Chapter 9: Technical Procedure for Extractions

1. Purpose / Scope - This procedure provides direction for the extraction techniques used in the Drug Chemistry Unit of the Wake County Bureau of Forensic Services.

2. Definitions

2.1. Quality control check - Periodic confirmation of the reliability of equipment, instrumentation, and/or reagents.

2.2. Reference Material - Material sufficiently homogeneous and stable with reference to specified properties, which has been established to be fit for its intended use in measurement or in examination of nominal properties

3. Materials and Reagents

3.1. Materials

3.1.1. Heat source
3.1.2. Beakers, vials, test tubes
3.1.3. Vortex mixer
3.1.4. Filter Paper
3.1.5. Funnel
3.1.6. Glass stirring rod
3.1.7. Graduated cylinder
3.1.8. Mortar and pestle
3.1.9. Pipettes with bulb
3.1.10. Bottles
3.1.11. pH Test paper
3.1.12. Litmus paper
3.1.13. Separatory funnel
3.1.14. Spatula
3.1.15. Water (Deionized)

3.2. Commercial Reagents

3.2.1. Acids, ACS grade

3.2.1.1. Hydrochloric acid
3.2.1.2. Sulfuric acid
3.2.1.3. Glacial acetic acid

3.2.2. Organic solvents - ACS, Optima or GC Resolv grade

3.2.2.1. Methanol
3.2.2.2. Chloroform
3.2.2.3. Acetone
3.2.2.4. Hexanes
3.2.2.5. Diethyl ether
3.2.2.6. Petroleum ether
3.2.2.7. Ethyl Acetate
3.2.2.8. Isopropanol
3.2.2.9. n-Heptane
3.2.2.10. Ethanol
3.2.2.11. Methylene chloride
3.2.2.12. Cyclohexane

3.2.3. Bases, ACS grade

3.2.3.1. Sodium hydroxide pellets
3.2.3.2. Sodium bicarbonate
3.2.3.3. Ammonium hydroxide

3.2.4. Drying agents, ACS or certified grade

3.2.4.1. Sodium sulfate, anhydrous
3.2.4.2. Magnesium sulfate, anhydrous

3.3. Prepared Reagents - Reagents may be prepared in any amount provided that the component ratios are kept constant. Reagents shall be labeled and stored according to the Drug Chemistry Unit Technical Procedure for Quality Assurance and Health and Safety Manual.

3.3.1. Dilutions or preparations of acids and bases may be prepared in the molarity or normality desired. Use the molarity or normality and the identity of the acid or base in the lot number. Label the container clearly with the identity of the acid or base and the molarity or normality.

Example: 5 ml of concentrated HCl is added to 95 ml of water

The initial normality of the HCl is 12N

The final normality is determined: (5 ml)(12 N) / (100 ml) = 0.6 N HCl

The lot number is 20120101HCl0.6NXXX

The container is clearly labeled: 0.6 N HCl

Example: 5 grams of sodium hydroxide pellets are dissolved in 100 ml of water

The formula weight of sodium hydroxide is 39.997

The normality (molarity) is determined:

(5 g / 0.1 L) (1 mol / 39.997 g) = 1.25 N NaOH

The lot number is 20120101NaOH1.25NXXX

The container is clearly labeled: 1.25 N NaOH

3.3.2. Storage: Closed container.

3.3.3. Expiration: Stock container: Three years
Use container: One year

3.3.4. Lot number: Eight-digit format year/month/day/identity/concentration/initials of preparer.

3.3.5. PQCC: Acceptable result: acidic or basic to litmus or pH paper

3.4. Ammoniated Organic Solvents

3.4.1. Preparation

3.4.1.1. Shake 10 milliliters ammonium hydroxide with 100 milliliters of organic solvent, e.g., hexane, chloroform or a prepared solvent mixture, e.g., 4:1 chloroform:hexane.

3.4.1.2. Allow layers to separate and draw off organic solvent during use.

3.4.2. Storage: Closed glass container.

3.4.3. Expiration: Stock container: One month
Use container: One month

3.4.4. Lot number: Eight-digit format year/month/day/Amm/solvent identification (ratio) /initials of preparer.
Example: 20120101AmmHex1:10XXX

3.4.5. PQCC: Acceptable result: basic to litmus or pH paper

3.5. Concentrated Sodium Hydroxide

3.5.1. Preparation

3.5.1.1. Add desired amount of water to a beaker or glass container.

3.5.1.2. Add Sodium Hydroxide pellets with stirring until solution is saturated (i.e. no more pellets will dissolve).

3.5.2. Storage: Closed glass container

3.5.3. Expiration: One year

3.5.4. Lot number: Eight digit format year/month/day/SatNaOH/initials of preparer.
Example: 20210514SatNaOHXXX

3.5.5. PQCC: Acceptable result: basic to litmus or pH paper

3.6. Acidified Organic Solvents

3.6.1. Preparation

3.6.1.1. Shake 10 milliliters concentrated hydrochloric acid with 100 milliliters of organic solvent, e.g., diethyl ether, chloroform or a prepared solvent mixture, e.g., 4:1 diethyl ether:hexane.

3.6.1.2. Allow layers to separate and draw off organic solvent for use.

3.6.2. Storage: closed glass container.

3.6.3. Expiration: Stock container: One month
Use container: One month

3.6.4. Lot number: Eight-digit format year/month/day/ Acidic/solvent identification (ratio) /initials of preparer.
Example: 20120101AcidicEther1:10XXX

3.6.5. PQCC: Acceptable result: acidic to litmus or pH paper

3.7. Organic solvent mixture

3.7.1. Preparation

3.7.1.1. Mix desired solvents in ratio desired. Mix prior to each use.

3.7.2. Storage: Closed glass container.

3.7.3. Expiration: Stock container: Three years
Use container: One year

3.7.4. Lot number: Eight-digit format year/month/day/solvent identification and ratio/initials of preparer.

Example: 20120101CHCl3IPA3:1XXX

3.7.5. PQCC: Observe for complete mixing.
Acceptable result: Only one layer is present.

3.8. Procedure

3.8.1. Samples may be extracted to isolate the compound of interest.

3.8.2. Consider the chemical properties, e.g., solubility, partition coefficient and dissociation constant, of the sample and the medium, e.g., pH, and determine the extraction technique. Consider the stability and volatility, especially when applying heat and/or strong acids or bases. Typically basic and acidic drugs are extracted at a pH 2 to 3 units above and below, respectively, the pK_a values of the drugs.

3.8.2.1. Extraction of Organic Acids and Bases

3.8.2.1.1. Dissolve the sample in an aqueous dilute acid solution and verify acidity with litmus or pH paper.

3.8.2.1.2. Wash the aqueous solution with an organic solvent chosen based upon the solubilities of the sample and medium to aid in the removal of any unwanted compounds in the medium. Repeat the wash if necessary.

3.8.2.1.2.1. If an organic base is the compound(s) of interest, discard the solvent washing.

3.8.2.1.2.2. If acid drugs are of interest or if the compound(s) of interest is unknown, retain the solvent washings and evaporate for further analysis.

3.8.2.1.3. Add a basic reagent to the aqueous solution and verify basicity with litmus or pH paper.

3.8.2.1.4. Add a suitable organic solvent to extract the basic compound(s) of interest.

3.8.2.1.5. Remove organic solvent from the aqueous solution for the intended analysis.

3.8.2.1.6. If the organic base being extracted is not volatile, evaporate the solvent in the fume hood, apply heat if desired and the compound of interest is compatible.

3.8.2.1.6.1. If excess moisture is a concern, dry the organic solvent over a drying agent, e.g., sodium or magnesium sulfate.

3.8.2.1.6.2. If the compound of interest is volatile, or if the salt form of the organic base is desired, add an acidified organic solvent dropwise until precipitation is complete, prior to evaporation.

3.8.2.1.6.3. Add a few drops of organic solvent if recrystallization is desired. Apply heat if desired and the compound of interest is compatible.

3.8.2.2. Extraction of Organic Bases

3.8.2.2.1. Dissolve the sample in water or an aqueous solution.

3.8.2.2.2. Add a basic reagent to the aqueous solution and verify basicity with litmus or pH paper.

3.8.2.2.3. Add a suitable organic solvent to extract the basic compound(s) of interest.

3.8.2.2.4. Remove the organic solvent from the aqueous solution for the intended analysis.

3.8.2.3. Extraction of Psilocybin Mushrooms

3.8.2.3.1. Break up an appropriate amount of mushrooms (approximately 2 grams if available)

3.8.2.3.2. Add water and Sodium Bicarbonate and mix/grind until a paste-like consistency is achieved.

3.8.2.3.3. Wash paste with ethyl ether.

3.8.2.3.4. Remove ethyl ether and evaporate in the fume hood.

3.8.2.3.5. Reconstitute in methanol for analysis.

3.8.2.4. Extraction of Cocaine Base

3.8.2.4.1. Place sample in a test tube or disposable glass vessel.

3.8.2.4.2. Add hexane (approximately 3 mL) and mix.

3.8.2.4.3. Add water (approximately 20 mL).

3.8.2.4.4. Remove hexane layer.

3.8.2.4.4.1. Repeat if necessary.

3.8.2.4.5. Evaporate the solvent if necessary for the intended analysis.

3.8.2.5. Extraction by Filtration/Solvent Wash

3.8.2.5.1. Place sample in filtering apparatus over a container.

3.8.2.5.2. Wash sample with several small portions of suitable solvent.

3.8.2.5.3. Alternatively, if a pipette with glass wool or cotton is used, a sample may be brought up in a suitable solvent first and then washed through the pipette into a container.

3.8.2.5.4. Suggested uses: Cocaine hydrochloride and inositol, Cocaine base and phenacetin, Oxycodone and Acetaminophen

3.8.3. Record the extraction technique used in the case file in sufficient detail to allow the technique to be repeated. Record the lot number of any prepared reagents used. When a specific target pH is desired check the pH with pH test paper and record the observed pH (ie. acidic, basic, pH-8).

3.8.4. Negative Control for GC/MS extractions

3.8.4.1. For each sample or batch of samples prepared to be analyzed by GC/MS, prepare a negative control extraction immediately prior to or concurrent with the sample(s) using the same techniques, reagents and materials in approximately the same amounts.

3.8.4.1.1. When using disposable vessels the negative control extraction may be performed in separate glassware from the sample extraction.

3.8.4.1.2. When using reusable vessels, perform the negative control extraction in the vessel immediately prior to performing the sample extraction.

4. Limitations - NA

5. Safety - NA

6. Records

6.1. Reagent log

7. References

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Document Revision History		
Revision Date	Prepared By	Revision
7/10/2023	L. Wiley	Corrected typo in 3.5.4 "…/day/NaOH/initials of preparer." to "…month/day/SatNaOH/initials of preparer."
11/7/2024	A. Abernethy	Document revised to reflect the agency name change from Raleigh/Wake City-County Bureau of Identification to Wake County Bureau of Forensic Services, effective December 1, 2024. Changed header and revision history format. No change to procedure content.