The Italian Association of Chemical Engineering Online at www.cetjournal.it

A publication of

Guest Editors: Aleš Bernatík, Bruno Fabiano Copyright © 2022, AIDIC Servizi S.r.l. ISBN 978-88-95608-88-4; ISSN 2283-9216

VOL. 90, 2022

DOI: 10.3303/CET2290133

IGUS – 60 Years of Successful International Collaboration on Safety of Unstable Substances

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IGUS is an international group of scientific experts dealing with the explosion risks of unstable substances. It was set up in 1962 with the aim to harmonise test methods used by different countries to identify and quantify the explosive properties of unstable materials. Since then, its scope has widened to thermal stability, reactivity and flammability of all kinds of unstable and reactive substances.

On the occasion of IGUS' 60th anniversary in 2022, this paper gives a closer look at the IGUS history, its aims and objectives and its current organization. Recent developments in the field of hazmat classification are highlighted as well as expected future developments.

Note. The views and opinions expressed in this paper are those of the individual authors at the time this paper was drafted and are not to be regarded as the policy position of their respective organizations or countries.

1. Introduction

IGUS is the International Group of Experts on the Explosion Risks of Unstable Substances. This group brings together independent experts from all over the world that discuss the risks of unstable dangerous substances. The aim of the group is to exchange information on the behaviour of these substances and to improve and promote safety in their production, storage, transport and use. IGUS works in support of the international bodies dealing with dangerous goods regulations. Full members of IGUS work for governments and independent research organizations. Experts from industry can and do attend meetings by invitation regularly.

IGUS' 60th anniversary in 2022 gives reason to have a closer look at the IGUS history, its aims and objectives and its current organization. Most important to highlight however are the recent developments in the field of hazmat classification.



Figure 1: The IGUS logo

2. History

In 1959, the European Productivity Agency of Co-operation initiated an international co-operative study on the explosive properties and behaviour of unstable substances. In 1961, the Organisation for Economic Co-operation and Development (OECD) approved the creation of an expert working group which first met in Paris on 8 and 9 May 1962. This group was then named the 'International Group of Experts on the Explosion Risks of Unstable Substances' (IGUS). Now, 60 years later, this group is still very actively operating.

The history and work of IGUS have been extensively discussed by Owen and Von Zahn (1974) and by Thomson and Von Zahn-Ullmann (1988). De Jong et al (2012) did this on the occurrence of IGUS' 50th anniversary.

Within IGUS, considerable discussions have occurred on topics like rationalisation of test methods since its first meeting in 1962. The first major collaborative exercise occurred in 1965 when seven member countries (Belgium, Canada, Germany, France, the Netherlands, the United Kingdom and the USA) tested 32 reference energetic materials by their national test methods and compared the results. This was the first major initiative in establishing the basis for internationally harmonised testing of energetic materials. Since then, harmonisation has moved forwards substantially with classification schemes being developed by the United Nations and various modal regulations now being aligned in respect to transport. The Globally Harmonized System of Classification and Labelling of Chemicals or GHS (United Nations, 2017) is a good example of the strong demand for harmonisation of classification and labelling.

Since 2005, IGUS no longer operates under the umbrella of OECD. Being an informal group, it proved to be rather difficult for IGUS to meet the criteria that OECD set for its formal working groups. However, IGUS continues to operate according to the same principles as it did before.

3. Aims and objectives

The main aim of IGUS is to support the development of adequate regulations and test methods in the field of hazardous substances, especially when aspects like explosive properties, thermal (in)stability, flammability and reactivity are involved. IGUS does so by stimulating the exchange of information and supporting international developments. The group itself is in the first place a group of experts that work together on the basis of their personal expertise, free of any political or economic interest. As such, the group does not directly participate in the work of the United Nations Committee of Experts on the Transport of Dangerous Goods (TDG) and the Globally Harmonized System (GHS) but during its 60 years of existence it has contributed to many improvements of test methods and criteria that are included in the Manual of Tests and Criteria (United Nations, 2017).

Over the years, IGUS has continued to offer scientists who are advisers to their governments, and others, a forum to exchange data and information. This exchange takes place primarily in support of the development of classification regimes to protect society from hazards of unstable substances and to remove barriers to international trade.

The aim of IGUS is defined as follows:

Exchange of information and thoughts on:

- prediction of explosive properties of industrial chemicals,
- · phenomenology of explosions,
- testing with regard to fire and explosion hazards, and
- · analysis of accident case histories,

in order to improve safe manufacture, handling, transport and storage of various kinds of dangerous materials. Schemes and criteria for the classification of dangerous substances come about through the negotiations of policy makers at meetings of international bodies responsible for agreements. These policy makers and international bodies are supported by technical experts. Whilst these experts support their colleagues' national positions, it is also extremely important that these experts have the means to exchange and discuss freely technical concerns in detail so that progress can be made at such meetings.

There are relatively few organisations where technical experts can share views and opinions at a formative stage and exchange information that benefits their institutes programmes, but IGUS is one such body. The relationships and respect among the members built up over the years enables valuable interchange to continue. Increasingly, the attendance of policy representatives and inspectors at IGUS meetings as well as joint open meetings with industry and universities speak for itself in respect to benefits perceived.

The working group meetings of IGUS are well-known for their open and friendly atmosphere and vivid

4. Organization

Currently, two working groups operate under the umbrella of IGUS:

Energetic and Oxidising Substances (EOS). This group deals with test methods, classification and safety aspects related to organic peroxides, self-reactive substances and other energetic substances, fertilisers, ammonium nitrate and oxidisers.

Explosives, Propellants and Pyrotechnics (EPP). This group deals with explosive properties, general test methods and thermodynamic ratings, phenomenology of explosions, safety and regulatory aspects related to explosives, pyrotechnics and propellants. This groups usually meets in conjunction with the annual meeting of the Chief Inspectors of Explosives (CIE).

Members of IGUS are experts from the OECD countries insofar as they are members of government laboratories and organisations or scientists who are involved in research and are governmental advisers on a permanent basis. Scientists from non-OECD countries may apply for membership as independents if similarly engaged. The working group meetings are also open for experts from industry, after being invited by the chairman, in some occasions closed sessions without industry participation are held.

Members participate at IGUS meetings as technical experts in their own right. They cannot commit their country's policy department in any way.

At the IGUS plenary meeting held every four years, national delegates elect the IGUS officers for the following four years. At the end of their term, they can be re-elected. The officers are nominated by their respective working groups and their appointment is confirmed by the plenary meeting. Together, the officers form the IGUS Steering Committee. IGUS officers for the 2020-2024 term are:

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EOS chairman	Wim Mak	TNO	The Netherlands
EOS co-chairman	Marcus Malow	BAM	Germany
EPP chairman	Ed de Jong	TNO	The Netherlands
EPP co-chairman	Shulin Nie	MSB	Sweden
IGUS secretary	Anna Martinez	HSE	United Kingdom

The IGUS website can be found at www.igus-experts.org.

5. Results and developments

Several members of IGUS act as scientific advisors to their national delegations at the UN Committee on TDG and GHS in Geneva (see unece.org/committee-experts-tdg-and-ghs). They have been able to use the outcome of discussions within IGUS as input for their national delegations. Several topics that were discussed and decided on in the UN meetings have been thoroughly discussed within IGUS and in a number of cases ad-hoc working groups under IGUS have worked on the preparation of proposals and informal documents that were submitted to the UN Committee. IGUS itself has no formal status at the UN Committee and cannot submit documents directly but proposals based on work done within IGUS have been taken to UN by different national delegations. The UN Committee has recognized the relevance of the work of IGUS.

5.1 Widening the scope of the UN Manual of Tests and Criteria

Since the introduction of the GHS as well as for instance the European CLP Regulation (European Union, 2008), the test methods that are included in the UN Manual of Tests and Criteria have gained importance. TDG and GHS classification now both rely on the same set of test methods, i.e. those included in the UN Manual. Therefore, the UN Manual was amended to accommodate not only TDG purposes but also GHS. For a part, this was a straightforward exercise as for instance all references to "packing group" (i.e. TDG only) were replaced by "packing group/category" (i.e. TDG and GHS). A more extensive exercise was to add references to the purple GHS book in those places where reference to the UN Model Regulations was made. Editorial amendments were necessary in numerous other places. Individual members of IGUS have participated in this work.

5.2 Classification of ammonium nitrate based fertilizers

Since the previous revision of the classification procedure for ammonium nitrate based fertilizers (ANBF) more than 20 years ago, this procedure was covered by two UN numbers, namely 2067 and 2071, and by the Special Provisions 186, 193, 306 and 307. It was noted however that the existing set of criteria quite often gave rise to misunderstanding and misinterpretation, leading to incorrect classification of ANBF. Even cases of deliberate misinterpretation and non-classification have been identified.

An ad-hoc working group of IGUS-EOS was tasked to improve the existing classification criteria and to harmonize the descriptions under the existing special provisions. No new criteria were to be added. The work took a couple of years and was done in cooperation with experts from industry.

The work resulted in the introduction of a dedicated flowchart and accompanying text as Section 39 of the UN Manual of Tests and Criteria. This is considered as a major step forward in promoting the safe transport of ANBF.

There are some issues that still need to be dealt with, see Section 6.5.

5.3 Review of GHS Chapter 2.1 on explosives

When the GHS was set up, the matters concerning to physical hazards were referred to TDG due to their experience when dealing those properties. Basically, the classification system for transport was transposed to GHS. However, since the transport classification is largely dependent on the packaging and GHS covers all sectors, such as storage and use, measures were required to address the hazards when explosives are no longer kept in their packaging. Furthermore, GHS applied the term 'unstable explosives' for substances and articles that failed test series 3 or 4, which was a misleading and incorrect term. Under the leadership of Sweden and with many IGUS members participating, a new system for the GHS application was set up for explosives that did not have a hazard division assigned. This term is used to make clear that the product is outside the transport packaging. The longstanding GHS practice of assigning to 'categories' is now also used for explosives. There are four categories: 1, 2A, 2B and 2C with corresponding criteria.

5.4 New method for the classification of solid oxidizers

Since long, UN test O.1 has been the method for the determination of oxidizing properties of solids. In this method, a potentially oxidizing solid is mixed with cellulose and, upon ignition, the burning time is determined as a measure for the oxidizing capability. This method has two major drawbacks: it is based on visual observation of the burning time, which is operator-dependent, and the reference substance potassium bromate is suspected carcinogenic. Years ago, within IGUS-EOS the discussion on a replacement was started and an ad-hoc working group was initiated.

After years of discussions and Round Robin studies, a new method has been introduced as UN test O.3. This method applies gravimetric determination of the burning time and it uses the non-hazardous calcium peroxide as reference.

For the time being both methods can be used for classification although it may be expected that the O.1 method is gradually phased out in future. At this moment, the O.3 method is still subject to further finetuning at UN level and in an ad-hoc EOS working group.

5.5 Revision of UN Test Series H

For a couple of years, an IGUS-EOS ad-hoc working group has discussed a necessary update of UN test series H. These test series comprise the methods for the determination of the self-accelerating polymerisation temperature (SAPT) and the self-accelerating decomposition temperature (SADT). It was noted that the test descriptions were not very up-to-date, that some equipment was prescribed very precisely while other equipment operating according to the same principles could be used as well and that the general introduction could easily lead to misunderstanding and misinterpretation.

In fact, test series H might well be the most challenging part of the UN Manual as its application requires a considerable level background knowledge as well as very specialized equipment. It was therefore attempted to improve the readability of the text and refine e.g. the important heat loss criteria.

Apart from updating the test descriptions and deleting over-specification, the following aspects were dealt with:

- For the Adiabatic Storage Test (test H.2, AST): to make the test description and the description of the equipment to be used more general and the introduction of a pressure adiabatic storage test.
- For the Isothermal Storage Test (test H.3, IST): to make the test description and the description of the equipment to be used more general.
- The introduction of standard heat-loss values for packagings and intermediate bulk containers (IBCs).

The proposal made by the ad-hoc working group were adopted by the UN Committee of Experts and introduced into the 7th edition of the UN Manual.

6. Future

As new technologies and new products emerge constantly, there will be a continuing need for technical discussions and exchange of knowledge to support international developments as well as sharing the knowledge about chemical hazards. Moreover, the chemicals business has become a truly global business with many countries involved. Therefore, reasonable international harmonization of test methods and criteria remains a very important issue as this may help removing barriers for trade.

The basis and principles of the current classification schemes were established in the 1960's. Since then, a lot has changed. New products have appeared on the market. Very often these products are not straightforward

compounds anymore but rather complex compositions. Existing products found new applications. During the past two decades or so, many chemical products were introduced on the market that do not fit well into the current classification scheme. Thus, further rationalisation of these schemes is really necessary. Also, incidents that occurred during transport or storage have taught us many lessons that need to be applied into the existing regulations and criteria. Last but not least, test methods and criteria that were state-ot-the-art when they were developed and introduced need further finetuning.

As far as we can see now, there are a couple of relevant issues that need to be resolved within the current decade.

6.1 Classification of polymerizing substances

Since a couple of years polymerizing substances have been recognized as a separate category of hazardous substance (UN numbers 3531 to 3534). This was initiated - due to different incidents happening e.g. with M/V Flaminia. The criteria for classification have been included in Special Provision 386. The main criterion for classification of such substances is the determination of the self-accelerating polymerisation temperature (SAPT) which is defined in analogy with the self-accelerating decomposition temperature (SADT) of organic peroxides and self-reactive substances.

The current classification criteria for polymerizing substances have some drawbacks that still need to be addressed:

- The new criteria do not apply to polymerizing substances that already have been classified in another hazard class (e.g. transport class 6 or 8). Thus, requirements for temperature control (if necessary because of thermal instability) that exist for 'new' substances do not exist for 'old' substances.
- The concept of SAPT was copied from the concept of SADT. While the SADT does not need to take into
 account aspects like loading temperature and stabilizer depletion these are aspects that are overlooked for
 in the application of the SAPT.
- The criteria in Special Provision 386 leave room for different interpretations.

6.2 Screening methods

Appendix 6 of the UN Manual of Tests and Criteria comprise a number of screening criteria for energetic substances. These criteria are very relevant as they can help to avoid unnecessary testing. These criteria were sometimes chosen on basis of intuition and best available knowledge at that moment. Since their introduction some 25 years ago, no further finetuning has been done. It can however be argued that some criteria may be overly conservative, e.g. the 500 J/g decomposition energy threshold for potentially explosive substances. Thermal analysis (TA) methods like Differential Scanning Calorimetry (DSC) are only briefly mentioned while significant knowledge has been gained since then about the proper application of TA methods and the use and application of TA results in calculation and modelling. This is a topic that is currently taken up in one of the existing ad-hoc working groups under IGUS.

6.3 Classification of energetic substances

Roughly, energetic substances (apart from organic peroxides) are either classified as explosive (transport class 1) or as self-reactive (transport division 4.1). Apart from the latter, there are a few substances that were identified as 'substance related to self-reactive substances' in the past but this designation has unfortunately been deleted many years ago. While these two possibilities probably were sufficient 60 years ago, we see more and more new substances that do not fit well or were the current criteria could lead to peculiar classifications. For instance, a rapidly deflagrating substance that is thermally stable needs to be classified for transport in class 1. If the same substance would have been thermally unstable (SADT below 75°C), it could have been classified for transport into division 4.1 that is usually more easy to transport than class 1. Also, substances that are provisionally accepted in class 1 but escape via test series 6 sometimes end up in division 4.1 as flammable solid while this not properly reflects their hazardous properties.

Many years ago an attempt was made to introduce dedicated UN numbers, like e.g. 'energetic substance n.o.s.' or even a separate division 4.4. This never came through but given the number of substances with functional groups (and thus potentially energetic) appearing on the market that may have some explosive properties but do not need to be classified as an explosive substance, further developments in this direction are urgently needed

6.4 Transport of energetic samples

Energetic materials that because of screening test results are considered to have potentially explosive properties, need to be tested according the UN Manual of Tests and Criteria, test series 1 and 2. These test series require a few kilograms of sample. The issue is that during the development stage of new substances

such large amounts are usually not available. Moreover, quite often such substances are for instance pharmaceuticals that may cost thousands of euros per gram. Last but not least, unlike for organic peroxides and self-reactive substances, there are no provisions for the transport of samples of explosive substances for the purpose of testing.

These observations underline the need for an enhancement of the screening procedures as well as the introduction of provisions for the classification and transport of energetic samples.

6.5 Classification of ammonium nitrate based fertilizers

As mentioned in Section 5.2, the classification criteria for ammonium nitrate based fertilizers (ANBF) have been rationalised some years ago. The remit of the ad-hoc working group that dealt with this topic was limited to a revision of the existing criteria and entries. No new criteria could be introduced. However, the new flowchart (UN Manual of Tests and Criteria, Section 39) still needs some finetuning and further rationalisation. There are some open ends, e.g. criteria or guidance is missing for fertilizers that are not classified as UN 2067 or 2071. Modern fertilizers like coated slow-release fertilizers have not been considered at all. An n.o.s. entry for fertilizers that need to be classified into class 5.1 but do not fit into the definition for UN 2067 is missing.

7. Conclusions

During the 60 years of its existence, IGUS has offered its members a unique forum for discussing necessary developments in the field of hazardous materials classifications. Members of IGUS have contributed to a number of new or revised classification criteria and test methods that appeared in the UN Manual of Tests and Criteria. In view of the ongoing international economic and scientific developments there is a continuous need for finetuning existing principles and developing new criteria and methods. Some topics that require attention are the classification of polymerizing substances, preliminary screening methods, the classification of energetic substances, the transport of potentially explosive samples and the classification of ammonium nitrate based fertilizers. IGUS will continue to play a role at the forefront of these developments.

Acknowledgments

The information in Sections 2 and 3 is partly extracted from a lecture held by Dr. B.J. Thomson (1998, 1999). Dr. Thomson was chairman of IGUS from 1985 till 1999. His lecture dealt with the history and achievements of IGUS.

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