IN-25-CR
72171
p 26

Report to:

National Aeronautics and Space Administration
George C. Marshall Space Flight Center
Marshall Space Flight Center, Alabama 35812

Order No. H-11942D

ANALYSIS OF PARTICULATE CONTAMINATION ON TAPE LIFT SAMPLES FROM THE VETA OPTICAL SURFACES

Submitted by:

McCrone Associates, Inc.

Mark S. Germani, Ph.D.
Principal Investigator

Date: 21 February 1992

MA Number: MA-21600

Copy 16 of 16

(NASA-CR-189918) ANALYSIS OF PARTICULATE CONTAMINATION ON TAPE LIFT SAMPLES FROM THE VETA OPTICAL SURFACES (McCrone Associates) 26 p

N92-19694

CSCL 07D

Unclas

G3/25 0072171

mccrone associates, inc.
850 PASQUINELLI DRIVE • WESTMONT, ILLINOIS 60559

ANALYSIS OF PARTICULATE CONTAMINATION ON TAPE LIFT SAMPLES FROM THE VETA OPTICAL SURFACES

INTRODUCTION

We received two plastic boxes, each containing nine tape lift samples. The boxes were labeled Parabola and Hyperbola and they had been sealed with tape. The samples in each box were labeled 1 through 9. You indicated in your letter dated 17 November 1991 that Sample 9 in each box was a control. You requested that we analyze Parabola samples 1, 4, 8 and 9 and Hyperbola samples 1, 5, 8 and 9.

The plastic boxes were opened in our Class 100 cleanroom. The tape lift samples consisted of a loop of tape stuck to the bottom of the plastic container. We cut out an approximately 1" long section of tape from the top of the tape loop. Each section of tape was mounted onto an adhesive covered 1" diameter aluminum block. The four Parabola and Hyperbola samples were placed into pre-cleaned plastic boxes and kept inside of zip-lock plastic bags. The remaining tape lift samples were kept in their plastic containers. The containers were resealed and placed back inside their plastic bags. The four Parabola samples are designated P1, P4, P8 and P9; the four Hyperbola samples are designated H1, H5, H8 and H9.

SAMPLE ANALYSIS

The samples were coated with a thin film of evaporated carbon. This is done to render the surface conductive so

that the samples can be examined in the scanning electron microscope. The samples were analyzed using our automated scanning electron microscope. Using this instrument, it is possible to perform unattended analysis of the particle contamination on the tape lift surfaces. Particles are detected by an increase in their backscattered electron signal above a preset video threshold. The video threshold is set at a fixed level above the average video signal from the tape lift surface at each field of view. The samples are analyzed at a magnification of 600X and each field of view is approximately 150 x 150 μm. We believe that under these conditions we are able to detect inorganic particles as small as 0.5 μm in diameter. For each particle, we determine its shape and size, location, and elemental composition as determined by energy-dispersive x-ray spectrometry. Particle data from each sample is transferred to a Dec Microvax II minicomputer.

Particles with less than 200 net x-ray counts are deleted from the data set. These are particles that do not exhibit any characteristic x-ray peaks in their energy-dispersive x-ray spectra. These may be organic or polymeric particles, or artifacts from features on the tape lift surface itself. For example, folds or cracks in the surface of the adhesive may be detected and "analyzed" by the automated scanning electron microscope. Cracks occur in the tape lift surface when it is carbon coated. These cracks can appear bright in the image due to electron beam charging.

Cluster analysis is used to determine the particle types for the remaining inorganic particles in the samples.

RESULTS

Table I is a summary of the particle analysis results from the tape lift samples for the VETA optical surfaces. This table contains the area analyzed, the number of inorganic particles >0.5 μm in diameter in the analysis area, particle loading, the average particle diameter, and the area fraction obscured by particles. The area fraction is determined based upon the average particle diameter for each sample and is not integrated over all particle sizes for the sample. The individual particle data can be obtained from the data files on the enclosed floppy disk.

The control samples, P9 and H9, had particle loadings of 2.07×10^{-6} and 4.94×10^{-7} particles per square micrometer, respectively. Samples P4, P8 and H5 had similar particle loadings to control sample P9, although they were somewhat higher than the value for control sample H9. Samples H8, P1 and H1 had significantly higher particle loadings than on either of the two control samples. Sample H8 had the highest particle loading at 1.41×10^{-5} particles per square micrometer, followed by sample P1 with a particle loading of 9.44×10^{-6} particles per square micrometer and sample H1 with a particle loading of 8.72×10^{-6} particles per square micrometer.

The samples had average particle diameters in the range of 1.5 to 4.8 μm. The area fraction obscured by particles in the two control samples were 3.7×10^{-6} and 2.1×10^{-6} for samples P9 and H9, respectively. All of the other samples had a higher area fraction obscured by particles than either of the control samples. These values ranged from a low of 5.5×10^{-6} for sample P8 to a high of 7.6×10^{-5} for sample P1.

Figures 1 through 8 are average particle size distributions for the samples. The particle size distributions are for average diameters from 0 to 20 μm with each bin representing 1 μm. The size data for the individual particles in each sample can be found in the data files on the floppy disk. Particles have irregular shapes. Shape factors are typically 1-3. Shape factor (SF) is defined as:

$$SF = \frac{P^2}{4\pi A}$$

where P and A are projected particle perimeter and area, respectively.

The cluster analysis results for the samples are given in Tables II through VII. The particle data sets from control samples P9 and H9 were combined for cluster analysis. This was done to provide a larger number of particles for cluster analysis. Together, the two sample data sets contain a total of 60 particles. Table II is the results of the cluster analysis for the combined control samples. This

table lists the cluster code, the percent number of particles in each cluster, the elemental composition for each cluster, and the possible materials represented by each cluster. The cluster codes listed in Tables II through VII correspond with the cluster codes that can be found in the data files on the floppy disk. In Tables II through VII, I have only listed those clusters that represent more than 5% of the total number of particles in a sample or a combined set of samples. For the most part, the samples contained a variety of particle types. Table III lists the cluster analysis results for sample H5. Table IV lists the cluster analysis results for the combined data sets from samples P4 and P8. Table V lists the cluster analysis results for sample H1. Table VI lists the cluster analysis results for sample P1. Table VII lists the cluster analysis results for sample H8.

The particle types in control samples P9 and H9 and samples H5, P4, P8 and H1 are very similar. These samples contain major amounts of magnesium silicate (talc), calcium carbonate, potassium chloride salt, and iron rich particles (iron oxide, iron metal, steel). The identification of the material for each particle type is based solely on the energy-dispersive x-ray analysis results. Energy-dispersive x-ray analysis provides data on elements with atomic number greater than 11 (sodium). We do not have any data on elements lighter than sodium such as carbon, oxygen, nitrogen. Therefore, these are not unambiguous material identifications. For example, in the case of particles that

contain major amounts of iron, we cannot distinguish whether these are iron oxide, iron metal or steel. The difference between iron metal and steel is only a small amount of manganese which we may not detect in very small particles. Therefore, the material identification should be used with caution. The major particle types in samples P1 and P8 are, however, somewhat different from the other samples and the controls. For example, the major particle types in sample P1 are aluminosilicate minerals and what we believe to be a calcium sulfate. These two particle types are not present in major amounts in the other samples or the controls. In the case of sample H8, the predominant particle type is sodium chloride salt. This particle type was also not present as a major particle type in the other samples and the controls.

You mentioned in a telephone conversation that the mirrors had been polished with cerium oxide and you thought that some of the particulate contamination may be cerium oxide which had not been cleaned from the mirror. The only sample in which we found a significant amount of cerium oxide is sample H8. The cerium oxide particle type represented only 7% of the total number of particles.

CONCLUSIONS

The results of our analysis of the particulate contamination on the tape lift samples would seem to conclude that samples P4, P8 and H5 are not significantly different in particle loading or particle type from the two control

samples, P9 and H9. Samples P1 and H8 are significantly different from the two control samples, both in particle loading and in the types of particles found on the tape lift samples. Sample H1 has a higher particle loading than either of the two control samples, but the particle types are generally similar to those found in the control samples.

APPENDIX A

Tables

TABLE I

Summary of Particle Analysis Results for Tape Lift Samples from the VETA Optical Surfaces

SAMPLE	AREA ANALYZED (μm^2)	PARTICLES ANALYZED*	PARTICLE LOADING (#/μm^2)	AVERAGE PARTICLE DIAMETER (μm)	AREA FRACTION OBSCURED BY PARTICLES
P1	2.34×10^{7}	221	9.44×10^{-6}	3.2 ± 4.0	7.6×10^{-5}
P4	2.46×10^{7}	59	2.40×10^{-6}	2.4 ± 3.3	1.1×10^{-5}
P8	2.53×10^{7}	44	1.74×10^{-6}	2.0 ± 2.7	5.5×10^{-6}
P9 (control)	2.32×10^{7}	48	2.07×10^{-6}	1.5 ± 1.5	3.7×10^{-6}
H1	1.25×10^{7}	109	8.72×10^{-6}	3.0 ± 3.7	6.2×10^{-5}
H5	2.04×10^{7}	41	2.01×10^{-6}	4.8 ± 4.8	1.5×10^{-5}
H8	2.47×10^{7}	349	1.41×10^{-5}	1.5 ± 1.6	3.3×10^{-5}
H9 (control)	2.43×10^{7}	12	4.94×10^{-7}	2.3 ± 1.3	2.1×10^{-6}

* Inorganic particles with total x-ray counts >200. Particles >0.5μm in diameter.

Ref: MA21600

TABLE II

Cluster Analysis Results for Tape Lift Control Samples for the Parabola and Hyperbola Mirror Surfaces

SAMPLE: Combined P9 and H9 control samples			
NO. OF PARTICLES: 60			
CLUSTER CODE	**PERCENT NUMBER OF PARTICLES**	**ELEMENTAL COMPOSITION**	**POSSIBLE MATERIAL**
2	11	Mg, Si	talc
9	8	Fe	iron oxide, iron metal, steel
7	7	Ca	calcium carbonate
6	6	Pb	lead metal
3	5	Zn	zinc metal
11	3	K, Cl	salt
16	3	Mo	molybdenum disulfide

Ref: MA21600

TABLE III

Cluster Analysis Results for Tape Lift Sample from Position 5 on Hyperbola Mirror Surface

SAMPLE:	H5		
NO. OF PARTICLES:	41		
CLUSTER CODE	PERCENT NUMBER OF PARTICLES	ELEMENTAL COMPOSITION	POSSIBLE MATERIAL
4	15	Mg, Si	talc
1	12	Cu, Zn	brass
6,8	12	K, Cl	salt
2	7	Ca	calcium carbonate

Ref: MA21600

TABLE IV

Cluster Analysis Results for Tape Lift Samples from Positions 4 and 8 on Parabola Mirror Surface

SAMPLE:	Combined P4 and P8		
NO. OF PARTICLES:	103		
CLUSTER CODE	**PERCENT NUMBER OF PARTICLES**	**ELEMENTAL COMPOSITION**	**POSSIBLE MATERIAL**
6,15	21	Fe	iron oxide, iron metal, steel
8	12	Mg, Si	talc
17	10	Pb	lead metal
1	9	Ca	calcium carbonate
9	8	K, Cl	salt

Ref: MA21600

TABLE V

Cluster Analysis Results for Tape Lift Sample from Position 1 on Hyperbola Mirror Surface

SAMPLE: H1			
NO. OF PARTICLES: 109			
CLUSTER CODE	**PERCENT NUMBER OF PARTICLES**	**ELEMENTAL COMPOSITION**	**POSSIBLE MATERIAL**
1	17	Fe	iron oxide, iron metal, steel
8	9	Ca	calcium carbonate
12	8	Al	aluminum metal, aluminum oxide
10	6	Mg, Si	talc

Ref: MA21600

TABLE VI

Cluster Analysis Results for Tape Lift Sample from Position 1 on Parabola Mirror Surface

SAMPLE: P1			
NO. OF PARTICLES: 221			
CLUSTER CODE	**PERCENT NUMBER OF PARTICLES**	**ELEMENTAL COMPOSITION**	**POSSIBLE MATERIAL**
3,25	12	Al, Si, Ca	aluminosilicate minerals
23	9	Ca, S	calcium sulfate
2	8	Fe	iron oxide, iron metal, steel
6	8	Mg, Si	talc
17	8	Ca	calcium carbonate
22	6	Na, Cl	salt

Ref: MA21600

TABLE VII

Cluster Analysis Results for Tape Lift Sample from Position 8 on Hyperbola Mirror Surface

SAMPLE: H8			
NO. OF PARTICLES: 349			
CLUSTER CODE	**PERCENT NUMBER OF PARTICLES**	**ELEMENTAL COMPOSITION**	**POSSIBLE MATERIAL**
1	54	Na, Cl	salt
17	8	Cu, Zn	brass
6	7	Mg, Si	talc
3	7	Fe	iron oxide, iron metal, steel
29	7	Ce	cerium oxide

Ref: MA21600

APPENDIX B

Figures

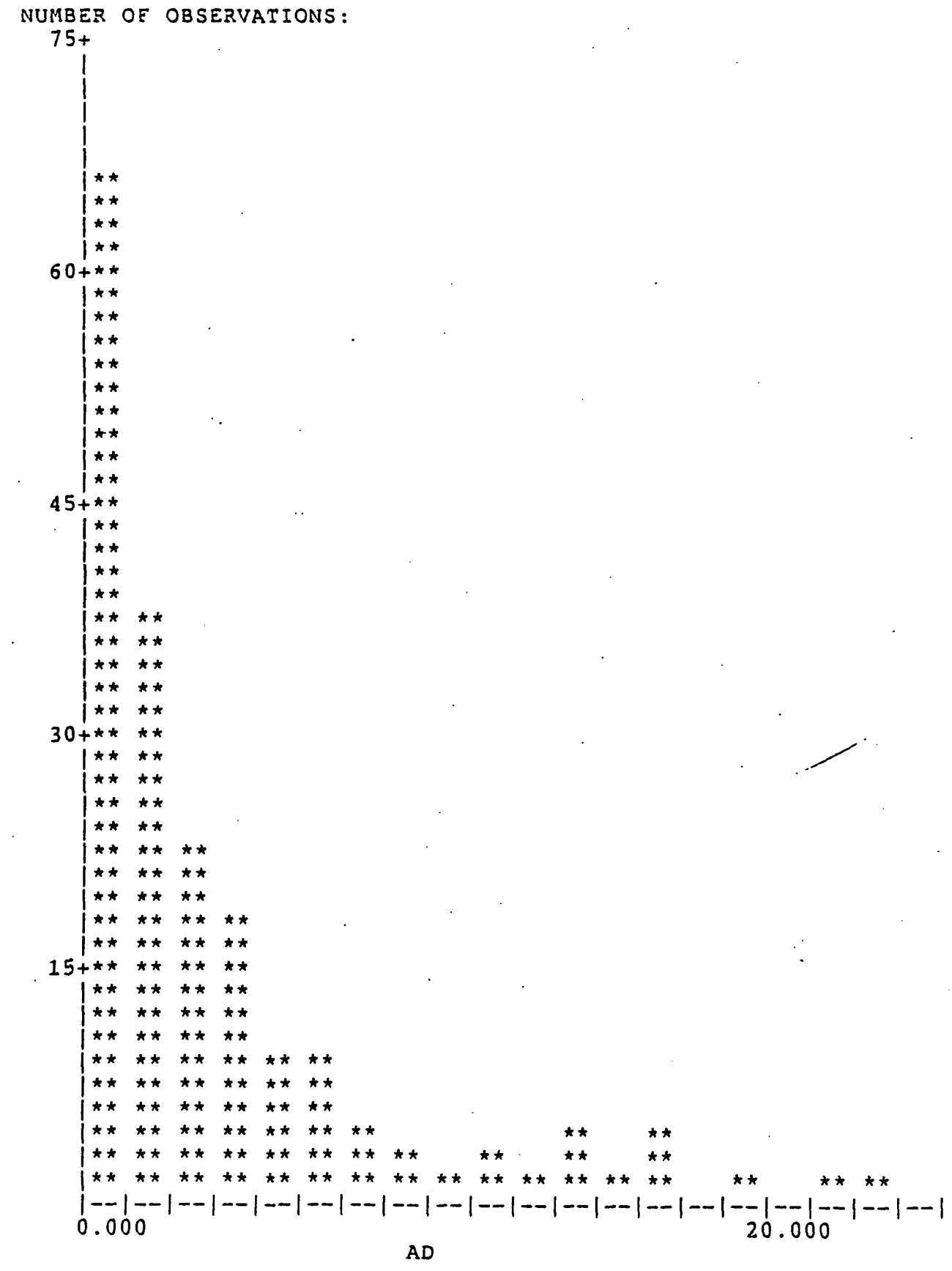


FIGURE 1
Tape lift sample P1.

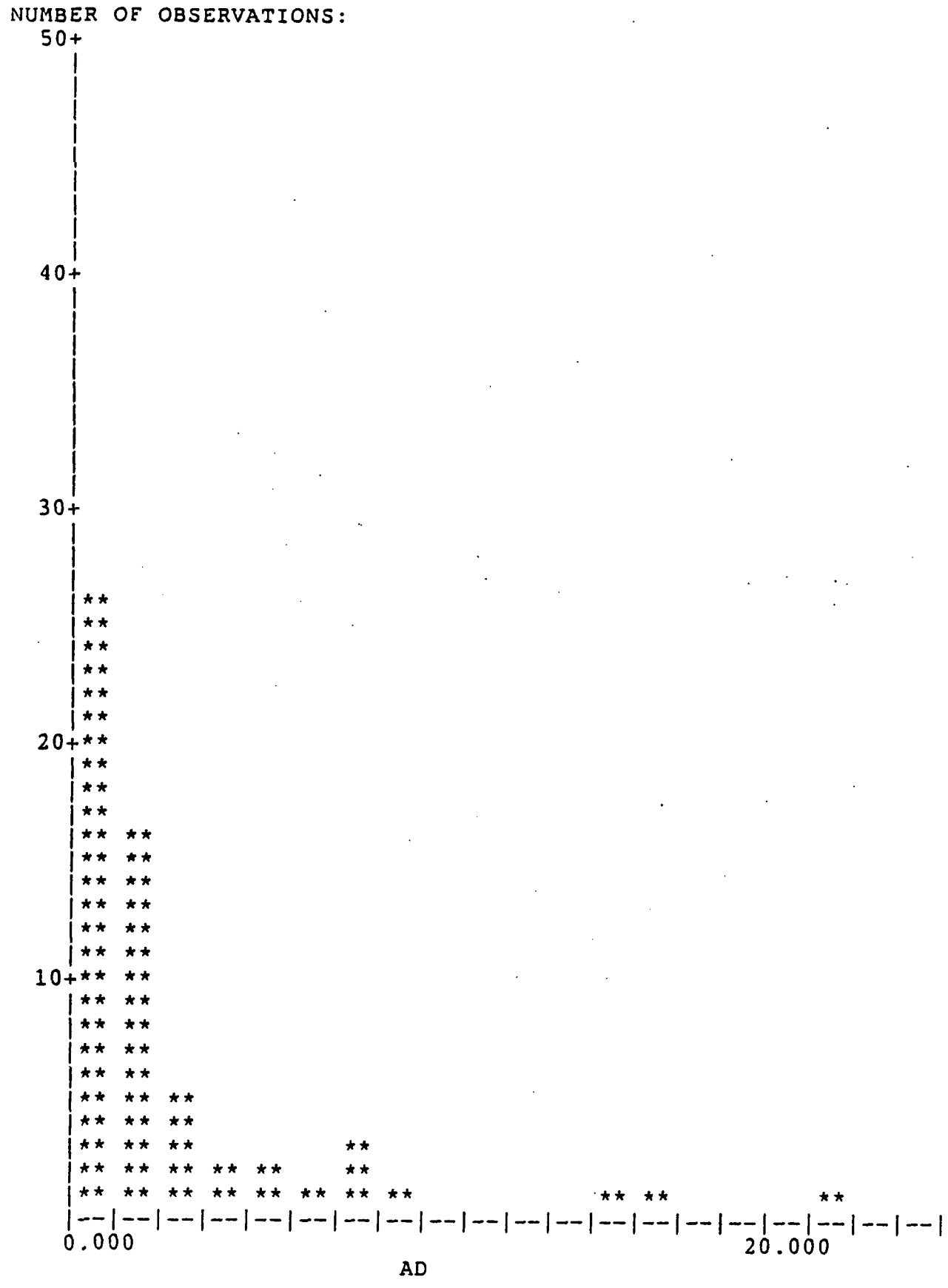


FIGURE 2
Tape lift sample P4.

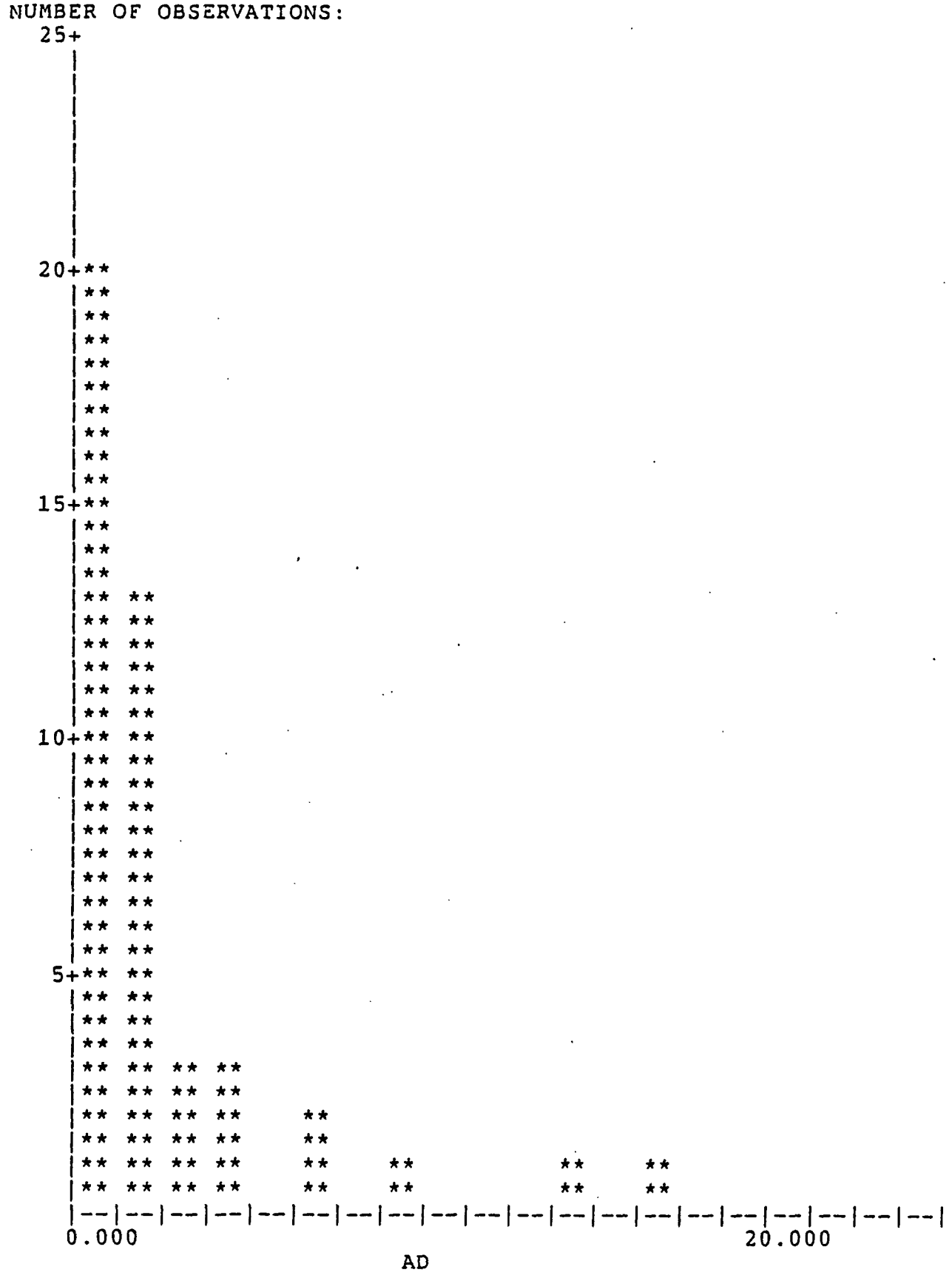


FIGURE 3
Tape lift sample P8.

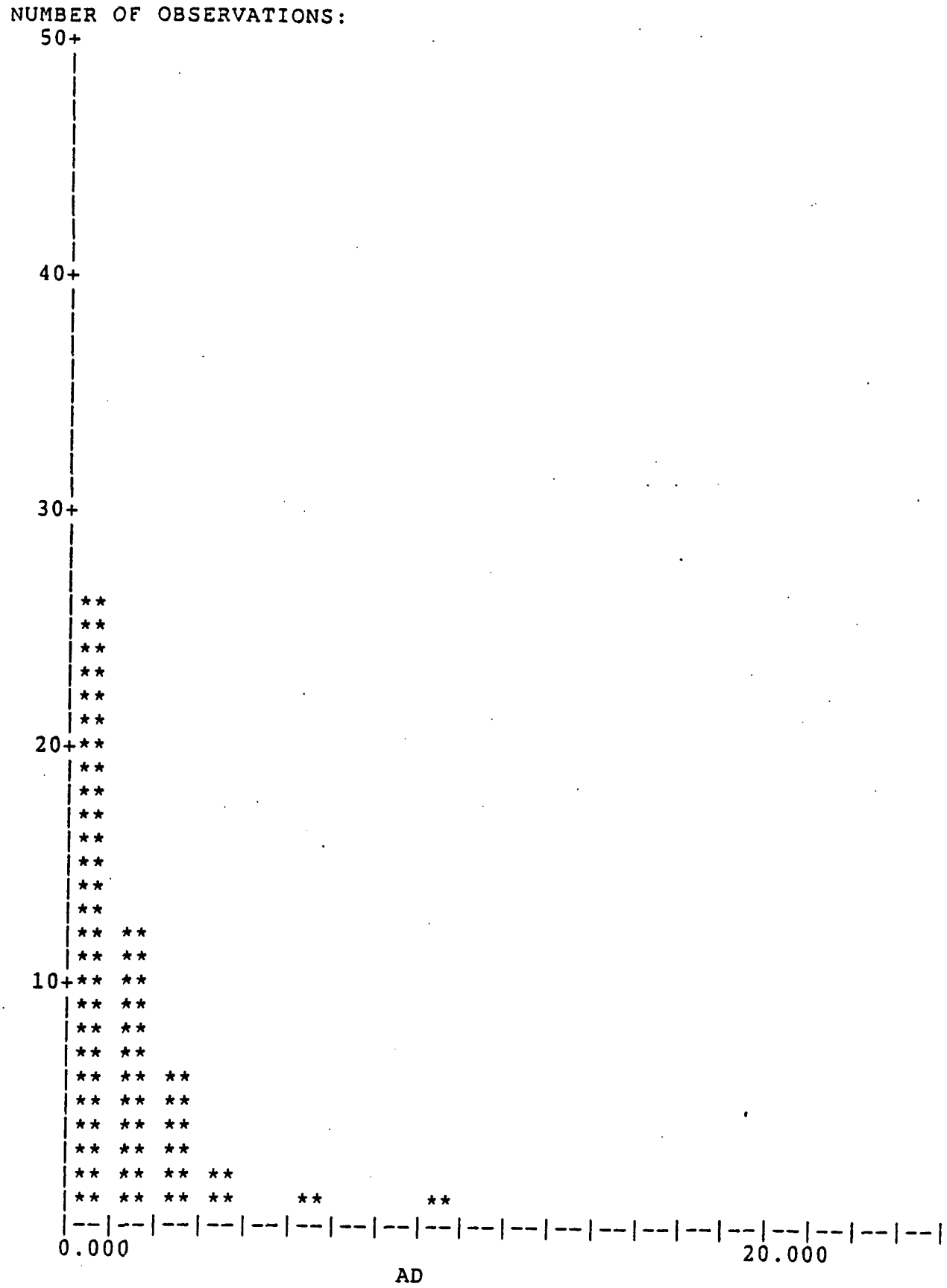


FIGURE 4
Tape lift sample P9.

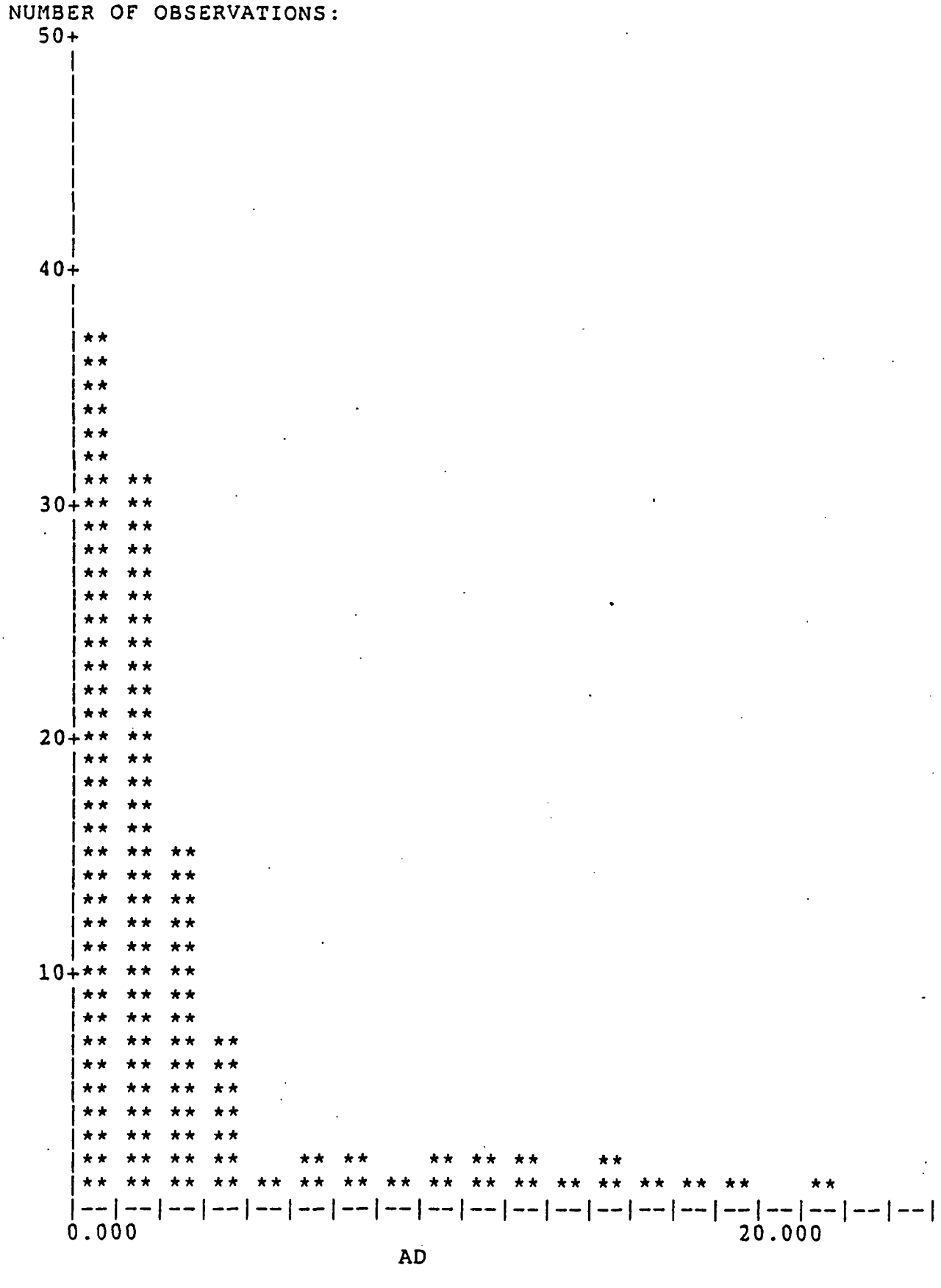
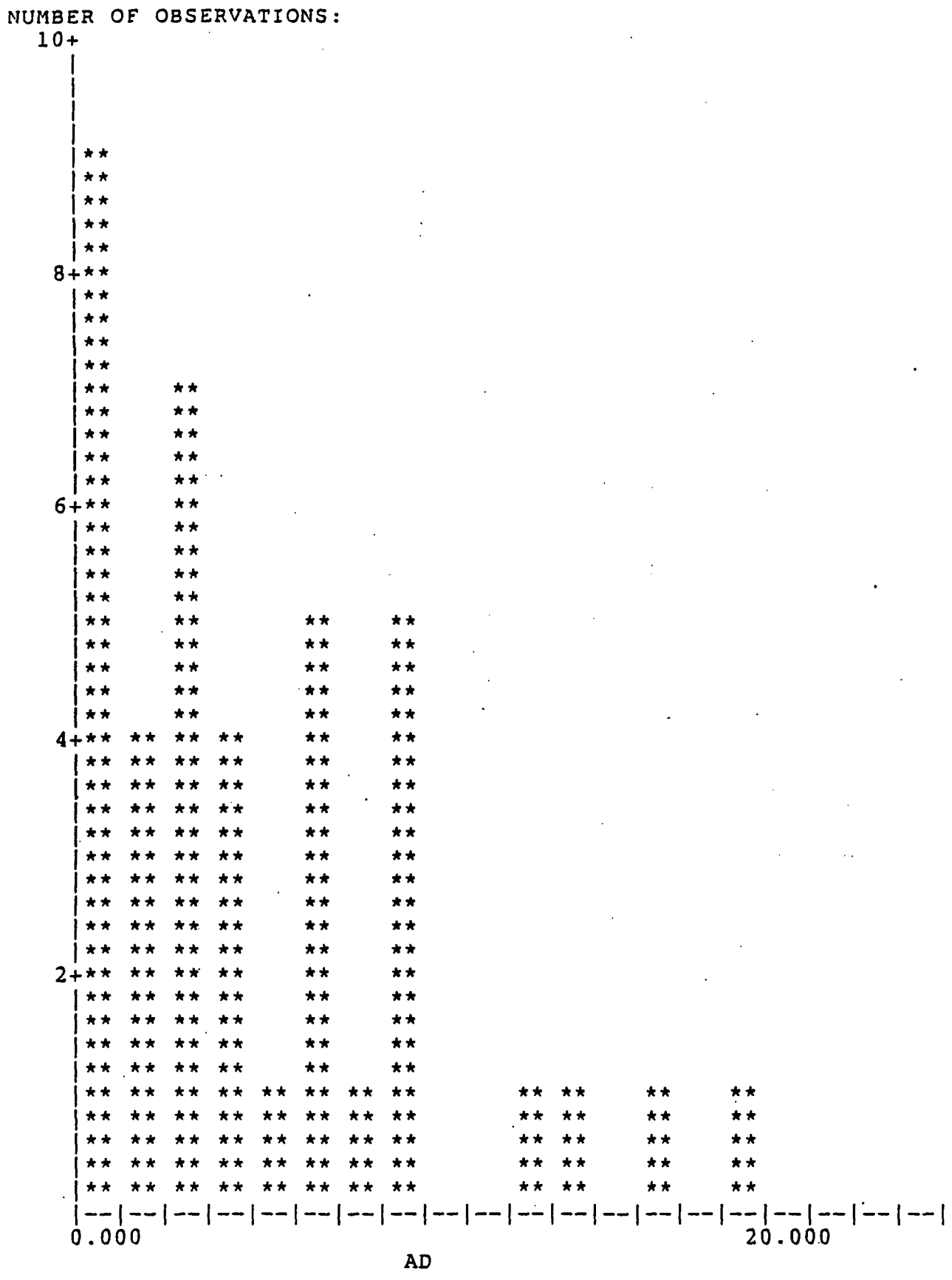


FIGURE 5
Tape lift sample H1.



Average Particle Diameter (μm)

FIGURE 6
Tape lift sample H5.

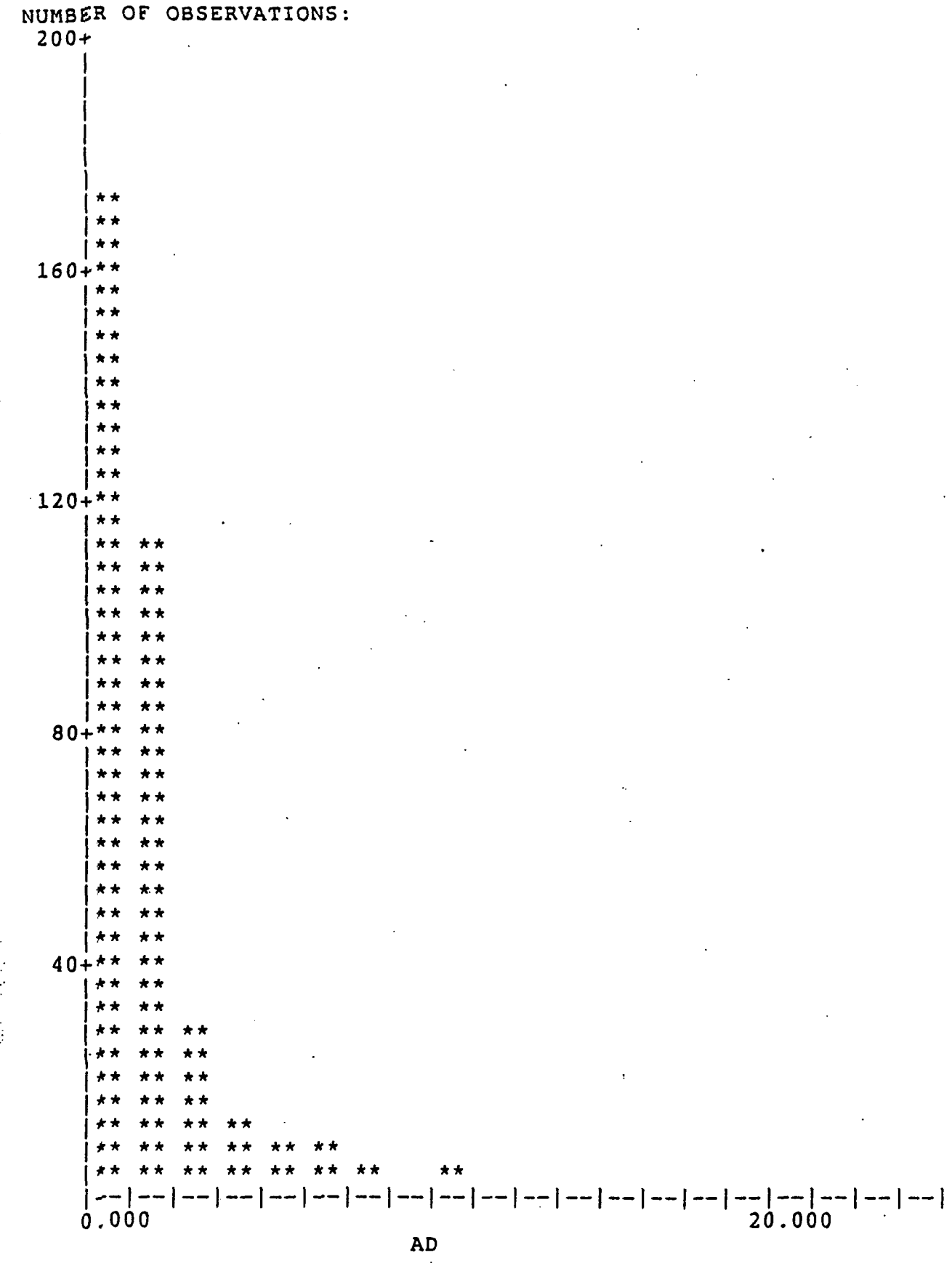


FIGURE 7
Tape lift sample H8.

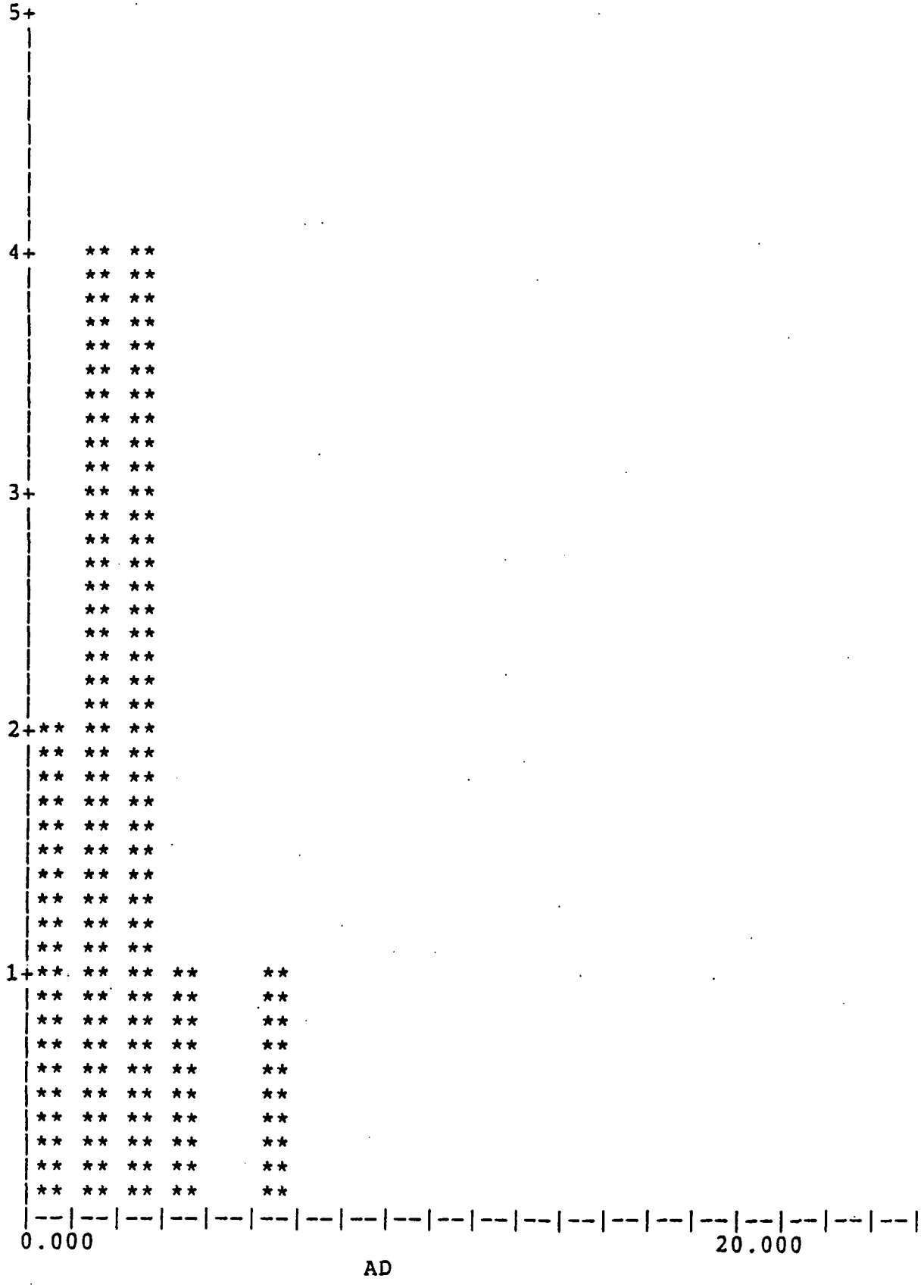


FIGURE 8
Tape lift sample H9.

NASA

Report Documentation Page

1. Report No	2. Government Accession No.	3. Recipient's Catalog No.

4. Title and Subtitle

ANALYSIS OF PARTICULATE CONTAMINATION ON TAPE LIFT SAMPLES FROM THE VETA OPTICAL SURFACES

5. Report Date

February 1992

6. Performing Organization Code

7. Author(s)

Mark S. Germani, Ph.D.
Principal Investigator

8. Performing Organization Report No.

MA21600FR

10. Work Unit No.

9. Performing Organization Name and Address

McCrone Associates, Inc.
850 Pasquinelli Drive
Westmont, IL 60559

11. Contract or Grant No.

H-11942D

13. Type of Report and Period Covered

12. Sponsoring Agency Name and Address

14. Sponsoring Agency Code

15. Supplementary Notes

16. Abstract

Automated electron microprobe analyses of particulate contamination on tape lift samples from the VETA optical surfaces. This report details the individual particle analyses and describes the contaminant particles on the hyperbola and parabola mirror surfaces.

ORIGINAL PAGE IS
OF POOR QUALITY

17. Key Words (Suggested by Author(s))	18. Distribution Statement
VETA particle contamination mirror surfaces	Unclassified - Unlimited

19. Security Classif. (of this report)	20. Security Classif. (of this page)	21. No. of pages	22. Price
Unclassified	Unclassified	25	