

International Carbon Black Association

UN/DOT Transportation Testing-Flammability of Solids / Self-Heating Solids

Client	International Carbon Black Association
Client location	1702 Brier Park Crescent Northwest, Medicine Hat, Alberta T1C 1T9, Canada
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1. PROJECT DETAILS AND TEST WORK APPROVAL STATEMENT

Quotation Number	28617
Job Number	19475
DEKRA Insight Facility	Chilworth Technology, Inc., 113 Campus Drive, Princeton, NJ 08540.
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Peer Reviewer	Yuan Dai, Senior Laboratory Specialist
Completion Date of Experimental Work	06.14.17

This report has been issued in digital format. In order to ensure that the integrity of the data is maintained, the signed hard copy (in the CTI archive) will be considered the source document and digital versions will be considered copies. All original test records are kept in a locked archive for a minimum of 10 years after the date of this report. Any remaining material(s) will be stored for a minimum of 1 month after the issue date of this report.

2. INTRODUCTION

This report contains test data for International Carbon Black Association regarding flammability/transport classification characteristics of **seven** samples named in the table on the following page. Specifically, the following work has been undertaken:

- UN/DOT Transportation Testing N.4 (Division 4.2) / Self-Heating Solids

This work is in response to quotation number 28617. A formal hazard assessment of the process / plant has not been conducted by Chilworth Technology and the consequences of specific process deviations have not been examined¹.

Detailed characterization of the material tested in this study is provided in Section 2 of this report (with results summarized in the conclusions section)².

¹ Process safety requires that all possible explosion, thermal stability and chemical reaction hazards are evaluated and that a suitable basis for safe operation is determined and implemented. Should the materials or processing conditions change then consideration should be given to re-assessment.

² A description of the test procedures together with full test results and information on their interpretation is given in the test sections of this report. The appendices provide background information on the subject matter. Chilworth Technology's Laboratories are ISO 17025 or GLP (Good Laboratory Practice) compliant and this study was carried out to the principles of ISO 17025.

3. SUMMARY OF TEST RESULTS AND RECOMMENDATIONS

3.1 Summary of Test Data

The results of testing completed on your samples are summarized below.

Sample	Self-Heating Substances – UN/DOT Test N.4 Division 4.2
a) N134	<u>NOT</u> a self-heating substance of Division 4.2
b) N762	<u>NOT</u> a self-heating substance of Division 4.2
c) N326	<u>NOT</u> a self-heating substance of Division 4.2
d) N220	<u>NOT</u> a self-heating substance of Division 4.2
e) N683	<u>NOT</u> a self-heating substance of Division 4.2
f) N660	<u>NOT</u> a self-heating substance of Division 4.2
g) N990	<u>NOT</u> a self-heating substance of Division 4.2

The results of testing are highly dependent on the composition and physical nature of the sample. For this reason, any change in manufacturing / handling procedures or composition should be accompanied by a review of the relevant data.

Chilworth Technology would be pleased to provide specific advice, including interpretation and application of experimental data. Site visits to discuss operational safety or to perform plant inspections and measurements can be arranged on request.

3.2 Interpretation of Results and Recommendations

This guidance is based purely upon the data collected from the current study and therefore may not encompass all factors which are necessary in order to fully support your chosen Basis of Safety for processing the material. The test work should be supplemented with a detailed hazard and risk assessment (incorporating a hazardous area classification study), definition and implementation of a Basis of Safety and control measures, and continuing auditing to ensure that places where the material is processed remain safe. If further clarification, or support in any of these activities, is required, Chilworth Technology would be pleased to assist.

4. SAMPLE INFORMATION

Product name	N134
CTI sample reference	19475a
Appearance	Coarse, black powder
Particle size	As received

Product name	N762
CTI sample reference	19475b
Appearance	Coarse, black powder
Particle size	As received

Product name	N326
CTI sample reference	19475c
Appearance	Coarse, black powder
Particle size	As received

Product name	N220
CTI sample reference	19475d
Appearance	Coarse, black powder
Particle size	As received

Product name	N683
CTI sample reference	19475e
Appearance	Coarse, black powder
Particle size	As received

Product name	N660
CTI sample reference	19475f
Appearance	Coarse, black powder
Particle size	As received

Product name	N990
CTI sample reference	19475g
Appearance	Coarse, black powder
Particle size	As received

5. SELF-HEATING SOLIDS

Test Objective and Information

The series of tests is conducted to define whether a substance will undergo spontaneous ignition or dangerous self-heating. This is indicated by a sample temperature rise of 60 K (or more) above the set oven temperature. If the substance does exhibit self-heating properties, then further tests are conducted to determine the transportation classification and appropriate packing group. The test is conducted in accordance with the UN Transportation of Dangerous Goods Recommendations, Test N.4

The cubic sample containers of 25 mm and 100 mm side lengths are employed. They are open at the top and manufactured from stainless-steel net, with a mesh opening of 0.053 mm. Each container is housed in an outer cubic container cover, manufactured from stainless-steel net with a mesh opening of 0.595 mm which is slightly larger than the sample container, so that the container fits in this cover. In order to minimise the effects of air circulation, another stainless-steel cage, manufactured with a mesh opening of 0.595 and 150 x 150 x 250 mm in size, houses both these baskets.

The test material, in its commercial form, is filled to the brim of the sample basket which is then tapped several times. If the sample has been seen to settle then more material is added. The container is housed in the covers and placed in the centre of the oven at the required test temperature

A hot air, circulating type oven with an inner capacity of approximately 120 litres and capable of controlling the internal temperature at $140 \pm 2^\circ\text{C}$ is utilised. In the initial test (with a 100 mm cube), the oven temperature is raised to 140°C and held isothermally for a test period of 24 hours. The temperature of the sample and oven are continually monitored / recorded with inconel sheathed thermocouples to provide accurate temperature measurement. One thermocouple is placed in the centre of the sample and the other between the sample container and the oven wall

Generic equipment information is contained in the table below.

Equipment Configuration / Settings for UN 4.2 Testing

Parameter	Setting / Configuration
Oven Type	Fan Assisted
Oven Volume	120 Litre (average)
Testing Range	$100^\circ\text{C} - 140^\circ\text{C}$
Thermocouple Type	K - Type
Data Acquisition system	DasyLab software
Test basket size	25mm^3 and 100mm^3 (With external $150 \times 150 \times 250\text{mm}$ wire cage)

Interpretation of results

A substance is classified as a self-heating substance of UN Class 4, Division 4.2 if a positive test result is observed in a 100 mm basket at 140°C. A positive result is defined as a sample temperature rise of 60°C (or more) above the oven temperature during a 24 hour period. From this point the following packing groups are then assigned to the sample:

- Packing group II : Assigned to any substance which gives a positive test result in a 25 mm basket at 140°C.
- Packing group III : Assigned to any substance which:
- a) gives a positive test result in a 100 mm basket at 140°C and a positive test result in a 100 mm basket at 100°C
 - b) gives a positive test result in a 100 mm basket at 140°C and a negative test result in a 100 mm basket at 120°C and is to be transported in packages of more than 3 m³ volume
 - c) gives a positive test result in a 100 mm basket at 140°C and a negative test result in a 100 mm basket at 100°C and is to be transported in packages of more than 450 l volume
- Not Division 4.2 : Assigned to any substance which:
- a) gives a negative test result in a 100 mm basket at 140°C.
 - b) gives a positive test result in a 100 mm basket at 140°C and a negative test result in a 100 mm basket at 120°C and is to be transported in packages of less than 3 m³ volume
 - c) gives a positive test result in a 100 mm basket at 140°C and a negative test result in a 100 mm basket at 100°C and is to be transported in packages of less than 450 l volume

5.1 100mm at 140°C Test Results for SAMPLE A: N134

Date 06.06.17
 Operator Y. Dai
 Test Standard United Nations Document, Recommendations on the Transport of Dangerous Goods
 UN Test Reference Test N.4

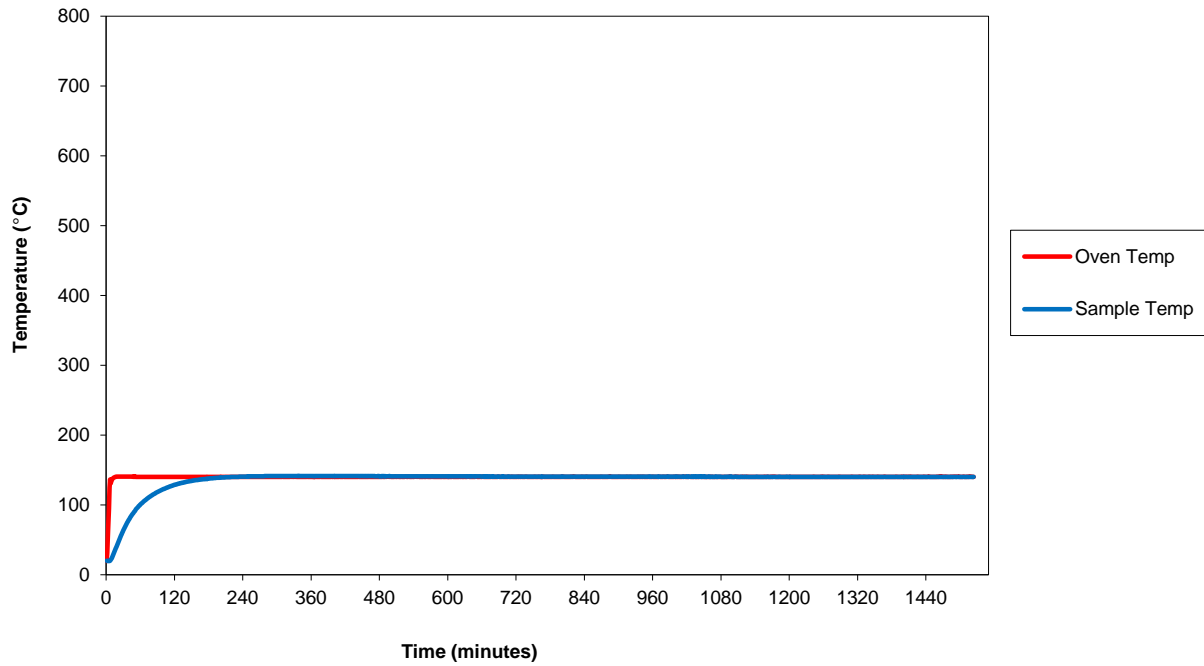
Results Not Self-heating substance of Division 4.2.

Table 5.1.1: Details of Test

Basket Number	Basket Size (mm ³)	Test Temperature (°C)	Sample Mass (g)
1	100	140	389.0

Comments: No exothermic reaction was observed during the test.

100mm Basket Test - Isothermal at 140°C
N134



5.2 100mm at 140°C Test Results for SAMPLE B: N762

Date 06.08.17
 Operator Y. Dai
 Test Standard United Nations Document, Recommendations on the Transport of Dangerous Goods
 UN Test Reference Test N.4

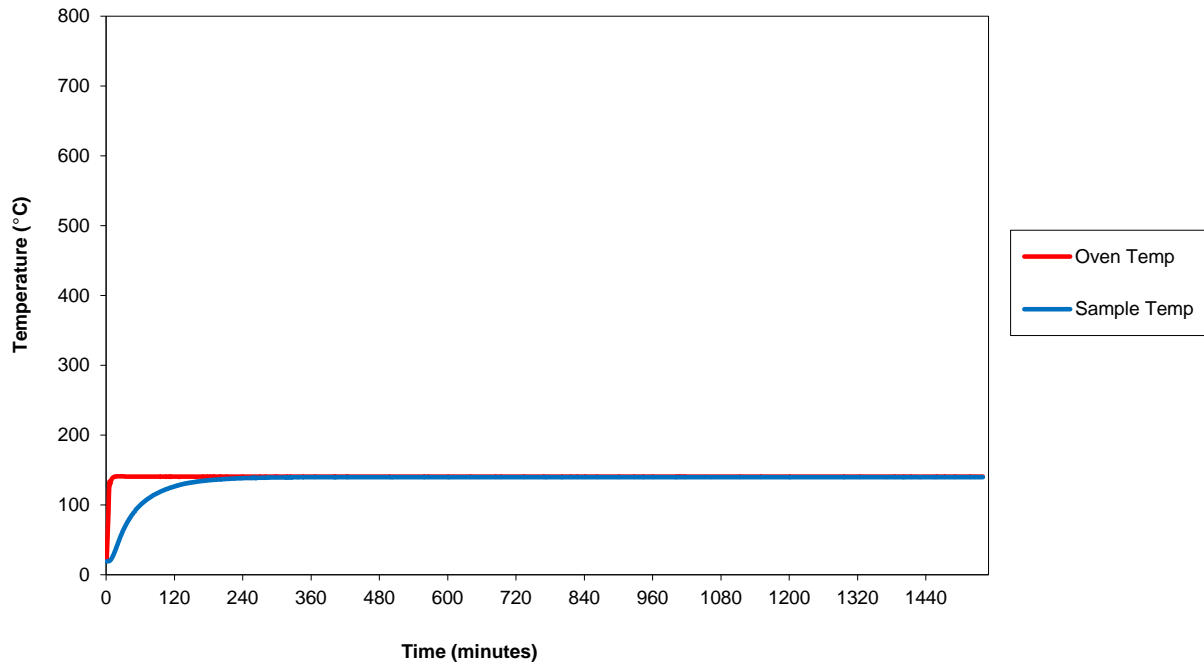
Results Not Self-heating substance of Division 4.2.

Table 5.2.1: Details of Test

Basket Number	Basket Size (mm ³)	Test Temperature (°C)	Sample Mass (g)
1	100	140	599.0

Comments: No exothermic reaction was observed during the test.

100mm Basket Test - Isothermal at 140°C
N762



5.3 100mm at 140°C Test Results for SAMPLE C: N326

Date 06.08.17
 Operator Y. Dai
 Test Standard United Nations Document, Recommendations on the Transport of Dangerous Goods
 UN Test Reference Test N.4

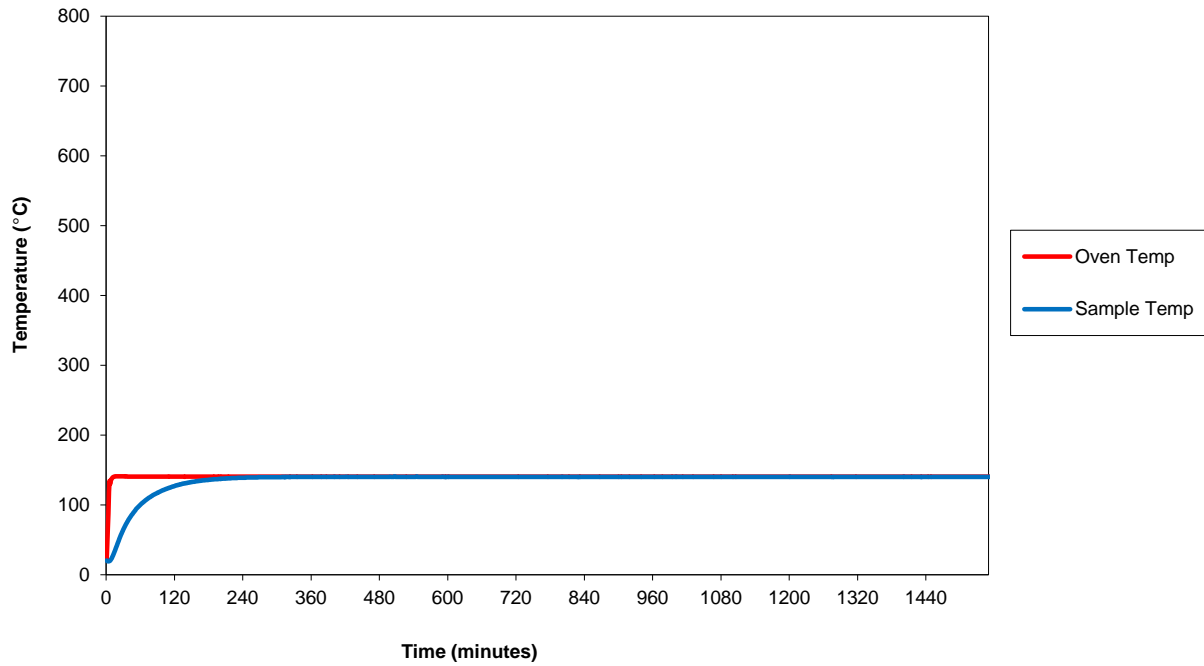
Results Not Self-heating substance of Division 4.2.

Table 5.3.1: Details of Test

Basket Number	Basket Size (mm ³)	Test Temperature (°C)	Sample Mass (g)
1	100	140	510.0

Comments: No exothermic reaction was observed during the test.

100mm Basket Test - Isothermal at 140°C
N326



5.4 100mm at 140°C Test Results for SAMPLE D: N220

Date 06.09.17
 Operator Y. Dai
 Test Standard United Nations Document, Recommendations on the Transport of Dangerous Goods
 UN Test Reference Test N.4

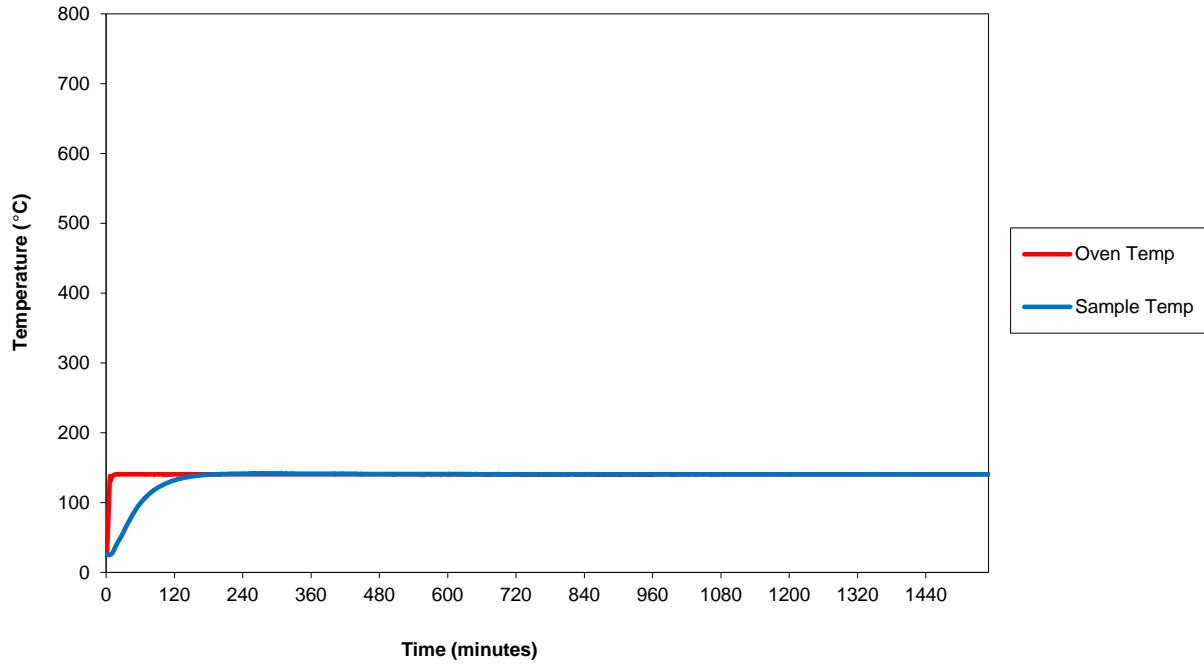
Results Not Self-heating substance of Division 4.2.

Table 5.4.1: Details of Test

Basket Number	Basket Size (mm ³)	Test Temperature (°C)	Sample Mass (g)
1	100	140	334.0

Comments: No exothermic reaction was observed during the test.

100mm Basket Test - Isothermal at 140°C
N220



5.5 100mm at 140°C Test Results for SAMPLE E: N683

Date 06.12.17
 Operator Y. Dai
 Test Standard United Nations Document, Recommendations on the Transport of Dangerous Goods
 UN Test Reference Test N.4

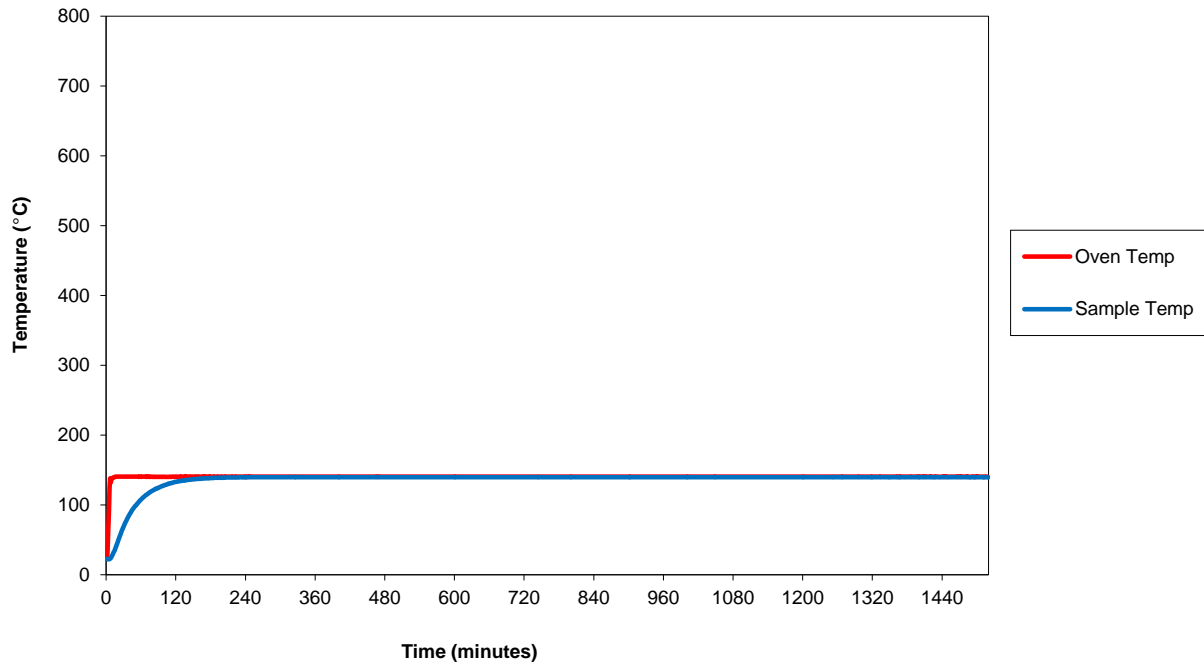
Results Not Self-heating substance of Division 4.2.

Table 5.5.1: Details of Test

Basket Number	Basket Size (mm ³)	Test Temperature (°C)	Sample Mass (g)
1	100	140	323.0

Comments: No exothermic reaction was observed during the test.

100mm Basket Test - Isothermal at 140°C
N683



5.6 100mm at 140°C Test Results for SAMPLE F: N660

Date 06.13.17
 Operator Y. Dai
 Test Standard United Nations Document, Recommendations on the Transport of Dangerous Goods
 UN Test Reference Test N.4

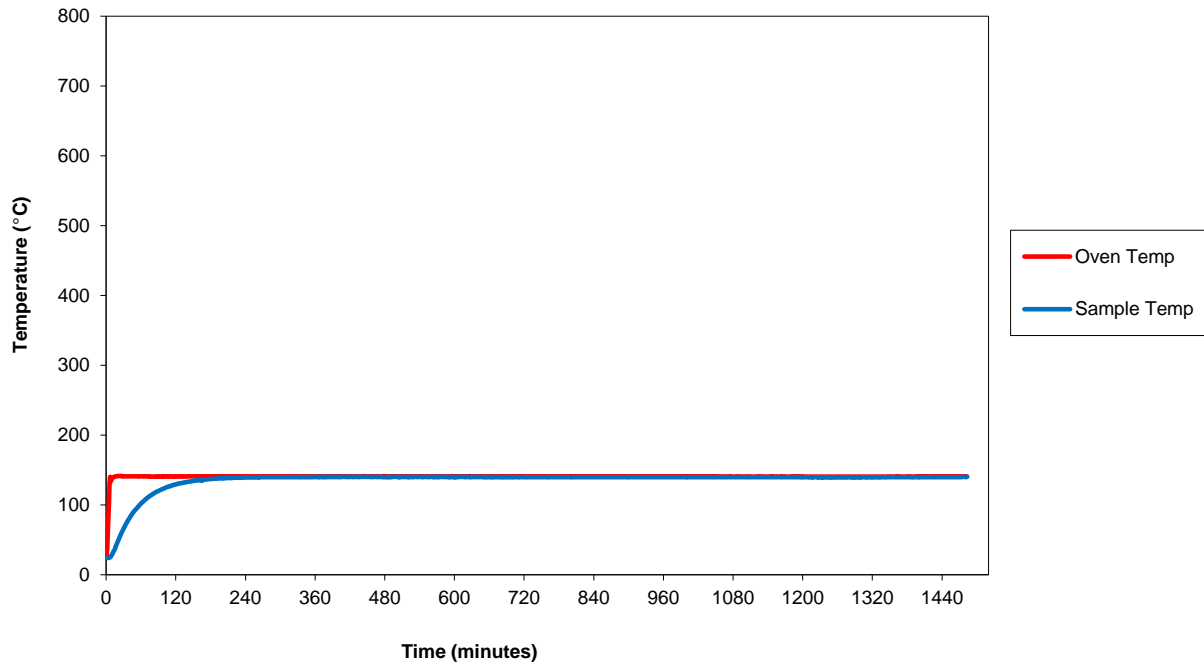
Results Not Self-heating substance of Division 4.2.

Table 5.6.1: Details of Test

Basket Number	Basket Size (mm ³)	Test Temperature (°C)	Sample Mass (g)
1	100	140	500.0

Comments: No exothermic reaction was observed during the test.

100mm Basket Test - Isothermal at 140°C
N660



5.7 100mm at 140°C Test Results for SAMPLE G: N990

Date 06.14.17
 Operator Y. Dai
 Test Standard United Nations Document, Recommendations on the Transport of Dangerous Goods
 UN Test Reference Test N.4

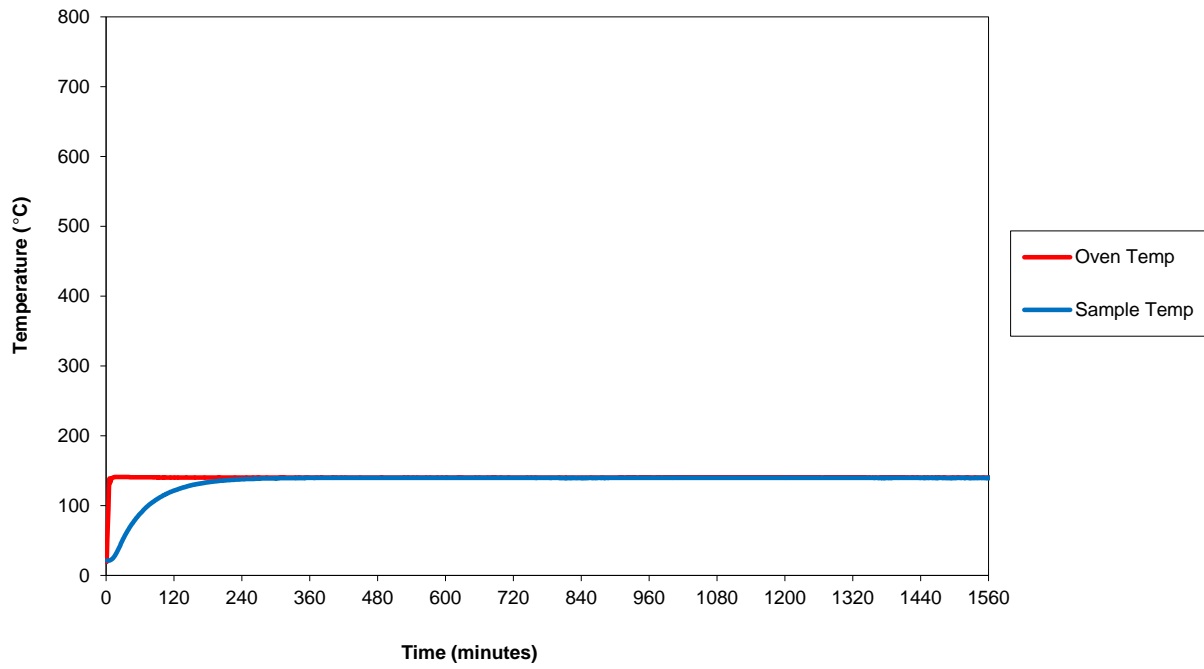
Results Not Self-heating substance of Division 4.2.

Table 5.7.1: Details of Test

Basket Number	Basket Size (mm ³)	Test Temperature (°C)	Sample Mass (g)
1	100	140	700.0

Comments: No exothermic reaction was observed during the test.

100mm Basket Test - Isothermal at 140°C
N990



6. APPENDIX

Legal Disclaimer and Liability

- (a) Limitation of Liability. The test procedures and/or consulting services conducted by Chilworth Technology (the “Company”) were performed under controlled laboratory conditions, which the Company considers reliable. Although the Company performed its testing services pursuant to reliable and generally accepted testing procedures in the industry, the Company does not guarantee or provide any representations or warranties with respect to Client’s use, interpretation or application of the test results and/or consulting services provided by the Company. Moreover, the results of the testing procedures are based upon certain assumptions, information, documents, and procedures provided by the Customer. AS SUCH, IN NO EVENT AND UNDER NO CIRCUMSTANCE SHALL THE COMPANY BE LIABLE FOR SPECIAL, INDIRECT, PUNITIVE OR CONSEQUENTIAL DAMAGES OF ANY NATURE WHATSOEVER, INCLUDING WITHOUT LIMITATION, ANY LOST REVENUE OR PROFITS OF THE CUSTOMER OR ITS CUSTOMERS, AGENTS AND DISTRIBUTORS, RESULTING FROM, ARISING OUT OF OR IN CONNECTION WITH, THE SERVICES PROVIDED BY THE COMPANY OR THE RESULTS OF ANY TESTS PERFORMED BY THE COMPANY. The Customer agrees that the Company shall have no liability for damages, which may result from Client’s use, interpretation or application of the test results and/or consulting services provided by the Company.
- (b) The Company’s pricing of the testing services provided does not contemplate that the Company shall have any liability resulting from its performance of the testing procedures, except as otherwise set forth in the Quotation from the Company. Accordingly, the Customer shall indemnify and hold harmless the Company, its shareholders, directors, officers, employees and agents (the “*Indemnified Parties*”) from and against any and all loss, cost, liability and expense, including reasonable attorney’s fees and costs, which any of the Indemnified Parties may incur, sustain or be subject to, as a result of any claim, demand, action, investigation or proceeding arising out of or relating to either: (a) the testing services provided by the Company; or (b) any material, equipment, specifications or safety information (or lack thereof) supplied to the Company (or which should have been supplied to the Company) by Customer and/or any failure of such materials, equipment, specifications and safety information to comply with any federal, state or local law or safety standard.
- (c) For additional terms and conditions, which apply with respect to the provision of this report, see the Quotation provided by the Company and executed by Customer. If any terms set forth in the Quotation conflict with the terms set forth herein, the terms set forth herein shall apply.