Source Rock Analyzer (SRA): Quick Start Guide

K. Fujine (10/20/09); revised/approved by D. Houpt (8/13/13)

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. 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 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: H_2, 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)

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 H_2, He, O_2, and air gas lines. **Note**: Verify gas gauge pressures using the gas flow meter.
2. Turn on the SR Analyzer power and PC.
3. Double-click the **TStationAcq.exe** shortcut icon on the desktop to open the software *Main Menu.*
4. 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.
5. Click **Configuration** and select the **Temperatures** tab.
6. 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**.

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: PWDR5, 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_580**

–***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 *Main Menu*.
2. Fill in the *Data File, Sample ID, Method, C#, Sample Weight,* and *Acq Type* fields using the weight recording chart as a reference.
3. Click **Exit** at the top right of the *Sequence Editor* 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 *Main Menu* > **Acquisition** to open the *Acquisition Setup* screen. Fill in the fields:
   –*Operator*: operator's last name (same as that to access LIMS).
   –*Sample Selection*: 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 *Data Folder* field.
3. Click **Next** to open the *Analysis* 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.

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)

–**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

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 *Processor* 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 *SR Loader* screen.
2. Open CSV and PDF files to be uploaded to LIMS.
3. Confirm *Text_id* and *Operator* name. Edit these fields if needed.
4. Click **Calculate** to calculate values for *PI, PC, OI,* and *HI*. 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.