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DOMESTIC PREPAREDNESS PROGRAM: TESTING OF MSA DETECTOR TUBES AGAINST CHEMICAL WARFARE AGENTS SUMMARY REPORT

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This report characterizes the chemical warfare (CW) agent detection potential of the commercially available MSA Detector Tubes. These detector tubes were tested against HD, GB and GA vapor at various conditions. This report is intended to provide the emergency responders concerned with CW agent detection an overview of the detection capabilities of these detector tubes.

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PREFACE

The work described herein was authorized under the Expert Assistance (Equipment Test) Program for the U.S. Army Soldier and Biological Chemical Command (SBCCOM) Program Director for Domestic Preparedness. This work was started in April 1999 and completed in November 1999.

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DOMESTIC PREPAREDNESS PROGRAM: TESTING OF MSA DETECTOR TUBES AGAINST CHEMICAL WARFARE AGENTS SUMMARY REPORT

1. INTRODUCTION

The Department of Defense (DOD) formed the Domestic Preparedness (DP) Program in 1996 in response to Public Law 104-201. One of the objectives is to enhance federal, state and local capabilities to respond to Nuclear, Biological and Chemical (NBC) terrorism incidents. Emergency responders who encounter a contaminated or potentially contaminated area must survey the area for the presence of toxic or explosive vapors. Presently, the vapor detectors commonly used are not designed to detect and identify chemical warfare (CW) agents. Little data are available concerning the ability of these commonly used, commercially available detection devices to detect CW agents. Under the Domestic Preparedness (DP) Expert Assistance (Test Equipment) Program, the U.S. Army Soldier and Biological Chemical Command (SBCCOM) established a program to address this need. The Design Evaluation Laboratory (DEL) at Aberdeen Proving Ground, Edgewood, Maryland, performed the detector testing. DEL is tasked with providing the necessary information to aid authorities in the selection of detection equipment applicable to their needs.

Several detectors were evaluated and reported during Phase 1 testing in 1998. Phase 2 testing in 1999 continues the evaluation of detectors including the MIRAN SapphIRe Portable Ambient Air Analyzer, MSA tubes, the APD2000, and the M90-D1-C Chemical Warfare Agent Detector.

2. OBJECTIVE

The objective of this test is to provide emergency responders concerned with CW agent detection an overview of the capabilities of the Mine Safety Appliances (MSA) detector tubes to detect chemical warfare agent vapors. Two types of MSA tubes were evaluated for their ability to detect CW agents at low concentrations. The phosphoric acid ester (PAE) tube for nerve agent detection and the mustard (HD) tube for blister agent detection. This summary report is one of several reports on the Phase 2 evaluations of detectors conducted during 1999.

3. SCOPE

The scope of this evaluation is to characterize the CW agent vapor detection capability of the MSA detector tubes. The agents used included Tabun (GA), Sarin (GB), and Mustard (HD). These were chosen as representative CW agents because they are believed to be the most likely threats. Test procedures followed those described in the Phase 1 Test Report[1]. The test concept was as follows:

a. For each selected CW agent, determine the minimum concentration levels (Minimum Detectable Level, MDL) where repeatable detection readings are achieved. The advertised sensitivity for the respective tube is used as a guide for detection sensitivity objectives.

b. Investigate the effects of humidity and temperature on the detection response.

c. Observe the effects of potential interfering vapors upon detection performance both in the laboratory and in the field.

4. EQUIPMENT AND TEST PROCEDURES

4.1. DETECTOR DESCRIPTION

Mine Safety Appliances Company (MSA/Auer) manufactures the detector tubes used in these evaluations. The detector tubes are slender glass tubes approximately 5 inches long that are filled with reagents and reagent-impregnated granular solids appropriate for the type of substance to be sampled. MSA produces tubes for measuring more than 120 gases and vapors. Two types of tubes for chemical agent detection were tested in this evaluation. They included the HD detector tubes and the Phosphoric Acid Ester (PAE) nerve agent detector tubes.

Operating procedures were followed according to the instruction sheet provided in each box of detector tubes[2]. The operational temperature range for the detector tubes is given as –5°C to 55°C (23°F to 130°F) with relative humidity conditions between 10% and 90%. The specification for storage and transport temperatures is up to 25°C (77°F). However, tubes were stored at room temperatures during the evaluation.

The MSA instruction sheet included in the box of tubes describes the chemical reaction and color change that takes place. In addition, the respective step by step sampling procedure for each tube type is presented on the instruction sheet. Figure 1 is a digital photograph of the MSA tubes beside their respective boxes.

Figure 1. MSA Detector Tubes

4.2. TUBE SAMPLING PROCEDURES

The agent vapor was drawn directly from the vapor generator into the prepared tube. This was accomplished manually by use of a Kwik-Draw™ bellow pump or other suction source that pulls the respective equivalent sample volume through the tubes. The tubes were activated as directed and color development was observed. Blank tests were run, as references, exactly as the agent tests by sampling the generator's conditioned air without agent at the respective temperature and humidity conditions. A positive response indicates the appropriate color change within the time requirement for the tube tested.

These semi-quantitative tubes are advertised capable of detecting low concentrations of CW vapors. The HD detection tubes are designed for detection of HD vapor down to 1 mg/m^3 (0.001 mg/l) using 50 pump strokes. The PAE nerve agent detector tubes are capable of detecting GB, GD, VX, GP, GA and GF in air to approximately 0.01mg/m^3 (0.00001 mg/l) using 10 pump strokes. Each pump stroke draws in approximately 100 milliliters of sample.

The Phosphoric Acid Ester (PAE) nerve agent detector tube contains two reagent ampoules and two reaction layers sealed in a glass tube marked with three red bands. The sealed ends of the glass tube are broken to begin use. The reagent ampoule at the three-band end is then crushed and its content is shaken onto the white cellulose layer that contains an enzyme. The tube is then inserted to the inlet of a Kwik-Draw™ pump or other suction source. Ten Kwik-Draw™ pump strokes or other suction source is used to draw in approximately one liter of sample air for analysis. Two minutes after the sample is collected the second reagent ampoule is crushed allowing the solution to pass through the yellow substrate layer. Then, the user is to shake the liquid to the white enzyme layer where the collected sample has reacted with the enzyme. A yellowish color will form before two minutes if less than detectable nerve agent is present. The white layer will remain white after the two minutes wait when the required concentration of nerve agent is detected.

The HD Detection Tube is used without preparation except to break off both glass tips. The tube is then attached to a suction pump to draw in approximately five liters of sample. The sample was drawn using fifty strokes of the Kwik-Draw™ pump or using a 1 liter per minute suction pump for 5 minutes. A reddish orange band will appear in the presence of the required concentration of HD. The intensity and broadness of the color band is directly related to the dosage sampled as shown on the outside of the box of MSA tubes.

4.3. AGENT CHALLENGE

The agent challenges were conducted using the Multi-Purpose Chemical Agent Vapor Generation System[3] with Chemical Agent Standard Analytical Reference Material (CASARM) grade CW agents. The vapor generator permits preconditioning of a detector with controlled humidity and temperature air before challenging it with similarly conditioned air containing the CW agent.

Agent testing followed successful blank tests of the detector tubes. Agent challenge begins after the generator's solenoids are energized to switch the air streams from the conditioned air only to the similarly conditioned air containing the agent. Three detector tubes were tested in succession under each condition. The agent's challenge time is the time it takes to pull the respective volume through the detector tube.

The detector tubes were tested with the agents GA, GB and HD at different concentration levels at ambient temperature and low relative humidity in an attempt to determine the minimum detectable level (MDL). Additionally, the detectors were tested at different relative humidity conditions (50% and 90%) and temperature extremes of -5°C and +55°C to observe potential temperature and humidity effects. The MSA tubes were also tested at 10°C because of their failure to detect at the colder temperatures.

The detector tubes were placed in the environmental temperature chamber for temperature conditioning before being used. Blanks were tested by sampling the dry conditioning air from the vapor generator first to observe temperature effects on the detector tubes in absence of chemical agent vapor. The detector tubes were then tested at the prescribed concentration of chemical agent. Blanks, agent challenges, and color development occurred in the same temperature environment to assure consistent temperature during the testing process.

4.4. AGENT VAPOR QUANTIFICATION

The generated agent vapor concentrations were analyzed independently and reported in mg/m^3. The vapor concentration was quantified by the manual sample collection methodology[3] using the Miniature Continuous Air Monitoring System (MINICAMS®) manufactured by O. I. Analytical, Inc., Birmingham, Alabama. The MINICAMS® is equipped with a flame photometric detector (FPD), and operated in phosphorus mode for the G agents and sulfur mode for HD. This system normally monitors air by collection through sample lines and subsequently adsorbing the CW agent onto the solid sorbent contained in a glass tube referred to as the pre-concentrator tube (PCT). The PCT is located after the MINICAMS® inlet. Here the concentrated sample is periodically heat desorbed into a gas chromatographic capillary column for subsequent separation, identification, and quantification.

For manual sample collection, the PCT was removed from the MINICAMS® and connected to a measured suction source to draw the vapor sample from the agent generator. The PCT was then re-inserted into the MINICAMS® for analysis. This "manual sample collection" procedure eliminates potential loss of sample through sampling lines and the inlet assembly in order to use the MINICAMS® as an analytical instrument. The calibration of the MINICAMS® is performed daily using the appropriate standards for the agent of interest.

4.5. FIELD INTERFERENCE TESTS

After the agent sensitivity tests, the tubes were tested outdoors in the presence of common potential interferents such as the vapors from gasoline, diesel fuel, jet propulsion fuel (JP8), kerosene, AFFF liquid (Aqueous Film Forming Foam used for fire fighting), household chlorine bleach and insect repellent. Also included were vapor from 10% HTH slurry (a chlorinating agent decontaminant), engine exhausts, burning fuels, and other burning material smokes.

The field tests were conducted at M-Field of the Edgewood Area of Aberdeen Proving Ground in July 1999. The detector tubes were each connected to a one liter per minute suction pump placed at various distances downwind from the source. For example, they were placed at 1-3 meters away for fumes tests or 25 meters for smoke tests depending on wind velocity at test time. The objective was to assess the ability of the detectors to withstand outdoor environments and to resist "false positive" indications when exposed to the selected "potential interference" substances.

Three of each type (PAE for nerve agent and HD) of tubes were exposed against each interferent. Blank tests of both detector tubes were performed in the 'clean' field environment away from known interferents to assure that the detector tubes did not yield false positives prior to "interferent' exposures.

4.6. LABORATORY INTERFERENCE TESTS

These tests were designed to assess the detector tube response to vapor from representative substances, and to show the CW agent detection capability of the tubes in the presence of the potential interference vapors from AFFF and diesel fuel. The interferents were chosen based on the likelihood of their presence during an emergency response by first responders.

The detector tubes were screened against "1% concentrations" of gasoline, JP8, diesel fuel, household chlorine bleach, floor wax, AFFF, Spray 9 cleaner, Windex, antifreeze, toluene, vinegar, and 25 PPM ammonia to observe potential interference with the detection reaction process. If the tubes gave false positive results at 1%, they were tested against an "0.1% concentration" of each interferent. To prepare the interferent test gas mixture, dry (<5% RH) air at 20°C was saturated with interferent vapor by passing it through the interferent liquid in a bubbler or by sweeping it over the liquid contained in a tube. Thirty milliliters of this vapor saturated air was then diluted to three liters of the conditioned air to produce the "1% concentration" of interferent. In the same manner, a 0.1% concentration of interferent was produced using three milliliters of vapor saturated air diluted to 3 liters of generator air to further test the detector tubes if they false alarmed at the higher concentration. The 25 ppm ammonia was derived by proper dilution of the 1% NH_3 vapor from an analyzed compressed gas cylinder. The 25 ppm ammonia concentration was chosen as representative of possible occurrences in typical CW protective shelters.

The CW agent detection capability of the MSA tubes in the presence of the potential interference vapors from AFFF and diesel fuel was assessed. The test mixture was prepared similarly to produce the 1% or 0.1% 'concentrations' of potential interference vapor but the prescribed concentration of CW agent from the agent generator was included in the test exposures.

5. RESULTS AND DISCUSSION

5.1. MINIMUM DETECTABLE LEVELS

The minimum detectable levels (MDL) for the two types of detector tubes (PAE for nerve agent and HD) for each agent at ambient temperatures and low (<10%) relative humidity (RH) are shown in Table 1. The current military requirements for CW agent detection (Joint Service Operational Requirements [JSOR] for CW agent sensitivity for point detection alarms) and the Army's established values for Immediate Danger to Life or Health (IDLH) and Airborne Exposure Limit (AEL) are also listed as references to compare the detector tubes' performance. The MDL was established by lowering the agent concentrations until there was no agent detection from the detector tubes. The MDL values were selected based on the lowest CW agent concentration exposure to produce positive color development consistently for three trials. MDL of the MSA tubes does not reflect the actual chemical agent concentration of the contaminated area, only the lowest level of detection capability, thus the MSA tubes cannot be used in decision making to lower the level of required personal protection.

Table 1. Minimum Detectable Level (MDL) of MSA tubes at Ambient Temperatures and Low Relative Humidity

AGENT	Concentration in milligrams per cubic meter, mg/m^3, With parts per million values in parenthesis (ppm)			
	MDL	JSOR*	IDLH**	AEL***
HD	2.97 (0.454)	2.0 (0.30)	N/A	0.003 (0.0005)
GA/GB	0.01 (0.002)	0.1 (0.017)	0.2 (0.03)	0.0001 (0.000015)

* Joint Service Operational Requirements for point sampling detectors.
** Immediate Danger to Life or Health values from AR 385-61 to determine level of CW protection. Personnel must wear full ensemble with SCBA for operations or full face piece respirator for escape.
*** Airborne Exposure Limit values from AR 385-61 to determine masking requirements. Personnel can operate for up to 8 hours unmasked.

When compared to the JSOR and IDLH values, the MDLs of the PAE nerve agent tubes for the nerve agents tested (GA/GB) are approximately an order of magnitude lower (better). The PAE nerve agent tubes were found capable of responding consistently to very low concentrations of nerve agents. The HD detector tubes were found to detect HD concentrations at approximately the JSOR level, however, army regulation AR 385-61 does not establish an IDLH for HD due to concerns over carcinogenicity. Neither the HD nor PAE nerve agent tubes detected to the AEL values.

5.2 TEMPERATURE AND HUMIDITY EFFECTS

Table 2 shows the effects of temperature and humidity changes on the minimum detectable levels for the MSA tubes evaluated. Tests were conducted at ambient temperatures and RH conditions of approximately 0, 50 and 90%. The detectors were also tested at temperature extremes of –5°C and +55°C.

The tubes successfully demonstrated CW agent detection at most of the temperature and humidity conditions. None of the tubes worked correctly in cold temperatures of -5°C. At -5°C, all the PAE nerve agent blank tubes evaluated showed false positives and the HD tubes showed false negatives. The tubes were then tested at modified cold temperatures of 10°C. The HD tubes required a higher concentration of HD before positive detections were observed at this temperature. The PAE nerve agent tubes, however, functioned properly at 10°C.

At 20°C and 90% RH, the HD tubes could not detect HD even at the relatively high concentration of approximately 10 mg/m³. High humidity negatively affected the HD tubes. The PAE nerve agent tubes, however, were still able to detect the nerve agents at the concentrations found at lower RH conditions.

A suction pump was used occasionally instead of the Kwik-Draw™ manual sample collection method, especially where 50 pump stokes were needed for the HD tubes. Both the Kwik-Draw™ bellow pump and other suction source pulling the respective equivalent sample volume through the tubes were used. Similar results were observed between the different techniques.

Table 2. Temperature and Humidity Effects on CW Detection Limits of MSA Tubes

Agent	Average Temperature °C	Relative Humidity %RH	HD Tubes		Phosphoric Acid Esters Tubes	
			mg/m³	Ppm	mg/m³	Ppm
HD	20	<10	2.97	0.454	No Capability	
HD	25	50	2.99	0.456		
HD	20	90	Fail			
HD	55	16	2.41	0.369		
HD	10	16	5.3	0.810		
HD	-5	16	Fail			
GA	20	<10	No Capability		0.014	0.0021
GA	20	50			0.014	0.0021
GA	20	90			0.014	0.0021
GA	55	14			0.009	0.0013
GA	10	2			0.011	0.0017
GA	-5	0			Fail	
GB	20	<10	No Capability		0.005	0.0008
GB	20	50			0.011	0.0017
GB	20	90			0.011	0.0016
GB	55	12			0.005	0.0007
GB	10	0			0.01	0.0015
GB	-5	0			Fail	

It should be noted that there was a high degree of difficulty in color development determination. Conflicting opinions occurred among several observers regarding whether or not the results were positive on exposures at the threshold detection concentration level, especially for the PAE nerve agent tubes. Unlike at higher concentrations where the PAE nerve agent tube would stay white for >2 minutes, the tubes showed a slightly "yellowish" white band at the time of reading at near the threshold concentration exposures. Blanks usually yielded a deeper yellowish color development except in cold temperatures. The failure of the tubes to work properly at -5°C reflected depressed enzyme activity. Blank PAE nerve agent tube tests at cold temperatures were indicating positive CW agent detection. When this occurred, the tubes could not be tested at that condition.

HD tubes had similar subjective results such as "slightly positive" or "slightly red". Also, as an example, several people could observe a color change and give different interpretations of tube response. Observers expressed different opinions on the color bandwidth and intensity that constitute a positive response in determining the minimal detection level.

5.3. FIELD INTERFERENCE

The results of the tube evaluations in the field tests are presented in Table 3. No false positives were found for any of the conditions tested. The ambient temperature and relative humidity levels during these tests were in the range of 26-36°C and 53-91% RH, with gentle wind.

Table 3. Summary of Field Interference Testing with MSA Tubes

Interferent	Temperature (°C)	Relative Humidity (%)	Phosphoric Acid Ester (PAE)	HD
Gasoline Exhaust, at Idle	30	57	Negative	Negative
Gasoline Exhaust, at Revved	31	68	Negative	Negative
Diesel Exhaust	36	53	Negative	Negative
Diesel Exhaust, at Revved	36	53	Negative	Negative
Kerosene Vapor	36	53	Negative	Negative
Burning Kerosene Smoke	26	91	Negative	Negative
JP8 Vapor	27	88	Negative	Negative
Burning JP8 Smoke	29	73	Negative	Negative
Burning Gasoline Smoke	29	73	Negative	Negative
Burning Diesel Smoke	29	73	Negative	Negative
Diluted AFFF Vapor	27	88	Negative	Negative
Insect Repellent	29	73	Negative	Negative
Diesel Vapor	27	88	Negative	Negative
Gasoline Vapor	27	88	Negative	Negative
10% HTH Vapor	27	88	Negative	Negative
Bleach Vapor	27	88	Negative	Negative
Burning Cardboard Smoke	26	91	Negative	Negative
Burning Cloth Smoke	26	91	Negative	Negative
Burning Wood Fire Smoke	26	91	Negative	Negative
Doused Wood Fire Smoke	26	91	Negative	Negative
Burning Tire Smoke	26	91	Negative	Negative

Note: Negative response reading indicates no color change for the HD tube = no agent detection. For the PAE nerve agent tubes, a negative response means a color change to yellow = no agent detection.

5.4. LABORATORY INTERFERENCE TESTING

Laboratory screening of potential interference is summarized in Table 4. These tests were conducted without using the CW agents in order to supplement the field interference testing, under a more controlled concentration. If a positive test occurred at the 1% saturation level, the concentration was reduced to 0.1% saturation and tested again. Those substances that did not cause false positive indications at the 1% level were not further tested at the 0.1% level. Diesel vapor at 1% and 0.1% concentration levels caused false positives on the PAE nerve agent tubes. All other tests were negatives. Although diesel fuel vapor did not cause positive indication when tested outdoors, it did cause the PAE nerve agent tubes to give false positives when tested in the laboratory. This suggested that the vapor concentration achievable in outdoor environment must be below the 0.1% saturation level. This reinforces the validity of choosing the 0.1% vapor mixture for laboratory screening testing as being meaningful.

Table 4. Results of Laboratory Interference Tests without Agents

Interference	HD Tubes		Phosphoric Acid Esters Tubes	
	1%	0.1%	1%	0.1%
AFFF	Negative	Not tested	Negative	Not tested
Bleach	Negative	Not tested	Negative	Not tested
Diesel	Negative	Not tested	Positive	Positive
Floor Wax	Negative	Not tested	Negative	Not tested
Gasoline	Negative	Not tested	Negative	Not tested
JP8	Negative	Not tested	Negative	Not tested
Spray 9	Negative	Not tested	Negative	Not tested
Vinegar	Negative	Not tested	Negative	Not tested
Windex	Negative	Not tested	Negative	Not tested
Ammonia	Negative	Not tested	Negative	Not tested

Table 5 presents the results of testing of GA, GB or HD in the presence of diesel fuel vapor or AFFF vapor at 20°C. Results indicate that these detector tubes were able to detect the CW agents in the presence of these potential interfering vapors if the interference concentration did not cause false positives.

Table 5. Results of Laboratory Interference Tests with Agents

Agent	Interferent	Concentration		Response	
		mg/m³	ppm	HD Tubes	Phosphoric Acid Ester Tubes
GA	0.1% AFFF	0.0075	0.0011	No Capability	Positive
GA	0.1% Diesel	-	-	No Capability	Not Tested *
GB	0.1% AFFF	0.0062	0.0011	No Capability	Positive
GB	0.1% Diesel	-	-	No Capability	Not Tested *
HD	1% AFFF	3.03	0.4643	Positive	No Capability
HD	1% Diesel	3.03	0.4643	Positive	No Capability

* Not tested due to positive indication for 0.1 % diesel fuel vapor

6. DISCUSSION

The MSA tubes, both the phosphoric acid ester tube for nerve agent detection and the HD tube for mustard detection, showed consistent results. The phosphoric acid ester tube detected GA and GB at a minimum concentration of approximately 0.01mg/m^3. The HD tube detected HD at a minimum concentration of approximately 3 mg/m^3. These detectors, however, gave false positives at cold temperatures of -5°C. High humidity also appeared to affect the HD tubes significantly. The HD tubes failed to detect agent at ambient temperature in 90% RH even at a high concentration of 10 mg/m^3 HD.

Civilian first responders and HAZMAT personnel use Immediate Danger to Life or Health (IDLH) values to determine levels of protection selection during consequence management of an incident. Army Regulation (AR) 385-61 provides IDLH and AEL values for GA/GB, and an AEL value for HD. AR 385-61 does not establish an IDLH for HD due to concerns over carcinogenicity. The MSA detector tubes demonstrated detection of G agents to meet the IDLH values, however, are unable to meet the AEL values for HD or GA/GB.

The MSA detector tubes are relatively inert to potential interference. Field interference evaluations did not produce any false positive indications. The potential interference substances tested in the laboratory only showed false positive indication when the PAE nerve agent tubes were exposed to the 0.1% or the 1% diesel vapor. Therefore, PAE nerve agent tubes were not tested against agent in the presence of diesel vapor. However, the HD tubes were unaffected by any of the interferents tested, and retained CW agent detection capability in presence of AFFF and diesel vapor. PAE nerve agent tubes retained CW agent detection capability in the presence of AFFF.

Neither MSA tube type would perform well in the cold temperature of -5°C. The PAE nerve agent tubes produced false positive indications on blank runs (absence of CW agent) and the HD tubes showed false negatives. In addition, the HD tubes did not perform well in high moisture conditions. The tubes failed to detect HD at high humidity even at much higher than the determined MDL concentration levels.

There was a large amount of subjectivity in determining the color change or lack of change for positive indication near the CW agent threshold levels. Under threshold level conditions, the results were not clearly distinguishable. Positive detection indications, however, were more distinguishable at CW agent vapor concentrations higher than threshold detection levels.

7. CONCLUSION

The effectiveness in using the HD tubes is questionable because of the vulnerability of HD tubes to fail under moderately "moist" conditions. Using the HD tubes in foggy, rainy, or even at low RH but higher temperatures (moisture content is high as compared to the 90%RH at 20^{o}C) situations will likely result in failures. The poor performance of both the PAE nerve agent and HD tubes at "cold" temperatures is also an issue of concern. At -5^{o}C, the HD tubes cannot detect HD and the PAE nerve agent tubes are producing false positive indications. It appears that the cooler temperature subdued the chemical reactions required for these tubes to function properly. The specified storage and transport temperatures requirement of the tubes of less than 25°C could pose a problem. It suggests that the tubes are subject to deterioration at higher temperatures. Users must recognize these limitations if these tubes are to be used.

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