

SRA Quick Start Guide

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Source Rock Analyzer (SRA):

Quick Start Guide (from SRA_QSG_371T_draft)

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Introduction

This quick start guide contains operational procedures for the Source Rock Analyzer (SRA). See the *SRA Analyzer User Guide and Advanced User Guide* for additional information.

Pyrolysis analysis is conducted on dried, ground sediments or borehole source rock for safety monitoring as well as for scientific purposes. The analysis yields information about thermal maturation and source of hydrocarbons at the drilled site, as well as petroleum potential. The data obtained from the Rock-Eval/TOC analyses are defined by the following parameters.

| Parameter | Unit | Definition/Indication |
|-----------|------------------------------------|--|
| S1 | mg HC/g rock | Free hydrocarbons (HC) |
| S2 | mg HC/g rock | HC resulting from kerogen cracking and/or high molecular weight HC |
| S3 | mg organic CO ₂ /g rock | Produced during low-temp pyrolysis of kerogen |
| S4 | mg carbon/g rock | Convertible carbon + residual carbon |
| Tmax | °C | Stage of maturation of the organic matter |
| TOC | wt% C/unit wt rock | Total organic carbon: composed of convertible carbon |
| HI | Normalized HC content | Hydrocarbon index: used to determine kerogen type (I, II, or III) |
| OI | Normalized oxygen content | Oxygen index: used to determine continental vs. other types of immature organic matter |
| PI | NA | Production index: conversion of kerogen into free HC; used to determine petroleum HC |
| S1/TOC | NA | Used to identify source or reservoir rocks |
| PC | NA | Pyrolysis carbon: used to determine if a source rock is oil-prone or gas-prone |
| RC | NA | Residual carbon: carbon present in kerogen (low potential to generate HC) |
| S2/S3 | NA | Used to determine kerogen type in absence of TOC data |

Apparatus, Reagents, & Materials

Hardware

The SRA system consists of the following main components (*Figure 1*): Autosampler, Main control unit: oven and detector temperature control unit and gas flow controllers, Infrared (IR) section, Combustion with gas separation and FID (pedestal, oven, and conversion with FID), and Cahn electrobalance dual balance system



Figure 1. Main Components of the SRA System.

Laboratory Apparatus for Sample Preparation

- Autosampler tray (1/4)
- Forceps, small spatula, and sample filler
- Crucible and crucible holder
- Weighing paper
- Recording chart

Reagents

- Methanol
- Compressed gases: H₂, 60–80 psi, He, 60–80 psi, Air, GC grade, 60–80 psi, and laboratory grade, 60–100 psi

Standards

- Colorado Oil Shale Silica (COSS) house standard
- Standard material (99986)
- Weatherford Laboratories SRA Rock Standard 533

Sample Preparation

Sediments and sedimentary rock samples logged into the LIMS system as **SRANL** are analyzed by SRA. During sample preparation, keep in mind that sample container numbers must correlate with autosampler place numbers. Freeze-dried bulk samples are crushed by hand or electric mill, then homogenized using a mortar and pestle. Prepare a weighing chart similar to that found below, and record sample ID, mass, and container number in the weighing chart.

| Text ID (parent) | Text ID (child) | Mass (mg) | Container/Autosampler number | LIMS code | SRA code |
|------------------|-----------------|-----------|-----------------------------------|-----------|----------|
| Blank | | | 99 (Calibration blank; always 99) | | BLK |
| STD99986 | OTHR137987 | 65.0 | 100 (Calibration STD; always 100) | QAQCSTD | STD |
| OTHR2764599 | OTHR346972 | 78.26 | 1 | UNK | TPH |
| OTHR2765092 | OTHR130746 | 70.63 | 2 | UNK | TPH |
| : | : | : | : | : | : |
| CV1-99986 | OTHR136947 | 65.0 | 5 | QAQCCV | TPH |
| OTHR348962 | OTHR147989 | 74.85 | 6 | UNK | TPH |
| OTHR389012 | OTHR384702 | 85.03 | 7 | UNK | TPH |
| : | : | : | : | : | : |
| CV2-99986 | OTHR427034 | 65.0 | 10 | QAQCCV | TPH |

Sample Analysis

Preparing the Instrument

The SRA must warm up for 3 hr before operation. Perform the following steps to prepare for sample acquisition (Note: the onboard technician will warm up the instrument):

1. Open H₂, He, O₂, and air gas lines. **Note:** Verify gas gauge pressures using the gas flow meter.
2. Turn on the SR Analyzer power and PC.
3. Individually test the H₂, O₂, He, and oxidation air gas lines. With a gas flow meter verify gas flows correspond to the following values: H₂: 65 ml/min, He: 100 mL/min, Air: 300 mL/min, Oxidation Air: 250 mL/min. H₂, and O₂ lines are beneath the FID cover. The helium and oxidation air line is connected to the base of the sample pedestal.
4. Double-click the **TStationAcq.exe** shortcut icon on the desktop to open the software *Main Menu*.
5. Click **Preview** to confirm oven and FID temperatures. Values will be <300°C. If after 10–15 min oven and FID temperatures have not increased, proceed to Steps 5 and 6.
6. Click **Configuration** and select the **Temperatures** tab
7. Set desired temperature and click **Send Temperatures Immediately**. **Note:** Except for this action, do not change any values on any tabs in the *Configuration* screen.

Setting up the Software

Prepare the software for data acquisition by creating a data folder, editing the method (if needed), and editing the sequence table.

Creating a New Folder

Data (CSV and ROW files) are saved into the expedition-specific data folder with the PAS file (sequence record) and PAR file (method) with respect to each sequence (batch).

Open the Data folder at C:\Program Files\Thermal Station\Data, and create a new folder named EXPxxx where xxx is the expedition number (or xxxx in the rare cases where the expedition has a letter designation with it).

Editing the Method

The method does not need to be edited for regular onboard measurements (i.e., safety monitoring) except for the standard information. The onboard laboratory specialist will edit the fields in the **Standard** tab if the standard material changes. In that case, one blank, one standard, and one known sample (house standard) must be measured to confirm calibration. The default method is **toc_580.SRA.sram**

Editing the Sequence Table

A sequence table represents an analytical batch. After each batch, the sequence table is saved into the expedition-specific data folder. A typical sequence table contains the following:

–**Data File** name (one for each QA/QC and unknown sample). Examples: Blank: OTHR468061, Standard: PWDR39980915, Calibration verification standard: PWDR468051, Unknown sample: textID = LIMS text ID.

–**Sample ID:** must be identical to the Text ID or the LIMS uploader will reject the file

–**Method:** **toc_580SRA.sram**

–**C#:** container number (must match autosampler place number, as recorded in the weight recording chart)

–**Sample Weight:** from weight recording chart (mg)

–**Acq Type:** acquisition type: **BLK** = cal blank, **STD** = cal standard, **TPH** = unknown or QA/QC samples

Edit the sequence table as follows:

| | |
|----|--|
| 1. | Click Sequence Editor on the <i>Main Menu</i> . |
| 2. | Fill in the <i>Data File</i> , <i>Sample ID</i> , <i>Method</i> , <i>C#</i> , <i>Sample Weight</i> , and <i>Acq Type</i> fields using the weight recording chart as a reference. |
| 3. | Click Exit at the top right of the <i>Sequence Editor</i> screen, then click Yes to save the sequence table. |
| 4. | Save the sequence into _folder C:\Program Files\Thermal Station_ (same level as the data folder), as EXPxxx.PAS . |

Running Samples

To analyze samples, fill in the *Acquisition Setup* screen and confirm sample information on the *Analysis* screen.

| | |
|----|---|
| 1. | Select <i>Main Menu</i> > Acquisition to open the <i>Acquisition Setup</i> screen. Fill in the fields: – <i>Operator</i> : operator's last name (same as that to access LIMS). – <i>Sample Selection</i> : select the samples to be run from the list shown on the screen. |
| 2. | Specify the data folder:–Select parent folder EXPxxx from the explorer window on the screen.–Enter subfolder name EXPxxx_sequence number in <i>Data Folder</i> field. |
| 3. | Click Next to open the <i>Analysis</i> screen. |
| 4. | Click Info at the bottom right to open detail information and confirm the information for the first sample. |
| 5. | Click Start Sequence above the sample window to start the run. The software will ask for confirmation of SRA function after starting the first measurement (e.g., autosampler arm position, turn on the FID, etc.). |

QA/QC

Calibrating the Instrument

The first sequence for an expedition must include a Blank and a Standard to calibrate the SRA each time the instrument is turned on after power off (e.g., loss of power). After this initial calibration, a Blank and a Standard should be measured once per day to verify calibration of the SRA. If the standard is outside the acceptable limits, the IR cell should be recalibrated.

Analytical Batch (Sequence)

An analytical batch is referred to as a sequence. Each sequence contains the following samples:

–**Calibration blank** (CB): contains no material; mass = 0; include at the beginning of the sequence

–**Calibration standard** (CS): ~80 mg of CS material; include 1 per 50 samples or every day

–**Calibration verification standard** (CV): 1/10 samples, either CS (60 mg), COSS (80 mg), or STD 999986 (60 mg), or STD 533 (PWDR3998091, 100 mg)

–**QA/QC blank**: empty, used crucible (mass = 1) or empty crucible as unknown (TPH)

–**Unknown sample(s)** (TPH): 50–70 mg powdered sample (max = 200 mg)

Data Handling

Results are stored in the LIMS database associated with an analysis code and an analysis component, component value, and component unit. Analysis codes and their components, definitions, and units are listed below. Use Spreadsheet Uploader to transfer data from a Sequence Report to LIMS. Complete fields in Spreadsheet Uploader with the following parameters: Analysis: SRANL, Instrument: SRA, Display Flag: T, and the rest according to the table below.

Table 1: LIMS components and units

| Analysis | Component | Definition | Unit | LIMS Component Unit |
|----------|-----------|---|----------------------------|---------------------|
| SRANL | HI | Hydrogen index (S2 x 100/TOC) | mg HC/g TOC | NONE |
| | OI | Oxygen index (S3 x 100/TOC) | mg CO ₂ /g TOC | NONE |
| | PC | Pyrolysis carbon (0.83 x [S1 + S2]/10) | mg HC/g rock | MG_PER_G |
| | PI | Production index ([S1/(S1 + S2)] x 100) | — | NONE |
| | S1 | HC vaporize < 330°C (free oil) | mg HC/g rock | MGHC_P_GC |
| | S2 | HC vaporize > 330°C (kerogen) | mg HC/g rock | MGHC_P_GC |
| | S3 | Carbon dioxide evolved | mg CO ₂ /g rock | MGHC_P_GC |
| | S4 | Residual carbon | mg HC/g rock | MGHC_P_GC |

| | | | |
|------|-------------------------------|-----|------------|
| mass | Sample mass | Mg | MILLIGRAMS |
| tmax | Maximum S2 (peak) temperature | °C | CELSIUS |
| toc | Total organic carbon | wt% | PCT_WEIGHT |

SRA results are printed to a PDF file, which, along with the corresponding CSV file, are uploaded into the LIMS.

Creating a PDF File

| | |
|----|---|
| 1. | Double-click the TStationProcessor shortcut icon to open the <i>Processor</i> screen. |
| 2. | Select File > Open Raw File to open data. |
| 3. | Select the data file to be reviewed and select File > Print to create the PDF file. Note: The PDF file must be uploaded to LIMS with the CSV file. |
| 4. | The file name should be same as the CVS file. Save the PDF file in the same folder as the CSV file. |

Uploading Data to LIMS

| | |
|----|---|
| 1. | Click SRAnalyzer Loader shortcut icon to open the <i>SR Loader</i> screen. |
| 2. | Open CSV and PDF files to be uploaded to LIMS. |
| 3. | Confirm <i>Text_id</i> and <i>Operator</i> name. Edit these fields if needed. |
| 4. | Click Calculate to calculate values for <i>PI</i> , <i>PC</i> , <i>OI</i> , and <i>HI</i> . Calculated values are shown on the right side of the screen. |
| 5. | Click Load to upload data to LIMS. |

Health, Safety, & Environment

All laboratory specialists must complete required safety training before operating the SRA instrument.

Compressed Gases

- Always wear safety glasses when handling and using compressed gases.
- Hydrogen gas is highly flammable. Be careful with ignition sources and oxidizers.
- Check for leaks in compressed gas lines.
- Use only wrenches approved for use on compressed gas tanks and lines.
- Ground all equipment and lines associated with hydrogen usage.

High Temperatures

- High oven and detector temperatures (325°–600°C) are reached during operation. Use proper heat protective equipment when operating the instrument.
- Make sure the FID cover is always installed during operation to protect from burns.