NIST NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY
U.S. DEPARTMENT OF COMMERCE

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03 February 2026

Standard Reference Material® 3672a
Organic Contaminants in Smokers' Urine
(Frozen)
CERTIFICATE OF ANALYSIS

Purpose: The certified values delivered by this Standard Reference Material (SRM) are intended for use in evaluating analytical methods for the determination of caffeine, creatinine, selected hydroxylated polycyclic aromatic hydrocarbons (hydroxylated PAHs) and phthalate, phenol, nicotine, volatile organic compound (VOC), herbicide, and pesticide metabolites in urine. All the constituents for which certified and non-certified values are provided are naturally present in the urine.

Description: A unit of SRM 3672a consists of five vials each containing 5 mL of frozen urine. The development of SRM 3672a was a collaboration between NIST and Division of Laboratory Sciences, Centers for Disease Control and Prevention (CDC).

Certified Values: These values are traceable to International System of Units (SI) unit of mass. The values are reported on an as-received basis [1,2].

Certified Mass Fraction Values in SRM 3672a

Analyte	Mass Fraction[(a)] (µg/g)
Caffeine[(b,d,e)]	1.0274 ± 0.0164
Creatinine[(c,d,f)]	809.7 ± 1.1

[(a)] Values are expressed as $x \pm U_{95\%}(x)$, where x is the certified value and $U_{95\%}(x)$ is the expanded uncertainty of the certified value.
[(b)] NIST gas chromatography mass spectrometry (GC-MS) analysis
[(c)] NIST liquid chromatography with tandem mass spectrometry (LC-MS/MS) analysis
[(d)] CDC LC-MS/MS analysis; data were converted from nmol/mL using the density of the materials which was measured to be 1.0052 ± 0.0001 g/mL and the molar mass of caffeine recorded as 194.191 ± 0.005 g/mol.
[(e)] The certified value is the DerSimonian-Laird consensus estimator of µ, $\bar{y}_{DL}$ [3] from two methods. The standard and approximate 95 % level of confidence expanded uncertainties were estimated using the bootstrap method [4]. The standard uncertainty is estimated as the standard deviation of the bootstrap values. A symmetrical expanded uncertainty is estimated as the half-width of the interval from the 2.5th percentile to the 97.5th percentile of the bootstrap values.
[(f)] The certified value is the arithmetic mean from one reference measurement procedure. The standard uncertainty of the mean is the standard deviation divided by the square root of the number of replications. The expanded uncertainty is estimated using the Student's t 0.95 confidence level for n-1 degrees of freedom as the coverage factor.

Non-Certified Values: Non-certified mass fraction values are provided in Appendix A.

Additional Information: Additional information is provided in Appendix B.

Period of Validity: The certified values delivered by **SRM 3672a** are valid within the measurement uncertainty specified until **01 March 2040**. The certified values are nullified if the material is stored or used improperly, damaged, contaminated, or otherwise modified.

Maintenance of Certified Values: NIST will monitor this SRM over the period of its validity. If substantive technical changes occur that affect the certification, NIST will issue an amended certificate through the NIST SRM website (https://www.nist.gov/srm). Before making use of any of the values delivered by this material, users should verify they have the most recent version of this documentation, available through the NIST SRM website (https://www.nist.gov/srm).

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Steven J. Choquette, Director
Office of Reference Materials

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Safety: This is a human-source material. Donors were tested for infectious bloodborne pathogens before donation and urine was filtered and sterility tested for infectious bacterial organisms. SRM 3672a is a Biosafety Level 2 material and should be handled according to applicable federal, state, and/or local regulations and according to policies and procedures of recipient's organization.

Storage: The SRM is stored at –80 °C at NIST. The urine is shipped frozen (on dry ice) and, upon receipt, should be stored frozen until ready for use. A freezer temperature of –20 °C is acceptable for storage for up to one week. If a longer storage time is anticipated, the material should be stored at or below –60 °C. The SRM should not be exposed to sunlight or ultraviolet radiation. Storage of thawed material at room or refrigerator temperatures may result in changes to analyte concentrations.

Use: SRM 3672a IS INTENDED FOR RESEARCH USE. Vials of the SRM to be analyzed should be removed from the freezer and thawed completely to room temperature (20 °C to 25 °C). After the material is thawed to room temperature, it should be used immediately. The material should be vortex mixed before aliquots are withdrawn.

REFERENCES

[1] Beauchamp, C.R.; Camara, J.E.; Carney, J.; Choquette, S.J.; Cole, K.D.; DeRose, P.C.; Duewer, D.L.; Epstein, M.S.; Kline, M.C.; Lippa, K.A.; Lucon, E.; Molloy, J.; Nelson, M.A.; Phinney, K.W.; Polakoski, M.; Possolo, A.; Sander, L.C.; Schiel, J.E.; Sharpless, K.E.; Toman, B.; Winchester, M.R.; Windover, D.; *Metrological Tools for the Reference Materials and Reference Instruments of the NIST Material Measurement Laboratory*; NIST Special Publication 260-136, 2021 edition; National Institute of Standards and Technology, Gaithersburg, MD (2021); available at https://nvlpubs.nist.gov/nistpubs/SpecialPublications/NIST.SP.260-136-2021.pdf (accessed Feb 2026).

[2] Boggs, A.S.P.; Blount, B.C.; Botelho, J.C.; Brosius, C.; Burdette, C.Q.; Calafat, A.M.; Camara, J.; Feng J.; Heckert, A.N.; Hoguet, J.C.; Huncik, K.; Morel Espinosa, M.; Ospina, M.; Ragland, J.; Reese, C.; Rybak, M.; Seyler, T.; Valentin-Blasini, L.; Wang, L.; Wood, E.S.C.; Xia, B.; *Certification of Standard Reference Material®s 3672a & 3673a: Organic Contaminants in Smokers' Urine (Frozen) & Organic Contaminants in Non-Smokers' Urine (Frozen)*; NIST Special Publication (NISTSP) 260-256; National Institute of Standards and Technology, Gaithersburg, MD (2025); available at. https://doi.org/10.6028/NIST.SP.260-256r1 (accessed Feb 2026).

[3] DerSimonian, R.; Laird, N.; *Meta-Analysis in Clinical Trials;* Control Clin Trials, Vol. 7(3), pp. 177–188 (1986).

[4] Efron, B.; Tibshirani, R.J.; *An Introduction to the Bootstrap*; Chapman & Hall: London, UK (1993).

If you use this SRM in published work, please reference:

Boggs ASP, Blount BC, Botelho JC, Brosius C, Burdette CQ, Calafat AM, Camara J, Feng J, Heckert AN, Hoguet JC, Huncik K, Morel Espinosa M, Ospina M, Ragland J, Reese C, Rybak M, Seyler T, Valentin-Blasini L, Wang L, Wood ESC, Xia B (2025) Certification of Standard Reference Material®s 3672a & 3673a: Organic Contaminants in Smokers' Urine (Frozen) & Organic Contaminants in Non-Smokers' Urine (Frozen). (National Institute of Standards and Technology, Gaithersburg, MD), NIST Special Publication (NISTSP) 260-256. https://doi.org/10.6028/NIST.SP.260-256r1

Certificate Revision History: 03 February 2026 (Addition of non-certified values; editorial changes); 14 May 2025 (Original certificate date).

Certain commercial equipment, instruments, or materials may be identified in this Certificate of Analysis to adequately specify 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 necessarily the best available for the purpose.

Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the Office of Reference Materials, 100 Bureau Drive, Stop 2300, Gaithersburg, MD 20899-2300; telephone (301) 975-2200; e-mail srminfo@nist.gov; or the Internet at https://www.nist.gov/srm.

* * * * * * * End of Certificate of Analysis * * * * * * *

APPENDIX A

Non-Certified Values: Non-certified values are suitable for use in method development, method harmonization, and process control but do not provide metrological traceability to the SI or other higher-order reference system. Non-certified mass fraction values are provided below.

Table A1. Non-Certified Mass Fraction Values for Selected Phthalate, Terephthalate, and 1,2-cyclohexane dicarboxylic acid diisononyl ester Metabolites in SRM 3672a

Analyte	Abbreviation	Mass Fraction ng/g[a]
mono-ethyl phthalate[b,c,d]	MEP	499 ± 31
mono-n-butyl phthalate[b,c,d]	MBP	13.95 ± 0.56
mono-isobutyl phthalate[b,c,d]	MiBP	6.75 ± 0.59
monobenzyl phthalate[b,c,d]	MBzP	5.5 ± 4.3
mono-hydroxybutyl phthalate[c,e]	MHBP	1.110 ± 0.048
mono-hydroxyisobutyl phthalate[c,e]	MHiBP	1.929 ± 0.052
mono-2-ethyl-5-hydroxyhexyl phthalate[b,c,d]	MEHHP	4.69 ± 0.91
mono-2-ethyl-5-oxohexyl phthalate[b,c,d]	MEOHP	4.8 ± 4.4
monooxononyl phthalates[c,e]	MONP	1.003 ± 0.030
mono-3-carboxypropyl phthalate[c,e]	MCPP	0.896 ± 0.043
mono-2-ethyl-5-carboxypentyl phthalate[b,c,d]	MECPP	6.24 ± 1.18
monocarboxyisooctyl phthalate isomers[c,e]	MCOP	3.253 ± 0.105
mono-2-ethyl-5-hydrohexyl terephthalate[c,e]	MEHHTP	8.31 ± 0.25
mono-2-ethyl-5-carboxypentyl terephthalate[c,e]	MECPTP	55.27 ± 1.32
cyclohexane-1,2-dicarboxylic acid monohydroxy isononyl ester[c,e]	MHiNCH	0.995 ± 0.055

[a] These values are expressed as $x \pm 2u(x)$, where x is a mean value and $u(x)$ is its associated standard uncertainty. While the best estimate of measurand mass fraction lies within the interval $x \pm 2u(x)$, neither the purity nor the identity of the calibrants used have been determined by NIST. For purposes of harmonization and process control, the imprecision and homogeneity components of uncertainty can be propagated as $u(x)/x$ relative standard deviations.

[b] NIST LC-MS/MS analysis

[c] CDC LC-MS/MS analysis; data were converted from ng/mL using the density of the materials which was measured to be 1.0052 g/mL ± 0.0001 g/mL.

[d] The non-certified value is the DerSimonian-Laird consensus estimator of μ, $\bar{y}_{DL}$ [3] from two methods. The standard and approximate 95 % level of confidence expanded uncertainties were estimated using the bootstrap method [4]. The standard uncertainty is estimated as the standard deviation of the bootstrap values. A symmetrical expanded uncertainty is estimated as the half-width of the interval from the 2.5th percentile to the 97.5th percentile of the bootstrap values.

[e] The non-certified value is the arithmetic mean from one procedure. The standard uncertainty of the mean is the standard deviation divided by the square root of the number of replications. The expanded uncertainty is estimated using the Student's t 0.95 confidence level for n-1 degrees of freedom as the coverage factor.

Table A2. Non-Certified Mass Fraction Values for Selected Polyaromatic Mono-Hydroxylated Hydrocarbons in SRM 3672a

Analyte	Abbreviation	Mass Fraction ng/g[(a)]
1-hydroxynaphtholene[(b,c,d)]	1-NAP	8.78 ± 0.31
2-hydroxynaphtholene[(b,c,d)]	2-NAP	14.70 ± 0.33
2-hydroxyfluorene[(b,c,d)]	2-FLU	0.973 ± 0.189
3-hydroxyfluorene[(c,e)]	3-FLU	0.5760 ± 0.0114
2-hydroxyfluorene + 3-hydroxyfluorene[(b,d)]	2&3-FLU	1.78 ± 0.23
1-hydroxyphenanthrene[(c,e)]	1-PHE	0.1401 ± 0.0030
2-hydroxyphenanthrene[(b,e)]	2-PHE	0.1053 ± 0.0046
3-hydroxyphenanthrene[(b,e)]	3-PHE	0.2482 ± 0.0061
2-hydroxyphenanthrene + 3-hydroxyphenanthrene[(b,c,d)]	2&3-PHE	0.345 ± 0.039
4-hydroxyphenanthrene[(b,e)]	4-PHE	0.0425 ± 0.0026
1-hydroxypyrene[(b,c,d)]	1-PYR	0.2455 ± 0.0177

[(a)] These values are expressed as $x \pm 2u(x)$, where x is a mean value and $u(x)$ is its associated standard uncertainty. While the best estimate of measurand mass fraction lies within the interval $x \pm 2u(x)$, neither the purity nor the identity of the calibrants used have been determined by NIST. For purposes of harmonization and process control, the imprecision and homogeneity components of uncertainty can be propagated as $u(x)/x$ relative standard deviations.

[(b)] NIST LC-MS/MS analysis

[(c)] CDC LC-MS/MS analysis; data were converted from ng/mL using the density of the materials which was measured to be 1.0052 g/mL ± 0.0001 g/mL.

[(d)] The non-certified value is the DerSimonian-Laird consensus estimator of μ, $\bar{y}_{DL}$ [3] from two or three methods. The standard and approximate 95 % level of confidence expanded uncertainties were estimated using the bootstrap method [4]. The standard uncertainty is estimated as the standard deviation of the bootstrap values. A symmetrical expanded uncertainty is estimated as the half-width of the interval from the 2.5th percentile to the 97.5th percentile of the bootstrap values.

[(e)] The non-certified value is the arithmetic mean from one procedure. The standard uncertainty of the mean is the standard deviation divided by the square root of the number of replications. The expanded uncertainty is estimated using the Student's t 0.95 confidence level for n-1 degrees of freedom as the coverage factor.

Table A3. Non-Certified Mass Fraction Values for Selected Caffeine Metabolites in SRM 3672a

Analyte	Mass Fraction μg/g[(a)]
Paraxanthine[(b,c,d)]	4.84 ± 0.38
Theobromine[(b,c,d)]	1.913 ± 0.121
Theophylline[(b,c,d)]	0.3538 ± 0.0054

[(a)] These values are expressed as $x \pm 2u(x)$, where x is a mean value and $u(x)$ is its associated standard uncertainty. While the best estimate of measurand mass fraction lies within the interval $x \pm 2u(x)$, neither the purity nor the identity of the calibrants used have been determined by NIST. For purposes of harmonization and process control, the imprecision and homogeneity components of uncertainty can be propagated as $u(x)/x$ relative standard deviations.

[(b)] NIST LC-MS/MS analysis

[(c)] CDC LC-MS/MS analysis; data were converted from ng/mL using the density of the materials which was measured to be 1.0052 g/mL ± 0.0001 g/mL.

[(d)] The non-certified value is the DerSimonian-Laird consensus estimator of μ, $\bar{y}_{DL}$ [3] from two methods. The standard and approximate 95 % level of confidence expanded uncertainties were estimated using the bootstrap method [4]. The standard uncertainty is estimated as the standard deviation of the bootstrap values. A symmetrical expanded uncertainty is estimated as the half-width of the interval from the 2.5th percentile to the 97.5th percentile of the bootstrap values.

Table A4. Non-Certified Mass Fraction Values for Selected Benzene and Furfural Metabolites in SRM 3672a

Analyte	Abbreviation	Mass Fraction ng/g[a]
t,t-Muconic acid [b,c]	MUCA	114.9 ± 2.6
N-Acetyl-S-(phenyl)-L-cysteine [b,c]	PhMA	1.574 ± 0.053
N-2-Furoylglycine [b,c]	N2FG	17131 ± 526
5-Hydroxymethyl-2-furancarboxylic acid [b,c]	HMFA	4716 ± 235
5-Hydroxymethyl-2-furoylglycine [b,c]	HMFG	1136 ± 53

[a] These values are expressed as $x \pm 2u(x)$, where x is a mean value and $u(x)$ is its associated standard uncertainty. While the best estimate of measurand mass fraction lies within the interval $x \pm 2u(x)$, neither the purity nor the identity of the calibrants used have been determined by NIST. For purposes of harmonization and process control, the imprecision and homogeneity components of uncertainty can be propagated as $u(x)/x$ relative standard deviations.

[b] CDC LC-MS/MS analysis; data were converted from ng/mL using the density of the materials which was measured to be 1.0052 g/mL ± 0.0001 g/mL.

[c] The non-certified value is the arithmetic mean from one procedure. The standard uncertainty of the mean is the standard deviation divided by the square root of the number of replications. The expanded uncertainty is estimated using the Student's t 0.95 confidence level for n-1 degrees of freedom as the coverage factor.

Table A5. Non-Certified Mass Fraction Values for Selected Mercapturic Acid and Related Biomarkers in SRM 3672a

Analyte	Abbreviation	Mass Fraction ng/g[a]
N-Acetyl-S-(2-carbamoylethyl)-L-cysteine[b,c]	2CaEMA	87.36 ± 2.22
N-Acetyl-S-(N-methylcarbamoyl)-L-cysteine[b,c]	MCaMA	279.9 ± 21.4
N-Acetyl-S-(benzyl)-L-cysteine[b,c]	BzMA	11.28 ± 0.37
N-Acetyl-S-(n-propyl)-L-cysteine[b,c]	1PMA	8.55 ± 0.68
N-Acetyl-S-(2-carboxyethyl)-L-cysteine[b,c]	2CoEMA	194.53 ± 2.54
N-Acetyl-S-(2-cyanoethyl)-L-cysteine[b,c]	2CyEMA	166.67 ± 1.72
N-Acetyl-S-(2-carbamoyl-2-hydroxyethyl)-L-cysteine[b,c]	2CaHEMA	11.00 ± 0.77
N-Acetyl-S-(2-hydroxyethyl)-L-cysteine a[b,c]	2HEMA	3.116 ± 0.141
N-Acetyl-S-(2-hydroxypropyl)-L-cysteine[b,c]	2HPMA	61.38 ± 1.68
N-Acetyl-S-(3-hydroxypropyl)-L-cysteine[b,c]	3HPMA	1126.0 ± 15.4
N-Acetyl-S-(3-hydroxypropyl-1-methyl)-L-cysteine[b,c]	3HMPMA	958.0 ± 16.7
N-Acetyl-S-(1 PHEnyl-2-hydroxyethyl)-L-cysteine[b,c] N-Acetyl-S-(2 PHEnyl-2-hydroxyethyl)-L-cysteine[b,c]	2HPhEMA	2.609 ± 0.098
2-Methylhippuric acid[b,c]	2MHA	83.9 ± 4.7
3-Methylhippuric acid + 4-Methylhippuric acid[b,c]	3MHA+4MHA	412.8 ± 5.1
Mandelic acid[b,c]	MADA	258.4 ± 15.2
Phenylglyoxylic acid[b,c]	PhGA	196.2 ± 6.9
2-Aminothiazoline-4-carboxylic acid[b,c]	2ATCA	83.3 ± 11.8
2-Thioxothiazolidine-4-carboxylic acid[b,c]	TTCA	33.25 ± 1.84

[a] These values are expressed as $x \pm 2u(x)$, where x is a mean value and $u(x)$ is its associated standard uncertainty. While the best estimate of measurand mass fraction lies within the interval $x \pm 2u(x)$, neither the purity nor the identity of the calibrants used have been determined by NIST. For purposes of harmonization and process control, the imprecision and homogeneity components of uncertainty can be propagated as $u(x)/x$ relative standard deviations.

[b] CDC LC-MS/MS analysis; data were converted from ng/mL using the density of the materials which was measured to be 1.0052 g/mL ± 0.0001 g/mL.

[c] The non-certified value is the arithmetic mean from one procedure. The standard uncertainty of the mean is the standard deviation divided by the square root of the number of replications. The expanded uncertainty is estimated using the Student's t 0.95 confidence level for n-1 degrees of freedom as the coverage factor.

Table A6. Non-Certified Mass Fraction Values for Selected Phenolic Metabolites in SRM 3672a

Analyte	Abbreviation	Mass Fraction ng/g[a]
2,4-Dichlorophenol[b,c]	2,4-DCP	14.34 ± 0.52
2,5-Dichlorophenol[b,c]	2,5-DCP	582.9 ± 23.5
Methyl Paraben[b,c]	M-PB	24.00 ± 1.03
Ethyl Paraben[b,c]	E-PB	7.98 ± 0.37
Propyl Paraben[b,c]	P-PB	3.947 ± 0.183
Benzophenone-3[b,c]	BP-3	6.83 ± 0.37
Bisphenol F[b,c]	BPF	6.70 ± 0.35
Bisphenol A[b,c]	BPA	1.028 ± 0.054
Bisphenol S[b,c]	BPS	1.111 ± 0.043

[a] These values are expressed as $x \pm 2u(x)$, where x is a mean value and $u(x)$ is its associated standard uncertainty. While the best estimate of measurand mass fraction lies within the interval $x \pm 2u(x)$, neither the purity nor the identity of the calibrants used have been determined by NIST. For purposes of harmonization and process control, the imprecision and homogeneity components of uncertainty can be propagated as $u(x)/x$ relative standard deviations.

[b] CDC LC-MS/MS analysis; data were converted from ng/mL using the density of the materials which was measured to be 1.0052 g/mL ± 0.0001 g/mL.

[c] The non-certified value is the arithmetic mean from one procedure. The standard uncertainty of the mean is the standard deviation divided by the square root of the number of replications. The expanded uncertainty is estimated using the Student's t 0.95 confidence level for n-1 degrees of freedom as the coverage factor.

Table A7. Non-Certified Mass Fraction Values for Nicotine and Smoking Related Metabolites in SRM 3672a

Analyte	Abbreviation	Mass Fraction ng/g[a]		
Total (R,S)-Nornicotine[b,c]	NNCT-total	62.01	±	1.38
Free (R,S)-Nornicotine[b,c]	NNCF-free	37.46	±	0.77
Total (-)-Nicotine[b,c]	NICT-total	1303	±	26
Free (-)-Nicotine[b,d]	NICF-free	984	±	12
Total (1'S,2'S)-nicotine N'-oxide [b,c]	NOXT-total	284.7	±	3.8
Free (1'S,2'S)-nicotine N'-oxide [b,c]	NOXF-free	258.0	±	5.7
Total N′-nitrosonornicotine[b,c]	NNNT-total	0.01458	±	0.00027
Free N′-nitrosonornicotine[b,c]	NNNF-free	0.00718	±	0.00027
Total (-)-Cotinine[b,c]	COTT-total	1454.4	±	24.2
Free (-)-Cotinine[b,d]	COTF-free	667	±	56
Total (S)-Cotinine-N-oxide[b,c]	COXT-total	157.05	±	1.80
Free (S)-Cotinine-N-oxide[b,c]	COXF-free	142.62	±	1.71
Total (-)-trans-3'-Hydroxycotinine [b,c]	HCTT-total	2746	±	45
Free (-)-trans-3'-Hydroxycotinine[b,d]	HCTF-free	1809	±	64
Total (R,S)Anabasine[b,c]	ANBT-total	8.801	±	0.164
Free (R,S)Anabasine[b,d]	ANBF-free	6.66	±	0.24
Total N′-nitrosoanabasine[b,c]	NABT-total	0.02211	±	0.00056
Total (R,S)-Anatabine[b,c]	ANTT-total	16.65	±	0.29
Free (R,S)-Anatabine[b,d]	ANTF-free	9.97	±	0.41
Total N′-nitrosoanatabine[b,c]	NATT-total	0.14594	±	0.00144
Free N′-nitrosoanatabine[b,c]	NATF-free	0.00934	±	0.00050
Total 4-hydroxy-4-(3-pyridyl)-butanoic acid [b,c]	HPBT-total	428.9	±	5.7
Free 4-hydroxy-4-(3-pyridyl)-butanoic acid [b,c]	HPBF-free	338.0	±	9.3
Total 4-(Methylnitrosamino)-1-(3 Pyridyl)-1-butanol[b,c]	NNAL-total	0.2675	±	0.0047
Free 4-(Methylnitrosamino)-1-(3 Pyridyl)-1-butanol[b,c]	NNALF-free	0.07431	±	0.00071
Total (-)-menthol β-D-glucuronide[b,c]	MEG	2103	±	57
Total 8-8-iso-prostaglandin $F_{2\alpha}$ [b,c]	8PGFT-total	0.2711	±	0.0041
Free 8-8-iso-prostaglandin $F_{2\alpha}$ [b,c]	8PGFF-free	0.1784	±	0.0033

[a] These values are expressed as $x \pm 2u(x)$, where x is a mean value and $u(x)$ is its associated standard uncertainty. While the best estimate of measurand mass fraction lies within the interval $x \pm 2u(x)$, neither the purity nor the identity of the calibrants used have been determined by NIST. For purposes of harmonization and process control, the imprecision and homogeneity components of uncertainty can be propagated as $u(x)/x$ relative standard deviations.

[b] CDC LC-MS/MS analysis; data were converted from ng/mL using the density of the materials which was measured to be 1.0052 g/mL ± 0.0001 g/mL.

[c] The non-certified value is the arithmetic mean from one procedure. The standard uncertainty of the mean is the standard deviation divided by the square root of the number of replications. The expanded uncertainty is estimated using the Student's t 0.95 confidence level for n-1 degrees of freedom as the coverage factor.

[d] The non-certified value is the DerSimonian-Laird consensus estimator of μ, $\bar{y}_{DL}$ [3] from two methods. The standard and approximate 95 % level of confidence expanded uncertainties were estimated using the bootstrap method [4]. The standard uncertainty is estimated as the standard deviation of the bootstrap values. A symmetrical expanded uncertainty is estimated as the half-width of the interval from the 2.5th percentile to the 97.5th percentile of the bootstrap values.

Table A8. Non-Certified Mass Fraction Values for Thiocyanate in SRM 3672a

Analyte	Abbreviation	Mass Fraction ng/g[a]
Thiocyanate[b,c]	SCN	4348 ± 79

[a] These values are expressed as $x \pm 2u(x)$, where x is a mean value and $u(x)$ is its associated standard uncertainty. While the best estimate of measurand mass fraction lies within the interval $x \pm 2u(x)$, neither the purity nor the identity of the calibrants used have been determined by NIST. For purposes of harmonization and process control, the imprecision and homogeneity components of uncertainty can be propagated as $u(x)/x$ relative standard deviations.

[b] CDC LC-MS/MS analysis; data were converted from ng/mL using the density of the materials which was measured to be 1.0052 g/mL ± 0.0001 g/mL

[c] The non-certified value is the arithmetic mean from one procedure. The standard uncertainty of the mean is the standard deviation divided by the square root of the number of replications. The expanded uncertainty is estimated using the Student's t 0.95 confidence level for n–1 degrees of freedom as the coverage factor.

Table A9. Non-Certified Mass Fraction Values for Organophosphate Ester Metabolites and Dialkylphosphates in Urine in SRM 3672a

Analyte	Abbreviation	Mass Fraction ng/g[a]
Bis(1,3-dichloro-2propyl) phosphate[b,c]	BDCIPP	1.778 ± 0.150
Diethylphosphate[b,c]	DEP	0.812 ± 0.124
Dimethyldithiophosphate[b,c]	DMDTP	0.1174 ± 0.0157
Dimethylphosphate[b,c]	DMP	1.083 ± 0.156
Dimethylthiophosphate[b,c]	DMTP	0.736 ± 0.084
Diethylthiophosphate[b,c]	DETP	0.194 ± 0.022

[a] These values are expressed as $x \pm 2u(x)$, where x is a mean value and $u(x)$ is its associated standard uncertainty. While the best estimate of measurand mass fraction lies within the interval $x \pm 2u(x)$, neither the purity nor the identity of the calibrants used have been determined by NIST. For purposes of harmonization and process control, the imprecision and homogeneity components of uncertainty can be propagated as $u(x)/x$ relative standard deviations.

[b] CDC LC-MS/MS analysis; data were converted from ng/mL using the density of the materials which was measured to be 1.0052 ± 0.0001 g/mL.

[c] The non-certified value is the arithmetic mean from one procedure. The standard uncertainty of the mean is the standard deviation divided by the square root of the number of replications. The expanded uncertainty is estimated using the Student's t 0.95 confidence level for n–1 degrees of freedom as the coverage factor.

Table A10. Non-Certified Mass Fraction Values for Glyphosate in Urine in SRM 3672a

Analyte	Abbreviation	Mass Fraction ng/g[a]
Glyphosate[b,c]	GLYP	0.2658 ± 0.0078

[a] These values are expressed as $x \pm 2u(x)$, where x is a mean value and $u(x)$ is its associated standard uncertainty. While the best estimate of measurand mass fraction lies within the interval $x \pm 2u(x)$, neither the purity nor the identity of the calibrants used have been determined by NIST. For purposes of harmonization and process control, the imprecision and homogeneity components of uncertainty can be propagated as $u(x)/x$ relative standard deviations.

[b] CDC LC-MS/MS analysis; data were converted from ng/mL using the density of the materials which was measured to be 1.0052 ± 0.0001 g/mL.

[c] The non-certified value is the arithmetic mean from one procedure. The standard uncertainty of the mean is the standard deviation divided by the square root of the number of replications. The expanded uncertainty is estimated using the Student's t 0.95 confidence level for n–1 degrees of freedom as the coverage factor.

Table A11. Non-Certified Mass Fraction Values for Neonicotinoid and N,N-diethyl-meta-toluamide (DEET) Metabolites in Urine in SRM 3672a

Analyte	Abbreviation	Mass Fraction ng/g[(a)]
3-(ethylcarbamoyl)benzoic acid	ECBA	16.29 ± 0.53
3-(diethylcarbamoyl)benzoic acid	DCBA	57.5 ± 2.8
5-Hydroxyimidacloprid	5-OH-IMI	0.856 ± 0.047
Acetamiprid-N-desmethyl	NDAC	0.1197 ± 0.0040

[(a)] These values are expressed as $x \pm 2u(x)$, where x is a mean value and $u(x)$ is its associated standard uncertainty. While the best estimate of measurand mass fraction lies within the interval $x \pm 2u(x)$, neither the purity nor the identity of the calibrants used have been determined by NIST. For purposes of harmonization and process control, the imprecision and homogeneity components of uncertainty can be propagated as $u(x)/x$ relative standard deviations.

[(b)] CDC LC-MS/MS analysis; data were converted from ng/mL using the density of the materials which was measured to be 1.0052 ± 0.0001 g/mL.

[(c)] The non-certified value is the arithmetic mean from one procedure. The standard uncertainty of the mean is the standard deviation divided by the square root of the number of replications. The expanded uncertainty is estimated using the Student's t 0.95 confidence level for n–1 degrees of freedom as the coverage factor.

Table A12. Non-Certified Mass Fraction Values for Selected Nonpersistent Pesticide Metabolites in Urine in SRM 3672a

Analyte	Abbreviation	Mass Fraction ng/g[(a)]
2,4-dichlorophenoxyacetic acid	2,4-D	0.2355 ± 0.0143
3-phenoxybenzoic acid	3-PBA	0.680 ± 0.059
para-nitrophenol	PNP	0.609 ± 0.027
trans-3-(2,2-dichlorovinyl)-2,2-dimethylcyclopropane carboxylic acid	trans-DCCA	0.831 ± 0.070

[(a)] These values are expressed as $x \pm 2u(x)$, where x is a mean value and $u(x)$ is its associated standard uncertainty. While the best estimate of measurand mass fraction lies within the interval $x \pm 2u(x)$, neither the purity nor the identity of the calibrants used have been determined by NIST. For purposes of harmonization and process control, the imprecision and homogeneity components of uncertainty can be propagated as $u(x)/x$ relative standard deviations.

[(b)] CDC LC-MS/MS analysis; data were converted from ng/mL using the density of the materials which was measured to be 1.0052 ± 0.0001 g/mL.

[(c)] The non-certified value is the arithmetic mean from one procedure. The standard uncertainty of the mean is the standard deviation divided by the square root of the number of replications. The expanded uncertainty is estimated using the Student's t 0.95 confidence level for n-1 degrees of freedom as the coverage factor.

Period of Validity: The non-certified values delivered by **SRM 3672a** are valid within the measurement uncertainty specified until **01 March 2040**. The non-certified values are nullified if the material is stored or used improperly, damaged, contaminated, or otherwise modified.

Maintenance of Non-Certified Values: NIST will monitor this material to the end of its period of validity. If substantive technical changes occur that affect the non-certified values during this period, NIST will update this certificate and notify registered users. Before making use of any of the values delivered by this material, users should verify they have the most recent version of this documentation, available through the NIST SRM website (https://www.nist.gov/srm).

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APPENDIX B

Source and Preparation: The SRM is a pool of human urine from more than 10 donors, over 18 years old who were not pregnant at the time of donation. In order for an individual to donate, they must smoke a minimum of one pack of cigarettes per day. Cigars, pipes, or other smoking methods are not prohibited, but cigarette requirements must be met. Donors were tested and found to be hepatitis B surface antigen negative, HBV NAT negative, HIV 1 and 2 antibody negative, HIV NAT negative, HCV antibody negative, syphilis negative, West Nile virus negative, zika virus RNA negative, and *T. cruzi* negative. Sterility testing was completed on the full lots post filtration at 0.2 µm to test for presence of infectious bacterial organisms. Urine was stored at 4 °C until homogenization in a 20 L carboy on top of a stirring plate. The pool was continually mixed with a stir bar and aliquoted into sterile amber glass vials with a cleaned and primed Hamilton diluter dispenser. Vials are capped with a butyl stopper and aluminum tear-away crimp cap and frozen at –20 °C until shipped to NIST.

Analysis: Value assignment of the mass fractions of analytes in SRM 3672a were based on single methods or the combination of measurements made by NIST using liquid chromatography with tandem mass spectrometry (LC-MS/MS) or gas chromatography tandem mass spectrometry (GC-MS/MS), and/or by CDC using LCMS/MS. -Methods are described in NIST.SP.260-256r1.

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