

iCAP 7400 ICP-OES operation sequence

1. Turn on the instrument (analyzer, water chiller, autosampler) and gases (Ar; N₂ should be kept on).
2. Tighten the pumps and tubing for rinse, sample introduction and drain. Make sure the sample and drain tubes are in the correct configuration.
3. Log into the computer and start Qtegra. Check status for all the interlocks through Dashboard. Optics temperature and Detector temperature are usually red when the instrument was just turned on. Everything else should be green.
4. (The next two steps are to check the pump and tube system.) Make sure your rinse and waste bottles are in position. Move the sipper to the Rinse position through the autosampler interface.
5. Set the pump speed to 50 rpm through the analyzer interface. And then watch for around three minutes to see if the sample introduction and drain flow are functioning. Bubbles should be consistently generated in the drain flow. Adjust the pump pressure or change the tubes when necessary. Stop then pump and rinse when the adjustment is completed.
6. While the Optics are being cooled down (usually takes 30 to 40 mins), fill each of your samples into the test tubes and place them in one of the rack in the autosampler. Rinse the test tubes well. Create and fill out your labbook. (The next four steps. Refer to Appendix for graphical illustrations.)
7. Select the elements of interest and appropriate wavelengths for each of them through Analytes.
8. Fill out your standards information.
9. Select the rack (and correct type) where your samples are placed through Cetac ASX-520.
10. Fill out the sample list. Start with Blank, low to high standards and then unknown samples. The position of each sample is determined by the Rack No. and the Vial No. Refer to Cetac ASX-520 to confirm the positions.
11. After all the interlocks on the Dashboard turned green, click the Blue "Get Ready" button to start warming up the instrument. DI water works fine for previous steps. But if your solvent is not DI water, you should also prepare around 2 L of essentially your blank to serve as Rinse. And switch to your actual rinse at this step.
12. Once the warm-up is complete, click the green button on your labbook to schedule it and click it on the schedule to start measurement.

13. After measurement is completed, shut down the instrument by clicking the blue “get ready” button on the Dashboard again. Turn off the instrument and water chiller, autosampler and Ar gas. Loosen all the pumps and tubes.

14. Analyze data and generate reports. Make sure your labbook and reports are saved into your own folder.

15. Log out your session. Fill out the Logbook and report any issues.

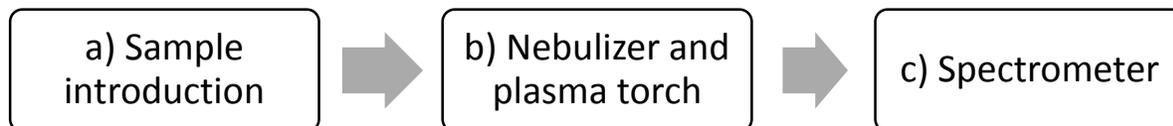
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More annoying notes:

1. With the plasma aspirated a blank sample for 15 minutes to allow the instrument to fully stabilize before analysis.
2. After an analysis is completed. A blank sample should be aspirated for five minutes to rinse. Deionized water should be aspirated for another minute after blank.
3. When organic solvent based samples are being analyzed, the final rinse should be the pure solvent. Air should be aspirated for two minutes to remove organic vapours.
4. After plasma has been turned off for at least 30 s to turn off the chiller.
5. Allow 5 minutes after switching off the plasma before disconnecting the electrical power.

ICP-OES is a widely used technique for the detection of trace metals. It atomizes the sample with inductively coupled plasma and then measures the emission intensity at wavelengths characteristic of particular elements. A flow chart illustrating the measurement process in an ICP-OES instrument is given below.



a) The sample solution is transported from external containers into the nebulizer by a peristaltic pump.

b) The sample is converted to aerosol by the nebulizer and the aerosol is atomized by the plasma torch.

c) The spectrometer quantifies the emission intensities at specified wavelengths.

Refer to literature and web resources for more background and application information on ICP-OES instrumentation. (Youtube and Wikipedia are not bad choices here.)

The instrument that we will be using is an iCAP™ 7400 radial model from Thermo Scientific equipped with a CETAC ASX520 auto sampler.

Appendix

Setting up the program-Qtegra

Data collection and analysis are conducted through the Qtegra program coupled with the instrument. The Dashboard panel of the program is shown below.

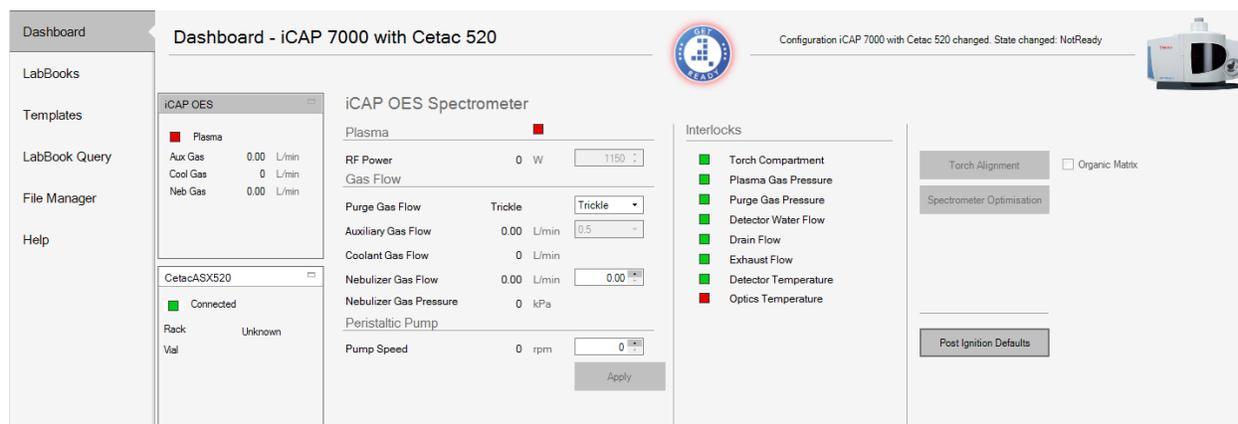


Figure 1: Qtegra-Dashboard

The Dashboard provides an overview of the status of the instrument. When the instrument is operational, all the interlocks should be green.

Each experiment on the instrument is carried out through a LabBook, which can be created from either a template or an existing LabBook.

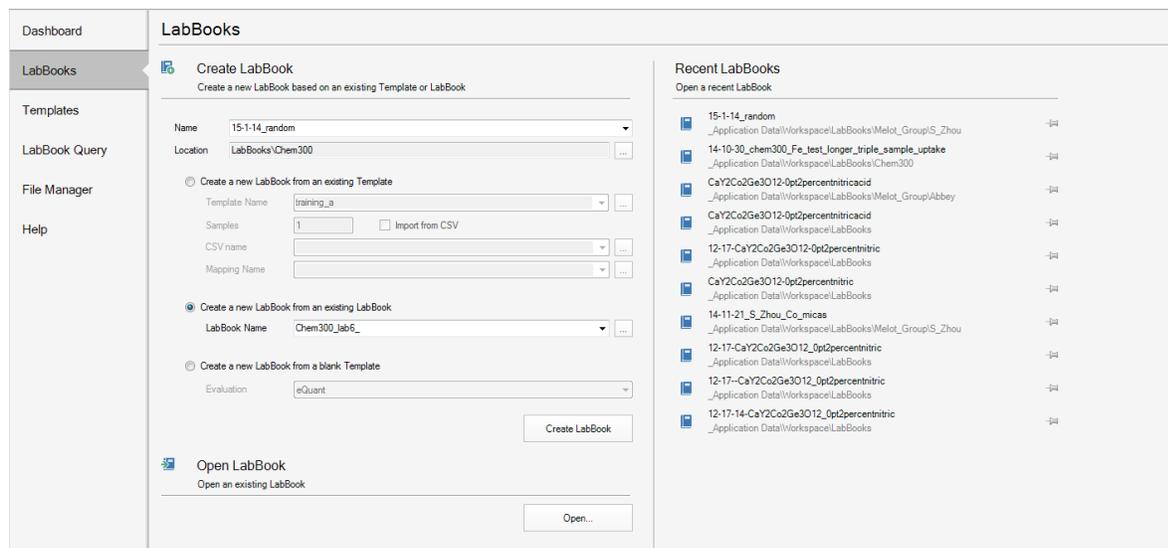


Figure 2: Qtegra-Creat a LabBook

The interface of a LabBook is shown below.

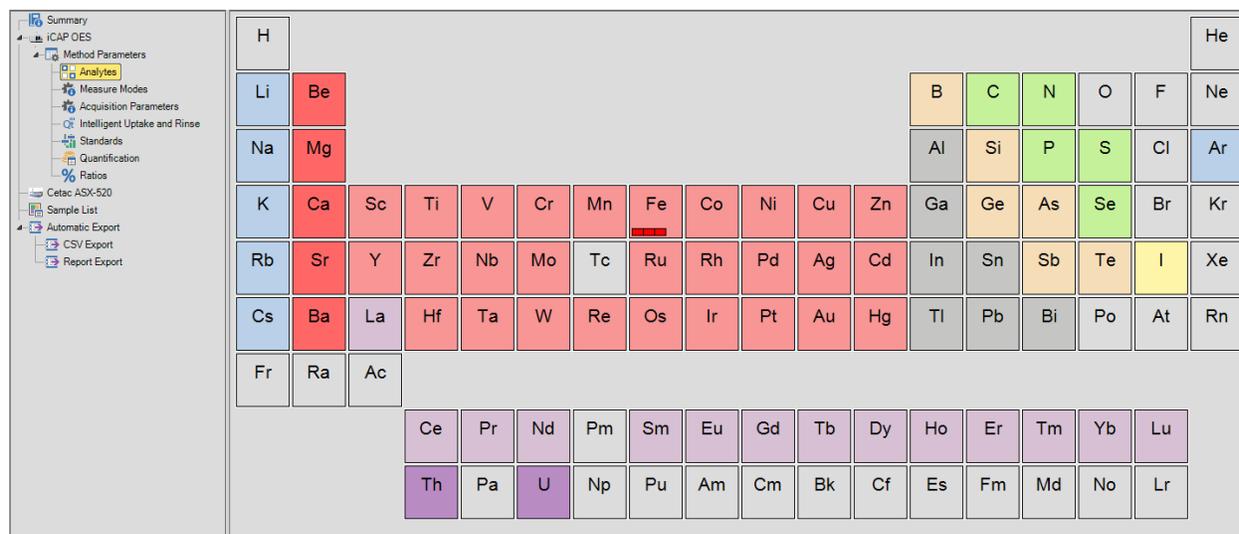


Figure 3: Qtegra-an unstarted LabBook

By clicking different items in the left panel, one can access or modify all the experiment parameters and the output (results).

For example, one can choose which element is to be analyzed in the experiment by clicking Analytes, after which a periodic table will show up. (Figure 3) Since each element has multiple

emission wavelengths, you can choose one or more wavelengths to analyze simultaneously. By right clicking an element, you can see the emission spectrum and relative intensity of that element at each wavelength that you can choose from. (Figure 4)

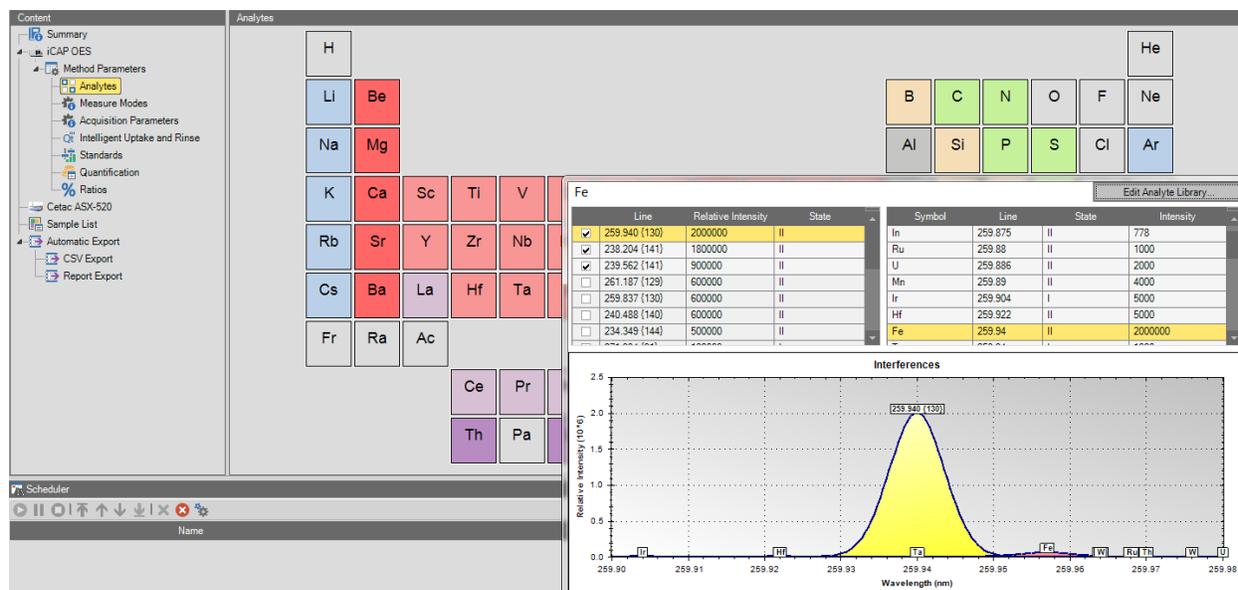


Figure 4: Qtegra-Emission spectra for Fe

You probably want to choose the peaks that are most intense and don't overlap with elements that your samples probably contain, Na for example.

Analysis of ICP-OES data

Once the experiment is completed, an "Evaluation Results" section will show up on the left panel of your LabBook. The results include the measured intensity of your sample at each of the wavelengths that you have selected.

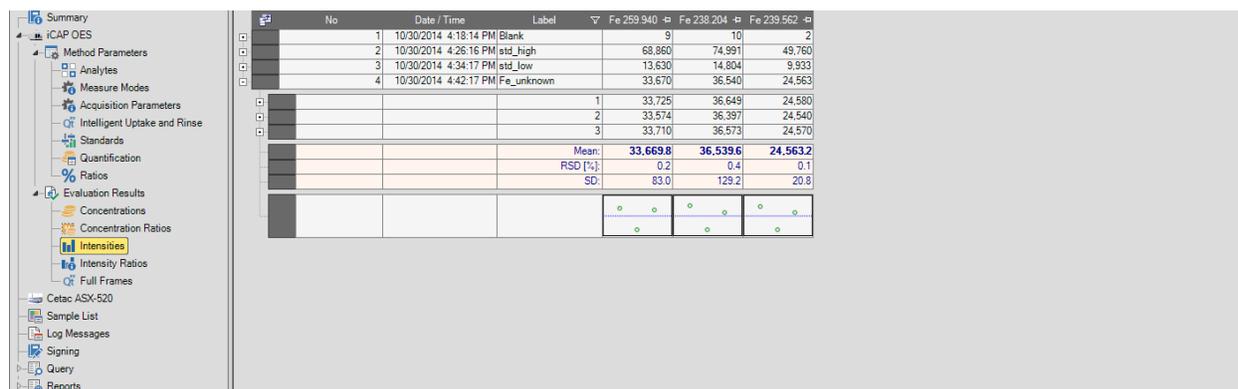


Figure 5: Qtegra-Evaluation Results-Intensities

You can, of course, calculate the concentration of your unknown with any set of intensity data and the concentrations of your standards. However, the program has done this automatically and the results are shown in the Concentrations section.

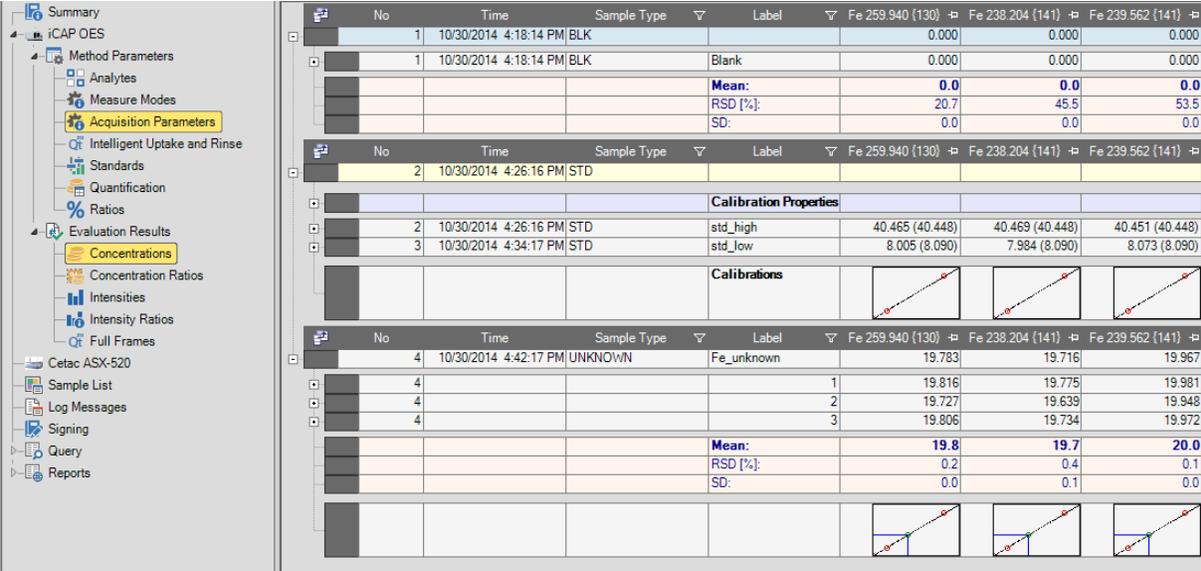


Figure 6: Qtegra-Evaluation Results-Concentrations

Each column shows the results for one of the wavelengths you have chosen. Each main row shows the results for one sample (a blank, standard, or unknown). By expanding the rows, one can also see the statistics of each result. This includes the RSD and SD for each measurement, the calibration curve of the standards.