CALIBRATION OF AUTO-PIPETTES, DISPENSERS AND SYRINGES IN THE GC-PESTICIDES GROUP

1. SCOPE AND APPLICATION

The accuracy of analytical procedures performed by pipettes, dispensers and syringes should be checked by calibrating these devices at least once per quarter. This SOP describes the procedure used to calibrate them.

2. SUMMARY OF THE METHOD

Pipettes, dispensers, and syringes are calibrated using the known density of toluene. Aliquots of toluene are pipetted and weighed. The density of toluene is used to calculate the actual volume pipetted and this is compared to the expected volume. The accuracy (% difference) of the mean volumes checked and the precision (% RSD) of replicate measurements are evaluated.

3. APPARATUS AND EQUIPMENT

3.1. Weighing vials

3.2. Toluene, Reagent grade

3.3. Analytical balance (accurate to 0.0001 g)

3.4. Calibrated thermometer

4. PROCEDURE AND DOCUMENTATION

4.1. All pipettes are identified by unique ID numbers or serial numbers. These ID numbers are documented on the pipette labels and in the pipette calibration worksheet. If you start using a new or different pipette, notify your supervisor and modify the worksheet accordingly.

4.2. There is a pipette calibration worksheet template for the GC pesticides workgroup located in the current year subfolder in the “Syringes and Dispenser Cali” folder on the GC drive at \\FLDEP1\DEAR\Labs\Lab5\Chemistry\Organic\GCgroup\. Open the “GCGroup-SyringeCal-1.1” excel file. Each pipette, dispenser or syringe is assigned one sheet in the file containing a calibration table for that specific device. Additionally, the first sheet contains a temperature/density table for toluene to which all calculations are linked. All syringes, pipettes and

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dispensers used by the GC group for both preparations and analysis are contained in this file.

4.3. In the calibration worksheet, identify the pipette that you are going to calibrate. For each pipette, the volumes to be used for the calibration must be entered in the next available line. For adjustable pipettes, that are used at different volumes, check the calibration at a low point (10% of the nominal volume) at the midpoint (50% of the nominal volume) and at the nominal volume to bracket the range of use. For a fixed-volume pipettes (10 mL pipettes used in preparation) or other fixed volume devices check the calibration at the volume used (usually nominal volume). If any changes to the worksheet are needed, contact your supervisor. Record the analyst, date, balance serial number, thermometer ID and temperature of the toluene used for the calibration.

Note: When using variable volume pipettes, the accuracy at the maximum, middle and minimum values are checked. If only one value is calibrated the use of the pipette is restricted to this specific volume. The volume must be documented on the pipette label (see 4.7).

4.4. Check the calibration of the balance to be used (see DEP SOP CM-011). Bracket balance weight checks around expected measured mass values.

4.5. Locate a vial /vessel that can hold the volume of toluene to be pipetted. Weigh the weighing vessel and tare balance. If you are calibrating a variable volume pipette, make sure the pipette is set to the correct volume. Pipette an aliquot of toluene into the vessel and **record the weight of the aliquot** in the appropriate cell. Tare the balance. Repeat for a total of five measurements of each volume checked. The volume in mL for each aliquot will be calculated and appear in the adjacent cell. The mean volume in uL, the RSD of the replicate measurements, and the % difference will be calculated automatically.

4.6. Check the calculated results in each column of the worksheet. The percent differences must not be greater than laboratory guidelines listed below the table on each sheet. If all the calibrations are acceptable, save the calibration worksheet and notify your supervisor to review the worksheet. If a calibration is not acceptable follow the corrective action procedure as described in section 5.

4.7. Save the calibration worksheet file. Report to the supervisor that the calibrations are completed and ready for review. The supervisor's duty is then to review the results and protect the new entries by converting all cells in the newly entered lines to the locked status. The locked status is password protected. Once all sheets are locked and password protected the file is saved and then saved again as pdf that carries the date stamp. The pdf file is saved as GCGROUP-SYRINGECAL-1.1_mm/dd/yy.

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4.8 Place labels with expiration dates on all calibrated devices.

5. QUALITY CONTROL AND CORRECTIVE ACTION

5.1. If a calibration result is not acceptable a corrective action has to be taken. The device in question pipette should be inspected for maintenance, adjustment, or repair, and the calibration must be repeated.

5.2. If the calibration fails again, a comment will be added to the worksheet indicating that the pipette cannot be adjusted and is removed from service. Notify your supervisor if a pipette could not be calibrated successfully. It must be repaired and re-calibrated or taken out of service. During review and repair, the failing pipette should be clearly labeled "OUT OF SERVICE". Pipettes that cannot be repaired should be removed from the laboratory.

6. CALCULATION

The following equation is used to calculate the percent difference between the nominal volume pipetted and the calculated volume. The worksheet will calculate this value automatically. The result is recorded in the "% Difference" column.

$$\%D = |(V_e - V_a) / V_e| * 100$$

V_e = Volume of toluene according to pipette setting (mL)

V_a = Average volume of toluene (mL)
$= \sum[V_n] / n$

V_n = Volume of n^{th} aliquot of toluene (mL)
$= (W_n$ / Density of toluene at given temperature) * 1000

n = Number of aliquots pipetted

W_n = Weight of n^{th} aliquot of toluene (g)

%D = Percent difference between volume of toluene according to the pipette setting and the calculated volume.

The %RSD is also calculated automatically in the worksheet as the standard deviation of the replicate measurements in µL divided by the mean volume in mL. The result is recorded in the "RSD of Vol." column.

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7. REFERENCES

7.1. DEP SOP CM-011, Gravimetric Analysis: Analytical Weight and Balance Calibration

Appendix of Significant Changes

02/06/2019 Added midpoint measurement to the adjustable volume pipettes.

01/28/2021 Expiration labels added.

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