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Density, Compressibility, Thermal Conductivity, and Mechanical Movement of Solid Hydrazine

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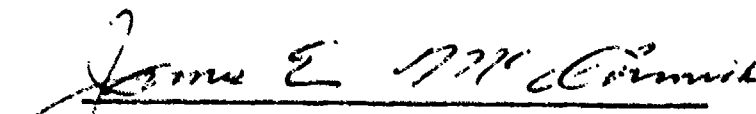
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FOR THE COMMANDER


JAMES E. MCCORMICK
Deputy for Space Defense Systems

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19. KEY WORDS (Continued)

20. ABSTRACT (Continued)

measured at various temperatures and pressures to obtain $\rho_s = 1 \times 10^{-4} \text{ ft}^3$ (ft³ atm), which is independent of temperature from -30 to -4°C and pressure from 0 to 30 lb/in². The thermal conductivity, k_s , of solid hydrazine was also measured; the result is $k_s = 2.52 \times 10^{-4} \text{ Btu/(sec ft}^2 \text{ }^\circ\text{F)}$. The foregoing values (except the elevation of freezing point) were compared with the corresponding values for liquid hydrazine. $\beta = 0.00252 \text{ deg}$

The rates of movement of solid hydrazine in straight and 120°-bent tubes at various temperatures by pressure exerted from one end were measured. The water added in hydrazine permits easier movement of solid because of the presence of eutectic liquid particles distributed in the solid. Extensive results on the gliding behavior are summarized. Recommendations are made to alleviate stresses caused by freezing and melting of hydrazine in confining tubes and valves.

* .0001 cu ft atm

PREFACE

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I. INTRODUCTION

Experimental results on the thermophysical and mechanical behavior of solid hydrazine are scarce. Such results are very useful in interpreting the failures caused by freezing and remelting of hydrazine in valves and constricted tubes on spacecraft. An estimation of the stresses caused by repeated phase changes in confined hydrazine requires the densities and compressibility coefficients of liquid and solid hydrazine as functions of temperature. Whereas most of the required data are available for liquid hydrazine, virtually no significant data exist for solid hydrazine. In addition, the mechanical movement of pure and water-containing solid hydrazine in straight and bent tubes is essential in estimating the stress-relief characteristics under relevant conditions of pressure and temperature.

II. DENSITY OF HYDRAZINE

The density of pure solid hydrazine was measured in the pyrex densitometer shown in Fig. 1. The experimental method, described elsewhere in detail (Ref. 1), consists of measuring the volume of a precisely known weight of hydrazine. The volume change with temperature was determined by measuring the level of a fluid in a capillary stem calibrated to read with an accuracy of ± 0.0001 ml. About 28 g of pure distilled hydrazine was placed in the densitometer, degassed under a vacuum, and weighed accurately to ± 0.0002 g. Half of the liquid hydrazine was frozen slowly under a vacuum; the remaining half, under 1 atm pressure of helium to form a bubble-free mass of solid. After freezing, the densitometer was evacuated and a known amount of a silicone oil was injected on solid hydrazine to the level shown in Fig. 1. Helium was then admitted to assure that solid hydrazine was surrounded by liquid silicone. The densitometer was inserted in a thermostat controlled to $\pm 0.005^\circ\text{C}$. The volume change was read with a cathetometer to ± 0.0001 ml or with an accuracy of 0.0003% of the total volume. After several measurements at various constant temperatures, the densitometer was taken out, brought to room temperature, and reweighed to determine the weight of silicone oil. In a separate experiment, the density and compressibility of silicone oil was measured also in order to calculate the volume of solid hydrazine from the combined volumes of silicone and hydrazine. In another separate experiment, it was found that liquid hydrazine and silicone oil are insoluble in one another.

Density values at various temperatures, obtained by the preceding method, are listed in Table 1. The data were fitted by the method of least squares to obtain the following equation for the density, ρ_s :

$$\rho_s(\text{solid, g/ml}) = 1.1869 - 0.000675 t \quad (t \text{ in } ^\circ\text{C}) \quad (1)$$

The maximum deviation of the data from this equation is 0.17%. The density from Eq. (1) at -5°C is 1.1903, whereas a single experimental value obtained

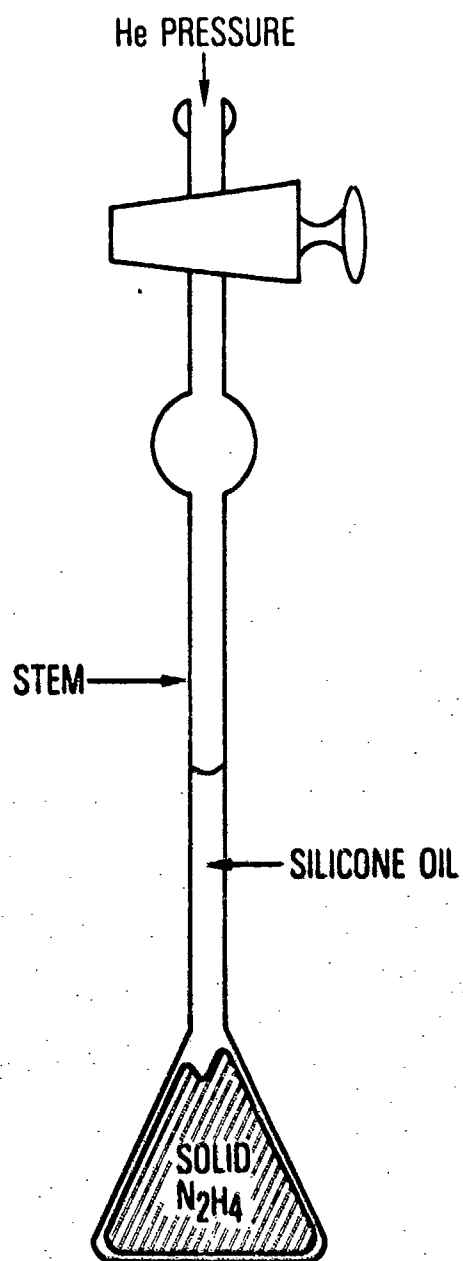


Fig. 1. Densitometer

Table 1. Density of Solid Hydrazine at Various Temperatures

Density, ρ_s , g/ml	Temperature, t, °C			
	-30.00	-20.00	-4.50	-4.20
Experiment No. 1 ^a	1.2052	1.2009	1.1891	
Experiment No. 2 ^a	1.2073	1.2027		1.1894
^a Two separate batches of hydrazine were frozen at different rates to ascertain reproducibility of results.				

in 1943 (Ref. 2) is 1.146; the difference is 3.7%. The density of liquid hydrazine, obtained previously by the authors' technique (Ref. 3), is as follows:

$$\rho_l(\text{liquid, g/ml}) = 1.02492 - 0.000865 t \quad (t \text{ in } ^\circ\text{C}) \quad (2)$$

A comparison of Eqs. (1) and (2) shows that hydrazine, as most substances, expands upon melting, whereas a few substances such as water, arsenic, bismuth, and antimony contract upon melting.

III. ELEVATION OF MELTING POINT WITH INCREASING PRESSURE

The melting point of hydrazine is 2.0°C (275.15 K) at 1 atm pressure (Ref. 4). As the pressure increases, the melting point also increases according to:

$$\frac{\Delta T}{\Delta P} = \frac{V(\text{liquid}) - V(\text{solid})}{\Delta H_m} \times 275.15 \quad (3)$$

where ΔT is the increase in mp for the corresponding increase, ΔP , in pressure in atmospheres; V , the molar volume, in ml; and ΔH_m , the molar heat of melting (Ref. 5), in ml atm/mole. From Eq. (1) and (2), $V(\text{solid}) = 27.03$ ml/mole and $V(\text{liquid}) = 31.32$ ml/mole and, from published compilations (Ref. 6), $\Delta H_m = 3025$ calories/mole or 124,902 ml atm/mole. Substitution of these values in Eq. (3) gives

$$\frac{\Delta T}{\Delta P} = 0.00945^\circ\text{C/atm} \quad (4)$$

For an arbitrarily selected value of $\Delta P = 600$ psig (pounds per square inch gage) = 40.83 atm, the value of ΔT is

$$\Delta T = 0.00945 \times 40.83 = 0.39^\circ\text{C} \quad (5)$$

The increase in the freezing point is 0.39°C (0.70°F) for an increase of 600 psig in pressure. For the same increase in pressure, the freezing point of water is depressed by 0.31°C, i.e., $\Delta T = -0.31^\circ\text{C}$. Additional examples are given in Ref. 5.

If the pressurization gas is nitrogen, the mole fraction of nitrogen, X_2 , at an arbitrarily selected pressure, 600 psig, and at 273.15 K, is 2.18×10^{-4}

in liquid hydrazine (Ref. 7). Dissolved substances (solutes) always depress the freezing point of a liquid solvent when the solid phase does not dissolve the solutes. In general, solid substances at ordinary temperatures do not dissolve solutes having molecular structures different from the solid solvent. The depression of the freezing point, ΔT_{fr} , for dissolved nitrogen is calculated from

$$X_2 = 0.02011 \Delta T_{fr} \quad (6)$$

The result from this equation is $\Delta T_{fr} = 0.011$. The derivation of Eq. (6) is shown in detail in Ref. 5, page 228. The net elevation of the freezing point by pressurization with nitrogen at 600 psig is therefore $0.39 - 0.011 = 0.38^\circ\text{C}$ (0.68°F).

The values of depression of the freezing point of hydrazine with added water, calculated from Eq. (6), are listed in Table 2. These values are in agreement with those by Audrieth and Ogg (Ref. 4).

Table 2. Freezing Points of Hydrazine Containing Water

H ₂ O, wt%	0	0.5	1.0	1.5	2.0
Freezing Point, °C	2.00	1.56	1.12	0.69	0.26

Hydrazine and water constitute a double-eutectic system. The hydrazine-rich eutectic has a freezing point of -54°C , signifying that the last minute quantity of water-bearing hydrazine freezes at -54°C . The amount of final liquid freezing at this temperature can be calculated from the phase diagram by the lever rule (Ref. 5).

IV. COMPRESSIBILITY COEFFICIENT OF HYDRAZINE

The densitometer was also used for measurements of the compressibility coefficient, β_s , of solid hydrazine. For this purpose, the densitometer was pressurized up to 14 psig, and the change in the liquid level was accurately measured. From the expansion of the container and the compressibility of silicone, determined in two separate experiments, β_s was calculated at -30.0, -20.0, and -4.2°C. The results for β_s are independent of pressure and temperature; the average value is as follows:

$$\beta_s = \frac{1}{V} \left(\frac{\Delta V}{\Delta P} \right) = 10 \times 10^{-5} \text{ per atm} \quad (7)$$

For liquid hydrazine the corresponding value from Ref. 8 is

$$\beta_l = 2.54 \times 10^{-5} \text{ per atm} \quad (8)$$

The values of β_s and β_l from Eq. (7) and (8) are comparable.

In general, β_s for most solids is lower than β_l for liquids, but available data are too scarce to make a general statement. In addition, β_s for solids is strongly dependent on crystal orientation when the measurements are made on single crystals.

V. THERMAL CONDUCTIVITY OF HYDRAZINE

The thermal conductivity of solid hydrazine was determined in the apparatus shown in Fig. 2. An upper section containing solid hydrazine between two plates and a thermostated bath, A, was made of plexiglass. Liquid hydrazine was frozen slowly to avoid bubble formation. A small space around the solid was evacuated and filled with silicone oil. The oil was brought to atmospheric pressure to fill any empty space in order to achieve uniform contact between stainless-steel plates and hydrazine. Two thermocouples, TC No. 1 and TC No. 2, were spaced accurately in hydrazine for measuring the temperature gradient. A lower thermostated bath, B, was used as a heat sink. The heat capacity of this bath was measured by precisely known amounts of electrical energy dissipated through a resistance coil.

The apparatus was first used with air in the hydrazine chamber (without solid hydrazine), and the rate of heat leak was determined. Solid hydrazine was then formed in the chamber, and the rate of heat leak across the solid was determined. The difference between two sets of measurements, with air, and with hydrazine, yielded the rate of heat transmission across solid hydrazine, denoted by dQ/dt' , where Q is the heat in calories, and t' , the time in seconds. The surface area of hydrazine, $A = 62.07 \text{ cm}^2$ and the thickness $\Delta X = 1.27 \text{ cm}$ (Fig. 2), was substituted in the following equation to obtain the thermal conductivity k_s :

$$\frac{dQ}{dt'} = -Ak_s \frac{\Delta T}{\Delta X} \quad (9)$$

where $\Delta T = 3.50^\circ\text{C}$ is the temperature difference as read by TC No. 1 and TC No. 2 (Fig. 2). The values of k_s , obtained from experimental measurements, are listed in Table 3.

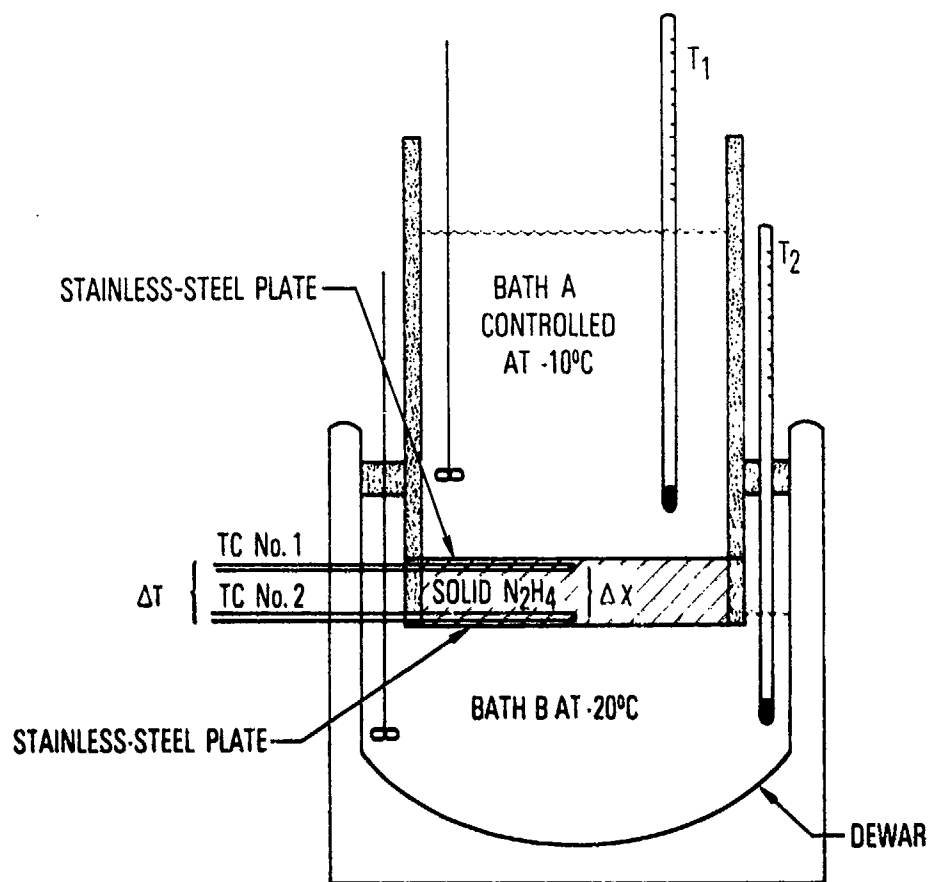


Fig. 2. Apparatus for determination of thermal conductivity

Table 3. Thermal Conductivity of Solid Hydrazine

Run No.	1A	1B	2A	2B	2C
$10^3 k_s$, calories/(sec cm °C)	3.58	3.69	4.07	3.63	3.78

The average result for k_s is given by

$$k_s = 3.75 \times 10^{-3} \text{ cal/(sec cm } ^\circ\text{C)}$$

$$k_s = 2.52 \times 10^{-4} \text{ Btu/(sec ft } ^\circ\text{F)}$$

For the liquid, k_l is 7.86×10^{-5} calories/(sec cm °C) or 5.66×10^{-6} Btu/(sec ft °F) from Ref. 8.

VI. MECHANICAL MOVEMENT OF PURE SOLID HYDRAZINE IN TUBES

Hydrazine frozen in straight and bent stainless-steel tubes forms a cohesive solid that is difficult to move by pushing from one end. When a sufficiently large pressure is exerted from one end of the confined solid, hydrazine moves at various rates, depending on its water content and temperature as well as the configuration of steel tube.

The apparatus (Fig. 3) was designed to investigate the rate of movement of solid hydrazine in stainless-steel tubes 0.25 in. in diameter. Liquid hydrazine was placed in a stainless-steel container, A, and a known length of solid was formed in the 0.25-in. tube, B, by lowering the temperature of the thermostat. A glass rod, C, was used to determine the length of frozen hydrazine and to monitor its movement when nitrogen pressure was transmitted through liquid hydrazine. Similar experiments were also carried out with bent tubes shown on the right side of Fig. 3. There are four variables controlling the rate of movement, i.e., pressure, temperature, configuration of tube, and time. At a given temperature and pressure, the rate of motion can be correlated on two-dimensional coordinates for a tube. However, because of requirements for extensive data, the movement of hydrazine versus pressure was plotted at various pressures, with each period of time at a fixed pressure as a parameter (Figs. 4 to 7, 10, 12, and 14). Figure 4 shows that at -40°F , no detectable motion begins before 1200 psig for a 108-mm-long solid. At this pressure, the solid moves at rapidly decreasing rates, and finally, at 1800 psig, the movement is slow and continuous, without a large decrease in the rate with time. At -30°C , the 88-mm-long solid bent to 120° shows very little motion at 2000 psig. Note that in one experiment, hydrazine frozen at 295 psig moved at a slower rate than hydrazine frozen at atmospheric pressure (Fig. 11). The reason is obviously that a less defective and more adhering solid was formed under 295 psig.

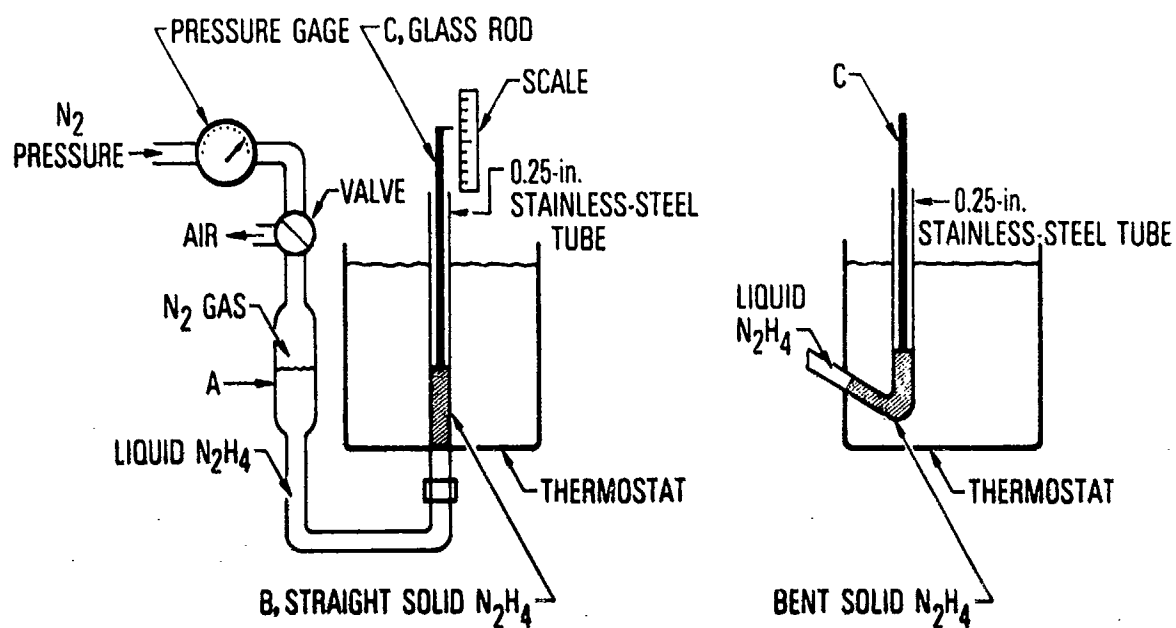


Fig. 3. Apparatus for measurement of movement of solid hydrazine

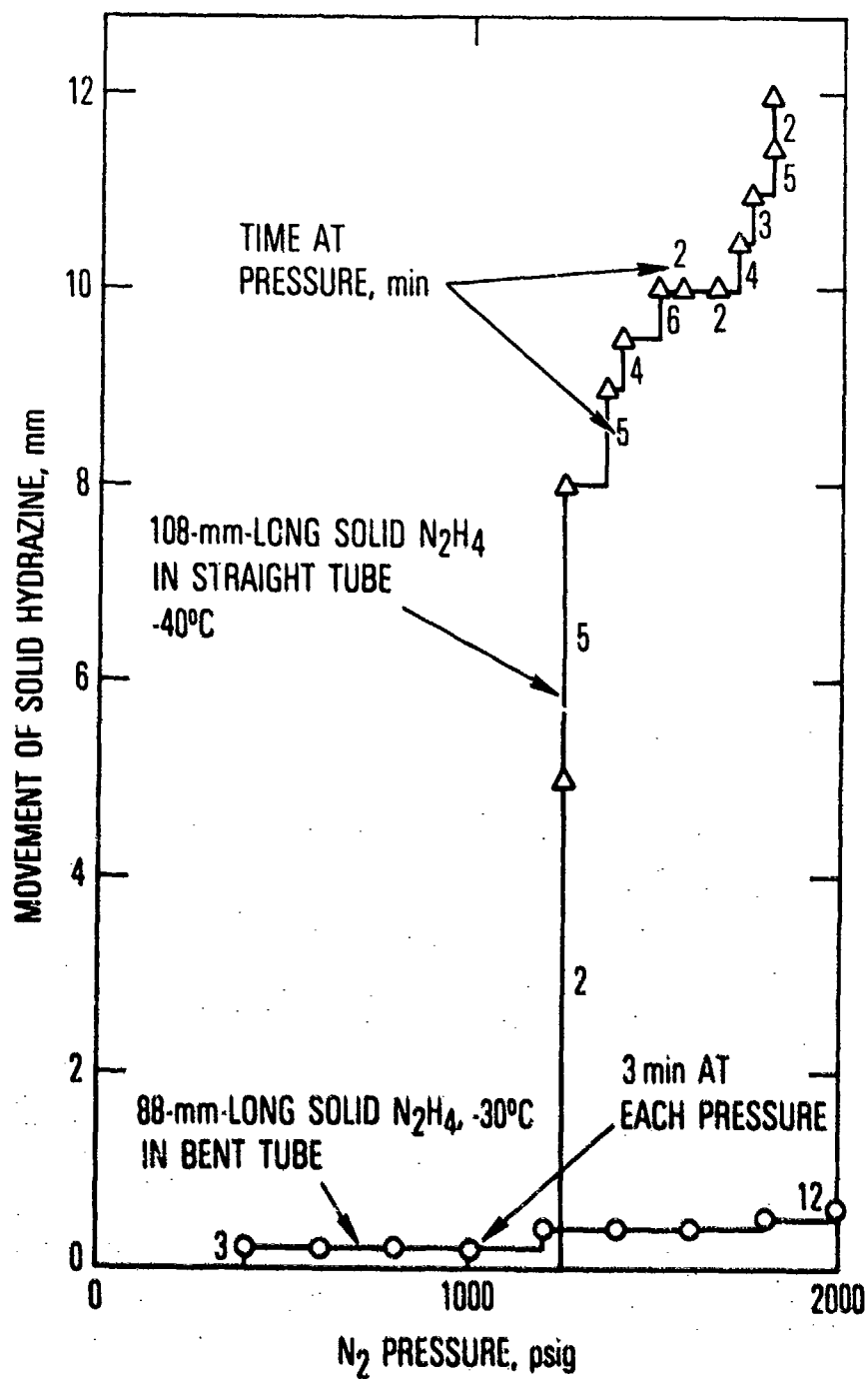


Fig. 4. Movement of pure solid hydrazine in 0.25-in. straight and 120° -bent stainless-steel tubes at $-40^\circ C$ ($-40^\circ F$) and $-30^\circ C$ ($-22^\circ F$)

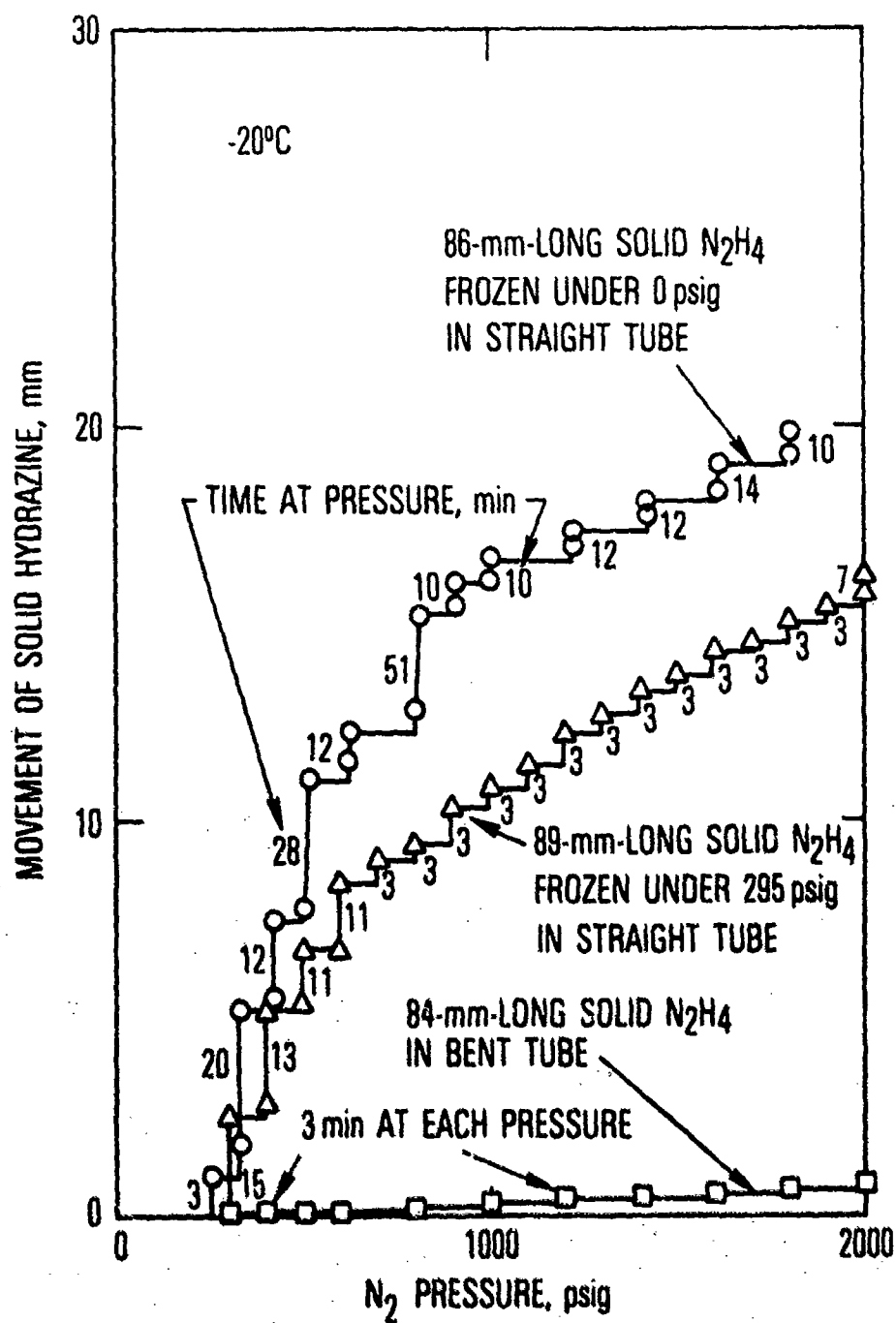


Fig. 5. Movement of pure solid hydrazine in 0.25-in. straight and 120°-bent stainless-steel tubes at -20°C (-4°F). Effect of freezing under pressure in straight tubes is shown by two upper curves.

Figure 6 shows that a 22-mm-long solid moves approximately four times as fast as a 94-mm-long solid in straight tubes at -10°C and 800 psig. Again, the bent tube shows approximately 10% of motion exhibited in the straight tube.

As the temperature approaches the freezing point of hydrazine, there is no difference between straight and bent solid hydrazine up to 800 psi at -1.1°C (Fig. 7). The 56-mm-long bent solid moves fast between 800 and 1000 psi, but thereafter it moves slower than the 85-mm-long straight solid.

A number of conclusions, drawn from Figs. 4 to 7 and shown in Fig. 8, are summarized as follows:

1. The movement at -1.1°C begins at 200 psig and increases in rate with increasing pressure (Fig. 8).
2. At -10 , -20 , and -40°C , increasing pressure decreases the rate of movement by locking-in hydrazine. The pressurized end expands, and liquid hydrazine seeps in and freezes to create a solid slightly larger in diameter on the pressurized end.
3. The initial rates of movement at -10 , -20 and -40°C are roughly comparable (0.33 to 0.48 mm/min) and attain lower values in the neighborhood of 0.1 mm/min with increasing pressure.
4. The movement begins at 250 psig for -10 and -20°C and at 1200 psig for -40°F .

The preceding conclusions are for the straight 0.25-in. stainless-steel tubes. For the tubes bent 120° , similar conclusions were drawn (Fig. 9):

1. The movement starts at 200 psig for -1.1°C and increases in rate as the pressure increases.
2. At -10°C , the rate first increases and then decreases with pressure.
3. At -20 and -30°C , the rates increase. It is likely that they decrease at a pressure much higher than the pressure limit for the apparatus. At -20°C , it is possible to speculate that the curve could have its maximum point in the range of 2000 to 3000 psi.

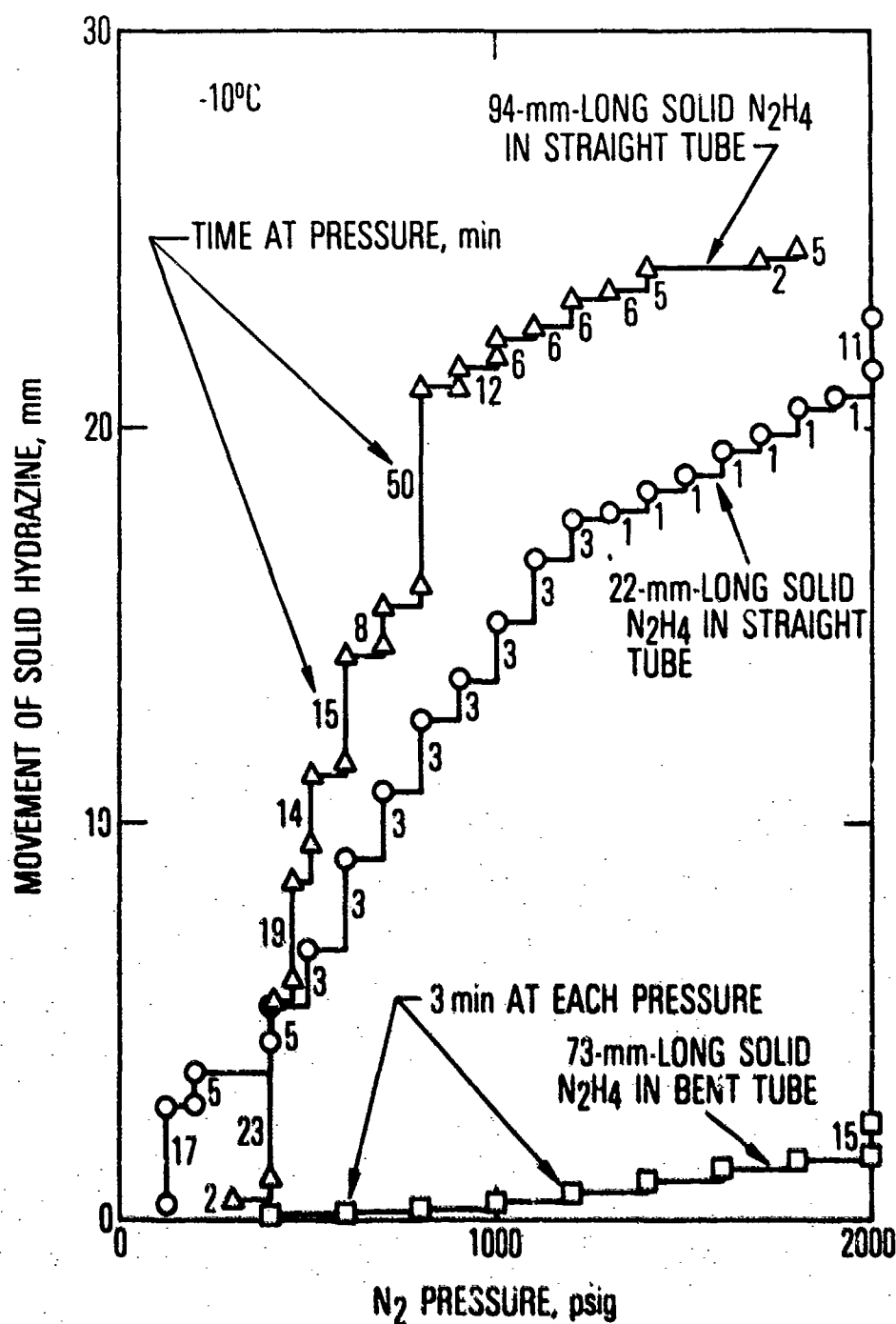


Fig. 6. Movement of pure solid hydrazine in 0.25-in. straight and 120°-bent stainless-steel tubes at -10°C (+14°F). Effect of length of solid hydrazine in straight tubes is shown by two upper curves.

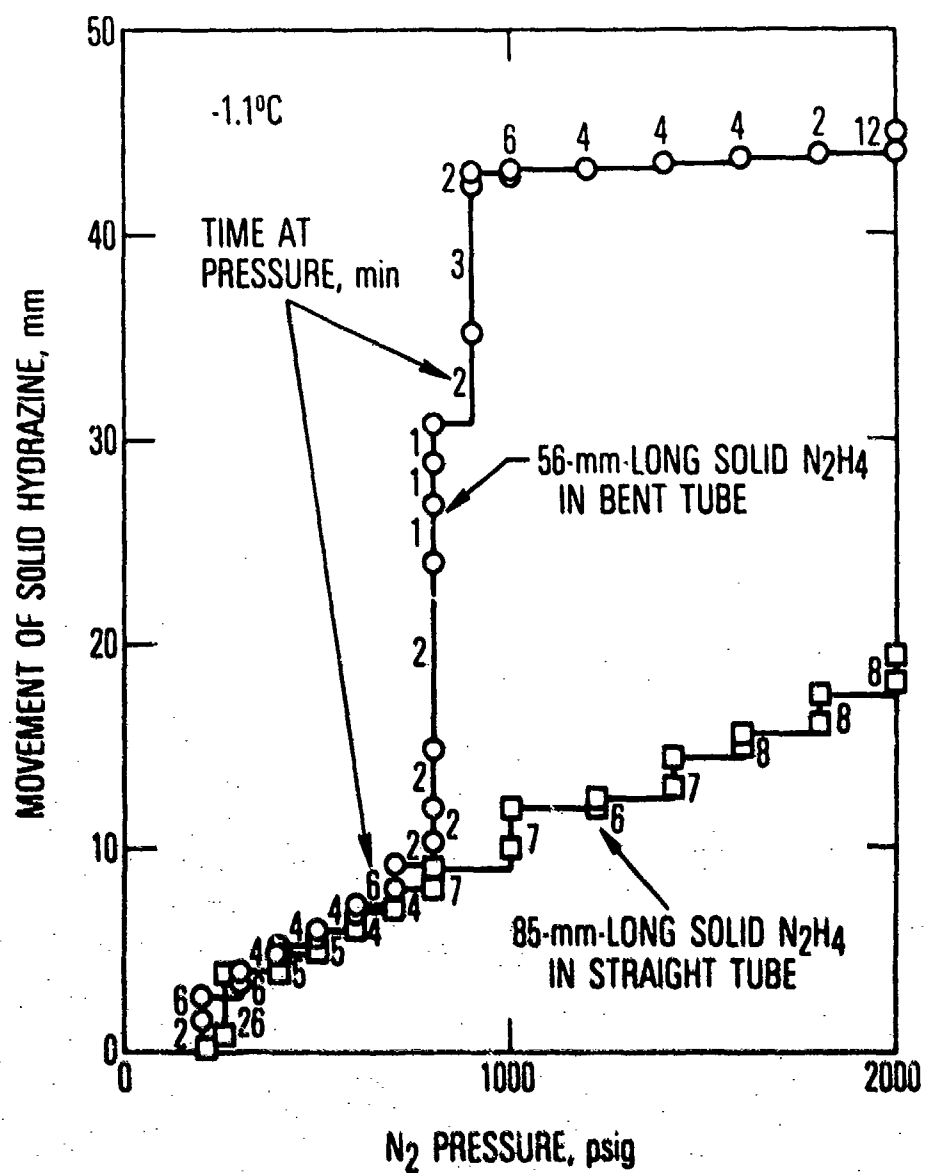


Fig. 7. Movement of pure solid hydrazine in 0.25-in. straight and 120° -bent stainless-steel tubes at $-1.1^\circ C$ ($+30^\circ F$)

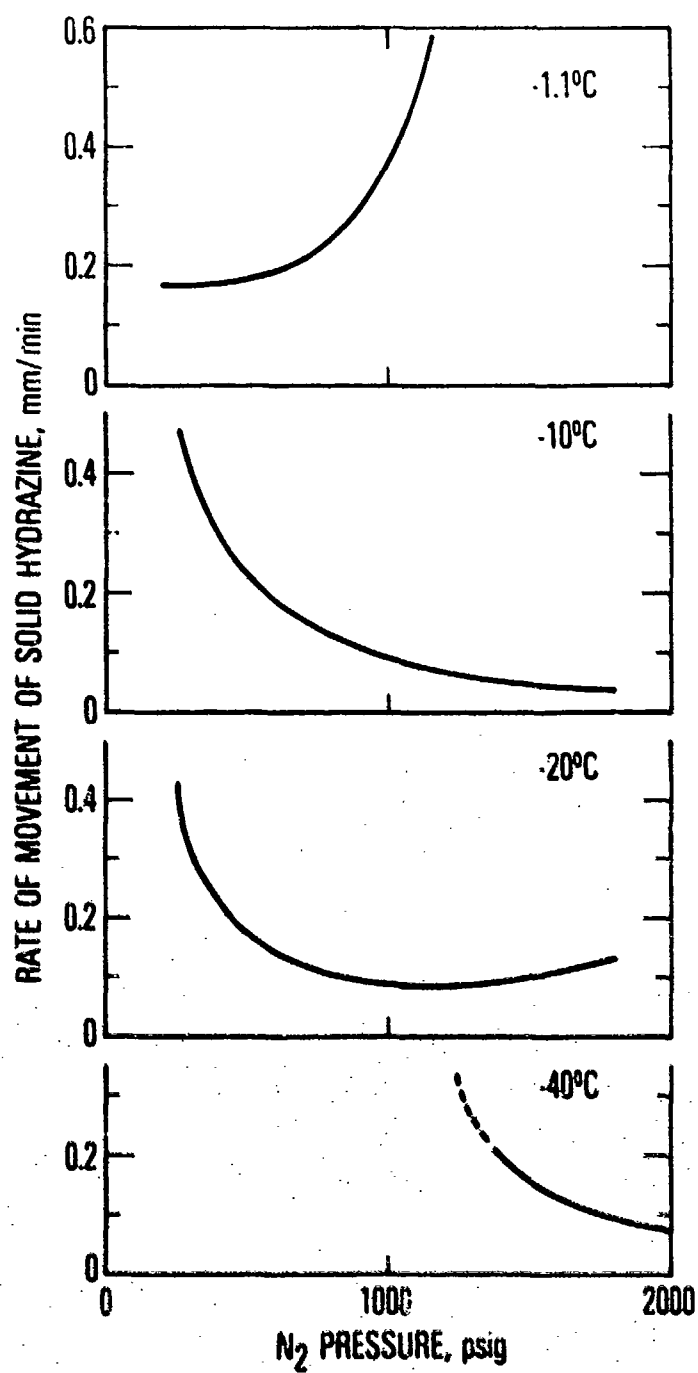


Fig. 8. Rate of movement of pure solid hydrazine in 0.25-in. straight stainless-steel tubes at various temperatures and pressures

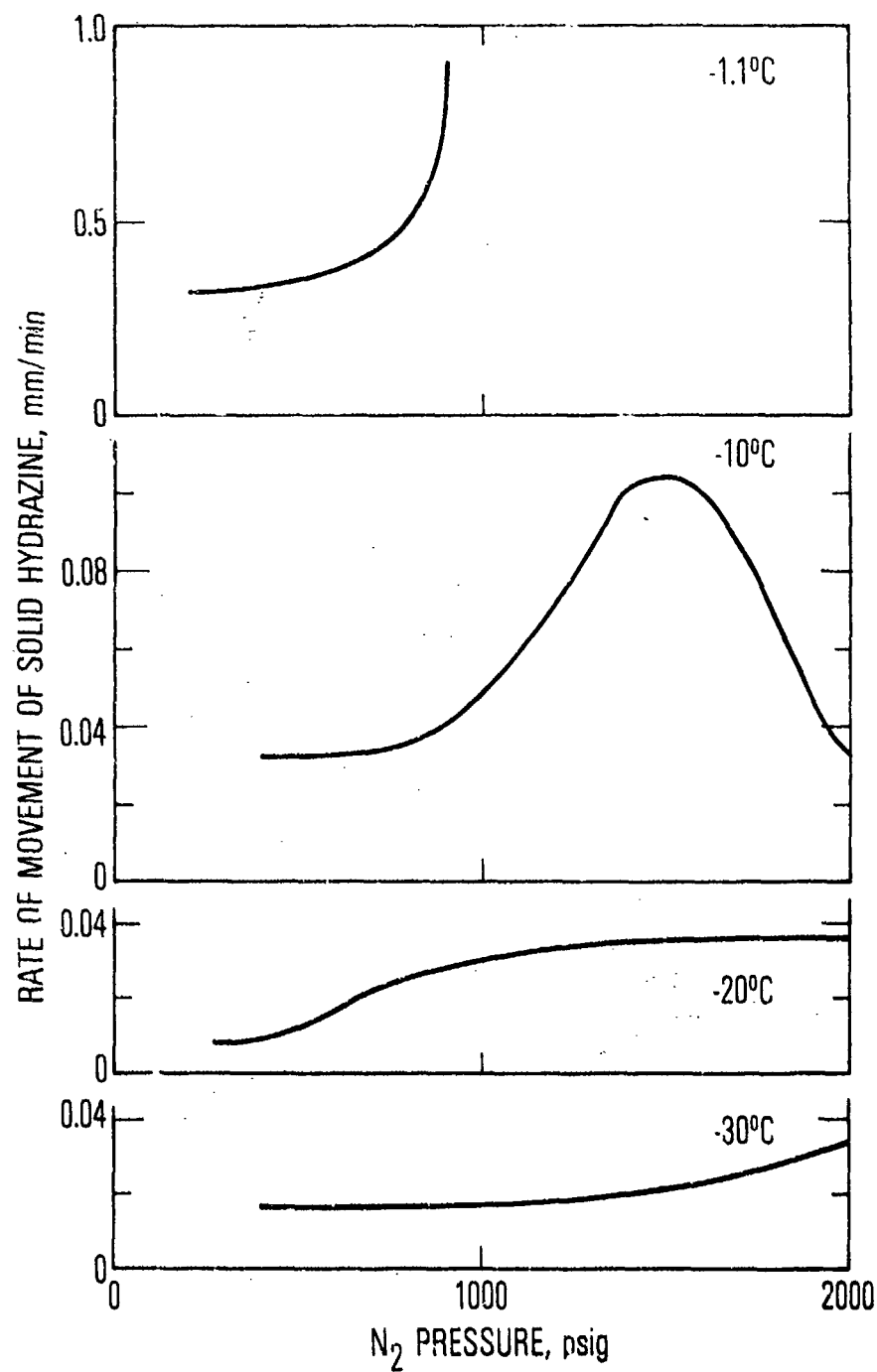


Fig. 9. Rate of movement of pure solid hydrazine in 0.25-in. 120°-bent stainless-steel tubes at various temperatures and pressures

VII. MECHANICAL MOVEMENT OF SOLID HYDRAZINE CONTAINING WATER

The rates of movement of solid hydrazine containing 1 and 2% water were investigated in straight and bent tubes. At -20°C , Fig. 10 shows that as the water content increases, the rate of movement increases in bent tubes as shown by the zigzag lines on the right side. The line on the left is for pure hydrazine in a straight tube for comparison. The rates of movement in bent tubes are plotted versus pressure in Fig. 11. At 1000 psig, the rates are 0.03, 1.64 and 1.73 mm/min for 0, 1, and 2% water, respectively, whereas at 2000 psig, the corresponding numbers are 0.035, 0.066 and 1.6 mm/min. Water forms $\text{N}_2\text{H}_4 \cdot \text{H}_2\text{O}$, which remains liquid in the grain boundaries and causes softening of hydrazine, as mentioned earlier.

At -10°C , the rate of movement is faster than at -20°C (Fig. 12). Again, higher water contents cause faster movement of solid hydrazine in the bent tubes. Figure 13 shows the rate of movement versus pressure at -10°C . The rate increases with increasing pressure for 2% water and reaches 0.297 mm/min at 2000 psig, whereas for pure hydrazine it increases to a maximum at 0.104 mm/min at 1500 psig and decreases to 0.033 mm/min at 2000 psig. (Fig. 13). At -5°C , the rate of movement is much more rapid than at -10°C (Figs. 14 and 15).

The foregoing results permit the calculation of stress and strain behavior of solid hydrazine as affected by temperature, water content, and configuration of stainless-steel tubes.

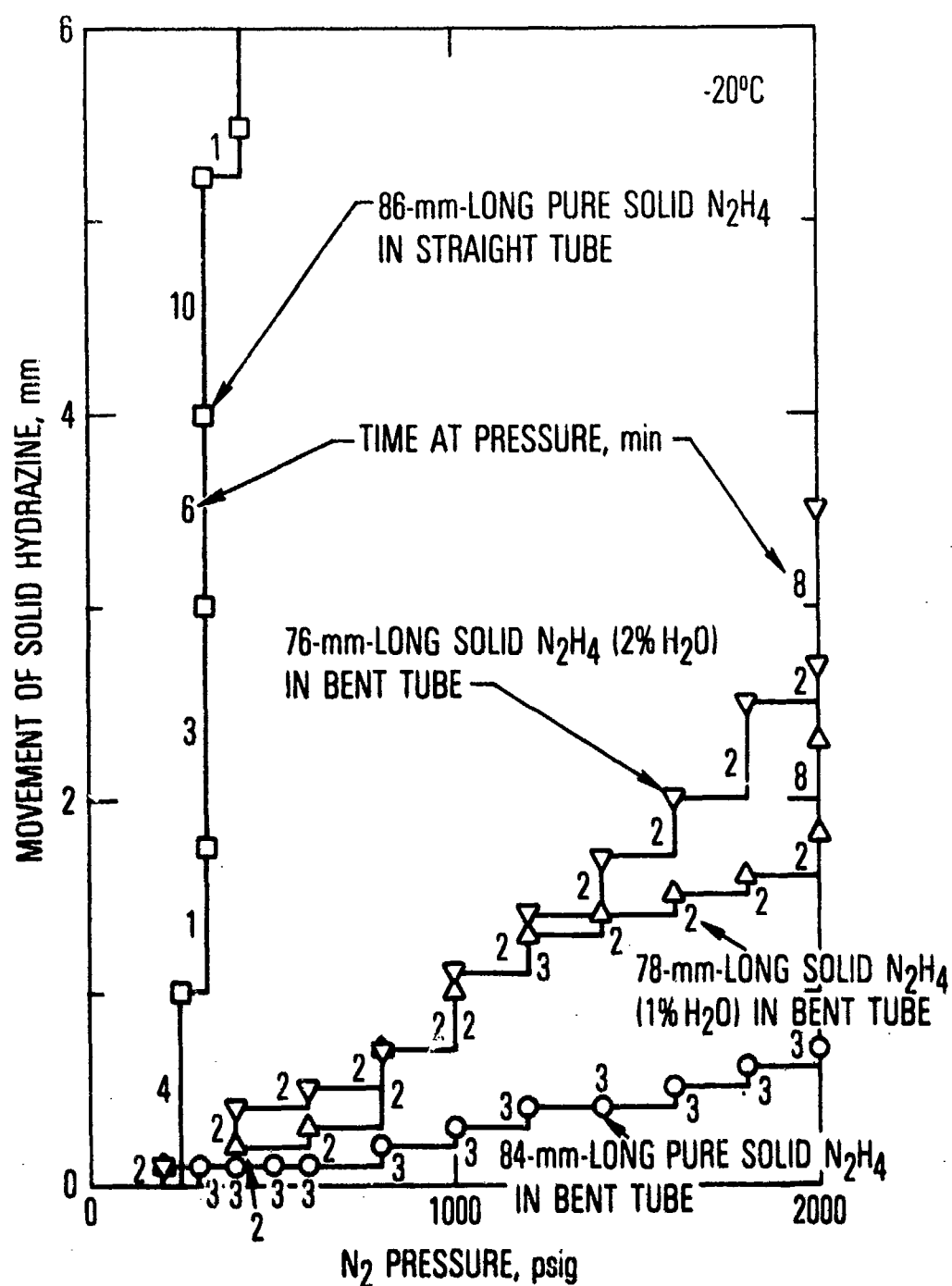


Fig. 10. Movement of solid hydrazine containing various amounts of water in 0.25-in. straight and 120°-bent stainless-steel tubes at $-20^{\circ}C$ ($-4^{\circ}F$)

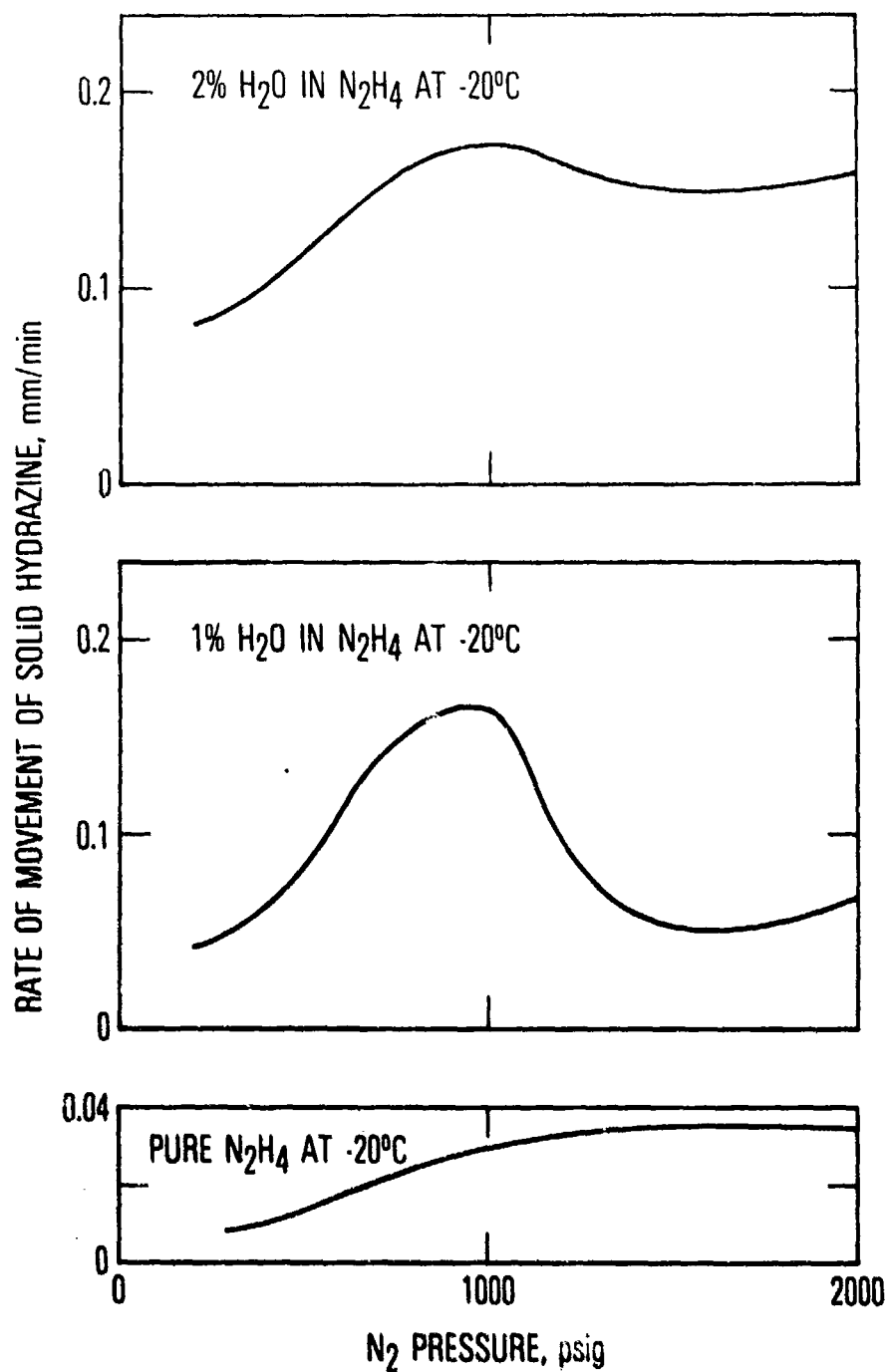


Fig. 11. Rate of movement of solid hydrazine containing various amounts of water in 0.25-in. 120°-bent stainless-steel tubes at -20°C (-4°F)

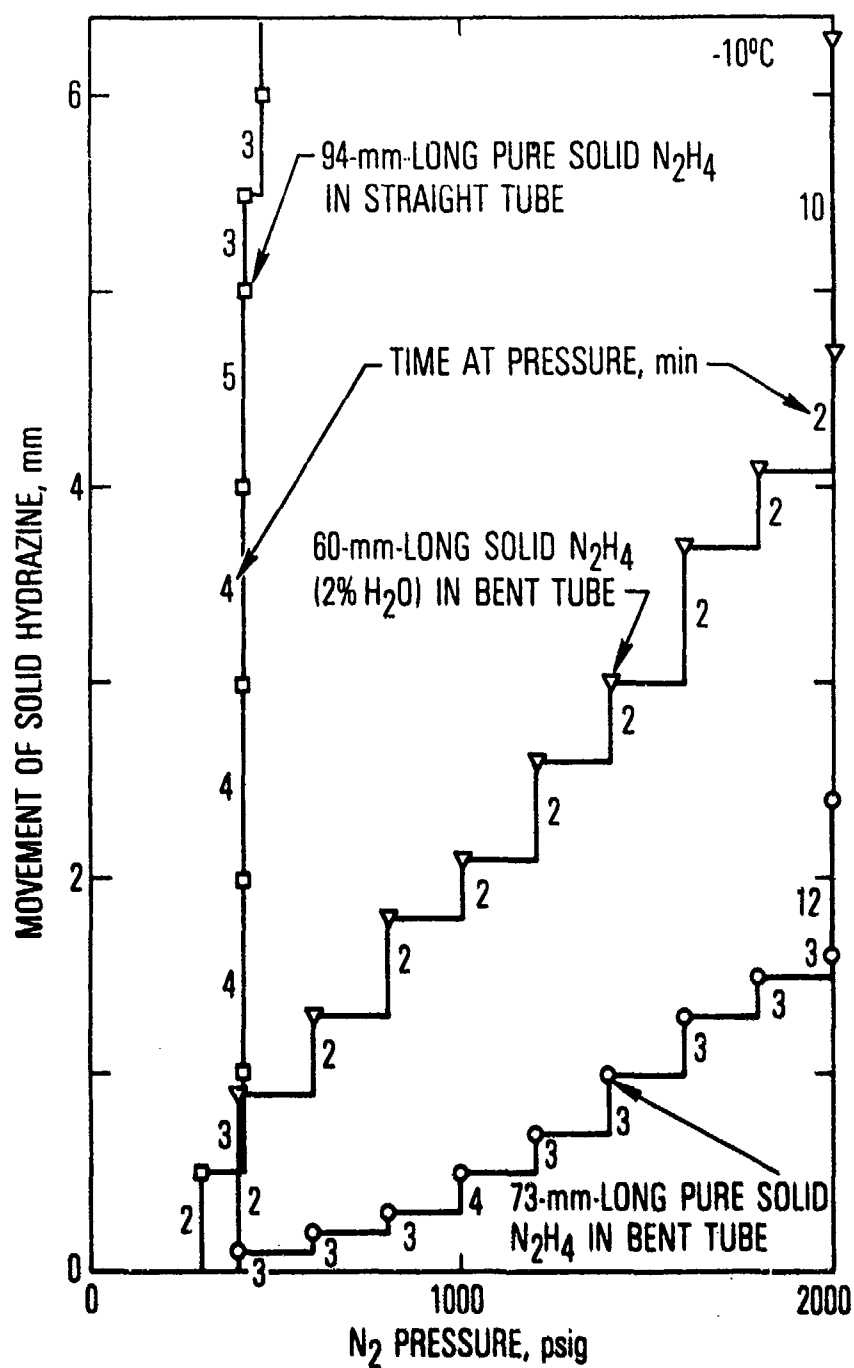


Fig. 12. Movement of solid hydrazine containing various amounts of water in 0.25-in. straight and 120°-bent stainless-steel tubes at $-10^\circ C$ ($+14^\circ F$)

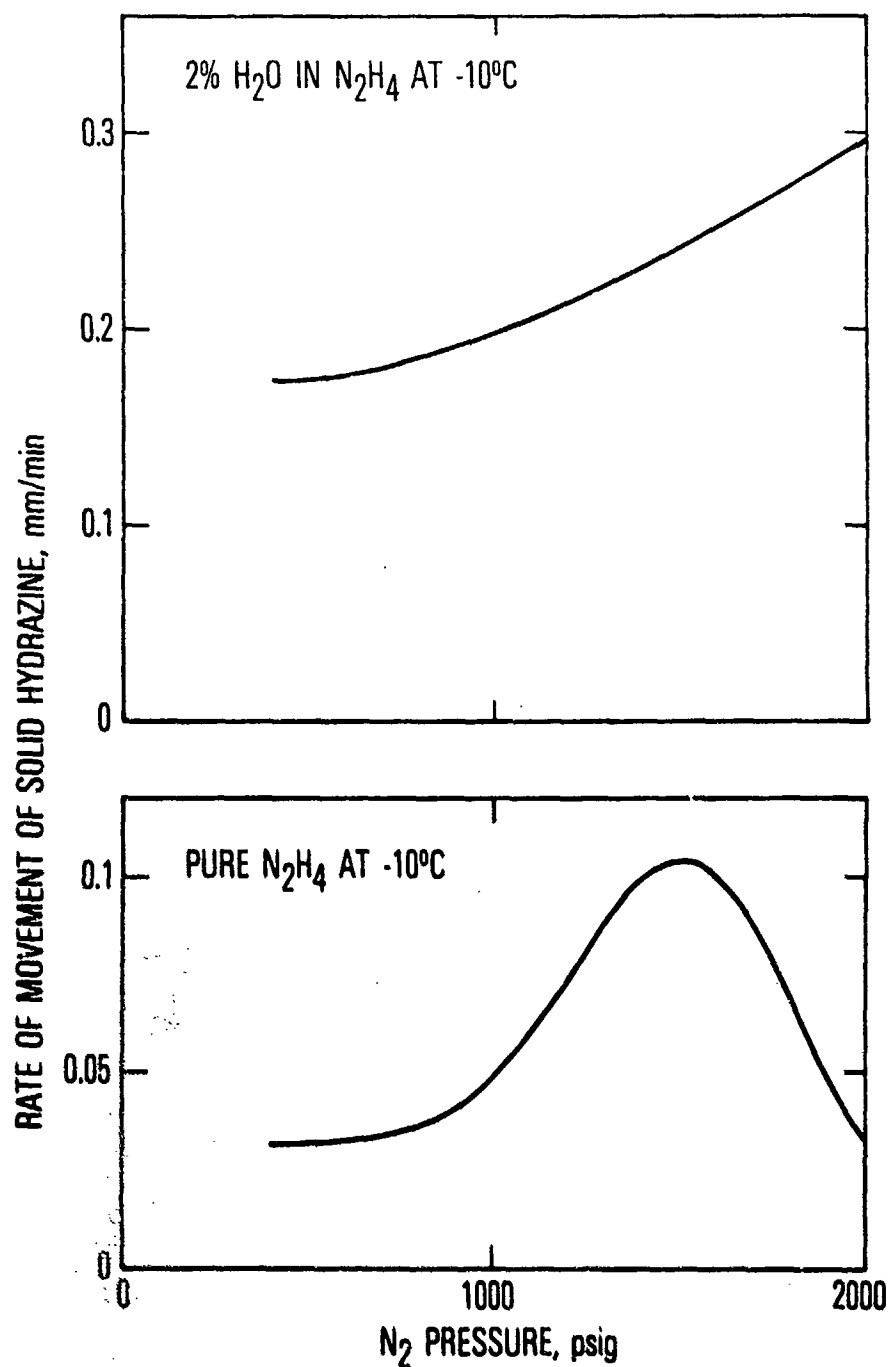


Fig. 13. Rate of movement of solid hydrazine containing various amounts of water in 0.25-in. 120°-bent stainless-steel tubes at -10°C (+14°F)

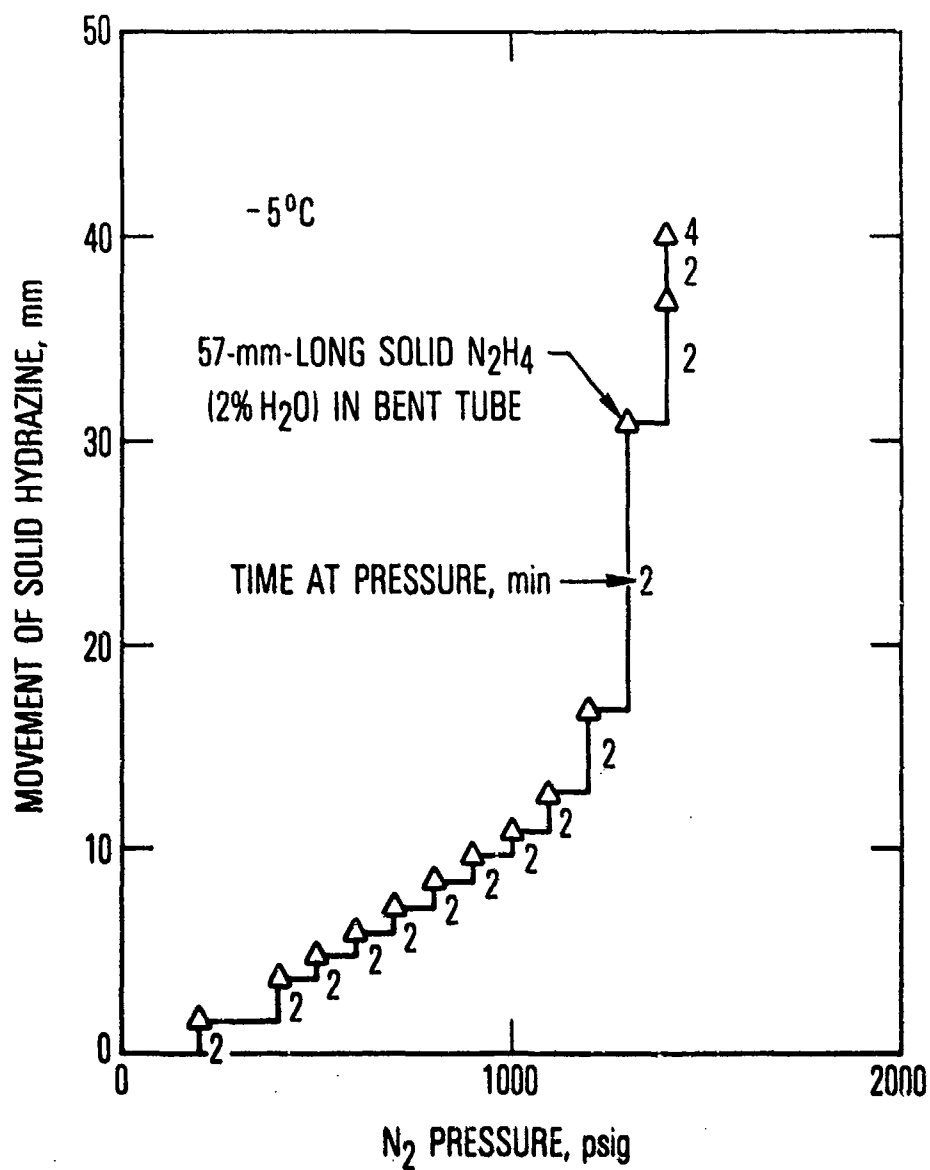


Fig. 14. Movement of solid hydrazine containing 2% water in 0.25-in. 120°-bent stainless-steel tube at -5°C (+23°F)

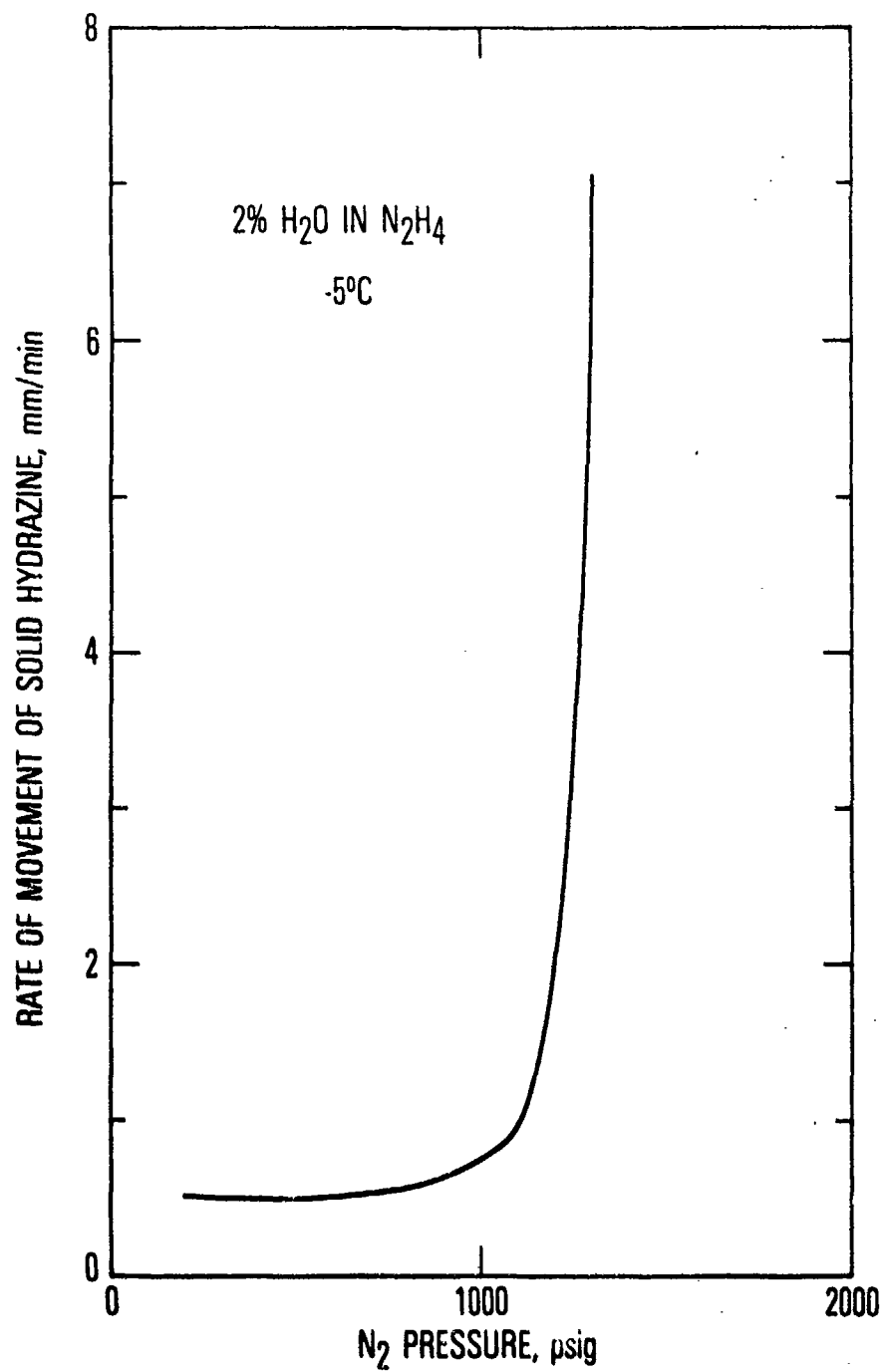


Fig. 15. Rate of movement of solid hydrazine containing 2% water in 0.25-in. 120°-bent stainless-steel tube at -5°C (+23°F)

VIII. RECOMMENDATIONS

The following recommendations are presented to minimize the danger of rupture in tubes and valves containing hydrazine that might be subjected to repeated freezing and melting:

1. Up to 1% water in hydrazine permits easier movement of solid hydrazine than 0% water. Addition of a permissible amount of water is therefore recommended.
2. Heavy wall tubes (about 0.035-in. thick) are preferable to the standard wall tubes (with 0.016-in. thickness) for lower strain on the tubes.
3. Some insulation on tubes and valves could permit slower rates of freezing and melting and minimize generation of excessive stresses.

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