

ABSTRACT

The research project is about the method validation for the determination of hexavalent chromium in seawater and other matrices by colorimetric method. This method is validated by referring to the APHA 3500 B- Cr Colorimetric Method. The current colorimetric method of determination of hexavalent chromium in wastewater is carried out by using UV-VIS Spectroscopy. This method is developed to determine the trace level of hexavalent chromium up to a minimum 0.001 mg /L or 1 ppb of hexavalent chromium in seawater, river water and industrial effluent by using the Discrete Analyzer. The calibration range is within 0 to 0.25 mg Cr ⁶⁺/L or 250 ppb. In the method validation study, various concentration level of hexavalent chromium were spiked in various matrices where the recovery that were obtained within 88 % to 105 %. The instrument detection limit is 0.001 mg/L or 1 ppb. The measurement uncertainty is 0.05 ± 0.005 mg/L Cr ⁶⁺. The hexavalent chromium is determined colorimetrically by reaction with diphenylcarbazide in acid solution where the red-violet color of unknown composition will be produced. The absorbance is measured at 550 nm.

ABSTRAK

Projek penyelidikan ini dijalankan untuk menentukan kepekatan Kromium heksavalen (Cr^{6+}) dalam air laut dan matriks lain dengan menggunakan kaedah *Colorimetric*. Kaedah ini dijalankan dengan merujuk kepada APHA 3500 B-Cr *Colorimetric Method*. Kaedah ini menggunakan konsep *UV-VIS Spectroscopy* untuk menentukan kromium heksavalen dalam air permukaan dan effluen perindustrian. Untuk menganalisa kepekatan Kromium heksavalen yang rendah sehingga ke peringkat ppb ($\mu\text{g}/\text{L}$), instrumen baru *Westco Smartchem Discrete Analyzer* boleh digunakan untuk meningkatkan sensitiviti malah dapat menganalisis kepekatan yang lebih rendah dan lebih jimat kos dan masa. *Westco Smartchem Discrete Analyzer* dapat menentukan kepekatan Kromium heksavalen sehingga $0.001 \text{ mg}/\text{L}$ atau 1 ppb Kromium heksavalen dalam sampel air laut, air sungai dan effluen perindustrian. Julat untuk kepekatan bagi graf kalibrasi adalah antara 0 hingga $0.25 \text{ mg Cr}^{6+}/\text{L}$ atau 250 ppb. Dalam projek penyelidikan ini, pelbagai tahap kepekatan Kromium heksavalen telah dijalankan dalam pelbagai matriks di mana peratus purata yang diperolehi adalah di antara 88% hingga 105%. Had penentuan bagi kaedah ini adalah $0.001 \text{ mg}/\text{L}$ atau 1 ppb. Kromium Heksavalen ditentukan dengan menggunakan kaedah *Colorimetric* di mana sampel akan bertindak balas dengan diphenylcarbazide dalam keadaan asid di mana warna merah-ungu akan dihasilkan. Sebatian yang berwarna merah-ungu ini akan dikesan pada gelombang 550 nm.

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LIST OF ABBREVIATIONS

Abs	Absorbance
APHA	American Protection Health Association
Cr	Chromium
Cr 6 +	Hexavalent Chromium
CrO ₄ ²⁻	Chromate
Cr ₂ O ₇ ²⁻	Dichromate
CV	Coefficient of Variation
DIPH	Diphenylcarbazide
EPA	Environmental Protection Agency
EU	European Union
Fe ³⁺	Ferric Ion
FeOCr ₂ O ₃	Chromite
H ₂ SO ₄	Suphuric Acid
ISO	International Organization for Standardization
IUPAC	International Union of Pure and Applied Chemistry
LCL	Upper Control Limit
LCMRL	Lowest Concentration Minimum Reporting Limit
LWL	Lower Warning Limit
MDL	Method Detection Limit
mg/L	Miligrams Per Litre
MWQCS	Marine Water Quality Criteria and Standards
MU	Measurement Uncertainty

N	Normality
NaOH	Sodium Hydroxide
nm	Nanometre (Wavelength)
ppb	Part Per Billion
ppm	Part Per Million
QC	Quality Control
QMS	Quality Management System
R ²	Linear Regression
R _m	Method Recovery
R _s	Sample Recovery
RSD	Relative Standard Deviation
SD or s	Standard Deviation
SQC	Statistical Quality Control
UCL	Upper Control Limit
µg	Micrograms
µg/L	Micrograms Per Litre
UV–Vis	Ultraviolet and Visible
UWL	Upper Warning Limit
V	Voltage
W	Watt
WHO	World Health Organization