



Standard Guide for Assessing the Environmental and Human Health Impacts of New Energetic Compounds¹

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INTRODUCTION

Sustaining training operations while maintaining force health is vital to national security. Research efforts are underway to identify energetic substances that have negligible environmental impacts and implement them in military munitions. This guide is intended to provide a standardized method to evaluate the potential environmental impacts of prospective candidate energetic substances. This guide is intended for use by technical persons with a broad knowledge of risk assessment, fate and transport processes, and toxicology to provide recommendations to the research chemist or engineer regarding the environmental consequences of use.

1. Scope

1.1 This guide is intended to determine the relative environmental influence of new munition constituents, consistent with the research and development (R&D) level of effort and is intended to be applied in a logical, tiered manner that parallels both the available funding and the stage of research, development, testing, and evaluation. Specifically, conservative assumptions, relationships, and models are recommended early in the research stage, and as the munition technology is matured, empirical data will be developed and used. Munition constituents may include fuels, oxidizers, explosives, binders, stabilizers, metals, dyes, and other compounds used in the formulation to produce a desired effect. Munition systems range from projectiles, grenades, rockets/missiles, training simulators, smokes and obscurants. Given the complexity of issues involved in the assessment of environmental fate and effects and the diversity of the munition systems used, this guide is broad in scope and not intended to address every factor that may be important in an environmental context. Rather, it is intended to reduce uncertainty at minimal cost by considering the most important factors related to the environmental impacts of energetic materials. This guide provides a method for collecting data useful in a relative ranking procedure to provide the munition scientist with a sound basis for prospectively

determining a selection of candidates based on environmental and human health criteria.

1.2 The scope of this guide includes:

1.2.1 Energetic materials and compositions in all stages of research, development, test and evaluation.

1.2.2 Environmental assessment, including:

1.2.2.1 Human and ecological effects of the unexploded energetics and compositions on the environment.

1.2.2.2 Environmental transport mechanisms of the unexploded energetics and composition.

1.2.2.3 Degradation and bioaccumulation properties.

1.2.3 Occupational health impacts from manufacture and use of the energetic substances and compositions to include load, assembly, and packing of the related munitions.

1.3 Given the wide array of applications, the methods in this guide are not prescriptive. They are intended to provide flexible, general methods that can be used to evaluate factors important in determining environmental consequences from use of the energetic substances.

1.4 Factors that affect the health of humans as well as the environment are considered early in the development process. Since some of these data are valuable in determining health effects from generalized exposure, effects from occupational exposures are also included.

1.5 This guide does not address all processes and factors important to the fate, transport, and potential for effects in every system. It is intended to be balanced effort between scientific and practical means to evaluate the relative environmental effects of munition compounds resulting from intended use. It is the responsibility of the user to assess data quality as

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well as sufficiently characterize the scope and magnitude of uncertainty associated with any application of 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

- D 5660** Test Method for Assessing the Microbial Detoxification of Chemically Contaminated Water and Soil Using a Toxicity Test with a Luminescent Marine Bacterium
- E 729** Guide for Conducting Acute Toxicity Tests on Test Materials with Fishes, Macroinvertebrates, and Amphibians
- E 857** Practice for Conducting Subacute Dietary Toxicity Tests with Avian Species
- E 943** Terminology Relating to Biological Effects and Environmental Fate
- E 1023** Guide for Assessing the Hazard of a Material to Aquatic Organisms and Their Uses
- E 1147** Test Method for Partition Coefficient (N-Octanol/Water) Estimation by Liquid Chromatography
- E 1148** Test Method for Measurements of Aqueous Solubility
- E 1163** Test Method for Estimating Acute Oral Toxicity in Rats
- E 1193** Guide for Conducting *Daphnia magna* Life-Cycle Toxicity Tests
- E 1194** Test Method for Vapor Pressure
- E 1195** Test Method for Determining a Sorption Constant (K_{oc}) for an Organic Chemical in Soil and Sediments
- E 1241** Guide for Conducting Early Life-Stage Toxicity Tests with Fishes
- E 1279** Test Method for Biodegradation By a Shake-Flask Die-Away Method
- E 1372** Test Method for Conducting a 90-Day Oral Toxicity Study in Rats
- E 1415** Guide for Conducting Static Toxicity Tests With *Lemna gibba* G3
- E 1525** Guide for Designing Biological Tests with Sediments
- E 1624** Guide for Chemical Fate in Site-Specific Sediment/Water Microcosms
- E 1676** Guide for Conducting Laboratory Soil Toxicity or Bioaccumulation Tests with the Lumbricid Earthworm *Eisenia Fetida* and the Enchytraeid Potworm *Enchytraeus albidus*
- E 1689** Guide for Developing Conceptual Site Models for Contaminated Sites
- E 1706** Test Method for Measuring the Toxicity of Sediment-Associated Contaminants with Freshwater Invertebrates

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. Terminology

3.1 Definitions of Terms Specific to This Standard:

3.1.1 *conception, n*—refers to part of the munition development process whereby molecules are designed through software and modeling efforts though not yet synthesized.

3.1.2 *demonstration, n*—refers to testing munition compounds in specific configurations that may use other substances to maintain performance specifications.

3.1.3 *engineering and manufacturing development, n*—involves the process of refining manufacturing techniques and adjusting formulations to meet production specifications.

3.1.4 *environmental, adj*—used to describe the aggregate of a receptor's surroundings that influence exposure, used in the holistic sense that may include human exposures in a variety of conditions.

3.1.5 *energetic materials, n*—chemical compounds or compositions that contain both fuel and oxidizer and rapidly react to release energy and other products of combustion. Examples of energetic materials are substances used in high explosives, gun propellants, rocket & missile propellants, igniters, primers, initiators, and pyrotechnics (for example, illuminants, smoke, delay, decoy, flare and incendiary) and compositions. Energetic materials may be thermally, mechanically, and electrostatically initiated and do not require atmospheric oxygen to sustain the reaction.

3.1.6 *munition, n*—refers to weapon systems or platforms that have a military application. Includes the use of energetic substances in addition to stabilizers, plasticizers, and other substances to the final combined formulation referred to as energetic material.

3.1.7 *production, n*—includes activities involved in the finalized manufacturing and use of the munition compound and accompanying system.

3.1.8 *synthesis, n*—process in which minute (gram) quantities of the energetic material are made, often using laboratory desktop equipment.

3.1.9 *testing and refinement, n*—includes preliminary small-scale tests to large-scale testing and range operations that require refined synthesis techniques within the research and development phase for new energetic compounds. Energetic materials may be combined with other ingredients at this stage to tailor specific performance properties.

4. Summary of Guide

4.1 In the evaluation of the probability of adverse environmental effects, measures of exposure are compared with measures of toxicity to evaluate relative risk. These methods and data requirements are balanced with the level of funding used in munition compound development. This guideline, therefore, provides a tiered approach to data development necessary for various levels of risk assessment. Often it results in a relative ranking of properties, not a robust estimation of exposure. Initially, physical/chemical properties necessary for fate, transport, and exposure estimation may be derived and estimated from conceptual compounds developed from computer model simulations. Quantitative structural activity relationships (QSARs) and quantitative structural property relationships (QSPRs) may be useful in estimating toxicity and

chemical properties, respectively. Following successful synthesis of compounds, key properties may be experimentally determined (for example, water solubility, vapor pressure, sorption (K_{oc}), octanol/water partition coefficients (K_{ow}), boiling point, and so forth). These properties can be used in a relative manner or quantitatively to determine potential for transport and bioaccumulation. Given the expense involved, toxicity studies are tiered, where lower cost *in vitro* methods are used early in the process and more expensive *in vivo* methods are recommended later in the development process. Acute mammalian toxicity data may be generated, along with soil, water, and sediment toxicity to invertebrates (Tier I tests). Earthworm bioaccumulation tests may also be conducted, along with an evaluation of plant uptake models. At advanced stages, sublethal mammalian testing shall be conducted along with avian and other limited vertebrate toxicity tests (Tier II tests).

5. Significance and Use

5.1 The purpose of this guide is to provide a logical, tiered approach in the development of environmental health criteria coincident with level and effort in the research, development, testing, and evaluation of new energetic materials. Various levels of uncertainty are associated with data collected from previous stages. Following the recommendation in the guide should reduce the relative uncertainty of the data collected at each developmental stage. At each stage, a general weight of evidence qualifier shall accompany each exposure/effect relationship. They may be simple (for example, low, medium, or high confidence) or sophisticated using a numerical value for each predictor as a multiplier to ascertain relative confidence in each step of risk characterization. The specific method used will depend on the stage of development, quantity and availability of data, variation in the measurement, and general knowledge of the dataset. Since specific formulations, conditions, and use scenarios are often not known until the later stages, exposure estimates can be determined only at advanced stages (for example, Engineering and Manufacturing Development; see 6.6). Exposure data can then be used with other toxicological data collected from previous stages in a quantitative risk assessment to determine the relative degree of hazard.

5.2 Data developed from the use of this guide are designed to be consistent with criteria required in weapons and weapons system development (for example, programmatic environment, safety and occupational health evaluations, environmental assessments/environmental impact statements, toxicity clearances, and technical data sheets).

5.3 Information shall be evaluated in a flexible manner consistent with the needs of the authorizing program. This requires proper characterization of the current problem. For example, compounds may be ranked relative to the environmental criteria of the prospective alternatives, the replacement compound, and within bounds of absolute environmental values. A weight of evidence (evaluation of uncertainty and variability) must also be considered with each criterion at each stage to allow for a proper assessment of the potential for adverse environmental or occupational effects; see 6.8.

5.4 This standard approach requires environment, safety, and occupational health (ESOH) technical experts to determine the risk and energetic materials researchers to evaluate the acceptability of the risk. Generally, the higher developmental stages require a higher managerial level of approval.

6. Procedure

6.1 *Problem Evaluation*—The first step requires an understanding of the current problem. Often, specific attributes of existing compounds drive the need for a replacement. For example, increased water solubility may indicate a propensity of the compound to contaminate groundwater. Environmental persistence and biomagnification may cause concerns regarding exposures to predatory animals and in human fish consumption. Increased vapor pressure may lead to significant inhalation exposures in confined spaces that would increase the probability of toxicity to workers or soldiers. A sound understanding of the factors principally attributed to the environmental problem is required to focus relative evaluation of these properties. A conceptualization of potential exposure pathways given specific chemical properties can be helpful in ascertaining likelihood for adverse effects. Guide E 1689 can be helpful in that regard. Table 1 provides stages of technical development of munition compounds and corresponding suggested data requirements.

TABLE 1 Life-Cycle Munition Development Stage Relative to the Collection of Data Important to the Evaluation of Environmental Criteria

Developmental Stage	Action	Data Requirement
Conception	Computer modeling (QSAR), computational chemistry	Chem/phys properties; toxicity estimates (mammalian and ecotoxicity)
Synthesis	Develop experimental chemical property data; conduct relative toxicity screen	Chem/phys properties (estimate fate, transport, bioaccumulation), in-vitro mammalian toxicity screen, in-vitro ecotoxicity screen (for example, luminescent bacteria)
Testing	Conduct Tier I mammalian toxicity testing	Acute/subacute rodent toxicity data; in-vitro cancer screen
Demonstration	Conduct Tier II mammalian toxicity testing; Tier I Ecotox screening	Subchronic rodent toxicity data; aquatic/plant/earthworm assays
Engineering and manufacturing development	Cancer studies ^A ; Tier II Ecotox studies, evaluate plant uptake	Rodent cancer evaluation; avian, amphibian studies; plant uptake models
Production	Evaluate exposure and effects	No additional data required ^B
Storage and use	Evaluate exposure and effects	No additional data required
Demilitarization	Evaluate exposure and effects	No additional data required

^A Only necessary if in-vitro screens are predominantly positive and potential for exposure is relatively high.

^B In certain cases, it may be necessary to verify predictions through environmental monitoring procedures.

6.2 Conception—At this stage of energetic material development, molecular relationships and characteristics are examined to evaluate the properties of a new material. These include molecular and electronic structure, stability, thermal properties, performance and sensitivity requirements, and decomposition pathways. Since these substances are still conceptual, no empirical data exist.

6.2.1 The predicted molecular and electronic structural properties can be used in quantitative structure-activity relationship (QSAR) or other approaches to determine chemical/physical properties relating to toxicity, fate, and transport. These properties can be gleaned from computer-modeled estimations using quantitative structure-property relationship (QSPR)-like or quantum mechanical models. The properties that are useful in estimating the extent of fate and transport include the following:

- 6.2.1.1 Molecular weight;
- 6.2.1.2 Water solubility;
- 6.2.1.3 Henry's law constant;
- 6.2.1.4 Vapor pressure;
 - (1) Liquid-phase vapor pressure;
 - (2) Solid-phase vapor pressure;
- 6.2.1.5 Affinity to organic carbon; sorption ($\log K_{oc}$);
- 6.2.1.6 Lipid solubility (octanol/water coefficient; $\log K_{ow}$);
- 6.2.1.7 Boiling point;
- 6.2.1.8 Melting point; and
- 6.2.1.9 Ionization potential.

6.2.2 When existing materials show promise as alternatives, conduct a literature search to determine first if Chemical Abstract Service (CAS) registry numbers are available. A comprehensive database available from the National Institute of Health can be used to search for this information (<http://chem.sis.nlm.nih.gov/chemidplus/>). These CAS numbers may then be used to search for chemical/physical property values and toxicity information without significant risk of confusion regarding synonyms. Other databases may provide information regarding chemical/physical properties and toxicity. See the suite available at <http://toxnet.nlm.nih.gov/>.

6.2.3 Models are available to predict environmental parameters with an inherent degree of uncertainty. It is important that this uncertainty be captured using a qualitative or semiquantitative approach (see 6.8). Examples of such models include those found in the EPI suite³ (<http://www.epa.gov/oppt/exposure/pubs/episuite.html>; (1)⁴) and can be helpful in obtaining values.

6.2.4 Henry's law constant is calculated using the following equation:

$$H = \frac{Vp(MW)}{S} \quad (1)$$

where:

- H = Henry's law constant ($\text{atm} \cdot \text{m}^3/\text{mol}$),
- Vp = vapor pressure (atm) at 25°C (298 K),

- MW = molecular weight (g/mol), and
- S = solubility in water (mg substance/L).

6.2.5 Octanol/water partition coefficients ($\log K_{ow}$) can be predicted through the use of QSPR models. Models that predict sorption (affinity to organic carbon; $\log K_{oc}$) are generally not required since $\log K_{oc}$ can be predicted from $\log K_{ow}$ values using the following equation:

$$K_{oc} = 10^{[0.0784 + (0.7919 + (\log K_{ow}))]} \quad (2)$$

where:

- K_{oc} = soil organic carbon-water partition coefficient (mL water/g soil), and
- K_{ow} = *n*-octanol/water partition coefficient (unitless).

6.2.6 QSAR approaches can also be used to estimate toxicological impact. Toxicity QSAR models can often predict many parameters before experimental toxicology testing but are dependant upon similar compounds that have toxicity data. These models produce estimates of toxicity (for example, rat subchronic no observed adverse effect levels (NOAELs)) are used to rank new energetic materials, not to evaluate them quantitatively. These methods provide a relatively fast, low-cost method for developing the minimum amount of environmental data necessary for an initial evaluation of environmental impacts. They can be used as a basis for go/no-go decisions regarding further development and can serve to focus further research. These rankings shall be based on measures of toxicity (for example, acute values such as LD50s, chronic/subchronic rat lowest observed adverse effect levels (LOAELs), and so forth). QSARs may also be used in a qualitative sense to evaluate the need for focused developmental, reproductive (for example, endocrine-like functional groups) in vivo testing. Compounds with structure suggesting specific toxicity should be qualified for further testing at advanced stages in munition development (for example, engineering and manufacturing development).

6.2.7 Following the problem evaluation procedure, pertinent properties are compared along with those of other candidate substances and, if applicable, with the currently used munition constituent marked for replacement. Estimates of the relative level of confidence (for example, high, medium, or low) shall also be assigned to each attribute. These qualifiers may be assigned a numerical weight and used in a semiquantitative approach. These substances are then ranked, evaluated based on absolute parameters, and/or assessed relative to the replacement substance configuration according to these criteria to provide the munition scientist with a prioritized list from which to focus efforts or provide general recommendations regarding their use in an environmental or occupational context or both.

6.3 Synthesis—Following the conceptualization and successful assessment of a new material, it must be made. Once it is shown that small amounts of a new energetic material can be produced, small-scale screening tests shall be performed to establish performance characteristics. If the material is found to be acceptable from a performance perspective, risks from an environmental and occupational perspective can be more reliably determined through experimentally determining chemical properties in small-scale tests using actual material. If the candidate is suitable for further consideration, performance

³ EPI Suite is a trademark of ImageWare Systems, Inc. 10883 Thornmint Road San Diego, CA 92127.

⁴ The boldface numbers in parentheses refer to the list of references at the end of this standard.

in gun or warhead configurations will be modeled to provide information on emissions. Amounts needed for each assay may need to be determined before initiation. These methods can be used to develop data that can increase confidence in risk (fate, transport, and toxicity) predictions. In addition, analytical chemistry methods are also needed at this stage.

6.3.1 Analytical chemistry and standard experimental methods can be used to develop the following data. The appropriate ASTM International standard is referenced where applicable.

6.3.1.1 *Water Solubility*—Test Method E 1148.

6.3.1.2 *Vapor Pressure*—Test Method E 1194.

6.3.1.3 *Log K_{oc}* —Test Method E 1195.

6.3.1.4 *Log K_{ow}* —Test Method E 1147.

6.3.1.5 *Boiling Point*—Organization for Economic Cooperation and Development (OECD) Test Guidelines 102 (2).

6.3.1.6 *Relative Toxicity*—Use of *in vitro* techniques.

6.3.2 Increased water solubility suggests a propensity for increased bioavailability and transfer to groundwater. This, however, shall be compared with the affinity to organic carbon, since sorption assists in retarding migration to groundwater. As mentioned, log K_{ow} values may be derived from log K_{oc} values (3); however, experimentally derived data are recommended at this stage, if feasible.

6.3.3 Increased vapor pressure and a lower boiling point suggest a greater propensity for inhalation exposures and can be compared in a relative sense. Molecular weight is valuable in determining exposure within and between organ systems (4, 5).

6.3.4 Relative acute toxicity can be evaluated using low-cost and rapid *in-vitro* basal cytotoxicity assays (for example, Neutral Red Uptake (NRU) <http://iccvam.niehs.nih.gov/methods/invitro.htm>). Relative acute toxicity can be evaluated using relatively low-cost *in-vitro* cell culture techniques (for example, MTT [3-(4,5-dimethylthiazol-2-yl)-2,5-diphenyltetrazolium bromide] assay, cell exclusion dyes, and propidium iodide (6, 7)). Specific assays that assess cellular function may be needed when toxicity for replacement compound is not mediated by changes in metabolism, necrosis or cell death. Screening-level ecotoxicological methods (8), Test Method D 5660 can be used to ascertain relative toxicity to the test organism and can be used for ranking purposes, though all have limitations (8, 9).

6.3.5 As before, these data are used to improve on the information and confidence estimates used in the previous evaluation. The relative weight of each ranking criterion depends upon the factors most important to the initial problem. Confidence estimates shall be used as ranking criteria in providing the hierarchical list of candidates.

6.4 *Testing*—This involves testing new energetic materials in various systems and configurations to determine the best formulations to achieve specific performance characteristics. This often requires varying the proportions of various compounds to achieve performance goals. Other substances, such as binders or plasticizers, are used to meet specifications. This requires an understanding of the dynamics of these mixtures insofar as they affect transport and fate (for example, products of combustion) as well as attributes of any introduced compounds to the mixture. Since larger masses/volumes of com-

pounds are needed at this stage, the probability for human exposure increases; therefore, it is important to have baseline human toxicity data (Tier I testing). At this stage, the following are important data to collect.

6.4.1 Sorption can be measured experimentally in various soil types using Test Method E 1195. Modeled approaches using available software systems could be used to estimate biodegradation, persistence, bioaccumulation, and toxicity, respectively (1).

6.4.2 Animal data are now needed since potential for human exposure is likely. Acute rodent studies shall be conducted before subacute and subchronic studies. Test Method E 1163 describes the up/down method to determine the lethal dose for 50 % of rats exposed to a single oral dose. Following the determination of the acute LD50, a 14-day range finding (subacute) study is required to refine sublethal levels of exposure for the 90-day subchronic tests (Test Method E 1372); data from the latter are required to determine a chronic benchmark (for example, reference dose). Study conduct and hence data quality is important. It is therefore recommended that mammalian toxicity studies are conducted consistent with good laboratory practices (GLPs). Extent of sublethal mammalian toxicity (NOAELs and LOAELs) needs to be identified. If the compound has properties consistent with exposures via inhalation routes, then the inhalation counterpart to these tests shall be conducted. The subchronic portion may be conducted coincident with the demonstration stage if it is more feasible to do so.

6.4.3 Identification of combustion products is important in characterizing exposure of those immediately exposed and resulting environmental loads. These methods are compound specific and involve consultation with munition scientists regarding the potential products of oxidation, reduction, and other processes important in attenuation and transformation in the environment. Some models and methods are available to address potential products but have assumptions specific to the design. These models can be used to produce a refined list of substances from which to investigate further. Rarely do products of combustion contribute significantly to environmental media concentrations (10); however, products of incomplete combustion (for example, pyrotechnics and smokes) may be important to specific receptors.

6.4.4 Propensity for persistence and transport can be estimated based on chemical physical properties and modeled approaches. Environmental half-lives may be estimated based on structure for various media and qualitative estimates can be made. Likelihood for transport may be estimated from water solubility (for example, solubility exceeding 1 g/L suggests the material is likely to contaminate groundwater). Affinity to organic carbon (K_{oc}) is also helpful in determining whether a compound is likely to reach groundwater. Vapor pressure, Henry's Law constant, and boiling point are useful for determining whether a compound is likely to volatilize or remain in water.

6.4.5 The potential for bioaccumulation/bioconcentration of organics may be predicted from the log K_{ow} . Organic compounds with log K_{ow} values < 4 do not generally bioaccumulate or biomagnify (1, 11, 12). Computer models exist to estimate

bioconcentration potential (body burdens in aquatic organisms **(1)**). Inorganics shall be evaluated separately.

6.5 Demonstration—At this stage, new energetic formulations are being designed and used in specific weapon system configurations. Therefore, greater masses of materials are being synthesized but not yet at a production capacity, and they have typically been blended into a composition consisting of several substances to tailor the performance and handling properties. Since workers and soldiers will be exposed at some level during testing, a greater investment in the program is required to proceed. Specific mammalian and ecotoxicity data are now needed to reduce uncertainty further to determine likelihood of adverse effects from environmental and occupational exposures (Tier II and Tier I, respectively; **Table 1**). This includes an assessment of products from natural attenuation in order to address sustainability issues. Toxicity data may be used to form the technical basis for toxicity clearances required in Health Hazard Assessments **(13)**. At this stage it is also cost-effective to provide a more robust dataset regarding fate and transport mechanisms. As such, the following are recommended.

6.5.1 Persistence or environmental half-lives can be more reliably determined using experimental methods and site-specific information (for example, ranges of soil types). The shake-flask test could be used to determine abiotic/biotic degradation rates of samples in natural water systems (Test Method **E 1279**). This test method would provide baseline information regarding environmental persistence in wetland or mesic environments. Accurate and meaningful estimates of persistence and transport are dependant upon local and site-specific conditions. Since these compounds may be used in a variety of climates and environmental media types, ranges of conditions that account for this variation are needed to provide useful results. Therefore, assumptions (for example, soil type, temperature, rainfall amount, and so forth) need to be bracketed to provide decision makers with an accurate representation of the potential for contamination given the range of environmental conditions. Since this requires a fairly complex assessment, therefore, models may be relied upon for results. Soil biodegradation protocols are available (for example, Ref **(14)** describes methods for determining mineralization rates). Since some compounds may not completely breakdown, the usefulness of these methods shall be determined relative to compound structure and resource availability.

6.5.2 To best confirm modeled exposure estimates, analytical methods will be needed in various matrixes. These methods may likely be built on those published for similar compounds given the chemical/physical properties determined previously. Regardless, some method development and/or refinement may be needed.

6.5.3 Toxicological information gathered from previous steps may be used with more specific exposure criteria to determine personal protective equipment and probability for risk. Rodent bioassays (for example, subchronic oral studies) may have been delayed from the testing stage if specific formulations were undecided. At this stage, sublethal toxicology information shall be complete and preliminary safe thresholds for exposure need to be established.

6.5.4 In-vitro methods are available to assess the potential of a compound to cause cancer. Cancer screen includes variations of the Ames test complemented with the umu test **(15)** and cytogenic assays (CHO) with and without S-9 fraction. S-9 is a liver homogenate added to the Ames cultures that provides an analysis of compound metabolism products also. Congruence of results using these assays would indicate the potential for cancer and warrant further in-vivo assays if the predominant outcomes suggests a propensity for cancer.

6.5.5 Models and laboratory models that predicted combustion and attenuation products shall be tested under field conditions to verify predictions. This requires quantifying the amount of products predicted to be present in various environmental media. All of these data requirements are used together to provide an accurate characterization of risks, which include occupational assessments as well as environmental.

6.5.6 Since there is a greater potential for environmental releases during the Developmental stage, some experimental ecotoxicity data are suggested. These environmental toxicity studies can be conducted at relatively minor cost and effort. Toxicity assays conducted with fish, invertebrates and plants can provide information regarding environmental consequences from release (for example, Guides **E 729**, **E 1415**, **E 1193**, **E 1023**). Knowledge regarding primary exposure routes gained from fate and transport analyses should be used to prioritize tests and media types. These tests are often focused on three primary endpoints, that is, mortality, growth, and reproduction.

6.6 Engineering and Manufacturing Development—Specific formulation and application has largely been decided at this stage; however, specifics regarding treatment of filler materials and the energetics themselves may be adjusted for manufacturing, occupational, or compliance reasons. Since most details regarding final formulation and use have been determined, specific information important in environmental fate and probability of adverse effects from occupational and/or environmental exposures shall be conducted through a focused risk assessment. However, an understanding of components used in the manufacturing process may now need to be evaluated from an occupational and compliance context. As before, data collected from previous stages can be used and combined with data collected at this stage; however, it will likely require further information relevant to understanding occupational and compliance issues associated with the use of raw materials, intermediates, and by-products of manufacturing. Before a new energetic material is fielded and used in large quantities on ranges, the following environmental criteria need to be considered.

6.6.1 Friability and dissolution rate depend on weather and final munition formulation. This information determines the relative influence of rainfall on the potential for distribution of residuals in soil. Methods described in Lever et al **(16)** may be useful in determining these factors.

6.6.2 Ecotoxicity evaluations need to be consistent with exposure route and duration (Tier II; **Table 1**). Acute tests for fish, macroinvertebrates, and amphibians can be conducted using exposures from two to eight days (Guide **E 729**) and provide data that can be used in a relative manner to compare

between formulations. Other aquatic assays that evaluate long-term, sublethal effects may also be used to evaluate toxicity, if appropriate (for example, Guides E 1193-97, E 1241-05, but see Guide E 1023-84 as a review), however, it is important to understand the relative influence of nitrogen and phosphorus as nutrients in these systems. Other guidelines exist to evaluate the toxicity and fate of compounds in sediment (Guides E 1525, E 1624, and Test Method E 1706). Earthworm toxicity studies have been used extensively and can be conducted using standard methods (Guide E 1676). These assays may also provide information regarding bioaccumulation. Avian acute and subacute methods have been suggested, standardized or both (17), (18), Practice E 857. Although many standards involve administering compounds in feed as the method of exposure, such methods introduce complications (19, 20). Oral dosing methods can be conducted precisely and are preferred; however, they are not without caveats. See Note 1.

NOTE 1—Oral dosing methods (for example, gavage) provide precise information on effects from oral exposures of mg compound/kg bodyweight/day. Bolus and matrix effects of vehicle have been proposed as limitations.

6.6.3 Models can be used to estimate chemical uptake in specific portions of plants (20-23). These models can be used in a relative manner to address exposure potential from plant ingestion. Experimental data can be collected if models suggest uptake could be significant (24).

6.7 *Production, Storage, Use, and Demilitarization*—It is likely that no further data are needed for these subsequent stages (production, storage and use, and demilitarization); however, other information may be important to adjust risk estimates. During production, it may be advisable to perform specific monitoring procedures to determine if occupational and environmental guidelines are met (for example, permissible exposure levels, threshold limit values, and authorized effluent levels). Since previous combustion models are limited, verification of model results may be needed to include other possible compounds. It is also advisable that experts in fate, transport, and toxicology review data at each development stage to provide optimal professional judgments regarding feasible alternatives.

6.8 *Further Applications*—This assessment, including prospective future characterization of ranges, can be used to estimate range sustainability and help bracket future potential

liabilities. Integrated approaches involving state-of-the-art fate, transport, and hazard modeling can be accomplished using models such as those found in the Adaptive Risk Assessment Modeling (ARAMS) system. This approach provides specific information to the decision makers to determine the degree of hazard. These data may also be integrated into a programmatic environmental safety and health evaluation (PESHE), National Environmental Policy Act (NEPA) documentation, toxicity clearances and the health hazard assessments (HHA) to better characterize health risks posed by a new energetic material. Further monitoring may be necessary during the life cycle to ensure that the product performs as predicted.

7. Precision and Bias

7.1 *Precision*—Precision is the closeness of agreement between test results obtained under prescribed conditions. Precise experimental values for specific chemical, biological, toxicological, and physical property information are important for proper characterization of results. The level of precision for each test is provided within the test methods where cited, where appropriate.

7.2 *Bias*—Bias is a systematic error that contributes to the difference between the mean of a large number of test results and an accepted reference value. It is important that a weight of evidence qualifier accompany each value derived in this process to provide for an accurate characterization of results (see 6.8). Values obtained through computation means are far less certain than those obtained experimentally or analytically, though values obtained through each model or test method have variation in certainty associated with them.

8. Measurement Uncertainty

8.1 Measurement uncertainty shall be captured through the same weight of evidence method used to address variability and other uncertainties (that is, differences between precision and bias; see 5.1, 7.1 and 7.2). The user shall be responsible for explaining the means used to partition bias from precision. The effort and expense to achieve this partition need not exceed what is commensurate with the complexity and degree of development of the project. The user should, when appropriate, assign an appropriate weighting scheme to each derived or extrapolated value.

9. Keywords

9.1 effects; energetics; environment; fate; health; life cycle

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