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5.1 Reagents

Alcoholic KOH

10% KOH in anhydrous methanol

Beam's Reagent

5g KOH diluted to 100 mL with ethanol

Citric Acid Buffer

7.8g anhydrous citric acid or 8.5g citric acid hydrate in 80 mL H2O.

14.2g Na2HPO4 in 200 mL H2O

Mix and dilute to 1000 mL H2O

pH approximately 5.3 (KEEP REFRIGERATED)

Cobalt Thiocyanate

2g Co(SCN)2 diluted to 100 mL H2O

OR

10g Co(SCN)2

490 mL H2O

500 mL glycerine

Dille-Koppanyi

#1 0.1g cobalt acetate

100 mL methanol

0.2 mL glacial acetic acid

#2 5 mL isopropylamine diluted to 100 mL with methanol

ADD 1:1 TO SAMPLE

m-dinitrobenzene

#1 0.2g m-dinitrobenzene diluted to 100 mL with isopropanol.

#2 2M methanolic KOH

ADD 1:1 TO SAMPLE

Dragendorf Spray

(a) 2g bismuth subnitrate

25 mL glacial acetic acid

100 mL H2O

(b) 40g potassium iodide

100 mL H2O

(c) 10 mL solution (a)

10 mL solution (b)

20 mL glacial acetic acid

100 mL H2O

Duquenois

1 g vanillin .

12 drops acetaldehyde

50 mL 95% ethanol

OR

9.4 g vanillin

5.9 mL acetaldehyde

473 mL (1 Pint) ethanol

Duquenois Spray

(a) .8g vanillin PREPARE FRESH

1 mL acetaldehyde

20 mL 95% ethanol

(b) HCl

Spray with (a) then (b).

Fast Blue 2B

50mg fast blue 2B

25 mL 95% ethanol

PREPARE FRESH.

Ferric Chloride

10g $FeCl_3$.

100 mL H_2O.

Fiegles

90 mL of a 1% solution of sodium nitroprusside

(nitroferricyanide)

10 mL acetaldehyde

MAKE FRESH EACH MONTH

FPN

5 mL 5% w/v $FeCl_3$ in H_2O.

45 mL 20% w/w perchloric acid in H_2O.

50 mL 50% w/w nitric acid in H_2O.

Froehde's

50 mg. molybdic acid or sodium molybdate

10 mL. conc. H_2SO_4

Furfural

(a) 1 mL furfural.

3 mL ethanol.

(b) 1 mL solution (a).

4 mL glacial acetic acid.

Gold Chloride

1g $HAuCl_4$

20 mL H_2O

Gold Chloride in 1g $HAuCl_4$.

Phosphoric Acid

7 mL phosphoric acid.

13 mL H_2O.

Gold Bromide

(a) 12 mL con. H_2SO_4

18 mL H_2O.

(b) 0.25 mL H_2O.

1.25 mL 48% HBr.

(c) 28.5 mL of solution (a).

1.5 mL of solution (b).

0.97 g gold chloride.

(d) Use 1 part solution (c) to 2 parts H_3PO_4.

0.1N HCl in Methanol

(for recrystallizing amphetamines and barbiturates): 0.83 mL con. HCl diluted to 100 mL with methanol.

Iodoplatinate Spray

0.25 g platinic chloride.

5 g potassium iodide.

Dilute to 100 mL H_2O.

Acidified Iodoplatinate

add 2 mL con. HCl to 100 mL iodoplatinate spray

Highly Acidified

Iodoplatinate:

12 mL of acidified iodoplatinate spray and 3 mL concentrated HCl

Liebermann's Reagent

5 g Sodium Nitrite.

50 mL con. H_2SO_4.

Mandelin's Reagent

1g ammonium vanadate.

100 mL conc. H_2SO_4

Marquis

4 mL con. H_2SO_4.

4 drops formaldehyde.

Mayers

0.68 g Mercuric chloride

2.5 g KI

100 mL H_2O

Mecke's Color Test

50 mg selenious acid dissolved in 10 mL con. H_2SO_4.

Mercuric Iodide

27% by volume con. HCl saturated with HgI_2.

Note: 27% by volume concentrated HCl is prepared by adding 27 mL of concentrated HCl to 73 mL of deionized water.

Mercurous Nitrate

30 mL H_2O saturated with $Hg(NO_3)_2$.

MAKE FRESH.

Mollisch

5% α-napthol in 95% ethanol.

Ninhydrin

0.05g ninhydrin.

25 mL 95% ethanol.

MUST BE LESS THAN 10 DAYS OLD.

Picric Acid

Crystal Test:

Saturated aqueous solution.

Platinum Chloride

5g platinic Chloride.

100 mL H2O.

Permanganate

15g potassium permanganate. 2g.

75 mL phosphoric acid. 5 drops.

dilute to 500 mL H2O. 100 mL

Silver Nitrate

5g AgNO3.

100 mL H2O.

Stannous Chloride

5g SnCl2.

10 mL conc HCl.

Dilute to 100 mL with H2O

50% H_2SO_4

125 mL con. H_2SO_4.

50 mL 95% ethanol.

75 mL H_2O.

TDTA

50mg di-p-toluoyl-d-tartaric acid (TDTA)

5 mL ethanol.

5 mL glycerin.

Dilute to 50 mL with H2O.

TLTA

50mg di-p-toluoyl-l-tartaric acid (TLTA)

5 mL ethanol.

5 mL glycerin.

dilute to 50 mL with H2O.

Van Urks

Color Test:

#1 0.4g p-dimethylaminobenzaldehyde.

10 mL con. acetic acid.

#2 0.34g Hg(NO3)2 in 10 mL con. HCl.

Strip Papers:

Soak filter paper strips in a solution of 0.8g of

p-dimethylaminobenzaldehyde in 100 mL 95% EtOH. Remove papers and allow to dry. Store in dark colored jar.

Van Urks Spray:

0.8g p-dimethylaminobenzaldehyde.

10 mL conc. H2SO4.

90 mL 95% EtOH.

5.39 Wagenaar's Reagent:

Add ethylenediamine to 5% CuSO4 until the solution becomes dark violet.

Wagner's Reagent

1.27g Iodine.

2.75g KI.

5 mL H2O.

Dilute to 100 mL with H2O.

Zwikker's Reagent

#1 0.5% CuSO4.

#2 5% pyridine in CHCl3.

5.2 Physical Identification

The US Food and Drug Administration requires pharmaceutical products in solid oral dosage forms to be marked with an imprint. This imprint, in conjunction with the product's size, shape and color, provides information about the drug product, active ingredients and manufacturer. Additionally, all pharmaceuticals have a unique National Drug Code (NDC) number which identifies the labeler, the product, and the package size and type.

The size, shape, color, and any physical markings on pharmaceutical products may give indications of the drug's identity. Label information on intact, factory-sealed pharmaceuticals may also give indications of the drug's identity.

5.2.1 Application to Casework

For the purposes of this section, pharmaceutical products include tablets, capsules, sublingual films, patches, injectable liquids, etc.

Physical identification of a pharmaceutical product may be used in the analysis of seized drugs as a selective test. This test cannot be used if markings are not present or are indistinct.

Physical identification of broken tablets may be performed if the fragments can be pieced back together to form an entire tablet and include all of the tablet markings.

5.2.2 Physical Identification Procedure

1. Record a detailed description of the appearance of the pharmaceutical preparation. Details such as color, shape, and imprints should be included. Product label information may be included when applicable.

   Information on the label of an intact, factory-sealed pharmaceutical preparation may be used as a physical identifier when recorded in the case record.

2. Compare the observed features of the pharmaceutical preparation to a literature reference. Suggested references include, but are not limited to: Drugs.com, Ident-a-Drug, NLM/NIH Pillbox, The Physicians Desk Reference, the Drug ID Bible, NDC List, and manufacturer's information.

5.2.3 Acceptance Criteria

A positive result occurs when the color, shape, and imprints of the pharmaceutical product match the information and photos in the literature reference. While color descriptions may be subjective, general agreement between the description and the reference is required. For example, peach/orange or turquoise/blue show sufficient agreement whereas peach/blue does not.

An inconclusive result occurs when any physical feature of the pharmaceutical product does not correspond to any literature reference or to the known or expected properties of a legitimate pharmaceutical product.

An inconclusive result may also occur if the results of instrumental analysis are not consistent with the drug content described in the literature reference.

5.2.4 Reporting

Reporting of Physical Identification of pharmaceutical products occurs in certain circumstances. Refer to CS-PM 2.4 for the entire description of seized drug analysis reporting guidelines.

5.2.4.1 Mimic Tablets

For tablets and capsules with markings consistent with a controlled substance but which have analytical results different from the manufacturer's specification as to content, use one of these examples:

"Observations of the physical characteristics and markings on the tablets are consistent with a pharmaceutical preparation containing hydrocodone. However, chemical analysis of 5 tablets did not show the presence of any controlled substance."

"Observations of the physical characteristics and markings on the tablets are consistent with a pharmaceutical preparation containing tramadol. However, chemical analysis of one tablet confirmed the presence of hydrocodone."

"Observations of the markings on the tablets are consistent with a pharmaceutical preparation containing alprazolam. However, physical characteristics of the tablets are not consistent with known pharmaceutical preparations of alprazolam. Chemical analysis of one tablet indicated the presence of zapizolam. Zapizolam is not controlled.

5.2.4.2 Drug or Preparation Specific Results

If consideration of non-controlled active ingredients or specific dosage form or specific combinations of ingredients is relevant, use the following example:

"Item 1 was found to contain butalbital, acetaminophen, and caffeine. Reference(s) indicated Item 1 is consistent with an exempt (non-controlled) preparation."

NOTE: this report statement requires chemical analysis of the pharmaceutical to demonstrate the presence of the non-controlled active ingredients. If no chemical analysis was performed, use a possible presence report statement, and clearly state that no confirmatory analysis was performed.

5.2.4.3 Possible Presence Reported with a Full Analytical Scheme

If a Physical Identification is performed and reported without a full analytical scheme, the report shall clearly state that full analysis (identification) was not performed. Use the following example:

"Physical identification of the tablets indicated acetaminophen, a non-controlled substance. No confirmatory tests were performed."

5.2.5 Documentation and Technical Review

The detailed physical description of the pharmaceutical product or label is in the case record.

A copy of the literature reference is in the case record or section object repository. The results of the physical ID (the drug or drugs in the pharmaceutical) will be indicated in the case record.

5.3 Color Tests

Color tests are preliminary tests which lead to a general classification of a drug or an exclusion from a particular drug category. Color tests are actually visible spectrophotometry, using the human eye as a detector. Absorption of light in the visible region of the electromagnetic spectrum by chromophoric groups formed during the reaction between a drug and chemical reagent can be observed as a color change.

5.3.1 Color Test Procedures

1. Place the color test reagent in a well of a spot plate.
2. Add a small amount of the test sample. It may be necessary to crush the material before adding it to the reagent.
3. A positive result is the observation of a specific color change after the addition of the sample.

Three commonly used reagents are the Marquis, Cobalt Thiocyanate, and Dille-Koppanyi.

5.3.2 Interpretation Criteria

A positive result occurs with a specific color change that is known to occur with a reference material of the same substance. A compilation of known color changes may be used for a reference of positive results.

A negative result occurs when no color change is observed.

An inconclusive result occurs when a color change is observed but is inconsistent with any known or expected color results.

5.3.3 Positive Test Results for Specific Substances

Substance	Marquis	Cobalt Thiocyanate	Dille-Koppanyi
Glutethimide			purple
Barbiturates			purple
Diphenylhydantoin			purple
Theophylline			purple
Amphetamine	orange		
Benzphetamine	orange	blue	
Mephentermine	orange	blue	
Mescaline	orange		
Methamphetamine	orange		
Phentermine	orange		
Amitriptyline	orange to brown	blue	
Tetracycline	orange to brown		
Chlortermine	orange (slow)	blue (fades)	
Pethidine	orange (slow)	blue	
Ethinamate	orange (slow)		
Doxepin	brown	blue	
Phenazocine	brown		
Ephedrine	yellow (slow)		
Diphenhydramine	yellow		
Mazindol	yellow	blue	
Dilaudid	yellow to purple	blue	
Ethyl morphine	yellow to purple		
Papaverine	yellow to purple		
STP	yellow to green		
Flurazepam	pink (slow)	blue	

Substance	Marquis	Cobalt Thiocyanate	Dille-Koppanyi
Perphenazine	pink (slow)	blue	
Salicylate	pink (slow)		
Chlorpromazine	red	blue	
Clindamycin	red	blue	
Pentazocine	red to olive		
MDA	olive to purple to black		
Codeine	purple	blue	
Heroin	purple (streaking)	blue	
Methapyrilene	purple	blue	
Methocarbamol	purple	blue	
Morphine	purple	blue	
Opium	purple (slow) to brown		
Propoxyphene	purple	blue	
Glyceryl Guaicolate	purple		
Butethamine		blue	
Cocaine		blue	
Dicyclomine		blue	
Diethylpropion		blue	
Methadone		blue	
Phencyclidine		blue	
Quinine		blue	
Methaqualone		blue	
Methylphenidate		blue	

5.3.4 Additional Color Tests

Samples	Reagents	Procedure	Result
Ascorbic Acid	$AgNO_3$	Dissolve 1 mg sample in 0.5 mL H_20; Add 3 drops $AgNO_3$	Gray; ppt
Diazepam	m-dinitrobenzene in MeOH-KOH	Add reagents to 1:1 to 1 mg sample	purple
Indoles (LSD) $Hg(NO_3)_2$	A. Van Urks B.Strip Test	Add 3 drops PDAB and 1 drop to 1 mg sample. Then add 1 drop conc. HCl crush 1 mg sample on PDAB paper; add 1 drop 1:5 HCl; heat	purple purple
Iron (Fe++)	$K_3Fe(CN)_6$	Dissolve 1 mg sample in 0.5 mL H_2O;add few crystals of $K_3Fe(CN)_6$ and 1 drop 1:5 HCl	blue green
Phenothiazines in 0.5 mL	FPN	Dissolve 1 mg sample in H_20; add 5 drops FPN reagent	pink
Secondary Amines	Fiegles	Add 3 drops Fiegles to 1 mg crushed sample, add 50 mg Na_2CO_3 to side of well and just touching the liquid	Blue, Red is neg.
Procaine & Benzocaine	Furfural	Add 3 drops furfural to 1 mg sample	red
Salicylate	10% $FeCl_3$	Dissolve 1 mg sample in 0.5 mL H_20; make basic then slightly acidic (pH 6); add 3 drops $FeCl_3$	purple
Strychnine	$K_2Cr_2O_7$	Dissolve 1 mg sample in 5 drops con. H_2SO_4 in spot plate; add 1 crystal $K_2Cr_2O_7$ and pull it through the acid	purple
Sugar	Mollisch	Dissolve 1 mg sample in 0.5 mL H_2O; add 1 drop Mollisch: agitate; slowly add 1 mL con. H_2SO_4	purple
Sulfur		Crush 1 mg sample on rough textured brown paper; add 2 drops con. HCl	Orange

5.4 Microcrystal Tests

When a suspected drug is reacted with an appropriate reagent (usually a heavy metal salt), a precipitate will form and the crystals of the precipitate have a characteristic shape which can be observed under a microscope. The relative shapes and sizes of the molecules and the number and the orientation of bonds in the molecules will affect crystal form. This allows a microcrystal test to be used as a Category B selective test in seized drug analysis.

Specific drug sections contain information on the how to conduct individual microcrystal procedures.

5.4.1 Quality Assurance

Microcrystalline test reagents follow the documentation and verification procedures found in CS-PM 3. All reagents must have been verified within the previous three months in order to be used for casework. Reagents which have not been verified shall not be used.

Observations of crystals will be recorded at the time they are made.

5.4.2 General Interpretation and Acceptance Criteria

The interpretation of microcrystal tests relies heavily on the experience the analyst has had with a particular test. If the analyst has no experience with a particular test, the unknown crystals must be compared with crystals produced from a known drug standard under the same conditions.

Impurities in the sample, concentration differences, and polymorphism can play a part in the appearance of deformed or irregular crystals.

A positive result occurs when crystals are formed that correspond to the known and expected crystals formed by a given drug with that reagent.

An inconclusive result occurs when indistinct or irregular crystals are formed.

A negative result occurs when no crystal formation is observed. Negative results may be useful for ruling out a particular class of drugs but have no independent value in an identification.

5.4.3 Documentation and Technical Reviews

Ensure the following are present in the case record for each microcrystal test performed:

1. The reagent identifier and date of last verification. Verification must have been performed in the three months prior to the testing of the item.
2. The item number and observations of each test of that item.

5.5 Extraction

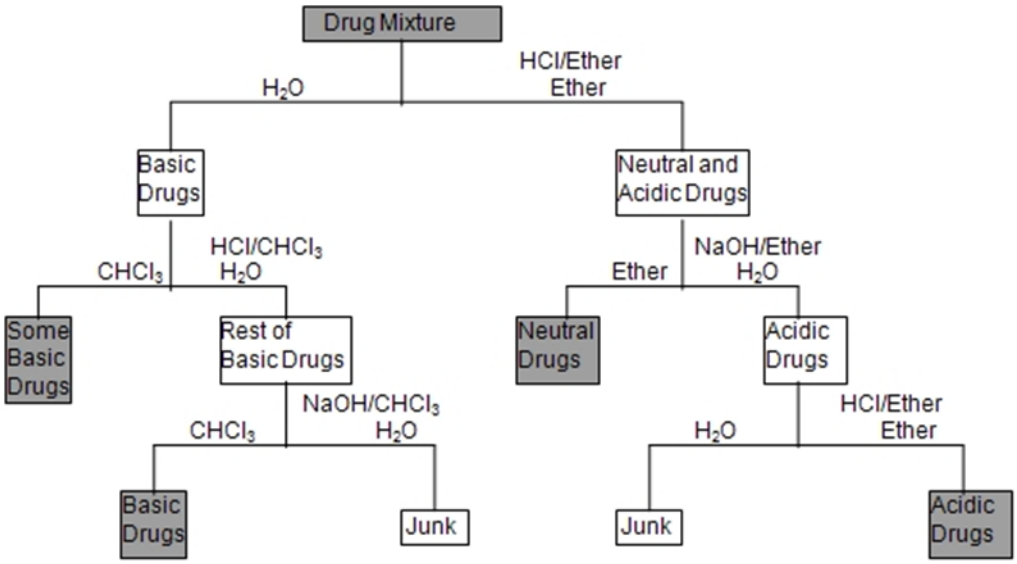
For infrared and mass spectral work, an extraction of a seized drug sample may be necessary to purify the controlled components.

Extraction methods include both dry extraction and solvent extraction.

In dry extractions the drug is simply dissolved from the dry drug mixture using a suitable solvent in which the excipients are insoluble.

In solvent extractions, two immiscible solvents are used to separate the components of a mixture depending on their solubilities in the solvents.

5.5.1 General Extraction Procedure for Acidic, Basic, and Neutral Drugs



Amphoteric drugs may be extracted from aqueous ammonia or carbonates using a suitable organic solvent.

The above extraction scheme is meant as a starting point for the analyst. Other acids, bases and organic solvents than those used in this scheme may yield better results in certain cases.

NOTE 1: A number of extraction procedures are included in this manual, but they are not meant to be all-inclusive, nor are they meant to be restrictive. The choice of extraction methods is dependent on the mixture of drugs and excipients present in a certain sample.

NOTE 2: It may be necessary to modify one of these procedures or to create a new one for a particular sample. Any extraction procedure used is validated or invalidated by the quality of the spectra of the extracted sample.

5.6 Gas Chromatography

5.6.1 Introduction

Gas chromatography (GC) is an instrumental separation technique. Components of a mixture are separated based upon the differences in the relative affinities of the analytes with a stationary phase on a solid support in the form of a chromatography column and a mobile carrier gas. A gas chromatograph is most often coupled with a flame ionization detector or mass spectrometer for the analysis of seized drugs.

5.6.2 Scope

This method describes the procedures for use and quality assurance of gas chromatographs in the Controlled Substances Unit.

This method includes qualitative test methods for direct comparison of retention times and for determination and comparison of relative retention times.

5.6.3 Referenced Documents

LOM 2.8

5.6.4 Terminology

Verification is confirmation that performance requirements are met by the instrumental technique.

Retention Time is the length of time from the time of injection until the time of maximum peak intensity.

Internal Standard is a compound purposely added to both samples and standards in order to provide a basis for qualitative or quantitative comparison.

5.6.5 Use and Application to Casework

Gas chromatography is regarded as a Category B test in the forensic analysis of seized drugs.

Operation of gas chromatographs shall be limited to authorized personnel. Other personnel may operate gas chromatographs under the direct supervision of authorized personnel. Refer to LOM 2.8.

5.6.6 Equipment and Reagents

Gas chromatographs with flame ionization detectors (FID) or with mass spectrometers (MS) are used.

Internal standards may be used with any GC method. If not specified in a method, tribenzylamine should be used. Caffeine may also be used; however, caffeine may not be used as an internal standard in casework that itself contains caffeine. Internal standards should be made to a concentration of approximately 1 mg/ mL.

5.6.7 Safety

The inlet(s) are heated zones and contact with inlet surfaces will cause burns. Avoid contact with GC inlets and use applicable tools or gloves to handle heated inlet components.

The oven vents at the back of the GC discharge hot air up to 450 °C (842 °F) during cooling. Avoid working behind the GC oven vents during cool down cycles.

Hydrogen is potentially explosive and must be used with extreme care. The use of hydrogen as a carrier gas must be reviewed in advance by the FSD Health and Safety Officer and installation of hydrogen systems must be performed to applicable codes and standards. The GC should be equipped with a hydrogen sensor prior to use of hydrogen carrier gas.

5.6.8 Sampling, Test Specimens, Test Units

Samples for GC analysis should be extracted or diluted with organic solvents. Injections should be free of solids, acids, and bases. Extraction, filtration and/or centrifugation may eliminate unwanted components.

Sample concentrations should be approximately the same as the concentration of the reference material solution. Visual inspection is sufficient for determining suitability of sample or reference concentration for qualitative methods.

5.6.9 Quality Assurance

Gas chromatographs shall be assigned a unique identification number. A label with the unique identification number shall be visible on the instrument. The specific instrument used to generate all case data will be clearly recorded in the case record.

The standard (either external or internal) that is analyzed with a case shall serve as a function verification of the instrument.

The chromatogram of the reference standard used for direct comparison shall be retained in the case record.

New procedures must be validated before use. New equipment, instruments, or methods that do not require validation must be verified prior to use. Validation and verification documentation will be stored electronically in Qualtrax. Laboratories may also retain a local copy. Historical validations and verifications may also exist in hard-copy form at the laboratory.

5.6.10 Maintenance

Maintenance of gas chromatographs shall be in compliance with all applicable FSD Maintained Instrument Maintenance Schedules and Policies.

Maintenance records shall be kept for all maintenance tasks appearing on the Instrument Maintenance Schedule.

5.6.11 Direct Comparison of Retention Time Procedure

Select an appropriate instrument method. Allow for full elution of analyte and internal standard peaks, ideally in the middle ¾ of the run time.

5.6.11.1 Injections

Use a split injection for most analyses. In general, a 67:1 split ratio is used.

a) It is generally appropriate to use a split ratio from 10:1 to 100:1.
b) Split ratios other than 67:1 shall be recorded in the case record.

NOTE 1: It is acceptable to document the split ratio used by including notation in the instrument method file name printed on the data. The split ratio recorded in the method file name will be formatted as the numerator of the ratio followed by the word "Split". Example: "…25split…" is understood to mean a 25:1 split ratio was used. For all other instances in the case record, the split ratio should be specified with both numerator and denominator, such as "25:1 split ratio."

NOTE 2: A split ratio specified in an analytical guide or procedure in this manual and used in the test satisfies this requirement and does not need to be documented again in the case record.

An injection volume of 0.1 uL to 1 uL is typically used. The volume injected shall be recorded in the case record.

Use an auto-sampler for sample introduction if practicable. Any manual injections shall be documented in the case record.

5.6.11.2 Blanks

Run a solvent blank prior to each case sample and reference material. Additional blanks may be added at the discretion of the analyst.

Investigate all significant peaks in solvent blank chromatograms and document the investigation in the case record.

a) If a drug, controlled substance, or related compound is detected in any concentration in a solvent blank, the blank and associated case sample shall be re-run.

b) If an interfering substance is present in a blank, such as a compound eluting at the retention time of an analyte in the case or standard, the blank and associated case sample shall be re-run.

c) If the peaks reappear in a blank that has been re-run, the blank should be replaced.

d) The instrumental data from the blank shall be kept in the case record with the associated case data.

5.6.11.3 Test Samples

Inject the test sample using the same instrument operating conditions as were used for the analysis of the solvent blank.

NOTE: This includes injection parameters and instrument method. This does not include method post-run conditions that occur after the temperature program has completed.

Compare the retention time of the case sample to the retention time of one or more reference materials. For comparison purposes, sample retention times shall be compared to the retention times of reference materials run within the same 24-hour period.

All comparisons between sample and reference material retention time shall be made only when the sample and reference material have been run under the same instrument method conditions and on the same instrument.

5.6.11.4 Derivatization

Derivatization may be necessary for some compounds that do not chromatograph well. Reference materials used for retention time comparison shall also have been derivatized by the same method.

5.6.12 Relative Retention Time (RRt or RRT) Procedure

5.6.12.1 Internal Standards

Prepare an internal standard solution of approximately 1 mg/ mL of tribenzylamine in methanol or other suitable solvent.

NOTE: Existing RRt methods that use caffeine as internal standard are acceptable until such time as the column phase is changed, the RRt of any analyte is re-measured, or caffeine is present or is of forensic significance in a given case.

5.6.12.2 Test and Reference Samples

Prepare a reference material sample and a case sample by using the internal standard solution as the final solvent into which the material is dissolved.

Prepare a sample of the internal standard solution for GC analysis as a solvent blank.

Select an appropriate instrument method.

Allow for full elution of analyte and internal standard peaks in the middle ¾ of the run time.

5.6.12.3 Injections

Use a split injection for most analyses. In general, a 67:1 split ratio is used.

a) It is generally appropriate to use a split ratio from 10:1 to 100:1.

b) Split ratios other than 67:1 shall be recorded in the case record.

NOTE 1: It is acceptable to document the split ratio used by including notation in the instrument method file name printed on the data. The split ratio recorded in the method file name will be formatted as the numerator of the ratio followed by the word "Split". Example: "…25split…" is understood to mean a 25:1 split ratio was used. In all other instances in the case record, the split ratio should be specified with both numerator and denominator, such as "25:1 split ratio."

NOTE 2: A split ratio specified in an analytical guide or procedure in this manual satisfies this requirement and does not need to be documented again in the case record.

Use an injection volume of 0.1 uL to 1 uL. The volume injected shall be recorded in the case record.

Use an auto-sampler for sample introduction if practicable. Manual injections shall be documented in the case record.

5.6.12.4 Blanks

Run a solvent blank prior to each case sample and each reference material. Additional blanks may be added at the discretion of the analyst.

Investigate all significant peaks in solvent blank chromatograms and document the investigation in the case record.

a) If a drug, controlled substance, or related compound is detected in any concentration in the solvent blank, the blank and associated case sample shall be re-run.

b) If an interfering substance is present, such as a compound eluting at the retention time of an analyte in the case or standard, the blank and associated case sample shall be re-run.

c) If the peaks reappear in a blank that has been re-run, the blank should be replaced, and case resampled prior to reanalysis.

Inject the test sample using the same instrument operating conditions as were used for the analysis of the solvent blank.

NOTE: This includes injection parameters and instrument method. This does not include method post-run conditions that occur after the temperature program has completed.

5.6.12.5 Calculate the sample relative retention time(s)

Compare the relative retention time of the case sample to the relative retention time of one or more reference materials.

All comparisons between sample and reference material relative retention time shall be made only when the sample and reference material have been run under the same instrument method conditions on the same instrument and without changing the phase of the capillary column between the analysis of reference material and that of the case sample.

NOTE: Reference material RRt are commonly compiled over a period of time for comparison with future case sample RRt.

5.6.12.6 RRt Compilations

Compilations of reference material RRt must be labeled with the instrument identifier and instrument method used, and the revision date.

The calculation of reference RRt shall be technically reviewed prior to addition to any list, repository, or compilation of RRt that is retained outside of the case record.

Documentation of technical review of reference material RRt shall be maintained and shall include the date of review and identity of the reviewer.

5.6.12.7 Derivatization

Derivatization may be necessary for some compounds that do not chromatograph well. Reference materials used for relative retention time comparison shall also have been derivatized by the same method.

5.6.13 Calculations

Retention time will be labeled to at least two decimal places. Calculations should be performed with all decimal places recorded.

NOTE: The software which labels peaks with retention time rounds the last digit retained according to standard rounding rules.

Relative retention time is calculated by dividing the retention time of the sample by the retention time of the internal standard. The result should be rounded to the appropriate number of significant figures following standard rules for rounding and significant figures.

Example: $RRt = \frac{Rt_{sample}}{Rt_{Internal\ Standard}}$

5.6.14 Quantitation

Gas chromatography may be used for quantitation. Quantitative procedures are analyte specific. Refer to the relevant procedure(s).

5.6.15 Acceptance Criteria and Interpretation of Results

Chromatographic peak(s) present in the unknown shall be reviewed for suitability for retention time comparison. A visual assessment is appropriate in most cases. Acceptance criteria for retention time comparisons are:

1) Peak shape is reasonably symmetrical and Gaussian.
2) Peak is fully resolved.
3) Peak is clearly distinguishable from baseline noise. If calculated, the minimum signal to noise ratio is 5.

The retention time or relative retention time of an unknown analyte is compared to that of a known reference material run under the same instrument method.

Retention times for an analyte and a reference material are in agreement if the retention time of the analyte is within +/- 0.030 minutes of the retention time of the reference material.

Relative retention time for an analyte and a reference material are in agreement if the relative retention time of the analyte is within +/-2% of the relative retention time of the reference material.

5.6.16 Reporting

No specific requirements.

5.6.17 Measurement Uncertainty

Measurement uncertainty is not typically expressed in numerical terms for qualitative tests such as gas chromatography.

Qualitative uncertainty, such as the inability to distinguish isomers, should be recorded and may be reported.

5.6.18 Documentation and Technical Review

5.6.18.1 Data

a) All chromatographic data shall be identified and correlated to the specific case, reference material, or task. Generally, the laboratory number and item number of the case, the unique reference material number, and/or the purpose of the data collection are sufficient.

b) Chromatographic data used to support a conclusion, result, or opinion shall be retained in the case record.

c) Retention times are labeled to show at least 2 decimal places.

d) The retention times compared are the result of tests performed with the same instrument method and on the same instrument.

e) Manual calculations of RRt shall be verified by the technical reviewer.

f) Chromatographic data that prompts a re-run or reanalysis of a sample will also be retained in the case record as rejected data. If data is rejected, the reason(s) must be recorded in the case record and each page of rejected data must be clearly marked as rejected data. Refer to the Casework Guidelines section of this manual.

5.6.18.2 Instrument and Method

a) The unique identifier of instrument used shall be recorded in the case record.

b) The instrumental parameters (injection parameters, column dimensions and phase, oven temperatures, ramps, and holds) shall be recorded in the case record or section object repository.

c) Split ratios shall be recorded if other than 67:1.

d) Splitless mode, if used, shall be recorded.

e) Manual injections shall be recorded.

5.6.19 References

Instrument Manuals

ASTM International, "Standard Practice for the Identification of Seized Drugs." ASTM E2329-17 (2017)

Scientific Working Group for the Analysis of Seized Drugs (SWGDRUG) Recommendations. Version 7.1, June 9, 2016.

5.7 Infrared Spectroscopy

Infrared spectrometry involves the measurement of the absorption of infrared energy by the molecule. Atoms in a molecule are in constant motion due to stretching and bending of chemical bonds. The region of the spectrum from 2.5 microns to 7 microns corresponds to the stretching of bonds. Many molecules have basically the same type of atoms bonded together, so stretching will cause little difference in spectrum appearance. The region from 7 to 14 microns and beyond corresponds to the bending of chemical bonds. Since small differences in atomic configurations cause drastic differences in the way atoms can be twisted relative to one another, this fingerprint region is generally unique for a molecule.

Infrared spectra can be obtained on samples in the gaseous, liquid, and solid forms, but most substances of interest to the drug analyst are solid at room temperature. Analyses of samples by use of potassium bromide pellets, ATR accessories, and infrared microscopes as described below are therefore the three most common techniques.

5.7.1 Controls

5.7.1.1 Instrument Background Checks

For FT-IR instruments background spectra should be acquired at sufficient frequency to assure high quality spectra. Indications that a background spectrum should be run are the appearance of carbon dioxide bands between 2400 and 2300 cm-1 and/or the broad, noisy water absorption bands located between 3800 and 2500 cm-1 and 1750 and 1500 cm-1.

5.7.1.2 Positive Controls

Positive controls are not typically used with this instrumentation. However, the function of the instrument must be periodically evaluated using an appropriate standard as specified in this manual.

5.7.1.3 Negative Controls

Negative controls generally are not used with this instrument.

5.7.2 Sample Preparation

Samples used for infrared spectrometry should be pure (typically 90% or better). Samples as submitted to the laboratory are frequently pure enough to be analyzed by FT-IR neat.

However, many samples require a purification process prior to analysis. This may be accomplished by a solvent extraction procedure as described in Section 4.6, "Extraction".

Often a single extraction of the sample from aqueous acid or base into chloroform or other suitable solvent will suffice to provide a pure sample for FT-IR analysis.

The solubility of a drug in a particular solvent frequently can be used to the analyst's advantage. Knowing the solubility of the drug and suspected impurities may be used to dissolve the drug and thus separate it from insoluble impurities when the appropriate solvent is used.

Solvent extraction options and other choices for sample purification, such as thin-layer chromatography, column chromatography, or sublimation may be used according to the training and experience of the analyst.

5.7.3 Infrared Spectroscopy Procedures

5.7.3.1 Potassium Bromide Pellets

The use of KBr pellets is a sampling method for infrared spectra of crystalline solids. Solids may also be examined as Nujol mulls or as deposited glassy films.

Crush and mix thoroughly approximately 2mg sample with 50mg anhydrous KBr. Press, preferably under vacuum, with approximately 8-10 tons pressure for about 1 min. Remove the pellet and place in the IR. Scan within the Mid-IR region of the electromagnetic radiation range. Compare spectrum with known reference spectra, noting peak maxima and relative peak intensities

Liquids are normally examined neat either smeared on a KBr disk or pressed between two KBr disks or plates.

5.7.3.2 ATR

The ATR apparatus is installed in the sample area of the FT-IR in approximately the same position as the pellet holder. It consists of a box containing mirrors and a crystal with an adjustable pressure plate on top.

With the ATR apparatus mounted, infrared beam from the instrument passes into the ATR apparatus and is reflected into the crystal. The beveled edge of the crystal determines the angle at which the beam enters it. After being reflected internally along the crystal and into the sample on top of the crystal the beam exits the crystal and is reflected out of the ATR apparatus toward the detector in the instrument.

The ATR accessory lends itself well to the analysis of liquids. Liquids are scanned after direct placement on the crystal surface and correct positioning of the anvil.

NOTE: The anvil and plate of the ATR apparatus must be thoroughly cleaned prior to each use.

5.7.3.3 Infrared Microscopy Procedure

Infrared microscopy allows high performance visual sample image discrimination while maintaining high infrared sensitivity in both reflectance and transmittance modes. Microscopic infrared spectroscopy is a fast, simple and nondestructive method of analysis. All samples can easily be recovered for preservation or further analysis. The microscope uses the infrared source and electronics from an FT-IR but has its own detector. Infrared reflectance is used for

data acquisition of opaque samples while transmittance is used for non-reflective samples as small as 10µm (micrometers).

The following is a general guideline for the use of the infrared microscope. Actual use of a particular microscope should be based on the manufacturer's instruction manual.

a) Fill detector with liquid N_2.
b) Initiate startup of spectrometer, microscope, and computer.
c) Run necessary initializing procedures.
d) Prepare sample by slicing, crushing, tearing or scraping and place on the microscope stage.
e) Looking through the microscope under visible light, pick the portion of the sample for which you desire a spectrum.
f) Obtain an infrared spectrum.

5.7.4 GC-IR

GC-IR provides an alternate approach to the identification of certain compounds. This technique is especially useful in discriminating positional isomers and some diastereomers. This instrument should be used when the analyst cannot distinguish isomers by using other techniques available in the laboratory.

5.7.4.1 General procedure for the Spectra Analysis DiscovIR GC IR

a) Liquid Nitrogen is added to the instrument MCT-A detector and large dewar prior to use
b) Method parameters shall be documented in the case record
c) Samples shall be dissolved in appropriate solvent and documented in the case record
d) An amount of sample (approximately 2 uL) is injected utilizing an autosampler
e) Solvent blanks shall be run prior to each sample in the same location where the sample is to be deposited on the disk
f) After the data is processed, the sample spectrum shall be compared to a known solid phase spectrum. For unknown compounds, a search of a library solid state transmission spectra may be conducted, however for an identification the unknown spectrum must be compared to a known standard spectrum taken under similar conditions.

5.8 Mass Spectrometry

Gas chromatography-Mass Spectrometry (GC-MS) is an instrumental separation and identification technique. Components of a mixture are separated by the gas chromatograph and detected by a mass spectrometer. The mass spectrometer ionizes the sample which subsequently fragments to produce charged ions. The charged ions are separated according to their mass to charge ratio (m/z). The m/z and the quantity of each charged ion is detected and a mass spectrum is produced. The retention times and the mass spectra from a sample may be compared to reference material for purposes of identifying the sample. GC-MS is a commonly used technique in the analysis of controlled substances due its sensitivity and selectivity, and its ability to analyze mixtures. Sensitivity is in the sub microgram range.

5.8.1 Scope

This procedure describes the methods for use and quality assurance of gas chromatograph-mass spectrometers in the Controlled Substances Unit

5.8.2 Use and Application to Casework

Mass spectrometry is regarded as a Category A test that can provide the highest level of selectivity through structural information. The selectivity of the test shall be considered by the analyst in the context of each examination as the ability to distinguish the analyte from similar compounds may be limited by the mode of operation, complexity of the sample, or concentration of the analyte.

Operation of gas chromatograph-mass spectrometers is limited to authorized personnel. Other personnel may operate GC-MS under the direct supervision of authorized personnel.

Specific test methods are found elsewhere in this manual; this guide should be used as a supplement to the specific test methods selected.

5.8.3 Equipment

Electron ionization (EI) mass spectrometer, coupled to a gas chromatograph.

5.8.4 Quality Assurance

The following quality assurance requirements are in place for all gas chromatograph-mass spectrometers in the Controlled Substances Unit.

5.8.4.1 Unique Identification Numbers

All GC-MS are assigned a unique identification number. This number shall be visible on the instrument and shall be referred to in records pertaining to the instrument.

5.8.4.2 Maintenance

Maintenance of gas chromatograph-mass spectrometers shall be in compliance with all applicable FSD Maintained Instrument Maintenance Schedules and Policies.

Maintenance records shall be kept for all maintenance tasks appearing on the Instrument Maintenance Schedule. Refer also to CS-PM 3.

5.8.4.3 Tuning

The tune of all mass spectrometers shall be checked and, if necessary, adjusted on a monthly basis. MS methods used in casework are based on a standard PFTBA tune. Refer to CS-PM 3 for the tuning process and acceptance criteria.

5.8.4.4 Function Verification

On any day the mass spectrometer is used, a function verification sample consisting of approximately 1 mg caffeine and 1 mg cocaine per ml shall be injected into the instrument and the spectra obtained. Refer to CS-PM 3.6 for acceptance criteria for function verifications.

5.8.4.5 GC-MS Data & Documentation

All GC-MS data generated in relation to a case are retained in the case record. Data should be marked “Not Used” or “Rejected” for clarity as applicable. If data are rejected, the reasons shall be recorded in the case record.

All GC-MS data shall be correlated to the specific task. Generally, the laboratory number and item number of the case or the reference material identifier is sufficient.

The instrument ID number and operating parameters must be recorded in the case record. An instrument method specified by procedure may simply be referred to by the method’s designated name without further elaboration.

5.8.5 Gas Chromatograph Operating Conditions

Oven temperature programs are typically used in the range of 80°C-280°C to elute samples. Oven ramp rates will vary, depending on the nature of the sample and on the GC column being used.

Isothermal conditions on the GC can be used if they achieve adequate peak shape and retention times.

Injector temperatures are nominally set between 225°C-250°C. Transfer lines are set at a similar temperature.

Analysts may store and use custom gas chromatographic methods based on sample characteristics and their own preferences and experience.

5.8.6 Mass Spectrometer Operating Conditions

Normal operating parameters are set by a manual tune or by the autotune function as described in CS-PM 3. The instrument is run in electron impact mode at or near 70 electron volts.

Scan ranges are generally set between m/z 40 and 550. The scan range may vary depending on sample molecular weight and the chromatographic method used.

Selected Ion Monitoring (SIM) may be used, but SIM mass spectra data have reduced discriminating power. SIM is useful to enhance sensitivity for chromatographic retention time comparisons.

5.8.7 Procedure

5.8.7.1 Prepare the sample.

Samples for GC/MS analysis should be extracted or diluted with organic solvents. Injections should be free of solids, acids, and bases. Extraction, filtration and/or centrifugation may eliminate unwanted components.

Transfer the extracted sample to an autosampler vial and affix a screw top or crimp cap. Vial inserts may be used for small sample volumes at the analyst's discretion. Prepare a second autosampler vial, containing only the solvent used, to serve as a solvent blank.

5.8.7.2 Select the instrument method.

Samples shall be injected by autosampler if practicable.

Sample volume should be 0.1 uL to 1 uL. The volume injected shall be recorded in the case record.

Instrument methods should be selected to allow for full elution of target analytes in the run time of the method.

Instrument methods typically use a split injection. It is appropriate to use a split ratio of 10:1 to 100:1. Refer to the section on Gas Chromatography in CS-PM 5 for split ratio documentation.

5.8.7.3 Inject and run the solvent blank, then the sample.

A solvent blank shall be run prior to each case sample. Additional blanks may be added at the discretion of the analyst.

The solvent blank shall be analyzed with the same instrument operating conditions as the case it precedes. This includes injection parameters and instrument method. This does not include method post-run conditions that occur after the temperature program has completed.

Significant peaks in solvent blank chromatograms shall be investigated and documented in the case record.

1. If a drug, controlled substance, or related compound is detected in any concentration, the blank and associated case sample shall be re-run.
2. If an interfering substance is present, such as a compound eluting at the retention time of an analyte in the case, the blank and associated case sample shall be re-run.
3. If the peaks reappear in a blank that has been re-run, the blank should be replaced.

5.8.8 Acceptance Criteria and Data Interpretation

Interpretation of chromatograms and GC retention time comparison procedures are found in the section on Gas Chromatography in CS-PM 5.

Comparison with known reference mass spectra is required. In order of preference, unknown mass spectra shall be compared with

1. Primary reference material, analyzed contemporaneously on the same instrument
2. Primary reference material, analyzed previously and spectrum retained in a Forensic Science Division laboratory user library
3. Primary reference material, analyzed by a laboratory accredited to ISO/IEC 17025.
4. Published reference spectra, including the following commercial Mass Spectral Libraries:
   a) NIST MS
   b) Cayman Mass Spectral Database
   c) Designer Drug
   d) SWGDRUG
   e) aafs

5.8.8.1 Acceptance criteria:

Identification is based upon a full scan mass spectrum.

The unknown spectrum must be compared to a known reference spectrum.

a) Ions across both spectra shall exhibit general visual correspondence of abundances and nominal m/z values
b) The spectra should have the same base peak.
c) The molecular ion (when observed) is present with corresponding isotope peaks.
d) Some compounds do not produce a molecular ion. In this case, comparison is made based on the fragment ions present.

The presence of extraneous ions in the unknown spectrum should be evaluated for significance, though background subtraction is strongly advised to minimize any background ions from appearing in the sample spectrum.

NOTE: Derivatization may be necessary for some compounds. Reference materials used for retention time or mass spectral comparisons shall also have been derivatized by the same method prior to comparison.

5.8.9 Reporting

No specific requirements.

5.8.10 Measurement Uncertainty

Measurement uncertainty is not typically expressed in numerical terms for qualitative tests such as mass spectrometry.

Qualitative uncertainty, such as the inability to distinguish isomers, should be recorded and may be reported.

5.8.11 Documentation and Technical Reviews

Ensure the following are documented in the case record:

1. The instrument used is recorded.
2. The instrumental parameters (injection parameters, column dimensions and phase, oven temperatures, ramps, and holds) are recorded in the case record or defined by the method given in the Controlled Substances Procedure Manual for that analyte.
3. Instrument processed data is present in the case record, including:
   a) Total Ion Chromatogram of sample and corresponding blank
   b) TIC showing each peak of interest with mass spectrum and library match to the spectrum presented.
   c) Any rejected data or unused data.

Ensure that interpretation criteria are met for identification by this method.

5.9 Rx-4 Automated Tablet and Capsule Counter

This procedure describes the use of the Rx Count Corp. Rx-4 Automatic Tablet Counter to obtain a count of tablets or capsules for evidence inventory purposes.

5.9.1 Function Verification

On any day the RX-4 is used for counting tablets or capsules for evidence inventory, a function verification test shall be performed.

Function verification material should be obtained by purchasing a commercially prepared bottle of at least 50 capsules or coated tablets. The function verification material shall be given a unique standard number. The number of dosage units in the bottle shall be determined by hand counting, and this number, along with the gross weight of the bottle and contents, shall be recorded on the standard documentation log form. The known number of dosage units will be the function verification standard for the Rx-4.

The Rx-4 will be function verified by loading and counting the number of tablets known to be in the function verification standard. Any discrepancy between the count supplied by the Rx-4 and the known number shall be resolved prior to use of the Rx-4 for evidence inventory purposes.

The function verification of the Rx-4 should be documented in the case record and should include the unique standard number and the count of the function verification as counted by the Rx-4.

The agreement between the count supplied by the Rx-4 and the known number of dosage units in the function verification standard will demonstrate that the device is functioning properly, and a count of dosage units for evidence inventory purposes can then be made.

5.9.2 Obtaining a Count for Evidence Inventory

<u>SAMPLE</u>

Remove the appropriate sample for testing from the population. Typically, this is one intact tablet or capsule.

Record the number of units sampled and/or withheld from automated counting. Since these withheld tablets or capsules are removed from the rest of the population before using the Rx-4, the number of withheld units must be added to the count produced by the Rx-4 tablet counter to determine the total population.

<u>CLEAN</u>

Clean the Rx-4 Tablet Counter. Allow any excess cleaning solvent to dry before loading tablets or capsules onto the turntable.

COUNT

Use the Rx-4 to count the remaining tablets or capsules for evidence inventory:

a) Place device on a level surface and turn the machine on.
b) Insert drawer.
c) Pour tablets on to the turntable.
d) Adjust separator tab as needed. Tab can be adjusted upwards for large tablets and down for small or flat tablets.
e) Select the Inventory Mode (INV) on the keypad.
f) Press “START”.
g) The number of tablets counted is displayed in the numeric display next to the keypad.
h) Record the count.
i) Press “STOP”.

REPEAT

Repeat the inventory count of the tablets to total three independent counts.

If the result of all three counts is the same, add the number of tablets removed for analysis to one Rx-4 count to calculate the total number of tablets.

If the result of one of the three counts done by the Rx-4 is different, the tablets must either be counted by hand, or three additional inventory counts should be made following the procedure above. For either method, the number of tablets removed for analysis shall be added to the inventory count in order to calculate the total number of tablets.

RECORD

Record the number of tablets in the case record.

If count was made by the automated counter, record the following information in the case record:

a) the unique identifier of the automated counting device
b) the unique function verification standard number and the function verification count
c) the number of evidentiary tablets withheld from counting
d) the inventory count of evidentiary tablets supplied to the counter
e) the sum total of evidentiary tablets (withheld + counted)

CLEAN

Clean the Rx-4 Tablet Counter upon completion of counting.

5.9.3 General Rx-4 Operating Instructions

Place device on a level surface and turn the machine on.

Insert drawer.

Pour tablets on to the turntable.

Adjust separator tab as needed. Tab can be adjusted upwards for large tablets and down for small or flat tablets.

Select the mode on the keypad.

a) Inventory Mode (INV) is used to count an entire amount of tablets. This mode should be used for most routine counting tasks.

b) Single Mode (SGL) is used to count a pre-determined number of tablets, dispensed into the top drawer. When the top drawer is removed, excess tablets on the turntable will drop into the bottom drawer. This mode is not routinely used for inventory.

c) Multiple Mode (MULTI) is used for counting multiple quantities from the same batch, dispensed into the top drawer. When the top drawer is removed, excess tablets will remain on the turntable. Once the drawer is reinserted, press start, and an additional count will be performed. To clear the turntable after the last count, leave the top drawer out and press CLEAR / INVENTORY / START. This mode is not routinely used for inventory.

Press “START”.

Record the count.

5.9.4 Cleaning and Maintenance

After each use, the Rx-4 Automatic Tablet Counter should be cleaned.

a) Turn off the power switch.
b) Tilt back the automatic gate assembly. Remove any residue from the bottom of the gate.
c) Lift the small sorting wheel from the center of the turntable.
d) Lift the main turntable disc from the device.
e) Clean the discs and plastic parts of the device with an isopropyl alcohol wipe, or a lint-free cloth. Clean all surfaces (top, bottom, edges).
f) Clean the drawers in the same manner.
g) Locate the sensor area in the square hole, and wipe both sets of sensors with an isopropyl alcohol wipe in a horizontal direction. Ensure that there are no other items such as fibers or hairs obstructing the area between the sensors.
h) Reassemble the unit.

5.9.5 Operating Tips

Count may be inaccurate if there is a momentary power failure. Count should be repeated if the power to the device is interrupted for any reason.

The device will count faster if it is loaded lightly with tablets. Do not overload or stack tablets on turntable.

Do not let the drawer overfill as it may partially block the sensor. Use the funnel drawer for bulk or direct-to-vial counts.

Adjust the separator tab according to tablet size: down for small or flat tablets, up for capsules or larger tablets.

Do not use the power switch to stop the turntable. Use the red “STOP” key on the keypad.

After turning the device off, allow a 20-second reset time before turning on again. The device may be left on all day if necessary.

Always ensure the collection drawers are in place when the machine is operating.

Maintain adequate clearance for the cooling fan.

Do not allow liquids to enter the machine.

5.10 Ultraviolet Spectrophotometry

Many organic compounds absorb ultraviolet energy, and the wavelength of this absorbed energy can be accurately measured. The sample is dissolved in a non-absorbing solvent and put into a quartz cell in a UV spectrophotometer. A beam of UV light passes through the sample and the wavelengths of light which are absorbed are recorded as peaks on a chart. The wavelength is measured in nanometers (10-9 m). The UV region is about 200nm wide (400-200 nm).

5.10.1 Controls

A blank is not required when using this instrumentation.

However, a solvent background must be run on single beam instruments prior to sample analysis. The background should be run using the same fused quartz cell that is used for the sample.

For a double beam instrument, a matched pair of fused quartz cells should be used, and the sample solution should be referenced against the solvent.

Negative controls are not typically used with this instrumentation.

Positive controls are not generally used with this instrumentation.

5.11 Thermo Scientific TruNarc Analyzer

5.11.1 Scope

This procedure describes the use of a Thermo Scientific TruNarc Analyzer hand-held Raman device to test exhibits submitted for controlled substances analysis in order to provide preliminary results in the form of a preliminary report.

5.11.2 Terminology

TruNarc denotes the Thermo Scientific TruNarc Analyzer, a hand-held Raman device used for the testing of seized drug exhibits.

5.11.3 Summary of Test Method

A seized-drug exhibit is presented to the TruNarc. The exhibit is bombarded with energy from a laser causing the molecular bonds within the exhibit to scatter the light from the laser. The scattered light with wavelengths different than that of the incident light is collected by the TruNarc and is then separated by wavelength by a dispersive spectrometer within the device.

A detector then measures the intensity of light at each wavelength and converts it to a spectrum. The spectrum is characteristic of a chemical compound. The spectrum from the exhibit is searched against entries in the TruNarc library.

The test is non-destructive, and spectra and library matches are printed and retained in the case record. Positive results for the following substances are reported in a preliminary report format for pretrial purposes:

- Amphetamine
- Cocaine
- Diazepam
- Heroin
- Methamphetamine
- MDMA
- Morphine
- Oxycodone
- Tramadol
- Gabapentin
- Methylphenidate

5.11.4 Application to Casework

Items suitable for TruNarc testing include light-colored powders, tablets and liquids. All suitable items should be considered for TruNarc testing upon receipt in order to provide preliminary investigative information to the agency.

Items generally not suitable for TruNarc testing include dark-colored materials, explosives, and plant materials

For weight-based penalty threshold cases, a full analysis is required per CS-PM 2.2, regardless of TruNarc results. TruNarc results may be used to guide decision making in the full analysis, but TruNarc results will not be reported per this procedure. Written authorization from the Prosecutor is required for TruNarc testing and exception to the full-analysis policy.

5.11.5 Interferences

Some samples fluoresce when illuminated with the laser. The fluorescence may interfere with the collection of the scattered light by the TruNarc and may result in an inconclusive result.

5.11.6 Equipment

Thermo Scientific TruNarc Analyzer with 250-350 mW 785 nm laser

Each TruNarc device will be uniquely identified by the manufacturer's serial number.

NOTE: Turn the device on by pressing the bottom button, then enter the four-digit laser code using the three buttons.

Type-H Solution Kit, optional

5.11.7 Sampling

Sample selection is performed with this test. The operator will select the areas of the exhibit to be scanned.

If testing a residual amount of material, shaking the exhibit to collect the material in a corner of the packaging can increase the surface area and the depth of material available for presentation to the TruNarc.

An exhibit may be tested multiple times. Also, several samples of an exhibit may be selected for testing. All scan data will be retained in the case record.

For visually inhomogeneous materials, the case record should describe what was tested in each sampling. For example: Scans 100-101 - brown powder, Scans 103-104 - white crystal.

5.11.8 Quality Assurance

5.11.8.1 Function Check

The self-check standard and self-check feature of the TruNarc are used to demonstrate the proper function of the device. The self-check shall be performed before and after the series of scans performed on any one exhibit.

Ensure that the self-check standard is closed on the nose cone of the TruNarc before performing the self-test. Press the middle button, labeled “Check.” Wait until the result is displayed.

The self-check function will produce a pass or fail result and display the result on the screen. The TruNarc is functioning properly if the self-check before and after the test produces a pass result. If the self-check does not pass, reposition the self-check standard and repeat the self-check.

If the self-check again does not pass, the device shall be placed out of service. The device may return to service following evaluation or repair, and a subsequent successful self-check.

5.11.8.2 Calibration

Calibration is not applicable to this procedure.

5.11.8.3 Conditioning

The TruNarc is powered by an internal, non-removable rechargeable battery. The battery is charged by connecting the AC charging cable to the device and plugging the opposite end of the cable into a 120V power outlet. The device may also be charged using the supplied USB cord connected to a computer. The battery provides approximately eight to ten hours of power and should be charged on an as-needed basis.

If necessary, use the TruNarc Admin software to set the date, time, and time zone before first use.

5.11.9 Test Procedure

a) Turn on the TruNarc Analyzer and activate the laser.
b) Perform self-check.
c) Press the nose of the TruNarc Analyzer to the specimen. Scan the specimen by pressing the top button, labeled “Scan,” twice. The scan progression is shown on the screen, followed by the processing progression. The scan will take between twenty seconds to two minutes.
d) Record the Scan Number in the case record, either in the worksheet or batch worksheet. If testing more than one exhibit as part of a batch of tests, record the unique identifier of the exhibit with the associated scan numbers. See Appendix 4.11-A for an example batch worksheet.
e) Repeat the scan of the exhibit and record the scan number in the case record.

f) Repeat the self-check.
g) The TruNarc analyzer records and stores all scan and self-check results.

5.11.10 Optional Type H-kit procedure

Some narcotics may emit high levels of fluorescent light when stimulated by the TruNarc laser, which obscures the spectral data. If a Raman spectrum is desired for these substances, the Type H-kit must be used to reduce the fluorescence of these substances.

a) To perform the Type H-kit procedure, open a vial and test stick package. Remove the test stick and hold it by the end.
b) Fill the scoop of the test stick with sample material.
c) Place the filled scoop into the vial of ethanol. Ensure that the end of the stick is immersed in the liquid and stir gently.
d) Remove the test stick from the vial. Any narcotic in the sample material has been adsorbed onto the metal wafer in the scoop.
e) Let the stick air dry for about 2 minutes without shaking the stick.
f) Ensure the TruNarc analyzer is ready to scan and self-check has been performed.
g) Press the scoop end of the test stick to the center of the nose cone. Press Scan to begin the scan.
h) Record the Scan number in the case record.
i) Repeat the scan of the exhibit and record the scan number in the case record.
j) Repeat the self-check.

5.11.11 Marking of the Items Tested

If two or more sub-items are present within one container, the specific sub-item(s) tested should be identifiable.

When possible, the sub-item scanned may be identified by physical description noted in the case record. For example, simply noting “the larger bag was tested” or “the blue tablet was tested” may provide sufficient information to identify which sub-item was scanned. This information should be recorded in the worksheet and/or batch worksheet.

Alternately, the scanned sub-item should be separated from similar untested items in the same container, such as by applying a heat-seal to isolate the tested sub-item.

When necessary, the container should be opened, and the scanned sub-item should be marked or otherwise identified.

5.11.12 Procedure to Upload Results to TruNarc Admin software

a) Open the TruNarc Admin software and turn on the TruNarc analyzer.
b) Connect the TruNarc to the computer with the USB cable.
c) At the prompt, click Yes to synchronize the data.
d) To view and print reports for an exhibit, locate the scan number or date of the scan.
e) Enter the Case ID information such as lab number and description if applicable.
f) Select the data to print, click the Report button.

g) To print the report, click the print button at the bottom of the report preview window.
h) To save reports to a PDF, click the save button at the bottom of the report preview window.

5.11.13 Interpretation of Results

The scan of an exhibit will result in one of the following Scan Result Types:

Scan Result	TruNarc Analyzer Identified…
Alarm	One or more substances of interest
Clear	A cutting agent and no substances of interest
Warning	A precursor or chemical used in manufacture of substances of interest Acetaminophen or aspirin that may be combined with a small amount of narcotic
Inconclusive	No identification of cutting agents or substances of interest

5.11.14 Alarm Result

If the Scan results of the two scans of an exhibit are in agreement as to the presence of cocaine HCl, cocaine base, cocaine, methamphetamine, diazepam, amphetamine, tramadol, morphine, MDMA (i.e. 3,4-MDMA), oxycodone, gabapentin, methylphenidate, or heroin, the analyte may be reported per this procedure.

If the two Scan results are not in agreement, or the analyte detected is not cocaine HCl, cocaine base, cocaine, methamphetamine, diazepam, amphetamine, tramadol, morphine, MDMA, oxycodone, gabapentin, methylphenidate or heroin, the exhibit shall be subjected to regular evidence processing procedures and/or the full analytical scheme for controlled substances.

5.11.14.1 Clear, Warning, or Inconclusive Result

If the scan results are “Clear,” “Warning,” or “Inconclusive,” the exhibit shall be subjected to regular evidence processing procedures and/or the full analytical scheme for controlled substances.

No interpretation of the spectra is necessary.

5.11.14.2 Limitations

The ability of the TruNarc to produce a result for a specimen is limited by the amount of analyte present, the distribution of the analyte in a specimen, the fluorescence of a specimen or analyte, and the availability of library spectra.

Inconsistent distribution of the analyte in the specimen may be mitigated by shaking the specimen and repeating the test procedure.

Cocaine and benzoylecgonine, a cocaine-breakdown product, produce spectra that are very similar. Benzoylecgonine is not in the TruNarc library and may produce a cocaine result. While benzoylecgonine is rarely encountered in seized drug evidence, this limitation will be reported.

“Clear,” “Warning,” or “Inconclusive” Scan results are affected by the limitations above. Consequently, exhibits producing “Inconclusive,” “Clear,” or “Warning” results must be subjected to the full analytical scheme for confirmation and should not be reported.

5.11.15 Reporting

The Results Table of the report should state the following

Weight:	"Not Measured"
Substance Identified:	"TruNarc Analyzer result. See Additional Information."
Schedule:	leave blank or "N/A"

5.11.15.1 Positive Results

The results shall be described in the Additional Information section with the following language. Modification of the item number, drug name, and/or schedule is permitted for accuracy or clarity.

> “Preliminary testing of Item 1 using TruNarc Analyzer indicated the presence of methamphetamine, a Schedule 2 controlled substance. This result is a preliminary indication as to the content of this item. A full analysis will be performed for this item upon request for trial. Contact this laboratory for additional information.”

Report the qualitative limitation for a result of cocaine, cocaine HCl, or cocaine base with the following language. Modification of the item number and/or drug name is permitted for accuracy or clarity.

> “Preliminary testing of Item 1 using TruNarc Analyzer indicated the presence of cocaine, a Schedule 2 controlled substance. Benzoylecgonine, a cocaine breakdown product, also will produce a cocaine result with this test. This result is a preliminary indication as to the content of this item. A full analysis will be performed for this item upon request for trial. Contact this laboratory for additional information.”

“Clear,” “Warning” or ”Inconclusive” TruNarc results should not be reported due to the limitations described above.

5.11.15.2 Additional Items

If additional Items are present in the submission, but not reported in the TruNarc report, use the following when applicable. Statements may be combined for accuracy or clarity:

> "Item 2 was tested using TruNarc Analyzer but did not indicate any reportable information."
>
> "Item 2 has been retained for full analysis and will be the subject of a supplemental report."
>
> "Item 2 will be analyzed separately and will be the subject of a supplemental report."
>
> "Item 2 may be resubmitted if full analysis is required."
>
> "Item 2 was not analyzed and may be resubmitted if additional analyses are requested."

In general, positive TruNarc results and full-analysis results should not be reported on the same lab report, whether the results pertain to the same or to different items. Positive TruNarc results should be reported within 36 hours of submission.

NOTE: It is appropriate to reference the previous testing and reporting of an item when the item is listed in the evidence description of a subsequent report. Example: Item 1 was previously analyzed and is the subject of Record 1.

5.11.16 Measurement Uncertainty

Qualitative results are generally not amenable to a numeric quantification of uncertainty.

5.11.17 Documentation & Technical Review

The following are required to be documented in the case record for all evidentiary scans performed:

- Serial number (unique identifier) of the TruNarc used
- Scan number, in the worksheet or batch worksheet
- Scan result (Clear, warning, alarm, or inconclusive)
- Spectrum and library spectrum

The spectrum page shall reference the sample scan number, date and time of the sample scan, the serial number of the device, and the Self-check dates and results.

The spectrum page should list the Case ID or unique laboratory identifier of the exhibit.

5.11.17.1 Report Requirements

The following are required in the laboratory report if the TruNarc result is reported.

- TruNarc Analyzer result

- Name of substance detected
- Report statements for weight, preliminary nature, and additional testing for trial
- Disposition of other items not reported

5.11.18 Safety

The Class 3B laser in the TruNarc can damage the eyes under direct or specular reflection exposure within the hazard zone of the laser. The hazard zone for the TruNarc extends 47.2" from the laser aperture along the path of the laser beam. Diffuse reflection is generally not harmful with Class 3B lasers.

5.11.18.1 Administrative Controls

- Never point the TruNarc at yourself or others.
- Never start the laser unless a sample fully covers the laser aperture.
- Always ensure appropriate deactivation of the laser by terminating a measurement prior to removing the sample from the laser aperture.
- When analyzing liquids through glass or clear containers ensure that the hazard zone of 47.2" is clear of reflective surfaces and other persons.

If the above administrative controls cannot be implemented, contact the FSD Health and Safety Officer for selection of appropriate safety eyewear.

The laser in the TruNarc has sufficient energy to ignite explosives and heat-sensitive materials. Do not test explosives or darkly colored materials with this device.

5.11.19 References

Appendix – 5-A TruNarc Analyzer Batch Worksheet

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5.11.20 Appendix

CS-PM 5-A TruNarc Batch Worksheet