
Shipboard Analytical Balance: User Guide

Manual Information

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Introduction

This user guide is designed to help onboard laboratory technicians and visiting scientists use the balance program for weighing samples on the ship's Mettler-Toledo (MT) XS204 and Cahn electrobalances. Detailed step-by-step procedures for using the balances with specific types of analyses and workflows are provided in instrument-specific user guides.

The shipboard laboratories have two different motion-compensated balance systems:

- Mettler-Toledo Excellence XS204 dual-analytical balance systems: range = 1~150 g
- Cahn Automatic Electrobalances: range = <1 g

Mettler-Toledo Excellence XS204 Analytical Balance

Onboard, three dual-balance sets of MT XS204 balances are installed, one each in the Core, Chemistry, and X-ray Laboratories. Software developed by IODP uses the two side-by-side balances (one for the unknown and one for a reference mass; *Figure 1*) to determine mass on the moving vessel. These balances can be used for masses ranging from ~1 g (possibly as low as 100 mg) to ~150 g. Although the XS-204 balances have a capacity of 200 g, ship heave will cause any sample or container weight to vary by a significant degree, so the user should keep samples well below that limit to avoid overloading the balances.



Figure 1. Mettler-Toledo XS204 Dual Analytical Balance System.

Cahn 29 and Cahn C31 Automatic Electrobalances

The Cahn 29 and C31 Automatic Electrobalances enable weighing of small masses (<1 g); take care not to overload them (*Figure 2*). The in-house motion compensated software enables users to accurately measure a small mass on the moving ship.

The Cahn electrobalances are located in the Chemistry Laboratory and the Microbiology Radiation Van on the ship.



Figure 2. Cahn C31 Electrobalance.

Instrument Calibration/Verification

To attain accurate results for subsequent measurements, the balance systems must be calibrated and that calibration verified. Calibration verification is run on a routine basis to ensure that the original instrument calibration is still valid. If calibration verification shows a problem in the system, any samples measured on that balance system since the last acceptable calibration verification must be reanalyzed.

Calibration

The balances are calibrated by IODP staff members using the balances' internal calibration routines. This calibration occurs in port when ship motion is minimal, or even on shore if possible.

The Mettler-Toledo (MT) balance software supports a secondary calibration based on the measurement of multiple mass standards, linear regression, and application of the coefficients to subsequent measurements. This method is based on older balance calibration methods where voltages were converted to masses in a customized system. We currently have no rationale to apply this secondary calibration as continued use has shown that the balance-provided calibration is accurate. Instead, we collect data from standards on a daily basis from the calibration verification process to build a data set over time and through variable sea states to evaluate the performance of MT dual balance system.

Calibration Verification

The user can verify calibration at any time by weighing a mass standard. A protocol of daily calibration verification, carried out by the responsible support staff, will provide the data necessary to make statements about the overall system performance.

Balance Start-Up

Turning the Balances On/Off

Before turning on the balance:

- Make sure serial cables are connected to both balances and PC.
- Make sure both balance trays are clear and the doors are closed.

Mettler-Toledo XS204

- Press and hold the On/Off button on the terminal until the balances start up or power down.

- To power down the MT balances, the software control program must be turned off.

Cahn Electrobalance

- To power up the Cahn electrobalance, press the power switch on the rear panel.
- Press the power switch on the rear panel to turn the Cahn electrobalance off.

Balance Software

The balances aboard the ship are used in a variety of ways, giving rise to separate software for each application.

- The moisture and density (MAD) software controls the MT XS204 balances in the core lab (see the MAD user guide for instructions on this software).
- The MT XS204 balances in the Chemistry lab are usually used to weigh reagents and use the .NET Balance Master software to make the mass determination.
- The MT XS204 balances in the X-ray lab are usually used to weigh loss-on-ignition (LOI) samples and use the Balance Master software to make the mass determinations; these values are uploaded to the LIMS using the Spreadsheet Uploader; the Balance Master software does not do so.
- The Cahn/29 and Cahn/31 electrobalances in the Chemistry lab are used to weigh samples for CHNS, Source Rock, coulometer analyses, and ICP hard rock/sediment preparation. The Cahn uses the Java Balance software to make the mass determination and record their values in the LIMS.
- The Cahn electrobalance in the Microbiology Radiation Van (Rad Van) is used to weigh small masses of isotopes and samples; operation of that balance is not covered in this guide.

Balance Master

The Balance Master program was created for the convenience of the chemistry technicians, who needed to be able to weigh out reagents without needing to upload those weights into the database, and was later adopted for the X-ray technician for LOI measurements as well. Balance Master is more streamlined and somewhat more user-friendly than the other balance programs.

Upon opening Balance Master, the initial screen (Figure 3) will be seen:

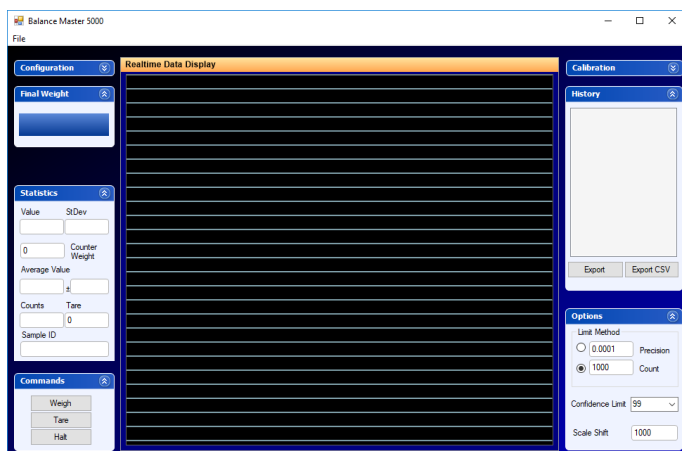


Figure 3. Balance Master initial screen.

Balance Master Configuration

The technician should ensure that Balance Master is configured properly. Click on the “Configuration” window (upper left in Fig. 3, above) to open the configuration screen as shown in Figure 4.

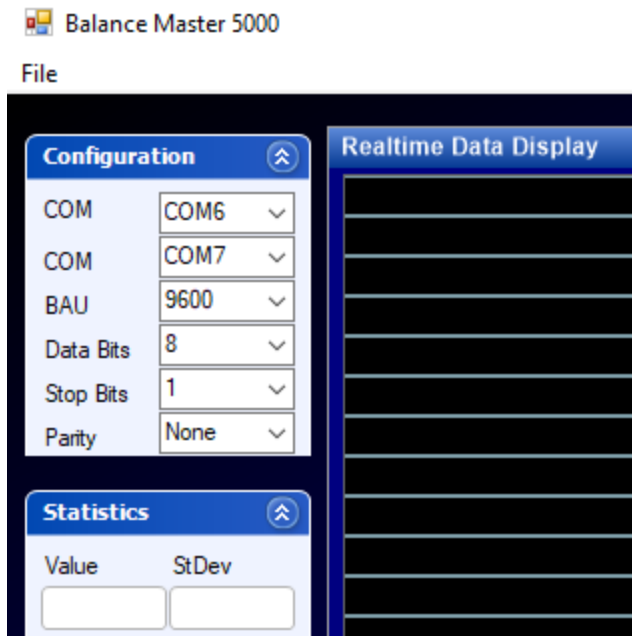


Figure 4. Balance Master configuration screen.

The two COM fields should be set to the appropriate COM ports of the two Metter-Toledo balances, taking care to ensure that the reference and unknown balances are selected appropriately. The communication settings for the XS-204 balances are: 9600 baud, 8 bits, 1 stop, no parity, as shown in Fig. 4.

Measurement Options

The user has the option of selecting either a set number of counts or a limit of precision for the balance measurement as shown in Figure 5, below. It is recommended that the user use the counting method, with a recommended minimum of 1000 (roughly 3 minutes) to capture data across a number of ship’s motion cycles.

Precision mode is not recommended because it is possible to reach a low precision value without sufficient measurement time; this problem can be seen in Figure 6, where the measurement stopped after only 31 data points.

If the user decides to use precision mode, they must also select a confidence limit (95, 99, or 99.5), which controls the multiplier for standard deviation used for the precision measurement.

The Scale Shift value should remain 1000 to keep the scale appropriate to the usual mass range being measured.

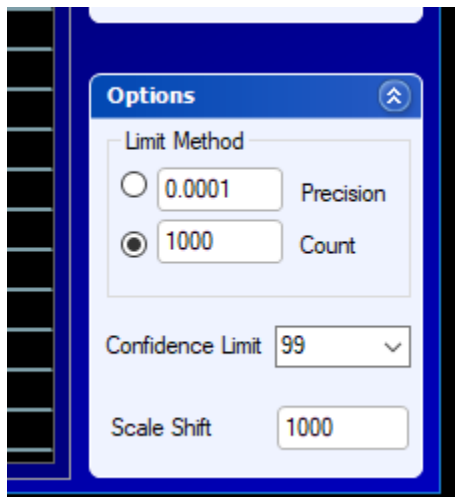


Figure 5. Measurement option window

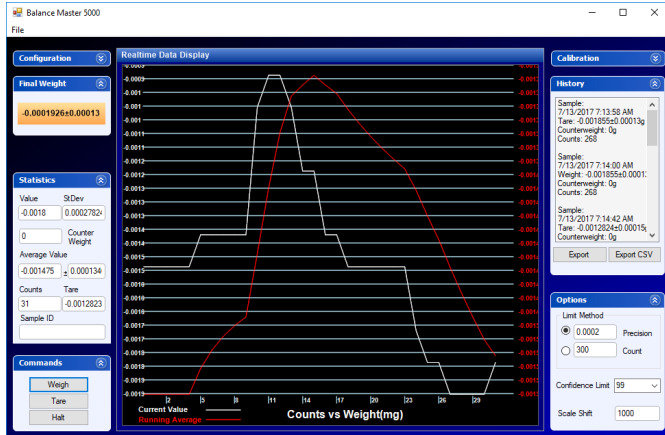


Figure 6. Precision-based problem. Note that although only 31 counts were captured, the weight uncertainty reached the defined 0.0002 grams simply by random happenstance. This is the reason the precision method is not recommended.

Taring the Balance

All of the paired balance systems on the ship must be tared via their control software, **not** by the tare buttons on the balances themselves. The software tare measures the difference between the averaged tare value of the reference and unknown balances. As seen in Figure 7, the tare value is -0.0012823 grams; this is the difference in the average tare value of the reference balance and the average tare value of the unknown balance.

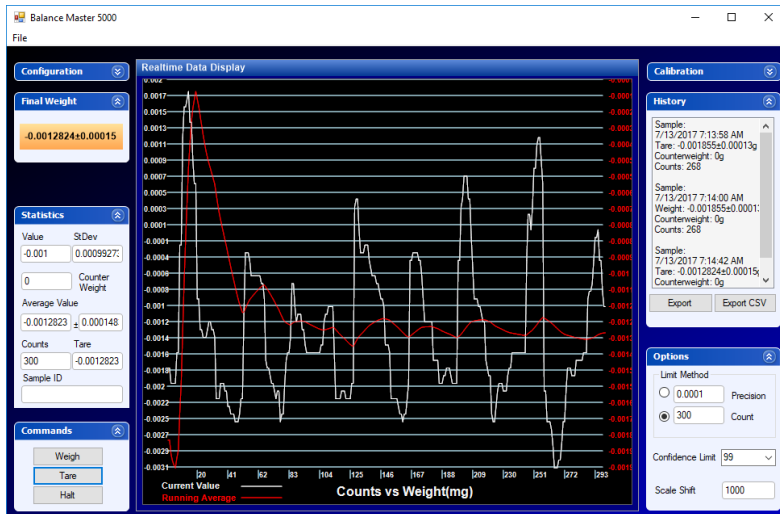


Figure 7. Completed tare step.

Making a Measurement

Once the tare is completed, the user can place the material to be weighed on the unknown (right-hand) balance, and a mass of standard reference masses approximately equal to the unknown to be weighed (refer to Precision Weighing at Sea for an explanation of this requirement). Enter the mass of the reference masses in the “Counter Weight” field, circled in red in Figure 8, below.

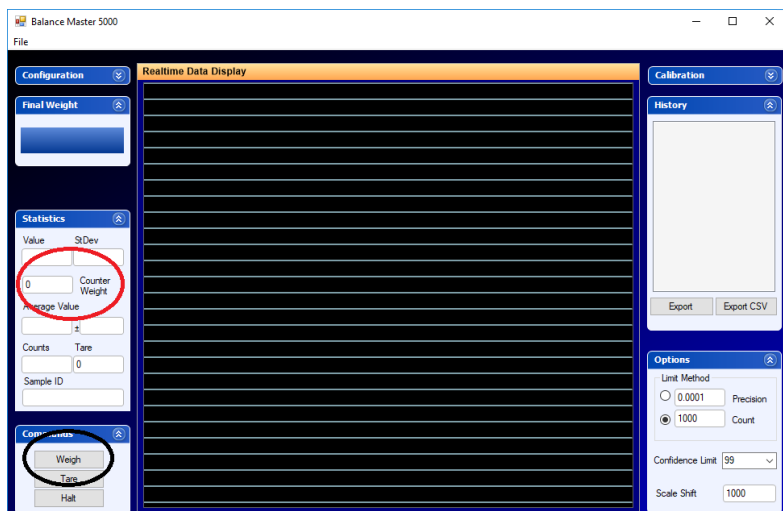


Figure 8. Counter Weight field (red circle) and Weigh command button (black circle).

Once the balances are loaded and the Counter Weight value is entered, press the “Weigh” button (circled in black in Figure 6, above) and the measurement will begin. It will stop automatically upon the completion of the measurement (after a set number of counts or a set precision value, depending on the options selected by the user).

Pro Tip

It is not necessary to wait for the measurement to complete if it is obvious that the reagent being weighed out is too much or too little. Press the “Halt” button, just below the “Weigh” and “Tare” buttons, add or remove reagent, and press “Weigh” again, repeating as necessary to reach the desired mass.

Final Weight

The final weight is expressed in grams \pm grams in the box on the upper left corner of the screen.

History and Export Features

The History window will show the last few measurements made by the balance, and the user can scroll through all of the masses measured since the software was started. It will clear if the software is closed.

The user can also use the “Export” button to export the data to a CSV file. (Do not use the “Export CSV” button as it does not work.) The CSV file contains a sample identifier (if used), the date and time of the measurement, the tare value (if taring) or the weight (if measuring), the value of counterweight used in the measurement, and the total counts of the measurement.

Calibration Feature

When the balance project began, the JRSO did not know if it would be necessary to calibrate the Mettler-Toledo XS-204 balances on top of the factory calibration, as they were being used in a manner different from the usual laboratory scenario. It was experimentally determined that no additional calibration is required, and this feature was never implemented. The user should ignore this window.

Java Balance Start-Up/Login

Use of the Java Balance software requires authentication to the LIMS, so once the software is started, a login screen will appear as shown in Figure 9. The link to this manual functions, but the instructions to email the shore-based developer account is out of date (any help requests should be routed to jr_developer@ship.iodp.tamu.edu instead). Although the login page states that it is for the dual Mettler-Toledo balances, this software is intended for use only on the Cahn electrobalances.

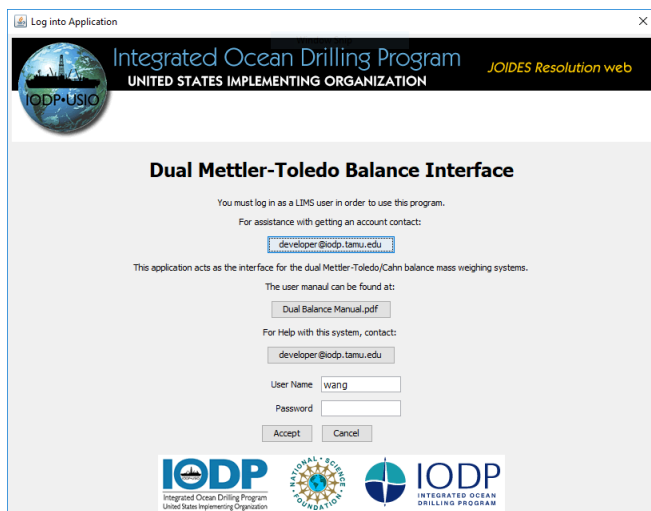


Figure 9. Java Balance login screen.

Once the user has logged in, the software will acknowledge login; click “OK” to proceed. The software will then which balance is being used, and then query whether to use the existing configuration profile or not as shown in Figure 10, below. Select Cahn (only) and use the previous configuration file if one exists.

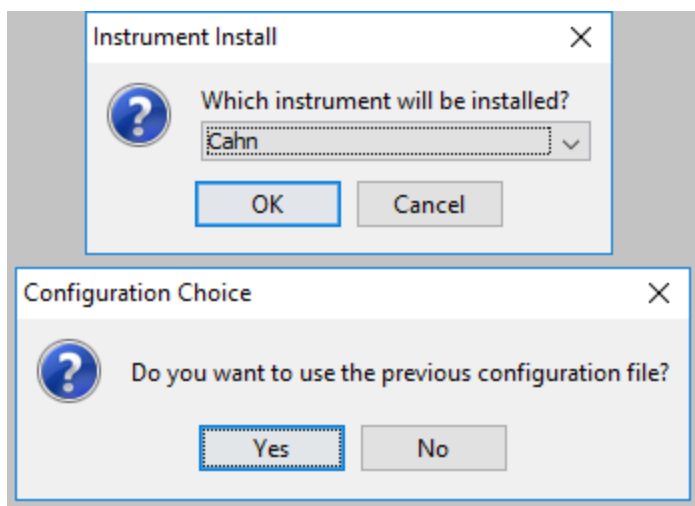


Figure 10. Instrument selection and configuration choice screens.

Java Balance Configuration

If this is the first time that the Java balance software is being used on this computer, the proper configuration values may need to be set for COM port, baud rate, etc. The proper settings for the Cahn balances are shown in Table 1, below.

| Property | Cahn Balances |
|--------------|---------------|
| Baud rate | 4800 |
| Parity | None |
| Data bits | 8 |
| Stop bits | 2 |
| Flow control | None |

Table 1. Communication settings for the Cahn electrobalance

These values can also be accessed from the instrument’s main panel by clicking on the Configuration button on the upper left corner of the panel. This will invoke the configuration screen as shown in Figure 11, below. Be sure that you understand what each parameter should be before changing any of the fields!

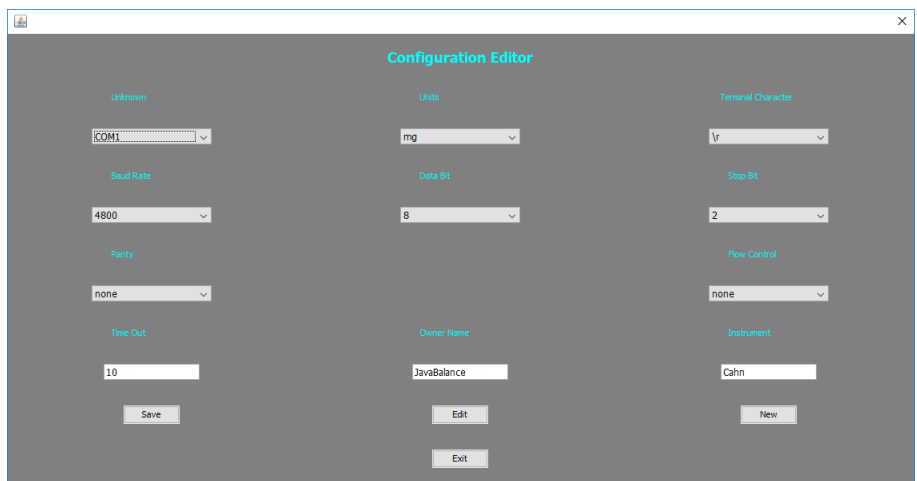


Figure 11. Java Balance configuration editor.

The user can choose between milligrams and grams for the data output; milligrams are recommended for the Cahn electrobalances. Once the configuration settings are correct, click “Save” in the lower left corner, followed by “Exit.” The Exit button will invoke the second configuration screen as shown in Figure 12. You will have to get a developer to help set the parameters on this screen.

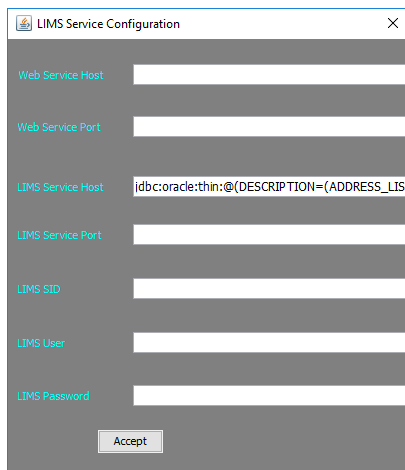


Figure 12. Java Balance second configuration editor.

Cahn Model 29 and Model 31 Electrobalance Calibration

The Cahn balances do not normally need to be calibrated even between expeditions, so the user must usually only verify the calibration is correct by measuring known masses.

If the Cahn must be calibrated, it should be done while the ship is still in port by following the calibration procedure in the Cahn balance manual. In summary, this requires zeroing the balance and weighing a 200 mg known mass, but these measurements are instantaneous and therefore very vulnerable to ship motion.

Once the Cahn is calibrated, it is necessary to measure a range of milligram-scale standard reference masses to ensure that the calibration was done properly. The ship is still bobbing up and down even in port, and if the calibration button is pressed at the wrong moment, the slope of the calibration could be off by a significant

percentage. It is necessary for the user to weigh a number of masses (e.g., 10, 20, 50, and 100 mg) to ensure that the balance calibration is linear and at least close to the origin.

If the calibration check shows that the balance is out of calibration and the ship is still in port, try again until you get a linear and accurate calibration.

If a calibration had to be performed at sea, it is unlikely that repeated attempts will be able to adjust it to “true.” Instead, the user can create a slope and intercept to correct the balance back to the proper readings. Measure known masses (e.g., 10, 20, 50, 100, and 200 mg) and determine the slope and intercept of the results against the known values, then edit the configuration file and add the experimentally-determined slope and intercept.

It will be necessary to close the software and re-open it for the new slope and intercept values to be used, and the user should then measure the same series of known masses used for calibration as unknowns to make sure that the correct factors are now in place.

Do **not** use the Java Balance calibration screen to do this, as it was set up for instantaneous, not time-series, measurements. Because of this, the last calibration shown may seem quite out of date; this is not a problem provided the user weigh known masses to ensure the balance is working properly.

Workflow

Once calibration is confirmed to be valid, the Cahn electrobalance workflow consists of the following procedures:

1. Set up test options.
2. Tare balance.
3. Weigh sample or reagent.
4. Assign the mass to a sample.
5. Save mass into the LIMS.

Test Options

Once the configuration is correct, the user may now select the test options by clicking the “Test Options” button. This will invoke the screen shown in Figure 13, below. The user can select between counts (strongly suggested) or standard deviation and enter the value for the selected parameter. The Cahn balance provides a mass at 1 Hz, so a count value of at least 180 is recommended to cover three minutes of the ship’s heave.

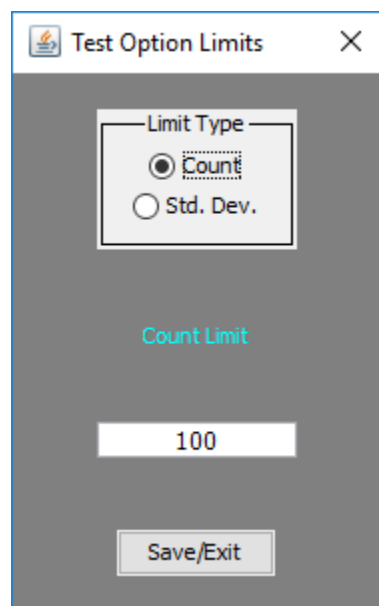


Figure 13. Java Balance test option screen. It is not recommended to use Standard Deviation mode.

Taring the Balance

Before the balance can be used, it is necessary to tare the balance via the software, **not** by the tare buttons on the balances themselves. The software tare measures the difference between the averaged tare value of the reference and unknown balance pans. Click “Tare” on the left hand side of the panel, followed by “Start” in the lower center panel. These buttons are circled in Figure 14, below.

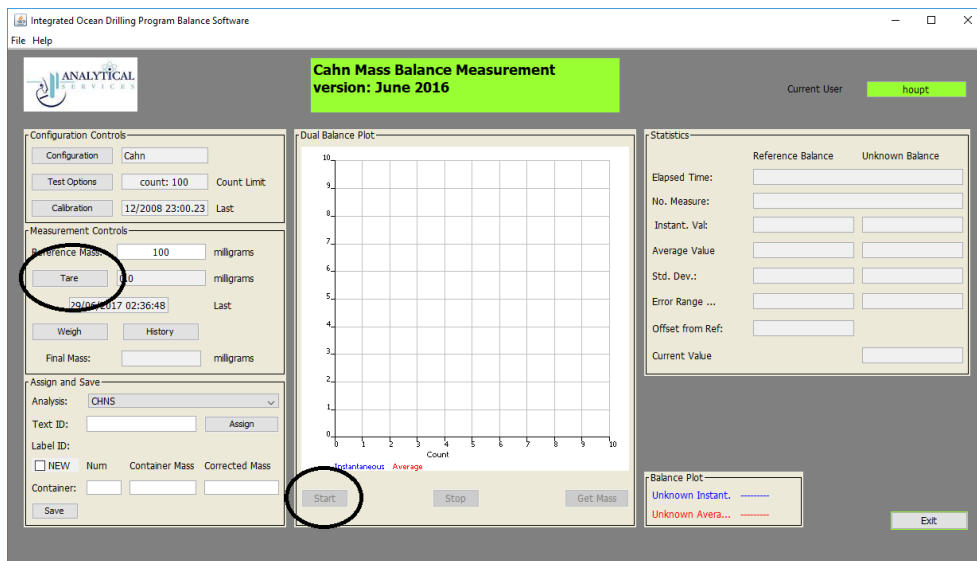


Figure 14. Taring the balance. Note that the “Start” button will be grayed out until the “Tare” button is pressed. The taring action will take as long as a regular measurement (~3 minutes). Once the selected counts have been reached, click “Stop” followed by the “Get Mass” button to the right of the “Start” button. This will save the tare value to the Tare field. See the notes in Counterweighting the Cahn, below.

Counterweighting the Cahn

Before taring the Cahn electrobalance, determine whether to tare with weighing paper or crucible:

| Reference Material | Analysis |
|--------------------|---|
| Weighing paper | CaCO ₃ by Coulometer TOC by SRA ICP sample Reagents (<1g) |
| Crucible | CHNS |

The Cahn returns the mass difference between the sample and reference/tare pans, so the best practice is to load the tare pan (right side) with a counterweight that is close to the mass of the container being used on the unknown pan. For example, if a folded piece of weighing paper is being used to collect the sample powder, place folding paper of the same size on the reference pan. **You must tare to these papers and/or crucibles, not to empty pans.**

When weighing ICP samples, it is often the practice of the technicians to tare the Cahn with weighing paper, then add a reference mass equal to the sample being weighed (100 mg) to the tare pan. This will cause the balance to show a negative number (roughly -100 mg), and then the sample powder can be added until the balance’s LED display is close to zero. Once this occurs, the mass measurement should be 0 mg ± 0.5 mg so that the ICP sample’s mass is 99.5-100.5 mg as specified in the hard rock preparation procedure. It is equally valid to have the LED show 0 and to add the 100 mg of sample, then measure the mass to ensure it’s 99.5-100.5 mg.

Weigh Sample or Reagent

Once the Cahn has been tared, add the unknown to the sample/unknown pan and click on the “Weigh” button as shown in Figure 15. The “Reference Mass” field is not used in the Java Balance software for the Cahn, so that field

can be ignored. Click “Start” and wait for the measurement to complete. Then click “Stop” and “Get Mass;” the final mass value should now be visible in the Final Mass field on the left side of the software panel.

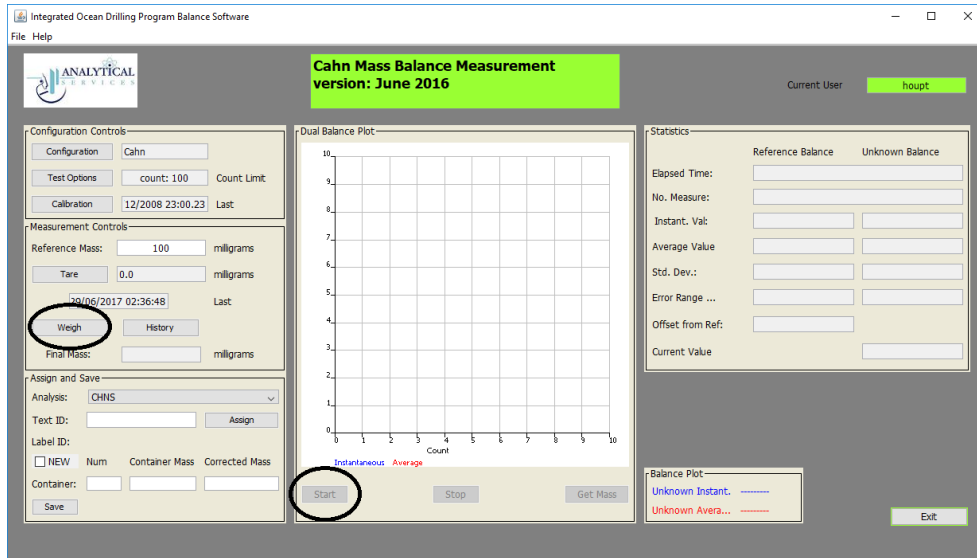
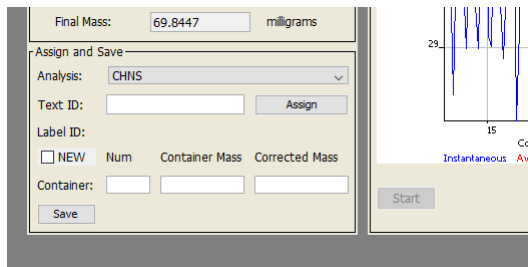


Figure 15. Making a measurement.

Assigning a Mass to a Sample and Saving to LIMS

Once the mass has been obtained, use the controls on the lower left corner of the software to select an analysis as shown in Figure 16, below. Note that the balance software will show “CHNS” at startup. If you are measuring CHNS samples, you must select away from this analysis, then back to it, in order to proceed. If you are measuring any other type of sample, simply select that analysis.



Put the cursor in the “Text ID” field and scan the sample’s barcode, then click “Assign.” This will invoke a new window with a list of samples that match that Text ID (normally only one sample unless it is a QA/QC standard) and you select it by double-clicking.

This will return to the main screen and should show the Label ID of the selected sample. If you are weighing a sample into a process that uses container numbers (and most do), enter the container number and then click “Save.” Almost all of the chemistry lab procedures use container numbers, and none of them have a mass, so leave the Container Mass field blank. That field was for Moisture and Density (MAD) analysis, for which the containers are pre-weighed, and is no longer applicable in Java Balance.

Quality Assurance/Quality Control

Calibration Verification

Calibration verification of the balance systems are carried out daily by the laboratory technical staff. Known masses should be measured at least daily to ensure that the balances are properly measuring mass; it is not

possible to see this from the values on the balance LED or LCD screens; measurements must be performed using the balance software and the time-averaged, reference-normalized methodology.

Be sure to weigh several weights to ensure that the balance performance is both accurate and linear across the desired mass range.

Expected Precision and Accuracy

The Mettler-Toledo XS-204 balances should be able to obtain a value within 10 milligrams of the true mass under fairly calm sea conditions and if the measured mass and reference mass are close to one another. Reproducibility should be less than 1% deviation between measurements.

The Cahn Model/29 and Model/31 electrobalances should be able to obtain a value within 0.05 milligrams of the true mass under fairly calm sea conditions, if not better. Reproducibility should be less than 1% deviation between measurements.

For both balances, when sea state is high, this level of performance will degrade. The user should measure reference masses to get an estimate of the uncertainty under high sea state conditions.

Maintenance

Mettler-Toledo XS204

Cleaning

- Clean the weighing pan, drip tray, housing, and terminal using a brush.
- Clean the weighing chamber:
 - Swivel the glasses of the draft shield away from the balance and remove from fastenings
 - Raise the front of the weighing pan and lift out of the guide
 - Pull the drip tray away from the balance
- Do not use cleaning agents containing solvents or abrasives
- Do not allow liquids to penetrate into the balance, terminal, or AC adapter

Errors

| Error Display | Problem | Resolution |
|------------------------------------|---|--|
| Wide bar with ends facing downward | Overload: weight exceeds balance capacity | Reduce weight on pan |
| Wide bar with ends facing upward | Underload | Ensure pan is in place, can move freely, and is not in a draft |
| 0.00000 g flashing | Error when switching on or zeroing | Remove items from weighing pan |
| Timeout | Taring/zeroing aborted - no stable result | Check for drafts or vibration |

Specifications

| | |
|--------------------------|----------------|
| Maximum load (g) | 220 |
| Readability (mg) | 0.1 |
| Taring range (g) | 0–220 |
| Repeatibility (sd) | 0.1 mg @ 200 g |
| Linearity (mg) | 0.2 |
| Eccentric load deviation | 0.3 mg @ 100 g |

Cahn Electrobalance

Cleaning

- Immediately clean all spilled material
- Wipe up spills with lintless paper cloth
- Remove stirrups and sample pans from hangdowns inside weighing compartment
- Secure hangdown and press the **Brake** button before moving balance

Specifications

| | |
|---------------------------------------|-------|
| Capacity (mg) | 250 |
| Weight range (mg) | 200 |
| Sensitivity (μg) | 1 |
| Electrical tare (mg) | 250 |
| Accuracy (μg) | 2 |
| Precision: ultimate (μg) | 1.0 |
| Precision: fraction of load (%) | 0.001 |

Additional Information

The following additional information is available upon request:

- Cahn Microbalance Instruction Manual (vendor)
- Mettler-Toledo XS204 Analytical Balances Operating Instructions (vendor)

Definitions

Accuracy - the agreement of a balance reading with the value as set by a reference standard.

Average Value – average value of all values taken by program.

Capacity - the maximum total load (sample + container) that the balance can handle. If load is increased beyond the rated capacity of the balance, the balance may become damaged, reducing accuracy and repeatability.

Configuration (setting) – set of instrument and software parameters set by instrument manufacturer and/ or software developer. For balance user purposes these are not changeable.

Calibration - standardization of measuring instrument: the checking of a measuring instrument against an accurate standard to determine any deviation and correct for error or determination whether or not characteristic of the instrument are within specifications. For Mettler-Toledo and Cahn electrobalance calibration is completed onshore with manufacturer.

Container # - sample container or holder number for particular measurement.

Current Value – net weight of material/sample being weighed.

Elapsed time – time in seconds that has elapsed since measurement started.

Error Range - the size of the error bar for each point.

History - shows a list of tare value or list of final weight (mass) number since last log-in.

Instant. Val. – value each balance is sending at the moment or instant.

NO. Measurement – number of measurements the software uses to determine final mass. Parameter set in Test Options.

Offset from Ref: - difference between the unknown and known balances.

Objective – reason for sample measurement, lists sample types.

Readability - the least significant digit that can be resolved from the readout under normal conditions.

Reference Mass – a known mass that is used to accurately determine unknown mass.

Repeatability - (Precision, as defined by Thermo/Cahn) is the standard deviation of 10 consecutive readings of the same mass under ideal conditions.

Sensitivity - smallest mass difference that can be detected by the balance.

Std. Deviation – standard deviation of all values taken by program

Tare – tells the program to ignore any weight currently on balance and measure new from that weight.

Test Option – sets parameters for current test. Includes number of counts required for measurement. This is how the balance knows when to stop taking measurements. Standard deviation limit and confidence limits are also shown here but are not changeable by the user.

Weigh – lets software know you are in 'weigh' mode, ready to weigh a sample.