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APS-7650G

Dilution Of Oil Samples By Weight

Technical Note

Preparing oil samples by weight every day is an arduous task for many laboratories.

There was no alternative to doing this work manually, until now.

The APS-7650G Gravimetric Sample Preparation Station handles this time-consuming process, allowing labs to free up personnel for more critical tasks.

Meets requirements for test methods ASTM D5185 and ASTM D4951

ASTM Provides Two Test Methods for Oils

Laboratories doing ICP-OES analysis of oil samples commonly reference two ASTM methods in particular. One method is ASTM D-4951 Standard Test Method for Determination of Additive Elements in Lubricating Oils by Inductively Coupled Plasma Atomic Emission Spectrometry. The other is ASTM D5185 Standard Test Method for Determination of Additive Elements, Wear Metals, and Contaminants in Used Lubricating Oils and Determination of Selected Elements in Base Oils by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES).

The two standards have many similarities, including the requirements for sample preparation. Both methods require that the samples and standards be diluted by mass with an appropriate solvent, typically kerosene, prior to analysis. Also, both recommend mixing the samples thoroughly after dilution to achieve a homogenous mixture. The precision of the dilution, cleanliness of the process, and final homogeneity of the samples are all critical to the success of these analyses, especially when working with new oil samples.

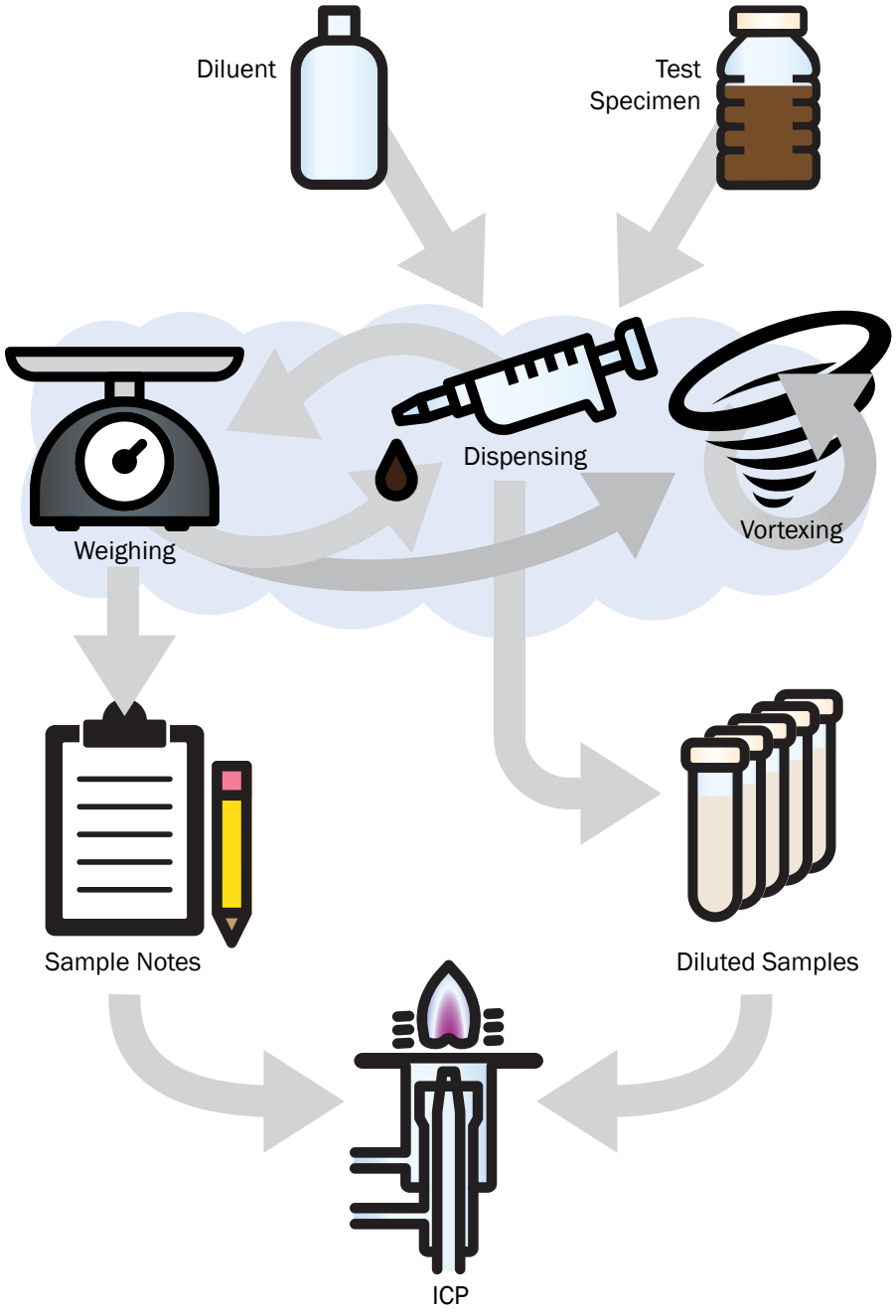


Figure 1. Overview of the manual dilution process

The process of preparing oil samples by hand is tedious, time consuming, and error prone.

1. Prep notepad with sample names
2. Place empty tube on scale
3. Tare scale
4. Pipette raw sample into tube
5. Record weight of sample on notepad
6. Carefully add diluent to 10x value
7. Record final weight on notepad
8. Cap sample (optional)
9. Mix sample tube on vortexer for 20-30 seconds
10. Check that sample is homogenous, if not, mix more
11. Place sample tube in rack
12. **Repeat above for each sample**
13. Place rack on autosampler
14. Type sample name, initial weight, and final weight into ICP software for each sample
15. Finally begin ICP analysis

Manual sample dilution by weight can take anywhere from 2 - 5 minutes per sample. Even a small sample load can take hours to prepare.

Much of the Sample Preparation Can Be Automated

The APS-7650G system automates dispensing, weighing, homogenizing, recording weights, and calculating dilution factors, making the process for the user much simpler. All one has to do is place the samples into a rack, load them onto the APS-7650G, create a sequence with sample names, and start the run. After that, the user is free to do other work around the lab.

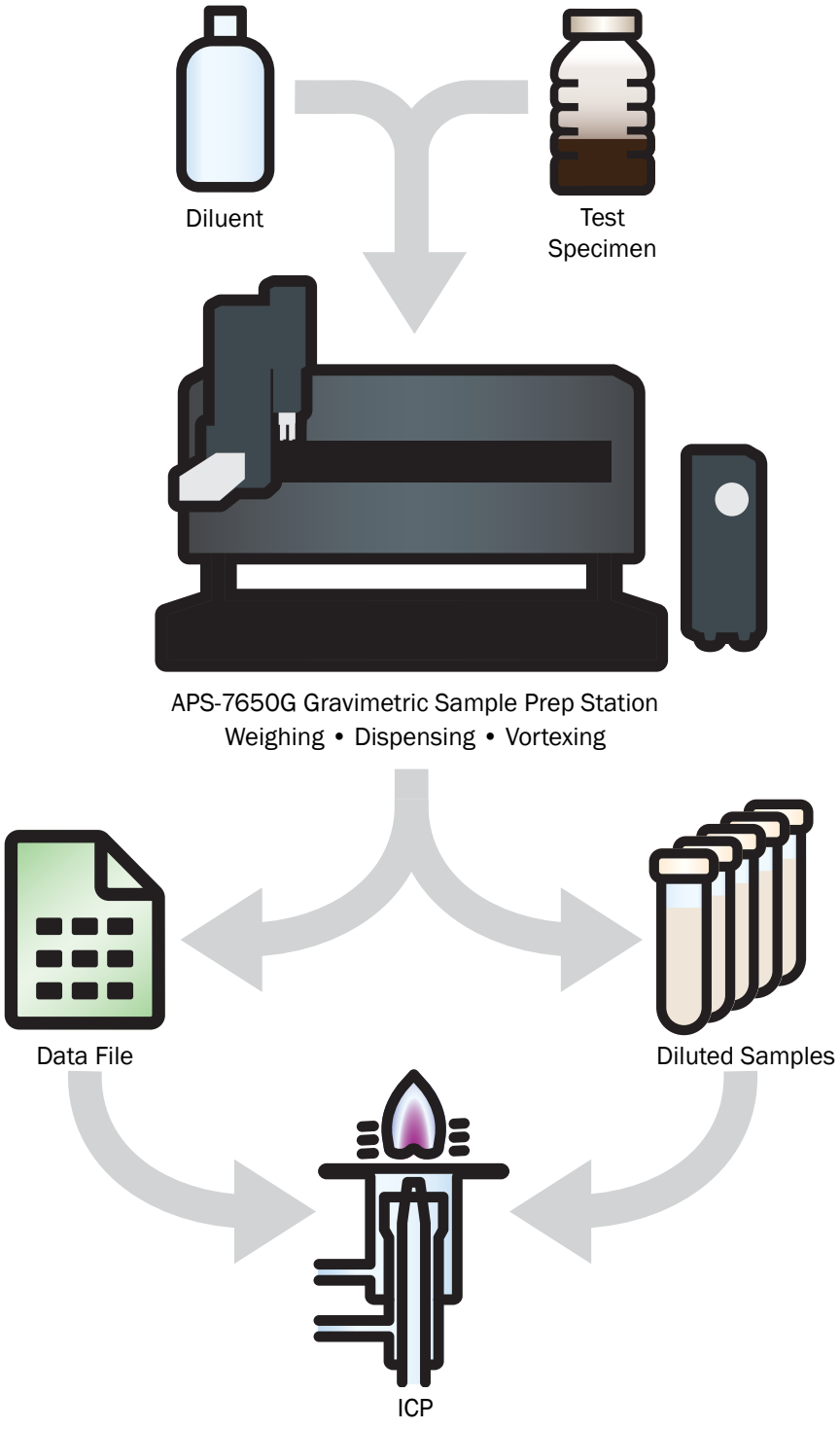


Figure 2. Overview of the automated dilution process

The automated system performs all the same steps as the manual process.

1. A fresh 1 mL wide bore pipette tip attaches to the built-in pipettor.

2. An empty sample vial is picked up using the gripper attachment.

3. A 1 mL air slug is aspirated into the pipettor before the test specimen is aspirated* into the pipette tip.

4. The empty vial is placed on the integrated balance and weighed.

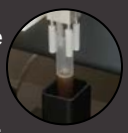
5. The test specimen is intelligently dispensed† into the vial on the balance to sufficient mass.

6. The pipette tip is discarded and the pipettor is emptied of remaining air.

7. The sample vial is transferred to the vortex vessel and mixing begins.

8. A precise volume of diluent is added over the duration of the vortexing to ensure accurate dilution.‡

9. The sample vial is returned to the balance and the diluted weight is measured. The dilution factor is calculated by the OilEase software.



10. The sample vial of diluted and homogenous sample is returned to its original position.

The process repeats until all samples in the sequence are completed. When the sequence is finished, the data is exported in an ICP ready format.

* There is a system pause during aspiration to ensure optimal sample volume for viscous samples.

† Intelligent dispensing occurs in two steps. First the sample volume is dispensed and a mass reading is taken. If mass is sufficient, the system moves onto the next step. If insufficient, the aspirated air slug is dispensed to evacuate the remaining oil.

‡ Diluent is dispensed from a highly precise sample syringe. The volume of diluent to be added is determined from the sample weight to ensure that the precise dilution factor is always accurately achieved.

Capabilities and Limitations

The APS-7650G meets the specific requirements of the ASTM standards for:

- Balance precision of 0.0001 g (D4951-09 §6.2, D5185-09 §7.1)
- Ensuring that all glassware and so forth do not contaminate (D4951-09 §X2.8)
- Maintaining consistent oil to solvent ratio when diluting (D4951-09 §X2.2, X2.19, D5185-09 §X1.20)

With the above process the APS-7650G fully meets the sample preparation requirements of ASTM D5185 and D4951. In addition, automating the process removes the opportunity for human error both in the sample preparation and in the data entry steps. By integrating the balance, pipettor, gripper and vortexer, the first of its kind APS-7650G can automate this arduous manual task, ensuring precise dilutions with zero carryover and full sample homogenization. This in turn leads to more reliable sample data and more available lab staff.

References

1. ASTM D4951-14 Standard Test Method for Determination of Additive Elements in Lubricating Oils by Inductively Coupled Plasma Atomic Emission Spectrometry
2. ASTM D5185-18 Standard Test Method for Multielement Determination of Used and Unused Lubricating Oils and Base Oils by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)

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