In-Process Hazard Classification of Explosives
Overview of ETUG Standard ETUG-GS01-15

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ABSTRACT
This paper provides an overview of the existing in-process hazard classification procedure that has been adopted by the Explosives Testing Users Group (ETUG) as ETUG-GS01-15 *ETUG Standard for In-Process Hazard Classification of Explosives*. The transport hazard classification division for explosive substances or articles that are packaged/configured for transport may not be valid when the substance or article is removed from the transport packaging for handling, processing/manufacturing, staging, or other unique conditions found in the various life cycle stages. This creates the need for "in-process" classification of explosives. The ETUG Standard utilizes standards from the UN MTC, US Department of Defense (DoD), Bureau of Alcohol Tobacco and Firearms (ATF), and other industry-accepted test methods/protocols to more closely simulate key in-process parameters for explosive substances and articles in-process classification testing. Tests that incorporate these key parameters are essential to determine proper in-process classification of explosives. Application of the ETUG Standard In-Process Classification protocol can provide accurate classification of explosives for the unique and varied configurations and conditions that may exist during manufacturing, remanufacturing, intermediate storage, and use. Such explosives classification will facilitate proper design and specification of process equipment and facilities and will therefore reduce the risk to personnel.

INTRODUCTION
This paper provides an overview of the existing in-process hazard classification procedures that have been adopted by the Explosives Testing Users Group (ETUG) as ETUG-GS01-15 ETUG Standard for In-Process Hazard Classification of Explosives. The ETUG-GS01-15 in-process hazard classification protocols for explosives are referenced in the NFPA 495 Explosive Materials Code, Section 5.3 Classification and Characterization of Energetic Materials Used in Process Operations, Annex A, Explanatory Material, A.5.3.1, as an example test protocol in which the in-process classification of materials are established by testing. The ETUG Standard is also incorporated by reference in the International Fire Code (IFC) Section 5605 Manufacture, Assembly and Testing of Explosives, Explosive Materials and Fireworks (2012).

This is also the primary standard of the Explosives Testing Users Group as the group is dedicated to improving and standardizing **in-process** material response characterization methods. This and other standards and procedures are published here: http://www.etusersgroup.org/library.

The ETUG Standard utilizes standard or modified UN MTC, US Department of Defense (DoD), Bureau of Alcohol Tobacco and Firearms (ATF), and other industry-accepted test methods/protocols to more closely simulate key in-process parameters for explosive substances and articles in-process classification testing. Key in-process parameters include composition, physical state, configuration (confinement or packaging), conditions (e.g., temperature, pressure, humidity, etc.), and normal and abnormal ignition sources. Tests that incorporate these key parameters are essential to determine proper in-process classification of explosives.

Accurate classification of explosive substances and articles is essential for all life cycle stages of explosives. Life cycle stages include manufacturing/remanufacturing, storage (including intermediate product storage), transport, and use. Proper classification requires an understanding of how the explosive will respond to the conditions found in each stage. The in-process material classification may be different for each operation within each stage. How explosives respond depends on 1) the initiation source and 2) the key parameters that govern the type of explosive effect that will occur. Initiation sources include impact, friction, electrostatic discharge (ESD), thermal heating, shock, etc. which may result from normal or abnormal events that could occur during the various life cycle stages. Key explosive parameters that may be present in the given life cycle stage include the composition, physical state, configuration/confinement, quantity, and environmental conditions (e.g., temperature, pressure, humidity, etc.). These initiation sources and parameters are identified and defined by performing a proper risk assessment. Explosive classification will only be accurate if it is based on testing and risk assessment that incorporates the unique aspects associated with each life cycle stage. For transport, the UN MTC includes testing protocol, configurations, and criteria based on acceptable transport risk and hazard communication. Risk assessment has a significant role in the classification of explosives in the other life cycle stages where the configurations and conditions of the explosive change.

Application of the ETUG Standard In-Process Classification protocol will provide accurate classification of explosives for the unique and varied configurations and conditions that may exist during manufacturing, remanufacturing, intermediate storage, and use. Such explosives classification will facilitate proper design and specification of process equipment and facilities and will therefore reduce the risk to personnel.

1. Scope

1.1 There are multiple stages in the life cycle of an explosive substance or article. In-process includes all processing and handling operations except for storage and transport. **Figure 1** show these stages which are grouped together into three categories: in-process (blue), transport (white), and storage (yellow).

1.2 The in-process stages in Figure 1 can include a myriad of operations. Examples of some of these operations are outlined in **Table 1**. Each in-process operation has unique risks and potential initiation scenarios. For classification, key parameters include composition of the substance, physical state, configuration (confinement or packaging), quantity, and conditions (e.g., temperature, pressure, humidity, etc.).

Each of these parameters can vary with the respective in-process operation, thus the in-process classification can also vary. In contrast, typically the composition, physical state, and configuration are static during transport and storage.

Note: Originally published in 2003 by Safety Management Services, Inc. This updated standard (ETUG-GS01-15) was adopted by consensus by the Explosives Testing Users Group at the annual meeting in October of 2015 as was the Test Method Matrix™ (TMM). The TMM documents this approach in detail with pictures and videos and is located here: http://www.etusersgroup.org/test-methods-matrix.

Table 1: Example In-Process Operations

Manufacturing Stage	· Staging
· Feeding	· Setup

· Mixing	· Functioning
· Blending	**Demilitarization Stage**
· Extruding	· OB/OD
· Pressing	· Contain Burn/ Detonation
· Casting	· Segmenting
· Curing	· Super Critical Water Oxidation
· Cutting/ Machining	· Cryo-washout
· Assembly/ Disassembly	· Cryo-fracture
· Waste handling/processing	· Hydrolysis
· Intermediate storage	**Decontamination Stage**
Use Stage	· Explosive Operating Buildings
· Unpacking	· Test Facilities/ Sites
· Handling	· Test Ranges
· Assembly	

1.3 The in-process classification scheme has two main categories: substances and articles. The test series outlined below will yield an In-Process (IP) 1.1 or 1.3 for a substance or IP 1.1, 1.2, 1.3, or 1.4 for articles for a given operation. There are six series of in-process tests for substances and articles, Series 1 – 4

are for substances and Series 5 for articles:

1.3.1 Series 1 has two purposes: first to determine the sensitivity of the substance to ensure safe handling during subsequent tests and second to characterize the material for further risk assessment when comparing the in-process energies to the substance's response to such energies.

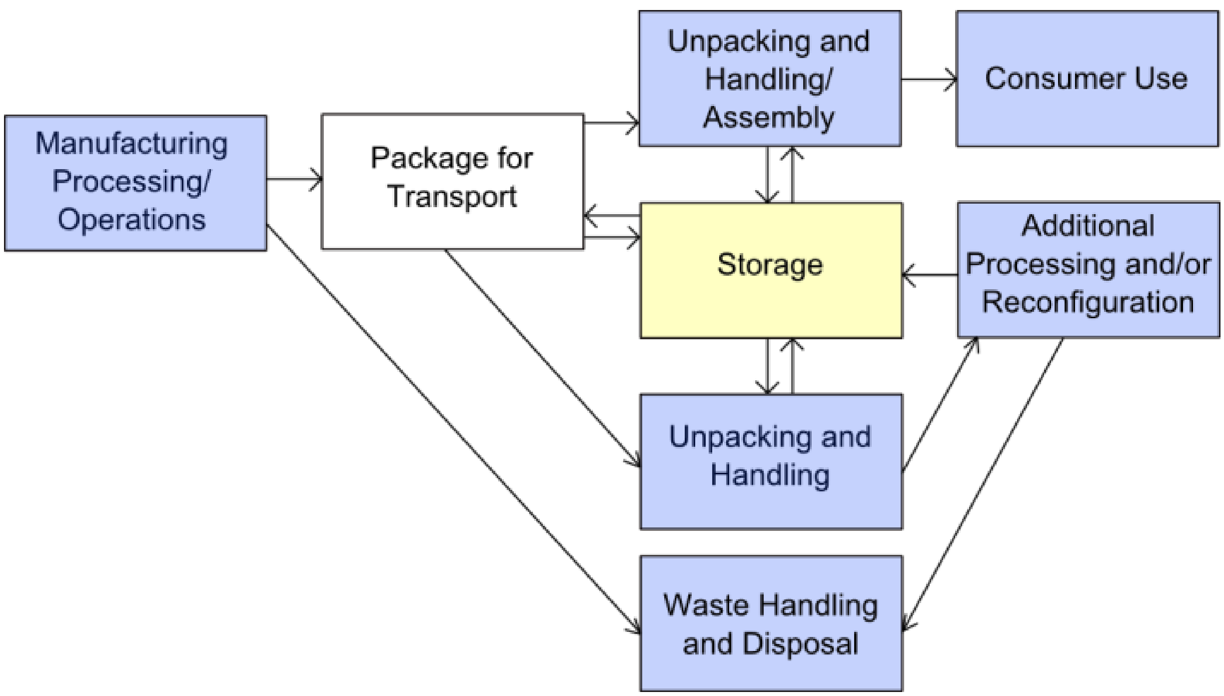

Figure 1. Explosive Life-Cycle: In-Process Stages Highlighted in Blue

1.3.2 Series 2 is equivalent to UN Series 1 in the Manual of Tests and Criteria and is to determine if a substance has explosive properties. This test series is not required for substances that are known to have such properties.

1.3.3 Series 3 determines if a substance can be considered for a classification other than IP 1.1.

1.3.4 Series 4 determines if the maximum credible event is a mass fire reaction (IP 1.3) when processing a substance.

1.3.5 Series 5 determines the hazard presented by in-process articles (IP 1.1, 1.2, 1.3, or 1.4).

1.4 **Figure 2** presents a flowchart for classifying substances and **Figure 3** is for articles. As stated above, unlike storage and transport, in-process substance or article composition, physical state, configuration, quantity, and conditions are specific to and can vary between operations. The in-process classification tests in each test series therefore incorporate both standard and modified United Nations Manual of Tests and Criteria (UN MTC), United States Department of Defense (US DoD), and United States Bureau of Alcohol, Tobacco, Firearms, and Explosives (ATF) classification tests where applicable and other tests accepted by industry. Risk assessment of each process operation facilitates the identification of credible worst-case composition, physical state, configuration, quantity, conditions and initiation scenarios (key parameters) that should be incorporated into in-process classification tests.

1.5 Note that characterization of the substance for safe in-process conditions is not treated here other than obtaining sensitivity data as a function of energy. Comparison of this data to the in-process energies is performed as part of a process hazards analysis/ process risk assessment. Such an assessment identifies failure scenarios, potential risks, safeguards, and recommendations for in-process risk reduction.

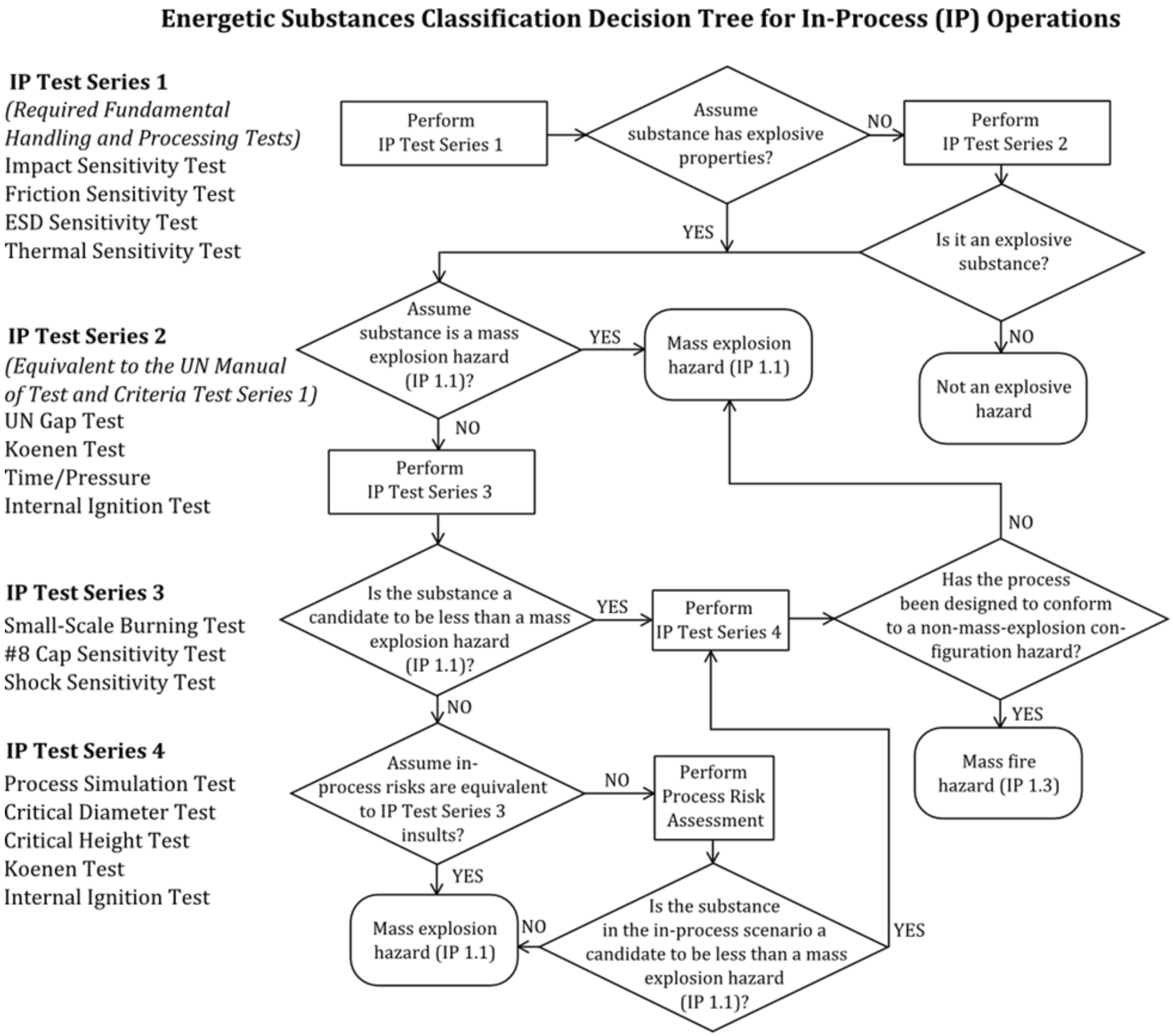

Figure 2: Energetic Substances Classification Decision Tree for In-Process (IP) Operations

2. Background

2.1 In the absence of a clear standard, UN MTC, US DoD, and ATF classification systems have sometimes been inappropriately applied to in-process conditions. It should be noted that an energetic material will typically provide the same or greater level of hazard within an in-process operation than in a transport or storage configuration. Therefore, the entities performing the in-process explosives operations may be under-assessing the hazard of the energetic materials in their processes by solely using the transport and storage classifications as a guide.

2.2 Alternatively, an entity performing in-process operations may assume that in-process operations always represent a high explosive hazard. This approach may lead to unwarranted restrictions or expense for some operations. This blanket approach may also lead to some unsafe practices, when an entity has experience with a less hazardous material and then switches to more hazardous material, both of which are identified as having the same level of hazard. Understanding the characteristics of the in-process material ensures that proper safety requirements are addressed early in the designing and planning stages of an in-process configuration.

2.3 The in-process protocol outlined in this paper, which utilizes standard or modified UN MTC, US DoD, and ATF test methods/protocols that are augmented by additional industry-accepted tests, can be used to produce proper in-process classifications. The intent is to perform testing that closely simulates key in-process parameters. Key in-process parameters include composition, physical state, configuration (confinement or packaging), conditions (e.g., temperature, pressure, humidity, etc.), and normal and abnormal ignition sources. These parameters are identified and defined by performing a proper risk assessment. These tests and criteria are applied to determine the hazard classification for the hazardous materials for a given in-process operation. This protocol is not intended to replace or modify UN MTC, US DoD, and ATF classification systems for transport or storage, but to assist the entity performing the in-process explosive operations with proper facility design and siting of modified or new facilities.

2.4 For an in-process explosive operation, the sensitivity or reactivity of the material may vary for each in-process operation. The in-process material classification may therefore be different for each operation. Therefore, the classification methodology should be applied to all in-process operations and reflect the credible worst-case operating parameters.

2.5 There are two main categories of explosives: substance and articles addressed by this classification scheme. Substances refer to the actual energetic material (powders, grains, pellets, etc.) Articles refer to items, which contain explosive substances (detonators, igniters, inflators, etc.) Both substances and articles can mass react depending on the explosive materials they contain, and the design configuration used. Different tests are needed to evaluate the propagation potential of substances and articles. The remainder of this paper is divided into two main sections addressing these two basic explosive categories. For an in-process operation, where the article is in-process, but the substance has been separated into individual units, a combination of the appropriate tests from the two sections is recommended.

2.6 When classifying in-process materials or articles, using process simulations, one must be careful to provide a worst-case configuration instead of a normal process upset or minimal-case configuration. It is also generally better to perform an over-test rather than a minimum worst-case test, so that every change in the in-process operating procedures does not require a re-test of the energetic substances or articles involved. In addition, adequate training and understanding of how the test results apply to specific process operations is critical. Otherwise, for example, if testing of a specific in-process condition or configuration reveals that the substance or article is an IP 1.4; personnel may misapply the IP 1.4 to the material regardless of the configuration found in other operations and the possible understate the potential hazard.

2.7 For classification purposes, one looks at both the sensitivity and reactivity of the substance or article. Sensitivity refers to how easily a material or article is initiated due to various stimuli. Reactivity refers to what type or size of event is produced once initiation occurs. In addition to in-process classifications as presented here, proper sensitivity and reactivity data for a substance or article coupled with risk assessment can also be used to improve the safety for explosives operations.

2.8 For example, sensitivity and reactivity data are used to determine and rank explosive hazards for the various in-process operations. Sensitivity data, when compared to the in-process potential, can determine the margins of safety and therefore the probability of initiation when performing in-process operations. Reactivity data is then used to determine the magnitude of an event should initiation occur.

3. In-Process Energetic Substances

3.1 The testing of energetic substances refers to the explosive, pyrotechnic or propellant substance, whether it is a molecular explosive, a mixture of ingredients, liquids, powders, pastes, emulsions, or combinations, etc. Only those tests required for in-process classification of explosive substances have been included below. As a reminder that these test series may differ from the those designated and used for transport classifications, the words "In-Process" have been used at the introduction of each test series.

3.2 Energetic Substances Classification Decision Tree for In-Process Operations

3.2.1 Figure 2 is a decision tree for classifying in-process energetic substances. when using the decision tree to classify in-process energetic substances, if a single test in Series 1 – 4 results in an explosive result, further testing in that series is not necessary. For example, in Test Series 3 (see Figure 2), if any of the tests are positive (a "go" reaction), the others need not be performed. A positive result in any one of the listed tests results in the determination that the substance is an explosive (Class 1 substance). Of course, one may elect to assume the worst-case result for any of the decision boxes rather than to perform the tests indicated in that box.

3.2.3 As shown in Figure 2, energetic substances found in in-process operations are either categorized as IP 1.1 or 1.3 explosives. An IP 1.2 designation, which implies a fragmentation hazard, has not typically been applied to substances. A Hazards Division 1.5 classification, which is designated as an insensitive, yet detonable substance, is difficult to justify for in-process operations where these materials may experience more initiation hazards than found in transport and storage applications. An IP 1.4 designation for substances is currently reserved for substances classified as Hazards Division 1.4 for transport and remaining in their original shipping container.

3.3 In-Process Test Series 1

3.3.1 Test Series 1 consists of tests that determine material characteristics fundamental in determining handling and processing hazards. Series 1 has two purposes: (1) to determine the sensitivity of the substance to ensure safe handling with larger samples during subsequent tests and (2) to characterize the material for further risk assessment when comparing the in-process energies to the substance's response to that energy.

3.3.2 There are two ways to characterize a substance to determine its sensitivity: (1) by comparison to a standard substance or (2) by a characterization curve that relates the energy to the probability of reaction. Transport classification depends primarily on comparison tests. In-process testing however primarily uses characterization curves to estimate the risk during handling and processing activities. Energies found while handling explosives at the test site or during in-process operations are compared to the characterization curves derived from performing the sensitivity tests in this series. It is important that the sensitivity test equipment used mimics key in-process parameters and repeatably controls the energy delivered to yield a probability of reaction as a function of impetus in engineering units.

3.3.3 Series 1 characterizes the impact, friction, and ESD sensitivity of the substance. Additionally the thermal sensitivity of the substance is obtained by completing an SBAT test that yields the exotherms and endotherms of the substance up to 350°C. Thermal screening that include a flame or hot-plate tests can also be completed.

3.3.4 The results from Series 1 testing coupled with a hazards analysis are used to safely handle and process the substance. The hazards analysis systematically evaluates the impact, friction, ESD, and thermal hazards in each in-process scenario. The data from Series 1 testing are compared to the normal and abnormal energy stimuli identified in a hazards analysis. A hazards analysis also identifies potential adverse consequences, existing engineering and administrative safeguards, and recommendations for mitigating the risk of in-process operations. As shown in Figure 2, energetic substances found in in-process operations are either categorized as IP 1.1 or 1.3 explosives.

Specific details of the Impact, Friction and ESD tests are found below and in the ET Users Group In-Process Document ETUG-GS01-15. Each test is completed at various energies or settings in engineering units to identify the transition from a no-reaction to a reaction. That plot or characteristic curve of the transition relates the probability of a reaction to the stimulus in in-process engineering units that can be easily related to the in-process potential. Such a test methodology facilitates quantitative hazards analysis when needed in addition to evaluating the sensitivity of the substance.

3.4 In-Process Test Series 1, Impact Test

3.4.1 Impact tests are used to determine the response of an energetic material when a moving mass impacts it. This test simulates impact scenarios found at the test site or in-process operations, wherein an energetic material is subjected to a collision between moving components of the processing equipment, by normal handling operations, or by the inadvertent dropping of tools or equipment.

3.4.2 Test machines are recommended that can mimic key in-process parameters, repeatably control the impact conditions from test to test, and can readily express the energy stimuli to the material in engineering units be used to test the material and obtain a characterization curve relating the energy stimuli to the reaction probability. The Modified Bureau of Mines (MBOM) Impact test methodology is recommended for in-process classification ("MBOM Impact Test" 2015; "MBOM Impact Test" 2018). Other equipment like the BAM Fallhammer test that can express the impact energy in engineering units could also be used ("BAM Fallhammer Impact Test" 2015).

3.5 In-Process Test Series 1, Friction Test

3.5.1 Friction sensitivity tests determine the response of an energetic material sample when subjected to frictional forces at a given velocity. This test simulates friction conditions that may occur in a process when an energetic material is subjected to a frictional force between moving components or during material handling or processing.

3.5.2 In the ABL Friction test the sample is placed on an anvil and pinched between the anvil and wheel each with specified surface finishes and materials mimicking in-process conditions. Force is applied to the anvil through a hydraulic ram attached to a stationary wheel. A pendulum strikes the anvil and it slides under the wheel. Observation is made as to the reaction outcome with human senses or an assisted method such as a gas analyzer or high-speed camera. Testing is continued at various friction settings to determine the characteristic curve relating the probability of reaction to the friction impulse. The in-process risk from friction initiation can then be estimated by comparison to the material response using engineering calculations ("ABL Friction Test" 2015; "ABL Friction Test" 2018; *"Department of Defense"* 2012, 5-3(c)).

3.6 In-Process Test Series 1, Electrostatic Discharge (ESD) Test

3.6.1 Although not typically a concern during transport (due to the materials being packaged), understanding ESD or electrostatic spark hazards for in-process scenarios is critical. A potential ESD hazard exists for in-process operations where machines or personnel may become charged and then discharge to an exposed energetic material. It should be noted that ESD testing is required by the US DoD for proper in-process classification of explosives handling operations.

3.6.2 ESD testing is used to determine the response of an energetic material when subjected to various levels of electrostatic discharge energy. The approaching needle method is most commonly used because it mimics in-process scenarios (ABL ESD). Electrostatic energy, stored in a charged capacitor at a given high voltage, is discharged to the test sample through an approaching needle. Sample reaction at a given test condition is evaluated by an operator or through use of operator-assisted devices such as a gas analyzer or high-speed camera with automatic reaction detection like GoDetect. Various algorithms can be used to obtain the characteristic curve for risk evaluation under in-process conditions ("ABL ESD Test" 2018; "Approaching Needle ESD Test" 2001).

3.7 In-Process Test Series 1, Thermal Sensitivity Test

3.7.1 In-process thermal tests determine the effects of time and temperature on energetic materials in the process configuration ("IP Thermal Test" 2018). Thermal tests generally consist of measuring a sample's response to either: (1) rapidly heating a milligram sample with a hot plate or flame to quickly determine its behavior upon ignition, (2) gradually increasing the temperature of a larger sample and recording the temperature at which the substance gives off heat (auto-ignition temperature), or (3) holding the substance at a given temperature for an extended period of time to evaluate stability at a given temperature (thermal stability).

3.7.2 Many different pieces of equipment can be used to obtain an auto-ignition temperature. The most conservative auto-ignition or thermal stability result is obtained when there are no heat losses from the sample, the sample is confined, and the heating rate is very slow. Some test equipment commonly used to determine the thermal sensitivity includes:

3.7.2.1 Accelerating Rate Calorimetry (ARC): Uses gram-sized quantities with high insulation, high confinement, with a heat-wait search routine. Auto-ignition temperatures from the ARC apparatus are typically the lowest.

3.7.2.2 Simulated Bulk Auto-Ignition Temperature (SBAT) (UN Test Method 3 (c) (ii)): Also has a high insulation value, gram-sized quantities, a slow heating rate, and the sample can be confined ("SBAT Thermal Stability Test" 2015).

3.7.2.3 Differential Scanning Calorimetry (DSC): Has a high rate of heat loss, milligram-sized quantities, and the sample can be confined.

3.7.2.3 Heating ovens: Variable insulation, quantities, confinement, and instrumentation.

3.7.3 With these variations between tests, there can be significant differences in the auto-ignition and thermal stability results. On-average, the SBAT gives auto-ignition temperatures just 9°C greater than the ARC. The DSC on the other hand typically yields auto-ignition temperatures 50°C greater than the ARC. The DSC provides a less conservative approximation of the auto-ignition temperature.

3.8 In-Process Test Series 2

3.8.1 The In-Process Series 2 tests are completed when the material is not assumed to have explosive properties or in other words if it is not known if the substance is an explosive. The test series is equivalent to the UN Manual of Tests and Criteria Series 1, with the exception that instead of testing the substance in the condition offered for transport, the in-process condition (or a worst-case scenario) is tested.

3.8.2 The tests determine if the substance has explosive properties such as propagating a shock or sustaining vigorous burning under pressure. The tests and the test details are not repeated here. They are found in Test Series 1 in the "Recommendations on the Transport of Dangerous Goods: Manual of Tests and Criteria," Sixth Revised Edition, United Nations, New York and Geneva, 2015.

3.8.3 If the substance fails any of the UN Manual Series 1 tests (shows explosive characteristics), it should be classified as an explosive (IP 1.1 or 1.3) for the in-process operation. If the material is shown not to have these explosive characteristics then the material may be classified as something other than an explosive.

3.9 In-Process Test Series 3

3.9.1 Test Series 3 is a compilation of tests to determine whether or not the material should be considered a high explosive (i.e. IP 1.1) or whether the material may be considered in a lower explosive category (e.g. IP 1.3). The test series contains three different tests: the Small-Scale Burning test, the #8 Cap Sensitivity test, and the Shock Sensitivity test:

3.9.1.1 Substances that fail the Small-Scale Burning test should be considered to have a mass or high explosion hazard (IP 1.1).

3.9.1.2 In in-process operations, a substance that fails the #8 Cap Sensitivity test (violently explodes) should be considered to have a mass or high explosion hazard. The #8 Cap Sensitivity test or the EIS Cap tests, as used by the UN MTC or US DoD are good tests for this purpose.

3.9.1.3 If the substance is sensitive to shock and there is a credible scenario for shock to impinge on the substance, the substance should be classed as IP 1.1. There are multiple tests to determine shock sensitivity including the UN Card Gap, NOL Card Gap, and EIS Gap. Typically, instead of placing the booster right next to the sample, an attenuator is placed between the shock source and the material such that the sample is subjected to a reduced shock event.

3.9.2 Should a material fail any of the Series 3 tests, it is considered an IP 1.1 substance. If the in-process credible worst-case initiation scenarios as defined by the process hazards analysis are such that the as tested shock or cap events are not credible, the substance still may be a candidate for an IP 1.3 designation.

3.10 In-Process Test Series 3, Small-Scale Burning Test

3.10.1 This test is used to determine if unconfined samples, once ignited, continue burning or transit to an explosion or detonation ("Small-Scale Burning Test" 2015; "Department of Defense" 2012, 5-3(f)). A bed of sawdust, containing small samples of test material, is ignited and monitored. The test is a "go" (positive) if explosion or detonation occurs. If the test results in an explosion, the substance is IP 1.1.

3.11 In-Process Test Series 3, Cap (No. 8) Sensitivity Test

3.11.1 The No. 8 Cap Test is used to determine susceptibility of energetic materials to detonation from the energy delivered by a standard detonator or #8 cap. Sample detonation is determined by examining the witness plate. The criterion for detonation (positive result) is that the witness plate is torn or penetrated ("Cap Sensitivity Test" 2015; "Department of Defense" 2012, 5-7(a)).

3.12 In-Process Test Series 3, Shock Sensitivity Test

3.12.1 This test evaluates if the substance will violently explode upon exposure to shock with approximately 70 kbar of pressure using the NOL Card Gap test with 70 cards or the EIS gap test UN Series 7(b). In both tests the substance is confined in a pipe and a pentolite booster is used with a PMMA spacer between it and the substance to attenuate the shock. The criterion for a "go" is that a clean hole is punctured through the witness plate. The test determines if a material will propagate an attenuated detonation ("NOL Card Gap Test" 1974; "Department of Defense" 1989, 5-2(j); "EIS Gap Test" 2012, 5-7(b)).

3.12.2 If the substance is sensitive to shock (at 70 cards) and there is a credible scenario for shock to impinge on the substance, the substance should be classed as IP 1.1.

3.13 In-Process Test Series 4

3.13.1 Series 4 tests further ensure that the in-process hazard is not an IP 1.1 type event. Not all tests in Series 4 are required to properly classify the in-process scenario as an IP 1.3. Series 3 tests should have already been completed and shown that the substance doesn't explode unconfined, is not cap sensitive, and is not overly shock sensitive in the in-process condition. However, if the substance doesn't explode unconfined when exposed to flame but may be cap or shock sensitive, a risk assessment can be completed and if it is shown that the normal and abnormal in-process energies are less energetic than the cap and shock scenarios tested in Series 3, Series 4 tests can be completed to potentially class the in-process scenario as an IP 1.3.

3.13.2 Similarly, the process risk assessment or hazards analysis also defines the normal and abnormal scenarios and the associated key parameters to be simulated in Series 4. Test conditions and configurations are identified for the associated tests to determine if a violent over-pressurization event occurs should the material be exposed to a fire or other initiation scenario. Series 4 tests evaluate those conditions including process simulation tests with the actual processing equipment during a worst-case condition to determine if the result is an IP 1.1 type event. Other tests include critical height and diameter to ensure the depth (or diameter) of the substance in process is not such that credible worst-case scenarios result in a violent explosion. The other tests (Koenen and Internal Ignition) are included to evaluate venting and confinement. Given there is not a scenario for the in-process condition to result in an IP 1.1 type event, the in-process classification is IP 1.3.

3.14 In-Process Test Series 4, Process Simulation Tests

3.14.1 In-process simulation tests evaluate those process operations and associated equipment where there is a chance for the confined or semi-confined substance to result in an explosion or an IP 1.1 type event. Tests are completed under conditions and worst-case in-process configurations, as identified through a risk assessment or process hazards analysis, with a safety factor. Test types may represent various available energy stimuli, including inadvertent bottom ignition/initiation, external fire (bonfire or fast cook-off), slow cook-off, bullet impact, etc. For example, the equipment used in the in-process operation, or an appropriate modification, is filled or tested in a worst-case scenario, typically by bottom ignition of the substance, and the resulting outcome evaluated for evidence of deflagration or explosion by video, instrumentation, or witness plates. Testing is typically completed such that a minor change in the process does not necessitate a retest. Should the results show that a violent explosion or over-pressure event does not occur, the in-process classification is IP 1.3 and no addition classification testing is required; otherwise, the configuration is IP 1.1 ("Process Simulation Tests" 2018).

3.15 In-Process Test Series 4, Critical Diameter Test

3.15.1 The critical diameter of an energetic material is the largest diameter at which steady-state detonation cannot be maintained. This test is typically completed for evaluation of process piping. The test uses varying diameter cylinders and a witness plate. A Comp C-4 or similar booster is used to initiate the sample. The test results are considered to be positive if the witness plate indicates propagation of a detonation. Normally, the test shall be completed after three "no-go" reactions are obtained at a diameter one increment below a diameter that previously yielded a positive result ("Critical Diameter Test" 2001; "Critical Diameter Test" 2018).

3.15.2 The in-process condition can be designed (or modified) to ensure that applicable process piping is not greater than the critical diameter or if it is, is appropriately classified as an IP 1.1 operation.

3.16 In-Process Test Series 4, Critical Height (Mass) Test

3.16.1 This test is used to determine the critical height at which a flame initiation transits to an explosive reaction (explosion or detonation). This test is sometimes referred to as a critical mass test because the results are dependent on the self-confinement provided by the mass of material. The test is used to evaluate the explosive risk of collections of the substance in hoppers or other similar equipment ("Critical Height Test" 2018). In this test, a flame initiator (match or bag igniter) is placed at the bottom of a pipe assembly filled with the test material. Pipes of varying lengths and diameters are used to contain the test material. The test is performed by selecting a diameter, and progressively changing the depth of the substance loading in the pipe until the material transitions from burning to explosion or detonation. The diameter is then changed and the progressive height variation testing is repeated. Normally, a curve can be fitted using the data, to predict the critical height for other diameters as well. A "go" reaction, for explosion, is one in which the pipe is damaged. The test is concluded at each diameter by running a minimum of three successive trials which produce a "no-go" result at a height below a level which produces a positive reaction (explosion). This level is referred to as the critical height at that diameter.

3.16.2 The in-process condition can be designed (or modified) to ensure that the height of material in the applicable process equipment is not greater than the critical height or if it is, is appropriately classified as an IP 1.1 operation.

3.17 In-Process Test Series 4, Internal Ignition (10-Gram Bag) Test

3.17.1 This test is similar to the internal ignition test in Series 2 except that a 10-gram bag igniter is used instead of the 20-gram bag. This test is used to evaluate the explosion risk of sealed vessels. As stated previously, either the pipe or at least one of the end caps must be fragmented into at least two distinct pieces for a positive result. Three trials are performed unless a transition from deflagration to explosion occurs earlier. The test determines if a material will explode or detonate when ignited under confinement ("Internal Ignition Test" 2015).

3.18 In-Process Test Series 4, Koenen Test

3.18.1 This test is used to determine the sensitiveness of a material to the effect of intense heat under vented confinement ("Koenen Test" 2015; "IP Koene Test" 2018). Results from this test can be coupled with modeling to estimate the venting required in-process to ensure against an IP 1.1 type event. In this test, the substance is placed in a steel tube with an orifice plate. The test apparatus is then placed in a protective steel box, and heated at a specified rate. A series of trials is conducted using different sizes of orifices. A "go" reaction is determined by examining the container. Conducting three successive "no-go" reactions with an orifice plate size above that which produced a positive result concludes the test. This orifice is called the limiting diameter. The limiting diameter may be used to evaluate the degree of venting required to avoid an explosion in the process.

3.19 Further Testing or Classification

3.19.1 Further delineation is usually made within some of the classifications provided here. Within the IP 1.1 category for in-process classification, for example, are primary explosives, secondary explosives, blasting agents, booster charges, and others. While such delineation is beyond the scope of this effort, much of the data necessary to identify these subdivisions is included in the tests identified above.

3.19.2 Some tests in addition to those presented here, which may also be appropriate, include: thermal analysis, compatibility, TNT equivalence, and others. Thermal tests provide the auto-ignition temperature, temperature of exothermic reactions, time-to-explosion, etc. Compatibility tests evaluate whether contact with certain chemicals (including such common things as perfume or tape) may cause undesirable consequences. TNT Equivalence evaluates the explosive output (overpressure and/or impulse) of an energetic substance to that of TNT, as used in facility siting and design.

3.19.3 A hazards analysis should apply the known data to the in-process operations. It can also be used to identify any additional data necessary to make process design decisions. For example, if a material is very sensitive, but is not very reactive, then appropriate handling and in-process precautions may be able to readily minimize propagation scenarios.

4. In-Process Articles

4.1 In-Process Articles Classification Decision Tree for In-Process Operations

4.1.1 In-process articles have different hazards in the above identified in-process stages than when stored or in transport. Series 5 tests evaluate the worst-case hazards of in-process articles to classify them as IP 1.1, 1.2, 1.3, or 1.4 depending on whether the primary hazard of the articles is mass explosion, fragmentation, mass fire, or no mass reaction. In-process classification differs from transport or storage scenarios although many of the tests below are modified versions of the UN MTC and US DoD common tests to determine the hazards of a packaged finished article. In order to assess in-process articles, which are not yet in their final transport configuration, modified versions of these tests in addition to others should be conducted. Figure 3 is a decision tree for characterizing in-process articles. This flowchart was developed by applying UN MTC and US DoD tests to in-process scenarios. These tests evaluate worst-case scenarios which can occur in in-process operations.

4.2 In-Process Test Series 5

4.2.1 Series 5 tests are used to determine the hazard presented by in-process articles (IP 1.1, 1.2, 1.3, or 1.4). Tests are completed under conditions and worst-case in-process configurations, as identified through a risk assessment or process hazards analysis, with a safety factor. The tests represent energy stimuli common to in-process articles in an in-process environment: exposure to heat (curing, conditioning), free-fall impact (falls from material handling equipment, handling error by personnel), ignition/initiation of a donor in-process article in a simulated or worst-case process configuration, and exposure to intense heating or flame (facility fire). The in-process articles are classed according to their highest applicable hazard based on the test results:

4.2.1.1 IP 1.1 if any test results in a propagating mass explosion.

4.2.1.2 IP 1.2 if any test results in a fragment hazard.

4.2.1.3 IP 1.3 if any test results in a mass fire hazard.

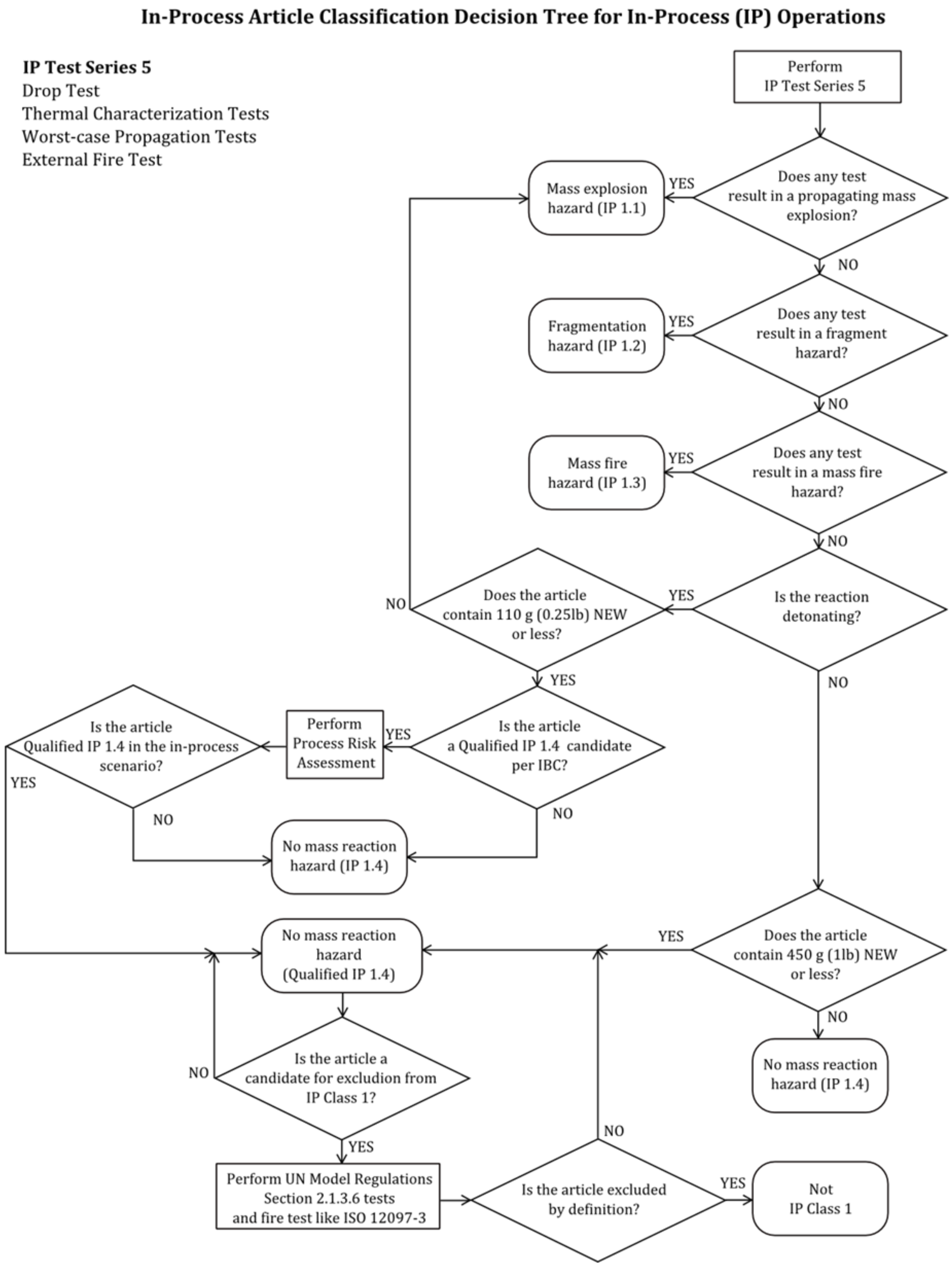

Figure 3: In-Process Article Classification Decision Tree for In-Process (IP) Operations

<u>4.2.1.4</u> IP 1.4 if no mass reaction hazard is observed in all tests and either 1) detonating articles contain 110 grams (0.25 lbs) net explosive weight each or less, or 2) deflagrating articles contain greater than 450 grams (1 lb) net explosive weight each.

4.2.1.4.1 IP "Qualified" 1.4 (see IBC Table 415.5.2 Note c) for deflagrating articles that contain 450 grams (1 lb) net explosive weight each or less and do not propagate a detonation or deflagration between articles. NOTE: The resulting hazard from a number of these articles is similar to that of 450 grams (1 lb) net explosive weight or less of unconfined IP 1.3 explosive in storage within the structure where operations are being performed since any reactions will be individual (not mass reactions) and any single event will involve 450 grams (1 lb) or less of explosive in a deflagration (including low-level explosions) but not in a detonation.

4.2.1.4.1 IP 1.1 for detonating articles that contain greater than 110 grams (0.25 lbs) net explosive weight each.

4.3 In-Process Test Series 5, Thermal Characterization Tests

4.3.1 These tests are used to determine an in-process article's thermal characteristics when subjected to worst-case normal/abnormal process conditions ("Thermal Characterization Test" 2018). The test conditions (test temperature(s), test duration(s), test quantity, sample configuration(s), pass/fail criteria, etc.) should reflect worst-case in-process conditions for defined failure scenarios identified in the process risk assessment with appropriate safety factors applied to test temperature(s) and duration(s).

4.3.2 Thermal sensitivity from Series 1 testing can also be used to evaluate the in-process risk of the substance(s) in the article. The process risk assessment or hazards analysis may also identify other thermal tests, such as the slow cook-off test for articles that may applicable for the specific process configuration.

NOTE: The UN MTC and US DoD Thermal Stability test for articles, performed at 75°C for 48 hours, is the prescribed test for determining the thermal stability of articles in transport. However, these test parameters for transport are likely not representative of the thermal stimuli available in a specific process.

4.4 In-Process Test Series 5, In-Process Drop Test

4.4.1 This test is used to determine whether an in-process article can withstand a free-fall impact without producing any significant fire or explosion hazards ("IP Drop Test" 2018). The UN MTC and US DoD 12-meter Drop test for articles, with a drop height of 12 meters, is the prescribed test for determining whether an article is too sensitive for transport. However, the drop height and test parameters for transport are likely not representative of the free-fall impact stimuli available in a specific process. Therefore, the test conditions (test quantity, configuration, temperature conditioning, drop height, impact surface, number of drops, pass/fail criteria, etc.) are defined by the process risk assessment or hazards analysis with appropriate safety factors. For example, a 1.2 meter (4 foot) to 1.8 meter (6 foot) drop height may be applicable for simulating handling error by personnel at ground level. Higher drop heights may be appropriate if the in-process articles are conveyed to higher/lower building levels with a drop potential from material handling equipment (conveyors, forklifts, elevators, etc.). Drop heights should be determined by the risk assessment.

4.4.2 The article is dropped at an orientation in which it is most likely to function on impact; if unknown, various impact orientations should be employed with the testing in the order of decreasing likelihood of producing a positive result. A minimum of three drops are made on identical units unless a decisive event (e.g. fire or explosion) occurs earlier. Typically, a positive result (i.e. fails the test) is a visible reaction of the energetics with effects apparent outside the in-process article. A rupture of the casing alone is not considered a positive result. Toxic vapors or other results outside the in-process article that may pose health or environmental hazards should be addressed.

4.4.3 An impact surface with a solid base and reasonably smooth surface (e.g., 75mm-thick steel plate on a 600mm-thick concrete foundation) may be representative of the worst-case scenario; alternative impact surfaces may be utilized as identified in the risk assessment.

4.5 In-Process Test Series 5, Worst-Case Propagation Test

4.5.1 This test is used to determine whether a reaction from an in-process article, which was accidentally fired or initiated, would propagate to other articles or parts of the process ("Stack Test" 2015; "Department of Defense" 2012, 5-5(c) and 5-5(d)). This test is conducted by placing articles in a worst-case configuration as defined by the hazards analysis (e.g. side-by-side, end-to-end, in a loose pile, within a container of robust construction, etc.). This test is similar to the stack test except that the articles are tested without packaging, as they are or may be found in the process, including during process upset. The in-process articles are placed on top of a steel witness plate. Sand-filled inert containers or sandbags may be positioned on the sides and top of the test articles for added confinement. Wire or clamps may be used to hold the articles in place. An article near the center of the articles is caused to function (donor). The test is assessed based on evidence of propagation of a mass explosion, fragmentation, mass fire, or no mass reaction. Normally at least two trials are conducted.

4.6 In-Process Test Series 5, External Fire Test

4.6.1 A stack of in-process articles in a worst-case operational configuration as defined by the process risk assessment is placed on a non-combustible surface (steel grate) above a lattice of dried wood soaked with kerosene or equivalent source ("External Fire Test" 2015; "Department of Defense" 2012, 5-5(e)). A wire basket or clamps may be used to hold the articles in place. Sufficient fuel is used to maintain a fully developed fire for 150% of the estimated time required to cause all articles to react. The test is assessed based on evidence of propagation of a mass explosion, fragmentation, mass fire, or no mass reaction.

5. Protective Enclosures

5.1 The use of technology to mitigate hazards is supported in civilian and military practice. Devices can be designed to fully contain or safely direct the deflagration and/or detonation effects of limited quantities of explosive material. Quantities of energetic material may thereby be afforded the same level of isolation and safety as provided by the traditional quantity-distance (Q-D) separations. A blast chimney is an example of these technologies. A blast chimney or other technology must be made to withstand the maximum possible event from the type and configuration of explosive contained within, if used in lieu of (Q-D) separations. Any overpressure discharged must be done so in a manner that safely directs or dissipates the effects thereof. A blast chimney or other technology must be made to withstand the maximum credible event from the type and configuration of explosive contained within, if used as workstation protection in lieu of separated or unattended operations.

6. Summary and Conclusions

6.1 Tests can be performed to determine in-process hazard classifications. Key test parameters are identified by a risk assessment to complete modified or standard UN MTC, US DoD, ATF, or other industry-accepted tests used for classification. Figures 2 and 3 are decision trees, which have been developed to aid code officials and users in classifying energetic substances and articles for in-process operations. Energetic substances in in-process operations are either categorized as IP 1.1 or 1.3 explosives. Substances which would otherwise be classed as Hazards Division 1.5 for transport should therefore be considered IP 1.1 for in-process classification. The IP 1.2 and IP 1.4 designations apply only to articles. The classifications of IP 1.1, 1.2, 1.3 and 1.4 are applied to in-process articles depending on whether the primary hazard of the articles is mass explosion, fragmentation, mass fire, or a minor event with no mass reaction.

6.2 Additional technology such as properly designed protective enclosures is a means to provide added safety or to minimize the effective quantity of an energetic material in an area. Also, further delineation is usually made within some of the classifications provided here. Additional tests may be necessary to obtain an adequate understanding of a material's explosive characteristics for purposes other than this in-process explosive classification system.

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