Drug Chemistry Section Drug Chemistry Procedure Manual Effective Date: August 3, 1998

Modification of C-7
Prepared By: I. L. Allcox
Approved By: I.L. Allcox
Supercedes: September 1, 1996

Name of Procedure:

Thin-Layer Chromatography 18:1 Developing Solvent

Suggested Uses:

The 18:1 Thin-Layer Chromatography Developing Solvent is used to separate and identify organic compounds. Suggested uses of this TLC solvent include opium alkaloids, ergot alkaloids, synthetic opiates, coca alkaloids and other acidic, basic, and neutral organic compounds and drugs.

Apparatus Needed to Perform Procedure Including Preparation of Reagent:

Thin-Layer Chromatography Plates Silica gel GF - fluorescent indicator

Thickness: 250 microns

Developing tank Micro pipets

UV light (long and short wave)

Fume hood

Gloves

Eye protection

Laboratory coat

Graduated cylinders

Reagent bottle

Funnel

Porcelain spot plate

Spatula

Chloroform

Methanol

Concentrated Ammonium Hydroxide

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Formula for Preparing Reagent:

1. Measure out 180 milliliters of ammonia saturated chloroform and 10 milliliters of methanol.

- 2. Pour measured solvents into reagent bottle and mix well.
- 3. Properly label reagent bottle.

Note: Ammonia saturated chloroform is prepared by shaking chloroform and concentrated ammonium hydroxide in a separatory funnel.

Quality Control Check:

A quality control check of this reagent will be performed using a known standard of heroin and following the application procedure listed below.

Expiration Date of Chemical Reagent:

The solvent can be used until depletion provided it is stored in an airtight reagent bottle.

Application of Procedure on Evidence:

- 1. A sample of the unknown (approximately 1-2 milligrams) is placed in the well of a porcelain spot plate and several drops of a suitable solvent are added to dissolve the sample.
- 2. A known standard (approximately 1-2 milligrams) is also dissolved in the well of a spot plate with several drops of a suitable solvent. (Known standard is selected after visual observation and/or a series of preliminary tests).
- 3. With a capillary pipet, several microliters of the unknown and standards solutions are placed side by side approximately 5 millimeters from the bottom of the TLC plate.

Application of Procedure on Evidence (continued):

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- 4. Add 18:1 solvent to the developing tank to a depth of approximately 2 millimeters and allow several minutes for atmosphere to equilibrate.
- 5. Allow all the spotting solvent to evaporate from the TLC plate.
- 6. Place the TLC plate in the developing tank and close the lid.
- 7. Allow the TLC plate to develop to the top of the plate.
- 8. Remove the TLC plate from the developing tank and allow the solvent to dry.
- 9. Visualize with UV light or an appropriate visualizing reagent.

Safety Concerns:

Always wear eye protection, gloves and a laboratory coat when preparing this TLC solvent.

Literature References:

Randerath, Kurt, **Thin-Layer Chromatography**, New York, Academic Press, 1968.

Moffat, A.C., <u>Clarke's Isolation and Identification of Drugs</u>, 2nd Ed., The Pharmaceutical Press, 1986, pp. 166-177.

Sperling, Albert, "Analysis of LSD," Microgram, Vol. III, No. 1, 1970.

Moore, Richard A., <u>Methods of Analysis</u>, Laboratory Division, Bureau of Narcotics and Dangerous Drugs, United States Department of Justice, pp. 63-67.

This procedure has been used in the Drug Chemistry Section since 1971.