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Consultancy



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Testing



Training

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Low Hazard Calcium Hydride Formulation

Flammable Gas Generation Test (A12)

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FOR AND ON BEHALF OF CHILWORTH TECHNOLOGY LIMITED

CONTENTS

	PAGE NO.
1. INTRODUCTION	3
2. SCOPE OF ASSESSMENT	3
3. SAMPLE INFORMATION	3
4. SUBSTANCES WHICH IN CONTACT WITH WATER EMIT FLAMMABLE GASES	4
4.1 Low Hazard Calcium Hydride Formulation	6
5. CONCLUSIONS	8

1. INTRODUCTION

This report contains test data for DanGoods Training & Consultancy Ltd relating to a sample of low hazard calcium hydride formulation. In this assessment, testing has been conducted in order to determine if the sample generates flammable gas when brought into contact with water. The test is conducted in accordance with EC Testing Method A12, performed to the EC's Classification, Packaging & Labelling of Dangerous Substances in the European Union Part 2, Testing Methods, Jan97. Directive 92/69/EC (Annex V to Directive 67/548/EC). The EC test method is equivalent to the method described in the current version (currently 4th revised edition) of the United Nations Transportation of Dangerous Goods Recommendations, Tests and Criteria Manual (test N.5).

The sample used in this study was supplied by DanGoods Training & Consultancy Ltd.

2. SCOPE OF ASSESSMENT

This assessment covers a flammable gas generation test (A12) on a sample of Low Hazard Calcium Hydride Formulation.

This work follows discussions between Mr D. Waight (DanGoods Training & Consultancy Ltd) and Mr D. Baker (Chilworth Technology Ltd) and is in response to quotation number 102527.

Safety in chemical manufacture requires that all possible operational hazards, i.e. the presence and possible ignition of flammable atmospheres, and chemical reaction hazards are evaluated and that a suitable basis for safe operation is determined and implemented.

Should the material characteristics or composition be changed then consideration should be given to re-assessment of the material.

3. SAMPLE INFORMATION

Sample name	:	Low Hazard Calcium Hydride Formulation
Composition	:	Contains calcium hydride < 5 %
Batch number	:	N/A
CTL sample reference	:	156508
Appearance	:	As received by Chilworth Technology Ltd the sample was observed to be a grey paste
Hazards	:	No significant hazard

4. SUBSTANCES WHICH IN CONTACT WITH WATER EMIT FLAMMABLE GASES

Introduction

The test is designed to determine the ability of a substance to emit flammable gases on contact with water by bringing it into contact with water under a variety of conditions. The test is conducted in accordance with EC Testing Method A12, performed to the EC's Classification, Packaging & Labelling of Dangerous Substances in the European Union Part 2, Testing Methods, Jan97. Directive 92/69/EC (Annex V to Directive 67/548/EC).

Test Procedure

The method consists of three preliminary tests to determine if a violent reaction with water occurs and a final test to determine the quantity of gas generated. If it is known that the sample does not react violently with water the preliminary tests need not be conducted. No further tests are required if a spontaneous ignition occurs at any stage.

Test 1

A small quantity of the sample (ca. 2 mm diameter) is placed in a trough of distilled water at 20°C. It is noted if any gas is evolved and whether spontaneous ignition of the gas occurs.

Test 2

A small quantity of the sample (ca. 2 mm diameter) is placed in the centre of a filter paper floated flat on the surface of distilled water at 20°C. The filter paper is to keep the sample in one place under which condition the likelihood of spontaneous ignition of the gas is greater. It is noted if any gas is evolved and whether spontaneous ignition of the gas occurs.

Test 3

The sample is made into a pile approximately 20 mm high and 30 mm in diameter with a hollow in the top. A few drops of water are added into the hollow. It is noted if any gas is evolved and whether spontaneous ignition of the gas occurs.

Test 4

The substance should be inspected for any particles of less than 500 µm diameter, if the powder constitutes more than 1 % (mass) of the total, or if the substance is easily crumbled, then the whole of the sample should be ground to a powder before testing begins. The test should be performed 3 times at ambient temperature (20°C) and atmospheric pressure. Water (10-20 ml) is put into a dropping funnel and 10 g of the substance is weighed into a conical flask. The tap of the dropping funnel is opened to let the water into the conical flask and a stop watch is started. The volume of gas evolved is measured and timed. The rate of evolution of gas is calculated over 7 hours at 1 hour intervals. If the rate of evolution is

erratic or is increasing after 7 hours, the measuring time should be extended to a maximum time of 5 days. If, at any time, the rate of gas evolution exceeds $1 \text{ l.kg}^{-1}.\text{h}^{-1}$ the test can be stopped. If the chemical identity of the gas is unknown it should be analysed. If the gas contains several flammable components and it is unknown whether the whole mixture is flammable, a mixture of the same composition should be prepared and tested according to EC test A11.

Interpretation of Results

The substance is considered hazardous if spontaneous ignition occurs during any of the tests or if there is an evolution of flammable gas at a rate greater than $1 \text{ l.kg}^{-1}.\text{h}^{-1}$.

For UN test N.5, a substance should be classified as UN Class 4, Division 4.3 if a spontaneous ignition occurs during any of the tests or if there is an evolution of flammable gas at a rate greater than $1 \text{ l.kg}^{-1}.\text{hr}^{-1}$. The sample can then be assigned to a packing group based on the following criteria:

- | | | |
|-------------------|---|--|
| Packing group I | : | assigned to any substance which reacts vigorously with water at ambient temperatures and generally demonstrates a tendency for the gas produced to ignite spontaneously, or which reacts readily with water at ambient temperatures such that the rate of evolution of flammable gas is equal to or greater than 10 l.kg^{-1} of substance over any one minute period. |
| Packing group II | : | assigned to any substance which reacts readily with water at ambient temperatures such that the maximum rate of evolution of flammable gas is equal to or greater than $20 \text{ l.kg}^{-1}.\text{hr}^{-1}$ and which does not meet the criteria for Packing Group I. |
| Packing group III | : | assigned to any substance which reacts slowly with water at ambient temperatures such that the maximum rate of evolution of flammable gas is greater than $1 \text{ l.kg}^{-1}.\text{hr}^{-1}$ and which does not meet the criteria for Packing Groups I or II. |

4.1 Low Hazard Calcium Hydride Formulation

Test System Employed : Gas burette system 1
 Date of Test : 22nd December 2008
 Test Operator : I. Livingston
 Sample Preparation : Sample tested as received

Test Objective

To test a sample of Low Hazard Calcium Hydride Formulation to determine if it generates flammable gas when in contact with water.

Test Information - Initial Tests

1. A small quantity of the sample was placed in a trough of distilled water. It was noted if any gas generation or a spontaneous ignition occurred.
2. A small quantity of the sample was placed in the centre of a filter paper which was floated on the surface of distilled water. It was noted if any gas generation or a spontaneous ignition occurred.
3. A small quantity of the sample was made into a pile with a hollow in the top and a few drops of distilled water were added to the hollow. It was noted if any gas generation or a spontaneous ignition occurred.

Test Results – Initial Tests

In each test some small bubbles were observed but no spontaneous ignitions occurred.

Test Information – Final Test

10 g of the sample was charged to a glass flask which was connected to an automated gas burette system. 20 ml of water was added rapidly to the flask which was then sealed. The rate and quantity of gas generated was measured for at least 1 hour or until the gas generation had ceased. The test was conducted three times.

Test Results – Final Test

A temperature / time plot for Test 3 is shown in Graph 4.1.a.

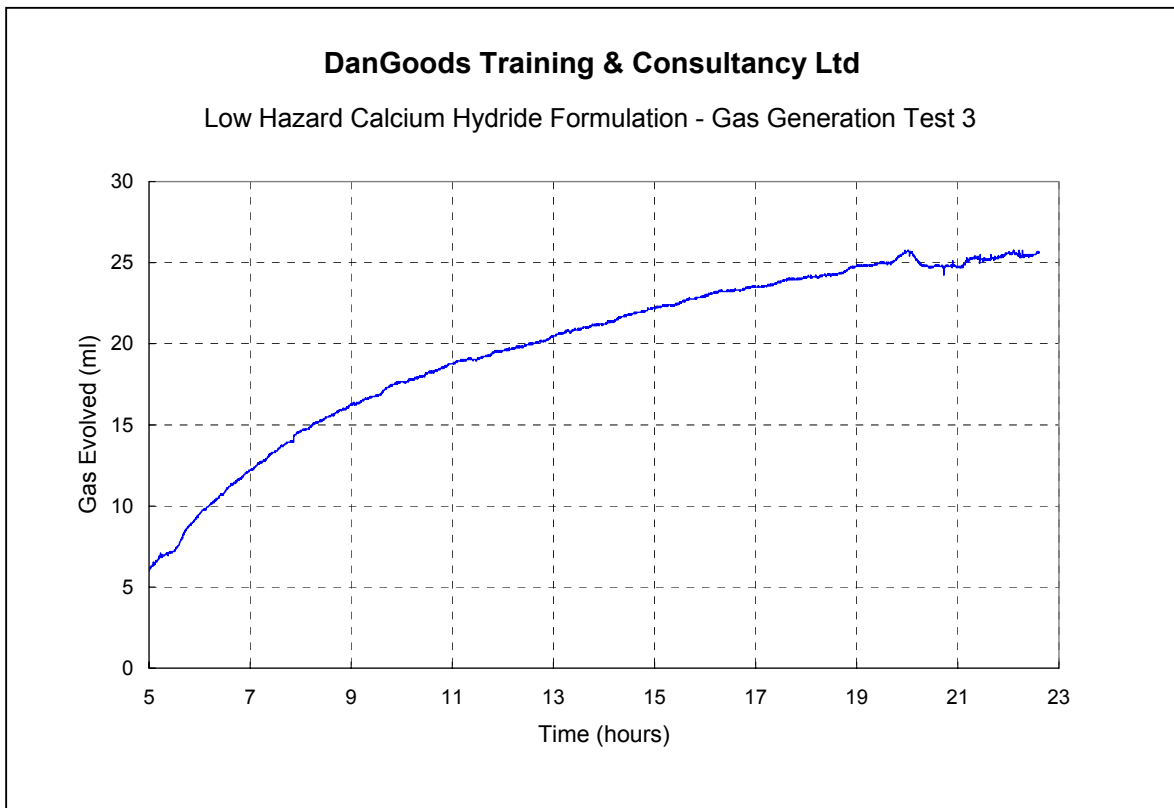
Table 4.1: Full Test Results

	Test 1	Test 2	Test 3*
Was spontaneous ignition observed?	No	No	No
Maximum rate of gas generation (l.kg ⁻¹ .h ⁻¹)	0.2	0.1	0.4

* in the final test the sample was spread out on the bottom of the flask to maximise the exposed surface area.

The maximum rate of gas generation was $< 1 \text{ l.kg}^{-1}.\text{h}^{-1}$ in all three tests. The sample is therefore considered to be 'non-hazardous' (according to EC test A12) and 'not division 4.3' (according to UN test N.5).

Graph 4.1.a



5. CONCLUSIONS

1. A flammable gas generation test was conducted on a sample of Low Hazard Calcium Hydride Formulation. The sample was found to have a maximum rate of gas generation of $< 1 \text{ l.kg}^{-1}.\text{h}^{-1}$ and is therefore considered to be 'non-hazardous' (according to EC test A12) and 'not Division 4.3' (according to UN test N5).