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COMITÉ D'EXPERTS DU TRANSPORT DES MARCHANDISES DANGEREUSES ET DU SYSTÈME GÉNÉRAL HARMONISÉ DE CLASSIFICATION ET D'ÉTIQUETAGE DES PRODUITS CHIMIQUES

Sous-Comité d'experts du transport des marchandises dangereuses

Vingt-neuvième session
Genève, 3-12 (matin) juillet 2006
Point 6 de l'ordre du jour provisoire

INSCRIPTION, CLASSEMENT ET EMBALLAGE

Transport de nitroguanidine humidifiée (n° ONU 1336) en grands récipients pour vrac (GRV) souples

Communication du Conseil international des associations chimiques (ICCA)

Rappel

1. La NITROGUANIDINE (PICRITE) HUMIDIFIÉE avec au moins 20 % (masse) d'eau (n° ONU 1336) est une matière explosible désensibilisée solide qui est classée dans la division 4.1, GE I.

2. Actuellement, cette matière peut être emballée conformément à l'instruction d'emballage P406, dans des emballages combinés ainsi que dans des fûts en plastique, en contreplaqué, en carton ou en métal, etc. Ni le Règlement type de l'ONU, ni les règlements modaux ne contiennent d'instruction d'emballage permettant d'emballer et de transporter le numéro ONU 1336 en GRV.

3. À la vingt-septième session du Sous-Comité, l'ICCA a proposé d'autoriser le transport de nitroguanidine humidifiée (n° ONU 1336) en GRV souples en modifiant une nouvelle instruction d'emballage IBC4xx (ST/SG/AC.10/C.3/2005/10 et INF.15). La proposition a été révisée en fonction des observations formulées par les représentants, et des résultats d'épreuves supplémentaires ont été fournis.

Introduction

4. La NITROGUANIDINE (PICRITE) HUMIDIFIÉE (n° ONU 1336) est une matière première importante pour l'industrie chimique. Le marché mondial pour cette matière est estimé à environ 7 000 tonnes avec une tendance à la hausse. De telles quantités nécessitent des procédures efficaces de transport, de stockage et de manutention. Cette demande peut être satisfaite au moyen d'emballages de plus grande taille qui permettent en même temps aux milieux professionnels d'accroître la sécurité sur le lieu de travail.

5. Le transport de nitroguanidine humidifiée en GRV souples s'effectue en Allemagne dans des conditions de sécurité depuis plus de 15 ans en vertu de dérogations nationales.

6. Cette pratique a été autorisée par le *Federal Institute for Materials Research and Testing* (BAM) sur la base d'épreuves complètes (essentiellement les séries d'épreuves 1, 2, 5 et 6 ainsi que des épreuves de stabilité et d'homogénéité du contenu en eau).

7. Selon la section 2.1.3.6.2 du Règlement type de l'ONU, une matière exclue de la classe 1 sur la base de la série d'épreuves 6 devrait être inscrite dans la liste des marchandises dangereuses avec une disposition spéciale indiquant le type d'emballage et la quantité maximale autorisée.

Propositions

8. Insérer «xxx» dans la colonne 6 pour le numéro ONU 1336.

9. Insérer dans le chapitre 3.3 la nouvelle disposition spéciale xxx ci-après:

xxx Cette matière peut aussi être transportée en GRV. Seuls les GRV souples (13H3, 13H4, 13H5, 13L3 et 13L4) satisfaisant au niveau de performance du groupe d'emballage I sont autorisés, dans les conditions suivantes:

i) La matière doit être approuvée par l'autorité compétente en ce qui concerne le mode et la durée du transport sur la base des résultats d'épreuves appropriées, de sorte que la quantité d'eau ne puisse être inférieure à 20 % et soit à tout moment répartie de manière homogène pendant le transport;

ii) Le GRV doit satisfaire aux dispositions générales de **4.1.1** et **4.1.2**;

iii) Le GRV doit être conçu et fabriqué pour empêcher la perte d'eau;

iv) La masse nette du GRV ne doit pas dépasser 1 000 kg;

v) Les dispositions en matière de séparation, pour chaque mode de transport, doivent reposer sur les principes énoncés dans la sous-section 7.1.2.3;

vi) Les GRV doivent être transportés dans des engins de transport fermés;

vii) Les GRV doivent être chargés sur l'engin de transport de manière à prévenir les frottements entre les GRV eux-mêmes et entre ces GRV et la paroi de l'engin de transport.

Justification

10. La «NITROGUANIDINE HUMIDIFIÉE avec au moins 20 % (en masse) d'eau» satisfait aux critères des épreuves de la série 2 de la première partie, section 12, du *Manuel d'épreuves et de critères*. Par conséquent, aucun risque d'explosion n'est indiqué, à condition que l'emballage retienne l'eau et que l'eau reste répartie de manière homogène. La matière ne semble pas humide, mais peut être fluide. Pour confirmer ceci, il a été vérifié que la teneur en eau du matériau emballé dans les GRV souples demeurait supérieure à 20 % à différentes hauteurs sur une période de quatre mois. Les épreuves confirment que les GRV souples utilisés conservent l'eau et qu'il n'y a pas de danger que le matériau se transforme en matière explosive sèche.

11. En outre, l'«épreuve du feu externe (brasier)» (épreuve 6c, *Manuel d'épreuves et de critères*, première partie, sect. 16) a été effectuée sur un GRV souple (13H3) rempli de 579 kg de nitroguanidine, humidifiée à environ 15 % d'eau. Lors de cette épreuve, aucun effet dangereux n'a été observé (pas d'explosion en masse, pas de boule de feu ou de jet de flamme, flux thermique inférieur à 4 kW/m^2 (résultat relevé: 0,21 kW/m^2 à une distance de 15 m) et pas de projection de matière).

12. Sur la base de ces résultats d'épreuves, le BAM a déclaré que la nitroguanidine, humidifiée avec au moins 20 % (masse) d'eau, emballée dans un GRV souple (13H3) satisfaisait aux critères d'exemption des dispositions de la classe 1.

13. L'expérience pratique accumulée en Allemagne pendant plus de 15 ans (environ 3 000 tonnes par an transportées sur plus d'un million de kilomètres par le rail et par la route sans qu'un seul accident ne se soit produit) a montré que ce transport pouvait être jugé sûr.

14. Les résultats d'épreuves, ainsi qu'on le voit dans les annexes 1 et 2, de même que l'expérience pratique montrent qu'il n'y a pas de risque de détonation ou d'explosion en masse dans les conditions de transport, même en cas d'incendie.

15. Des données d'épreuves supplémentaires et de nouvelles informations seront communiquées dans un document informel distinct avant la fin de mai 2006.

Annex 1 (ENGLISH ONLY)

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Test Report and Expert Opinion on the testing of Nitroguanidine, wetted with approx. 15% water by mass, in a flexible IBC according to Test 6 (c) of the UN Test Manual

File reference	II.2-135/06
Copy	No. 1 of 2
Date	17 February 2006
Applicant/ Client	Dynamit Nobel GmbH Explosivstoff- und Systemtechnik Kalkstraβe 218 51377 Leverkusen
Application	7 February 2006
File ref.	-
Received on	9 February 2006
Subject of expert report	Testing of Nitroguanidine, wetted with approximately 15% water by mass, in a flexible IBC according to Test 6 (c) of the UN Recommendations on the Transport of Dangerous Goods, Manual of Tests and Criteria, Fourth Revised Edition, United Nations New York and Geneva, 2003

This Expert Report comprises pages 1 to 5

1. Background

In their letter of 31 January 2006, the company Dynamit Nobel, Explosivstoff- und Systemtechnik, requested that the Bundesanstalt für Materialforschung und –prüfung (BAM) carry out testing according to Test 6 (c) of the UN Manual of Tests and Criteria on Nitroguanidine, wetted with approx. 15% water by mass, packaged in a flexible intermediate bulk container (IBC), type 13H3, with lattice box.

A 579 kg sample of Nitroguanidine, packaged in a flexible IBC, type 13H3, with lattice box, was received for testing by the BAM open-air experimental facility on 16 January 2006.

2. Composition

The company stated the test specimen consisted of Nitroguanidine, wetted with 15.3% water (an appropriate Certificate of Analysis was provided).
Determination of the water content by BAM through Karl Fischer titration showed that the water content was in the range 16.8% to 18.4% by mass.

3. Preliminary remarks

Nitroguanidine (picrite), dry or wetted with less than 20% water by mass, is a Class 1 substance and assigned to UN number 0282, Class 1.1 D, of the Dangerous Goods Regulations.
Nitroguanidine, wetted with not less than 20% water, by mass, is a “desensitized explosive” of the Dangerous Goods Regulations and has been assigned the UN number 1336, Class 4.1.
The company Dynamit Nobel requested an investigation of whether Nitroguanidine, wetted with not less than 20% water, by mass, and packaged in a flexible IBC, type 13H3, with lattice box, satisfied the requirements for exemption from Class 1 in that packaging, primarily for land transport.
Dynamit Nobel requested that a batch containing approx. 15% water by mass be used for the test. The BAM agreed to this proposal, as combustion tests performed by BAM with smaller quantities of Nitroguanidine, wetted with not less than 20% water by mass, showed that the combustion times were very long.
The reduced water content results in more rapid combustion and thus shortens the duration of the test. The test results of Nitroguanidine, wetted with approx. 15% water by mass, can be used in the sense of Test 6 (c) for an assessment of the behaviour of Nitroguanidine, wetted with not less than 20% water, by mass, in a flexible IBC, type 13H3, with lattice box.

4. Test methods

The methods used are those described in the UN Recommendations on the Transport of Dangerous Goods, Manual of Tests and Criteria, Fourth Revised Edition, New York and Geneva, 2003.
Transport regulations allow the exemption of a substance in the envisaged packaging on the basis of the results of Test Series 6.
Test Series 6 comprises three test methods:

- 6 (a) Single package test
- 6 (b) Stack test
- 6 (c) External fire (bonfire) test

These tests are to be performed in the sequence a to c. It is not necessary to perform all of the tests of Test Series 6 (Tests 6(a) and 6(b)) if it has been demonstrated that the substance does not undergo detonation or deflagration even under conditions of greater strain (with stronger confinement than the intended packaging).
On the basis of test results and experience of BAM with dry Nitroguanidine and with Nitroguanidine, wetted with not less than 20% water, by mass, concerning their ability to propagate a detonation upon initiation with a detonator – which for Nitroguanidine is the most severe test condition for Test 6 (a) – a detonative conversion can be ruled out if the explosive power in the Trauzl Test (UN Test F.3) is only very low. Nitroguanidine, wetted with approx. 15% water by mass, was found to exhibit a lead block expansion of 19 ml per 10 g substance in UN Test F.3. In view of the low explosive power found (dry Nitroguanidine gives a lead block expansion of 309 ml for 10 g substance), there was no need to carry out UN Tests 6(a) and 6(b) using a standard detonator.
UN Tests 6(a) and 6(b) using an igniter were not carried out as studies of BAM with dry Nitroguanidine did not show any mass explosion upon such initiation.

5. External fire (bonfire) test – UN Test 6 (c)

5.1. Test conditions and description

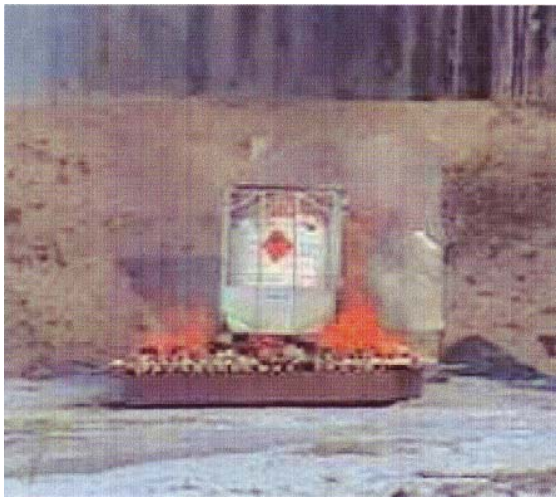
The test was carried out on 17 January 2006 at BAM's open-air facility in Horstwalde under dry conditions at temperatures of -6 °C to -4 °C and wind speeds of 0 m/s to 2 m/s.

The type 13H3 flexible IBC was placed directly on a wooden pallet to which the lattice box was secured. Metal sheets were fitted in the lower part of the lattice box to protect the stack from being damaged by fork lifts (see Figure 1). According to the manufacturer, the IBC contained 579 kg Nitroguanidine, wetted with approx. 15% water by mass. The IBC and its pallet were placed on a steel support in a steel tray measuring 2.8 m x 3.0 m, with sufficient wood and wood wool underneath. Approximately 20 liters of a diesel/petrol mixture were poured over the wood stack just before it was ignited.

Figure 1 Test set-up

The fire was recorded through two video cameras, and the heat radiation was measured through two sensors positioned 30 meters from the fire and further two sensors 40 meters from the fire.

The substance burned with a very small flame or reacted without flame. After a short period, the IBC material burnt through below and some of the contents trickled out into the wood pile during the combustion process.



After approx. 27 minutes the substance was converted down to the height of the protective sheets. The released substance in part extinguished the wood fire. However, the Nitroguanidine wetted with approx. 15% water by mass burnt in full. Combustion was complete after approx. 140 minutes.

The time required for conversion of the whole mass is determined as follows:

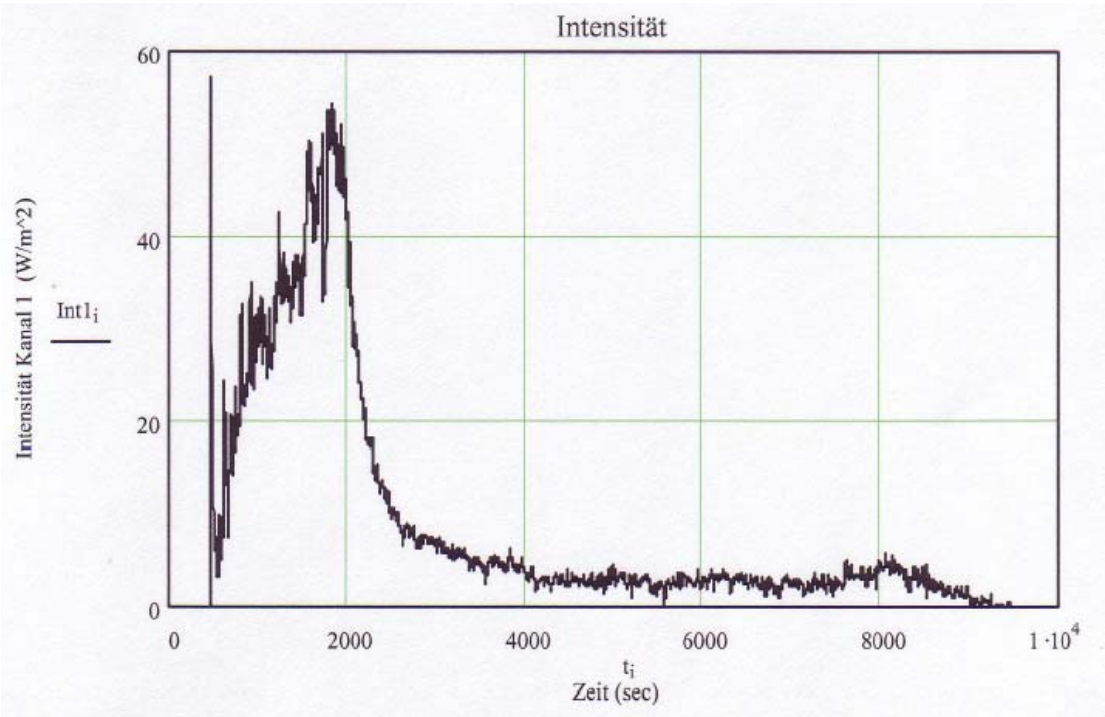


Figure 4 Thermal flux at 30 m distance. Ignition occurs at 443 sec. The first peak represents the ignition peak.

From the course of the thermal flux (Figure 4), the maximum was attained after 1820 seconds (approx. 23 minutes upon ignition). The thermal flux then decreased because the flames of the wood fire were no longer acting directly on the substance. After approx. 60 minutes, the thermal flux had fallen to 5% of the maximum value. At this time there were only residual quantities of the substance still present. The thermal flux at a distance of 15 m was calculated to be 0.21 kW/m^2, based on the measured value at a distance of 30 m. The upper limit (criterion for the 6 (c) Test) of 4 kW/m^2 at a distance of 15 m was not reached.

5.2. Evaluation of results according to the UN Test Manual

The test was evaluated on the basis of the criteria of UN Test 6 (c) with the following outcome:

- No mass explosion, no fireball and no splinters
- Thermal flux < 4 kW/m^2 at a distance of 15 m
- No projection from the product during combustion, with combustion being quiet and slow

The thermal flux at a distance of 15 m was below 4 kW/m^2, and the combustion time was > 35 s per 100 kg of substance.

6. Expert opinion on the transport of Nitroguanidine, wetted with not less than 20% water by mass, in a flexible IBC with lattice box*

When evaluating the test results it must be borne in mind that the following conditions need to be satisfied for Nitroguanidine, wetted with water, in order to be classified as a "desensitized explosive substances" (see, for example, Annex A to the ADR, marginal 2401, No. 21, Annex to the Federal Gazette Part II, No. 44 of 22 October 1998):

1. The water must be homogeneously distributed within the explosive substance. During transport, no segregation shall occur that would reduce the desensitization.
2. The explosive substances wetted with water must not be detonable through a standard detonator (standard electric blasting cap 0.6 g PETN) or undergo mass explosion upon initiation with a powerful booster charge.

The above conditions can only be satisfied with sufficient certainty if the Nitroguanidine is wetted with not less than 20% water by mass, so that, in the opinion of BAM, a general exemption from Class 1 based on Test Series 6 can only be considered if the water content is not less than 20% by mass.
The results of the UN Test 6 (c) for Nitroguanidine, wetted with approx. 15% water by mass, in a flexible IBC with lattice box, show that the thermal flux at a distance of 15 m is < 4 kW/m^2 and the combustion time is > 35 s for 100 kg. On the basis of the test results for Nitroguanidine, wetted with approx. 15% water by mass, it can be concluded that Nitroguanidine, wetted with not less than 20% water by mass, packed in a flexible type 13H3 IBC with lattice box, satisfies the criteria of UN Test 6 (c) for exemption from Class 1.

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*) This Expert Report has been drafted to the best of our knowledge and belief, objectively and without any external influence on the results. The BAM reserves the right to change, supplement or revoke this Expert Report if there are important reasons for doing so (e.g. because of substantial new findings).

Legends:

Figure 1: Test set-up

Figure 2: Approx. 3 minutes after ignition

Figure 3: After combustion for approx. 13 minutes

Figure 4:

Intensity - Channel 1

Time

Thermal radiation intensity at a distance of 30 m. Ignition occurs at 443 seconds. The first peak represents the ignition peak

Annex 2 (ENGLISH ONLY)

BAM
Bundesanstalt für
Materialforschung
und –prüfung

Your letter of: 31 October 2005
Our ref.: II.2-711/05
Tel. (030) 8104 4413
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Email: Fachgruppe-II.2@bam.de
Date: 9 December 2005

Bayer Industry Services GmbH & Co. OHG
Verfahrens- und Anlagensicherheit
For the attention of Dr. Heitkamp
SUA-VA1 Building B 407
51368 Leverkusen

Nitroguanidine

Dear Dr. Heitkamp

We refer to your letter of the above date and report the test results as follows:

1. Nitroguanidine, technically pure (data from "Explosivstoffe" #9, 1961)

Steel tube test (Koenen test)	Limiting diameter < 1.0 mm
Friction sensitivity (BAM)	None up to 360 N
Impact sensitivity (BAM)	None up to 50 J
BAM 2" steel tube (50 g booster) (UN test 1(a)(i), UN Test Manual, 1st edition)	Full propagation
BAM 1" steel tube (NP 8)	Full propagation
Trauzl lead block test	305 ml

2. Nitroguanidine, technically pure (additional test data)

UN Test 1(a)(iii) Gap Test (gap 0 mm) (UN Test Manual, 1st edition)	"+"
UN Test 2(a)(iii) Gap Test (gap 57.2 mm) (UN Test Manual, 1st edition)	"-"
UN Test 2(a)(iii) Gap Test (gap 50.8 mm) (UN Test Manual, 1st edition)	"+"
UN Test 5(a) Cap Sensitivity Test (0.6 g PETN) (UN Test manual, 3rd edition)	"+"

3. Nitroguanidine, wetted with more than 20% water (homogeneously distributed)

UN Test 1(a)(i) BAM steel tube test	No propagation
(50 g booster, UN Test Manual, 1st edition)	"-"
UN Test 5(a) Cap Sensitivity Test (0.6 g PETN)	"-"
(UN Test Manual, 3rd edition)	
UN Test 6(c) with approx. 200 kg in an FIBC	"-"
(UN Test Manual, 3rd edition)	

It follows from the test data presented that technically pure Nitroguanidine is a Class 1 substance under the Dangerous Goods Regulations. Nitroguanidine, wetted with at least 20% water (homogeneously distributed), however, may be regarded as a "desensitized explosive" under the known preconditions and assigned to Class 4.1.

Yours sincerely

[Signature]

Dr. Wehrstedt
Regierungsdirektor
Specialist Group II.2 "Reactive substances and substance systems"
Working Group "Explosive substances of the chemical industry"
