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StandardGuide for Analytical Testing of Substances of Very High Concern in Materials and Products[1]

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1. Scope

1.1 This guide contains a list of potential test methods for the analysis of Substances of Very High Concern (SVHC) as designated by the European Chemical Agency (ECHA), in October 2008. Substances of Very High Concern are defined in Article 57 of the European Union Regulation #1907/2006. Information on the test methods cited is publicly available and is drawn from a variety of sources. This guide is intended to assist in the selection of test methods that are applicable for the SVHCs identified.

1.2 The specific SVHCs covered within this guide are compiled from the ECHA Candidate List of Substances of Very High Concern. This list is also referred to as the REACH Candidate List.

1.3 This guide specifically addresses methods for the analysis of SVHCs in products. It is not intended to cover the many and varied analysis challenges associated in the manufacturing environment.

1.4 *Limitations:*

1.4.1 This guide is intended to provide a compilation of available test methods for the SVHCs listed on the ECHA Candidate list and is not intended to be exhaustive. The test methods within this guide are not the only ones available for any specific substances and this guide does not recommend any specific test method.

1.4.2 Test methods for specific substances at the detection limits required for REACH reporting are not always available. In some cases, it is necessary to deduce the quantity of substance present through the analysis and quantification of its elements. Although this approach is routinely used some degree of uncertainty exists in the final result due to the reduced specificity of the test method.

1.4.3 Although this guide is intended to be updated on a periodic basis to capture new developments in the field, there is no assurance that the information provided is the most current.

1.5 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1257 Specification for High-Gravity Glycerin

D1971 Practices for Digestion of Water Samples for Determination of Metals by Flame Atomic Absorption, Graphite Furnace Atomic Absorption, Plasma Emission Spectroscopy, or Plasma Mass Spectrometry

D3335 Test Method for Low Concentrations of Lead, Cadmium, and Cobalt in Paint by Atomic Absorption Spectroscopy

D4309 Practice for Sample Digestion Using Closed Vessel Microwave Heating Technique for the Determination of Total Metals in Water

D5831 Test Method for Screening Fuels in Soils

E1621 Guide for X-Ray Emission Spectrometric Analysis

F2576 Terminology Relating to Declarable Substances in Materials

2.2 *European Commission:*[3]

Article 57 of the European Union Regulation #1907/2006

2.3 *International Electrotechnical Commission TC111:*[4]

IEC 62321 Electrotechnical Products – Determination of Levels of Six Regulated Substances (Lead, Mercury, Cadmium, Hexavalent Chromium, Polybrominated Biphenyls, Polybrominated Diphenyl Ethers)

[1] This guide is under the jurisdiction of ASTM Committee F40 on Declarable Substances in Materials and is the direct responsibility of Subcommittee F40.02.02 on WK19940 on SVHCs.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Europese Commissie, B-1049, Brussels, Belgium, http://ec.europa.eu/index_en.htm.

[4] Available from International Electrotechnical Commission (IEC), 3, rue de Varembé, P.O. Box 131, CH-1211 Geneva 20, Switzerland, http://www.iec.ch.

2.4 *Joint Industry Guide (JIG):*[5]

JIG-101 Material Composition Declaration for Electrotechnical Products. Ed 4.0, 2011

3. Terminology

3.1 *Definitions:*

3.1.1 Terms and definitions related to declarable substances in materials may be found in Terminology F2576.

3.1.2 Terms and definitions in the guide not found in Terminology F2576 are found in a common dictionary or other reference documents such as the ASTM Dictionary of Engineering Science & Technology.[6]

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *article*—"an object which during production is given a special shape, surface or design which determines its function to a greater degree than its chemical composition" as defined in Article 3(3) of the REACH Regulation.

3.2.2 *brominated flame retardant*—a group of brominated organic compounds that are used to inhibit initiation of a fire.

3.2.3 *Joint Industry Guide*—An industry standard of the Electric Industries Alliance that "*establishes the relevant substances as well as reporting thresholds that the industry agrees should govern material content disclosures*."[5]

3.2.4 *phthalates—also known as phthalate esters*, are esters of phthalic acid primarily used as a plasticizer for polyvinyl chloride or as solvents for many different types of consumer products.

3.2.5 *plasticizer*—any of a group of substances used in plastics and other materials to control viscosity, flexibility or softness of the finished product.

3.2.6 *Substances of Very High Concern (SVHC)*—substances that have hazards of serious consequences and meet the criteria for carcinogenic, mutagenic and reproductive toxic substances of category 1 and 2.

3.2.6.1 *Discussion*—SVHC can be persistent, bioaccumulative and toxic (PBT) substances or very persistent and very bioaccumulative (VPvB) substances. Other substances giving rise to an equivalent level of concern as potential SVHC include endocrine disruptors.

3.3 *Acronyms:*

3.3.1 *AAS*—Atomic Absorption Spectrometry

3.3.2 *AES*—Atomic Emission Spectrometry

3.3.3 *AFS*—Atomic Fluorescence Spectometry

3.3.4 *BFR*—Brominated Flame Retardant

3.3.5 *CAS*—Chemical Abstract Services

3.3.6 *CMR*—Carcinogenic, Mutagenic and Toxic to Reproduction

3.3.7 *CPSC*—United States Consumer Product Safety Commission

3.3.8 *ECD*—Electron Capture Detection

3.3.9 *ECHA*—European Chemicals Agency

3.3.10 *ECNI*—Electron Capture Negative Ion

3.3.11 *EEE*—Electrical and Electronic Equipment

3.3.12 *EIA*—Electronic Industries Alliance

3.3.13 *EPA*—United States Environmental Protection Agency

3.3.14 *EU*—European Union

3.3.15 *FID*—Flame Ionization Detection

3.3.16 *GC-MS*—Gas Chromatography-Mass Spectrometry

3.3.17 *GFAA*—Graphite Furnace Atomic Absorption Spectrometry

3.3.18 *HAFID*—Hydrogen Atmosphere Flame Ionization Detection

3.3.19 *HFAA*—Heptaflurobutyric acid anhydride

3.3.20 *HIPS*—High Impact Polystyrene

3.3.21 *HPLC*—High Performance (or Pressure) Liquid Chromatography

3.3.22 *ICP-MS*—Inductively Coupled Plasma – Mass Spectrometry

3.3.23 *ICP-OES*—Inductively Coupled Plasma – Optical Emission Spectrometry

3.3.24 *IR*—Infrared Spectrometry

3.3.25 *NIOSH*—United States National Institute for Occupational Health and Safety

3.3.26 *PVC*—Polyvinyl Chloride

3.3.27 *REACH*—Registration, Evaluation and Authorization of Chemicals

3.3.28 *RoHS*—Restriction of the Use of Certain Hazardous Substances in Electrical and Electronic Equipment

3.3.29 *XRF*—X-ray Fluorescence Spectrometry

4. Summary of Guide

4.1 This guide provides a list of test methods for the determination of the Substances of Very High Concern as identified in the REACH Candidate list. Screening methods are discussed in Appendix X2.

4.2 This guide provides identifiers for each substance such as chemical name, synonyms, chemical formulas, CAS and EU numbers.

4.3 Common uses for each of the substances are identified.

5. Significance and Use

5.1 The REACH Candidate list classifies substances as SVHCs thus making them subject to possible authorization. Compliance to the REACH regulation requires that any identified SVHC be present at a concentration of less than 0.1 % (w/w) of the total article weight to avoid triggering a reporting obligation. This guide is intended to assist in the identification of available test methods for quantitative analysis of the substance(s) of interest.

5.2 When possible, industry accepted standard test methods are cited. However, industry vetted test methods are not

[5] Available from the Consumer Electronics Association, 1919 S. Eads St. Arlington, VA 22202, http://www.ce.org

[6] ASTM Dictionary of Engineering Science & Technology, 10th Edition. Available from www.astm.org.

available for all of the substances contained in the REACH Candidate List. Thus, some caution and due diligence must be exercised when applying some of the methods listed in this guide.

5.3 In some cases, test methods for the identification and quantification of a specific substance are not available. An example would include $CoCl_2$. Methods currently in practice involve the individual determination of Co and Cl concentrations and use other sources of information or chemical judgment to assign the expected $CoCl_2$ concentration. This approach obviously has its limitations and pitfalls and must be used judiciously.

5.4 Under the REACH regulation, EU manufacturers, importers or distributors of articles containing more than 0.1 % (w/w) of a substance that the Agency has listed as being an SVHC shall provide their customers with the name of the substance and information allowing the safe use of the article. Producers and distributors of articles containing SVHC shall also supply the same information to consumers, upon request. In situations where this information is not readily available from the supply chain it is incumbent upon the supplier to collect this information through actual chemical analysis or other means. This guide is intended to assist in the selection of appropriate test methods in the event that chemical analysis is required.

6. Substance List and Uses

6.1 The substances listed in Table 1 were derived from the ECHA SVHC Candidate List published in October 2008. Any substances that have been subsequently added after October 2008 are not included.

6.2 The CAS or EU numbers are unique identifiers for the substance. It is possible for a particular substance to have one or more commonly used names.

6.3 Common uses of the substance help to identify in which products or materials these substance is likely to be found. Note that the list of common uses is not exhaustive.

7. SVHC Test Methods

7.1 This guide is not intended to be exhaustive in the identification of available test methods. The intent is to provide guidance and some examples of available test methods relevant to the required analysis. Some of the SVHCs do not have industry standard test methods associated with them. Literature citations of these non-standard methods are meant to be for information only.

7.2 Standard test methods do not exist for all of the SVHCs listed. Therefore, the test methods cited for those substances must be validated for analytical accuracy before use in regulatory compliance demonstration. Moreover, when a method is applied outside of its intended scope, validation of the altered method is required. Considerations such as sample matrix, analytical requirements, etc. for the intended analysis will determine its applicability. All deviations from the published method must be clearly noted.

NOTE 1—If a standardized method is used beyond the stated scope, that addition must be validated. All deviations from published methods must be documented.

7.3 In many cases, test methods that are specific for a particular substance have not been developed. Therefore, the approach to quantitative analysis is not straightforward. A combination of test methods and the use of logical assumptions are required. These assumptions shall be clearly articulated because they will determine the limitations of the approach.

7.4 Screening methods can be utilized to determine whether a detectable amount of a substance is present. In many situations the application of a screening process can obviate the need to perform full quantitative analysis. See Appendix X2 for more information on screening methodologies.

7.5 In some cases, sample preparation methods are dependent upon the physical state of the sample to be analyzed. For many of the test methods, several sample preparation references are cited when applicable.

7.6 In some cases, limitations in the sample preparation method cited in Appendix X1.2 can produce a sample for analysis that is non-optimal. Incomplete extraction, incomplete digestion, loss of analyte, etc. often require adjustments to the sample preparation methods to obtain the desired analytical result. At a minimum the limitations in the sample preparation method shall be accounted for, when possible, in the calculation of the analytical result. Additionally, if these adjustments to the method do not produce a satisfactory sample for analysis, it may be necessary to develop an alternative method. Validation of the sample preparation method will thus be required.

7.7 Detection limits for the test methods are given when available and for reference only. It is necessary when reporting analytical results that they conform to the definition given by the reporting requirement.

7.8 Under certain circumstances it is advisable to employ external technical consulting services if the analysis being addressed is beyond the expertise of internal resources. There exist a number of laboratories that specialize in analysis of regulated substances for a fee.

8. Keywords

8.1 anthracine; arsenic oxide; brominated flame retardants; candidate list; cobalt dichloride; hexabromocyclododecane; lead hydrogen arsenate; MDA; musk xylene; phthalates; REACH; short chain chlorinated paraffins; sodium dichromate; Substances of Very High Concern; tributyltin oxide; triethyl arsenate

TABLE 1 SVHC Substances and Their Common Uses

Substance Name	Synonym	CAS Number	EU Number	Chemical Formula	Common Uses
4,4'-Diaminodiphenylmethane	4,4'-Methylene-dianiline, MDA	101-77-9	202-974-4	$C_{13}H_{14}N_2$	Converted to methylenedipheny diisocyanate (MDI). MDI used for polyurethane production. Other uses include: (1) hardener for epoxy resins and adhesives, (2) basic ingredient of colorant, and (3) intermediate for high-performance polymer.
5-tert-Butyl-2.4.6-Trinitro-m-Xylene	Musk Xylene (2,4,6-Trinitro-1,3-dimethyl-5-t-butylbenzene)	81-15-2	201-329-4	$C_{12}H_{15}N_3O_6$	Used as a fragrant particularly for consumer cosmetic products
Alkanes, C10-13, chloro	Short Chain Chlorinated Paraffins (SCCP)	85535-84-8	287-476-5	$C_xH_{(2x-y+2)}Cl_y$ where x=10-13 and y=1-13	May act as a secondary plasticizer or flame retardant in PVC. Other uses include: (1) metal working lubricant, (2) rubber parts, (3) paints, (4) sealant, (5) leather work, and (6) fiber.
Anthracene	Paranaphthalene	120-12-7	204-371-1	$C_{14}H_{10}$	An intermediate in the production of dyes; used in the manufacturer of pyrotechnic products
Benzyl butyl phthalate	Phthalic acid, benzyl butyl ester	85-68-7	201-622-7	$C_{19}H_{20}O_4$	Plasticizer in flooring material such as PVC foam. Other uses are as a plasticizer in traffic cones, food conveyor belts, artificial leather, etc.
Bis(2-ethylhexyl)phthalate	Dioctylphthalate	117-81-7	204-211-0	$C_{24}H_{38}O_4$	Commonly used as a platicizer in manufacturing of articles made of PVC, resins, rubbers, packaging materials, some paper products, and various medical devices, including the blood bags. It can also be found in some hydraulic fluids or dielectric fluids in capacitors. It is often found in coatings, pigments, textiles, or used as a solvent in light sticks.
Bis (tributyltin) oxide	Distannoxane, hexabutyl-	56-35-9	200-268-0	$C_{24}H_{54}OSn_2$	Primarily used as a biocide for boats, mildew proofing for leather work
Cobalt dichloride	Cobaltous Chloride	7646-79-9	231-589-4	$CoCl_2$	This substance can be used for (1) humidity indicator, (2) absorbent of ammonia gas, (3) gas mask, (4) production for vitamin B12, (5) trace amount of nutrient factor for food, (6) trace amount of element for nitric-acid pesticides, (7) solvent in purifying magnesium. Additionally, it may be used for packaging.
Diarsenic pentaoxide	Diarsenic Pentoxide	1303-28-2	215-116-9	As_2O_5	This substance can be used for (1) colorants, (2) metal refining, (3) special glass production, and (4) antiseptic agent for wood.
Diarsenic trioxide	Arsenic Trioxide	1327-53-3	215-481-4	As_2O_3	This substance can be used for (1) decolorants for glasses and enamel, (2) purifying and oxidizing agent in production of special glasses and lead crystal, (3) antiseptic agent for wood, and (4) medicine for leukemia.
Dibutyl phthalate	1,2-Benzenedicarboxylic acid di-n-butyl ester	84-74-2	201-557-4	$C_{16}H_{22}O_4$	Common plasticizer used in various polymers to keep crystals from forming. It is also used in paints, pigments, or printing inks as an adhesive agent. It is soluble in various organic solvents. This property allows it to be used as fixatives in perfumes or as an ectoparasiticide.
Hexabromocyclododecane	Cyclododecane, hexabromo-isomers	25637-99-4	247-148-4	$C_{12}H_{18}Br_6$	Used as a brominated flame retardant for polystyrene materials, e.g., HIPS case material and packaging.
Lead hydrogen arsenate	Lead Acid Arsenate	7784-40-9	232-064-2	$(AsO_4H)Pb$	Insecticide or pesticide
Sodium dichromate dihydrate	Sodium Bichromate	7789-12-0	234-190-3	$Na_2Cr_2O_7.2H_2O$	Used for: (1) production of other chromium compounds, (2) production of inorganic chromate pigments, (3) preservative supplement, finishing of metal plating, (4) production of vitamin K.
Triethyl arsenate	Arsenic acid Triethyl Ester	15606-95-8	427-700-2	$(C_2H_5O)_3AsO$	Used for integrated circuit manufacturing as an intermediate for n-type doping of semiconductors.

TABLE 2 Test Method(s) for 4, 4'-Diaminodiphenylmethane

Chemical Formula	$C_{13}H_{14}N_2$
Test Method	**United States Department of Labor – OSHA Organic Method #57**
Description	This method is applicable to samples in air matrix. Analysis is performed by analyzing for the heptafluorobutyric acid anhydride (HFAA) derivative of $C_{13}N_{14}N_2$ by gas chromatography using an electron capture detector.
Technique	GC-ECD
Sample Prep Method	Air extraction using sulfuric acid treated glass fiber filters. Derivatization of $C_{13}H_{14}N_2$ with HFAA. Extraction with toluene.
Analyte	HFAA derivative of $C_{13}H_{14}$ N_2 in toluene.
Detection	Electron capture detection of the HFAA derivative of $C_{13}H_{14}N_2$
Detection Limit	8.1 ng/sample (10 ppt or 81 ng/m^3 of air) for overall procedure; 3.2 pg/injection for analytical procedure.
Range	Not provided

TABLE 3 Test Method(s) for Anthracene

Chemical Formula	$C_{14}H_{10}$
Test Method	**EPA Method 8310**
Description	EPA Method 8310 is used to determine the concentration of certain polynuclear aromatic hydrocarbons (PAH) in ground water and wastes.
Technique	HPLC
Sample Prep Method	EPA Method 3540 or 3550 for solid samples
Analyte	$C_{14}H_{10}$ extracted into an appropriate solvent
Detection	Ultraviolet (UV) – Fluorescence
Detection Limit	0.66 µg/L
Range	Not provided
Test Method	**EPA Method 8270D**
Description	EPA Method 8270D is used to determine the concentration of semi-volatile organic compounds in extracts prepared from many types of solid waste matrices, soils, air sampling media and water samples
Technique	GC-MS
Sample Prep Method	EPA Method 3535, 3542 and 3561
Analyte	$C_{14}H_{10}$ extracted into an appropriate solvent
Detection	Mass spectrometry – primary ion at m/z 178
Detection Limit	10 µg/L
Range	Not provided
Test Method	**EPA Method 8100**
Description	EPA Method 8100 is used to determine the concentration of certain polynuclear aromatic hydrocarbons
Technique	GC-FID
Sample Prep Method	EPA Method 3540 or 3550 for solid samples
Analyte	$C_{14}H_{10}$ extracted into an appropriate solvent
Detection	Flame ionization
Detection Limit	1 µg/L
Range	Not provided

TABLE 4 Test Method(s) for Arsenic Pentoxide

Chemical Formula	As_2O_5
Analytical Approach	No test method is known for detecting As_2O_5 directly. Elemental Arsenic is detected and calculated as if all of the As is from As_2O_5.
Test Method	**EPA Method 7010**
Description	Metals in solution may be readily determined by graphite furnace atomic absorption spectrophotometry. The method is simple, quick, and applicable to a large number of metals in environmental samples including, but not limited to, water, domestic and industrial wastes, extracts, soils, sludges and sediments.
Technique	GFAA
Sample Prep Method	Acid digestion. Reference: EPA Method 3051A, Microwave assisted digestion of sediments, sludges, soils and oils; ASTM Practices D1971; ASTM Practice D4309.
Analyte	As (elemental)
Detection	Absorption at 193.7 nm
Detection Limit	1 µg/L
Range	Not provided
Caveats	Aluminum may interfere depending upon background correction method. Worst case scenario attributes that all the As has reacted with O. Knowledge of the system, either from manufacturer, engineering knowledge or other sources, is required to assume less than this worst case.
Test Method	**EPA Method 6020**
Description	This method is of wide applicability for analysis of a variety of elements in different types of matrices using Inductively Couple Plasma/Mass Spectrometry.
Technique	ICP-MS
Sample Prep Method	EPS Method 3051A, Microwave assisted digestion of sediments, sludges, soils and oils
Analyte	As (elemental)
Detection	Mass spectrometry of primary ion at m/z 75
Detection Limit	0.01 µg/L
Range	0.1 µg/L to 1000 µg/L
Caveats	Worst case scenario attributes that all the As has reacted with O. Knowledge of the system, either from manufacturer, engineering knowledge or other sources, is required to assume less than this worst case.
Test Method	**NIOSH Manual of Analytical Methods, Method 7901**
Description	This method was developed for particulate arsenic compounds
Technique	GFAA
Sample Prep Method	Digestion is required for all samples with the exception of those where the As is dissolved. A matrix modifier shall be added to all digestates prior to analysis to minimize volatilization losses during drying and ashing. Reference: US EPA Method 3051A, Microwave assisted digestion of sediments, sludges, soils and oils; ASTM Practices D1971, ASTM Practice D4309. Similar standardized methods for AAS sample preparation may be used.
Analyte	As (elemental)
Detection	Absorption at 193.7 nm
Detection Limit	0.06 µg
Range	0.3 µg to 13 µg
Caveats	Aluminum may interfere depending upon background correction method. Worst case scenario attributes that all the As has reacted with O. Knowledge of the system, either from manufacturer, engineering knowledge or other sources, is required to assume less than this worst case.