ENVIS Centre for Biogeochemistry

NEWSLETTER

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EDITORIAL

This issue of the newsletter comes on the occasion of World Environment Day on June 5. This year the capital is already experiencing acute water and power problems and we need not stress again the implication to the other environmental issues confronting us now. What worth Tsunami finally bidding adieu and an active volcano suddenly showering ashes and gases in the Andaman chain of island, we are always on our toe to face one or other kind of nature's outburst.

Our regular future on book of abstract of Biogeochemistry was brought out early this year and it has been receiving very good reader response.

Following the visit of a Review committee and their comments, our website has been modified and readers are encouraged to get back to us on their input about the content of our site as well as the mode of presentation. Due to space limitation, we are unable to give scope for animation based web design but we have now made our site user friendly.

We are also gradually introducing tutorial type of information on various aspects of Biogeochemistry in our website. In our site we have also now given direct links to several site that have database related to our thrust areas.

This issue of the newsletter contain usual futures such as invited article, book review, recent publications and conference news. Our website now contain the complete list of our ENVIS Library holding.

Happy reading!

Prof. V. Subramanian
Editor

INVITED ARTICLE

Environmental Applications of X-Ray Spectroscopy

By

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Environmental Analysis

Global concern over the environment continues to drive increasing levels of environmental legislation. In particular there are demands for a reduction in the release of toxic elements, notably heavy metals, into the environment, and into everyday things like our food. One of the challenges in attempting to monitor the use and disposal of these elements is the fact that they are used in such a diverse range of industries. This is particularly apparent in the fast-growing Asia Pacific economies. So, whether it is the disposal of refuse, the use of fertilisers in agriculture or the reclamation of contaminated land, any analytical technique used to monitor elements of environmental concern needs to be able to deal with both highly diverse material types and element concentrations.

Fortunately, X-ray analytical techniques, and X-ray fluorescence (XRF) spectrometry in particular, are ideally suited to environmental applications. This is because:

- they are able to handle a wide variety of sample types and physical forms – for example, solid pieces, pressed powders, loose powders and granules, liquids and thin films/filters can all be placed directly into the XRF instrument and analysed with a single calibration
- sample preparation is simple, inexpensive and fast
- calibrations cover a wide dynamic concentration range (from 100% to sub-ppm levels)
- they are able to target a wide range of elements, particularly those of environmental concern
- measurements are highly precise
- analysis is non-destructive – making re-analysis of critical samples possible

Not surprisingly, therefore, XRF is being used to support a wide range of environmental applications in geology, agriculture, use of fossil and secondary fuels, industrial emissions monitoring, recycling, waste disposal, land reclamation, catalyst production/recycling, and electronics and electrical appliance manufacture.

X-ray diffraction (XRD) is also of value in another area of environmental and pollution concern. As developing nations begin to legislate and enforce environmental safety and protection regulations, and as companies institute corporate and socially responsible actions, pollutants and health hazards such as contaminated air or respirable silica, can be analyzed for the presence of poisons or carcinogens, using XRD.

Choosing Instrumentation

A number of different types of XRF instrument are available, from bench-top energy-dispersive (EDXRF) spectrometers to flexible, high performance wavelength-dispersive (WDXRF) instruments. Choosing a system for a particular application depends on a number of criteria, including:

- range of elements under test
- sample throughput
- speed of analysis
- required detection limits

EDXRF spectrometers, such as PANalytical's Epsilon 5, have been developed specifically to address the heavy-metal analysis requirements of many environmental applications. Excelling in medium-heavy metals analysis, the Epsilon 5 is ideal for the measurement of contaminants in soils and sludges. The Axios-Advanced, a WDXRF spectrometer, has the capability of being able to analyse carbon and nitrogen in addition to the normal range of medium-heavy metals. This

added capability is achieved by the incorporation of light-element specific PX-crystals to the basic instrument configuration. The Axios-Advanced can therefore provide a full range of elemental analysis options from carbon to uranium.

At the other end of the spectrum are benchtop spectrometers, such as PANalytical's MiniPal. These instruments are most suitable for situations where sample throughput is low, say around 20 samples in a normal work-day. Their portability means they are ideal for on-site applications such as the appraisal of contaminated mine-site waters.

Application Example 1: Analysis of common heavy-metal contaminants in soils and sludges using EDXRF
The work described below was carried out to show the accuracy and precision of EDXRF when analyzing heavy metal contaminants in soils and sludges. Measurements are accurate and precise and the stability of the instrument is such that individual calibrations can be used for months. All analysis was performed on an Epsilon 5 spectrometer. A series of soil and rock standards were used to calibrate the application.

These were the GSS-, GRS-, and GSD-series geochemical reference materials (Institute of Geophysical and Geochemical Prospecting, People's Republic of China), together with NIST-2709, NIST-2710, NIST-2711 (National Institute of Standards and Technology, USA) and the soil standards SO-1, SO-2, SO-3 and SO-4 (Canadian Certified Reference Materials Project). Three powdered sewage sludge reference materials (WT-H, WT-L and WT-M) were also added to the calibration.

The samples themselves were analysed in the form of pressed powder pellets – prepared by oven drying, then pulverising for 20 minutes in a planetary ball mill with 20% wax/styrene additive, and 12 g of the mixture finally being pressed into 36mm diameter pellets using a hydraulic press operated at 20 tonnes pressure. The measuring program was set up in the software using the Epsilon 5 Wizard – the latter guides the user through the steps required to calibrate the application and proposes the polarization targets and instrument settings for optimal analysis of the selected analyte. For each of these instrument conditions a measurement time of 200s was specified.

The accuracy of the results is presented in Table 1 and analytical precision, in terms of the repeatability of 20 consecutive measurements and the reproducibility of measurements carried out over 10 days, is shown in Table 2.

Typical detection limits for heavy metals in soil are given in Table 3. Lower detection limits for Cd can be obtained with longer measuring times. These results show the EDXRF to be capable of analysing low levels of heavy metal contaminants in both soils and sludges. Measurements are accurate and precise and the stability of the instrument is such that individual calibrations can be used for months.

Table 1: Accuracy

	Calibration Range (ppm)	Calibration RMS (ppm)	GSS-1		GSD-7		WT-H (Sludge)	
			Certified	Measured	Certified	Measured	Certified	Measured
As	0.23-412	1.39	33.5	34.7	84	84	146	146
Cd	0.03-55	0.63	1.05	0.92	4.30	4.39	55	56
Cr	4.8-1340	17.44	62	58	122	119	1340	1256
Cu	4.1-3140	10.53	21	20.9	38.0	36.0	3140	3106
Mo	0.09-92	0.93	1.40	1.22	1.40	1.02	78	74
Ni	1.6-1140	5.42	20.4	19.7	53	54	1140	1147
Pb	4.4-2290	7.57	98	89	350	358	2290	2278
Zn	16-6360	10.98	680	679	238	246	6360	6359

The Lower Limit of Detection is calculated from

$$LLD = \frac{3}{s} * \sqrt{\frac{Rb}{t}}$$

Where:
s = sensitivity (cps/ppm)
Rb = background countrate (cps)
t = live time (s)
** The LLDs quoted are typical for soil samples. LLDs for individual samples vary according to sample composition.*

Table 2: Analytical Precision

Table 3: Detection Limits

Application Example 2: Analysis of carbon and nitrogen in soils using WDXRF

The work was carried out to illustrate how WDXRF can compare to dedicated, combustion based carbon and nitrogen analysers. All measurements were performed using an Axios-Advanced spectrometer. The application was calibrated using the GSS-series of geochemical reference materials (Chinese Institute of Geophysical and Geochemical Prospecting, PRC). Soil samples were oven-dried (105°C), then pulverised for 20 seconds in a planetary ball mill and pressed at 20 Nm-2 for 30 seconds into 40 mm diameter pellets, before being loaded into the spectrometer. The accuracies of the calibrations for carbon and nitrogen are presented in Table 4. The calibration RMS value is a statistical comparison (1 sigma) of the certified chemical concentrations of the standards with the concentrations calculated by the regression of the calibration procedure. Analytical repeatability data for ten separate preparations of a soil sample are shown in Figures 1 and 2. Detection limits are shown in Table 5.

These results - comparable to those achieved by dedicated, combustion-based carbon and nitrogen analysers - clearly show that Axios-Advanced is capable of analysing carbon and nitrogen at the low levels commonly found in soil samples (0.5 – 2.5% C and 0.02 – 0.15% N). Measurements are accurate and precise and the method benefits from simple, essentially hazard-free sample preparation.

Table 4: Calibration Quality

Table 5: Typical detection limits obtainable in 100s

Application Example 3: Analysis of Cr, Fe, Cu and Zn in waters from eontaminated mine sites using EDXRF

The work described below was carried out to prove the performance of EDXRF in analysing both low atomic number elements (using Kα lines) and high atomic number elements (using Kα lines) in water. All measurements were performed on a MiniPal spectrometer. Synthetic standard solutions were mixed in various ratios and pipetted into P1 ‘de Kat’ cells and loaded into the spectrometer. Two excitation conditions were used to acquire the spectra (Table 6). All elements were measured using their Kα lines except Ag, Cd, Sn and U, which were measured using their Lα1 lines, and Pb where the Lβ1 line was used. Table 7 shows the calibration results for Cr, Fe, Cu and Zn, and Figure 3 shows how repeat analyses of a single sample of contaminated water give excellent repeatability.

Table 6: Excitation conditions

Elements	kV	µA	Filter	Medium	Counting time(sec)
As, Bg1, Cu, Pb, U, Zn	30	100	Mo	Helium	300
Ag, Al, Bg2, Cd, Cr, Fe, Ni, Sn	15	30	Al_thin	Helium	300

The data show that EDXRF is clearly capable of analysing waters precisely, allowing rapid assessment of contaminated drainage from mine sites and landfills. Analysis of both low atomic number elements (using Kα lines) and high atomic number elements (using Lα lines) is possible.

Conclusion

While these examples give a clear insight into the use of XRF spectroscopy for the analysis of both heavy-metal contaminants and light elements of environmental interest the technique has the potential for numerous additional environmental applications. Advanced software programmes and simple sample preparation requirements combine to make XRF a cost-effective user-friendly technique suitable for routine use.

Figure 1: Analytical repeatability: carbon data for ten preparations of a soil sample

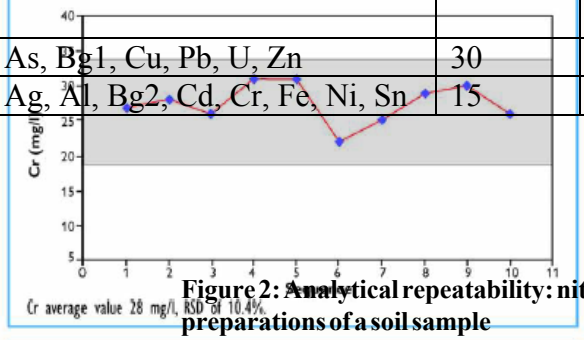
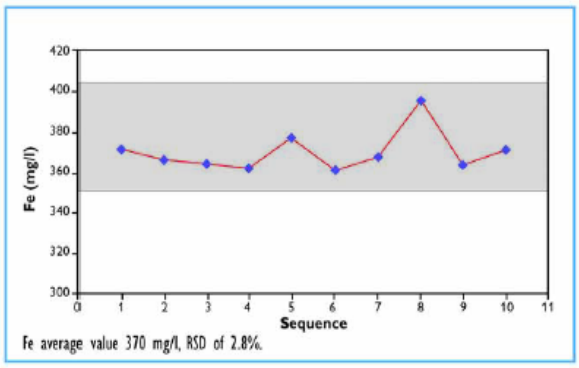


Figure 2: Analytical repeatability: nitrogen data for ten preparations of a soil sample



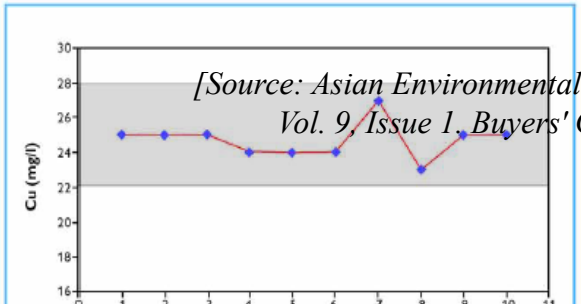


Figure 3: 10 analyses of contaminated water

[Source: Asian Environmental Technology, Vol. 9, Issue 1, Buyers' Guide 2005]

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BOOK REVIEW

Proceeding of the National workshop cum conference held from 19th to 20th May, 2005 in Delhi on "Emerging Technology for Controlling Groundwater Pollution: Drinking water Perspectives". Published by Allied Publishers Private Limited, New Delhi, 66p.

This Souvenir cum Book of Abstracts is edited by Dr. Ashok. K. Keshari of IIT, New Delhi. The publication is brought out with the support from IIT Delhi, Ministry of Environment and Forests, AICTE, RGNDWM and CPCB.

The growing population along with urbanization, the ground water contamination problems are also growing at alarming rate and are becoming complicated to tackle. The ground water problems vary in magnitude and size depends upon the availability of the resource and the utility. The ground water is getting polluted by natural and anthropogenic use depend upon the land use pattern changes (land fill impacts on ground water quality) over the decades. These problems are to be addressed holistically with an eye on the judicial utilization of ground water resource and along with various ways to recycle the contaminated waters such as bioremediation and other management options, ground water regulation and protection. These are the issues addressed in this two days seminar and brought out as a volume.

The topics covered and discussed under various themes are: Ground water problems and issues; Technology for controlling ground water pollution; Ground water quality monitoring and hydro geological investigations, Drinking water supply and treatment technology; ground water chemistry and sustainable development of ground water resources.

The volume incorporates the very important issues by very eminent and experiences people working on this field. Some of the interesting papers are- ground water contamination with poisonous chemicals, ground water quality deterioration around land fill sites in Delhi, fertilizer impacts on ground water quality, adaptation of modeling techniques in controlling groundwater pollution, methodology to estimate nitrogen loading from Septic systems, geo-statistical analysis of ground water quality, GIS based ground water pollution study for NCR Delhi, hydro geochemical and isotopic characteristics of ground water of NCT Delhi, ground water quality variations in hard rocks and sedimentary terrains including mining areas like L.St mining areas of India, innovative insitu water treatment technologies for drinking water technology for drinking water supply, iron minerals in polluted aquifers: their environmental significance, As in ground waters of Bengal Delta plains, application of factor analysis to ground water contaminations, new technologies in water shed management, water harvesting structures in Rajasthan and artificial recharging for ground water security and other many other interesting papers on ground waiter management.

This volume covers wide range of aspects of ground water management from various parts of India with contribution form various scientist and researchers working currently on this aspect. It is a must for any institution and universities to have this volume for the ready reference for the people who are working on ground water aspects.

By **Dr. AL. Ramanathan, SES, JNU.**

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NEWS BRIEFS

Listed below are some of the recent news items collected from various newspapers, reports, etc., and the detailed news items are updated on our website.

- Biomedical waste programme in TN, The Asian Age, May 28, 2005
- Crores down the drain but no saving Ganga, The Hindu, May 28, 2005
- City of Wonder is a city of Waste, The Hindu, May 25, 2005
- Poison plants get away with murder, The Telegraph, May 25, 2005
- Report lists pollution hotspots, The Hindu, May 24, 2005
- Low-cost technology converting seawater into potable water launched, The Hindu, May 24, 2005
- Goa delays removal of River Princess, The Indian Express, May 20, 2005
- Uttarkashi breaks disaster jinx with development bouquet, The Pioneer, May 17, 2005
- Coral reef barrier effective against tsunami: Study, The Pioneer, May 17, 2005
- Saudis lobby to limit chemical liability, International Herald Tribune, Bangkok, May 17, 2005
- Project to assess carbon absorption in rubber plants, Business Line, May 16, 2005
- Plan(t)s to mop up air pollutants, The Pioneer, May 10, 2005
- Melting glaciers may cause water shortage, The Pioneer, May 9, 2005
- Green Care products soon, The New Indian Express, May 5, 2005
- Subansiri project to control State floods, The Assam Tribune, May 3, 2005
- Pollution unlimited, The Kashmir Times, Jammu, April 28, 2005
- Study shows environmental pollutants affect sperm, Planet Ark (Internet), Australia, April 28, 2005
- Action plan for Ganga basin not enough: CPCB, The Indian Express, April 27, 2005
- DMC develops monsoon model, The New Indian Express, April 27, 2005
- Commercial use of fly ash can reduce pollution threat, Business Line, April 25, 2005
- Expect draft environment policy next week, The Indian Express, April 23, 2005
- Brahmaputra ranks 4th in historic floods, The Assam Tribune, April 23, 2005
- Govt urged to act on effluent discharge into Yamuna. The Tribune, April 21, 2005
- Marble work causing pollution. Central Chronicle, April 20, 2005
- Green body to review air pollution action plan. The New Indian Express, April 15, 2005
- A solution to environmental problems. The New Indian Express, April 9, 2005
- Villagers protest work on desalination plant. The New Indian Express, April 6, 2005
- Rs 10 crore plan for reconstruction of shelter belts. The Hindu, April 6, 2005
- Kangra belt prone to massive quakes: seismologist. The Tribune, April 5, 2005
- A great earthquake may be 'overdue' in the Himalayas. The Hindu, April 5, 2005
- Metro planning to sell 'carbon credits'. The Hindu, April 4, 2005
- State agencies blamed for delay in hazardous waste disposal. The Hindu, April 4, 2005
- Population growth, urbanisation main pollutants. The Assam Tribune, April 4, 2005
- Scientists to study quake-prone Himalayas. The Tribune, April 3, 2005
- Humans, animal 'major polluters'. Business Line, April 2, 2005
- Eyebrows raised over recycled water. The Age (Internet), Australia, April 2, 2005
- London left underwater without climate action. Edie (Internet), UK, April 1, 2005

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SOME RECENT PUBLICATIONS

Some Recent Publications in Journals Related to Environmental Biogeochemistry

A. Porteous (2005): Why energy from waste incineration is an essential component of environmentally responsible waste management. Waste Management. 25(4): 451-459.

Aimin Zhou, Hongxiao Tang and Dongsheng Wang (2005): Phosphorus adsorption on natural sediments: Modeling and effects of pH and sediment composition. Water Research. 39(7): 1245-1254.

Alan H. Stern (2005): A review of the studies of the cardiovascular health effects of methylmercury with consideration of their suitability for risk assessment. Environmental Research. 98(1): 133-142.

Alena L. Pribyl, James H. Mccutchan, William M. Lewis and James F. Saunders III (2005): Whole-system estimation of denitrification in a plains river: a comparison of two methods. Biogeochemistry, 73(3): 439 – 455.

Andrew J. Hamilton (2005): Species diversity or biodiversity? Journal of Environmental Management. 75(1): 89-92.

Andrew Turner and Edward Mawji (2005): Octanol-solubility of dissolved and particulate trace metals in contaminated rivers: implications for metal reactivity and availability. Environmental Pollution. 135(2): 235-244.

Anjali Srivastava, B. Sengupta and S.A. Dutta (2005): Source apportionment of ambient VOCs in Delhi City. Science of The Total Environment. 343(1-3): 207-220.

Anoop Singh, S.B. Agrawal and Dheeraj Rathore (2005): Amelioration of Indian urban air pollution phytotoxicity in Beta vulgaris L. by modifying NPK nutrients. Environmental Pollution. 135(1): 385-395.

Atsuyuki Ohta, Noboru Imai, Shigeru Terashima and Yoshiko Tachibana (2005): Application of multi-element statistical analysis for regional geochemical mapping in Central Japan. Applied Geochemistry. 20(5): 1017-1037.

C. Valeo, S.H. Skone, C.L.I. Ho, S.K.M. Poon and S.M. Shrestha. 2005: Estimating snow evaporation with GPS derived precipitable water vapour. Journal of Hydrology. 307(1-4): 196-203.

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Carla S. Neuberger and George R. Helz (2005): Arsenic(III) carbonate complexing. Applied Geochemistry. 20(6): 1218-1225.

Charles R. Hart, Larry D. White, Alyson McDonald and Zhuping Sheng (2005): Saltcedar control and water salvage on the Pecos river, Texas, 1999–2003. Journal of Environmental Management. 75(4): 399-409.

Christopher M.B. Lehmann, Van C. Bowersox and Susan M. Larson (2005): Spatial and temporal trends of precipitation chemistry in the United States, 1985–2002. Environmental Pollution. 135(3): 347-361.

Cong Tu and Lena Q. Ma (2005): Effects of arsenic on concentration and distribution of nutrients in the fronds of the arsenic hyperaccumulator Pteris vittata L.. Environmental Pollution. 135(2): 333-340.

Daniel P. Faith (2005): Global biodiversity assessment: integrating global and local values and human dimensions. Global Environmental Change Part A. 15(1): 5-8.

Declan Conway (2005): From headwater tributaries to international river: Observing and adapting to climate variability and change in the Nile basin. Global Environmental Change Part A. 15(2): 99-114.

Devendra P. Saroj, Arun Kumar, Purnendu Bose, Vinod Tare and Yashodhan Dhopavkar (2005): Mineralization of some natural refractory organic compounds by biodegradation and ozonation. Water Research. 39(9): 1921-1933.

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E. Peters, H.A.J. van Lanen, P.J.J.F. Torfs and G. Bier (2005): Drought in groundwater—drought distribution and performance indicators. Journal of Hydrology. 306(1-4): 302-317.

G. Bianchini, E. Marrocchino, R. Tassinari and C. Vaccaro (2005): Recycling of construction and demolition waste materials: a chemical–mineralogical appraisal. Waste Management. 25(3): 149-159.

H.V. Ram Babu and M. Prasanti Lakshmi (2005): A Note on Geophysical Mapping of the Granulite Terranes Surrounding the Godavari Basin. Journal of The Geological Society of India. 65(2): 211-216.

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Recently held/forthcoming Conferences and Workshops

Training Course on Decision support system for Water Resources Planning during September 19-23, 2005. Organised by National Institute of Hydrology, Roorkee. Contact: Dr. Sharad K. Jain, Scientist F and Head, WRS Division, National Institute of Hydrology, Roorkee - 247667. Tel: (01332) 276417, Fax: (01332) 272123. Email: skj@nih.ernet.in.

Summer School on Environmental Advocacy during May 20-29, 2005 at Kodaikanal, Tamil Nadu. Contact: The Registrar, Mother Theresa University, Kodaikanal, Tamil Nadu. Phone: (04542) 241122. Email: treescella@yahoo.co.in. Website: www.treesmadurai.org.

Emerging Technology for Controlling Groundwater Pollution: Drinking Water Perspectives during May 19-20, 2005. Organised by IIT Delhi, New Delhi. Contact: Dr. Ashok K. Keshari, Department of Civil Engineering, IIT, Hauz Khas, New Delhi – 110 016. Phone: 26591236(O), 26591548®, Fax: 26581117. Email: akeshari@hotmail.com.

National Seminar on Natural Resources, Health Care & Natural Hazards during June 2-3, 2005 at Vishakhapatnam. Contact: Prof. M. V. Subba Rao, Dept. of Environmental Sciences, Andhra University, 50-120-8/1, Seethammadhara North Extn. Visakhapatnam – 530 013. Phone: 91-891-2552888. Fax: 91-891-2560479. Email: mvsubbarao_enra@yahoo.co.in.

Closing water and resources cycles: options for gas treatment during June 26-July 1, 2005 at Wageningen, The Netherlands. Contact: Euro Summer School Secretariat, Markus Lenz, Sup-department of Environmental Technology, Wageningen University, P. O. Box 8129, 6700 EV Wageningen, The Netherlands. Phone: +31-317-484993, Fax: +31-317-842108. Email: resource.ete@wur.nl.

Training Programme on Sedimentation of Reservoirs control & Management during July 05-08, 2005 at ESCI Campus, Hyderabad. Contact: Dr. S. Nagabhushana Rao, Director, Engineering Staff College of India, Old Bombay Road, Gachi Bowli, Hyderabad – 500 032. Phone: 23000465/ 23000466, Fax: 040-23000336. Email: esci_ic@yahoo.com, esci_ic@escihyd.org. Website: www.escihyd.org.

International Symposium on Recent Advances in Water Resources Development and Management (RAWRDM-2005). During November 23-25, 2005 at IIT, Roorkee. Contact: Dr. Deepak Khare or Dr. S. K. Mishra, Department of Water Resources Development and Management, Indian Institute of Technology, Roorkee – 247 667. Phone: 91-1332-285393, 285457. Fax: 91-1332-27173, 273560. Email: kharefwt@iitr.ernet.in.

Eleventh National Water Convention on May 11, 2005 at New Delhi. Organised by National Water Development Agency(NWDA). Sponsored by Ministry of Water Resources, GOI. Contact: Shri. K. P. Gupta, Superintending Engineer, NWDA, Room No. 211, Palika Bhawan, R. K. Puram, New Delhi - 110 066. Phone: 91-11-24672398, 24671681, Fax: 24671681, 24672398, Email: tfsp@rediffmail.com.

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