Name of Procedure:

Quantitative Analysis of Methamphetamine by Gas Chromatography

Suggested Uses:

To determine the number of dosage units in a sample of methamphetamine. Under the North Carolina Controlled Substances Act, possession of more than one hundred dosage units of methamphetamine is a felony. Methamphetamine is often encountered in the form of a powder of clandestine origin. The amount of methamphetamine contained in the powder must be determined before the number of dosage units can be calculated.

Methamphetamine is manufactured by Abbott Laboratories and sold as a pharmaceutical preparation (Desoxyn) in tablets containing 5 mg, 10 mg, and 15 mg of methamphetamine hydrochloride. For the purpose of determining the number of dosage units in a powder (or liquid) containing methamphetamine, a dosage unit will be defined as 15 mg of methamphetamine hydrochloride, or an equivalent amount of another form of methamphetamine.

Apparatus Needed to Perform the Procedure:

Safety Glasses
Fume Hood
Gloves
Varian 3400 Gas Chromatograph
Spectra-Physics 4270/4290 Integrator
Chloroform
Volumetric flasks
Volumetric pipets
Electronic Balance
10 μl syringe
Operation Manual for a Varian 3400 Gas Chromatograph
Operation Manual for a Spectra-Physics 4270/4290 Integrator
DB-5 Megabore Column, ID 0.53 mm, 30 meters, 1.5 microns
Helium
Hydrogen
Compressed Air
Methamphetamine Hydrochloride

Lidocaine Hydrochloride
General Operational Procedures:

1. Varian 3400 Gas Chromatograph set up:

   Helium - 7 psi (18 ml/min) flow rate
   Split Ratio - 1:10
   Air and Hydrogen - preset flow rates by manufacturer

Turn the air and hydrogen controls until they are fully open and ignite the detector.

2. Method Parameters: Temperature Program

Initial Column Temperature	140 degrees C
Initial Column Hold Time	2 minutes
Column Pressure	7.0 psi
Program 1 Final Column Temperature	260 degrees C
Column Rate in degrees/minute	15.
Column Hold Time	5 minutes
Inject Temperature	250 degrees C
Detector Temperature	275 degrees C
FID Atten	1
FID Range	11
FID Autozero	Yes
Initial Relays	-1
Relay Time in Minutes	0.03
Program 1 Relays	1
Complete Time	15 minutes

3. Spectra-Physics 4270/4290 Integrator Method set-up:

File Name	Methamphetamine Quantitation	
TT=.01	TF=PM	TV=0
TT=.01	TF=NE	TV=1
TT=.01	TF=II	TV=1
TT=2.	TF=II	TV=0
TT=2.	TF=NE	TV=0
TT=2.	TF=PM	TV=1
TT=11.	TF=ER	TV=1
Method Number	2	
Number of Levels	NV=0	

General Operationl Procedures: (Continued)

RT=3.43 RF=.17 CN=Methamphetamine

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RT=9.87   RF=1      CN=Lidocaine
RRT REF Peak:          3.43
INT STD PEAK:          9.87
Sample Table:          default setting
Analyst:               default setting
Injections/Sample      1
Samples between calib: 100
Conc Units             mg/ml
SAM IDX              default setting
SI                   default setting
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Calibration Standards:

1. Prepare a 0.2 mg/ml internal standard solution by weighing 200. mg of lidocaine hydrochloride, placing the lidocaine hydrochloride in a 1 liter volumetric flask, and bringing the flask to volume with chloroform. Store the internal standard solution in in a properly labeled and sealed container in a refrigerator. The internal standard solution may be used until depleted.

2. Prepare a 1.0 mg/ml methamphetamine stock solution by weighing 50. mg of methamphetamine hydrochloride, placing the methamphetamine hydrochloride in a 50 ml volumetric flask, and bringing the flask to volume with the internal standard solution.

3. Prepare 0.3 mg/ml, 0.2 mg/ml, 0.15 mg/ml, 0.1 mg/ml, and 0.05 mg/ml calibration standard solutions of methamphetamine hydrochloride by dilution of the methamphetamine stock solution with the internal standard solution. Store the calibration standard solutions in properly labeled and sealed containers in a refrigerator. The calibration standard solutions may be used until depleted.

Calibration and Determination of Response Factor:

1. Run each calibration standard (1 µ-liter injection) at least twice on the gas chromatograph.

Calibration and Determination of Response Factor: (Continued)

2. For each calibration run, divide the area of the methamphetamine HCl by the area of the internal standard (lidocaine) and plot these numbers against the concentrations of the calibration standards. Confirm that a linear relationship exists between these variables by plotting on a graph. Concentrations of methamphetamine HCl that fall within this linear range - 0.05 mg/ml to 3.0 mg/ml - may be determined by this procedure.

3. For each calibration run, divide the area of the internal standard (lidocaine) by the area of the methamphetamine HCl and multiply by the concentration of the calibration standard. This calculation determines the response factor for that calibration run. Average all of the response factors for the calibration standards to determine the average response factor. Program this response factor into the Spectra-Physics Integrator Method set-up.

4. The retention times and the response factor should be verified by the use of a calibration standard for each analysis. If the retention times or response factor vary by more than 5% from the values listed in the "General Operational Procedures", a new calibration run should be initiated and the new values for the retention times and response factor programed into the Spectra-Physics Integrator Method set-up.

Sample Preparation, Analysis, and Calculations:

1. Weigh approximately 15 mg of sample into a 50 ml volumetric flask.

2. Bring the flask to volume with the internal standard solution.

3. Verify that the calibration of the instrument is correct by the use of at least one calibration standard (1 µ-liter injection).

4. Run a 1 µ-liter solvent blank on the instrument.

5. Inject 1 µ-liter of the sample. The results of the analysis will be printed on the chromatogram as a concentration of methamphetamine HCl in mg/ml.

Sample Preparation, Analysis, and Calculations: (Continued)

6. To calculate the amount of methamphetamine HCl in the sample:

$$\frac{[\text{meth. mg/ml}] \times 50 \text{ ml}}{\text{wt of sample in flask}} = \% \text{ of methamphetamine HCl in sample}$$

Therefore,
(% of meth. in sample) x total wt of sample = total wt of methamphetamine HCl

And to calculate the number of dosage units of methamphetamine HCl in the sample:

$$\frac{\text{total wt of meth. (mg)}}{\text{15 mg/dosage unit}} = \text{total number of dosage units of methamphetamine HCl}$$

7. If another form of methamphetamine is present in the sample, use the appropriate conversion factors to determine the equivalent amount of methamphetamine HCl and the corresponding number of dosage units in the sample.

Safety Concerns:

Safety glasses, gloves, a lab coat, and a fume hood are essential when preparing solutions.

General maintenance should be conducted when the instrument is unplugged and cool to protect against burns and electrical hazards.

Literature References:

Arky, Ronald and Davidson, Charles S., **1998 Physicians Desk Reference**, Medical Economics Company Inc., Montvale, NJ.

Spectra-Physics SP4270/4290 Users Guide, Spectra-Physics, 1986,

San Jose, California.

Varian 3300/3400 Gas Chromatograph Operator's Manual, Varian Associates, Inc., Walnut Creek, California, 1988.

Literature References: (Continued)

Skoog, Douglas A., **Instrumental Analysis**, Third Ed., Saunders College Publishing, New York, 1985, pp 727- 783.

Willet, John, **Analytical Chemistry by Open Learning - Gas Chromatography**, John Wiley & Sons, 1987.

Bonelli, E. J. and McNair, H. M., **Basic Gas Chromatography**, Varian Aerograph, Walnut Creek, California, 1967.