Request for Information (RFI) Technical Specification
RFI No. 2022-10-7-MARTIN R1
Design, Build, Integrate and Deliver an Automated Platform for Soil Extractions and Analyses

Battelle Pacific Northwest Division as operator of Pacific Northwest National Laboratory (PNNL) requests information to begin planning for the design and installation of four components of a larger platform for automated soils analysis to be purchased by the Environmental Molecular Sciences Laboratory (EMSL) user facility. The completed system would include a software platform to schedule and implement the automated workflow, and interact with EMSL's LIMs system, NEXUS. The four custom design components would support: (i) automated sieving (2 or 4 mm mesh) of soil samples, and weighing and aliquoting multiple sub-samples (Component 1, Figure 1); (ii) parallel extractions on soil samples, pH testing and acidification (Component 2, Figure 2, Protocol 1 (Appendix 1); (iii) total organic carbon/nitrogen analysis (Component 3, Protocol 3 (Appendix 2); and (iiii) solid phase extraction (Component 4, Figure 4, Appendix 3).

Multiple outputs from Component 1 will be handed off to staff for separate non-automated analyses (Figure 1). Aliquots from Component 1 will be also move into Component 2 for extractions and conditioning. Samples leaving Component 2 (Figure 2) will leave the system and enter one or more total organic carbon/nitrogen analysis (Component 3) and automated solid phase extraction stations (Component, 4). Samples leaving Component 4 move directly to one or more mass spectrometry platforms via autosamplers.

The completed automated system will replace the collection of spatially isolated, human-administered systems used to conduct the suite of analyses that comprise the organic matter capability now. The automated organic matter and soil organic matter analysis system should add modularity, expandability, robotic liquid handling, robotic sample handling, sample tracking, remote monitoring and scheduling, and comprehensive metadata and data collection through integration of scheduling software with EMSL's NEXUS (LIMS) system.

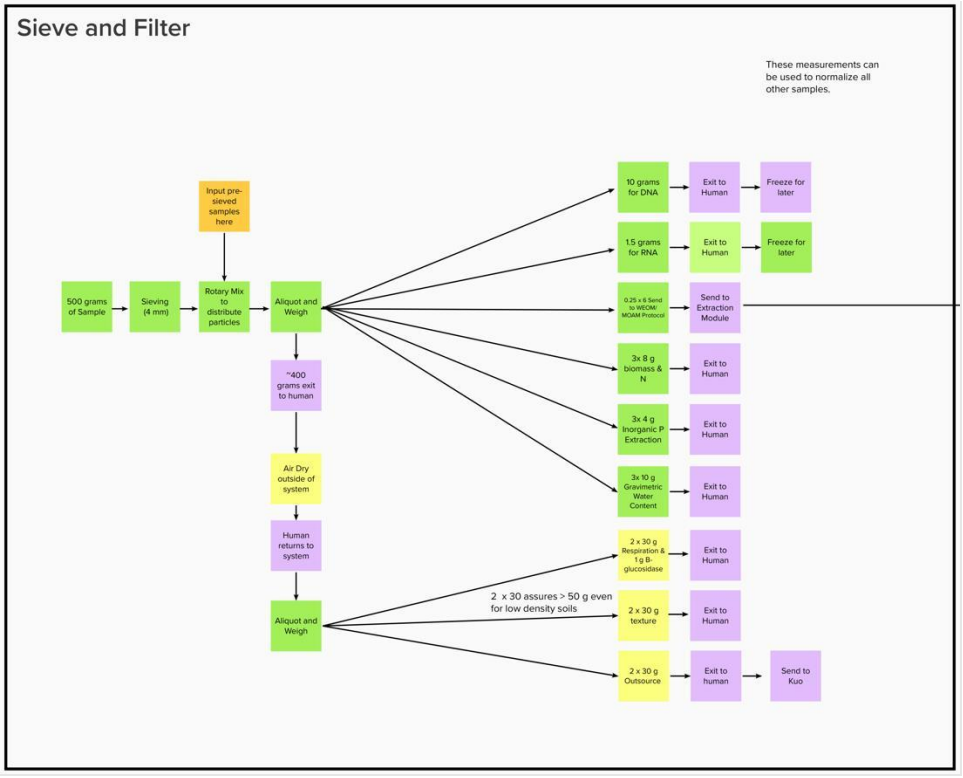

Figure 1: Process diagram of Component 1 of the automated soil analysis system.

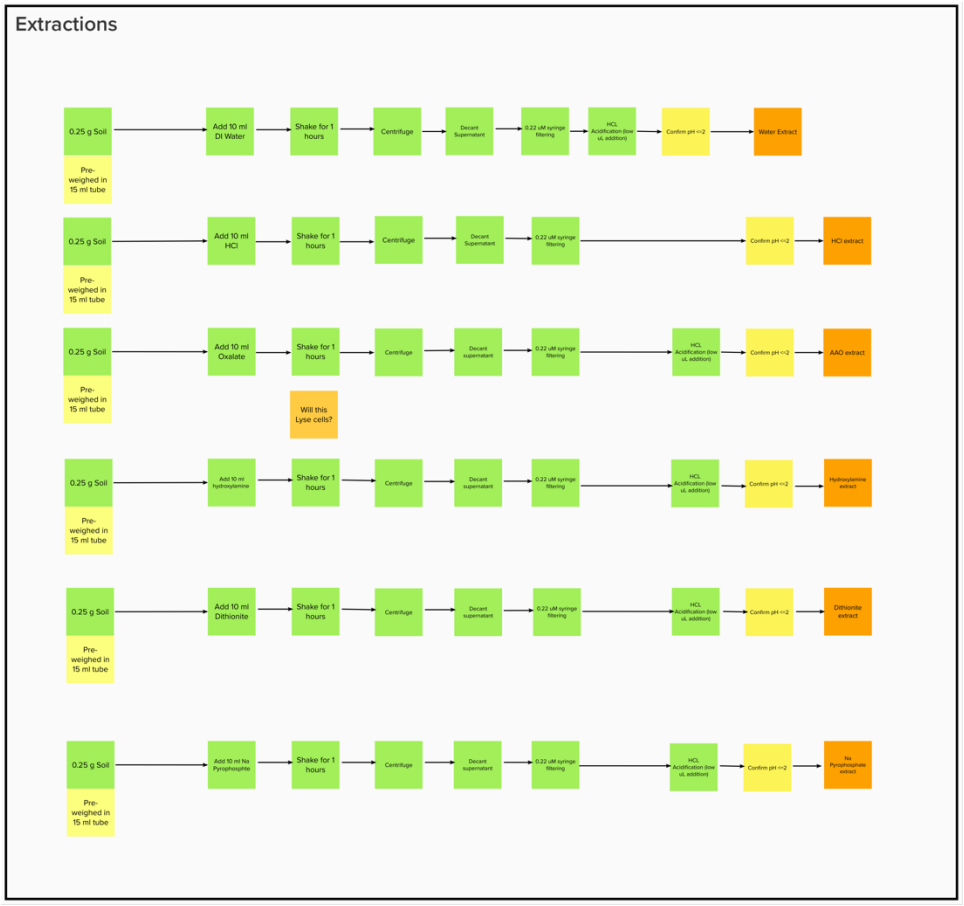

Figure 2: Process diagram of Component 2 of the automated soil analysis system showing use of 15 ml tubes and and low volumes of soil and extractants. System should be designed for use with either 15 ml or 50 ml tubes. Use of 50 ml tubes would increase solvent volumes to 30 mls/extraction. The protocol can be found in Appendix 1.

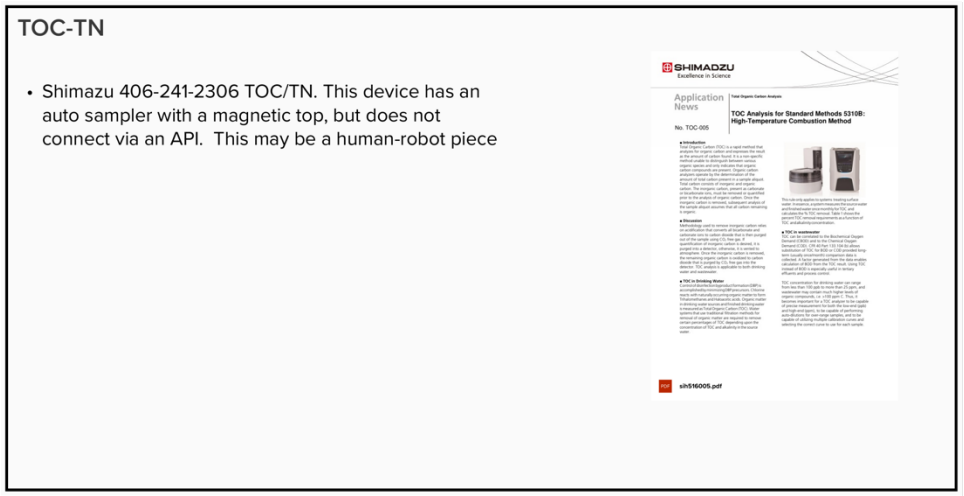

Figure 3: Instrument diagram of Component 3 of the automated soil analysis system. This component would contain or more Shimazu 06-241-2306 Total Organic Carbon/Total Nitrogen analysis devices, or similar device

by another manufacturer, integrated with robotics. Appendix 2 contains the general workflow description for this component.

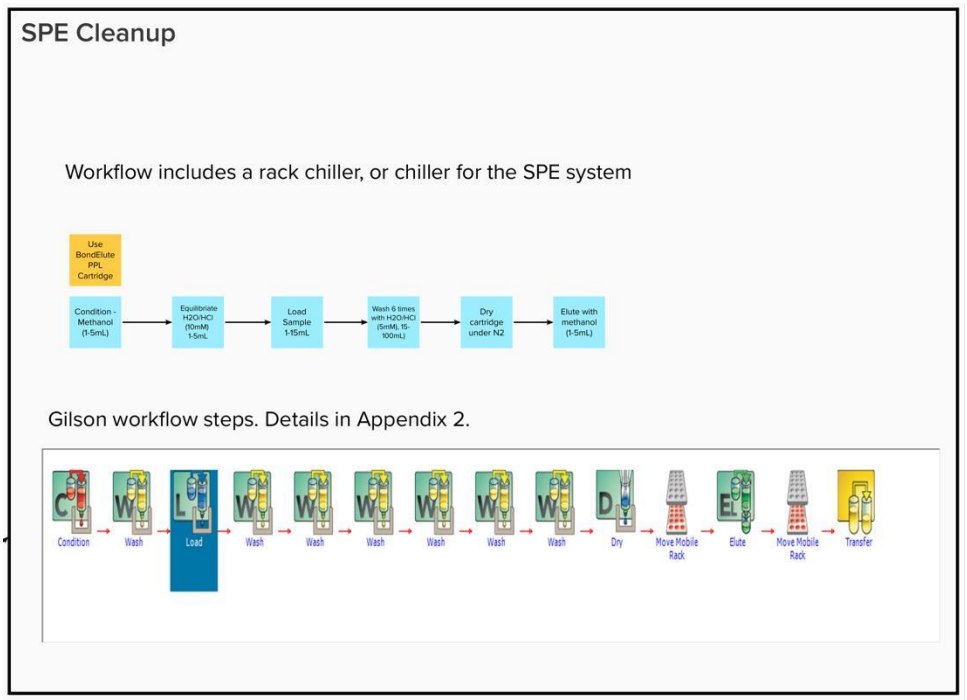

Figure 4: Process diagram of Component 4 of the automated soil analysis system showing a solid phase extraction system managed by one or more Gilson 274 devices. Appendix 3 contains the current workflow implemented in the Gilson device (the Powerpoint pdf) and the timing (the Excel document).

Facility Information

The facility infrastructure and space will have the following characteristics:

Possible Location One: See floor plan in Appendix 4

- General Information:
  - Square footage: 1130 SF
  - Ceiling heights: 12 ft
  - Floor to cable tray: ~8 ft
  - Floor to lowest ceiling extension: ~6' 4"
- Electrical:
  - Power
    - 3ea. 120/208V, 150A panel boards
    - 1ea. 120/208V, 225A panelboard
    - 120 and 208 distributed throughout lab
  - Data
    - PNNL Intranet, Cat 5e cabling or better
  - Other
    - 208V, 1 ph, 40A pin and sleave outlet in overhead service carrier
    - Laser Interlock system
    - 480V is available from continuous bus duct above adjacent utility corridor
- Mechanical:

    - Ventilation Rate (Air Exchanges) 23ACH
    - Cooling Capacity (Btuh) 60,000
    - Facility Chilled Water (flow rate, pressure, temp) – 37GPM, 45PSI, 47F (AT SERVICE PLATES)
    - Deionized water
    - 2ea, flexible exhaust snorkels
- Laboratory Gasses:
    - 2 ea. Flammable gas cabinets (3 bottles each) in adjacent corridor and piped into lab with ¼" stainless tubing
    - 4 nonflammable gas bottle anchor points in adjacent corridor and piped into lab with ¼" stainless tubing
    - Building Compressed air 110 psi
    - Building Nitrogen gas 100 psi
- Architectural Features:
    - Fume hoods: 1ea. 6' fume hood
    - Sinks: 1 ea, with glass drying rack
    - Casework: 16 lf. Base and upper laboratory casework
    - Special features
        - 1 ea. ¼ Ton overhead crane rail with trolly
        - 3 ea. 2" SS vacuum tubes from lab to vacuum pump rack in utility corridor
        - Overhead cable tray system with 4 service drop locations

Possible Location 2:

- General Information:
    - Square footage: 1130 SF
    - Ceiling heights: 12 FT
    - Floor to cable tray: ~8 ft
    - Floor to lowest ceiling extension: ~6' 4"
- Electrical:
    - Power
        - 2ea. 120/208V, 200A panelboards
        - 1ea. 120/208V, 300A panelboard
        - 120V and 208V outlets distributed throughout lab
    - Data
        - PNNL Intranet, Cat 5e cabling or better
    - Other
        - 208V, 1 ph, 40A pin and sleave outlet in overhead service carrier
        - Laser Interlock system
        - 480V is available from continuous bus duct above adjacent utility corridor
- Mechanical:
    - Ventilation Rate (Air Exchanges) 23ACH
    - Cooling Capacity (Btuh) 60,000BTUH
    - Facility Chilled Water (flow rate, pressure, temp) 37GPM, 45PSI, 47F
    - Deionized water
    - 3ea, flexible exhaust snorkels
- Laboratory Gasses:
    - 1 ea. Flammable gas cabinets (3 bottles each) in adjacent corridor and piped into lab with ¼" stainless tubing
    - 10 nonflammable gas bottle anchor points in adjacent corridor and piped into lab with ¼" stainless tubing
    - Building Compressed air 110 psi
    - Building Nitrogen gas 100 psi
- Architectural Features:
    - Fume hoods: 1ea. 4' fume hood
    - Sinks: 1 ea, with glass drying rack

- Casework: 14 lf. Base and 10 lf. upper laboratory casework
- Special features
  - 1 ea.¼ Ton overhead crane rail with trolly
  - 1 ea. ½ Ton overhead crane rail with trolly
  - Overhead cable tray system with 10 service drop locations

Integration Needs

Robotic access points to and between the components is expected. The design should allow for expansion to increase capacity. Vendor control software should support connection to EMSLs and PNNL secure networks and allow remote access to another Vendors scheduling software that will manage operation of the integrated system and instruments as necessary

Sample Platforms, Sample Handling and Labeling Needs

Samples for analysis in the automated organic matter pipeline arrive in EMSL in multiple formats and currently move through the analysis in multiple formats: multi-well plates, and tubes ranging from 1 ml to 50 ml. Components 1 and 2 should support use of 15 and 50 ml tubes. Component 1 inputs into the sieving system will have to accommodate up to 500 grams of soil.

Bar coding at the sample level required for tracking stored samples should be a part of the scheduling, tracking and data collection elements of the workflow. Labeling will be done outside of the automated system.

Anticipated Minimum Throughput Needs

Scalability to increase throughput is an essential characteristic of the automated soil organic matter workflow. In Phase I, Component 1 will be able to accept and process 16 soil samples per day, passing multiple aliquots in racks out of the system. Each of the 16 soil samples with produce 6 aliquots (One each for the 6extractions in Component 2, for a total of 96 samples) that pass to Components 2. All 96 samples then pass to Components 3 and 4. Phase 1 throughput is therefore 16 incoming soil samples producing 96 samples for analysis on the FTICR mass spectroscopy platform. Component 1 should be designed to process 80 soil samples per day (will produce 480 samples for FTICR analysis) though the initial throughput will be 16 samples per day in Phase I. Components 2, 3 and 4 should be designed for expansion to handle 480-samples per day (all samples derived from 80 soil samples).

Data and Metadata Capture Needs

EMSL operates a laboratory information management system (LIMS) that captures data from analytical instrumentation and provides secure access to the data by EMSL Staff and external collaborators. The automated system is expected to support or manage capture of instrument performance, sample ID, analytical results in real-time or near real-time, instrument calibration and quality control information, and associate these data with parallel data and metadata stored on EMSL's LIMS system (NEXUS). The vendor software is expected to provide APIs to support communication with NEXUS, and an SDK (Software Development Kit) to guide development of the needed communication layer between the vendor system and NEXUS.

Anticipated Minimum Performance Parameters for the Complete System. Some may not apply to Components 1 and 2.

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Key Performance Parameter	System Expectations
Modularity and Expandability	Key instruments and devices can be moved into and out of the workflow and additional instruments, liquid handling or other devices added to increase throughput.
Connectivity to Unique Instruments	Driver development and connectivity through APIs is straightforward through developer kits. No software or hardware specific barriers to integration with

	uncommon or newly engineered instruments exist. Connectivity too non-benchtop instruments possible.
Scheduling Software Connectivity and Security	Scheduling software can support user authentication, can connect with EMSLs NEXUS system and be operated securely, remotely.
Sample Labelling and Tracking	System registers samples at multiple steps in the workflow by reading bar-codes
Data and Meta Data Capture and Transfer	System collects instrument performance metrics, sample ID's, sample treatments and analytics results and transfers the information to EMSL's LIMS system.
Integration with ML Algorithms	Scheduling software can interface with analytic software to support real-time decisions regarding sample processing or analyses
Data and Systems Security	Application and network security includes role-based access control and/or integration with existing PNNL user management systems. API, RPC, and other network communications support encryption. Applications support logging for auditing purposes.
Sample Platforms	Supports multiple tube formats.

Appendix 1

Parallel Extraction Protocol.

Mineral dissolution extractions:

i) DI water
ii) 0.5 M HCl.
iii) 0.25 M hydroxylamine solution (0.25 M NH_2OH + 0.25 M HCl)
iv) HCl-dithionite (57.4 mM sodium dithionite shakes overnight, then rinses with the 0.05M HCl for 1 hour and centrifuged again. Supernatants from both extractions were combined)
v) 0.1 M sodium pyrophosphate

Protocol: Extractions will be conducted separately.

a) Briefly, 0.25 g air dry soil is weighed in 15ml centrifuge tubes and mixed with 10mL of each extractant, respectively.
b) Samples are shaken for 1 hours.
c) Samples are centrifuged at room temperature for 10 min at 4000 rpm.
d) The supernatant is filtered using a 0.22 μm syringe filter.
e) 4 ml of extract is stored under 4ºC for TC TOC TIC and for ICP-OES (Fe, Al, Si, Ca).
f) 6 ml of extract is stored under 4ºC for FTICR/MS.
g) air dried-solid is stored for TC TOC TIC, elemental composition, and archive.

Appendix 2

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Application News

No. TOC-005

Total Organic Carbon Analysis

TOC Analysis for Standard Methods 5310B: High-Temperature Combustion Method

■ Introduction

Total Organic Carbon (TOC) is a rapid method that analyzes for organic carbon and expresses the result as the amount of carbon found. It is a non-specific method unable to distinguish between various organic species and only indicates that organic carbon compounds are present. Organic carbon analyzers operate by the determination of the amount of total carbon present in a sample aliquot. Total carbon consists of inorganic and organic carbon. The inorganic carbon, present as carbonate or bicarbonate ions, must be removed or quantified prior to the analysis of organic carbon. Once the inorganic carbon is removed, subsequent analysis of the sample aliquot assumes that all carbon remaining is organic.

■ Discussion

Methodology used to remove inorganic carbon relies on acidification that converts all bicarbonate and carbonate ions to carbon dioxide that is then purged out of the sample using CO_2 free gas. If quantification of inorganic carbon is desired, it is purged into a detector, otherwise, it is vented to atmosphere. Once the inorganic carbon is removed, the remaining organic carbon is oxidized to carbon dioxide that is purged by CO_2 free gas into the detector. TOC analysis is applicable to both drinking water and wastewater.

■ TOC in Drinking Water

Control of disinfection byproduct formation (DBP) is accomplished by minimizing DBP precursors. Chlorine reacts with naturally occurring organic matter to form Trihalomethanes and Haloacetic acids. Organic matter in drinking water sources and finished drinking water is measured as Total Organic Carbon (TOC). Water systems that use traditional filtration methods for removal of organic matter are required to remove certain percentages of TOC depending upon the concentration of TOC and alkalinity in the source water.

This rule only applies to systems treating surface water. In essence, a system measures the source water and finished water once monthly for TOC and calculates the % TOC removal. Table 1 shows the percent TOC removal requirements as a function of TOC and alkalinity concentration.

■ TOC in wastewater

TOC can be correlated to the Biochemical Oxygen Demand (CBOD) and to the Chemical Oxygen Demand (COD). CFR 40 Part 133.104 (b) allows substitution of TOC for BOD or COD provided long-term (usually once/month) comparison data is collected. A factor generated from the data enables calculation of BOD from the TOC result. Using TOC instead of BOD is especially useful in tertiary effluents and process control.

TOC concentration for drinking water can range from less than 100 ppb to more than 25 ppm, and wastewater may contain much higher levels of organic compounds, *i.e.* >100 ppm C. Thus, it becomes important for a TOC analyzer to be capable of precise measurement for both the low-end (ppb) and high-end (ppm), to be capable of performing auto-dilutions for over-range samples, and to be capable of utilizing multiple calibration curves and selecting the correct curve to use for each sample.

Source Water TOC (mg/L)	Source Water Alkalinity (mg/L as $CaCO_3$)		
	0 – 60	60 – 120	Greater than 120
2.0 – 4.0	35.0 %	25.0 %	15.0 %
4.0 – 8.0	45.0 %	35.0 %	25.0 %
Greater than 8.0	50.0 %	40.0 %	30.0 %

Table 1: TOC removal requirements

■ Procedure

Standard Methods 5310B describes the procedure and requirements for analysis by high-temperature catalytic oxidation (HTCO) with non-dispersive infrared (NDIR) detection. The sample is homogenized and diluted as necessary, and a small amount is injected into a heated reaction chamber packed with a platinum catalyst. Water vaporizes, and organic carbon compounds are oxidized to CO_2 and H_2O. The CO_2 from the oxidation of organic and inorganic carbon are measured by the NDIR.

■ Experimental

A 1000 ppm C stock standard was prepped by dissolving 2.125 g of potassium hydrogen phthalate (KHP) in 1 L of DI H_2O. Two high standards were prepared by diluting this stock standard to concentrations of 1 ppm and 100 ppm, and two calibration curves were generated using auto-dilution of these standards. These curves are presented in Figures 1 and 2, with a summary provided in Tables 2 and 3. Instrument parameters are shown in Table 4.

Calibration Standard (ppm)	Std. Dev.	RSD
0	0.09218	7.82%
1	0.03950	1.17%
2.5	0.07136	1.00%
5	0.13870	1.01%
10	0.47930	1.79%

r	0.9998
Det. Limit	0.03 ppm

Table 2: Calibration curve summary for "Drinking Water" curve

Calibration Standard (ppm)	Std. Dev.	RSD
0	0.09164	10.94%
10	0.49500	0.19%
25	0.48790	0.74%
50	2.12100	1.61%
100	3.25300	1.21%

r	0.9999
Det. Limit	0.04 ppm

Table 3: Calibration curve summary for "Wastewater" curve

Parameter	Value
Analysis Method	NPOC (non-purgeable organic carbon)
Furnace Temperature	680 °C
Injection Volume	50 µL
Acid Addition	2%
No. of Injections	3/5

Table 4: Instrument parameters

Additionally, a 5 ppm check standard (for drinking water) and 50 ppm check standard (for wastewater) were both analyzed after calibration. The results are summarized in Table 5:

Check Standard	Result	Recovery	Std. Dev.	RSD
5 ppm	4.960	100.8%	0.11780	0.854%
50 ppm	49.39	101.2%	1.98700	1.505%

Table 5: Analysis of check standards

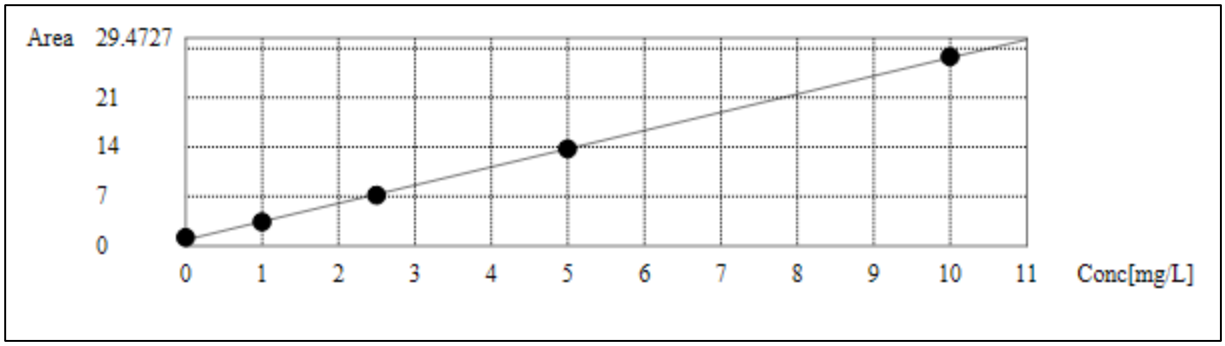

Figure 1: "Drinking Water" Calibration Curve

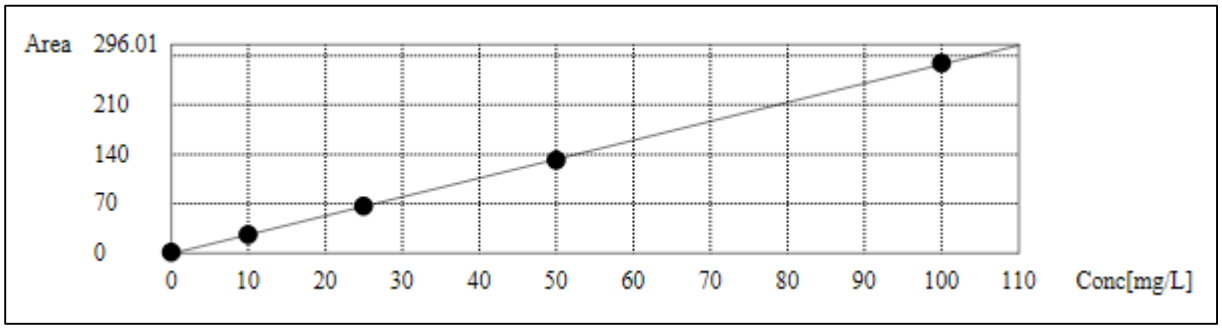

Figure 2: "Wastewater" Calibration Curve

■ Conclusion

The Shimadzu TOC-L total organic carbon analyzer is ideal for TOC measurement by Standard Methods 5310B. The TOC-L, with a 680 °C furnace and platinum catalyst, utilizes high-temperature catalytic oxidation to completely oxidize organic carbon.

Moreover, the ability to auto-generate a calibration curve from a single standard, coupled with the capability to auto-dilute and re-inject over-range samples, ensures that one method can analyze all samples.

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Appendix 3

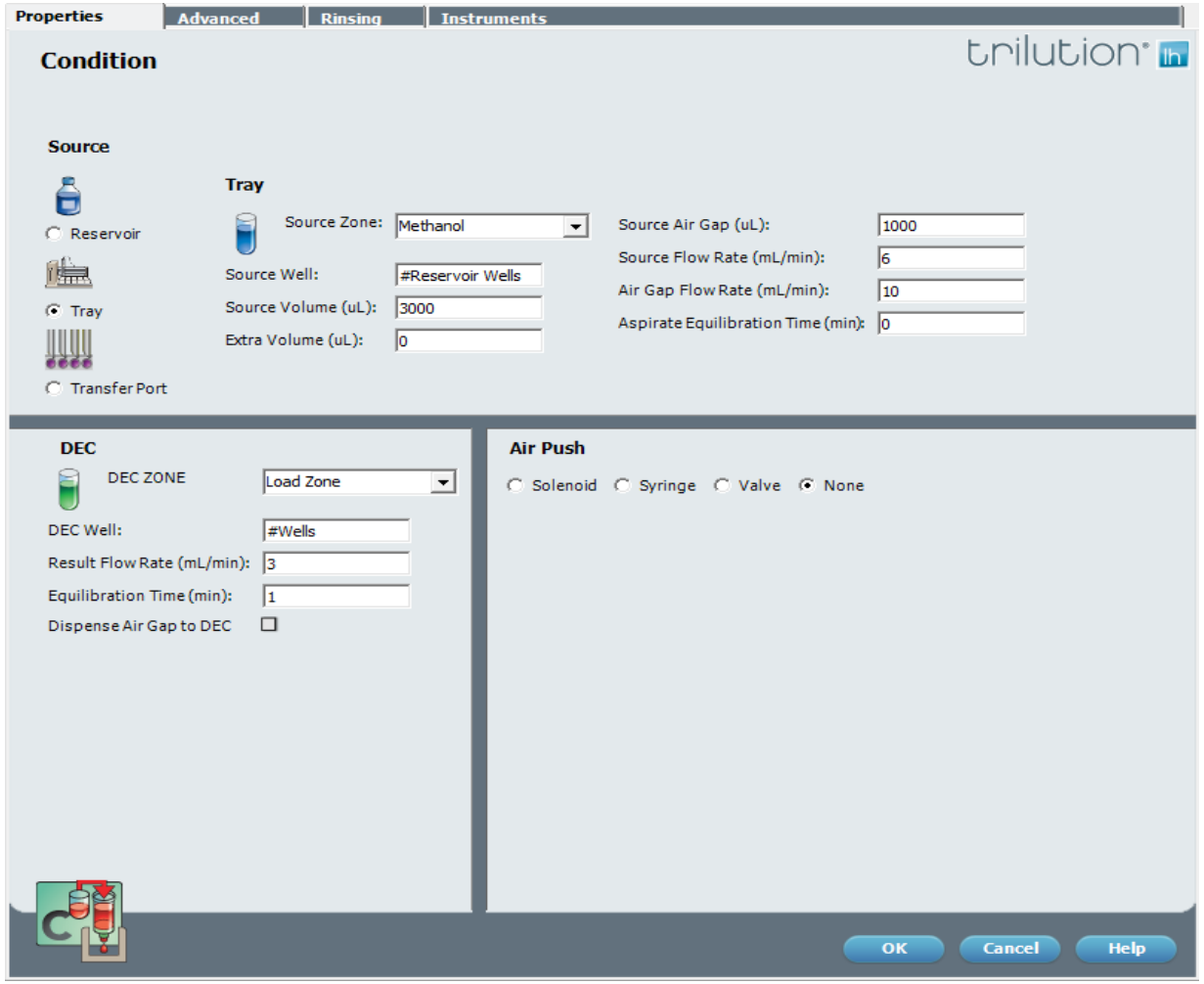
Gilson Liquid Handler SPE Method

Jason Toyoda

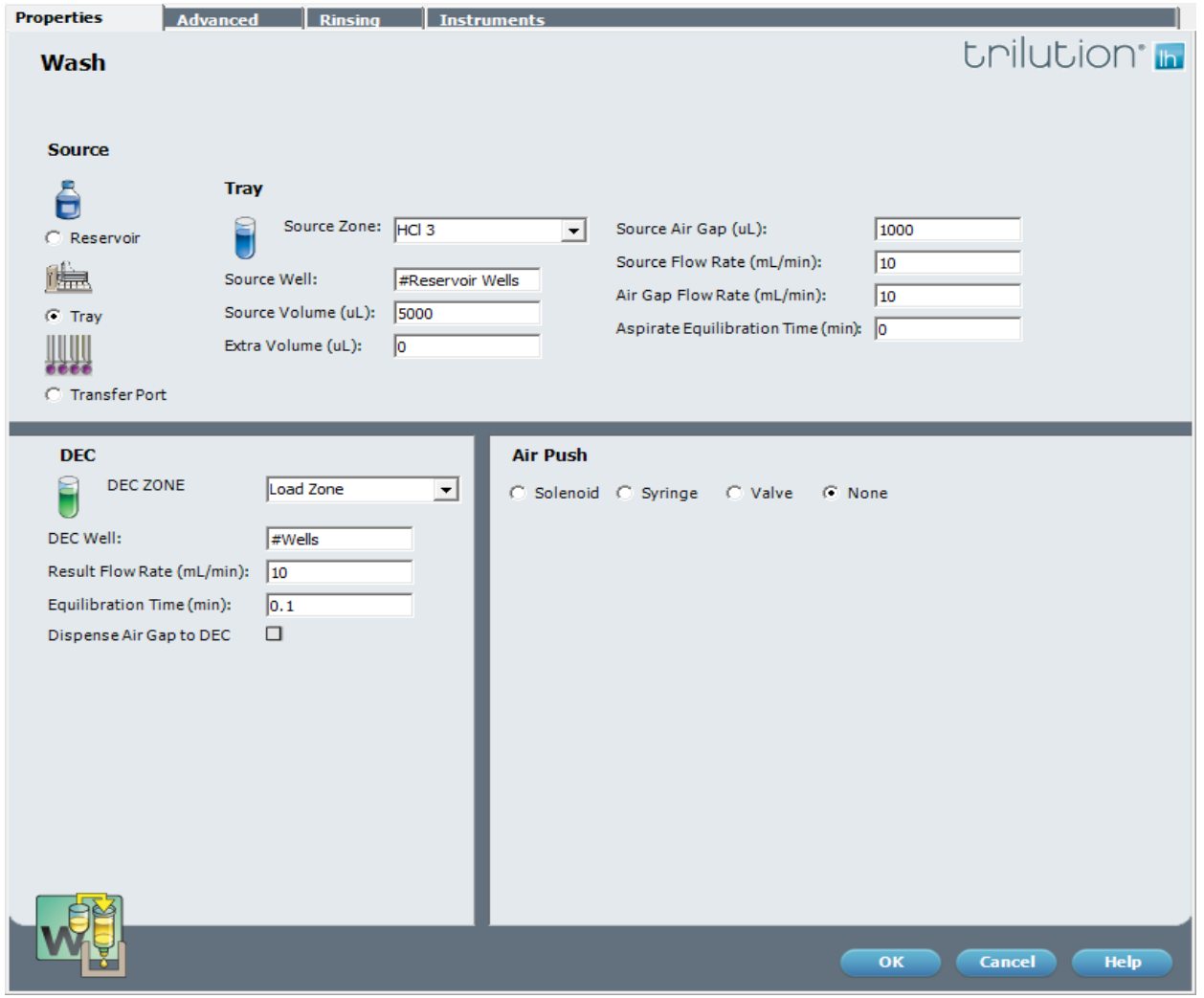
Entire Method for 5mL Sample Volume and 50mL Rinse

*These methods can easily be changed for different sample volumes and different wash volumes

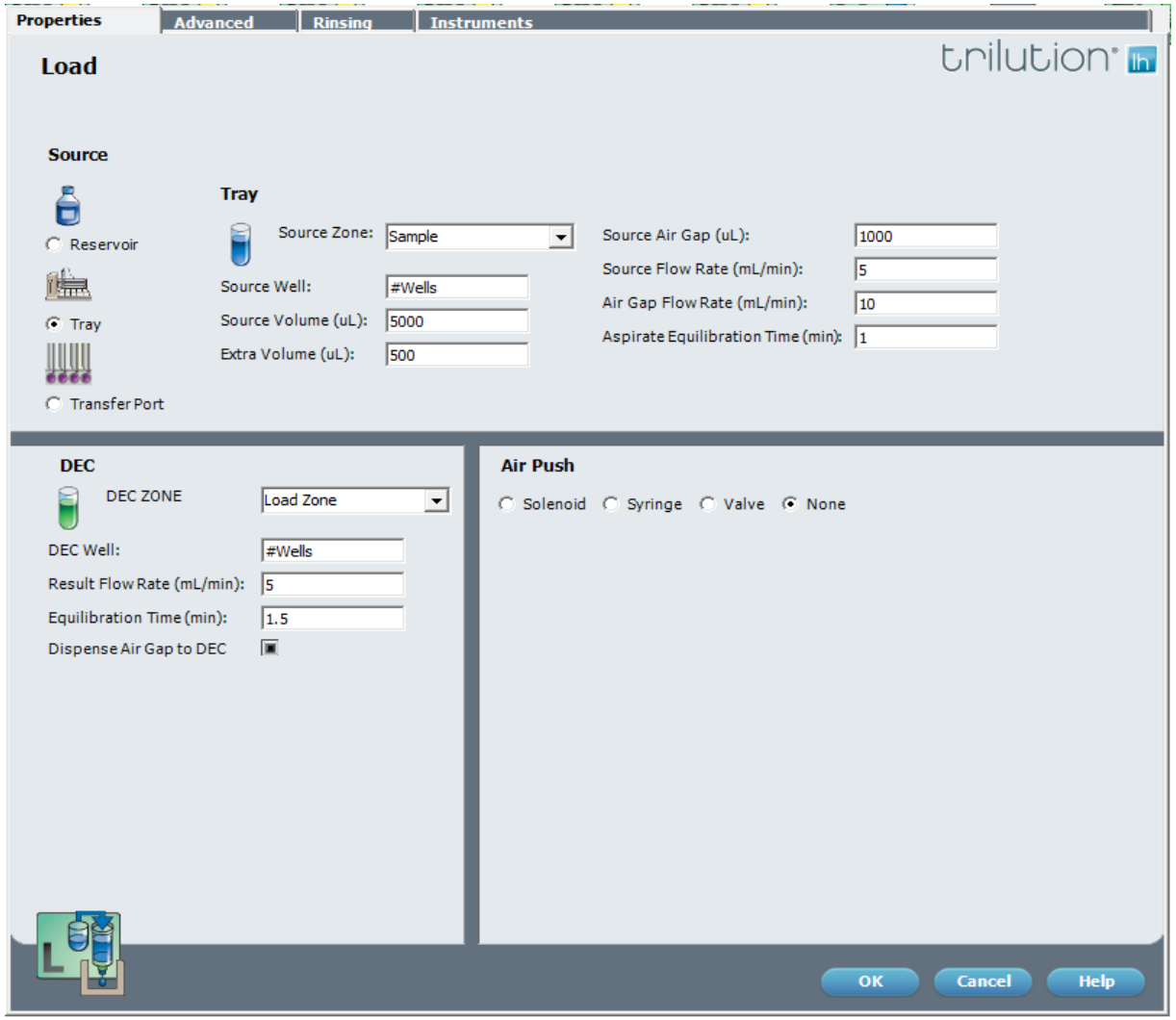
Cartridge Conditioning



Cartridge Pre-Wash

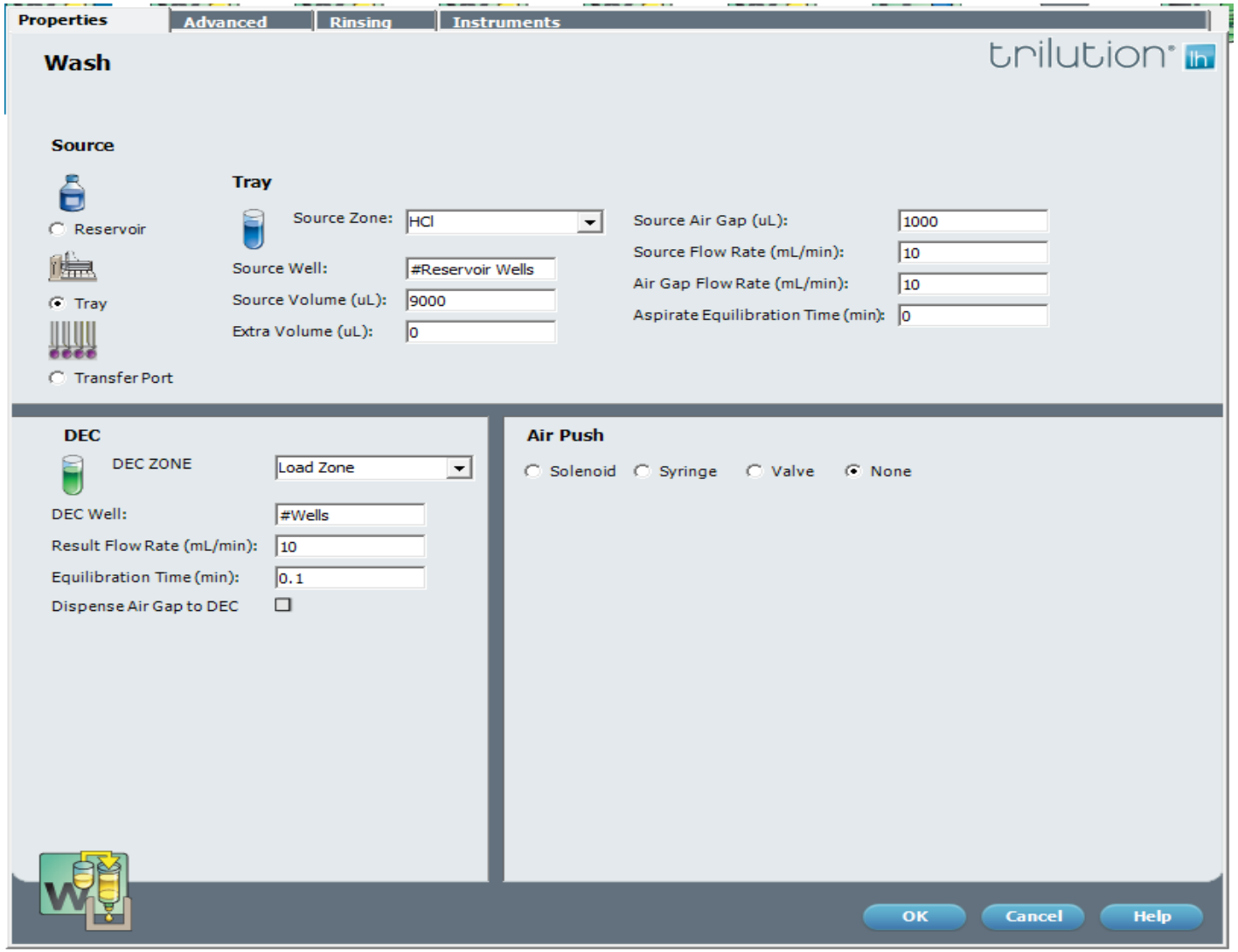


Sample Loading

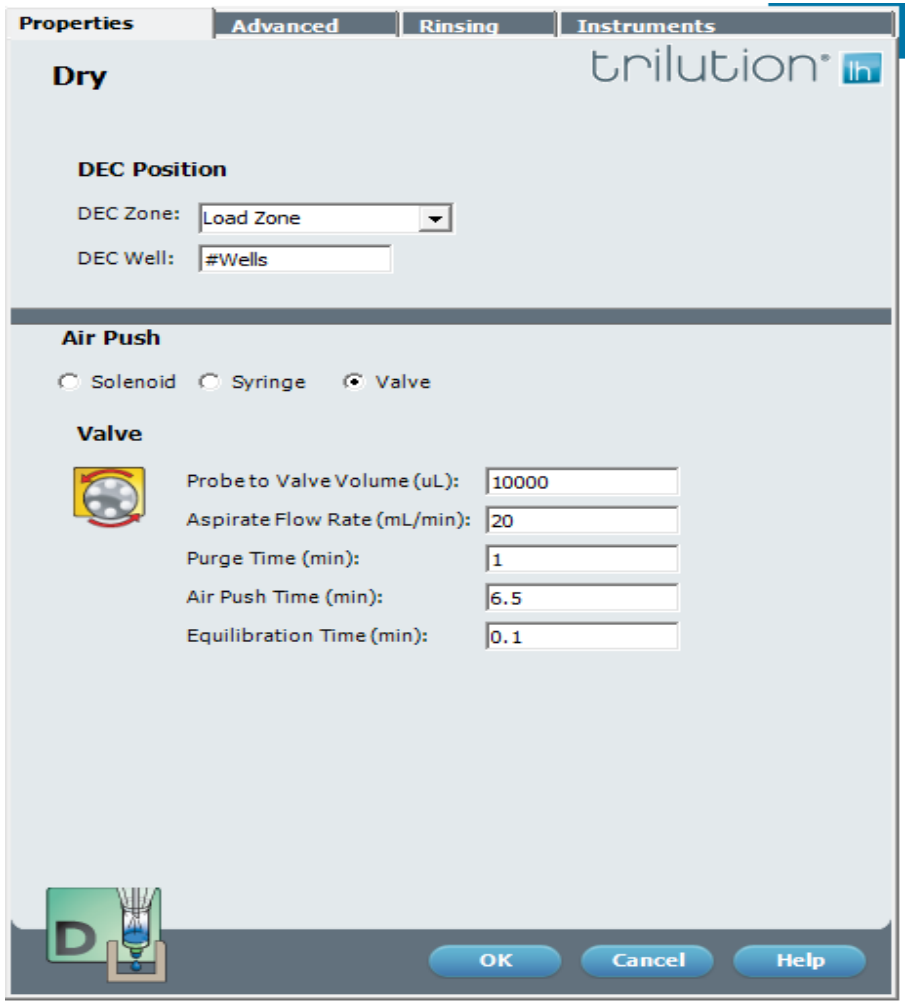


Wash Steps

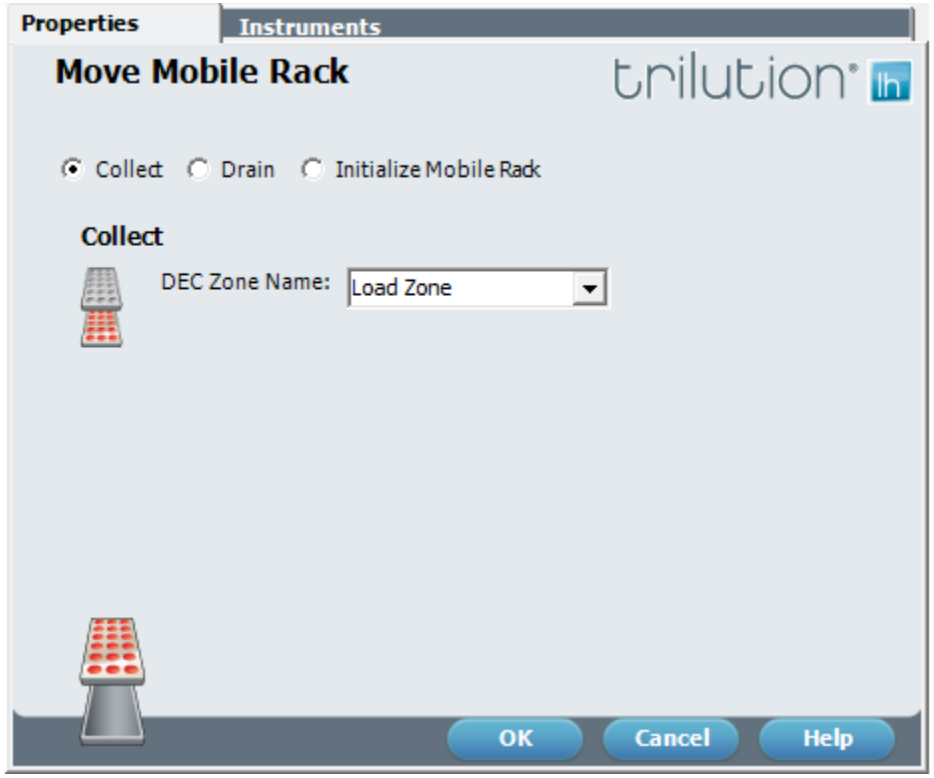
- Currently five 9mL rinses plus an extra 5mL rinse



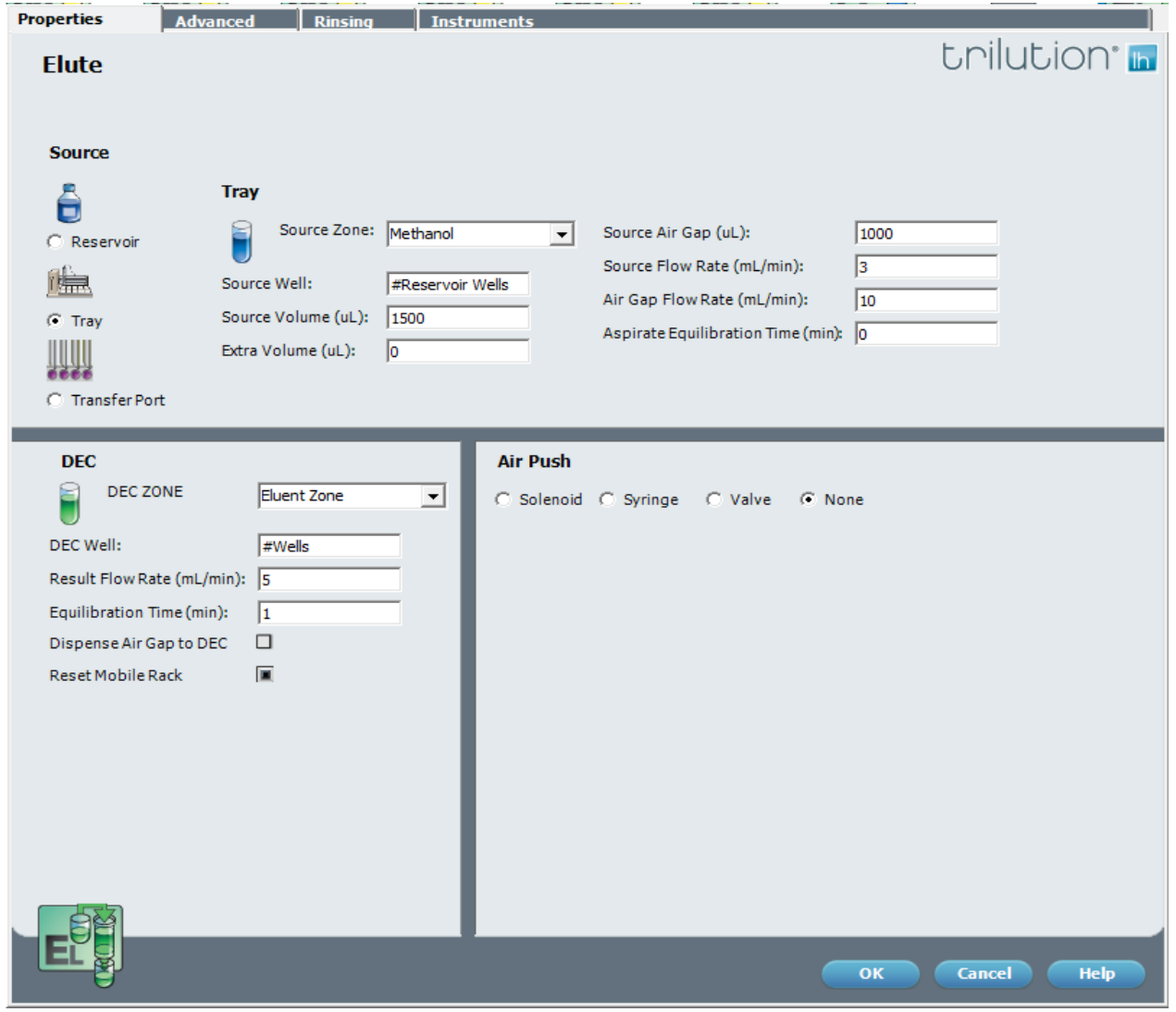
Drying Step (using house N2(g) line)



Mobile Rack Move to Collection Zone

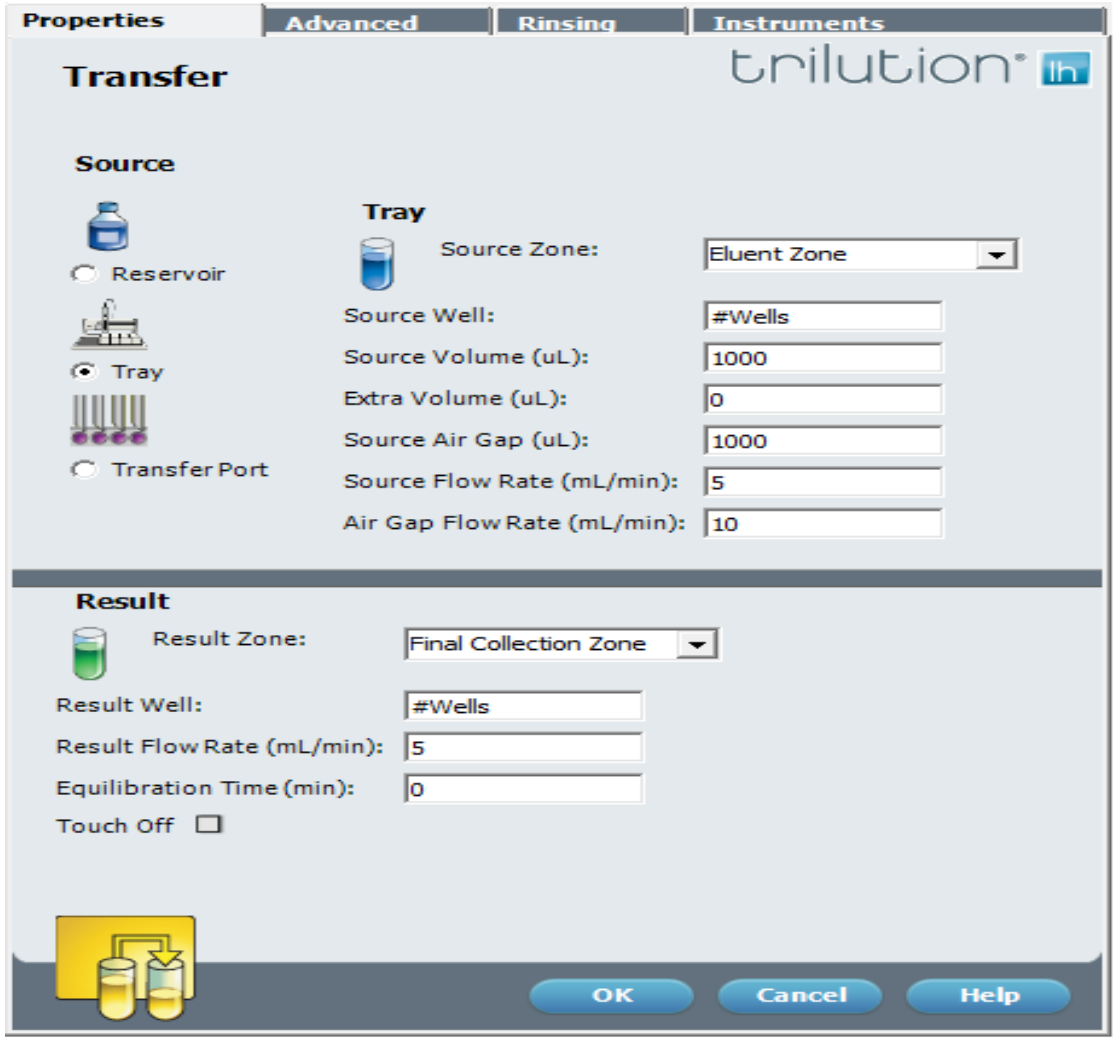


Elution Step



Mobile Rack Move Back to Drain/Waste

Sample Transfer to Microsolv Vials



Step

1: Conditioning Step

		Aspirate	Air Gap	Decant	Equilibration	Time (per 4 sample set)	Total Time with Probe Rinsing Steps (4 Sample Set)
Methanol	**Volume**	3mL	1 mL	3mL			
	Rate	6mL/min	10ml/Min	3mL/min			
	Time (min)	0.5	0.1	1	1	2.6	4 min

2: Load

		Aspirate	Air Gap	Decant	Equilibration	Time (per 4 sample set)	
Sample	Volume	5.5mL	1 mL	5mL			
	Rate	5ml/min	10ml/Min	5mL/min			
	Time (min)	1.1	0.1	1	2.5	4.7	6 min

3: Wash (5X 9mL+ one 5mL Rinse)

		Aspirate	Air Gap	Decant	Equilibration	Time (per 4 sample set)	
HCL and Water	Volume	9mL	1 mL	9mL			
	Rate	10ml/min	10ml/Min	10ml/min			
						6	7min (X5 +5min =40 minutes)
	Time (min)	0.9	0.1	0.9	0.1		

4: Dry

		Aspirate	Air Gap	Decant	Equilibration	Time (per 4 sample set)	
N2 gas	Volume	10mL					
	Rate	20mL/min					
	Time (min)	0.85	1	6.5	0.1	8.45	9.5 min

4: Elute

		Aspirate	Air Gap	Decant	Equilibration	Time (per 4 sample set)	
Methanol	Volume	1.5mL	1mL	1.5mL			
	Rate	3mL/min	10mL/min	5mL/min			
	Time (min)	0.5	0.1	0.3	1	1.9	3 minutes

5: Transfer to microsolve tube

		Aspirate	Air Gap	Decant	Equilibration	Time (per 4 sample set)	
	Volume	1mL	1mL	1mL			
	Rate	5mL/min	10mL/min	5mL/min			
	Time (min)	0.2	0.1	0.2	0	0.5	1.5 minutes

Total Time (min)	52.15
Total Time Adjusted for Cleaning Steps (min)	64*

*This time does not scale 1:1 with increase in number of samples. A set of 20 samples following this method is about 3 hours and 30 minutes

Appendix 4