



## SPECIFICATION

### **Supply and delivery of a Stable Isotope Ratio Mass Spectrometer (IRMS) coupled to an elemental analyser (EA) and gas chromatography (GC)**

#### 1. Scope

This Specification describes the requirements for an isotope ratio mass spectrometer (IRMS) coupled to an elemental analyser (EA) and gas chromatography (GC) via interface(s) with helium carrier gas flow permitting on-line continuous-flow (CF) operation (hereinafter referred to as the "System").

The End-User of the System is the Atomic Energy Board of Sri Lanka, Colombo, Sri Lanka.

The System shall enable the End-User Institute to establish a national centre for nuclear agriculture, to develop and implement programmes on the use of nuclear technology applications in the field of soil, water and plant nutrient studies, crop variety improvement and associated management technologies.

#### 2. Applicable Documents

The following documents shall be applicable for this Specification to the extent specified hereinafter:

- 2.1. CODEX Guidelines on the use of Mass Spectrometry (MS) for Identification, Conformation and Quantitative Determination of Residues, CAC/GL 56-2005.
- 2.2. ISO/IEC 17025 General requirements for the competence of testing and calibration laboratories.

In the event of conflict between the documents listed above and the content of this Specification, the content of this Specification shall take precedence to the extent of the conflict.

#### 3. Definitions, Acronyms, and Abbreviations

The following definitions, acronyms, and abbreviations shall apply throughout this Specification unless defined otherwise hereinafter:

IRMS = Isotope ratio mass spectrometer

GC = Gas chromatography

EA = Elemental analyser

EQ = Carbon dioxide/hydrogen gas equilibration unit

PC = Personal Computer

#### 4. Functional and Performance Requirements

The System shall meet the following functional and performance requirements:

- 4.1. Be able to perform isotope ratio analysis of gases at natural isotope composition range for elements H-C-N-O (see Table 1 below), with an option to extend it later, if required, to gases containing sulfur, e.g. S, SO<sub>2</sub> and SF<sub>6</sub>. The IRMS shall be able to perform isotope analysis both in Dual Inlet (DI) and Continuous Flow (CF) modes of sample gas introduction.



- 4.2. IRMS Faraday cup collectors, magnetic sector-field analyser, electron impact ion source and continuous-flow inlet.
- 4.3. The collector configuration shall permit analyses of gases and ions of the given mass-to-charge ratios ( $m/z$ ) with sufficient overlap of major and minor peaks (Table 1).

Table 1:

Gas Configuration	Isotope ratio(s) to be measured	Masses to be measured
H <sub>2</sub>	$\delta^2\text{H}$	2, 3
CO	$\delta^{13}\text{C}$ , $\delta^{18}\text{O}$	28, 29, 30
N <sub>2</sub>	$\delta^{15}\text{N}$	28, 29, 30
CO <sub>2</sub>	$\delta^{13}\text{C}$ , $\delta^{18}\text{O}$	44, 45, 46
O <sub>2</sub>	$\delta^{17}\text{O}$ , $\delta^{18}\text{O}$	32, 33, 34

- 4.4. The electron impact ionization source shall at a minimum fulfil the parameters as outlined in Table 2. The ion source shall be computer controlled, supporting manual and automatic tuning.
- 4.4.1. A magnetic sector field generated by an electromagnet, which allows simultaneous C, H, N, analysis.
- 4.4.2. A universal triple collector, suitable for N<sub>2</sub>, O<sub>2</sub>, CO<sub>2</sub>, and measurements and the collectors for H/D to measure H<sub>2</sub>.
- 4.4.3. Automatic ion source control electronics to allow optimal “tuning” for multiple reference gas species.
- 4.4.4. A reliable, secure and robust vacuum system.

Table 2.

Sensitivity	Sensitivity CF mode: $\leq 1200$ molecules per CO <sub>2</sub> + ion (factory test results accepted).
Relative isotope ratio dependence on sample size (linearity)	$\Delta R/R < 0.02 \text{ ‰ per nA } (\delta^{13}\text{C})$ $\Delta R/R < 0.2 \text{ ‰ per nA } (\delta^2\text{H})$
Precision continuous-flow	$^2\text{H}(\text{H}_2) \leq 0.2 \text{ ‰}$ $^{13}\text{C}(\text{CO}_2), ^{15}\text{N}(\text{N}_2) \leq 0.06 \text{ ‰}$ $^{18}\text{O}(\text{CO}_2) \leq 0.06 \text{ ‰}$ $^{18}\text{O}(\text{CO}) \leq 0.3 \text{ ‰}$
H3+correction factor	$\leq 8\text{ppm} / \text{nA (HD)}$
H3+stability	$\leq 0.03\text{ppm}$
Mass Resolution	(10% valley definition), $m/\Delta m \geq 100$



4.5. The GC peripheral / Gas chromatograph shall meet the following requirements:

- 4.5.1. The IRMS shall be coupled to a gas chromatography system (GC) via interface by combustion/pyrolysis for  $^{13}\text{C}$  and  $^2\text{H}$ . An easy and rapid switching between the combustion/pyrolysis modules is required.
- 4.5.2. The GC shall perform split/splitless and on-column injections into 0.32mm and 0.25mm columns to analyse organic molecules of variable polarity (from hydrocarbons to fatty acids).
- 4.5.3. The automatic injector shall perform injection volumes from 0.5 to 2  $\mu\text{l}$  from a total sample volume of 50 to 1000  $\mu\text{l}$ . The tray shall hold 100 conical bottom (tapered) vials of 10 X 32 mm.
- 4.5.4. The precision required with this interface for the measurement of  $^{13}\text{C}$  and  $^2\text{H}$  shall be respectively of 0.1 ‰ and 2 ‰ for sequences of 12 measurements on standards of the alkanes type (C18 - C31), sterols and fatty-acids. Each sequence of measurement shall be carried out by increasing the concentration of the standard solutions (from 1  $\mu\text{g ml}^{-1}$  to 1000  $\mu\text{g ml}^{-1}$ ). For sample performance parameters please refer to table 3.

Table 3:

GC Analytical performance based on analysis of:	Isotope ratio	Value
n-alkane mixture	$^{13}\text{C}/^{12}\text{C}$ (as $\text{CO}_2$ ) (1 $\sigma$ )	$\leq 0.2$ ‰ (n = 5)
	D/H (as $\text{H}_2$ ) (1 $\sigma$ )	$\leq 3.0$ ‰ (n = 5)
vanillin	$^{18}\text{O}/^{16}\text{O}$ (as CO) (1 $\sigma$ )	$\leq 0.8$ ‰ (n = 5)
caffeine	$^{15}\text{N}/^{14}\text{N}$ (as $\text{N}_2$ ) (1 $\sigma$ )	$\leq 0.5$ ‰ (n = 5)

- 4.5.5. The System shall not use chemical traps for water removal.
- 4.5.6. Fitted with a Flame Ionisation Detector (FID) or a Mass Selective Detector (MSD).
- 4.5.7. Heated Split/split-less injector (ambient to 350°C).
- 4.5.8. Temperature programmable oven (ambient to 350°C).
- 4.5.9. All temperature settings and gradients shall be programmable from the GC instrument software.



4.6. The Elemental Analyser shall meet the following requirements:

- 4.6.1. The IRMS shall be coupled to a solids/liquids elemental analyser (EA) preparation module with an auto-sampler to determine accurately the elemental weight percentages of N, C and  $^{13}\text{C}$  and  $^{15}\text{N}$ . Shall also determine,  $^{18}\text{O}$  and  $^2\text{H}$  with easy and rapid switching between the different combustion/pyrolysis modules.
- 4.6.2. Allow measurements from large amounts of sample material (solid material from 0.1 mg to 250 mg, filter samples of up to 47 mm diameter), in a sample receptor of a minimum of 10 mm diameter. No diameter reduction shall exist in the connection from the sample receptor to the combustion reactor.
- 4.6.3. The measurements shall cover a range of concentrations for the elements and operate over a wide dynamic range with simultaneous analyses of C, N and S. Accurate measurement of low concentrations of the major elements is needed (in the order of a few  $\mu\text{g}$  for C and N).
- 4.6.4. The auto-sampler shall have a configuration to avoid sample contamination and atmospheric contamination.
- 4.6.5. Precision required with the interface for the measurement of  $^{13}\text{C}$ ,  $^{15}\text{N}$  and  $^{34}\text{S}$  shall be at least and respectively 0.1 ‰, 0.15 ‰, and 0.2 ‰ for sequences of 12 measurements on reference material and standards for elemental analysis (acetanilide, sulfamic acid, benzoic acid etc.). Each sequence of measurement shall be carried out by increasing material size (from 0.1 mg to 250 mg) and concentrations of standards. These measurements,  $^{13}\text{C}/^{12}\text{C}$  (as  $\text{CO}_2$ ) combustion 50  $\mu\text{g}$  C ( $1\sigma$ )  $\leq 0.1$  ‰ ( $n = 10$ ).
- 4.6.6.  $^{15}\text{N}/^{14}\text{N}$  (as  $\text{N}_2$ ) combustion 50  $\mu\text{g}$  N ( $1\sigma$ )  $\leq 0.15$  ‰ ( $n = 10$ ).
- 4.6.7.  $^{18}\text{O}/^{16}\text{O}$  (as CO) High Temp conversion 50  $\mu\text{g}$  O ( $1\sigma$ )  $\leq 0.3$  ‰ ( $n = 10$ ).
- 4.6.8.  $^2\text{H}/^1\text{H}$  (as  $\text{H}_2$ ) High Temp conversion 50  $\mu\text{g}$  H ( $1\sigma$ )  $\leq 2.0$  ‰ ( $n = 10$ ).
- 4.6.9. The continuous-flow inlet shall have an isolation valve.
- 4.6.10. Have a mass filter to allow hydrogen isotope analysis under high-helium background conditions (continuous flow with helium carrier gas).
- 4.6.11. Computer controlled switching between two amplifier resistors on each ion-beam channel shall be possible allowing analysis of samples with highly enriched minor isotopes.
- 4.6.12. Be of bench-top design
- 4.6.13. The amplifier shall offer a wide voltage range providing a 1000 fold working beam intensity.



- 4.6.14. Be able to achieve a m/z 18 water background level of less than  $5 \times 10^{11}$  nA (200uA trap current).
- 4.6.15. Offer automatic tuning for all reference gas species and permit manual tuning if required.
- 4.6.16. Be able to automatically switch between GC and EA inlets.
- 4.6.17. Capable of producing CO<sub>2</sub>, N<sub>2</sub> and SO<sub>2</sub> in standard mode for simultaneous measurement of  $\delta^{13}\text{C}$ ,  $\delta^{15}\text{N}$  and  $\delta^{34}\text{S}$  from a single direct analysis of a bulk food sample at natural percent element levels e.g. bovine muscle protein.
- 4.6.18. Capable of producing H<sub>2</sub> and CO in standard mode for simultaneous measurement of  $\delta^2\text{H}$  and  $\delta^{18}\text{O}$  from a single bulk food sample pyrolysis.
- 4.6.19. Permit accurate analysis of oxygen isotopes from CO in nitrogen containing food samples, e.g. bovine muscle protein. It shall provide complete baseline separation to avoid any isobaric interference at m/z 28.
- 4.6.20. The pyrolysis furnace shall be capable of maintaining a constant temperature of  $\geq 1400^\circ\text{C}$ .
- 4.6.21. Have a thermal conductivity detector.
- 4.6.22. Integrated PC-software control for unattended analysis of batches of samples.
- 4.6.23. Permit automated sample peak dilution.
- 4.6.24. Carousel capacity  $\geq 50$  samples.
- 4.6.25. Automated reference gas injection.
- 4.6.26. Automatic determination and application of H<sub>3</sub>+correction.
- 4.7. The GC Auto sampler shall meet the following requirements:
  - 4.7.1. Integrated PC-software control for unattended analysis of batches of samples.
  - 4.7.2. Capable of Internal Standard Injection Mode.
  - 4.7.3. Sample loading  $\geq 50$  vials of 2 ml capacity
  - 4.7.4. Syringe range of 1 - 10  $\mu\text{L}$ .
  - 4.7.5. Variable injection volume of 0.1  $\mu\text{L}$  to 5  $\mu\text{L}$ .
- 4.8 The GC-IRMS Interface shall meet the following requirements:
  - 4.8.1. An interface for quantitative combustion and pyrolysis/high temperature conversion of individual organic compounds and for independent reference gas injection.



- 4.8.2. User friendly integrated PC-software control for synchronized (auto-sampler, GC, and interface) acquisition, analysis, storage, retrieval and manipulation of methods and data files.
- 4.8.3. Reactor for combustion to at least 1000°C.
- 4.8.4. Reactor for pyrolysis at high temperature conversion to at least 1450 °C.
- 4.8.5. Ability to use modified reactor tube packings e.g. to analyse fluorinated derivatives.
- 4.8.6. Automated recharge of oxidation reactor with O<sub>2</sub>.
- 4.8.7. Automated reference gas injection.
- 4.8.8. Automatic determination and application of H<sub>3</sub><sup>+</sup> correction.
- 4.9 The PC, Software and Printer shall meet the following requirements:
  - 4.9.1 PC with a minimum of 8 Gb RAM and 1Tb HD capacity for efficient routine operation.
  - 4.9.2 PC Monitor of at least 17”.
  - 4.9.3 Colour printer.
  - 4.9.4 Data acquisition software for continuous flow measurements and off-line data manipulation.
  - 4.9.5 Software shall be user friendly, provide automatic, multi-point isotopic calibration and output sample delta values with respect to internationally accepted reference scales.
  - 4.9.6 Software shall permit automatic drift and blank corrections if required. Drift correction shall be possible between definable sample positions within a sequence.
- 4.10 The following consumables shall be included:
  - 4.10.1. Spare filament.
  - 4.10.2. Spare reactors for compound-specific isotope analyses of 2H, 13C, 15N and 18O using GC interface.
  - 4.10.3. Spare reactors for EA-IRMS isotope analyses of 2H,13C, 15N,18O.
  - 4.10.4. Reagents, catalysts and capsules.
  - 4.10.5. Range of appropriate tools for IRMS and peripheral maintenance.
- 4.11 Un-interruptible Power Supply (UPS):
  - 4.11.1. Input: AC 220-240V, 50 Hz 40A max.
  - 4.11.2. Output: AC 220V, 50 Hz 27A max.
  - 4.11.3. Battery: 192 v Dc, 16 x12 V17AH.



5. Marking

The System shall have all safety markings in English language.

6. Packing

The System, for the shipment by air to the End-User, shall be packed in accordance with international standards that are applicable for the shipment by air of this kind of equipment.

7. Quality Requirements

7.1 The System shall be manufactured, shipped and installed in accordance with the Suppliers ISO quality assurance system or an equivalent quality assurance system.

7.2 The Supplier shall document the compliance with this quality assurance system.

7.3 The instrument shall operate under normal environmental conditions of temperature and humidity levels in Sri Lanka.

8. Testing and Acceptance

8.1 The System, after installation, shall be tested by the Supplier together with the End-User to demonstrate that the performance meets the manufacturer's performance specifications and the minimum requirements specified herein as determined by the IAEA and the End-User.

8.2 The results of the testing of the System shall be documented by the Supplier in an acceptance protocol that shall be signed by the End-User.

9. Installation and Training

9.1 The Supplier shall install the System at the laboratory of the End-User.

9.2 The Supplier shall provide one week's training for up to three staff of the SLAEB in the operation and maintenance of the System immediately after the installation of the System or at a mutually agreeable time and location.

10. Deliverable Data Items

The Supplier shall provide at least two complete sets of operation and servicing manuals and technical drawings in the English Language.