NATO STANDARD

ADivP-03

STANDARD TO QUANTIFY THE CHARACTERISTICS OF GRANULAR CARBON DIOXIDE (CO_2) ABSORBENT MATERIAL FOR DIVING AND HYPERBARIC APPLICATIONS

Edition A Version 2

SEPTEMBER 2016

NORTH ATLANTIC TREATY ORGANIZATION

ALLIED DIVING OPERATIONS PUBLICATION

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NORTH ATLANTIC TREATY ORGANIZATION (NATO)

NATO STANDARDIZATION OFFICE (NSO)

NATO LETTER OF PROMULGATION

15 September 2016

1. The enclosed Allied Diving Operations Publication ADivP-03, Edition A, Version 2 - STANDARD TO QUANTIFY THE CHARACTERISTICS OF GRANULAR CARBON DIOXIDE (CO_2) ABSORBENT MATERIAL FOR DIVING AND HYPERBARIC APPLICATIONS, which has been approved by the nations in the Military Committee Maritime Standardization Board (MCMSB), is promulgated herewith. The agreement of nations to use this publication is recorded in STANAG 1411.

2. ADivP-03, Edition A, Version 2 is effective upon receipt and supersedes ADivP-03, Edition A, Version 1 which shall be destroyed in accordance with the local procedure for the destruction of documents.

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4. This publication shall be handled in accordance with C-M(2002)60.

Edvardas MAŽEIKIS
Major General, LTUAF
Director, NATO Standardization Office

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NATO Nations

Each Nation may replace this page with its own National Letter of Promulgation

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RECORD OF RESERVATIONS

CHAPTER	RECORD OF RESERVATIONS BY NATIONS

Note. *The reservations listed on this page include only those that were recorded at the time of promulgation and may not be complete. Refer to the NATO Standardisation Document Database for the complete list of existing reservations*

RECORD OF SPECIFIC RESERVATIONS

NATION	SPECIFIC RESERVATIONS

Note. *The reservations listed on this page include only those that were recorded at time of promulgation and may not be complete. Refer to the NATO Standardisation Database for the complete list of existing reservations.*

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CONVENTIONS USED IN THIS PUBLICATION

CHANGE SYMBOLS

Revised text in changes is indicated by a black vertical line in either margin of the page, like the one printed next to this paragraph. The change symbol indicates added or restated information. A change symbol in the margin adjacent to the chapter number and title indicates a new or completely revised chapter.

WARNINGS, CAUTIONS, AND NOTES

The following definitions apply to warnings, cautions, and notes used in this manual:

WARNING

AN OPERATING PROCEDURE, PRACTICE, OR CONDITION THAT MAY RESULT IN INJURY OR DEATH IF NOT CAREFULLY OBSERVED OR FOLLOWED.

CAUTION

AN OPERATING PROCEDURE, PRACTICE, OR CONDITION THAT MAY RESULT IN DAMAGE TO EQUIPMENT IF NOT CAREFULLY OBSERVED OR FOLLOWED.

Note. *An operating procedure, practice, or condition that requires emphasise .*

WORDING

Word usage and intended meaning throughout this publication is as follows:

“Shall” indicates the application of a procedure is mandatory.

“Should” indicates the application of a procedure is recommended.

“May” and “need not” indicates the application of a procedure is optional.

Will” indicates future time. It never indicates any degree of requirement for application of a procedure

CHAPTER 1 - INTRODUCTION

0101. Aim

The aim of this agreement is to provide for the assessment of granular carbon dioxide absorbent, used for diving and hyperbaric applications within NATO, against a standard set of methods.

0102 Agreement

Participating nations agree to use the details in this publication as standard methods for the assessment of granular carbon dioxide absorbent material for diving and hyperbaric applications.

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CHAPTER 2 – DETAILS OF AGREEMENT

0201 General

1. All equipment shall be primarily specified and dimensioned in Standard International Units.

2. The methods specified in Annex A provide a basic standard for establishing the quality of granular carbon dioxide absorbent material used for diving and hyperbaric applications.

3. Samples shall be taken at random and the number of samples analysed for each test clearly stated.

0202 Principle Characteristics doe Absorbent Material

1. Carbon dioxide absorbent granule size.

2. Dust load.

Definition: The Dust Load is the mass of particles of a diameter less than the aperture of the finest sieve in use for that grade of absorbent.

3. Friability.

4. Carbon dioxide absorbent activity.

Definition: The Activity Time is the time taken for the carbon dioxide level, measured at the outlet of the test sample, to reach 0.50 % v/v.

5. Flow resistance.

6. Volatile (water) content.

7. Initial carbonate content.

8. Level of contaminants.

0203 NATO Carbon Dioxide Absorbent Grades

1. The absorbent material may be classified either by its performance characteristics or by a NATO grade.

2. The criteria for various NATO grade absorbents are presented in Annex B.

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ANNEX A – GRANULAR CARBON DIOXIDE ABSORBENT MATERIALS DETERMINATION OF PRINCIPLE CHARACTERISTICS

A101 Warning

Carbon dioxide absorbents consisting of (or containing) lithium hydroxide are harmful by inhalation and corrosive to skin and eyes. It is essential that the tests described in these procedures are conducted in a suitable ventilated facility and that operators wear appropriate personal protective equipment.

A102 Granule Size, Dust and Friability

The tests to determine the size range and dust load shall be conducted in accordance with the following procedures:

1. Sieves. The sieve mesh sizes shall be to an approved standard, in the range of 5.60 mm to 0.60 mm. The configuration of the sieves, used to determine the granule size and distribution, are determined by the NATO grade of absorbent against which the material is compared, as shown in Table A-1. Sieve nests shall include a correctly fitting lid and receiver.

Table A-1: Sieve Sizes and Nest Assembly Used for All NATO Grades

Aperture Size* (mm)	Small Grain	Large Grain
5.60	-	√
4.75	-	√
2.80	√	-
2.00	√	√
1.40	√	-
0.60	√	√

***ISO 3310-1:2000 Part 1: Test sieves of metal wire cloth**

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2. Shaker. A mechanical shaker shall be used which causes the particles to bounce and turn so as to present different orientations to the sieve.

3. Balance. The balance used to weigh the samples must have an accuracy of 0.1 g or better.

4. Method – Granule Size and Dust Load:

a. Assemble the nest of appropriate clean dry sieves in the order of aperture size, with the coarsest at the top. Distribute 100 ± 0.1 g of sample on the top sieve. Record the amount of sample used. Attach the lid and shake the assembled sieves for 5 minutes.

b. Remove the nest of sieves from the shaker and weigh the amount of material retained on each sieve, and the dust passing into the receiver at the base of the nest. Any granules lodged in the meshes of a sieve shall be included as the material retained on the sieve.

5. Method – Friability:

a. The friability test procedure is as specified for the granule size and dust load test, but the sample is shaken for 60 minutes. The same sieve sizes shall be used for both the sieve and friability test.

b. Remove the nest of sieves from the shaker and weigh the amount of material retained on each sieve, and the dust passing into the receiver at the base of the nest. Any granules lodged in the meshes of a sieve shall be included as the material retained on the sieve.

7. Reporting. Report the mass of material on each sieve, and the dust in the receiver, as a percentage of the sample mass used.

A103 Determination of Activity

1. The tests to determine the activity shall be conducted at 20 °C ± 1 °C and in accordance with the following procedure:

2. General. A mixture of nitrogen and carbon dioxide is passed through a bed of absorbent, of specified dimensions, at a constant rate and the effluent (outlet) gas analysed for carbon dioxide. The time taken from the start of the test until the concentration of carbon dioxide at the outlet reaches 0.50 % v/v carbon dioxide is measured. This time shall be defined as the ACTIVITY of the absorbent.

3. Test Apparatus. The test rig set up is shown in Figure A-1.

4. Test Gas Mixture. 5.0 % ± 0.05 % carbon dioxide in nitrogen.

5. Carbon Dioxide Gas Analysers:

a. Inlet carbon dioxide sampling: Monitor inlet carbon dioxide level with a closed loop analyser. The analyser must be suitably ranged and calibrated using certificated calibration gas mixtures of carbon dioxide in nitrogen.

b. Effluent (outlet) carbon dioxide sampling: Monitor the effluent gas mixture with an analyser that has been suitably ranged and calibrated using certificated calibration gas mixtures of carbon dioxide in nitrogen.

6. Activity Tube. The tube, as shown in Figure A-2, is to be a 30.0 mm internal diameter glass tube and calibrated with a bed volume of 105 ml ± 0.5 ml.

7. Method:

a. **Snow Storm Filling**. Transfer a random sample of absorbent granules, sufficient to fill the tube to the 105 ml filling mark, into the snow storm filling apparatus (Figures A-3 and A-4). The sample shall be poured at an even rate into the sample tube located in the base holder. Ensure the granules fill the tube evenly to the calibration mark. The mass (g) of sample added to the tube must be recorded.

b. **Testing.** Pass a flow of 3.0 ± 0.1 $l \cdot min^{-1}$ of homogenous test gas mixture, maintained at 20 ± 1 °C, through the humidifiers before passing through the glass absorption tube. This tube shall be vertical, with the test gas flowing top to bottom. A sample of the effluent gas exiting the absorption tube shall be drawn off and passed into a carbon dioxide analyser, at a sufficient rate to provide accurate analysis.

Note. *The sample gas must pass through a drying agent that will not absorb carbon dioxide [e.g. $MgClO_4$] before passing into the gas analyser.)*

c. **Data Recording.** The output signal from the inlet sample analyser and effluent sample analyser may be recorded on a suitably calibrated and scaled chart recorder or computerised data acquisition system. The time taken for the effluent gas to reach a level of 0.50 % v/v carbon dioxide shall be noted.

d. **Number of Samples.** For each material the activity test shall be repeated a minimum of 3 times.

A104 Capacity Equivalents

1. The test to determine the flow resistance shall be conducted at 20 °C ± 1 °C and as part of the activity test:

2. Test Apparatus. The test rig set up is shown in Figure A-1. A differential pressure monitor with a minimum accuracy of ± 0.01 mbar shall be connected to the top and the bottom of the activity tube.

3. Method. A baseline differential pressure should be recorded with the activity test gas flowing and no absorbent in the activity tube. Then, during the full duration of the activity test the differential pressure across the activity tube shall be measured.

4. Data Recording: The output signal from a differential pressure transducer may be recorded on a suitably calibrated and scaled chart recorder or computerised data acquisition system. Report the maximum differential pressure recorded minus the baseline differential pressure.

A105 Determination of Volatile Content

1. The test to determine the volatile (water) content shall be conducted in accordance with either of the following procedures.

2. <u>Automatic Volatile Content (Infrared Balance):</u>

a. **Method.** Transfer approximately 10 g of sample onto the infrared balance and note the mass (M_{A1}). Start the automatic drying cycle and note (if available) the percentage volatile reading when the sample is dry and/or the mass of the dried sample (M_{A2}).

b. **Calculation.** The percentage volatile content may be calculated using:

$$Volatile\ matter, \% = \frac{\left(M_{A1} - M_{A2}\right)}{M_{A1}} 100$$

3. Volatile Content (Manual System):

a. **Method.** Transfer approximately 100 g of sample to a previously weighed (M_{M1}) clean dry squat-form weighing dish and accurately weigh (M_{M2}). Remove the lid and place the dish, with the lid alongside, in a well-ventilated oven maintained at 150 ± 5 °C. After 120 minutes replace the lid and transfer the dish to a desiccator, containing freshly activated silica gel. Allow to cool for 30 minutes. Release the lid on the dish momentarily and re-weigh (M_{M3}).

b. **Weighing Dish.** The dish shall be a minimum of 100 mm diameter and minimum depth of 20 mm. The dish shall be able to withstand temperatures in excess of 150 °C.

c. **Calculation.** The percentage volatile content may be calculated using:

$$Volatile\ matter, \% = \frac{\left(M_{M2} - M_{M3}\right)}{\left(M_{M2} - M_{M1}\right)} 100$$

A106 Determination of Initial Carbonate Content

1. The test to determine the initial carbonate content of the absorbent shall be conducted in accordance with the following procedure:

2. General. A sample of absorbent material is dissolved in hydrochloric acid. Carbon dioxide gas released from any absorbent that has already reacted is collected and the volume measured.

3. Balance. The balance used to weigh the samples must have an accuracy of 0.1 g or better.

4. Method:

a. Assemble a suitable reaction vessel such that fluid and solid material may be added to it and any evolved gas collected and measured.

b. Add approximately 5 g of sample to a reaction vessel and record the mass (M_C).

c. Add approximately 50 ml of 6 molar hydrochloric acid (half diluted concentrated acid) and collect any evolved gas. Allow the collected gas to equilibrate with ambient laboratory temperature (nominally 20°C) and record the volume (V_C) in ml).

5. Calculation. The percentage carbonate may be calculated using:

$$\%\ Carbonate = \frac{\left(\left(\frac{273.15}{(Ambient(C) + 273.15)}\right) \times \left(\frac{(Ambient(mbar) - vp)}{1013.25}\right) \times Vc\right) \times}{M_C}\ constant$$

- *vp* = water vapour pressure (mbar)
- soda lime constant = 0.45
- LiOH constant = 0.33

A107 Determination of Contaminants

1. The tests to determine contaminants shall be conducted in accordance with the following procedure:

2. General. Absorption indicators are not required or effective for diving absorbents and should be classed as a contaminant.

3. **Indicators.** The presence of an indicator may be identified by observing the absorbent during the activity test and recording any colour change that occurs.

A108 Results

The testing report may be presented as shown in Table A-2 but shall contain at least the same level of information regardless of the final format.

Figure A-1. Schematic Carbon Dioxide Absorbent Test Rig

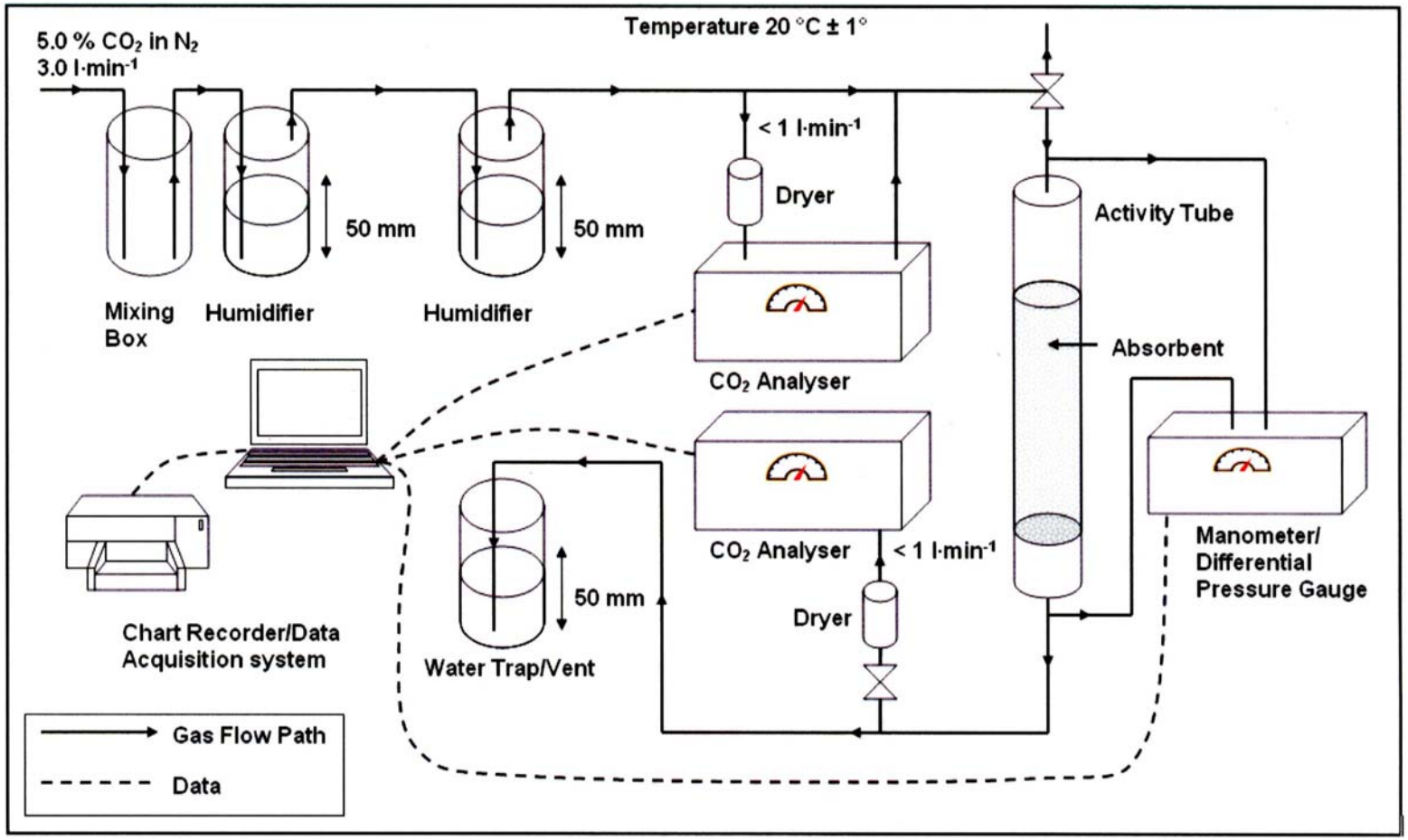

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Figure A2. Absorbent Activity Tube

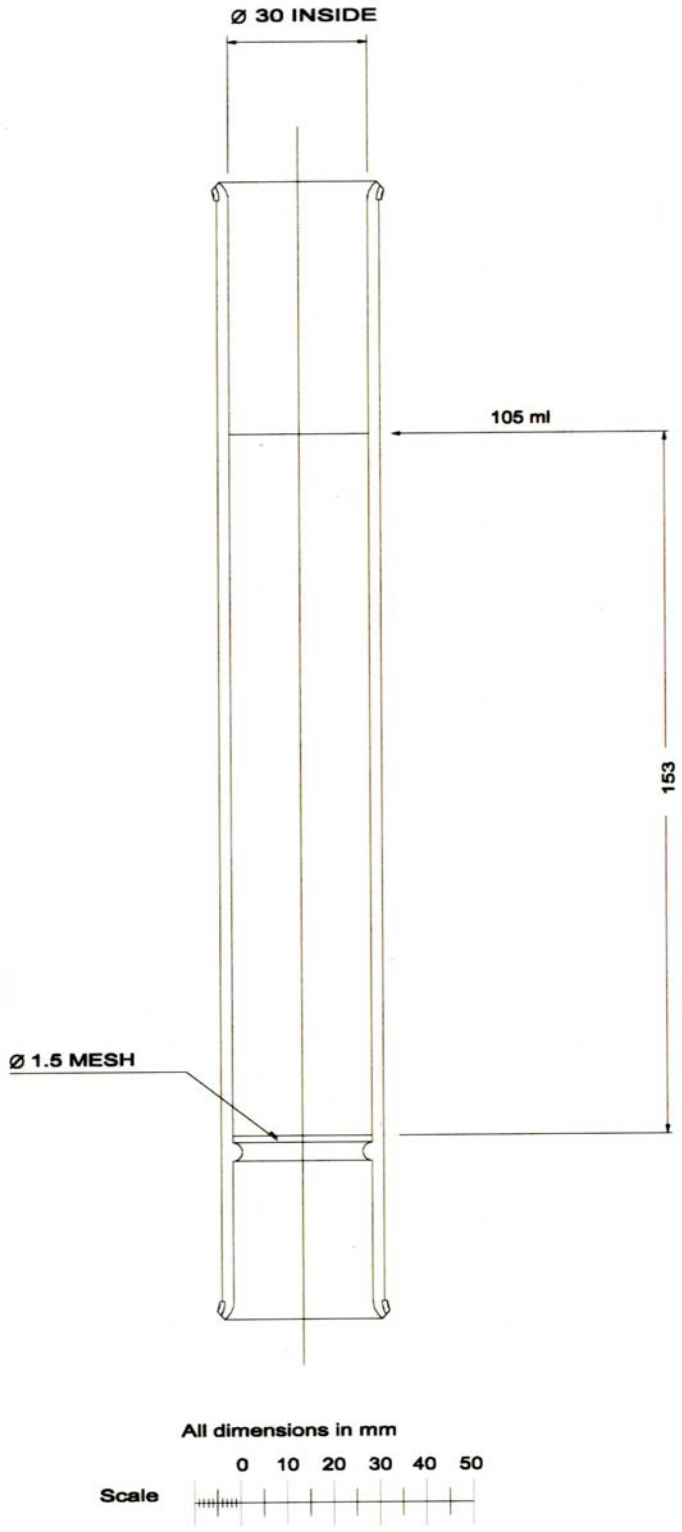

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Figure A3. Snow Storm Filler for Activity Tube

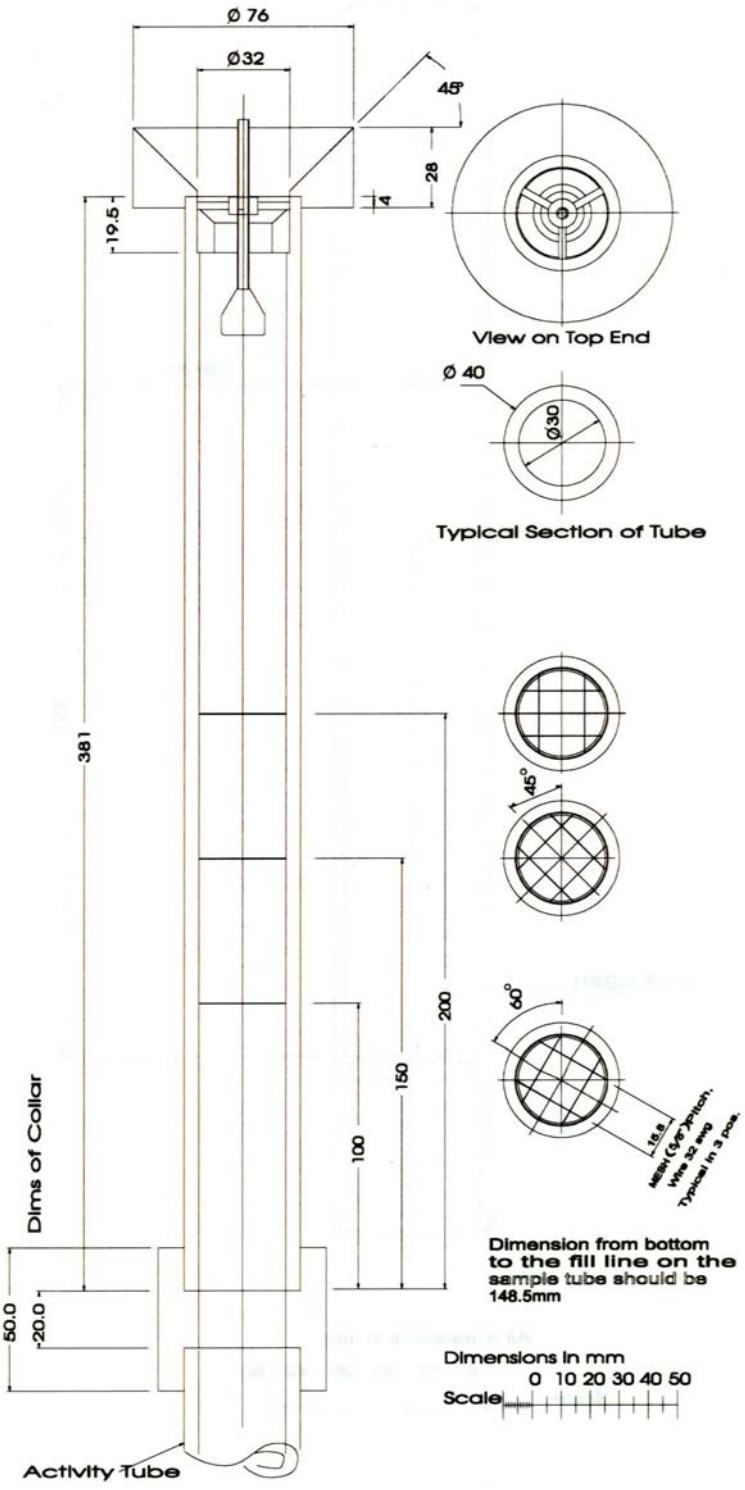

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Figure A4. Snow Storm Filler – Throat Detail

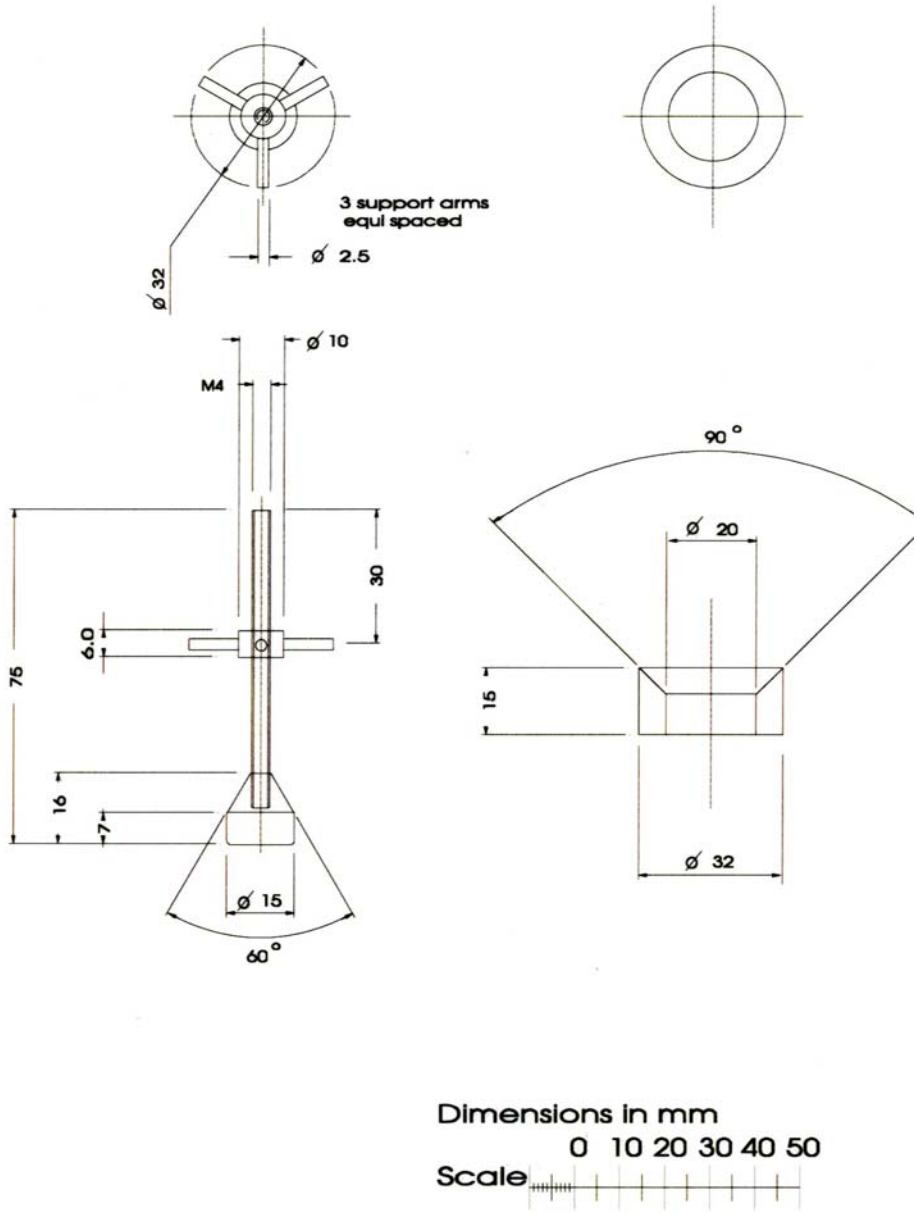

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Table A-2. Carbon Dioxide Absorbent Test Report

Time, Date	
Test Reference	
Material Type/Name	
Manufacturer	
Batch No	
Date of Manufacture	

TEST RESULT

Granule Size/Friability	**Granule Size Distribution %**	**Friability Distribution %**
Retained on mm Sieve		
Retained on mm Sieve		
Retained on mm Sieve		
Retained on mm Sieve		
Retained on mm Sieve		
Retained on final 0.6 mm Sieve		
Dust Load (within receiver)		

Activity Test	**Time to Reach 0.50 % v/v CO_2 minutes**
Activity	
Number of samples tested	

Flow Resistance	**Maximum flow resistance mbar**
Flow resistance	
Number of samples tested	

Volatile Content	**Volatile Content %**
Volatile content	
Number of samples tested	

Table A-2: Carbon Dioxide Absorbent Test Report (Continued)

Initial Carbonate Content	**Carbonate Content %**
Carbonate content	
Number of samples tested	

Contaminants	**Indicator Colour Change**
Contaminant	
Number of samples tested	

Remarks:

Analyst: Date:

Laboratory Manager: Date:

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ANNEX B - CRITERIA NATO CARBON DIOXIDE ABSORBENT GRADES

Table B-1. Criteria for NATO Carbon Dioxide Absorbent Grades

Test	**Limits**					
	NATO S-H[1]	**NATO S-L[2]**	**NATO L-H[3]**	**NATO L-L[4]**	**NATO Li-S[5]**	**NATO Li-L[6]**
GRANULE / FRIABILITY SIZE (%)						
Retained on 5.60 mm sieve	-	-	1.0 max	1.0 max	-	1.0 max
Retained on 4.75 mm sieve	-	-	7.0 max	7.0 max	-	7.0 max
Retained on 2.80 mm sieve	1.0 max	1.0 max	-	-	1.0 max	-
Retained on 2.00 mm sieve	30.0 max	30.0 max	76.0 min	76.0 min	30.0 max	76.0 min
Retained on 1.40 mm sieve	48.0 min	48.0 min	-	-	48.0 min	-
Retained on 0.60 mm sieve	20.0 max	20.0 max	15.0 max	15.0 max	20.0 max	15.0 max
Dust Load	1.0 max	1.0 max	1.0 max	1.0 max	1.0 max	1.0 max
Friability	3.0 max	3.0 max	3.0 max	3.0 max	3.0 max	3.0 max
ACTIVITY (Time minutes)	≥ 90	≥ 60	≥ 90	≥ 60	≥ 125	≥ 125
FLOW RESISTANCE (mbar)	≤ 1.4	≤ 1.4	≤ 1.2	≤ 1.2	≤ 1.4	≤ 1.2
VOLATILE CONTENT (%)	14 - 20	14 - 20	14 - 20	14 - 20	< 1.0	< 1.0
INITIAL CARBONATE CONTENT (%)	6.0 max	6.0 max	6.0 max	6.0 max	6.0 max	6.0 max
CONTAMINANTS						
Indicator	None	None	None	None	None	None

Absorbent Designations:

1 Soda lime, small grain, high activity.

2 Soda lime, small grain, low activity.

3 Soda lime, large grain, high activity.

4 Soda lime, large grain, low activity

5 Lithium hydroxide, small grain.

6 Lithium hydroxide, large grain.

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