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ABSTRACT

This paper describes a study of relative humidity effects on the adhesion of small particles to surfaces. Ambient dust ranging in size from less than one micron up to 140 microns was used as test particles. Relative humidities of 33% to 100% were used to condition test surfaces after loading with the test particles. A 20 psi nitrogen blowoff was used as the removal mechanism to test for particle adhesion. Particles were counted before and after blowoff to determine retention characteristics. Particle adhesion increased drastically as relative humidity increased above 50%. The greatest adhesion changes occurred within the first hour of conditioning time. Data is presented for total particle adhesion, for particles 10 micron and larger, and 50 microns and larger.

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INTRODUCTION

This is a study of small particle behavior related to microbial burden of spacecraft surfaces prior to final sterilization. Particles that have highly resistant microorganisms attached to them are of prime interest to this study. Since such particles are very few in number compared to total particle contamination, they are very difficult to identify for study. For this reason, total particle contamination is being studied relative to particle accumulation and retention on surfaces. Only a small number of particles bearing very hardy microorganisms would be expected to accumulate on a spacecraft during assembly in a very clean area; however, one to ten percent of the type of microorganism reported by Favero in USPHS Report No. 32 dated January 1971, could survive a 24-hour heat cycle at 125°C. In order to identify factors whose control would lead to a lower particulate burden (and hence lower bioburden) we believe that a rigorous analysis of spacecraft surface particle loading is necessary. This is particularly critical for spacecraft surfaces that cannot be rigorously cleaned before final sterilization. Many such surfaces are covered with reflective coatings and may be lightly wiped or vacuum brushed which removes only lightly adhering contamination.

EFFECTS OF HUMIDITY STUDY

A series of experiments was conducted to determine the effects of relative humidity on particle contamination adhesion to surfaces. Almost all naturally occurring particles are affected in some manner by water vapor in the surrounding air, particularly as the water content of air increases. Of special interest are those particles that pick up moisture from the air which partially or totally dissolve and then form a very strong bond with the surface on which the particles are located. At elevated humidities this occurs quickly - in a very few minutes. These particles cannot be effectively removed by dry wipes, vacuum cleaning or other cleaning methods that are permitted on many spacecraft surfaces. Thus, the final microbial burden of a spacecraft can be affected by the relative humidity of the air in which it was assembled.

These experiments were set up to study relative humidity effects from thirty-three percent to one hundred percent. Glass dessicators (Figure 1) containing the following saturated salt solutions were used for conditioning chambers.

H_2O	Water	100%
$NH_4H_2PO_4$	Ammonium Phosphate	93%
KBr	Potassium Bromide	84%
NaCl	Sodium Chloride	76%
$NaNO_2$	Sodium Nitrite	66%

Figure 1. Humidity Conditioning Chambers

$Na_2CR_2O_7 \cdot 2H_2O$	Sodium Dichromate	52%
$MgCl_2 \cdot 6H_2O$	Magnesium Chloride	33%

Test particles were used that simulated as nearly as possible the type particles expected in a clean room environment. Test particles were obtained by sieving building vacuum cleaner dust to exclude particles larger than 140 microns. After sieving, the test particles were stored in dry air over a dessicant bed until use.

Test surfaces were 1" x 1" highly polished metal foils cemented to 1 x 3 inch glass microscope slides (Figure 2). Test surfaces were etched to permit photographing the exact same area before and after "blowoff" (a simulated environmental removal of particles to test retention ability).

A 3.3 cu. ft. particle loading chamber (Figure 3) was used to load the test surfaces prior to conditioning at the various relative humidity levels. An agitator fan was located near the bottom of the chamber and a glass tube was used to feed test particles into the fan inlet during loading. A horizontal rack was positioned in the upper half of the loading chamber to hold test slides during loading.

A blowoff fixture (Figure 4) was used to retain test slides during "blowoff." The fixture consisted of a 1/8" diameter jet located 1/2" above the test strip. Dry nitrogen was used as the blowoff gas which was controlled by a solenoid valve and timer. Nitrogen pressure was regulated to 20 psi during blowoff.

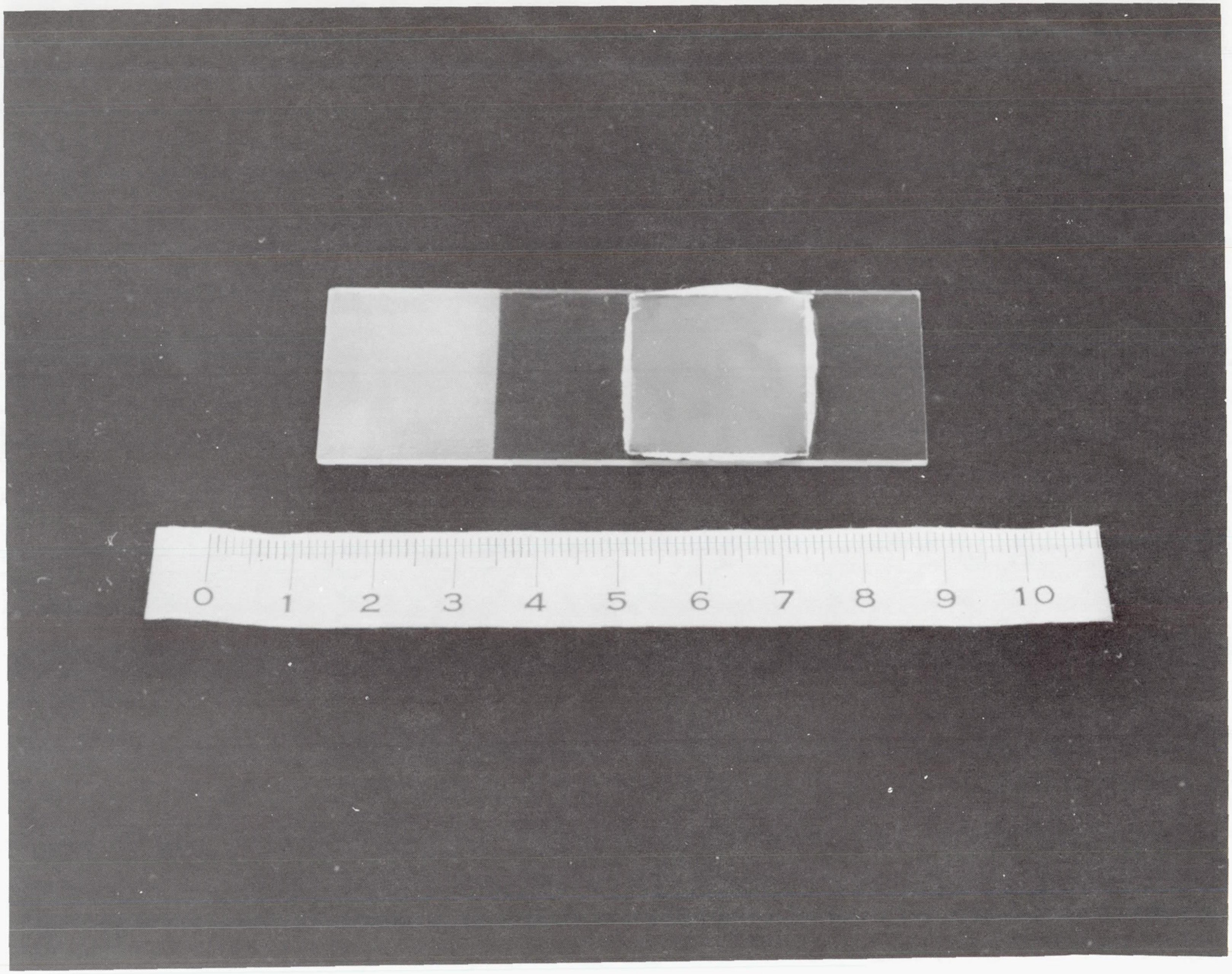

Figure 2. Test Surfaces - 25 x 75 mm

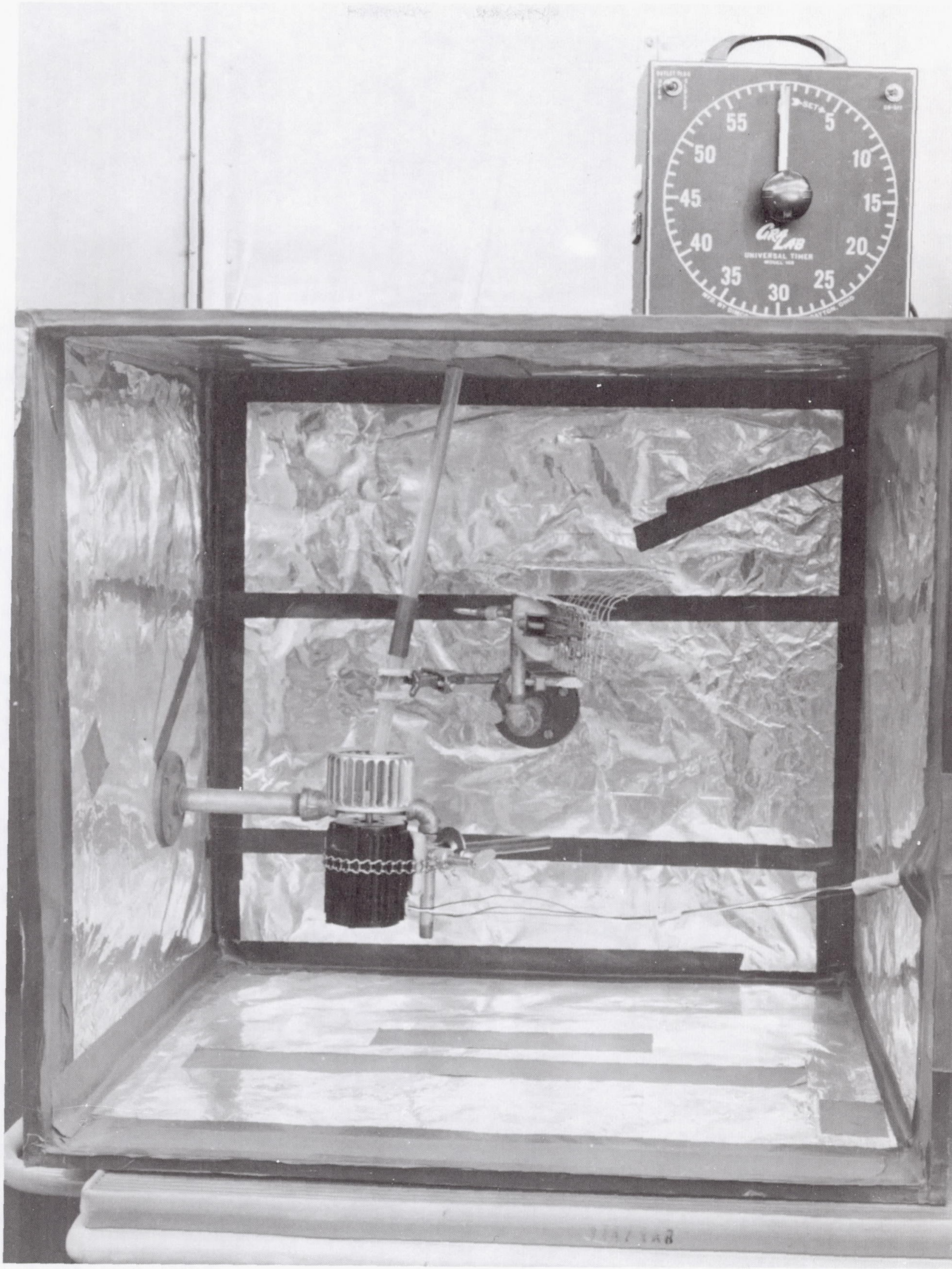

Figure 3. Loading Chamber

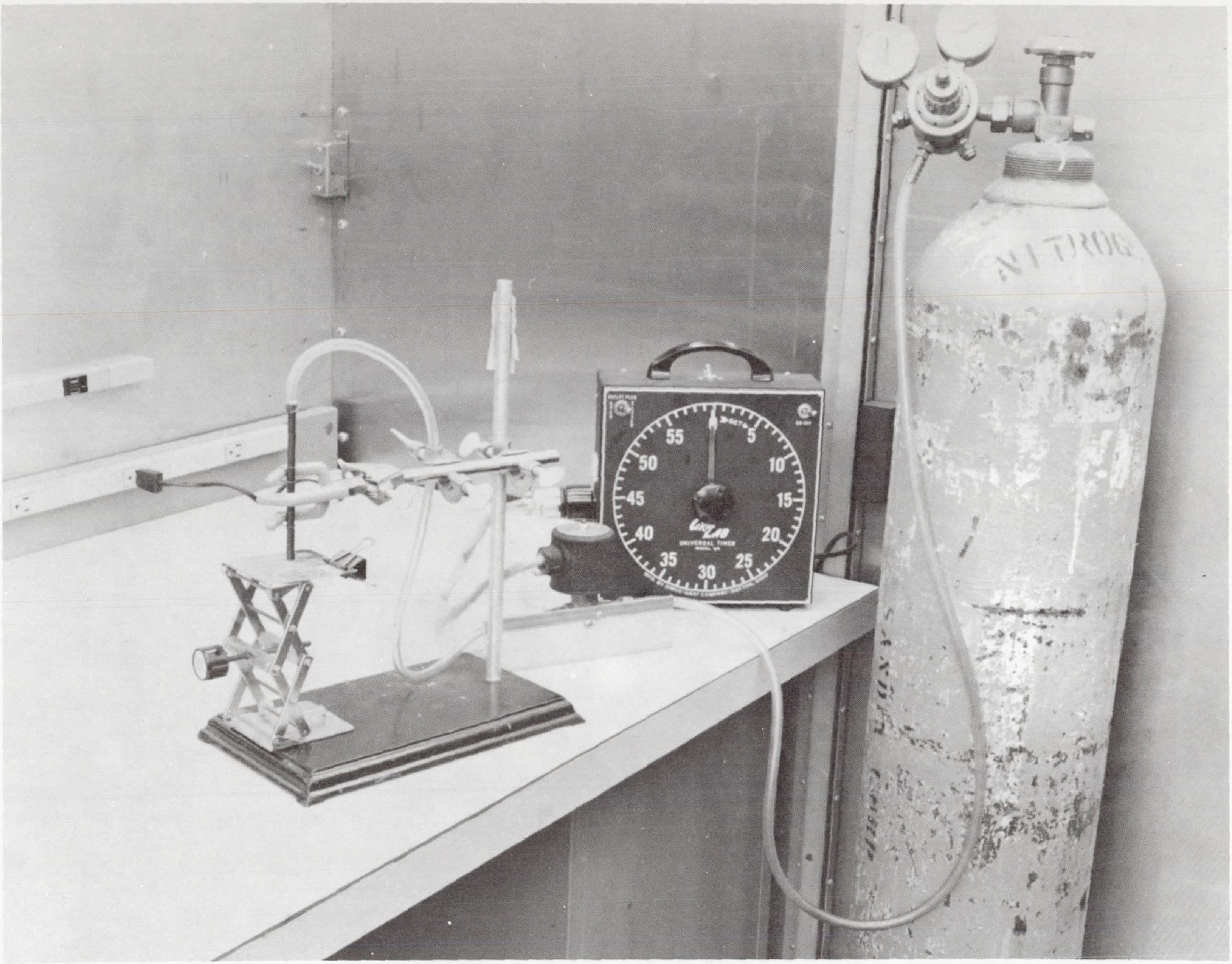

Figure 4. Test Blowoff Fixture

The microscope-camera system is a Leitz Ortholux equipped with Leitz Ultra-Pak vertical illumination equipment (Figure 5). This combination with the highly polished foils provides an excellent high contrast dark field illumination system. The system will resolve particles less than one micron size. A 4" x 5" Polaroid camera back is used for photographing test slides. A magnification of 90x is used for photographs to be counted and higher magnifications for individual particle analysis.

Figure 5. Microscope-Camera System

EXPERIMENTAL PROCEDURE

The following sequence of steps was followed during the experiment:

1. Twelve clean slides were placed on the loading chamber rack.
2. The timer was set for 60 seconds to start blower.
3. Two ml (approximately 1.2 gm) test particles were released into the loading tube during first 30 seconds of the loading cycle.
4. After the load cycle, the 12 test slides were carefully removed and placed in the humidity control chamber. Every effort was made to avoid air currents, drafts, vibration and rapid movement of the test slides during handling. One slide was removed from the humidity control chamber after each of the following conditioning periods: 84%, 93%, and 100% - 5, 10, 15, 30 minutes; 1, 2, 4, 8, 24, 48, 72 hours. 33%, 52%, 66%, 76% - 30 minutes; 1, 2, 4, 8, 24, 72, 200, 720 hours.
5. After removal from the humidity control chamber, each slide was immediately photographed. Then it was exposed to a "blowoff" treatment for 10 seconds at 20 psi. (Following "blowoff" it was immediately rephotographed. Two separate defined areas were photographed before and after "blowoff").
6. Particles, as recorded on the photographs, were sized and counted in ranges of (a) less than 10 microns, (b) 10 microns and larger, and (c) 50 microns and larger. The lower limit of particle size count was approximately 1 micron. The area photographed from each

slide was approximately 1.6 mm^2. Approximately 120 particles were counted per 1.6 mm^2 before blowoff (initial load).

7. Four test slides were loaded, photographed, then subjected to "blowoff" procedure, rephotographed, and counted for reference or control.

RESULTS

Particle count data was converted to "Percent Particles Remaining After Blowoff," designated as "Retention %" and defined as

$$\text{Retention \%} = \frac{\text{Count after blowoff}}{\text{Count before blowoff}} \times 100.$$

Data are presented in graph form in Figures 6 through 12 for individual relative humidity levels. Results are plotted as "Retention %" versus the time (in hours) of conditioning at the various humidity levels. At relative humidity levels of 76%, 84%, 93% and 100%, "Retention %" is plotted for the particle size categories "total particles," "10 microns and larger," and "50 microns and larger," marked as curves A, B and C. The "50 microns and larger" is not shown for relative humidity levels below 76% since there was very little retention of large particles at these humidities. Figure 13 shows a comparison plot of total particles for all relative humidity levels.

This series of experiments indicates that the major effect of relative humidity on particle retention occurs within one hour at any of the relative humidity levels investigated. At higher humidity levels, particles become firmly attached to the surface in a few minutes. Dis-association of the particles (Figures 14, 15, 16) occurred at all humidity levels; (occurring much more rapidly at the higher humidity levels). The dissociation or "breakup" of particles left large numbers of small particles adhering to the test surface that were much harder to remove than the original, or parent, particles.

EFFECTS OF RELATIVE HUMIDITY ON SURFACE PARTICLE RETENTION

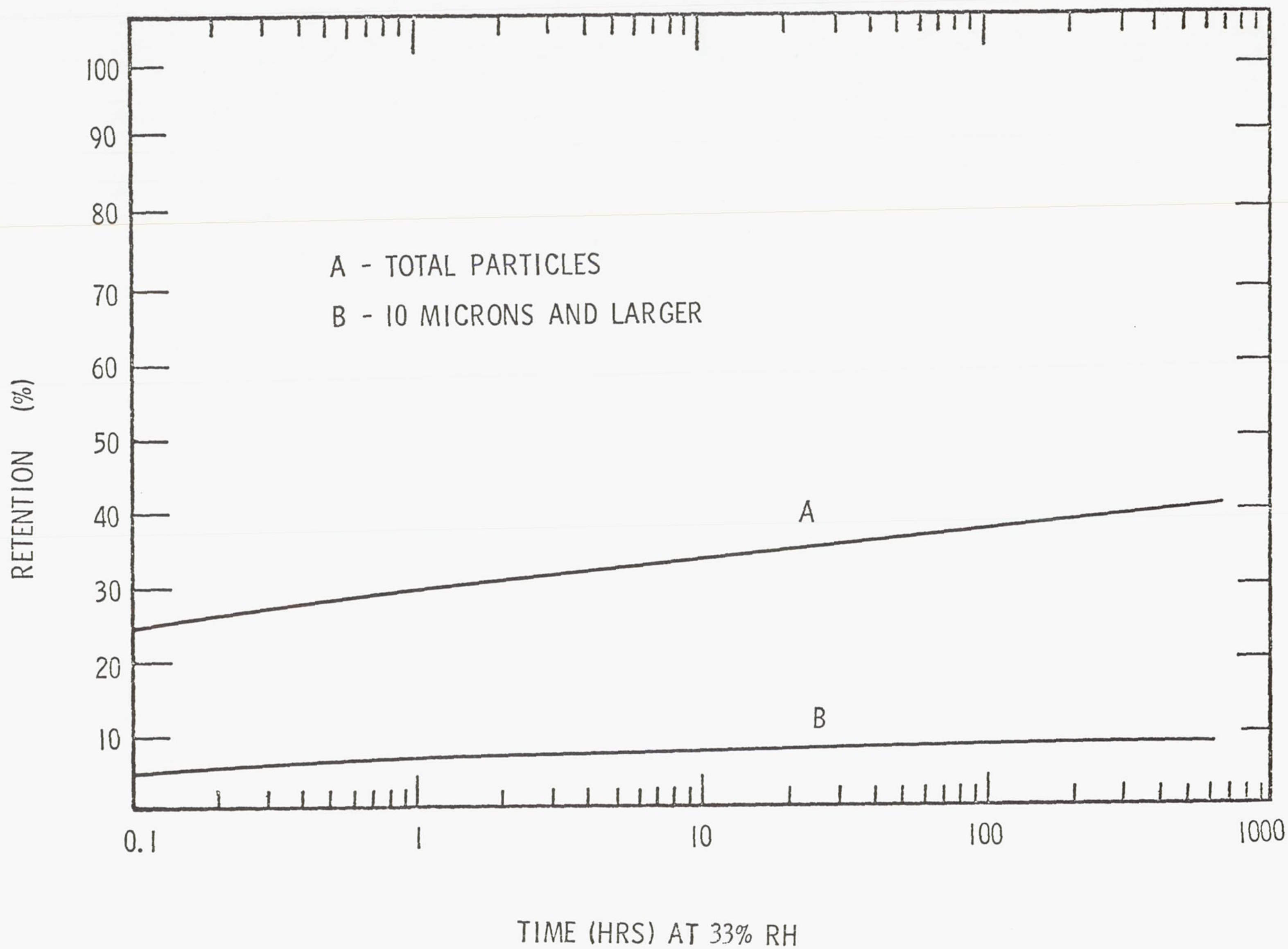

Figure 6

EFFECTS OF RELATIVE HUMIDITY ON SURFACE PARTICLE RETENTION

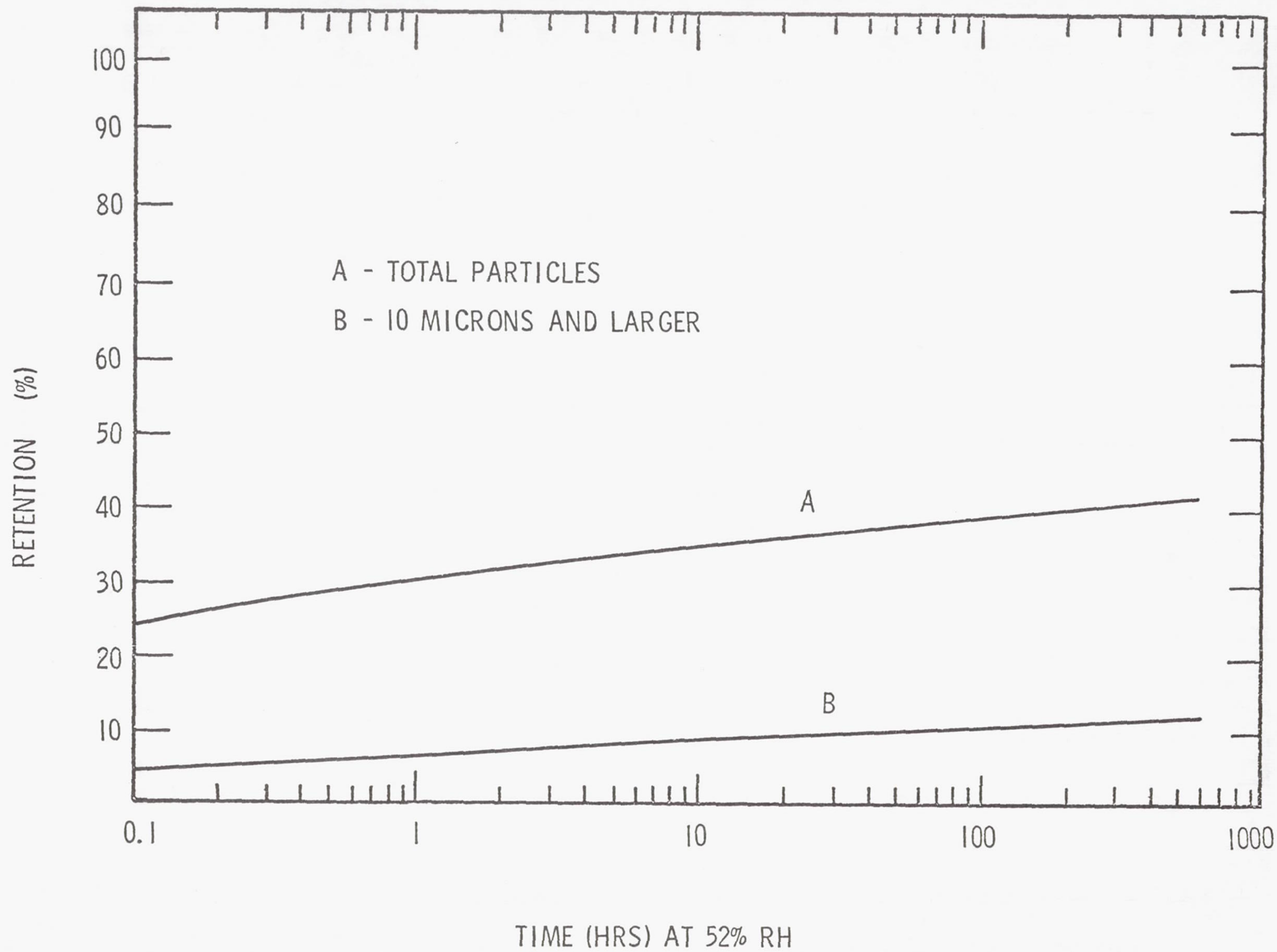

Figure 7

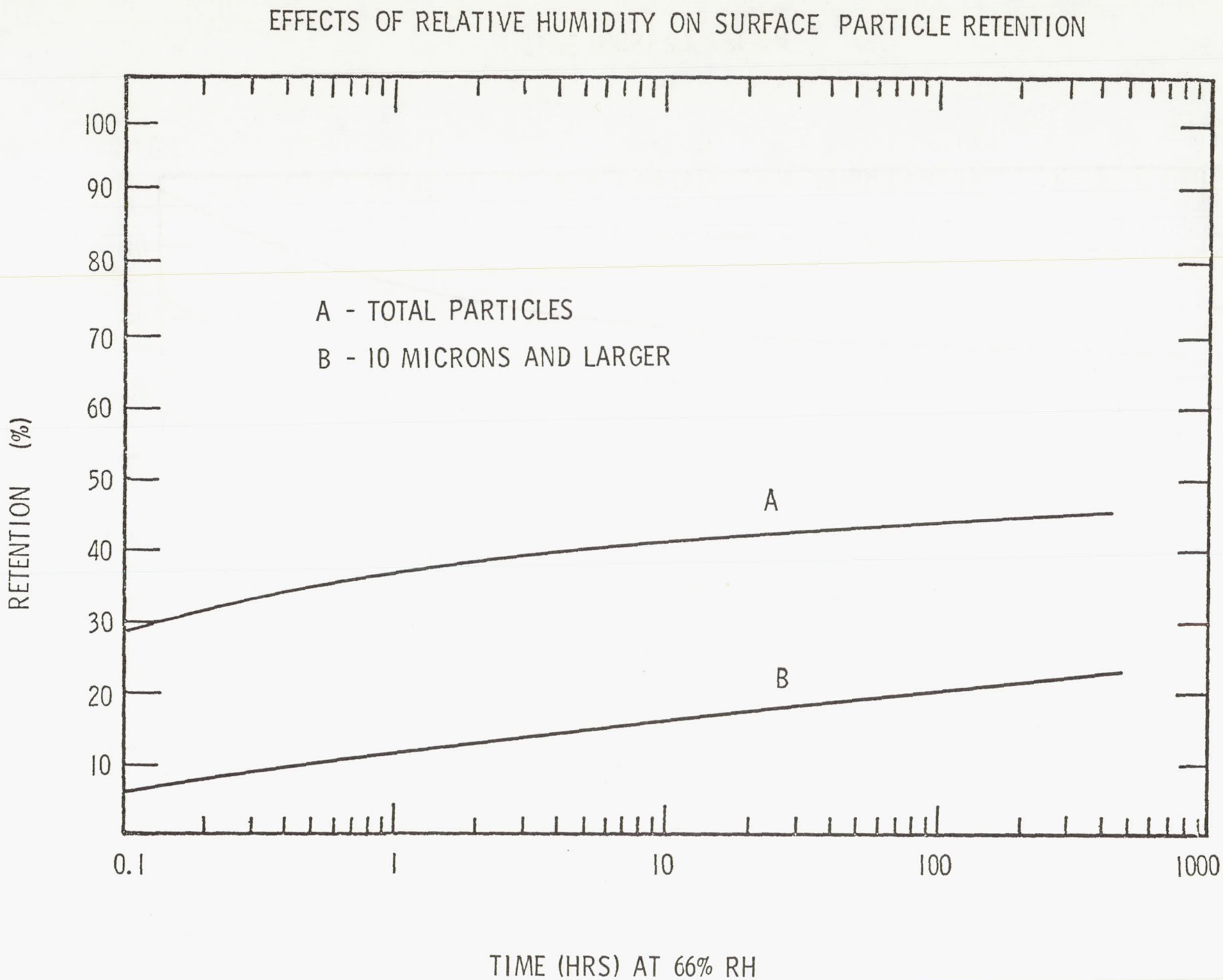
EFFECTS OF RELATIVE HUMIDITY ON SURFACE PARTICLE RETENTION
A - TOTAL PARTICLES
B - 10 MICRONS AND LARGER
RETENTION (%)
100
90
80
70
60
50
40
30
20
10
A
B
0.1
1
10
100
1000
TIME (HRS) AT 66% RH

Figure 8

EFFECTS OF RELATIVE HUMIDITY ON SURFACE PARTICLE RETENTION

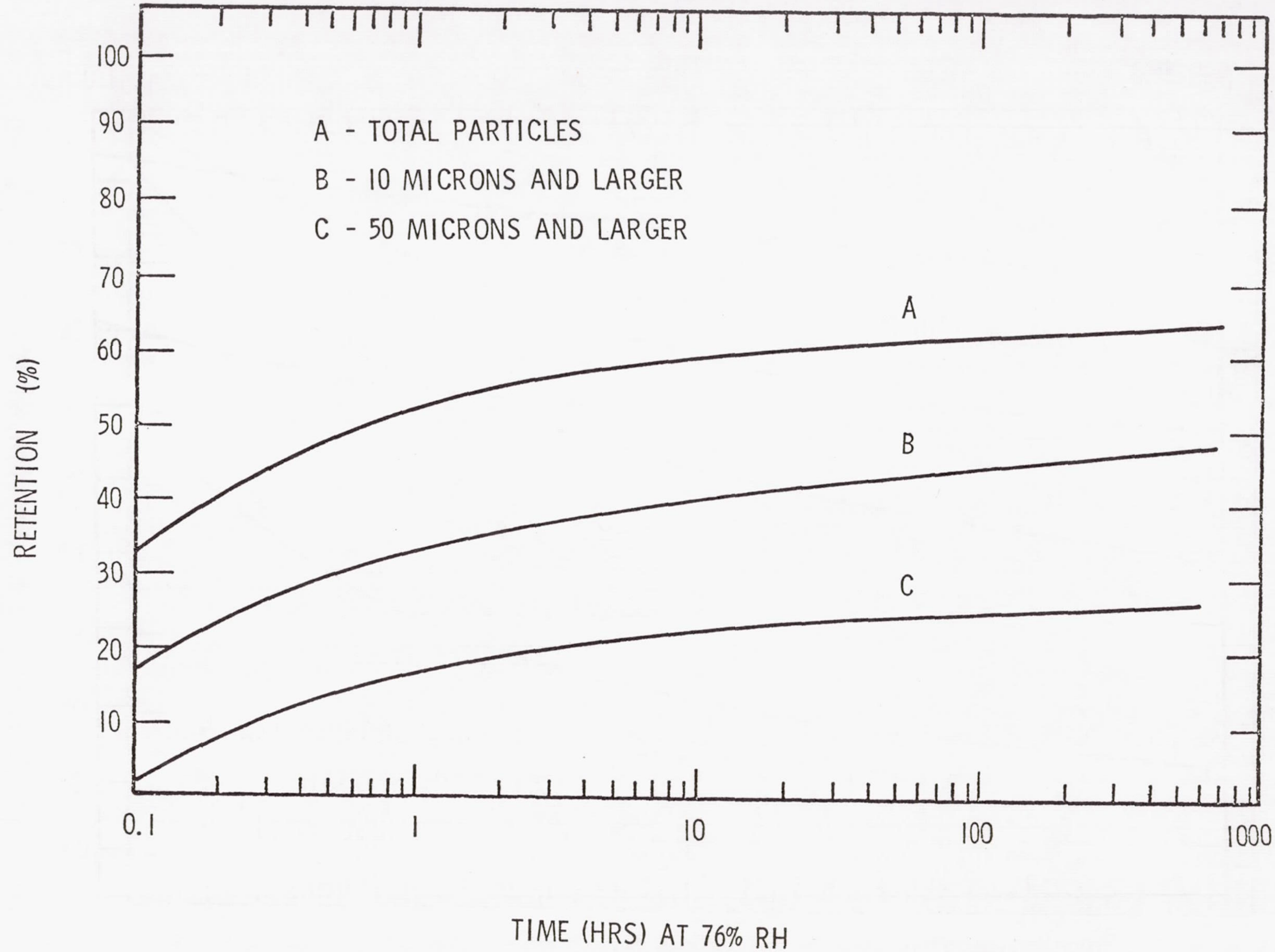

Figure 9

EFFECTS OF RELATIVE HUMIDITY ON SURFACE PARTICLE RETENTION

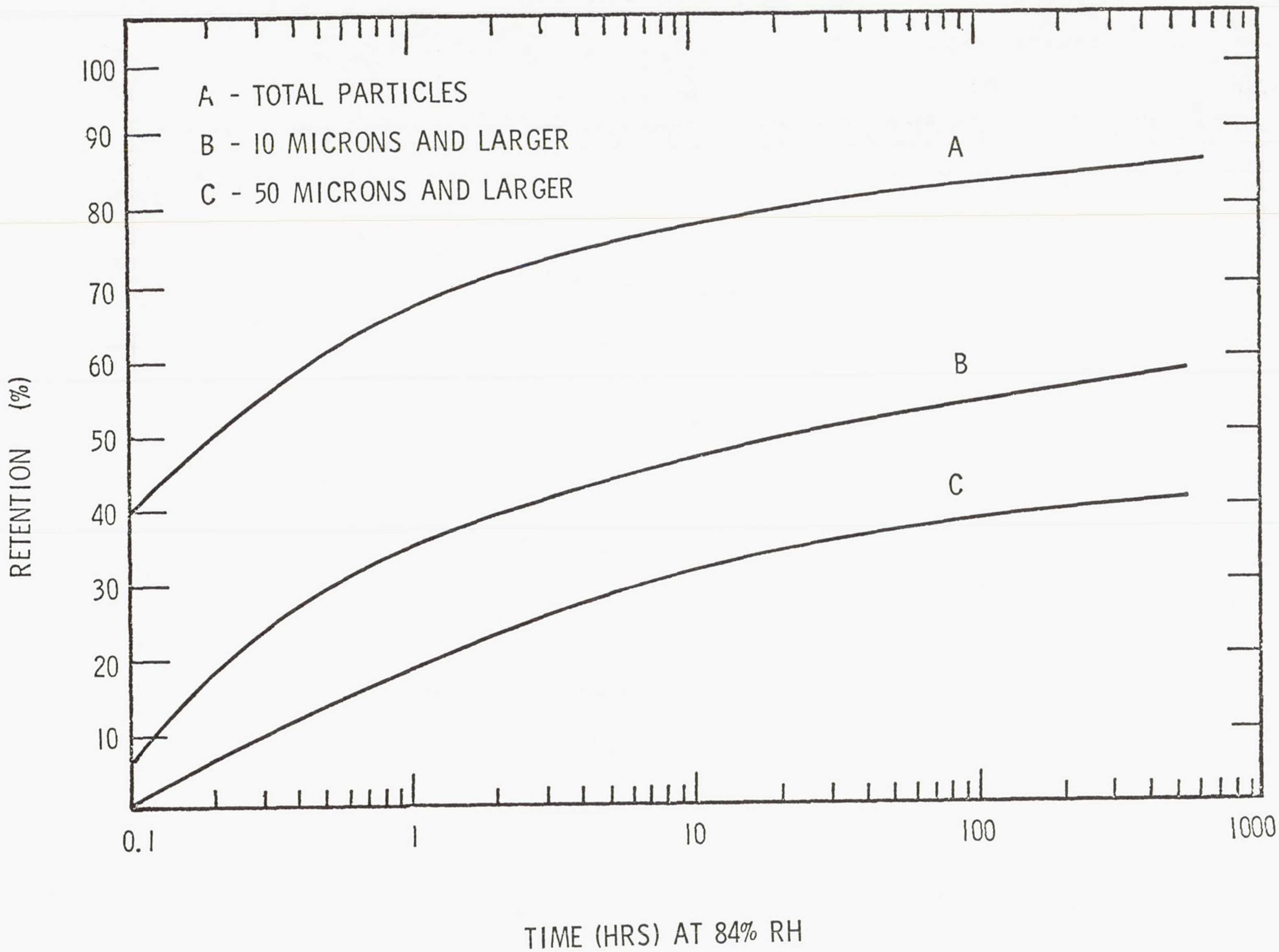

Figure 10

EFFECTS OF RELATIVE HUMIDITY ON SURFACE PARTICLE RETENTION

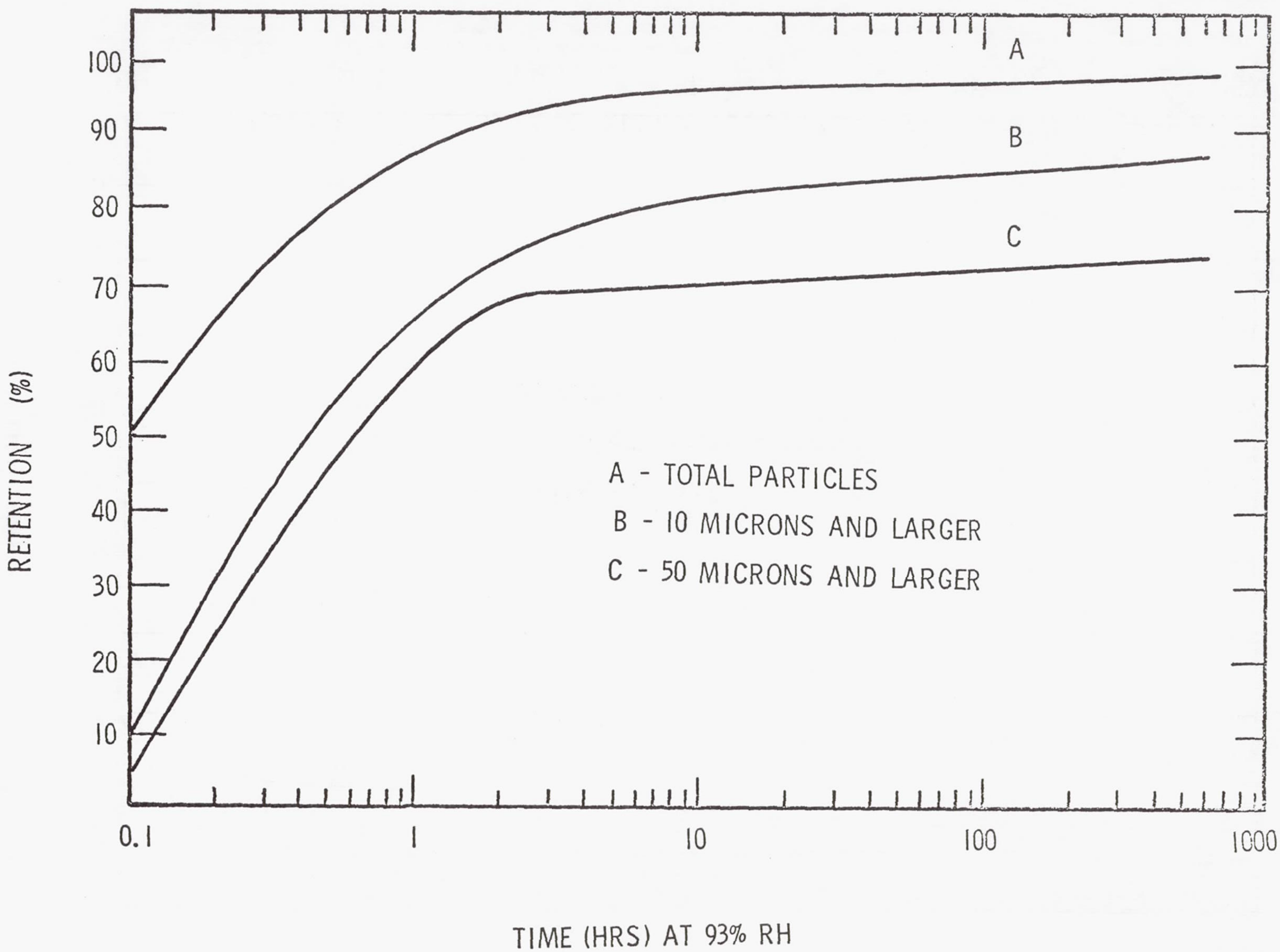

Figure 11

EFFECTS OF RELATIVE HUMIDITY ON SURFACE PARTICLE RETENTION

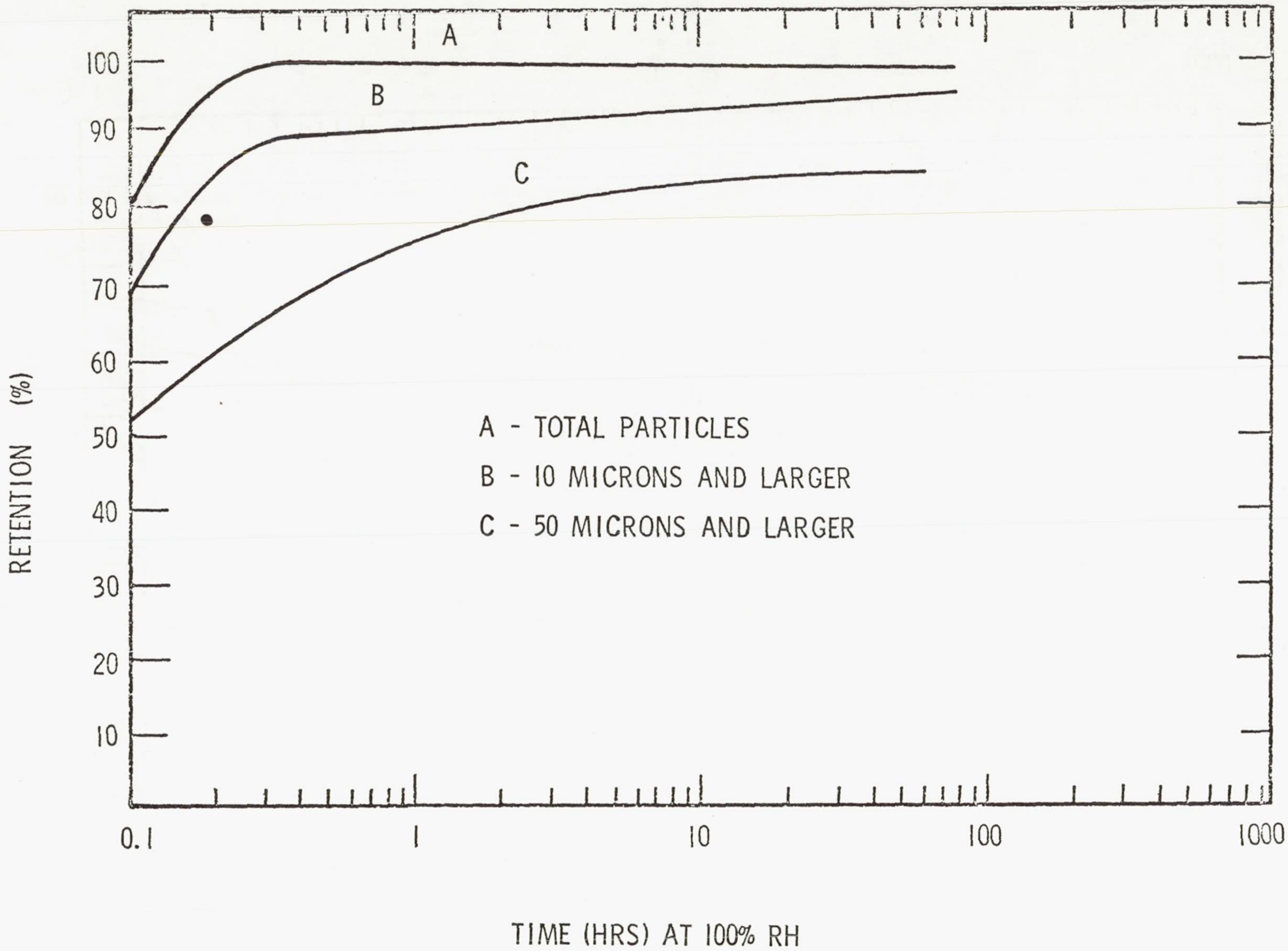

Figure 12

EFFECTS OF RELATIVE HUMIDITY ON SURFACE PARTICLE RETENTION

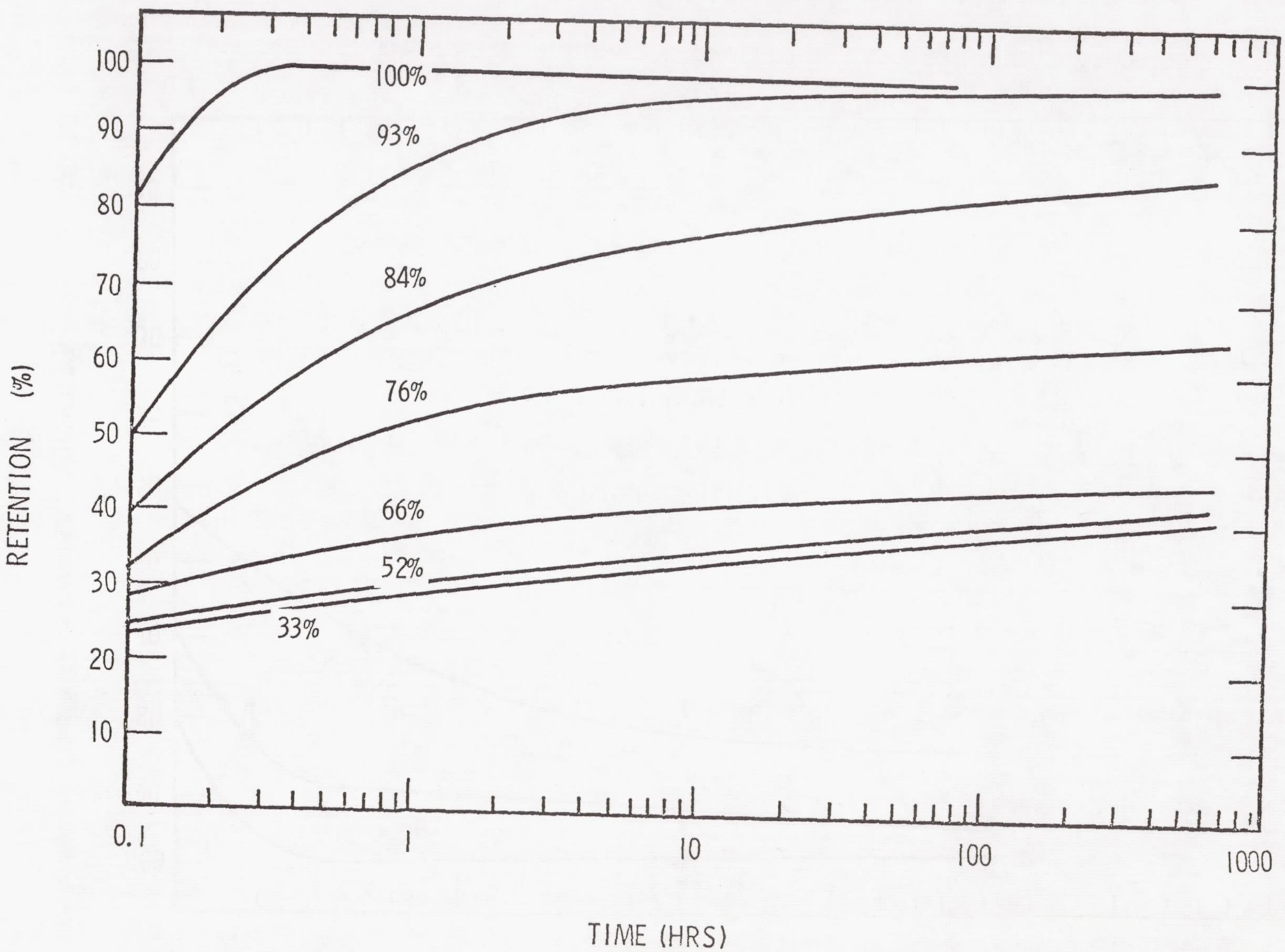

Figure 13

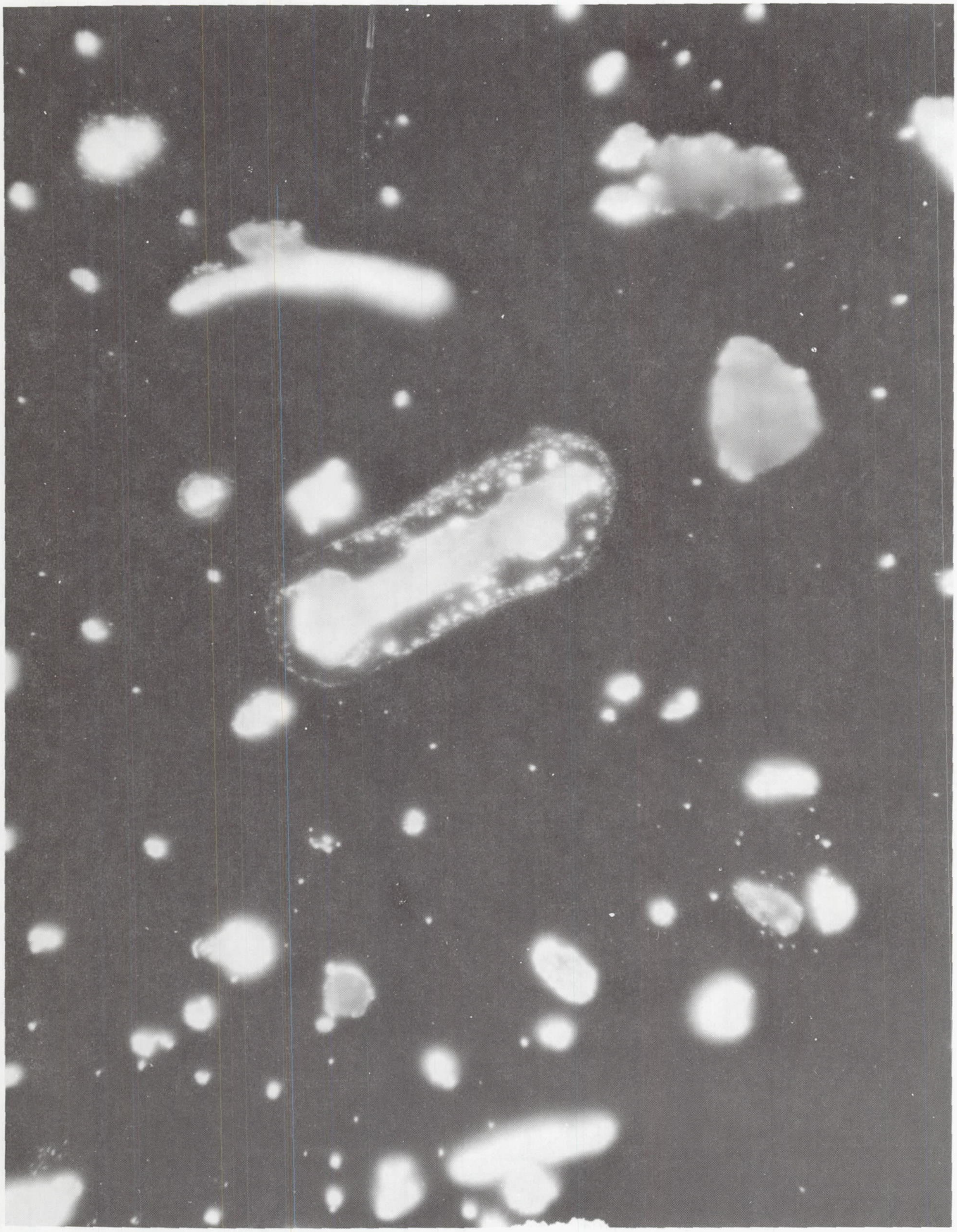

Figure 14. Dissociated Particles 280x

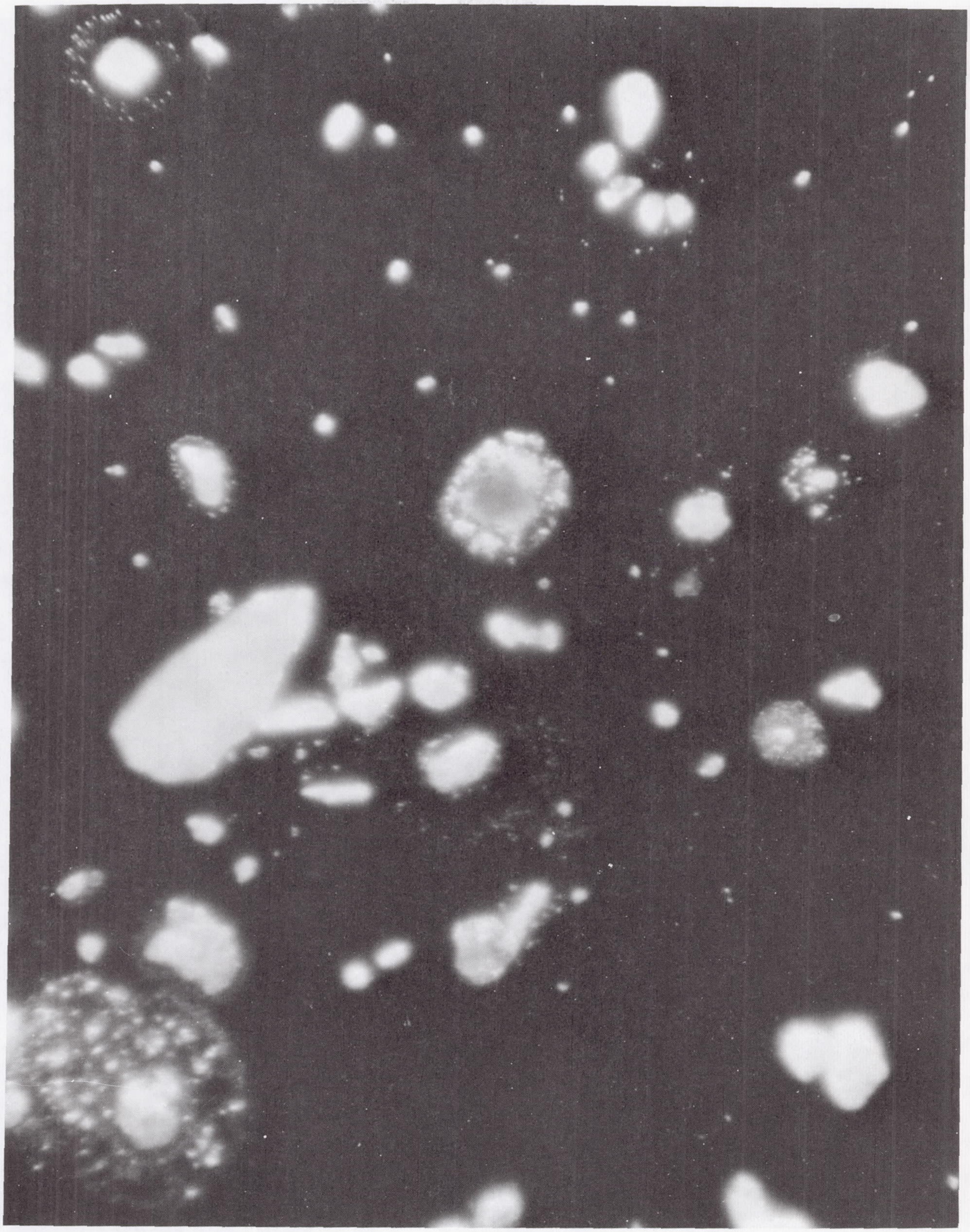

Figure 15. Dissociated Particles 280x

Figure 16. Dissociated Particles 500x

These experiments show relative humidity to be a very important factor in surface particle retention which relates directly to total loading factors for a spacecraft. The hygroscopic property of particles - the ability to pick up moisture from air - appears to be a major factor in the adhesion of particles to surfaces. Molecular, electrostatic and other forces account for only approximately 20% of the test particles retained on the test strips.

As a result of this study, it may be seen that particle removal (and therefore bioburden loss) from a spacecraft surface is most easily facilitated either by cleaning or natural environmental removal factors when the surface has not been exposed to high humidity environments. In particular, it would appear that spacecraft surfaces should not be exposed to environments with relative humidity above about 50% for even short periods of time.

This study calls attention to another aspect of hygroscopic particles that collect water from the surrounding air. Corrosion or oxidation can occur as "holes" or "pits" in a surface as a result of collected water. Should the hygroscopic particle cause a change in pH on absorption of water, then accelerated corrosion of the surface could be expected.

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