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Level IV Data Package

MWH Group 239631

Method: EPA 7196: CRVI7196

2805060291
2805060294

SM 7198A QC Check List

Hexavalent Chromium

Cr VI

Analyst ALZS

Analysis date 5/6/08

Instrument: HACH DR/4000V

Review date 5/14/08

No All Samples analyzed within 24 Hr hold time

All Samples within range of calibration curve, or diluted to within range

QC

CCB/Blank < 1/2 MRL

MRL within +/- 50%

All other calibration points within +/- 10%

LCS/LCSD recovery within +/- 15%

MS/MSD recovery within +/- 30%

MS/MSD RPD is within +/- 20%

CCV within +/- 10%

The pH of the samples are 7

No more than 20 samples per batch

MS is run at a frequency of 1 every 10 samples and MSD is run at frequency of 1 every 20.

No QIR needed for failed QC

No Special Det Code noted on the cover sheet

WB mm 5/14/08

Instrument: Hach DR 4000			Cr-VI by EPA Method 7196A								
Analyst: AZS			Date: 5/6/2008		Start time: 4:52 PM		End time: 5:00 PM				
Cal. Stock Std: Use Built in Curve			Cal. Stock Std exp. Date: 6/4/08		Cal. Working Std: AZS080404-1						
			LCS Stock Std exp. Date: 6/4/08		LCS Working Std: AZS080404-2						
Correlation Coeff: _____			Slope: _____		Y-Intercept: _____		LCS True Value: 0.05 ppm				
Sample	I.D.	Dilution		Turb Bik			Calc.	Reported	Sampling	pH	Comments
		sample	total	(same dilution)							
		mls	mls	abs	abs	res abs	mg/L	mg/L	date		
Blank		10	10	0.000			0.000	0.000			
Std 1 (0.005 ppm)		10	10	0.012			0.006	0.006			120% Limit: 50 - 150%
Std 2 (0.020 ppm)		10	10	0.040			0.022	0.022			110% Limit: 90 - 110%
Std 3 (0.050 ppm)		10	10	0.098			0.053	0.053			102% Limit: 90 - 110%
Std 4 (0.200 ppm)		10	10	0.385			0.210	0.210			105% Limit: 90 - 110%
Std 5 (0.500 ppm)		10	10	0.927			0.506	0.506			101% Limit: 90 - 110%
LCS - 1		10	10	0.105			0.056	0.056			112% Limit: 85 - 115%
1	FB-1	10	10	0.004			0.002	0.002	5/5/8	7	HOLD
	MS	10	10	0.104			0.057	0.057			112% Limit: 70 - 130%
	MSD	10	10	0.103			0.056	0.056			108% Limit: 70 - 130%
2	2805060291 MD-1	2	10	0.315			0.172	0.860			
3	2805060291 M-44	2	10	0.319			0.174	0.870			
4			10								
5			10								
6			10								
7			10								
	0.020 ppm/CCV	10	10								Limit: 90-110%
	Blank/CCB	10	10								
8			10								
9			10								
10			10								
	MS		10								Limit: 70 - 130%
11			10								
12			10								
13			10								
14			10								
15			10								
16			10								
	0.020 ppm/CCV	10	10								Limit: 90-110%
	Blank/CCB	10	10								
17			10								
18			10								
19			10								
20			10								
	LCS - 2	10	10	0.101			0.055	0.055			110% Limit: 85 - 115%
	0.020 ppm/CCV	10	10	0.040			0.022	0.022			110% Limit: 90 - 110%
	Blank/CCB	10	10	0.002			0.001	0.001			

Analytical Reviewed By:

QC Reviewed by:

Reagent: HACH Perma Chem Chroma Ver3

Entered By:

EXP Date: 8/10

Lot # A7324

**Standard
Preparation
Worksheet
&
Certificate of
Analysis**

Reagent Preparation Documentation

Reagent: Cr VI std 5 ppm
 Date Received/Prepped: 4/4/81 5/15/81 6/6/81 1 1
 Date Expired: 6/4/81 5/16/81 8/6/81 1 1
 Manufacturer: _____
 Storage Condition: _____

MW #: AZ5080404-1
 By: AZS
 Matrix: 1
 Amount: 200 ml
 Lot #: R201090

Component	Comment	Standard	Concentration
	1ml 1000 ppm std		
	into 200 ml DI		
	add 20 drops H ₂ SO ₄		

Comment: _____

Reagent: Cr VI QC 5 ppm
 Date Received/Prepped: 4/4/81 1 1 1 1
 Date Expired: 6/4/81 1 1 1 1
 Manufacturer: _____
 Storage Condition: _____

MW #: AZ5080404-2
 By: AZS
 Matrix: _____
 Amount: 200 ml
 Lot #: R201632

Component	Comment	Standard	Concentration
	1ml 1000 ppm std		
	into 200 ml DI		
	pres with 20 drops H ₂ SO ₄		

Comment: _____

Reagent: 100 ppb Hg 1 source
 Date Received/Prepped: 4/5/81 1 1 1
 Date Expired: 6/3/81 1 1 1
 Manufacturer: _____
 Storage Condition: _____

MW #: AZ5080404
 By: AZS
 Matrix: 1
 Amount: 100 ml
 Lot #: ME0804003

Component	Comment	Standard	Concentration
	0.5 ml 20 ppb std		
	100ml DI acidified with HNO ₃		

5

Comment: _____

HACH COMPANY



An ISO 9001 Certified Company

P.O.Box 389
Loveland, CO 80539
(970) 669-3050

Certificate of Analysis

Page 1

COMMODITY: Chromium Reference Standard Solution 1000 | 10 mg/L as total Cr
COMMODITY NUMBER: 14664-42 MANUFACTURE DATE: DATE OF ANALYSIS:
LOT NUMBER: A5005 12/31/2004 1/4/2005

<i>TEST</i>	<i>SPECIFICATIONS</i>	<i>RESULTS</i>
Hexavalent Chromium Concentration	995 to 1005 ppm	1001.0 ppm
pH of the solution	12 to 14	12.0

The expiration date is Jan 2010

The item 1466442 is traceable to NIST standards SRM 136e Potassium Dichromate LOT N/A.

R 201090

Certified by _____

A handwritten signature in black ink, appearing to read "Paul Kleinwolterink".

Paul Kleinwolterink
Analytical Services Chemist



195 Lehigh Avenue, Suite 4
 Lakewood, New Jersey 08701 - USA
 inorganicventures.com

CERTIFICATE OF ANALYSIS

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1.0 INORGANIC VENTURES is an ISO Guide 34:2000 registered Certified Reference Material (CRM) Manufacturer (Certificate #883-02). The certificate is designed and the data is determined in accordance with ISO Guide 31:2000 (Reference Materials-Contents of Certificates and Labels), ISO Guide 34:2000 "Quality System Guidelines for the Production of Reference Materials," and ISO Guide 35:1989 "Certification of Reference Materials - General and Statistical Principles."

2.0 DESCRIPTION OF CRM **1000 µg/mL Chromium (+6) in H2O**

R# 201632

Catalog Number: CGCR(6)1-1, CGCR(6)1-2, and CGCR(6)1-5
 Lot Number: **Z-CR02152**
 Starting Material: (NH4)2Cr2O7
 Starting Material Purity (%): 99.989259
 Starting Material Lot No: F04N14
 Matrix: H2O

3.0 CERTIFIED VALUES AND UNCERTAINTIES

Certified Concentration: 1000 ± 3 µg/mL

Certified Density: 0.999 g/mL (measured at 22° C)

The Certified Value is based upon the most precise method used to analyze this CRM. The following equations are used in the calculation of the certified value and the uncertainty:

$\text{Certified Value } (\bar{x}) = \frac{\sum x_i}{n}$	$\bar{x} = \text{mean}$
	$x_i = \text{individual results}$
	$n = \text{number of measurements}$
$\text{Uncertainty } (\pm) = \frac{2[(\sum s_i)^2]^{1/2}}{(n)^{1/2}}$	$\sum s_i = \text{The summation of all significant estimated errors}$ (Most common are the errors from instrumental measurement, weighing, dilution to volume, and the fixed error reported on the NIST SRM certificate of analysis.)

The independent samples t-test was used to determine if there is agreement between the above assay methods at the 95% confidence interval. Both methods were compared and showed agreement within the stated uncertainties. This agreement is a confirmation of the accuracy of this CRM.

4.0 TRACEABILITY TO NIST AND VALUES OBTAINED BY INDEPENDENT METHODS

· "Property of the result of a measurement or the value of a standard whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons all having stated uncertainties." (ISO VIM, 2nd ed., 1993, definition 6.10)

· This product is Traceable to NIST via an unbroken chain of comparisons. The uncertainties for each certified value are reported, taking into account the SRM uncertainty error and the measurement, weighing and volume dilution errors. In rare cases where no NIST SRMs are available, the term 'in-house std.' is specified.

4.1 Assay Method #1 **1000 ± 3 µg/mL**
 Redox NIST SRM 136e Lot Number: 980702

Assay Method #2 **1001 ± 4 µg/mL**
 ICP Assay NIST SRM 3112a Lot Number: 990607

- 4.2 **BALANCE CALIBRATION** - All balances are checked daily using an in-house procedure. The weights used for testing are annually compared to master weights and are traceable to the National Institute of Standards and Technology (NIST). The NIST Traceability numbers are 692476 - Class 1 and 692476A - Class 2. The NIST test number is 822/260017-98. All analytical balances are calibrated every 4 months. The balances are calibrated with a class 1 and/or class 2 analytical weight set. These weights are tested annually by a NIST / NVLAP accredited calibration lab. The NIST test number is 822/260017-98.
- 4.3 **THERMOMETER CALIBRATION** - The thermometers used in the determination of the final densities are calibrated vs standard thermometer No. 903-2680 which was certified in accordance with the procedures outlined by ASTM E77-87 and NIST Monograph 150 using NIST Test Nos. and Std Nos.: 769543, 217368/769543, 217368/P14452, 176240/P14452, 176240. Thermometers which are not calibrated vs standard thermometer No. 903-2680 are traceable to NIST Identification Nos. 92564, 119016, 471047 and NIST test report Nos. 811/258522, 811/2557078, and 236090.
- 4.4 **GLASSWARE CALIBRATION** - An in-house procedure is used to calibrate all Class A glassware used in the manufacturing and quality control of CRM's.

5.0 TRACE METALLIC IMPURITIES (TMI) DETERMINED BY ICP/MS AND ICP-OES IN µg/mL

CRM's solutions are tested for trace metallic impurities by Axial ICP-OES and ICP-MS. The result from the most sensitive method for each element, is reported below. Solutions tested by ICP-MS were analyzed in an ULPA-Filtered Clean Room. An ULPA-Filter is 99.9985% efficient for the removal of particles down to 0.3 µm.

<u>Q</u> Al < 0.00090	<u>M</u> Dy < 0.01185	<u>Q</u> Li < 0.00002	<u>M</u> Pr < 0.00059	<u>M</u> Te < 0.05923
<u>M</u> Sb < 0.00099	<u>M</u> Er < 0.00987	<u>M</u> Lu < 0.00079	<u>M</u> Re < 0.00197	<u>M</u> Tb < 0.00059
<u>M</u> As < 0.01974	<u>M</u> Eu < 0.00592	<u>Q</u> Mg < 0.00030	<u>M</u> Rh < 0.00197	<u>M</u> Tl < 0.00197
<u>M</u> Ba < 0.01974	<u>M</u> Gd < 0.00197	<u>M</u> Mn < 0.00790	<u>M</u> Rb < 0.00197	<u>M</u> Th < 0.00197
<u>M</u> Be < 0.00099	<u>M</u> Ga < 0.00197	<u>Q</u> Hg < 0.01500	<u>M</u> Ru < 0.00395	<u>M</u> Tm < 0.00079
<u>M</u> Bi < 0.00079	<u>M</u> Ge < 0.01185	<u>M</u> Mo < 0.00395	<u>M</u> Sm < 0.00197	<u>M</u> Sn < 0.00987
<u>Q</u> B < 0.01000	<u>M</u> Au < 0.00592	<u>M</u> Nd < 0.00395	<u>M</u> Sc < 0.01974	<u>Q</u> Ti < 0.00100
<u>M</u> Cd < 0.00592	<u>M</u> Hf < 0.00395	<u>M</u> Ni < 0.01579	<u>M</u> Se < 0.01579	<u>M</u> W < 0.01974
<u>Q</u> Ca < 0.00027	<u>M</u> Ho < 0.00099	<u>M</u> Nb < 0.00099	<u>Q</u> Si < 0.20000	<u>M</u> U < 0.00395
<u>M</u> Ce < 0.00987	<u>M</u> In < 0.01974	<u>n</u> Os	<u>M</u> Ag < 0.00395	<u>Q</u> V < 0.02000
<u>M</u> Cs < 0.00059	<u>M</u> Ir < 0.00987	<u>M</u> Pd < 0.00987	<u>Q</u> Na < 0.00300	<u>M</u> Yb < 0.00197
<u>s</u> Cr	<u>Q</u> Fe < 0.01000	<u>Q</u> P < 0.04000	<u>M</u> Sr < 0.00099	<u>M</u> Y < 0.07897
<u>M</u> Co < 0.00592	<u>M</u> La < 0.00099	<u>M</u> Pt < 0.00395	<u>i</u> S	<u>Q</u> Zn < 0.00400
<u>M</u> Cu < 0.01185	<u>M</u> Pb < 0.00592	<u>Q</u> K < 0.32485	<u>M</u> Ta < 0.01382	<u>M</u> Zr < 0.00987

M - Checked by ICP-MS Q - Checked by ICP-OES i - Spectral Interference n - Not Checked For s - Solution Standard Element

6.0 INTENDED USE

- For the calibration of analytical instruments including but not limited to the following: ICP-MS, ICP-OES, FAAS, GFAA, XRF, and DCP
- For the validation of analytical methods
- For the preparation of "working reference samples"
- For interference studies and the determination of correction coefficients
- For detection limit and linearity studies
- For additional intended uses, contact Technical Staff

7.0 INSTRUCTIONS FOR THE CORRECT USE OF THIS REFERENCE MATERIAL

Storage & Handling - Keep tightly sealed when not in use. Store and use at $20 \pm 4^\circ\text{C}$. Do not pipet from container. Do not return portions removed for pipetting to container.

Atomic Weight; Valence; Coordination Number; Chemical Form in Solution - 51.9961; +3, 6; $\text{Cr}_2\text{O}_7^{2-}$

Chemical Compatibility - Stable in HCl , HNO_3 , H_2SO_4 , HF , H_3PO_4 . Avoid basic media. Stable with most metals (Except Ba and Pb) and inorganic anions in acidic media.

Stability - 2-100 ppb levels stable for months in 1% HNO_3 / LDPE container as *Total Cr*, however, the stability of this oxidation state is unknown. 1-10,000 ppm solutions chemically stable for years in 0 - 1% HNO_3 / LDPE container.

Cr Containing Samples (Preparation and Solution) - Metal (soluble in HCl); Oxides/Ores (Chrome ore/oxides are very difficult to dissolve. The following procedures [A-D] are commonly used:

A. Fusion with KHSO_4 and extraction with hot KCl . The residue fused with Na_2CO_3 and KClO_3 , 3:1.

B. Fusion with Na_2SO_4 and NaF , 2:1.

C. Fusion with magnesia or lime and sodium or potassium carbonates, 4:1.

D. Fusion with Na_2O_2 or NaOH and KNO_3 or NaOH and Na_2O_2 .

Nickel, iron, copper, or silver crucibles should be used for D. Platinum may be used for A, B and C).

Organic Matrices (Ash at 450EC followed by one of the fusion methods above or sulfuric/hydrogen peroxide acid digestions may be applicable to non oxide containing samples).

Atomic Spectroscopic Information (ICP-OES D.L.s are given as radial/axial view):

Technique/Line	Estimated D.L.	Order	Type	Interferences (underlined indicates severe)
ICP-OES 205.552 nm	0.006 / 0.0008 $\mu\text{g}/\text{mL}$	1	ion	<u>Os</u>
ICP-OES 284.325 nm	0.008 / 0.0007 $\mu\text{g}/\text{mL}$	1	ion	
ICP-OES 276.654 nm	0.01 / 0.001 $\mu\text{g}/\text{mL}$	1	ion	<u>Cu, Ta, V</u>
ICP-MS 52 amu	40 ppt	n/a	M+	36S16O, 36Ar16O - The 50Cr, 53Cr, 54Cr lines suffer from many more potential interferences from sulfur, chlorine and argon compounds of oxygen, nitrogen and carbon.

8.0 **HAZARDOUS INFORMATION** - Please refer to the enclosed Material Safety Data sheet for information regarding this CRM.

9.0 **HOMOGENEITY** - This solution was mixed according to an in house procedure and is guaranteed to be homogeneous.

10.0 QUALITY STANDARD DOCUMENTATION



10.1 ISO 9001:2000 Quality Management System Registration - QMI Certificate Number 010105

Recognized by:

Registrar Accreditation Board (ANSI-RAB)

Standards Council of Canada (SCC)

Dutch Council for Accreditation (RVA)

Entidad Mexicana de Acreditacion, a.c.(EMA)

Members of IQ Net International Certification Network:

Argentina (IRAM), Australia (QAS), Austria (ÖQS), Belgium (Avinter), Brazil (FCAV), Canada (QMI), Hong Kong (HKQAA), Columbia (ICONTEC), Czech Republic (CQS), Denmark (DS), Finland (SFS), France (AFAQ), Germany (DQS), Greece (ELOT), Hungary (MSZT), Ireland (NSAI), Israel (SII), Italy (CISQ), Japan (JQA), Korea (KSA-QA), Netherlands (KEMA), Norway (NCS), Poland(PCBC), Portugal (APCER), Singapore (PSB), Slovenia (SIQ), Spain (AENOR), Switzerland (SQS)

10.2 ISO/IEC 17025 - 1999 "General Requirements for the Competence of Testing and Calibration"

- Chemical Testing - Accredited A2LA Certificate Number 883.01

10.3 ISO/IEC Guide 34 - 2000 "General Requirements for the Competence of Reference Material Producers"

- Reference Materials Production - Accredited A2LA Certificate Number 883.02

A2LA Mutual Recognition Agreement Partners:

Australia (NATA), Austria (BmWA), Belgium (BELTEST) (BKO-OBE), Canada (SCC), Chinese Taipei (CNLA), Czech Republic (NAO), Denmark (DANAK), Finland (FINAS), France (COFRAC), Germany (DAR), Hong Kong (HKAS), Ireland (NAB), Italy (SIT) (SINAL), Japan (JAB) (JNLA), Republic of Korea (KOLAS), The Netherlands (RvA), New Zealand (IANZ), Norway (NA), Portugal (IPQ), Singapore (SAC-SINGLAS), Spain (ENAC), Sweden (SWEDAC), Switzerland (SAS), United Kingdom (UKAS) and United States (NVLAP) (ICBO ES)

10.4 10CFR50 Appendix B - Nuclear Regulatory Commission

- Domestic Licensing of Production and Utilization Facilities

10.5 10CFR21 - Nuclear Regulatory Commission - Reporting Defects and Non-Compliance

10.6 MIL-STD-45662A (Obsolete/Observed)

11.0 DATE OF CERTIFICATION AND PERIOD OF VALIDITY

11.1 Shelf Life - The period of time during which the concentration of the analyte(s) in a properly packaged, unopened, and unused standard stored under environmentally controlled and monitored conditions will remain within the specified uncertainty range. Shelf life is limited primarily by transpiration (loss of water from the solution) and infrequently, by chemical instability. Transpiration studies of chemically-stable solutions performed at the manufacturer's facility show a CRM shelf-life of twenty one months for solutions packaged in 125-mL low density polyethylene bottles. When stored under special conditions that minimize transpiration and instability, the shelf life can be extended past this limit.

11.2 Expiration Date - The date after which a CRM should not be used. Routine laboratory use of a CRM increases transpiration losses and the chance of contamination which affect the integrity of the CRM and limit its useful life. Manufacturer concurs with state and federal regulatory agencies' recommendations that solution standards be assigned a one-year expiration date.

Certification Date: December 06, 2006

Expiration Date:

EXPIRES
11/06/09

12.0 NAMES AND SIGNATURES OF CERTIFYING OFFICERS

Certificate Prepared By: Nick Maida, Product Documentation Administrator

Nick Maida

Certificate Approved By: Katalin Le, QC Manager

Katalin Le

Certifying Officer: Paul Gaines, PhD., Senior Technical Director

Paul Gaines