

Presentation of the implementation of use of
reference materials in an application – Arsenic by ICP-MS

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Laboratory for
industry and
power plant
chemistry



Environmental
laboratory



REFERENCE MATERIALS

RM

Used in method:

validation,
calibration,
estimation of measurement uncertainty,
training,
internal quality control,
proficiency testing



Validity of measurement:

according customer requirements,
with validated methods and equipments,
qualified and competent staff,
traceability and measurement uncertainty,
proficiency testing,
QC and QA procedures,
accreditation



Types of reference materials:

RMS support measurements of:

chemical composition,
biological,
clinical,
physical,
engineering,
sensory properties



Pure substances: purity / impurities

Standard solutions, gas mixtures: prepared gravimetrically, used for calibration

Matrix reference materials: major, minor, trace chemical constituents, real matrices, synthetic mixtures

Physico-chemical reference materials: density, viscosity, melting point etc

Other:

CRM

RM



RM's FOR ELEMENT ANALYSIS

10580 ICP Multi Element Standard VI
11355 ICP Multi Element Standard IV

Perkin Elmer N812-2038
Perkin Elmer N812-2014
QC PLUS⁺ Quality Control,
VKI, QC Municipal Sludge A

Custom Grade Multielement Solution
CCS-1, Inorganic Ventures

Custom Grade Multielement Solution
CCS-2, Inorganic Ventures

AccuTraceTM Reference Standard

Baker Instra Analyzed Arsenic Atomic Absorption Standard



Reference materials	Type	Reference materials	Type
ALL-MO,	ALL-MO reference water	GBW 08628b	simulated rain water
ALLARD,	reference water	GRM-02	rain water from Southern Ontario
ALLARD,	reference water	ION-20	water
ANT-B 2778,	coal	LGC 6176	
AR 1517,	cement AR1517	QC- PLUS+ QCP-MTL	water, heavy metals
AR 2758,	coal	QC WW4, TOC	VKI water
AR 5170,	cement AR 5170	VAPO/JPT,	peat
ASSO 3193-85	dolomitized limestone	VAPO/SSLT,	ash
BCR 38,	ash	VTT 100 005/00	wood-peat ash
BCR 40,	coal	ÄMPÄRI slag	bottom slag
DP 1016,	paper	ÄMPÄRI, Partek 152	lime stone
DP 704,	paper, aged	ÄMPÄRI, VNJ 009510	REF
DP n. 700	old paper	ÄMPÄRI, ash	coal fly ash
GYP-A,	gypsum	ÄMPÄRI,	coal
IAEA V-10,	hay	ÄMPÄRI, wood	saw dust
ICMIX2-100,	water	ÄMPÄRI, cement	
KALKSTEIN II 8-34	lime stone	ÄMPÄRI, peat ash	peat fly ash
LIETE, L1-6/98	sludge	ÄMPÄRI, peat	peat
MAA, M1-6/98	soil	ÄMPÄRI, peat	VAPO peat
NBS 1648,	dust	T02-218-07	REF
NIST 1632b,	coal	NBS 1632a,	NBS bituminous coal
NIST 1633a,	ash	QCP-MTL, 03/2000	water, heavy metals
NIST1632c,	coal		
OLSO,	saline reference water		
OLSO, laboratorio	saline reference water		



RM FOR ARSENIC ANALYSIS

ICP Multi Element Standard VI CertiPUR LOT. OC316171
As (Arsenic) 100 +/- 5 mg/l; NIST Standard Reference
Material SRM 3103a Batch Code 891003




CALIBRATION STANDARD FOR ARSENIC ANALYSIS

Arsenic (III) Oxide, Purity 99.999%, Starting Material Lot No. R999A, Matrix 2 % HNO₃, Certified Value 1000.0 mg/l, Standard Uncertainty +/- 0.2 %, Confirmation by ICP vs. NIST SRM 3103a



ARSENIC BY ICP-MS VALIDATION DATA

Selectivity:	 $(b_{\text{SAM}}/b_{\text{CAL}}) < 2,5 \%$	SAM = Standard addition on a real sample CAL = Calibration curve of standards b = Regression coefficient ($y = bx + a$)
Accuracy:	97 %	Mean recovery from real samples spiked with the analyte standard (D-chart) or the mean of the RM
Repeatability:	1,7 %	Relative standard deviation of replicates
Laboratory reproducibility:	7 % (water) 5 % (soil)	Relative standard deviation of the recovery Medium time scale
Measurement uncertainty:	20 % (water) 15 % (soil)	$U = k * (u_{\text{bias}}^2 + u_{\text{lab. reprod.}}^2 + u_{\text{RM}}^2)^{1/2}$ k = 2
Detection limit:	0,01 ug/l	$3 * SD_{\text{bl}} / b$
Determination limit:	0,02 ug/l	$6 * SD_{\text{bl}} / b$
Linearity:	$R^2 = 999984$	Multiple correlation coefficient
Measuring range:	0,1 – 500 ug/l	Range covered by standards
Sensitivity:	3200 c/s/ug/l	b from calibration curve

