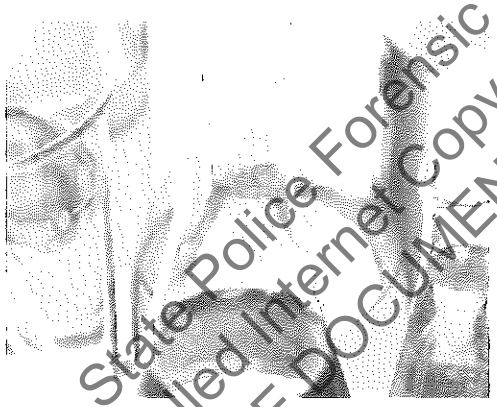
Idaho State Police
Forensic Services

Approval for Quality System Controlled Documents



Discipline/Name of Document: Controlled Substances
#11 Analytical Method for the Quantification of Methamphetamine Using GC/MS with Internal Standards

Revision Number: 5

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APPROVED BY: _____ 11/20/08
Quality Manager — Date Signed

#11
Analytical Method
For the Quantification of Methamphetamine Using GC/MS with Internal Standards

1.0.0 Background

Under normal circumstances quantification of a substance's purity is not part of the analytical scheme used by the Idaho State Police Forensic laboratories. By special request this analysis can be performed. Typically this analysis is performed on casework that will ultimately be tried in federal court. This analytical method was derived from the principles and methods detailed in EPA publication "SW-846" and the states of Oregon and Utah's quantitation analytical methods.

2.0.0 Scope

The following procedures have only been approved for the analysis of methamphetamine.

3.0.0 Equipment and Reagents

3.0.0 Gas Chromatograph/ Mass Spectrometer (GC/MS) and corresponding software.

3.1.0 Solid methamphetamine hydrochloride. The purity is to be documented with a certificate of analysis from the vendor.

3.3.0 ACS grade chloroform stabilized with either ethanol or pentene.

3.4.0 Class A volumetric flasks.

3.5.0 1.0 ml Gastight® type syringes. Syringes that are used to generate the standard calibration curve will have their accuracy checked before each use via section 7.9.0 of this AM. The verification must encompass the expected working range of the syringe, 200ul and 800ul. Syringes that fail to meet the acceptance value of (+/-) 3% will be replaced.

3.6.0 Internal standard. With a ratio of 1.3 ml of (98% or greater) n-tridecane per 1 L chloroform, prepare at least one liter. Each sequence of samples and standards must be made with the same internal standard.

3.7.0 0.5 N sodium carbonate solution. Add 2.7g of sodium carbonate to 100mls of water.

4.0.0 Generation of Standard Curve

A six point calibration curve will be generated.

4.1.0 Prepare a standard stock solution of approximately 2,000 ug/ml. Accurately weigh approximately 40-50 mg of methamphetamine, add to a 25ml volumetric flask and dissolve and bring to volume with the internal standard. Calculate the concentration.

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4.2.0 Using the syringe, auto-sampler vials, and stock standard prepare an additional five 1.0 ml standards. Into five autosampler vials place 0.1, 0.2, 0.4, 0.6, 0.8 ml of stock std and then dilute to 1.0 ml using the internal standard. The undiluted stock standard must be one of the points on the curve. If the stock standard point does not fall within the linear range of the instrument then a more dilute stock standard is prepared and a new curve is run or the acquisition parameters of the instrument can be altered, i.e. split ratio, and the original curve rerun.

4.3.0 Add approximately 100 ul (3 drops) of a 0.5N sodium carbonate solution to each vial and mix.

4.4.0 Using the GC/MS software set up the calibration acquisition parameters and tables. The curve is to be generated using linear regression with the points weighted using the inverse square. For Hewlett Packard/Agilent Chemstation software, the parameters and tables are found in the data analysis/ calibration section.

5.0.0 Sample Preparation

One of the basic requirements in determining an accurate quantification is that the sample must be homogenous. The sample must also be prepared using the same extraction procedure that was used in generating the standard curve.

5.1.0 Initially rough grind the sample with a mortar and pestle until the entire sample will pass through a US No. 4 sieve. Roll and quarter the sample until a representative sub sample of about 10 grams is obtained. Grind the sub sample until a fine powder is formed. **NOTE:** If the sample is less than 10 grams then grind the entire sample into a fine powder.

5.2.0 Using an analytical balance that is accurate to at least 0.1 milligram, accurately weigh out an amount of sample that is equal to, or less than, what was used for the stock standard, and place into a 25 ml volumetric flask. Add internal standard, dissolve, and bring to volume.

5.3.0 Into an auto sampler vial aliquot approximately one milliliter of sample extract, add approximately 100 ul of 0.5 N Na_2CO_3, mix and analyze.

5.4.0 Samples are to be run in duplicate (two separate weighings and extractions). The sample exhibiting the lowest response is used for calculating the result. The duplicate results must have a Relative Percent Difference (RPD) of less than 10%, if they are not then extract a new pair of samples and analyze.

$$RPD = \frac{|R1-R2|*100}{A}$$

Where R1 = Result of first run in percent
R2 = Result of second run in percent
A = Average of R1 and R2

5.5.0 If a sample(s) is to be forwarded to another laboratory for quantitative analysis, the originating laboratory will analyze the sample(s) qualitatively, prepare the sample(s) as per 5.1.0 above, then send a maximum of 1g per sample to the laboratory doing the quantitative analysis.

5.5.1 If permission is granted from the federal prosecutor, samples may be analyzed as a composite. The samples will be composited at the originating laboratory by mixing all of the samples that tested positive qualitatively for methamphetamine and the resultant mixture is processed per section 5.1.0.

6.0.0 Calculation and Reporting of Final Results

6.1.0 Calculation

Using the equation of the valid curve, calculate the concentration in the vial (the computer software should do this). Use the following equation to calculate the concentration of the analyte in the original sample:

$$\frac{(\mathbf{A}\ \text{ug/ml}) \times (\mathbf{Milliliters\ of\ solvent})}{(10) \times (\mathbf{B}\ \text{mg})} = \mathbf{C}\ \%\ \text{analyte}$$

A = Concentration given by curve
B = Weight of sample used, in milligrams

If C is less than 48 % then the sample is re-extracted and reanalyzed using a larger sample size. For calculating any re-extraction use the weight, in mg, of methamphetamine used to make the stock standard. The calculated result of the re-extraction must be greater than 48%.

6.2.0 Reporting

Using the formula:

C x D x (0.90)= X

Where **C**=result from equation in 6.1.0
D= total weight of sample in grams

Report the result as "the sample contains at least X grams of methamphetamine *calculated as the hydrochloride salt*"

7.0.0 Notes and QA/QC

7.1.0 The curve must be linear as defined by a correlation coefficient of 0.998 or better. The correlation coefficient is generated by the Agilent (Hewlett-Packard) Chemstation software.

7.2.0 The area counts of the internal standard should be consistent from the beginning to the end of the run (+/- 10% of the mean).

7.3.0 A new curve will be generated before each quantitation sequence. A sequence is defined as a batch(s) run consecutively without the introduction of non-quantitation samples. A batch is defined as up to twenty injections. At the end of each batch a positive control will be run, the results of which must be (+/-) 7% of the stated value. The Relative Percent Difference (RPD) will be calculated for each batch of positive controls:

$$RPD = \frac{|R1-R2|}{E} * 100$$

Where R1 = calculated result of the first positive control run after the generation of the curve.

R2 = calculated result of positive control run at the end of the batch, or sequence if two or more batches are run together.

E = Expected value

The RPD will be less than 14%.

7.4.0 Injector should have a split liner with a glass wool plug.

7.5.0 A positive control will be analyzed each time a curve is generated. The positive control will come from a source other than what was used to generate the curve. Another in-house standard from a different lot, if available, and prepared by a different analyst is to be used as the positive control. To a 100ml volumetric flask add approximately 0.1g of methamphetamine, that has been accurately weighed, then dissolve and bring to the mark with internal standard. The positive control is made with the same batch of internal standard as the rest of the run. Aliquot one milliliter into a auto-sampler vial and add sodium carbonate solution.

7.6.0 The accuracy of the curve is validated when the value of the positive control is within (+/-) 7% of the stated value.

7.7.0 The calibration curve, chromatogram and quantitation report of the positive controls, chromatogram(s) and quantitation report(s) of all samples, and chromatograms of all applicable blanks are to be kept in the case notes. Chromatograms of standards used to generate the curve do not need to be kept.

7.8.0 Each time a quantitative analysis is performed a data pack will be sent to the discipline leader. This data pack will include; copies of the calibration curve, all quantitation reports, the calculated positive control RPD's for each batch, and the calculated internal standard mean.

7.9.0 For the 1.0 ml syringe weigh 10 replicate aliquots of water at 200 ul and 800 ul. Calculate average recovery and standard deviation at each level. For the purpose of the calculations, the density of water is 0.998 g/ml. The acceptance criteria are (+/-) 3%.

8.0.0 History

Revision #	Issue or review date	History	Author or Reviewer
0	5/24/02	Original Issue	D.C. Sincerbeaux
1	8/27/02	Add #	D.C. Sincerbeaux
2	1/10/03	Added 7.7 and 7.8	D.C. Sincerbeaux
3	9/30/05	Added 4.4.0, Changed 5.1.0, 5.2.0, 5.3.0, 4.3.0, 7.3.0 and renumbered 7.0. Added 7.10.0	D.C Sincerbeaux
4	8/08/08	added 3.5, 3.6, 5.3, 5.5.0, 5.5.2 6.2, 7.8, 7.9. Changed 4.1, 4.2, 4.4, 6.1, 6.2, 7.1, 7.3, and 7.5	D.C. Sincerbeaux
5	11/20/08	Changed 5.4.0 and 6.0.0	D.C. Sincerbeaux