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Discipline/Name of Document: Toxicology
5.12 Solution Preparation

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Section Five
Quality Assurance

5.12 Solution Preparation

5.12.1 BACKGROUND

Refer to references.

5.12.2 SCOPE

This section describes the proper preparation of solutions and buffers used in the extraction of drug compounds from blood and urine specimens.

5.12.3 EQUIPMENT AND SUPPLIES

5.12.3.1 Glassware
Adequately sized beakers, volumetric flasks, graduated cylinders and volumetric pipettes

5.12.3.2 Laboratory balance

5.12.3.3 pH Meter and/or Indicator Strips

5.12.3.4 Appropriate buffer solutions for pH meter

5.12.3.5 Stirring hotplate

5.12.3.6 Magnetic stirrers

5.12.3.7 Safety Equipment

- Chemical Fume Hood
- Acid Resistant Apron
- Laboratory Coat
- Safety Goggles and/or face Shield
- Laboratory Gloves

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5.12.4 REAGENTS

All chemicals must be ACS Grade or equivalent.

5.12.4.1 Acids

- Acetic, Glacial
- Hydrochloric
- Phosphoric
- Sulfuric

5.12.4.2 Salts

- Ammonium Chloride
- Potassium Hydroxide
- Potassium Phosphate Monobasic
- Potassium Phosphate Dibasic

- Sodium Acetate Trihydrate
- Sodium Bicarbonate
- Sodium Hydroxide
- Sodium Phosphate Monobasic
- Sodium Phosphate Dibasic
- Sodium Tetraborate Decahydrate

5.12.4.3 Enzyme

- β-Glucuronidase (Patella vulgata)

5.12.4.4 Solvents

- Methanol

5.12.5 PROCEDURES

Preparation of the following solutions must be recorded on corresponding preparation log.

Note: Appropriate safety equipment must be worn during the preparation of solutions to prevent exposure to caustic/corrosive solutions. The order of the addition of chemicals may be crucial to prevent exothermic reactions.

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5.12.5.1 Acetic Acid

5.12.5.1.1 **0.1M/100mM Acetic Acid (500mL)**
Place approximately 300mL distilled/deionized (DI) water into a 500mL volumetric flask. Add 2.9mL **glacial acetic acid**, mix. QS to 500mL.
Solution is stable for six months.

5.12.5.1.2 **1.0M Acetic Acid (500mL)**
Place approximately 400mL DI water into a 500mL volumetric flask. Add 29mL **glacial acetic acid**, mix. QS to 500mL.
Solution is stable for six months.

5.12.5.1.3 **20% Acetic Acid (500mL)**
Place approximately 300mL DI water into a 500mL volumetric flask. Add 100mL glacial acetic acid, mix. QS to 500mL.
Solution is stable for six months.

5.12.5.2 Ammonium Chloride

5.12.5.2.1 **Saturated Ammonium Chloride (500mL)**
Place approximately 300mL DI water in a beaker and heat/stir over low heat. Add **ammonium**

chloride until the solution is saturated. QS to 500mL.
Solution is stable for 6-months.

5.12.5.3 Borate Buffers

5.12.5.3.1 **Saturated Borate Buffer , pH >9.5 (500mL)**
Place ≅250mL DI water into a 500mL volumetric flask. Stir while adding **sodium tetraborate (≅60g)** until solution is saturated. QS.
Solution is stable for six months.

5.12.5.3.2 **Borate Buffer, pH 9.2**
Place ≅500mL DI water into a 1000mL beaker. Heat and stir while adding 50g sodium tetraborate ($Na_2B_4O_7 \cdot 10\ H_2O$). Once dissolved, allow to cool. Bring volume up to ≅950mL with DI water. Verify pH and adjust as necessary to pH 9.2 ±0.2 with 1N NaOH/KOH or 1N HCl. Place solution in 1000mL volumetric flask and QS with DI water.
Solution is stable for six months.

5.12.5.3.3 **Borate Buffer, pH 12**
Place ≅500mL DI water into a 1000mL beaker. Heat and stir while adding 50g sodium tetraborate ($Na_2B_4O_7 \cdot 10\ H_2O$). Once dissolved, allow to cool. Bring volume up to ≅ 900mL with DI water. Add 25mL NaOH and stir. Verify pH and adjust as necessary to pH 12 ±0.2 with 10N NaOH/KOH or 6N HCl. Place solution in 1000mL volumetric flask and QS with DI water.
Solution is stable for six months.

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5.12.5.4 β-Glucuronidase

5.12.5.4.1 **β-Glucuronidase (10mL) {UCT}**
Transfer appropriate units β-Glucuronidase (Patella vulgata) lyophilized, 5,000 units/ml, powder to a clean volumetric flask. QS with 1.0M or 100mM Acetate buffer (pH 5) and mix well. Store at 2-8°C. Allow to come to room temperature prior to use.
Solution is stable for ~1-month.

5.12.5.5 Hydrochloric Acid

5.12.5.5.1 **0.1M/100mM Hydrochloric Acid (500mL)**
Place approximately 300mL DI water into a 500mL volumetric flask. Add 4.2mL **concentrated**

hydrochloric acid, mix. QS to 500mL. Solution may also be prepared by diluting 1N HCl.
Solution is stable for one year.

5.12.5.5.2 **1N HCl (500mL)**
Place approximately 400mL DI water into a 500mL volumetric flask. Add 42mL **concentrated hydrochloric acid**, mix. QS to 500mL.
Solution is stable for one year.

5.12.5.5.3 **1% HCl in Methanol (10mL)**
Add approximately 5mL of methanol to a 10mL volumetric flask. Pipet 100µL of **concentrated HCl**, QS and mix.
Solution is stable for one-month.

5.12.5.6 Potassium Hydroxide (KOH)
Note: The addition of KOH to water will generate significant heat, exercise due caution.

5.12.5.6.1 **1N/1M Potassium Hydroxide (1000mL)**
Gradually add 56.0g **potassium hydroxide** to approximately 800mL DI water; stir on stir plate to dissolve. QS to 1000mL.
Solution is stable for one year.

5.12.5.6.2 **1.0M Potassium Hydroxide (100mL)**
Dissolve 5.6g **potassium hydroxide** in approximately 80mL DI water in a 100mL volumetric flask. QS to 100mL.
Solution is stable for one year.

5.12.5.6.3 **2N/2M Potassium Hydroxide (1000mL)**
Gradually add 112.0g **potassium hydroxide** to approximately 800mL DI water; stir on stir plate to dissolve. QS to 1000mL.
Solution is stable for one year.

5.12.5.6.4 **6N Potassium Hydroxide (1000mL)**
Gradually add 337g **potassium hydroxide** to approximately 600mL DI water, stir on stir plate to dissolve. QS to 1000mL.
Solution is stable for one year.

5.12.5.6.5 **10N Potassium Hydroxide (500mL)**

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Gradually (!) add 280g **potassium hydroxide** to approximately 300mL DI water in a minimum of a 800mL beaker, stir on stir plate to dissolve. Allow solution to cool (this takes awhile) and QS in a 500mL volumetric flask.
Solution is stable for one year.

5.12.5.6.6 **11.8N Potassium Hydroxide (1000mL)**
Gradually(!) add 662g **potassium hydroxide** to approximately 600mL DI water, stir on stir plate to dissolve. Allow to cool (this takes awhile) and QS in a 1000mL volumetric flask.
Solution is stable for one year.

5.12.5.7 Potassium Phosphate Buffers

5.12.5.7.1 **Saturated Potassium Phosphate Buffer (1000mL)**
Place approximately 1000mL DI water in a beaker and heat/stir over low heat. Add **potassium phosphate monobasic** until the solution is saturated. Allow solution to cool. Adjust pH to approximately 1.8 with **concentrated phosphoric acid**.
Solution is stable indefinitely at room temperature.

5.12.5.7.2 **0.1M/100mM Potassium Phosphate Buffer (100mL) - Adjusted to pH 6**
Dissolve 1.36g **potassium phosphate monobasic** in ≈90mL DI water in a 150mL beaker. Adjust to pH 6.0 with 1.0M **potassium hydroxide.** QS in a 100mL volumetric flask.
Solution is stable for 6-months.

5.12.5.7.3 **100mM Potassium Phosphate Buffer (1000mL) Adjusted to pH 6 (Varian)**
Weigh 13.6g of potassium phosphate monobasic (KH_2PO_4) into a ≥1000mL beaker. Add ≅900mL DI water. Stir to dissolve. Adjust pH to 6.0 ±0.1 with 1M KOH while stirring. Bring up to volume with DI water in an 1000mL volumetric flask.
Solution is stable for 1 month. Store in glass container.

5.12.5.7.4 **100mM Potassium Phosphate Buffer (1000mL) Adjusted to pH 8 - 9 (Varian)**

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Weigh 13.6g of potassium phosphate monobasic (KH_2PO_4) into a ≥1000mL beaker. Add ≅900mL DI water. Stir to dissolve. Adjust pH to 8 - 9 with 10M KOH while stirring. Bring up to volume with DI water in an 1000mL volumetric flask.
Solution is stable for 1 month. Store in glass container.

5.12.5.8 Sodium Acetate

5.12.5.8.1 **1.0M Sodium Acetate (100mL)**
Dissolve 13.6g **sodium acetate** in 90mL DI water in a 100mL volumetric flask. QS to 100mL with DI water.
Solution is stable for 6-months.

5.12.5.8.2 **0.1M/100mM Sodium Acetate (100mL)**
Place 10mL **1.0 M sodium acetate** in a 100mL volumetric flask. QS to 100mL with DI water.
Solution is stable for 6-months.

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5.12.5.9 Sodium Acetate Buffers

5.12.5.9.1 **0.1M/100mM Acetate Buffer, pH 4.5 (500mL)**
Dissolve 2.93g **sodium acetate trihydrate** in 400mL DI water in a 600mL beaker. Add 1.62mL **glacial acetic acid**, and mix well. Adjust to pH 4.5±0.1 with **100mM sodium acetate, glacial acetic acid** or **100mM acetic acid.** QS to 500mL in a 500mL volumetric flask.
Solution is stable for six months.

5.12.5.9.2 **0.1M/100mM Acetate Buffer, pH 5.0 (500mL)**
Prepare as with pH 4.5 buffer (5.12.5.9.1). Adjust pH to 5.0 ±0.1.

5.12.5.9.3 **1.0M Acetate Buffer, pH 5.0 (500mL)**
Dissolve 42.9g **sodium acetate trihydrate** in 400mL DI water. Add 10.4mL **glacial acetic acid** and stir well. QS to 500mL. Adjust to pH 5.0±0.1 with **1.0M sodium acetate** or **1.0M acetic acid**.
Solution is stable for six months.

5.12.5.9.4 **2.0M Acetate Buffer, pH 4.8 (1000mL)**
Dissolve 141.4g **sodium acetate trihydrate** in ≈800mL DI water. Add 55.2mL **glacial acetic acid**. Adjust to pH 4.8 and QS to 1000mL.

Solution is stable for six months.

5.12.5.10 Sodium Bicarbonate

5.12.5.10.1 **50mM Sodium Bicarbonate, pH 11 (500mL)**
Dissolve 2.1g **sodium bicarbonate** in 500mL DI water.
Solution is stable indefinitely at room temperature.

5.12.5.11 Sodium Hydroxide (NaOH)
Note: The addition of NaOH to water will generate heat, exercise due caution.

5.12.5.11.1 **0.45N NaOH (500mL)**
Gradually add 9g **NaOH** in 500mL DI water. (Caution: Exothermic)
Solution is stable for one year.

5.12.5.11.2 **2N NaOH (1000mL)**
Place approximately 250mL DI water into a 1000mL beaker. Gradually add 80g **NaOH.** Transfer to 500mL volumetric flask and QS to 500mL. (Caution: Exothermic)
Solution is stable for one year.

5.12.5.11.3 **10N NaOH (500mL)**
Place approximately 400mL DI water into a 1000mL beaker. Gradually add 200g **NaOH.** Transfer to 500mL volumetric flask and QS to 500mL. (Caution: Exothermic)
Solution is stable for one year.

5.12.5.12 Sodium Phosphate

5.12.5.12.1 **100mM Sodium Phosphate Dibasic (200mL)**
Dissolve 2.84g **sodium phosphate dibasic** in ≈160mL DI water. QS to 200mL and mix.
Solution is stable for 1 month. Store in glass container.

5.12.5.12.2 **100mM Sodium Phosphate Monobasic (200mL)**
Dissolve 2.76g **sodium phosphate dibasic** in ≈160mL DI water. QS to 200mL and mix.
Solution is stable for 1 month. Store in glass container.

5.12.5.13 Sodium Phosphate Buffers

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5.12.5.13.1 **0.1M/100mM Sodium Phosphate Buffer (100mL) Adjusted to pH 6**

Dissolve 1.70g **sodium phosphate dibasic** (Na_2HPO_4) and **12.14 sodium phosphate monobasic** ($NaH_2PO_4 \cdot H_20$) in approximately 800mL DI water in a 1000mL volumetric flask. QS to 1000mL. Adjust to pH 6.0 ±0.1 with 100mM **monobasic sodium phosphate** (to lower pH) or 100mM **dibasic sodium phosphate** (to raise the pH).

Solution is stable for 1 month. Store in glass container.

5.12.5.13.2 **0.1M/100mM Phosphate Buffer, pH 6 (250mL)**

Dissolve 0.42g **sodium phosphate dibasic** (Na_2HPO_4) and **3.03 sodium phosphate monobasic** ($NaH_2PO_4 \cdot H_20$) in approximately 200mL DI water in 250mL volumetric flask. QS to 250mL. Adjust to pH 6.0±0.1 with 100mM **monobasic sodium phosphate** (lowers pH) or 100mM **dibasic sodium phosphate** (raises pH).

Solution is stable for 1 month. Store in glass container.

5.12.5.14 Sulfuric Acid

5.12.5.14.1 **0.05M/0.1N Sulfuric Acid**

Place approximately 800mL distilled/deionized (DI) water into a 1L volumetric flask. Add 2.7mL **concentrated sulfuric acid**, mix. QS to 1L.

Solution is stable for one year.

5.12.5.14.2 **1N Sulfuric Acid (H_2SO_4) (500mL)**

Place approximately 400mL DI water into a 500mL volumetric flask. Add 14mL **concentrated H_2SO_4**. QS to 500mL, mix.

Solution is stable for one year.

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5.12.6 QUALITY ASSURANCE

5.12.6.1 Refer to toxicology Analytical Method 5.2 for balance intermediate check and calibration requirements.

Note: Balances properly monitored by drug discipline analysts fulfills quality assurance requirements. Additional check need not be performed.

5.12.7 REFERENCES

5.12.7.1 Shugar, G.J., Shugar, R.A. and Bauman, L. *Grades of Purity of Chemicals* pp. 145-154, *pH Measurement*. pp. 232-234. *in:* Chemical Technicians' Ready Reference Handbook, McGraw Hill: New York, 1973.

5.12.7.2 Ansys, Inc. SPEC Extraction Methods

5.12.7.3 United Chemical Technologies, Inc. Applications Manual.

5.12.7.4 Certify Methods Manual, pp. 8 - 12, Varian Sample Preparation Products, January 2001.

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Revision History

Section Five
Quality Assurance

5.12 Solution Preparation

Revision # Issue Date History

Revision #	Issue Date	History
0	05-07-2007	Combined urine solution preparation (2.6) and blood solution preparation (3.8).
1	08-20-2008	Removed obsolete solutions, added reference for balance check requirements, clarifications.

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Idaho State Police
Forensic Laboratory Training Manual
Cocaine

1.0.0 HISTORY

Archaeological artifacts show that the use of coca was widely accepted in ancient cultures of South American Indians. Paintings on pottery, ornaments depicting pictures and symbols of the coca bush and its leaves, as well as sculptured wood and metal objects dating as far back as 3000 BC on the coast of Ecuador indicate the use of coca in both civil and religious rituals. Relatively recent studies of the antiquity of the use and cultivation of coca indicate that the coca plant is native to the eastern Andes Mountains. Until this day, the natives in the area continue the custom of chewing coca.

A French chemist, Angelo Mariani, introduced Europe to the coca leaf by importing tons of coca leaves and using an extract from them in many products such as his "Coca Wine." Cocaine, as obtained from the coca leaves, was first discovered by Gaedecke in 1855 and rediscovered by Niemann in 1859, at which time he gave the compound the name cocaine. The local anesthetic properties of cocaine were demonstrated first by Wohler in 1860; however, it was not used medically until 1864 as a topical anesthetic in the eye.

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2.0.0 TAXONOMY

The French botanist Joseph de Jussieu made the first taxonomical reference to coca[1] in 1750. He assigned the plants to the genus **Erythroxylum**. Later, Lamarck, another French botanist, published six new species, including the famous **Erythroxylum coca** Lamarck, in 1786. Today, the full taxonomical classification is:

CATEGORY	TAXON
Division (phylum)	**Spermatophyta**
Class	**Dicotyledons** (shrubs and trees)
Order	**Geraniales**
Family	**Erythroxylaceae**
Genus	**Erythroxylum**

[1]Note that other literature sources seem to credit Patrick Brown as the founder of the genus **Erythroxylum** (in 1756).

Species One	**Erythroxylum coca** Lamarck
Variety of Species One	**Erythroxylum coca var. ipadu** (Plowman)
Species Two	**Erythroxylum novogranatense** (Morris) Hieronymus
Variety of Species Two	**Erythroxylum novogranatense var. truxillense** (Rusby) Plowman

The spelling of the genus name **Erythroxylum** is considered a hybrid of Greek and Latin. Linnaeus changed the spelling of the generic name to **Erythroxylon**, a pure Latin form. Nevertheless, botanists consider **Erythroxylum** as the proper spelling since it was derived in accordance with the rules of nomenclature.

Excluding the wild species of coca, all cultivated coca plants are currently considered to belong to two distinct species of **Erythroxylum** including one variety within each of the species. Specific areas of cultivation for each follows:

1. **Erythroxylum Coca Lam. (or E. coca)** -- This is the most important species from which almost all-commercial cocaine is derived. This species is native to the eastern Andes extending from Ecuador south to Bolivia. This was the first type of cultivated coca to be collected and studied by Europeans and was referred to as Huanuco or Bolivian coca after the main areas of cultivation. Like many plants that have been cultivated for thousands for years, E. coca is now rarely found in a truly wild state.

A particular variety of E. coca that warrants a brief mention since it has gained some recent notoriety is the coca of the Amazon Valley that, until today, continues to be cultivated on a very small scale by a number of Indian tribes. It belongs to the species of E. coca, but differs from the typical Andean species and is described as a variety, i.e., **Erythroxylum coca**, var. ipadu.

2. **Erythroxylum novogranatense** (Morris) Hieronymus -- This is the second species[2] of cultivated coca and is commonly known as Colombian coca. This species is cultivated throughout the mountains of Colombia. The species name "novogranatense" refers to the geographical origin of the plant, Nueva Grenad, the old colonial name for Colombia. There appears to be little cocaine production based upon this species.

During the early 1900s, the Europeans created a cocaine industry on one of the major islands of Indonesia (i.e., Java). Dutch farmers adapted the coca species **Erythroxylum novogranatense** to their soil and climate using modern agricultural techniques. Commonly referred to as "Java" coca, its total alkaloidal content was found to be higher than Colombian coca; however, its true cocaine

[2]Older scientific publications (prior to 1976) refer to the studies of Morris who described this type of coca plant as a variety of E. coca. The German botanist Hieronymus was the first to recognize it as a distinct species.

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content was found to be much lower than Colombian coca. This variation in alkaloidal content is attributed to changes in climate, soil, and possibly other environmental conditions. The Java coca industry practically collapsed just prior to World War II because of declining prices for coca leaves from other countries.

3. **Erythroxylum novogranatense var. truxillense** (Rusby) Plowman -- This is one recognized and distinct variety within the species **Erythroxylum novogranatense**. It is well known as Trujillo coca[3] since it is cultivated near the city of Trujillo in northern Peru on the slopes of the Andes (as well as the nearby desert coast of Peru). This variety of (Colombian) coca is particularly rich in methyl salicylate (oil of wintergreen) and other flavoring compounds that are used in the production of coca-flavored beverages. (Coca-Cola is a popular beverage that incorporates decocainzed extracts of Trujillo coca in its preparation.)

3.0.0 MORPHOLOGICAL AND ECOLOGICAL CHARACTERISTICS

Bolivian (or Huanuco) coca leaves are usually large and thick, broadly elliptic in shape, pointed at the apex, and dark green in color. The underside of the leaves has two lines (parallel) to the midrib. These leaves are not considered characteristic of this species since they are present to a degree in the other species of coca. The leaves are usually deciduous after the current season's growth. The plant grows as a small to medium size bush usually planted in rows on large, hillside plantations. This species thrives on moist, cool mountain climates.

Colombian coca can be distinguished from Bolivian coca by its smaller, narrower, thinner, bright yellowish-green leaves, which are usually rounded at the apex. Contrary to the Bolivian plant, the Colombian coca plant holds its leaves on the branches after a season's growth. This habit gives Colombian coca a larger, bushier, and robust appearance. In addition, Colombian coca prefers the hotter, drier climate and thrives at lower elevations. Dried Colombian leaves also have a more noticeable odor of methyl salicylate than Bolivian leaves.

Trujillo coca is morphologically similar to Colombian coca. Trujillo coca has smaller, narrower, and slightly thicker leaves that are darker green at maturity.

Refer to Table 1 for additional characteristics for both varieties of **Erythroxylum coca** and **Erythroxylum novogranatense**.

The genus **Erythroxylum** is the only natural source of the alkaloid cocaine and related compounds. Almost all commercial coca leaves and cocaine are derived from the species **Erythroxylum coca** Lam.; **Erythroxylum novogranatense** (Morris) Hieronymus, because of its quick adaptability to different climates, is most likely the dominant species that is illegally cultivated for the illicit drug trade.

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[3]Sometime referred to as Truxillo or Peruvian coca.

SPECIES OF COCA AND THEIR CHARACTERISTICS

Genus, Species	ERYTHROXYLUM COCA		ERYTHROXYLUM NOVOGRANATENSE	
Varieties	**coca**	**ipadu**	**novogranatense**	**truxillense**
Origin	montaña region of eastern Andes; Ecuador, Peru, and Bolivia, mainly between 500 - 1500m	western Amazon of Brazil, Colombia, and Peru	Colombia, Venezuela and Central America, Sierra Nevada de Santa Marta and rugged mountains of Cauca and Hucha	desert coast of Peru and in adjacent arid valley of the Rio Maranon, Truxillo region on the north coast of Peru
Description of plant and/or leaves	pointed leaves, parallel longitudinal lines on leaf undersides	tall, spindly shrub with long weak branches and relatively large elliptical leaves which are blunt or rounded at the apex; flowers have a shorter flusher pedicel and a markedly denticulate staminal tube only short styled morphs	large bush plant with small, narrow, thin, and bright yellow-green leaves which are rounded	up to 3m tall with multiple trunks reaching 4 cm in diameter; branches are dense erect and spread leaves narrowly elliptical to oblong-lanceolate 20-65 mm long; medium to light green above pale green to glossy green beneath and midrib fluted with slight medial ridge
Odor	grassy or haylike		wintergreen	wintergreen
Climate	favorable tropical environment with high rainfall, moderate temperatures and well drained mineral rich soils; moist, cool	does not like intense heat or poorly drained soils, short-lived	hot, seasonably dry habitat resistant to drought	has been cultivated in arid, desert climate and wet montaña habitat of Colombia; even more tolerant to drought; prefers desert conditions
Adaptability	very little	very little	will survive under a wide range of environmental conditions; Resistant to drought	
Means of propagation	seeds	cuttings	seeds	seeds
Commercial uses	most important commercial species providing by far the largest supply of coca leaves and cocaine; 95% of Peru's crop	used for chewing	illegal in Colombia; grown illegally for coca chewing and cocaine production	principal variety used in beverage industry owing to its high content of essential oils and flavors-several hundred tons exported to N.Y. for preparation of extracts, used in making Coca-Cola
% Alkaloids	0.5 - 1.0	unknown	1.0 - 2.5	1.0 - 2.5
% Cocaine of Total alkaloid content	70 - 90	very little	20 - 50	20 - 50

Table 1

Note that the genus **Erythroxylum** also includes a number of wild species of coca (reportedly about 200), most of which contain minimal quantities of cocaine and some of which contain no detectable quantities of cocaine.

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4.4.0 CHEMISTRY

4.1.0 Coca leaves contain three basic groups of alkaloids, most of which are present in the form of esters. The content of these alkaloids will vary depending on the particular species of coca used, age of the plant, where it is grown, how it was cultivated, and when the leaves were picked. A higher total alkaloid content but with a smaller cocaine content reportedly occurs when the leaves are picked at an early stage of development; the reverse occurs when the leaves are picked when fully developed. Another important factor affecting the variation of an alkaloidal extract is the manner in which the alkaloids were refined from the leaves. Listed in specific groups are some of the popular alkaloids found in coca leaves:

Derivatives of Ecgonine

a. *l*-Cocaine (Methylbenzoylecgonine)
b. cis- and/or trans-Cinnamoylcocaine[4] (cis- and/or trans-Methylcinnamoylecgonine)
c. α and/or β-Truxillenes (Methyl α and/or β-truxilloylecgonine or α and/or β-cocaine)
d. Methylecgonine (Ecgonine methyl ester)
e. Methylecgonidine

Derivatives of Tropine (and ψ-Tropine)[5]

a. Tropococaine (Benzoyl-ψ-tropeine or ψ-tropine benzoate)

Derivatives of Hygrine

a. Cuscohygrine
b. Hygroline
c. Hygrine

In addition to *l*-cocaine, cinnamoylcocaine and the truxillenes are considered the most important coca alkaloids for pharmaceutical use. Hygrine and its related alkaloids are reported to be present in significantly large quantities in both varieties of **E. novogranatense** (i.e., Colombian and Trujillo coca). Hygrine is a tertiary aminoketone, hygroline is the secondary alcohol related to hygrine, and cuscohygrine is a diaminoketone containing two N-methyl groups.

The pharmaceutical industry produces pure cocaine semi-synthetically. The ecgonine conversion process employed eliminates the need to separate cocaine from related ecgonine alkaloids and produces a much greater yield of cocaine. Briefly, the procedure involves:

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[4] Cinnamylcocaine and cinnamoylcocaine have been used interchangeably in the literature.

[5] Esters of tripine are called tropeines. ψ is the Greek symbol for pseudo.

1. Extracting the coca alkaloids previously mentioned from dried coca leaves and isolating the mixture of ecgonine alkaloids.

2. Converting the group of alkaloids to ecgonine via hydrolysis with dilute hydrochloric acid:

Cocaine ⟶ *l*-Ecgonine•HCl + MeOH + Benzoic Acid

Cinnamoylcocaine ⟶ *l*-Ecgonine•HCl + MeOH + Cinnamic Acid

Truxillene ⟶ *l*-Ecgonine•HCl + MeOH + Truxillic Acid

(Tropococaine is excluded since it would hydrolyze to pseudotropin and not ecgonine.)

3. Isolating and purifying the ecgonine and converting it to its free base.

4. Converting the ecgonine to *l*-cocaine by benzoylating the ecgonine with benzoic anhydride to benzoylecgonine followed by methylation with methyl iodide.

An alternate commercial process converts the isolated alkaloids to ecgonine methyl ester (or methyl ecgonine) by hydrolyzing the alkaloids in methyl alcohol-hydrogen chloride. The ester is isolated and benzoylated directly to *l*-cocaine. Either industrial process provides the following approximate yield:

Dried Coca Leaves ⟶ Cocaine HCl
145 kgs ⟶ 1 kg

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In clandestine cocaine laboratories in South America, the crude cocaine process also seeks to eliminate most of the coca alkaloids except cocaine. The cured process is an extensive operation. It generally consists of:

1. a "pasta" laboratory where all the coca alkaloids are extracted from a batch of coca leaves and converted to a water soluble (sulfate salt) pasty material called "pasta" or "sulfate" or coca paste;

2. a "base" laboratory (optional) where the coca paste is treated to remove most undesirable substances and to provide cocaine base;

3. a "crystal" laboratory where the cocaine base is converted to cocaine hydrochloride.

Very often, however, the illicit laboratory "cook" abbreviates or overlooks a critical step of the process and causes other alkaloids, impurities, and cocaine decomposition products to become part of the final product. The approximate yield from this illicit process can be summarized as follows:

Dried Coca Leaves ⟶ Coca Paste ⟶ Coca Base ⟶ Cocaine HCl
240 kgs ⟶ 1.4 kg ⟶ 0.9 kg ⟶ 1 kg

If chemical conditions become too acidic or basic, cocaine can degrade, as illustrated in Figures 1 and 2, and the degradation products may recombine to form new products. For example, benzoic acid and methyl alcohol will combine to yield methyl benzoate, a sweet smelling, volatile liquid. Similarly, cinnamoylcocaine and truxilline can decompose, producing products that can recombine to form new products, such as

CHEMICAL STRUCTURE OF COCAINE

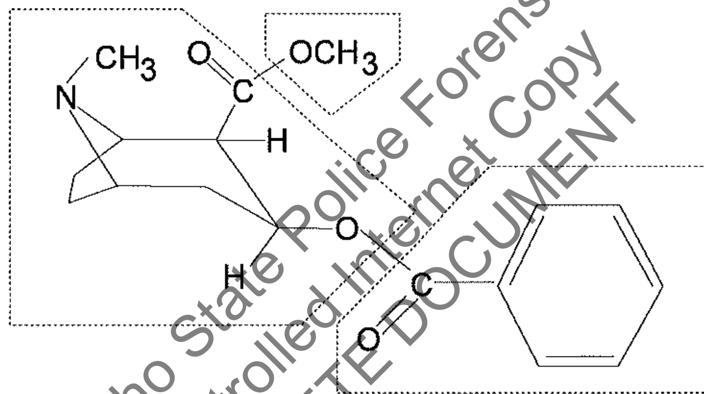

Figure 1

The cocaine molecule is made up of three building blocks. If chemical conditions become too acidic or too basic the cocaine molecule readily breaks down into these three pieces.

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DECOMPOSITION OF COCAINE

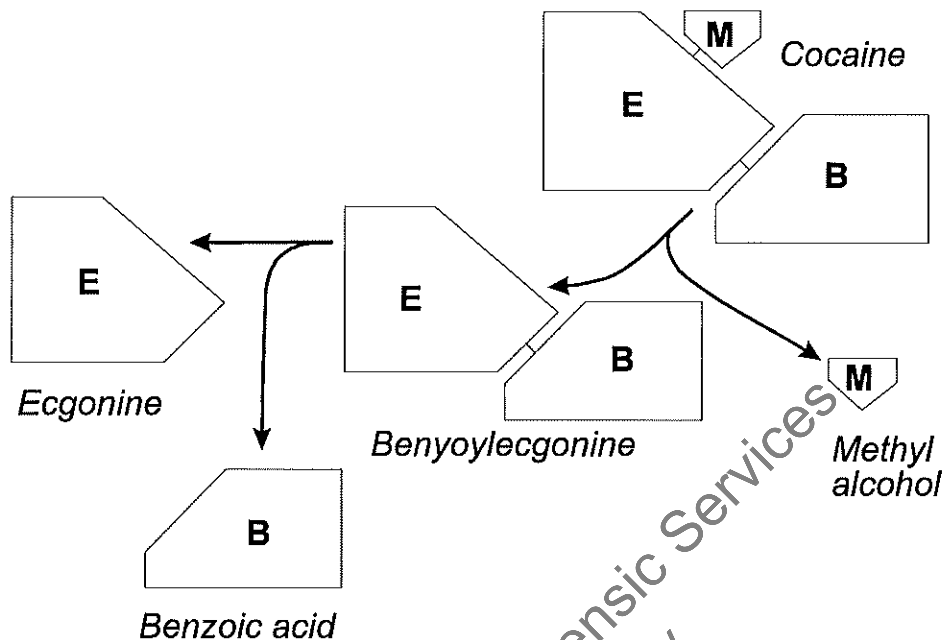

Figure 2

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methyl cinnamate and methyl truxillate. Contrary to earlier studies, benzoylecgonine, ecgonine, pseudococaine, and cocaethylene are believed to be produced during the refining process and are not naturally occurring in the coca plant.

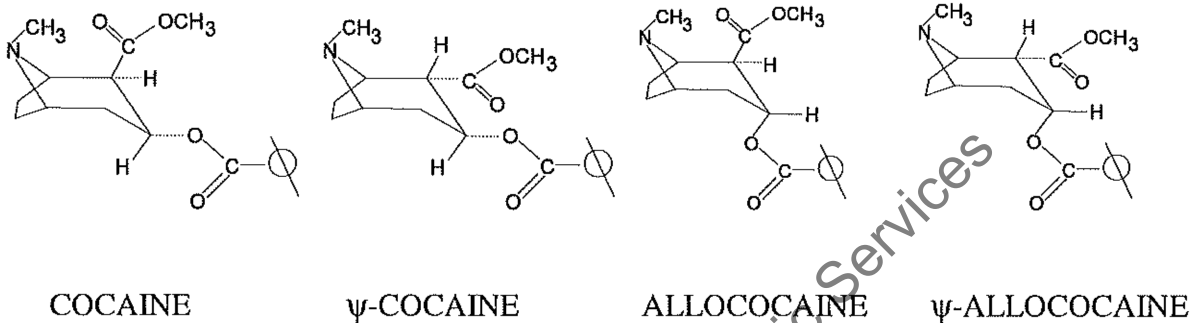
4.2.0 SYNTHESIS OF COCAINE

In 1923, using 2-carbomethoxytropanone as an intermediate, Willstatter prepared (±) cocaine along with pseudococaine. At least four synthetic methods have been reported for the synthesis of 2-carbomethoxytropanone. These syntheses range from the difficult cyclization of 1-methyl-2,5-dicarboethoxypyrrolidine, to the simplest, at least in principle, reaction of succindialdehyde, methylamine, and β-ketoglutaric acid to yield the target compound 2-carbomethoxytropanone. Willstatter's synthesis in 1923 involved the formation of the dipotassium salt of monomethyl-β-ketoglutarate, which was then reacted with methylamine and succinaldehyde to obtain the 2-carbomethoxytropanone. The methyl esters of (±) pseudoecgonine and (±) ecgonine are formed by the reduction of 2-carbomethoxytropanone using a sodium-mercury amalgam; fractional crystallization can be used to separate the two ecgonine methyl esters. The final step in the reaction scheme is between the (±) ecgonine methyl ester and benzoyl chloride to yield (±) cocaine.

4.3.0 STEREOCHEMISTRY

During the period 1898 to 1923, Willstatter and his collaborators were able to obtain pseudococaine by transformation of (-) cocaine and by total synthesis. At

the time of this early work, stereochemistry was in its infancy. Based on chemical studies regarding ecgonine and tropine, Willstatter incorrectly assigned pseudococaine a structure that has both the C-2 carbomethoxy group and the C-3 benzoyloxy group axial. Instead, later studies show that pseudococaine has both the carbomethoxy group at C-2 and the benzoyloxy group at C-3 in equatorial position.



Two other cocaines, allococaine and allopseudococaine, were synthesized and characterized by Findley in the 1950s. Again, problems arose with the nomenclature used in representing the stereochemistry of these compounds. Using Findley's assignments, the carbomethoxy group at C-2 and the benzoyloxy group at C-3 were equatorial-axial, respectively, for allococaine and axial-axial for allopseudococaine. More recent nomenclature places allococaine and cocaine with the same stereochemistry at the C-2 position, that is, the carbomethoxy group is axial. Pseudococaine and allopseudococaine have the same spatial relationship at the C-2 position; viz., the carbomethoxy group is equatorial. Thus, the cocaine molecule has four asymmetrical centers which give rise to eight stereoisomers arranged as four diastereoisomeric pairs -- *d,l*-cocaine, *d,l*-pseudococaine, *d,l*-allococaine, and *d,l*-allopseudococaine.

5.0.0 SAMPLE ANALYSIS

The detection and identification of cocaine through acceptable analytical methods generally presents no problems; however, in most instances, pure cocaine is not presented for analysis. Even when diluents and adulterants have not been added to the cocaine, the sample usually contains impurities resulting from the original isolation of the alkaloid. For example, cis- and trans-cinnamoylcocaine are usually present in illicit cocaine samples. Benzoylecgonine may be present in many cocaine samples and should be eliminated by a basic extraction since its UV is similar to that of cocaine. Note that the detection of benzoyl ecgonine (as well as ecgonine) by UV, GLC, or TLC can be difficult because of the polar nature of the acid group at C-2.

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A second complication in the analysis of cocaine samples results from the presence of adulterants. Many of the "caines," such as benzocaine, lidocaine, procaine, and tetracaine, are commonly encountered.

From a practical standpoint most of the analyses conducted at the ISP Laboratory will utilize capillary GC/MS for the identification of cocaine. The high resolution capabilities of capillary GC will easily separate cocaine from most common adulterants and diluents.

6.0.0 EXERCISES

1. Prepare a sample of "crack" cocaine from cocaine hydrochloride.
2. Obtain a sample of procaine base mixed with cocaine base. Via Infrared analysis, prove that the sample contains cocaine base. (In other words, separate the cocaine from the procaine without converting either to its salt.
3. Obtain an infrared spectrum of Cocaine Sulfate. Compare with the spectra of Cocaine HCl and Cocaine base.

7.0.0 DISCUSSION QUESTIONS

1. Is cocaine is a stimulant or a narcotic?
2. Name the two countries that produce the majority of cocaine.
3. Name one country prominent in the smuggling of cocaine.
4. Draw the structure of the four cocaine isomers and show the equatorial/axial relationship for the carbomethoxy group and the benzoyloxy group for each isomer.
5. Draw the structures of procaine, lidocaine, tetracaine, and benzocaine. Are they related to cocaine?
6. Describe the process of preparing "crack" cocaine.
7. Draw the cocaine molecule and indicate what portion of the molecule the 82, 182, and 272 ions arise from.
8. Describe the process by which Colombian chemists convert the crude cocaine base to refined cocaine hydrochloride.

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8.0.0 REFERENCES

1. CND Analytical, Inc., (1990), Analytical Profiles of Cocaine, Local Anesthetics and Common Diluents found with Cocaine, pp. 1 - 11.

2. Jatlow, P.I. "Drug of Abuse Profile: Cocaine," Clinical Chemistry, Vol. 33, No. 11(B), 1987

3. Smith RM. "The Mass Spectrum of Cocaine," Journal of Forensic Science, 42(3): 475-480, 1997

4. Heffley, S.R. "Cocaine Legal Note," Internal KBI Document

5. Roberson, J.C. "Legal and Analytical Aspects of Cocaine," Microgram, Vol. XI, No.3, Mar. 1978, pp. 45-46

6. Allen, A.C., Cooper, D. A., et al "The Cocaine Diastereoisomers," Journal of Forensic Science, Vol.26, No.1, Jan. 1981, pp.12-26

7. Gelsomino, R., and Raney, J.K., "NMR Identification of Cocaine," Microgram, Vol. XII, Dec. 1979, pp. 222-225.

8. Ruybal, R., "Differentiation of d- and l-Cocaine by Microcrystalline Test," Microgram, Vol. XV, Sept. 1982, pp. 160-163.

9. Sorgen, G.J., "Identification of d- and l- Cocaine," Microgram, Vol. XVI, Aug. 1983, pp. 126-131.

10. Siegel, J.A., Cormier, R. A., "The Preparation of d-Pseudococaine from l-Cocaine," Journal of Forensic Sciences, Vol. 25, No. 2, Apr. 1980, pp. 357-365.

11. Hufsey, J., and Cooper, D., "Synthetic Cocaines," Microgram, Vol. XII, Dec. 1979, pp. 231-239.

12. Kerr, K. "A Simple Procedure for Separating Cocaine Base From Procaine Base," Microgram, Vol. XXIII, No. 5, May 1990, pp. 93-95.

13. Galka, K.I., "Separation of Cocaine from Benzocaine," Microgram, Vol XXIII, May 1990, pp. 97-98.

14. Clark, C.C., and Allen, A.C., "Quantitative Separation of Cocaine-Lidocaine Mixtures," Microgram, Vol. VII, 1974, pp. 96 - 99.

15. Lebelle, M., Callahan, S., etal, "Comparison of Illicit Cocaine by Determination of Minor Components," Journal of Forensic Sciences, Vol. 36, No. 4, Jul. 1991, pp. 1102-1120.

16. Casale, J. F., Waggoner, R. W., "A Chromatographic Impurity Signature Profile Analysis for Cocaine Using Capillary Gas Chromatography.," Journal of Forensic Sciences, Vol. 36, No.5, Sept. 1991, pp.1312-1330.

17. Ensing, J. G., Hummelen, J. C., "Isolation, Identification and Origin of Three Previously Unknown Cogners in Illicit Cocaine," Journal of Forensic Sciences, Vol.36, No. 6, Nov. 1991, pp. 1666-1687.

18. Ensing, J.G., deZeeuw, R. A., Detection, Isolation, and Identification of Truxillines in Illicit Cocaine by Means of Thin-Layer Chromatography and Mass Spectrometry," Journal of Forensic Sciences, Vol 36, No. 5, Sept 1991, pp. 1299-1311.

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19. Carpenter, A., Laing, R. R., "Cocaine in Bleach: Destroying the Evidence, Identification of Degredation Products" Microgram, Vol. XXVII, No. 8, Aug, 1994, pp. 249-252.

20. Casale, J. F., "Methyl Esters of Ecgonine: Injection-Port Produced Artifacts from Cocaine Base (Crack) Exhibits," Journal of Forensic Sciences, Vol. 37, No. 5, Sept. 1992, pp. 1295-1310.

21. Moore, J.M., "Some Observations On the Quantitative Analysis Of Purportedly Pure Cocaine And Heroin Samples," Microgram, Vol. V, pp. 38-46.

22. Cartier, J., Gueniat, O., "Headspace Analysis of Solvents in Cocaine and Heroin Samples," Science & Justice, 37(3), 1997, pp. 175-181.

23. Ard, E., "Problems With IR Spectra of Cocaine Hydrochloride Case Samples," Microgram, Vol. XXI, No. 2, Feb. 1988, pp. 29-33.

24. Santos, N. A., "Moisture Absorption Problems Associated With The Packaging of Cocaine Hydrochloride," Microgram, Vol. XXV, No. 2, Feb. 1992, pp. 28-32.

25. Kiser, W. O., "Analysis for Cocaine Base," Microgram, Vol. XXI, No. 2, Feb. 1988, p. 28.

26. Scott, L.J., "Specific Field Test For Cocaine," Microgram, Vol. VI, 1973, pp. 179-181.

27. Fasanello, J., Higgins, P., "Modified Scott Test For Cocaine Base Or Cocaine Hydrochloride," Microgram, Vol. XXIII, No. 4, Apr. 1990, pp. 137-138.

28. Kaufman, M.S., "Household Bleach as a Test for Drugs," Microgram, Vol. XXIII, No. 4, Apr. 1990, pp. 75 - 77.

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