



# National Institute of Standards & Technology

## Certificate of Analysis

### Standard Reference Material<sup>®</sup> 2379

#### Drugs of Abuse in Human Hair I

This Standard Reference Material (SRM) is intended primarily for use in validating methods for determining cocaine (COC), benzoylecgonine (BZE), cocaethylene (CE), amphetamine (AMP), methamphetamine (MAMP), and phencyclidine (PCP) in human hair and similar materials. This SRM can also be used for quality assurance when assigning values to in-house control materials. The SRM is a drug-free human hair material to which drugs of abuse have been added. A unit of SRM 2379 consists of one bottle containing approximately 100 mg of hair segments.

**Certified Mass Fraction Values:** The certified mass fraction values for the target compounds in SRM 2379 are provided in Table 1. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or taken into account [1]. The certified concentration values for each level are based on the agreement of results from gas chromatography – mass spectrometry (GC-MS) and liquid chromatography – mass spectrometry (LC-MS). Values are reported on an as-received basis in mass fraction units.

**Expiration of Certification:** The certification of **SRM 2379** is valid, within the measurement uncertainty specified, until **01 January 2017**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see “Instructions for Storage and Use”). However, the certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

**Maintenance of SRM Certification:** NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

The overall direction and coordination of the analytical measurements leading to the certification of this SRM were performed by M.J. Welch and K.W. Phinney of the NIST Analytical Chemistry Division.

Certification measurements were performed by J.L. Prendergast, L.T. Sniegoski, and S.S-C. Tai of the NIST Analytical Chemistry Division. Additional measurements were performed by M.M. Schantz of the NIST Analytical Chemistry Division and B.E. Lang of the NIST Biochemical Science Division.

Statistical consultation was provided by N.-f. Zhang of the NIST Statistical Engineering Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

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*Revision History on Last Page*

## NOTICE AND WARNING TO USERS

Warning: For laboratory use only.

## INSTRUCTIONS FOR STORAGE AND USE

**Storage:** The SRM should be stored under refrigeration at temperatures between 4 °C and 8 °C.

**Use:** A quantity of hair appropriate for a particular analysis should be withdrawn and weighed on an analytical balance prior to extraction and analysis.

## SOURCE, PREPARATION AND ANALYSIS<sup>1</sup>

**Preparation:** SRM 2379 is human hair from a single individual. The hair was manually cut into short segments and then soaked for 19 days in a 0.01 mol/L solution of HCl in equal volumes of water:dimethylsulfoxide containing the target analytes. The hair was removed from the mother liquor, rinsed several times with methanol, and allowed to air dry.

**NIST Analyses for the Target Drugs of Abuse:** For each of the analytes, measurements from two independent methods based on GC-MS and LC-MS were used. One GC-MS method was used for measurement of AMP and MAMP; another GC-MS method was used for the other four compounds. For the GC-MS methods [2], hair samples were spiked with deuterated analogs of each of the analytes and then extracted with 0.1 mol/L HCl at 45 °C for 24 h. The extracts were neutralized and put through commercial solid-phase extraction cartridges designed for urine drug testing following the manufacturer's directions. After the eluants were collected and dried, COC, BZE, CE, and PCP were dissolved in N,O-bis(trimethylsilyl)acetamide and analyzed together. AMP and MAMP were derivatized with heptafluorobutyric acid anhydride and analyzed together. The GC-MS was equipped with a 30 m low-polarity GC column, and measurements were performed using electron ionization and selected ion monitoring. For the LC-MS method, all six compounds were measured together in a single LC-MS run. Hair samples were spiked with deuterated analogs of each of the analytes and extracted with 0.1 mol/L HCl at 45 °C for 24 h. The extracts were neutralized and put through solid-phase extraction cartridges that were different from those used for the GC-MS method. No derivatization was necessary for the LC-MS measurements. The LC was equipped with a deactivated C<sub>18</sub> column, and compounds were eluted using a gradient mobile phase of 0.1 % (volume fraction) acetic acid in water and methanol. The electrospray ionization conditions were optimized for each analyte. For both GC-MS and LC-MS methods, three sets of samples were measured for all of the analytes. Each set consisted of two samples each from three bottles of SRM 2379. Calibration standards were run before and after the samples, and a standard curve was generated by a least-squares fit of all of the standards measurements for a particular set.

**Homogeneity Assessment:** For the sample sizes used for the certification measurements (20 mg to 25 mg), there was no evidence of within-bottle inhomogeneity. There also was no correlation between fill order and concentrations measured for any of the analytes.

**Stability Measurements:** Analyses of the target analytes were performed using the GC-MS and LC-MS approaches previously described. The certified concentration values for amphetamine and cocaine were updated based upon the stability measurements.

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<sup>1</sup>Certain commercial materials and equipment are identified to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are the best available for the purpose.

**Certified Mass Fraction Values:** The uncertainty provided with each value is an expanded uncertainty about the mean to cover the measurand with approximately 95 % confidence, consistent with the ISO Guide and its Supplement 1 [3–7]. The expanded uncertainty is calculated as  $U = ku_c$ , where  $u_c$  is the combined uncertainty and  $k$  is a coverage factor corresponding to approximately 95 % confidence for each analyte [3]. For the certified values shown below,  $k = 2$ .

Table 1. Certified Mass Fraction Values for Analytes in SRM 2379

Analyte	Mass Fraction (ng/mg)
Amphetamine	5.49 ± 0.28
Benzoylecgonine	4.01 ± 0.31
Cocaethylene	2.67 ± 0.24
Cocaine	6.84 ± 0.54
Methamphetamine	5.20 ± 0.27
Phencyclidine	6.24 ± 0.42

#### REFERENCES

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- [2] Welch, M.J.; Sniegowski, L.T.; Allgood, C.C.; Habram, M.; *Hair Analysis for Drugs of Abuse: Evaluation of Analytical Methods, Environmental Issues, and Development of Reference Materials*; J. Anal. Toxicol., Vol. 17, pp. 389–398 (1993).
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**Certificate Revision History:** 24 September 2012 (Revised certified mass fraction values for cocaine and amphetamine, editorial changes, and an extension of the certification period); 08 November 2002 (Original certificate issue date).

*Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the SRM Program: telephone (301) 975-2200; fax (301) 948-3730; e-mail [srminfo@nist.gov](mailto:srminfo@nist.gov); or via the Internet at <http://www.nist.gov/srm>.*