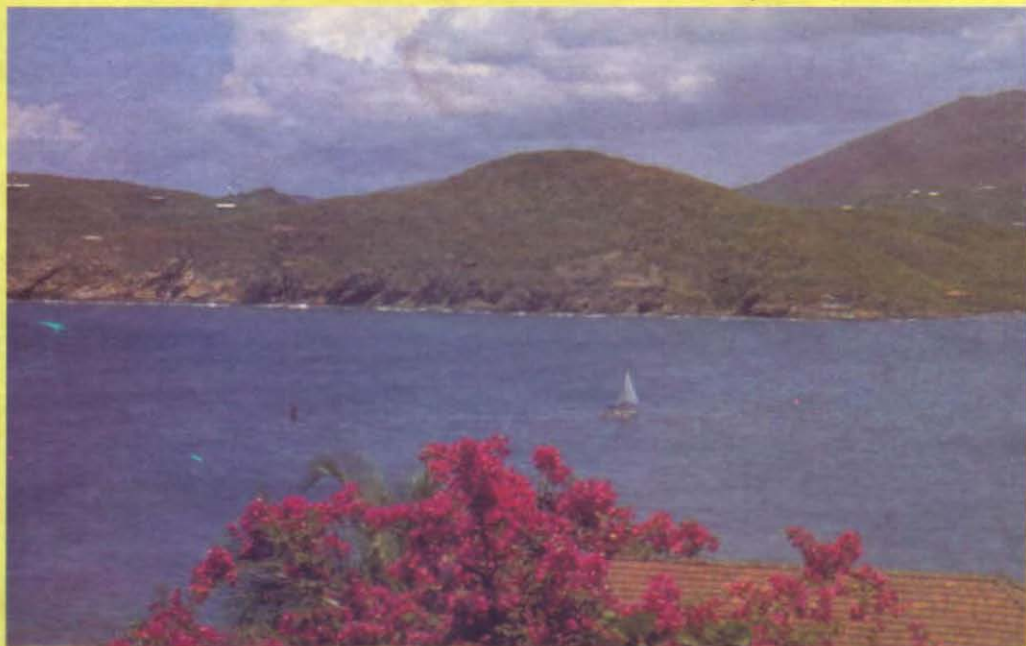


PROCEDURAL MANUAL (Ver. 1.0)
Measurement of
Radiocarbon & Tritium in waters and
Cesium-137 & Lead-210 in sediments



National Institute of Hydrology

Roorkee - 247 667 (UA)

2002

PROCEDURAL MANUAL (Ver. 1.0)
Measurement of
Radiocarbon & Tritium in waters and
Cesium-137 & Lead-210 in sediments



National Institute of Hydrology

Roorkee - 247 667 (UA)

2002

Preface	i
Acknowledgments	iii

Part 1. Measurement of Radiocarbon in Natural Water Samples

1.0	Introduction	1-1
1.1	¹⁴ C Dating Procedure	1-4
2.0	Groundwater Sampling for ¹⁴C Dating	1-4
2.1	Chemicals and Sampling equipment	1-5
2.2	Sampling procedure	1-5
3.0	Sample Preparation	1-7
3.1	Preparation of CO ₂	1-10
3.1.1	Measurement of CO ₂ volume	1-11
3.1.2	CO ₂ extraction from Oxalic acid	1-12
3.2	Benzene Synthesis	1-12
3.2.1	Preparation of Acetylene from carbon dioxide	1-12
3.2.2	Trimerization of Acetylene into Benzene	1-14
3.2.2.1	Activation of Catalyst (Al ₂ O ₃ +Cr ₂ O ₇)	1-14
3.2.2.2	Trimerization of acetylene	1-15
3.2.2.3	Recovery of Benzene trapped in catalyst	1-16
3.3	Reaction Summary	1-17
3.4	CO ₂ Absorption Method	1-18
3.4.1	Absorption Procedure	1-20
3.4.2	Cleaning of Carbosorb absorption column	1-21
4.0	Radioactivity Measurement	1-22
4.1	Sample Size	1-22
4.2	Procedure of cleaning of Teflon vials	1-23
4.3	Standard Sample	1-23
4.3.1	Primary Modern Reference Standard	1-23
4.3.2	New Reference Standard	1-24
4.3.3	Sample for Measurement of Background Activity	1-24
4.4	Counting Procedure	1-26
4.5	Quenching	1-27
4.6	Noise reduction in Liquid Scintillation Counter	1-27
4.7	Data filtering	1-28
4.8	Counting and Data Synthesis	1-30
5.0	Age Determination	1-31
5.1	Age Equation and Counting Errors	1-31
5.2	Age Correction for Contamination	1-33
5.2.1	DIC in unsaturated zone	1-34
5.2.2	Carbonate dissolution	1-36
5.2.3	¹³ C as a tracer to correct ¹⁴ C activity in DIC:	1-38

5.3	¹⁴ C Age correction Models	1-38
5.3.1	Statistical Correction:	1-40
5.3.2	Alkalinity correction model	1-40
5.3.3	Chemical Mass Balance (CMB) correction	1-41
5.3.4	¹³ C mixing model	1-43
5.3.5	Matrix Exchange Model	1-46
5.4	Reporting of ¹⁴ C age	1-48

Part 2. Measurement of Tritium (³H) in natural water samples

1.0	Introduction	2-1
2.0	Sampling	2-2
3.0	Pretreatment of Samples	2-2
4.0	Enrichment of Samples	2-3
5.0	Distillation of Enriched Samples (Secondary-distillation)	2-6
6.0	Preparation for counting	2-7
7.0	Counting	2-7
7.1	Minimum Detectable Concentration of Tritium	2-13
7.2	Estimation of Tritium Concentration	2-14
7.3	Determination of Error in Tritium Measurement	2-16
7.4	Drawbacks in the Estimation of Tritium by Spiking Method	2-19

Part 3. Estimation of rates of sedimentation and useful life of lakes / reservoirs by ¹³⁷Cs dating technique

1.0	Introduction	3-1
2.0	Scope	3-2
3.0	Sample Collection	3-2
3.1	Need for representative samples	3-2
3.2	Optimum number of samples	3-2
3.3	Sampler description	3-4
3.4	Sample preservation, storage and numbering system	3-4
3.5	Precautions	3-5
3.6	Maps, sample records	3-5
4.0	Sample Processing	3-5
4.1	Determination of bulk density	3-5
4.2	Oven drying	3-6
4.3	Determination of dry density	3-6
4.4	Moisture content	3-6
4.5	Powdering and sample homogenization	3-7
4.6	Determination of organic contents	3-7
5.0	Activity Measurement	3-8

5.1	Some basics of ^{137}Cs measurement by γ -spectrometry	3-8
5.2	HPGe detector - Multi-channel Gamma ray system	3-8
5.3	MAESTRO32 program	3-9
5.3.1	Preset Limits	3-10
5.3.2	Adjust controls	3-10
5.3.3	Spectrum Stabilisation	3-11
5.3.4	Spectrum Calibration	3-12
5.3.5	Activity Calibration	3-12
5.3.6	Developing protocol	3-14
5.4	Precautions	3-15
5.5	Estimation of measurement errors	3-15
6.0	Data Processing	3-16
6.1	^{137}Cs activity	3-16
6.2	Activity matching	3-17
6.3	Estimation of Rate of sedimentation	3-17
6.3.1	Uncertainty in estimated rates	3-18
6.4	Estimating the useful life of a lake	3-18
6.4.1	Estimation of mean depth of a lake	3-19
6.4.2	Uncertainty in estimated mean depth of a lake	3-19
6.4.3	Uncertainty in estimated useful life of a lake	3-19
References		3-20

Part 4. Estimation of rates of sedimentation in lakes / reservoirs by ^{210}Pb dating technique

1.0	Introduction	4-1
2.0	Scope	4-3
3.0	Sample Collection and Processing	4-4
4.0	Measurement of ^{210}Pb concentration in sediment samples	4-4
4.1	Lead-210 separation from sediment samples	4-5
4.2	Lead-210 measurement using Liquid Scintillation Counter	4-7
5.0	Lead-210 measurement by other methods	4-9
5.1	Lead-210 measurement by Alpha Spectrometry	4-9
5.1.1	Digestion	4-9
5.1.2	Filtration	4-10
5.1.3	Plating	4-10
5.2	Lead-210 measurement by Gamma Spectrometry	4-11
6.0	Models for ^{210}Pb profiles interpretation	4-12
6.1	Constant initial concentration (CIC) model	4-12
6.2	Constant rate of supply (CRS) or constant flux (CF) model	4-14
6.3	Constant Flux Constant Sedimentation Rate (CFCS) model:	4-15
References		4-17