# 30 QUALITY ASSURANCE

## 30.1 Introduction:

30.1.1 The purpose of this section is to provide a uniform Quality Assurance Program for the Drug Analysis Section of the Virginia Division of Forensic Science. It is to establish a baseline or reference point of reliability and system performance.

30.1.2 It is expected that the analyst will report any unacceptable or anomalous behavior of any of our analytical systems immediately to either their section supervisor or the appropriate Instrument Specialist (Primary Operator). It is further expected that appropriate corrective actions will follow ASAP and be properly documented.

30.1.3 For further detail, refer to the appropriate Quality Manual Section.

## 30.2 Reagents:

30.2.1 Chemicals and solvents used in qualitative reagents should be of at least ACS reagent grade or better.

30.2.2 Solvents used to dissolve samples or standards should be a high quality, low residue solvent (e.g., HPLC grade, OMNISOLV, OPTIMA or better).

30.2.3 Water used in reagent preparation should be of either deionized (DI) or reverse osmosis (RO) quality.

30.2.4 Stock solutions of general color test reagents and TLC sprays will be made up as needed. After they are made, they will be checked with the compound listed below in Table 2 and results, date and initials will be recorded in the log book. This verification and documentation will be repeated every three months during the shelf life of the reagent (2 years).

30.2.5 Individual chemists may have unique reagents other than the ones listed in Table 2 and it will be their responsibility to check them with an appropriate compound and document accordingly. For single use reagents, this documentation may be in the case file.

TABLE 2: Common Reagents and Appropriate Check Compounds

| REAGENT | CHECK COMPOUND |
|---|---|
| Duquenois | Marijuana |
| Marquis | Heroin |
| Froehdes | Heroin |
| Meckes | Heroin |
| Cobalt Thiocyanate | Cocaine |
| Ehrlichs | LSD |
| Iodoplatinate | Cocaine |
| Iodoplatinate/Ceric Sulfate | Caffeine |
| $KMnO_4$ | Acetaminophen |
| Fast Blue B and Fast Blue BB | Hash Oil or Marijuana |

## 30.3 Standards:

30.3.1 Primary and quantitative reference standards should normally be at least of USP-NF quality. This applies to both powders purchased from a manufacturer/supplier and pharmaceutical preparations.

30.3.1.1 Primary standards are those purchased from a reputable manufacturer.

30.3.1.2 Secondary standards are those which are obtained or synthesized within the laboratory. These may be from previously analyzed case samples.

30.3.1.3 Quantitative standards have a specified concentration and are purchased from a reputable manufacturer.

30.3.2 For all standards, a qualified examiner will be responsible for obtaining a mass spectrum, IR or other suitable definitive instrumental data (data provided by the manufacturer is not sufficient) for all standards obtained for drug lab use. The hard copy data will be filed. The hard copy should include the following data:

- Lot# or log book code
- Standard name
- Concentration, as appropriate
- Amount injected (for MS only)
- Analyst's initials and date

30.3.3 After the examiner gathers the data and insures that it agrees with known published spectra or that the data is consistent with the unique compound represented from both a chemical and data interpretation perspective, label the standard bottle in red ink with "MS", "IR", etc., the date and initials. Include the source of the standard and record all pertinent information in the Standards File.

30.3.4 If an examiner needs a standard from a new lot that has not been documented in this fashion, the examiner must perform the above procedure prior to using it for drug case work.

30.3.5 When positive results are achieved in casework, the corresponding standard(s) must be properly documented in the case file. Standards used for TLC should be documented in the case notes, whereas standards used for instrumental methods may be documented either in the case notes or on the data for the standard. If the same standard is used for both types of tests, it only needs to be documented once in the case file.

**30.4 Balances:**

30.4.1 All analytical and toploading balances will be checked weekly for accuracy using Class S-1 weights or better. Record the weights in the log book with the date and analyst's initials.

30.4.2 All high capacity balances will be checked monthly for accuracy using Class S-1 weights or better. Record the weights in the log book with the date and analyst's initials.

30.4.3 The below listed balances are intended as examples of a balance class or type with appropriate check weights:

TABLE 3: Balances and Appropriate Check Weights

| BALANCE TYPE | BALANCE EXAMPLES | CHECK WEIGHTS |
|---|---|---|
| Analytical | Mettler AE 160<br>Sartorius Basic | 10.0 (± 0.2) mg,<br>1.000 (± 0.0005) gram,<br>100.0000 (± 0.002) grams |
| Toploading (± 0.01) gram | Mettler PE 2000<br>Mettler PE 1600<br>Mettler PB302<br>Ohaus Port-O-Gram<br>Sartorius 2100 | 1.00 (± 0.02) gram,<br>10.00 (± 0.05) grams,<br>100.00 (± 0.05) grams |
| Toploading (± 0.001) gram | Ohaus Explorer<br>Mettler PB303 | 1.000 (± 0.002) gram,<br>10.000 (± 0.005) grams,<br>100.00 (± 0.005) grams |

| | | |
|---|---|---|
| High Capacity | A.N.D. Electronic | 1000 (± 1) grams |

30.4.4 Accuracy and precision must be established after a balance has been put into service after purchase, calibration, maintenance or repair.

30.4.4.1 The check weights listed in Table 3 are weighed and recorded five times.

30.4.4.2 The mean and % relative standard deviation (%RSD) are calculated for each weight.

- %RSD = 100 * (standard deviation / mean)

30.4.4.3 Acceptance Criteria:

30.4.4.3.1. The accuracy of each weight should meet the criteria in Table 3.

30.4.4.3.2. %RSD must be less than or equal to 5 %.

**30.5 Thin Layer Chromatography:**

30.5.1 The TLC bath solutions will be made up as needed and recorded in log book with date and initials.

30.5.2 The baths will be refreshed daily.

30.5.3 The performance is checked on sample(s) by running the standard along with the sample(s).

**30.6 Gas Chromatographs:**

30.6.1 Weekly

30.6.1.1 Change all septa (unless a Merlin Microseal is installed). Record in the log book, date and initial.

30.6.1.2 The column performance is checked on sample(s) by injecting the standard prior to the sample(s).

30.6.2 Monthly

30.6.2.1 Run DFTPP, Methamphetamine HCl, Cocaine and Heroin standards. The concentration of these standards should be 2 mg/mL or less. Record in log book, date and initial. Maintaining hardcopies of the GC runs and integration data may be substituted for logbook, if desired. The chromatogram should demonstrate good chromatographic performance. Acceptable results must be documented in either the logbook or on the data. Any performance discrepancies or degradation must be reported immediately to the supervisor. Store hardcopy of data for approximately one year.

30.6.3 Every 3 Months

30.6.3.1 Change injection port liners.

30.6.3.2 Clean FID detectors. Low usage FID detectors will be cleaned only as needed.

30.6.4 Yearly

30.6.4.1 Remove columns, clean injection ports and FID detectors. Reinstall or replace columns as needed.

30.6.4.2 Clean or replace split vent line and clean solenoid on HP5890 series gas chromatographs.

30.6.4.3 Replace the split vent traps for Agilent 6890 series gas chromatographs.

**30.7 Drug Analysis Mass Spectrometers:**

30.7.1 Daily

- Autotune - Use "Freshtune" and "Standard Spectra Autotune" for 5972 and 5973 GC/MS systems for routine "seized" drug analysis. Quicktune or its equivalent may be used as determined by the primary operator. Specialized tunes may be used on a case by case basis as determined by the primary operator and the examiner in concert with the Section Chief.

  Table 4: Standard Spectra Autotune Acceptance Criteria

| TUNE PARAMETER | SPECIFIC PARAMETER | ACCEPTANCE RANGE |
|---|---|---|
| Ion Abundance/Ratios | 69 | = Base peak |
| Ion Abundance/Ratios | 219 | 35-99 % of the 69 ion |
| Ion Abundance/Ratios | 502 | >1% of the 69 ion |
| Peak widths | 0.55 amu | ± 0.05 amu |
| Mass assignment | 69.00, 219.00, 502.00 amu | ± 0.10 amu |
| Isotope Ratios | Ratio of mass 70 to 69 | 0.5 – 1.6% |
| Isotope Ratios | Ratio of mass 220 to 219 | 3.2 – 5.4% |
| Isotope Ratios | Ratio of mass 503 to 502 | 7.9– 12.3% |

- Run Background
- Store hard copy of daily autotune and retain for 5 years. The background data file may be retained electronically.
- Check number of injections made, change septum if necessary or perform maintenance on "Merlin Microseal" if needed and record in log book.
- If GC/MS is used for GC retention time data, run appropriate standards as needed and retain hardcopy for lab files. Lab standards must be retrievable and maintained for the same time period as the case files. They may be kept in the laboratory until the case files are sent to archives. At that time the lab standard files must be archived along with the case files.

30.7.2 Weekly

- Run Methamphetamine HCl, DFTPP, Cocaine and Heroin reference standards and store representative peaks and spectra in file. Concentration should be 2 mg/mL or less. The chromatogram should demonstrate good chromatographic performance. Acceptable results must be documented in either the logbook or on the data.
- Copy data of Autotune, background and reference standards and send to central lab. Retain hard copy for lab files at least one year.
- Check Helium and other gas supplies, replace as needed.
- Replace injection port liner as needed.

30.7.3 Biweekly

- Archive data files to magnetic tape, a second hard drive or to writeable CDROM. Retain for at least five years.

30.7.4 Monthly

- Check PFTBA level and do full source clean, if needed.
- Check mechanical pumps oil level.
- Clean injection port if needed.
- Record any maintenance performed in log book, date and initial.

30.7.5 Semi Annually

- Perform preventive maintenance as required, record in log book, date and initial.
- Archive important macro files to magnetic tape, removable disk or writeable CDROM. Include macros, methods and user libraries for HP 5972 and 5973 systems.

30.7.6 Yearly

- Replace GC column with a new one. Record in log book, date and initial.

**30.8 HP 1100 LC/MS:**

30.8.1 Liquid Chromatograph

30.8.1.1 Due to the varied nature of chromatographic conditions employed with this device and the various ionization methods normally used, a strict regimen of procedures cannot be followed. It is expected that appropriate standards and valid theoretical applications will be employed to establish a particular compound's behavior under the applied conditions. The instrument components shall be maintained to manufacturer's specifications, using repair/replacement guidelines set forth by the manufacturer, or to maintain optimum operating conditions.

30.8.2 Mass Spectrometer

30.8.2.1 Prior to collecting data for any analysis, as needed, the manufacturer's "checktune" program or "autotune" must be run. If the instrument completely fails the checktune program, a full "reset to defaults" autotune should be run. Hard copies of tune data shall be retained for five years.

30.8.2.2 The instrument shall be maintained to manufacturer's specifications, using repair/replacement guidelines set forth by the manufacturer, or to maintain optimum operating conditions.

30.8.3 Data/Method files Archival

30.8.3.1 Data files shall be archived to writeable CD-ROM, or other suitable medium, on a monthly basis and retained for approximately five years. Method files shall be archived semi-annually.

**30.9 Support HP 5973 GC/MS:**

30.9.1 Prior to collecting data for any analysis, as needed, the manufacturer's "freshtune" and "autotune"* programs must be run. Specialized tunes may be used as determined by the primary operator. A background run shall also be done.

* Autotune maximizes instrument sensitivity over the mass range, using PFTBA masses 69, 219 and 502. Use this tune for applications requiring maximum sensitivity that do **not** require the traditional abundance ratios of 69, 219 and 502. - HP Chemstation Help (G1701AA ver. A.03.00)

30.9.2 Hard copies of tunes and backgrounds shall be retained for five years.

30.9.3 Methamphetamine, DFTPP, Cocaine and Heroin reference standards, in a suitable concentration for this instrument, should be run after installation of a new or cleaned injection port liner.

30.9.4 The instrument shall be maintained to manufacturer's specifications, using repair/replacement guidelines set forth by the manufacturer, or to maintain optimum operating conditions.

30.9.5 Data/Method files Archival

30.9.5.1 Data files shall be archived to writeable CD-ROM, or other suitable medium, on a monthly basis and retained for approximately five years. Method files shall be archived semi-annually.

**30.10 FTIR:**

30.10.1 Daily

30.10.1.1 The throughput of the bench will be documented daily. Both the maximum and minimum values of the interferogram, the location of the centerburst, and the gain setting will be recorded in the log book, date and initial.

30.10.1.2 The location of the centerburst must be stable at 1024 ± 16, if not, then align the bench and perform maintenance if needed.

30.10.1.3 If the sum (absolute value) of the maximum and minimum is less than 8.0 at a gain of 1.0, then align the bench and perform maintenance if needed.

30.10.1.4 The VAL-Q software checks a number of different performance characteristics including collecting a polystyrene spectrum and checking the peak-to-peak noise. The VAL-Q check will be run daily. If the instrument does not pass any of the VAL-Q acceptance criteria, perform maintenance as needed. A hardcopy of the report will be generated and stored in the laboratory for five years.

30.10.1.5 An uncorrected standard of procaine hydrochloride will be run on the ATR accessory and searched against a library containing uncorrected standards collected using the ATR accessory. The match should be 95% or greater. A hardcopy of the standard and library match will be generated and stored in the laboratory for five years.

30.10.1.6 A record of all samples will be kept in a logbook which includes the date, FS Lab# and/or file name, and the initials of the user.

30.10.2 Weekly

30.10.2.1 The bench will be aligned weekly using the "Align Bench..." function and the throughput values will be documented following the same criteria as listed above.

30.10.2.2 The throughput of the GC/lightpipe will be documented weekly. The oven temperature will be set to 200º C, the lightpipe temperature will be set to 270º C, and the transfer line temperature will be set to 270º C. Both the maximum and minimum values of the interferogram, the location of the centerburst, the gain setting will be recorded in the log book, dated and initialed. If the sum (absolute value) of the maximum and minimum energy is less than 8.0 at a gain of 1.0, then perform maintenance as needed.

30.10.2.3 The noise level of the GC/lightpipe will be checked weekly. A spectrum will be taken while the GC/lightpipe is in the same configuration as the throughput check, taking 128 scans with the resolution set to 8 $cm^{-1}$ and the gain set to 1.0. Select the 2200-2100 $cm^{-1}$ range and measure the noise using

"Analyze" then "Noise". The Peak-to-Peak noise value should be below 0.10, if not, perform maintenance as needed. Record in logbook, date and initial.

30.10.3 Monthly

30.10.3.1 Perform maintenance on the computer and peripherals as needed. Record in log book, date and initial.

30.10.3.2 Archive data files to a second hard drive or to writeable CDROM. Retain for at least five years.

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