1.0 Principle: Proper function of laboratory equipment and instrumentation is essential to ensure the forensic defensibility of the testing process. The proper function of most terminal instruments (e.g. GCMS, Headspace-GC) will be demonstrated on a per-batch basis with appropriate blank and control materials, see specific TX SOPS. Other important equipment, such as analytical balances, manual and automated pipettes may require monitoring (e.g. refrigerator temperature, pipette function).

2.0 Procedure: Analysts shall ensure that the equipment used in forensic sample analysis were appropriately validated, and are functional for use. It is also the responsibility of the analysts to ensure that documentation regarding such validation and functional operation (e.g. pipette checks, refridgerator temperature records) are kept up to date.

2.1 Repeat Pipette Validation:

At any time if there is a precieved issue with a certified pipette, it can be checked in-house by determination of accuracy and precision. Variable-volume pipettes are checked at the low, mid and high volume points. Note; a variable volume pipette may be checked at a single fixed volume point, however, the "validation sticker" must indicate the validation is only for that volume and that the validation is nullified if the pipette set volume is altered.

All pipettes are marked with a unique identifier, and documentation of pipette validation is maintained in "Validation of Miscellaneous Equipment, notebook in the Toxicology Laboratory:

Toxicology Pipettes: "TX" and a number; e.g."TX-2"

Note: Class A Volumetric Pipettes need not be validated. However, such pipettes shall be discarded if the tips are cracked, chipped or otherwise broken or damaged.

2.1.1 Determination of accuracy and precision:

2.1.1.1 Using an appropriate, certified balance, pipette and weigh 10 portions of room temperature D.I. water, of the target validation volume. Record the weights determined on a "Pipette Calibration" form. Assure that the read out of the balance used is sensitive enough to allow for an accurate calculation of standard deviation (see section E.1 below).

2.1.1.2 Complete the "Pipette Calibration" form and calculate the % Recovery, Standard Deviation, Coefficient of Variation, Average Deviation and Pipette Accuracy.
Note: an excel spreadsheet set up to calculate the above is available on the S drive. When using this spreadsheet the analyst should review the cell calculations to assure they account for the needed data points:
s:/toxi pipette validation/pipette validation worksheet master(TX-14:2)

2.1.3 If performance of the pipette is acceptable, The Pipette Calibration form must be reviewed and signed by a second analyst, and a validation sticker attached to the pipette.

2.1.3.1 The validation sticker will have the date of validation, the pipette number, the initials of the person performing the validation; and (in cases were only a specific volume is validated) the volume for which that pipette is validated.

2.1.4 The validation paperwork will be filed in the appropriate section of the 'validation of miscellaneous equipment' notebook, located in the Toxicology section.

2.1.5 Annual Verification

2.1.5.1 Pipettes will be certified annually by a contract vendor. The vender most be an ISO certified company or equivalent. The certificate of validation/calibration will be maintained within the laboratory section in the 'validation of miscellaneous equipment".

2.1.5.2 Pipettes that are validated/calibrated by an outside vender will be verified in-house prior to use for casework, as follows.

2.1.5.3 The vendors certificate will be reviewed by the section Supervisor or QM to determine (this review will be annotated by initialing and dating the certificate):

2.1.5.3.1 The found condition, was it acceptable or out of range

2.1.5.3.2 The as left condition, was it acceptable or did it fail

2.1.5.4 If the "as found" condition was acceptable and the "as left" condition was acceptable, the above method (see 2.1) will be used however, the check will be performed only at the mid-point of the pipettes range, or if the pipette is primarily used for a specific volume the check will be at that volume. The acceptance criteria as listed on worksheet (TX14:2).

2.1.5.5 If the "as found" condition was unacceptable inform the Deputy Director of the problem. Based on the nature of the issue and how the pipette is used (qualitative v. quantitative) a QAR may be initiated to determine if any case findings need to be reevaluated.

2.1.5.6 If the "as left" condition is unacceptable mark the pipette as out of service or dispose of the pipette.

Note: If the full range of a variable-range pipette is not found acceptable, the pipette must be annoataed with appropriate use limits, or marked as out of service or disposed of.

2.2 Automated pipettes;

Automated pipettes will be validated annually in-house (or sooner if a problem is perceived), by the mechanism noted above (sections 2.1.1-2.1.5 above). Note: when possible, such devices may be validated by performance. E.g. the automatic pipetter-diluter used for dilution of volatile samples may be validated by performance on certified reference material. (Example; 10 CRM samples in sequential batches within acceptable range would indicate functional validity).

2.3 Analytical Balances;

2.3.1 Analytical balances will be calibrated annually by a contracted vendor that is ISO certified or equivilent. A certification sticker provided by the contracted vender will be placed on the balance if it is acceptable for use.

Note: Moving Balances: Balances which need to be moved between annual validations will be checked by the
section supervisor or designee using NIST traceable certified weights. The

balance's suitability for use will be demonstrated by weighing the certified weights in the normal working range of the balance; the values must be within 1% of the expected value for the balance to be considered acceptable for use. Values outside of acceptable ranges will require calibration by the contracted vender.

2.4 Day of Use Checks;

Balances used on any given day for casework will be verified with a check mass. Check masses are NIST traceable weights available for daily use. The day of use the analyst will weigh a check mass and record the value on the 'Analytical Balance Check Sheet' (TX-14:1) located next to the balance and complete the form as appropriate. If the weight is out of range a 'do not use' sign is placed on the balance and the analyst reports this to the section supervisor, or Deputy Director immediately. The balance will be investigated to determine if it is acceptable for use.

2.5 Masses

2.6.1 Masses used as daily check weights (gram sizes) will be calibrated an approved contracted vendor no less than every 2 years. The calibration paperwork will be on file with the Quality Section.The acceptable range, for masses used in the daily check, are based on the mass corrections obtained during the annual certification.. For this the upper and lower uncertainty values are used to calculate the acceptable range which is -1% of the lower reported value and +1% of the upper reported value.

2.6 Thermometers

The thermometers used in this laboratory are used only for non-essential temperatures such as for recording refrigerator temperatures. Current procedures have no specific temperature ranges which must be maintained.

2.6.1 Thermometers will be verified annually. The reading will be recorded on the Annual Thermometer Check worksheet (TX-14:5).

2.6.2 Thermometers will be designated by TX for the section with a number designation after the letter designation(example TX#1) or by the serial number of the device. This designation will be written on a sticker on the thermometer itself or on the container directly related to the thermometer, when the serial number is used it is etched into the devise by the manufacturer.

2.6.3 Using a NIST traceable thermometer (with valid certification) place both the NIST traceable thermometer and the one to be checked into a refrigerator or appropriate device for ~1/2hour. The reading of both will be taken and recorded on the annual Thermometer Validation' worksheet (TX14:5). The thermometer is acceptable for use if the thermometer being validated is +/-2° of the reference thermometer. Note: for freezers the reference thermometer will be in a range appropriate for the device. When verifying digital thermometers associated with waterbaths the device should be turned on and set to its normal setting, the comparision to the NIST traceable standard should be made after the set temperature is reached and stable.

2.6.4 Thermometers that are verified will have a green validation sticker placed on them with the date verified and the analyst's initials.

2.7 Refrigerators

Refrigerators are used to store some standards, reagents and biological evidence. The temperatures are monitored weekly to ensure that there is not a gradual temperature drift which may indicate refrigerator failure.

2.7.2 Temperatures of refrigerators will be monitored once weekly and recorded on the temperature record log form (TX-14:4) attached to each specific unit. At the end of each year the log will be filed in the 'validation of miscellaneous equipment' note book.

3.0 Controls

The paperwork generated by the above processes will be periodically reviewed by the section supervisor to verify compliance with the procedures.

4.0 Definitions

4.1 Accuracy; the degree of conformity of a measured or calculated quantity to its actual (true) value.

4.2 Precision; the measure of the degree of mutual agreement among a series of individual measurements, values or results.

4.3 Standard Deviation; the square root of the variance or a measure of statistical dispersion measuring how spread out the values are.

4.4 Percent Recovery; a measure of accuracy, this compares the average value from the expected value expressed as a percentage.

4.5 Variance; measure of statistical dispersion indicating how values are spread around the expected value.

4.6 Average Deviation: used to show reproducibility, a measure of the absolute difference of a data set from a given point usually the mean of the data set.

5.0 Calculations: It is suggested that the calculations listed below be performed through an Excel spread sheet. (See sample Appendix A)

5.1 Standard Deviation

$$\sigma = \sqrt{\mathrm{E}((X - \mathrm{E}(X))^2)} = \sqrt{\mathrm{E}(X^2) - (\mathrm{E}(X))^2}$$

5.2 Accuracy: 100 - % recovery

5.3 Percent Recovery: $\frac{\text{(expected value – average value)}}{\text{(expected value)*100}}$

5.4 Variance: V = (σ / average value)*100

5.5 Average Deviation: $\frac{1}{n}\sum_{i=1}^{n}|x_i - \overline{x}|$

6.0 Equipment Failure

6.1 Any equipment which fails a validation procedure or in the case of refrigerators starts to work outside of normal working parameters will be immediately reported to the section supervisor or designee.

6.2 The section supervisor (or designee) will take needed steps to correct any instrument failures or take the equipment out of use. Major equipment failures need to be reported to the Deputy Director so that replacements can be arranged if needed.

Note: If for any reason equipment is sent out of the laboratory for repair, the Lab will demonstrate proper function of the equipment as shown above.

7.0 Records

7.1 Validation records: forms completed to document the validation of any miscellaneous equipment will be filed in the 'validation of miscellaneous equipment' note book for the individual section. These records will be maintained for a minimum of ten years.