

**Committee of Experts on the Transport of Dangerous Goods
and on the Globally Harmonized System of Classification
and Labelling of Chemicals**

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**Recommendations made by the Sub-Committee
on its forty-seventh, forty-eighth and
forty-ninth sessions and pending issues:
electric storage systems**

**Possible categorization of lithium batteries for transport
according to their hazard and effects when reacting**

Transmitted by the expert from France¹

(Revised edition of the first study contained in INF.31)

STUDY REPORT

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**OPPORTUNITY OF HAVING A CLASSIFICATION
SYSTEM FOR THE TRANSPORT OF BATTERIES**

BASIC SPECIFICATIONS FOR A TESTING PROGRAM

INERIS

*maîtriser le risque |
pour un développement durable |*

Opportunity of having a classification system for the transport of batteries
Basic specifications for a testing program

Accidental Risks Division

People involved in the study: P. Goncalves

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1 INTRODUCTION

Within the frame of technical support performed by INERIS to the French administration regarding the transport of batteries, detailed results were obtained in 2013 from various batteries during abuse testing and test specifications were defined to potentially classify battery for transport. They showed that different behaviors were observed when the batteries were crushed, pierced or exposed to a fire.

It was then proposed, in accordance with the French Department of Transport of Dangerous Goods to look at the opportunity of having a classification system for the transport of batteries depending on the behavior when exposed to a fire.

The current report presents investigations performed by INERIS regarding the relevance of having a classification of batteries for transport and describes basic specifications for performing a testing program ahead.

2 RELEVANCE OF HAVING A CLASSIFICATION OF BATTERIES FOR TRANSPORT

This chapter presents a discussion on the relevance of having a classification of batteries for transport.

2.1 BATTERY MARKET

The origin of creating such a classification comes most notably from the great variety of battery types being transported in terms of their geometry, chemical composition and design which leads to varying behavior when batteries are subjected to accidental situations.

2.1.1 Geometry

In terms of cell geometry, four configurations¹ are currently used on the market (Figure 1):

- Cylindrical cell: generally used for small cells. It is constructed by superimposing anode-separator-cathode-separator bands which are coiled around a central pivot,
- Prismatic cell: used for capacity greater than 10Ah. Its housing is rigid with protective elements (e.g.: safety valve),
- Button cell: hardly used anymore for rechargeable lithium batteries,
- Pouch cell: have a soft housing which is sealed close to the electrode-separator stack and allows for potential warping due to internal cell pressure.



Figure 1: Various lithium cell formats: cylindrical, prismatic and pouch

¹http://batteryuniversity.com/learn/article/types_of_battery_cells

2.1.2 Chemical composition

Regarding chemical composition, we will differentiate cathode and anode materials in the study below.

Currently, five² cathode materials are mostly present on the market:

- Cobalt Oxide, LiCoO_2 (LCO): a material still widely used in current batteries due to its high energy density, its low self-discharge, but high cost (due to the Cobalt) and low thermal stability (runaway risk),
- Nickel-Manganese-Cobalt Oxide, $\text{LiNi}_{0.33}\text{Mn}_{0.33}\text{Co}_{0.33}\text{O}_2$ (most widely used formula) (NMC): used more and more (particularly in electric vehicles), replacing cobalt oxide thanks to its greater thermal stability and lower cost,
- Nickel-Cobalt-Aluminum Oxide, $\text{LiNi}_{0.8}\text{Co}_{0.15}\text{Al}_{0.05}\text{O}_2$ (NCA): used more and more for stationary or mobile applications thanks to its high energy density and its long stability over time, but has a relatively high cost.
- Manganese Oxide, LiMn_2O_4 (LMO): used more and more thanks to its lower cost relative to cobalt oxide cathodes, but has long-term cyclability which is inferior to other chemistries,
- Iron Phosphate, LiFePO_4 (LFP): used more and more thanks to its very high thermal stability and its capacity to withstand high power levels, but has low energy density resulting from lower usage voltage than the preceding oxides.

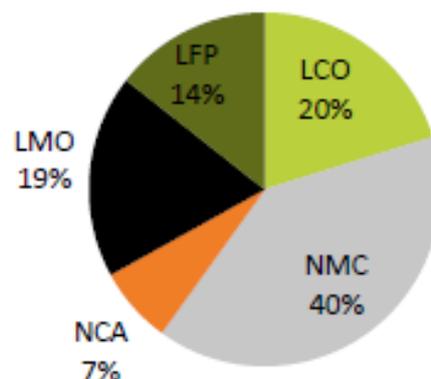


Figure 2: Lithium battery distribution forecast as relates to cathode material in 2020³

² N. Nitta, et al., Matter Today (2014)

³ The Worldwide battery market 2011-2015, Avicenne Energy Presentation, Batteries 2012

As for anodes, two materials² are marketed on a large scale:

- Graphite: anode material present in the vast majority of current lithium batteries, thanks to its great abundance on earth, its low cost and strong thermal conductivity,
- Lithium Titanate, $\text{Li}_4\text{Ti}_5\text{O}_{12}$ (LTO): used more and more thanks to its high thermal stability, its capacity to withstand strong currents and its long life, despite a much higher cost and lesser energy density than graphite.

Instinctively, the chemical composition could, during an external fire test, have an influence on the observed effects. Indeed, depending, for example, on the quantity of electrolyte and on the chemical compounds used, the quantities of gases produced and the fire intensity could widely vary.

Another type of lithium battery that is worth mentioning is the Lithium-Metal-Polymer battery developed by Batscap company and used in the BlueCar electric vehicle. This battery is made up of a metallic lithium anode and a vanadium oxide, carbon and polymer-based cathode.

2.2 STUDY OF THE RELEVANT EFFECTS FOR BATTERY CLASSIFICATION

During the various fire tests performed at INERIS, the following effects were observed:

- Toxic effect due to the emission of gases produced during combustion,
- Thermal effect due to heat released by the burning battery,
- Mechanical effect due to material spatter.

In light of the results obtained at INERIS, the effect of overloading does not seem relevant and need not be taken into account. Battery carriage classification could therefore be done according to relevant criteria (e.g.: emitted heat flux...) for the 3 effects listed above.

To determine the relevance of reaching a classification, the external fire tests previously done at INERIS must be analyzed to see if relevant classification criteria exist and to precisely define them. The following tests were done:

- At the cell, module, module stack or pack level,
- On items of varying chemistries (NMC, LFP...),
- On items of varying geometries (cylindrical, prismatic...).

2.2.1 Toxic Effect

Since the amount of gas emitted is an extensive variable, a first thought regarding toxic effects is that they are greatly dependent on the amount of energy of the sample subjected to the external fire test (Figure 3).

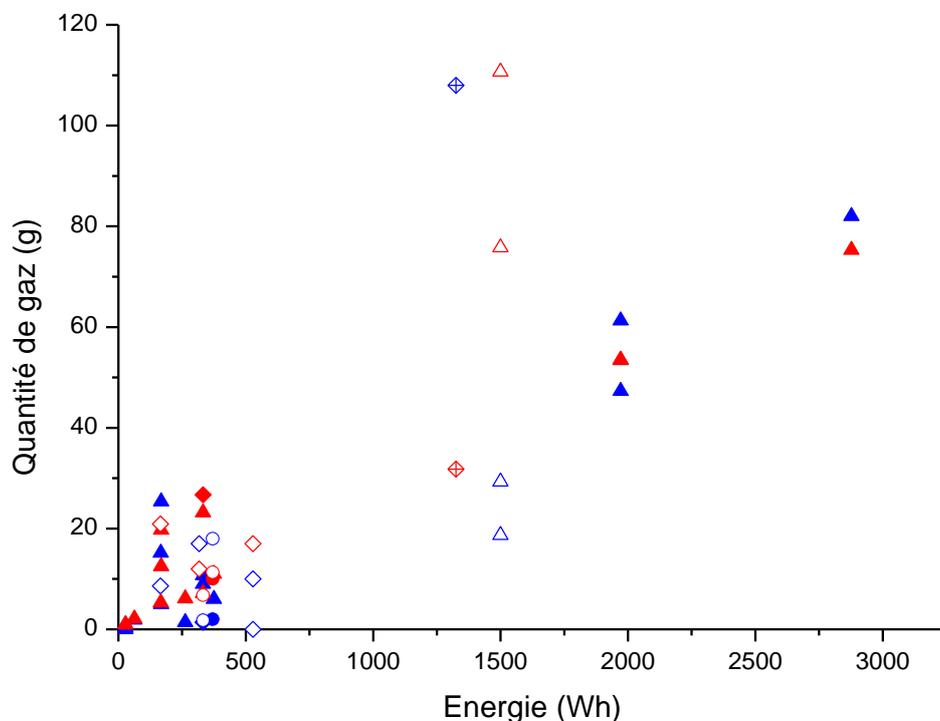
Indeed, within the framework of battery transport, the values of energy transported can vary enormously and are: in the order of:

- In the order of one Watt-hour for the transport of a few button-type or 18650 batteries,
- Up to several Megawatt-hours for, for example, a truck fully loaded with car batteries.

Any prospective classification will therefore need to take into consideration the amount of energy transported per unit of transport.

Figure 3 shows the amounts (in g) of carbon monoxide (CO) and hydrogen fluoride (HF) measured during the tests performed at INERIS as a function of:

- The energy of the tested sample,
- The geometry (*pouch*: full markers, *prismatic*: empty markers and *cylindrical*: locked markers),
- The chemical composition (*NMC*: triangle, *LMO*: round and *LFP*: diamond).



As expected, the analysis shows a strong disparity between total amounts of gas emitted during the various external fire tests performed at INERIS. We also observe a good relationship between the amount of emitted gas and the energy of the sample subjected to fire.

This result shows that a classification of batteries for transport as a function of their toxic effect during an external fire test could at first appear to be relevant. Indeed, the amount of transported energy will directly affect the amount of toxic smokes emitted during an accidental event and the transported load would be considered toxic or not depending on this amount of energy.

However, it would be inappropriate to consider only the amount of transported energy as a factor influencing a battery's toxicity when subjected to fire. To illustrate this, Figure 4 shows the various amounts of gas measured during the external fire tests performed at INERIS, expressed in energy units (mg/Wh).

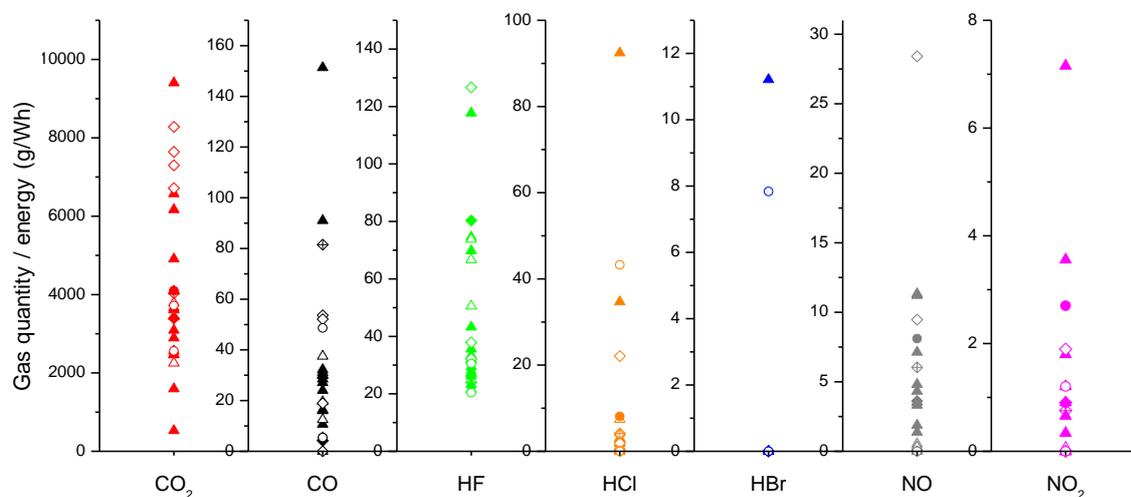


Figure 4: Amounts of various gases emitted, in energy units, during the cell, module or pack fire tests at INERIS

The results analysis shows a strong heterogeneity in the values of amount of gas emitted during an external fire test. A battery's toxic effect therefore strongly depends on a large number of parameters such as the amount of energy of the tested sample, the chemical composition, the sample structure and the tested batteries' various states of charge. One notices that for two samples sharing the same chemistry and geometry, the amount of gas emitted can vary by a factor of 10.

A classification of transported batteries as a function of their toxic effect when exposed to an external fire test therefore seems conceivable and relevant.

Future investigations would be necessary to go ahead and would consist of:

- Defining a reliable and sound test protocol to be able to test batteries and gather data needed to evaluate their toxic effect,
- Identifying the parameters relevant to classification,
- For these parameters, defining one or more thresholds that will allow the classification of batteries according to one or more toxicity classes.

2.2.2 Thermal effect

During external fire tests, the study of a battery's thermal effect is done using a fluxmeter placed at a certain distance from the tested sample. The value obtained from this measurement is a heat flux value (in kW/m²) received by the fluxmeter. By nature, this value is very dependent on the distance at which the fluxmeter is placed. Since all external fire tests done at INERIS were not performed according to the same protocol, the distance of the fluxmeter varied tremendously and the measured flux value could not be taken at face value to make a comparison between the batteries' thermal effects.

To be able to make such a comparison, one must be able to trace back to the power emitted by the flame produced by the tested battery. To do this, a model has to be chosen. The point source model⁴ where the flame is equivalent to a point source, centered on its axis, radiating with power equivalent to that of the entire flame (Figure 5) is a relevant model for this study.

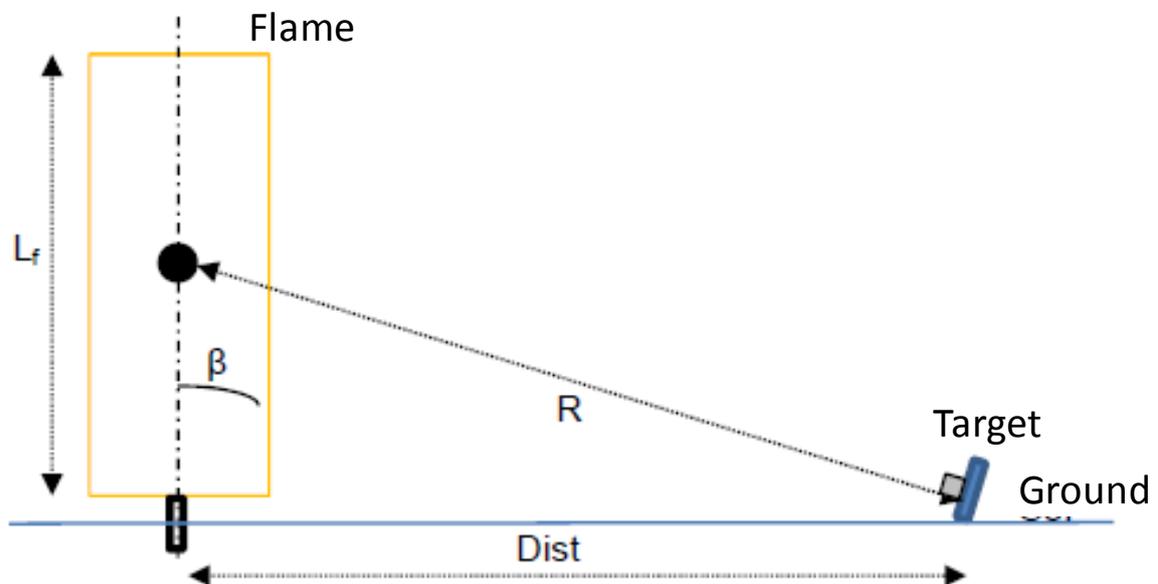


Figure 5: Point source model schematic

⁴ Report n° DRA-14-133133-02917A: Knowledge and tool formalization in the major risk area (DRA-76); Ω -8; Torch Fire: <http://www.ineris.fr/>

Radiative thermal flux density, Φ_c , received by a target placed at a distance R from the flame and perpendicular to the radiation at the point source is given by the following formula:

$$\Phi_c = a \frac{P_r}{4\pi R^2} \quad \text{Equation 1}$$

with:

- Φ_c = Flux received by the target, [W/m²]
- P_r = Power radiated by the flame, [W]
- R = Distance between the center of the flame and the target, [m]
- a = Atmospheric transmissivity or atmospheric attenuation factor, [-]
- $4\pi R^2$ = Solid angle of sphere with radius R centered on the point source, [-]

In our case study, the atmospheric attenuation value will be equal to 1 (upper-bound case) by making the following assumptions:

- The fluxmeter is positioned very close to the flame (a few meters),
- The tests are done in a closed and ventilated vessel chamber, therefore condensation in the air is considered to be non-existent.

During the external fire tests done at INERIS, the measured flux values are not constant with time and vary significantly. For our study, it was therefore decided to take the maximum value measured during the test. The power radiated by the flame with therefore be equal to:

$$P_r = \Phi_c(max) * 4\pi R^2 \quad \text{Equation 2}$$

As in the toxic effect study, a first thought regarding thermal effects is that they are greatly dependent on the amount of energy of the sample subjected to the external fire test.

Figure 6 shows the values of power radiated by the flame as measured during the test as a function of:

- The energy of the tested sample,
- The geometry (*pouch: full markers, prismatic: empty markers and cylindrical: locked markers*),
- The chemical composition (*NMC: triangle, LMO: round and LFP: diamond*).

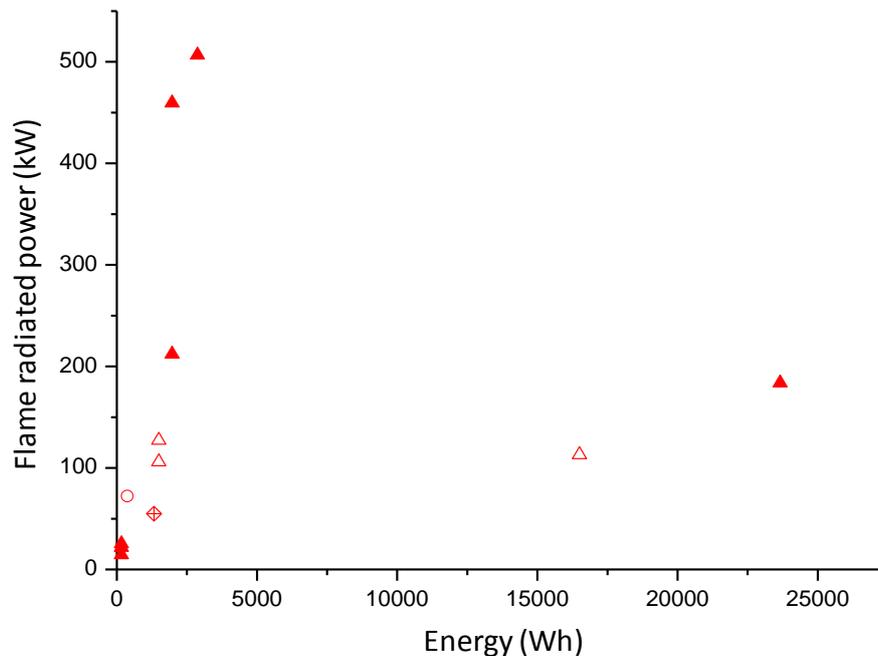


Figure 6: Power radiated by the flame of samples subjected to the external fire test at INERIS

Indeed, the analysis shows a dependence of the power radiated by the flame as a function of the energy of the sample subjected to fire. However, this dependence is much more pronounced at low energy levels (< 5000 Wh) than at high energy levels. Because the two samples were full battery packs and had energy levels above 15,000 Wh, it is likely that the fact that the active material (i.e. the modules) was isolated from the flame by an exterior metal casing caused the radiated power during these tests to be inferior to what would be expected at such energy levels. **In order to create a classification for battery transport, it therefore seems essential to handle single cell or module transport separately from full battery pack transport.**

In light of this result, a classification of batteries for transport as a function of their thermal effect during an external fire test could at first appear to be relevant. Indeed, the amount of energy and the type of merchandise transported (pack, cell, ...) will directly affect the amount of radiated power during an accidental event, and a transported load may or may not be considered a thermal risk depending on this energy.

However, it would be inappropriate to consider only the amount of transported energy as a factor influencing a battery's radiated power when subjected to fire. To illustrate this, Figure 7 shows the various amounts of gas measured during the external fire tests performed at INERIS, expressed in energy units (mg/Wh).

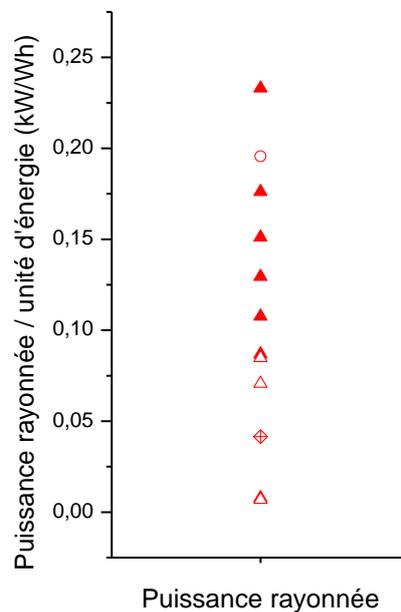


Figure 7: Emitted radiated power, in energy units, during the cell, module or pack fire tests at INERIS

The results analysis shows a strong heterogeneity in the values of amount of radiated power emitted during an external fire test. A battery's toxic effect therefore strongly depends on a large number of parameters such as the amount of energy of the tested sample, the chemical composition, the sample structure, the tested batteries' various states of charge... One notices that for two samples sharing the same chemistry and geometry, the radiated power per energy unit can vary by a factor of 20.

Significantly varying behavior was also observed as a function of the samples subjected to fire: single active elements (cells or modules) or full battery packs.

A classification of transported batteries as a function of their thermal effect according to the results of an external fire test therefore seems conceivable and relevant.

Further investigations would be necessary to go ahead and would consist of:

- Defining a reliable and sound test protocol to be able to test batteries and gather data needed to evaluate their thermal effect,
- Identifying the parameters relevant to classification,
- Defining one or more thresholds that will allow the classification of batteries according to one or more classes of thermal effect.

/!\ In light of the results above, the protocol and/or the thresholds which will be defined in the classification for battery transport may vary as a function of the type of item to be transported (cell, module, pack).

2.2.3 Missiles effect

During the INERIS external fire tests, only one tested sample projected material at significant distances. The sample for which the spatter effect was observed is the only sample of cylindrical geometry subjected to an external fire test at INERIS, which can lead to believe that this particular geometry is more prone to create missiles. Furthermore, according to a report⁵ published by the NFPA (National Fire Protection Association), this geometry presents the most risk for projectile danger. A more in depth study of this effect must therefore be done through a test campaign which INERIS will undertake to demonstrate, or not, the impact of this geometry.

At first glance, a battery transport classification as a function of missile effects according to the results of an external fire test seem relevant nonetheless, and future work within this operation will be to:

- Define a reliable and sound test protocol to be able to test batteries and gather data needed to evaluate the spatter effect,
- Identify parameters relevant to classification,
- If possible, define one or more thresholds that will allow the classification of batteries according to one or more classes of spatter effects.

⁵<http://www.nfpa.org/research/fire-protection-research-foundation/reports-and-proceedings/hazardous-materials/other-hazards/lithium-ion-batteries-hazard-and-use-assessment>

3 BASIC SPECIFICATIONS FOR PERFORMING A TESTING PROGRAM

3.1 METHODOLOGICAL APPROACH

Chapter 2 clearly demonstrates that the effects observed on batteries subjected to fire, in particular thermal and toxic effects, and to a lesser extent, missile effects, should allow the establishment of a classification method.

The development of this classification method could rely on the following steps.

Firstly, relevant parameters must be selected for each effect defined above. These parameters must be selected for their appropriateness as regards the objective of defining a classification system for battery transportation. As the principles for current tests in the Manual of Tests and Criteria of the Recommendations on the Transport of Dangerous Goods are such that they can be carried out by a large number of laboratories, the parameters selected for a prospective classification must also be easily measurable.

After having defined the relevant parameters for classification, threshold values must be recommended. These values will allow the classification of batteries according to one or more classes with regards to toxic, thermal or missile effects.

For toxic effects, we may rely on what is done in the classification for the transport of toxic and infectious substances (Class 6)⁶. Indeed, the classification of the substances is realized by using, as reference parameter, the gas concentration in the ambient air for a defined exposure time of 60 minutes.

For thermal or missile effects, we may rely on the work done in the classification for the transport of explosives (Class 1). Indeed, for the study of the thermal effect, an external fire test⁷ is performed using the thermal flux as the parameter used for the classification of the various samples tested.

For the missile effects, the external fire test⁷ was performed using three aluminum screens (size and type of aluminum were clearly defined) placed 4 m from the edge of the tested samples as a means of measuring the missile effect.

⁶ *Class 6: Toxic and infectious substances.* "Recommendations on the transport of dangerous goods, Model Regulations."

⁷ Test 6 (c): External fire (bonfire) test. "Recommendations on the transport of dangerous goods, Manual of Tests and Criteria."

Moreover, for toxic or thermal effects, the parameters chosen for the transport classification (gas concentration and thermal flux) are the same as the ones defined on a ministerial decree⁸ and used for industrial site hazard studies in France. Therefore, these parameters seem very relevant.

3.2 BROAD PRINCIPLES OF THE TESTING PROGRAM

As defined in the previous paragraphs, the protocol to be implemented in order to come up with a prospective classification of batteries for transport must:

- Be resilient, to allow the results to be reproduced,
- Be as simple as possible to implement in order to allow a maximum number of laboratories to carry it out,
- Allow the gathering of all data required to calculate relevant parameters to be defined within the classification context.

Preliminary thoughts on this test protocol are described in the paragraphs below.

3.2.1 Tested samples

Batteries are already subjected to a number of tests prior to authorization for transport:

- T1: Altitude
- T2: Thermal stability
- T3: Vibrations
- T4: Shock
- T5: Short-circuit
- T6: Impact
- T7: Overcharge
- T8: Forced discharge

All these tests are performed on batteries (cell, module or pack) without packaging. Nevertheless, during the external fire tests performed for the classification of other dangerous goods, the samples are tested with their transport packaging. Therefore, the test campaign at INERIS could be performed on batteries inside their packaging.

⁸ Order of 09/29/05 related to the evaluation and the accounting for the probability of occurrence, the kinetics and the intensity of the effects, and the severity of consequences of potential accidents in the hazard studies of classified installations and subjected to authorization

3.2.2 Fuel

As the external fire tests aim to achieve a classification for battery transport, the fuel to be used for the test must be consistent with this objective. Therefore, an automobile fuel (diesel or gasoline) will be used for the test.

3.2.3 Exposure time

In order to be the most representative of a most critical case, the battery will be completely on fire for the external fire test. The fuel fire will therefore be maintained under the tested sample until the battery is fully combusted. The end of combustion will be determined with the absence of flames on the sample.

3.2.4 Gas analysis

Gas analyses can be performed in several ways in order to reach a final total amount of gas:

- Continuous analysis: physicochemical methods, FTIR...
- Discontinuous analysis: bubbling...

To demonstrate measurement reliability during the tests to be performed at INERIS, continuous and discontinuous gas analyses will be done measuring the various gases, known to be toxic that may be emitted during a battery fire: CO₂, CO, HF, NO_x...

3.2.5 Thermal flux measurement

The thermal flux emitted by the battery fire will be measured with a fluxmeter placed at a certain distance from the tested sample. As the thermal flux value can vary greatly depending on the sample being tested, the distance at which the fluxmeter will be placed may also vary. However, to be able to assume the point source model (see §2.2.2), the distance must be greater than five times the equivalent diameter of the tested sample.

During the INERIS test campaign, several fluxmeters will be placed at different distances in order to verify that the different values measured will allow reverting back to an identical radiated power.

3.3 INERIS TEST CAMPAIGN

3.3.1 Testing program objectives

INERIS has already completed a large number of external fire tests (see §2). However, in order to be able to come up with a battery transport classification, a testing program must be carried out because, during the previous tests:

- The procedures used were not all identical and varied by client (fuel, battery state of charge...),
- Few tests were done with a fluxmeter to measure the thermal effect and most were done on cells which all had the same composition and geometry (pouch cells with NMC chemistry),
- No test was performed with a means to analyze missile effects,
- Very few tests were performed on cylindrical cells which make up a large part of the current battery market (especially for portable applications) and which are prone to missile effects.

The objectives of the future INERIS test campaign are therefore:

- To test the protocol to be implemented and prove that it is sound, reliable and that it will gather all the required data by repeating it several times on a same sample.
- To modify the protocol if it does not meet one of the conditions listed above and to test the new protocol, once again, on several identical samples,
- To study the impact of the battery state of charge on its response to the external fire test in order to define the state of charge to be used for the classification test protocol.
- To conclude on the impact of the battery's geometry and chemical composition by using a same test protocol for all tests.
- To check the impact of the tested sample's energy value on the batteries' toxic, thermal and spatter effects. This relationship will allow to conclude, or not, through a battery carriage classification test, on the possibility of performing a test on a sample whose energy level is inferior to the amount transported.
- To demonstrate that the cylindrical cells are indeed more prone than other geometries to spatter effects and to conclude on the relevance of including this effect in a prospective classification.
- To perform tests on a wide variety of batteries while being very exhaustive regarding the data measured (thermal flux, continuous gas analysis...), to define, once the data is gathered, the parameters most relevant to classification (thermal power or energy, total or kinetic amounts of gases...).

3.3.2 Supply of batteries

To perform this test campaign, batteries must be supplied. With the objective of obtaining a relevant classification for lithium battery transport, the tests must be performed on a representative current market sample, specifically in terms of chemistry and geometry (see §2.1). To perform the tests and to be able to observe the effects (specifically thermal or toxic), it was decided that the tested samples must have an energy level of at least 300 Wh.

Thanks to the study in §2.1, it seems that to represent actual market conditions, it is appropriate to obtain batteries with:

- Cylindrical, prismatic and pouch geometries,
- Cobalt Oxide, Nickel-Manganese-Cobalt Oxide, Nickel-Cobalt-Aluminum Oxide, Manganese Oxide and Iron Phosphate cathode materials,
- Graphite and Lithium Titanate anode materials.

Supply of Lithium-Metal-Polymer batteries is difficult because these batteries are manufactured by Batscap company for internal use only.

There are five cathode chemical compositions, two anode chemical compositions and three cell geometries to be tested. In order to be the most representative and the most exhaustive possible, and to reach the objectives detailed in the previous paragraph, the following test sequence is proposed which presents a forty-five tests program:

- Three tests with one same cell (cathode 1, anode 1 and geometry 1) with the same energy value to demonstrate the soundness and reliability of the test protocol. Here, we are assuming that the protocol will be modified twice and that the total number of tests for this sequence is nine.
- Five tests with the same cell as previously (cathode 1, anode 1 and geometry 1), but with varying states of charge in order to determine the state of charge value to use during the tests. Each test will be repeated twice, for a total of ten tests.
- Two tests with the same cell as previously (cathode 1, anode 1 and geometry 1), but with different energy assemblies to demonstrate, for the same cell, the relationship between the thermal and toxic effects and the energy of the sample subjected to fire. Each test will be repeated twice, for a total of four tests.
- Four tests on cells of varying cathode chemical composition (cathode 2, 3, 4 and 5) but with the chemical composition of anode 1 and the same geometry 1 to verify the impact of chemical composition on the effects. Each test will be repeated twice, for a total of eight tests.
- One test on a cell with a different anode chemical composition (anode 2) but with the chemical composition of cathode 1 and the same geometry 1 to verify the impact of chemical composition on the effects. This test will be repeated twice.
- Two tests on cells with the same chemical composition (to be defined) but with different geometries (geometry 2 and 3) to verify the impact of geometry on the effects. Each test will be repeated twice and will be performed on three selected chemical compositions, for a total of twelve tests.

3.3.3 Financial estimate

Suppliers have been identified for the following provision:

- Cylindrical, pouch and prismatic batteries for the LiFePO_4 cathode and a graphite anode,
- Cylindrical and pouch batteries for $\text{LiNi}_{0.33}\text{Mn}_{0.33}\text{Co}_{0.33}\text{O}_2$ and LiCoO_2 cathodes and a graphite anode,
- Pouch batteries for a LiMn_2O_4 cathode and a graphite anode,
- Cylindrical batteries for a $\text{Li}_4\text{Ti}_5\text{O}_{12}$ anode and a LiFePO_4 cathode.

The cost of cells, taking into consideration unforeseen events and the tests to be performed with the above-mentioned cells, is approximately € 20,000.

Suppliers have not yet been identified for the following provisions:

- Prismatic cells for two different cathode materials other than LiFePO_4 and a graphite anode,
- Cylindrical or pouch cells with a graphite anode and a $\text{LiNi}_{0.8}\text{Co}_{0.15}\text{Al}_{0.05}\text{O}_2$ cathode,
- Cylindrical cells with a graphite anode and a LiMn_2O_4 cathode.

In order to perform all the tests, the total cost of cell provisioning will therefore be approximately € 30,000.

The cost of developing the protocol and the performance of the tests within three test campaigns is estimated at € 160,000.

Processing the data from these tests is estimated at € 120,000.

The total operation is therefore estimated at € 310,000.

4 CONCLUSION

The study of INERIS external fire test results indicates classification of batteries for transport as a function of their toxic and thermal effects as being particularly relevant.

Regarding a classification as a function of missile effects, a more in depth study will have to be done to demonstrate the relevance of this criterion and to define a test protocol which would allow gathering all information required for a prospective classification.

No over-pressure effect was detected during the various tests performed at INERIS. This effect therefore does not appear to be relevant for the aimed classification.

In order to carry out this classification, the future work will consist in:

- Defining of a reliable and resilient test protocol (used fuel, gas analysis method, ...),
- Performing the tests on a representative current market sample,
- Defining the reference threshold values that will be used to classify the batteries.



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pour un développement durable*

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