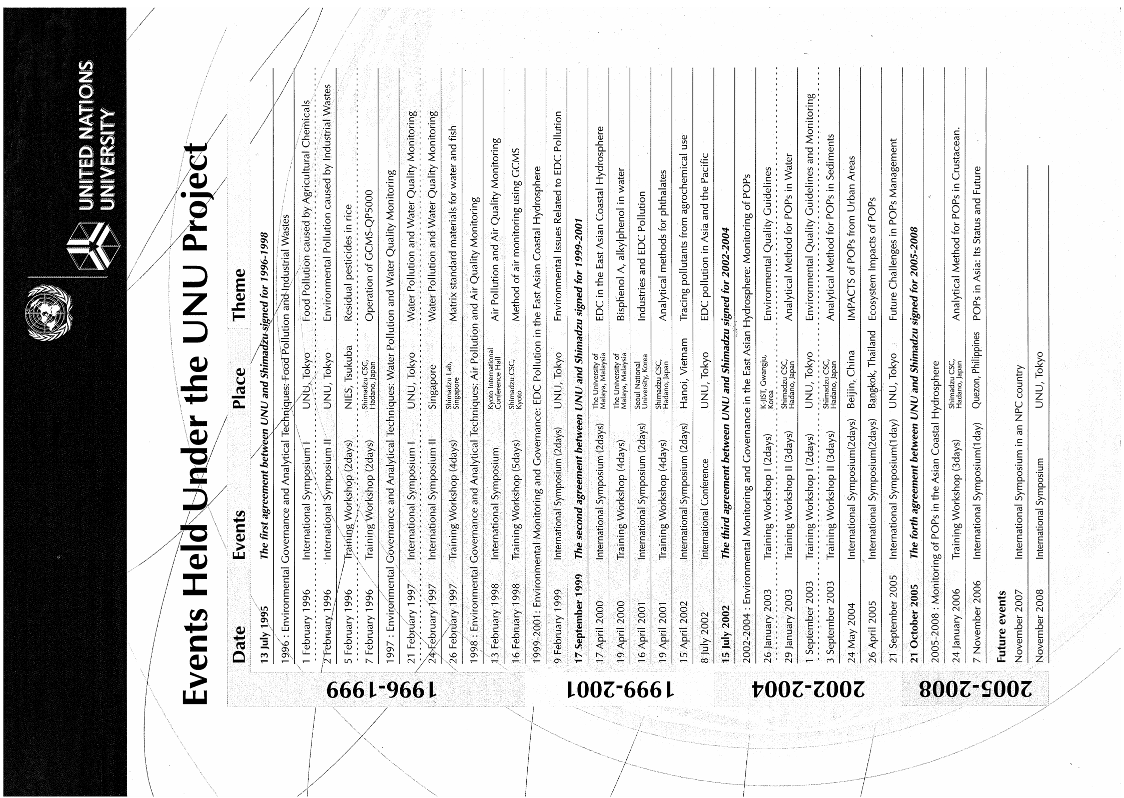
Pham Manh Hoai, Phan Tien Hung, Tran Thi Lieu, Pham Hung Viet, 2001, “Dien Bien Du Luong Cac Thuoc Tru Sau Co Clo Tai Cua Song Ba Lat, Viet Nam”, Tap chi Phan tich Hoa, Ly va Sinh hoc Tap 6, So1, 20-25.



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Events Held Under the UNU Project

	Date	Events	Place	Theme
1996-1999	**13 July 1995**	***The first agreement between UNU and Shimadzu signed for 1996-1998***		
	1996 : Environmental Governance and Analytical Techniques: Food Pollution and Industrial Wastes			
	1 February 1996	International Symposium I	UNU, Tokyo	Food Pollution caused by Agricultural Chemicals
	2 February 1996	International Symposium II	UNU, Tokyo	Environmental Pollution caused by Industrial Wastes
	5 February 1996	Training Workshop (2days)	NIES, Tsukuba	Residual pesticides in rice
	7 February 1996	Training Workshop (2days)	Shimadzu CSC, Hadano, Japan	Operation of GCMS-QP5000
	1997 : Environmental Governance and Analytical Techniques: Water Pollution and Water Quality Monitoring			
	21 February 1997	International Symposium I	UNU, Tokyo	Water Pollution and Water Quality Monitoring
	24 February 1997	International Symposium II	Singapore	Water Pollution and Water Quality Monitoring
	26 February 1997	Training Workshop (4days)	Shimadzu Lab, Singapore	Matrix standard materials for water and fish
	1998 : Environmental Governance and Analytical Techniques: Air Pollution and Air Quality Monitoring			
	13 February 1998	International Symposium	Kyoto International Conference Hall	Air Pollution and Air Quality Monitoring
	16 February 1998	Training Workshop (5days)	Shimadzu CSC, Kyoto	Method of air monitoring using GCMS
1999-2001	1999-2001: Environmental Monitoring and Governance: EDC Pollution in the East Asian Coastal Hydrosphere			
	9 February 1999	International Symposium (2days)	UNU, Tokyo	Environmental Issues Related to EDC Pollution
	17 September 1999	***The second agreement between UNU and Shimadzu signed for 1999-2001***		
	17 April 2000	International Symposium (2days)	The University of Malaya, Malaysia	EDC in the East Asian Coastal Hydrosphere
	19 April 2000	Training Workshop (4days)	The University of Malaya, Malaysia	Bisphenol A, alkylphenol in water
	16 April 2001	International Symposium (2days)	Seoul National University, Korea	Industries and EDC Pollution
	19 April 2001	Training Workshop (4days)	Shimadzu CSC, Hadano, Japan	Analytical methods for phthalates
	15 April 2002	International Symposium (2days)	Hanoi, Vietnam	Tracing pollutants from agrochemical use
	8 July 2002	International Conference	UNU, Tokyo	EDC pollution in Asia and the Pacific
2002-2004	**15 July 2002**	***The third agreement between UNU and Shimadzu signed for 2002-2004***		
	2002-2004 : Environmental Monitoring and Governance in the East Asian Hydrosphere: Monitoring of POPs			
	26 January 2003	Training Workshop I (2days)	K-JIST, Gwangju, Korea	Environmental Quality Guidelines
	29 January 2003	Training Workshop II (3days)	Shimadzu CSC, Hadano, Japan	Analytical Method for POPs in Water
	1 September 2003	Training Workshop I (2days)	UNU, Tokyo	Environmental Quality Guidelines and Monitoring
	3 September 2003	Training Workshop II (3days)	Shimadzu CSC, Hadano, Japan	Analytical Method for POPs in Sediments
	24 May 2004	International Symposium(2days)	Beijin, China	IMPACTS of POPs from Urban Areas
	26 April 2005	International Symposium(2days)	Bangkok, Thailand	Ecosystem Impacts of POPs
	21 September 2005	International Symposium(1day)	UNU, Tokyo	Future Challenges in POPs Management
2005-2008	**21 October 2005**	***The forth agreement between UNU and Shimadzu signed for 2005-2008***		
	2005-2008 : Monitoring of POPs in the Asian Coastal Hydrosphere			
	24 January 2006	Training Workshop (3days)	Shimadzu CSC, Hadano, Japan	Analytical Method for POPs in Crustacean.
	7 November 2006	International Symposium(1day)	Quezon, Philippines	POPs in Asia: Its Status and Future
	Future events			
	November 2007	International Symposium in an NPC country		
	November 2008	International Symposium	UNU, Tokyo	