PARTICLE COUNTING: AN ANALYSIS OF THE PROBLEM

OF CALIBRATION AND UNCERTAINTY

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CHAPTER I

INTRODUCTION

The capability to accurately estimate the parameters of the distribution of sizes of small particles (i.e., 1 to 100 micrometres) is very important in a number of diverse fields. In medicine, blood cell counts are based on the size difference of red and white cells. The ultimate characteristics of sintered products in powder metallurgy is critically dependent on particle size. Of special concern to the author, the technology of contamination control in fluid power systems is totally dependent on accurate, easily obtained, counts of various size particles entrained in fluid samples.

There are a number of techniques employed in particle counting. Manual counts using microscopes have been used for many years. By the late 1960's, it had been recognized that microscopic counting techniques were neither accurate enough nor sufficiently economical to support the research and development effort required to evaluate the filters and components used in fluid power systems. The opto-electronic automatic particle counter was the most promising technique for a general methodology if a suitable standard method could be developed for calibration and use of the instrument.

The Fluid Power Research Center (FPRC) of Oklahoma State University undertook the task of developing and promulgating such a standard, and, in 1973, the American National Standard B93.28-1973 was adopted. This

standard is presently being circulated for ballot as an international (ISO) standard. As is the case with all such standards, the method proposed is a compromise of the technology in order to meet political constraints. The standard incorporates the use of Shewhart \overline{X} and R charting which was virtually unknown to the participating committee members both in the U.S.A. and abroad. At the time the standard was promulgated, the requirements were considered far too severe. The standard is now under attack as unacceptably lenient.

This is a healthy sign, reflecting the growing sophistication of the industry. The scope and nature of the problems of particle counting are much more generally understood, and the environment is conducive to the acceptance of statistical control techniques.

There are two considerations in applying statistical control aspects to automatic particle counting: (1) the physical nature of counting process including optical and electronic effects, and (2) the nature of the size vs. frequency of occurrence of various contaminants encountered. This thesis presents the results of an extensive investigation of the counting phenomena, an analysis of a large body of data from actual particle counts, and proposes a set of statistical quality control techniques to be incorporated into a revised proposed standard method.

CHAPTER II

SURVEY OF RELATED TOPICS

Over one hundred papers, books, and monographs relating to particle counting were reviewed in preparation of this thesis. Only fifteen made any mention of calibration or uncertainty. Nine of the papers were concerned with particle distributions, another nine addressed particle behavior, and the balance either advanced new particle counting methods or reviewed existing techniques. The lack of concern with the metrological aspects is surprising in view of the widespread interest in particle counting. Prior to the introduction of the first automatic particle counter in 1956 by W. H. Coulter, all particle counting was performed manually by microscope examination which may explain the absence of calibration studies.

As early as 1929 it was recognized that the most useful information about particulate contamination would be in the form of a mathematical relationship between particle size and frequency of occurrance [1]. Subsequently a number of investigations have postulated appropriate models including the Hyperbolic [2] and the lognormal [3]. These probabilistic models are intellectually appealing since most naturally occurring contaminants are of essentially infinite supply. However, the automatic particle counter yields only the number of particles greater than a selected size. There is no way to determine the total number of particles in a sample for estimating

relative frequency. A model proposed by Cole [4] provides a useful transformation of the log-normal. This equation is very tractable and particularly well suited for graphical analysis. The model is of the form

$$n = A \int_{-\infty}^{\ln X} \exp(-B\ln^2 X)$$
(2-1)

where A and B are emperical constants for the contaminant in question. This model has been generally accepted in the Fluid Power Industry and is an integral part of several American National Standards dealing with contamination control. Particle counting is concerned principally with the estimation of the parameters of this model and any method of calibration must be relevant to this application.

The major impetus for standardization came with the space program when strict cleanliness standards were imposed on various components being manufactured at different locations. A research group at Oklahoma State University was working on contamination sensitivity and filtration for N.A.S.A. and an evaluation of automatic particle counters was a part of the study. The study by Zaloudek [5] is one of the earliest attempts to quantify the uncertainty of an automatic particle counter. Zaloudek presented data which strongly supported the proposal by Michaelson [6] that the appearance of particles in the counter was a Poisson process.

In the late 1960's both the National Fluid Power Association and the Society of Automotive Engineers began the development of calibration standards for automatic particle counters. The SAE standard which was published in 1970 as Aerospace Recommended Practice (ARP) 1192 was

an outgrowth of earlier work with microscopic counting and in fact relied on correlation with microscopic results. The limitations of the latter technique were widely known and as a result ARP 1192 received little acceptance.

The N.F.P.A. Standard was directly the result of a broadly based study by the Fluid Power Research Center at Oklahoma State University which included a series of International Round Robin Tests using known and unknown (i.e., undisclosed) samples. The procedure uses an arbitrary calibration standard, A.C. Fine Test Dust, the distribution of which is accepted knowledge and conforms to the log-log² model. This standard was adopted as an American National Standard in 1973.

At the time the ANSI standard procedure was developed it was not uncommon to observe a 200% variation in counts of the same fluid by different laboratories. The round robin test of 1971-72 showed a coefficient of variation of 74% for laboratories not using the proposed procedure. Within two years of publication a large number of users had become sufficiently expert in the science of particle counting to detect serious deficiencies in the procedure. Primarily these problems are the result of the use of statistical procedures which do not discriminate against a process which does not have an origin in a stable region. If the particle counting process has an extremely large variance, or is operating in an invalid concentration region when the calibration procedure is started, the results are unrealistic.

The objective of this thesis is to develop analytical descriptions of the counting process and to apply these models to the structuring of a fundamental and general calibration and control procedure for automatic particle counters.

CHAPTER III

THE COUNTING PROCESS AND SOURCES OF ERROR

All of the automatic particle counting devices in use today exhibit a degree of non-repeatibility and error. Although the actual counting technique varies widely between the different types of instruments, the concepts employed are similar and the basic sources of error are common to all. The total light reduction principle is by far the most widely used technique and will be used to illustrate the problems associated with reducing the uncertainty of particle counts.

The basic particle counting device is illustrated in Figure 1. A stream of fluid is passed through a transparent chamber (usually quartz) across which a collimated beam of light is focused. A photoelectric device (typically a photo diode) measures the intensity of the light passing through the fluid. If a particle whose index of refraction is sufficiently different from the fluid [7] passes between the light source and the detector, the light is scattered and/or blocked resulting in a reduction of intensity which is directly proportional to the projected area of the particle. The output of the photo detector is amplified and conditioned electronically to provide a train of pulses such as that shown in Figure 2. A level detector(s) determines whether or not the pulse exceeds a preset value. If it does, a counter is incremented. The levels correspond to the intensity reduction caused by a circular cross section particle of the specified





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Figure 2. Electrical Output From Sensor

diameter. The result is the number of particles greater than a specified diameter which passed the sensing zone.

To the user, the variation in counts obtained from repeated samples is in the form of scatter of the counts about a mean value, the deviation of the mean from a known or expected correct count, and the failure of the observed count to increase proportionately with increasing particle concentration. This latter condition is generally called saturation and is depicted in Figure 3.

From an analytical viewpoint the sources of error can be grouped into three categories: (1) procedural, often erroneously called operator induced, which are effects not related to the actual counting phenomenon; (2) functional, which are limitations of the counting device; and (3) inherent variation in the counting process.

Generally the particle counting procedure consists of obtaining a sample of contaminated fluid from the system in a suitable container, passing a known portion of the sample through the sensing zone and reporting the result in terms of a standard volume, usually one millilitre. If the count exceeds the saturation level discussed above, the fluid is diluted with clean fluid and the process repeated. If the sample container is not extremely clean, the background contamination in the container will add to the actual count. Any variation in measurement of the fluid volume passing the sensor results in a directly proportional error in the perceived count. If dilution is required the inaccuracies of the volume measurements will reflect directly on the magnitude of the count and if the sample is not properly mixed, very erratic results are obtained. In addition, the particles tend to either settle or float in the fluid and care must be

9.



NUMBER OF PARTICLES NA

Figure 3. The Saturation Phenomenon

taken to assure proper dispersion of the particles. Until very recently these procedural effects were the major source of uncertainty in particle counts and in fact may still be significant. However, in 1970 a sample container cleanliness verification procedure was developed at the Fluid Power Research Center which has since been adopted as an international standard. Improved particle counters which incorporate photo-electric volume measuring have sharply reduced the variance from this source.

The requirement for dilution imposes a severe burden on the particle counting technician. Dilutions of 40 to 1 and higher are not uncommon and this requires very precise measurement of small volumes of fluid.

The functional errors of the particle counter are related both to design variables and the condition of the counter. The counter may simply fail to count correctly due to erratic behavior of the counting or display electronics. Variations in the reference level voltages will result in counts which represent a particle diameter other than that intended. Due to the extreme nonlinearity of the particle distribution a reduction in level would result in a positive increase in count much larger than the decrease associated with an equivalent upward shift in level. It should be noted that the signal from the photo detector is a function of the area and not the diameter and therefore if linear amplification is used the change in perceived particle size is proportional to the square root of the change in threshold setting.

The signal from the photo detector is a function of the rate at which the light source in the sensor is blocked. This is in

turn a function of the size of the particle and the flow rate. If the amplifiers and associated circuitry do not exhibit acceptable frequency response, the output for a given particle size is lower for increasing flow rate. This effect is the same as increasing the threshold level so that increasing flow causes reduced counts. If the apparatus is calibrated in too high a flow region it will be operating within only a portion of the normal electronic regions.

The lowest particle size which can be counted accurately is for any particular instrument configuration ultimately determined by the signal to noise ratio. Any counter exhibits a random signal which results from vibration of sensor elements, electromagnetic interference and thermal emission. Instrument design and environment influence this phenomenon to a considerable degree but it cannot be eliminated; there is a threshold below which the counter will register the passage of a particle even when the sensor is empty. Electromagnetic fields may induce electrical impulses of a magnitude well above the normal noise level. While these might properly be called procedural problems related to the environment, the sensitivity to such phenomena are so strongly related to circuit design that they must be considered functional.

All of the light blocking counters currently in use employ assyncronous counting. Therefore it is essential that the signal from the photo detector fall below the threshold level for a sufficient length of time for the comparator and counter circuits to reset. This implies that no particle or group of particles with a total area greater than the size of interest may be in the sensor during the reset interval.

If all the sources of error described thus far were eliminated one would still expect some variation in repeated counts (unless by some means all the fluid in the population were counted) due to the natural difference in samples. In addition there exists a possibility that two or more particles will be passing through the sensor simultaneously. This phenomenon is called coincidence and is widely discussed in the literature and among users of particle counters. Coincidence is offered as an explanation for nearly every major defect including saturation.

It is apparent that not all of the potential sources of error can be of equal significance. In order to determine the appropriate statistical control techniques for evaluating particle count uncertainty, it is necessary to determine the degree to which each potential error can occur and the influence of the error on the resulting count. This is developed in the next chapter.

CHAPTER IV

ANALYSIS OF THE ERROR PROBABILITIES

In order to evaluate the error probabilities it is necessary to postulate a model for the appearance of the particles in the sensing zone. Most investigators have proposed the Poisson model. This is attractive both for the tractability of the mathematics and from experience with similar mechanisms encountered in industrial and natural processes. Analysis of a large amount of particle count data from various sources shows excellent agreement with this hypothesis.

The Poisson is a distribution on the number of occurrances in some interval or area [7]. The probability mass function is

$$P(X = x; \lambda) = \frac{e^{-\lambda} \lambda^{X}}{X!}; X = 1, 2, ..., n.$$
 (4-1)

Lamda has units of occurrances per unit opportunity. In the case of estimating the coincidence probability this is particles per sensor volume which can be estimated as particles per millilitre of fluid multiplied by the volume of the sensing zone in millilitres. Thus for a particular particle size lambda can be estimated if the distribution is known. For example using Cole's model for A.C. Fine Test Dust the number of particles greater than 10 micrometers is

$$N > 10 = Ke^{-\beta \ell n^2 D} = 144$$
 particles per microgram , (4-2)

where

K = 1751.9, and

 $\beta = 0.4714.$

In a 28 mg/ ℓ solution of test fluid this would yield a Lambda of .11 with a sensor volume of 2.58 x 10⁻⁵ ml. The probability of 2 or more particles residing in the sensor simultaneously is given by

P(2 or more) = 1 - P(1 or less) = 1 -
$$\sum_{K=0}^{1} \frac{e^{-\lambda} \lambda^{K}}{K!}$$
. (4-3)

Unfortunately, repeated tests by a number of investigators including the particle counter manufacturer have shown that the ratio of observed count to expected count under these conditions is about .90 which implies a .10 coincidence probability. A careful analysis of the requirement for coincidence error reveals that this is almost certainly not occurring in any realistic particle counting situation.

If any two or more particles of a size greater than the threshold size (D) appear in the window at the same time, a count will be missed. If a particle of size \geq D and any quantity of particles \leq D appear simultaneously, no error occurs. However, if two or more particles of size \leq D, whose combined area is greater than the area associated with D, appear during an interval when no particle greater than D is in the sensor, a false count will be recorded (see Figure 4).

Analyzing multiple occurrances of particles in various size intervals requires that Lambda be a function of particle size and that the probabilities be evaluated for an infinite number of intervals. This



Figure 4. Coincidence Error

of zero occurrances in an interval of time t is

$$P(\circ) = e^{-\lambda t} \qquad (4-5)$$

where λ has the same meaning as described earlier. To translate Lambda from the volume domain to the time domain requires the consideration of flow through the sensor which is 30 ml per minute in current practice. Thus

$$= \frac{.11 \text{ particles}}{2.58 \times 10^{-5} \text{ ml}} \times \frac{30 \text{ ml}}{\text{min}} \times \frac{60 \text{ min}}{\text{sec}} = 2131.78$$
(4-6)

considering the particle to be a point (a reasonable assumption for particles which are substantially smaller than the sensing volume) the time for a particle to transit the sensor is

$$\frac{2.58 \times 10^{-5} \text{ ml}}{.5 \text{ ml/sec}} = 5.16 \times 10^{-5} \text{ sec} . \tag{4-7}$$

The particle counter requires a reset time of 12 μ s so that the total time between particles must be 63.6 x 10⁻⁶ sec. Therefore the probability that reset will occur is

$$e^{-2131.78(63.6 \times 10^{-6})} = .87$$
 (4-8)

which shows excellent correlation with published data [8, 9]. The equation can be generalized as follows

$$P(\text{count}) = e^{-N_D(V_S + QT_R)}$$
(4-9)

where

$$N_{D}$$
 = actual number of particle \geq size D,
 N_{c} = sensor volume,

Q = flow rate, and

 T_{p} = minimum time to reset.

The error rate is sensitive to flow rate, especially for large reset times, which has also been documented by Moore [10].

The reduction in particle count due to increases in flow rate may also be caused by roll-off of the amplifier circuits as a function of the frequency response. However, the rate of change of the area shaded by a particle is a function only of the sensor configuration, particle size and particle velocity, thus the count loss due to frequency response characteristics is independent of particle concentration in the fluid. The effects of roll-off and reset error can therefore be separated.

If the coincidence and reset constraints are met, a lower bound can be placed on the counting uncertainty by virtue of the Poisson assumption. As has been implied in the earlier discussion, the parameter Lambda can be estimated from the particle counts. The unbiased point estimate of Lambda [11] can be determined as the ratio of total occurrances to the total opportunity. In the volume domain this is

$$\lambda = \frac{\text{particle count x volume of sensor}}{\text{volume counted}} .$$
(4-10)

Probabilities of various particle counts can be computed readily using Equation (4-1). As a practical matter we are seldom concerned with a single count; the use of averages of five counts is the common practice. For this case we utilize the central limit theorem and the characteristics of the Poisson which dictate that the mean and the variance are both equal to Lambda [11], so that the confidence limits on the mean are

$$n + \frac{1}{2} c_{\alpha/2}^2 \pm \sqrt{(n c_{\alpha/2}^2 + \frac{1}{4} c_{\alpha/2}^4)}$$
 (4-11)

where c_{α} is the upper α point of the unit normal distribution and n is the estimate of λ . For large values of n this can be approximated by

$$n + c_{\alpha/2} \pm \sqrt{n} \quad . \tag{4-12}$$

The effects of volume measurement and dilution error are linear with particle count while sampling, mixing and cleanliness problems will most likely be more pronounced in the smaller particle sizes. There is no way to predict such errors individually from an unknown sample but various experimental designs incorporated into the calibration procedure can assign expected upper limits to these effects.

For example if solutions of A. C. Fine Test Dust of 2, 5, 10, 20, and 50 are diluted by 2 to 1, 5 to 1, etc., the resulting counts should be within the same limits as repeated samples from 1 mg/ ℓ solution. The variation in excess of this amount must be attributed to procedure. The uncertainty associated with procedural defects must be incorporated in any estimate of the accuracy of the particle counting process.

Industrial specifications that require contamination assessment for measuring performance criteria must be based on the assumption that reproducible and accurate particle counting is performed. Each of the potential sources of error must be evaluated and controlled by a methodology which is essentially free from subjective judgement.

The next chapter presents a general procedure which meets this objective.

CHAPTER V

PROPOSED CONTROL PROCEDURE FOR PARTICLE COUNTING

The extent of the uncertainty which can be tolerated in any measurement is related to the intended use of resulting information. The tape measure which is wholly satisfactory to the carpenter, finds little use in a precision machine shop. In the same manner, a laboratory attempting to identify small differences in filters would require much greater precision and repeatibility in particle counting than would be needed for field contamination assessment of operating systems.

The flow rate and concentration effects inherent in the particle counter impose a direct trade off between minimal uncertainty and operating flexibility. The design of many particle counters is such that narrow confidence intervals would dictate such low concentration that virtually every sample would require dilution. The limitation of the procedural capabilities would effectively eliminate such instruments. In the recommendations which follow, this problem is avoided by addressing only the question of establishing .90 confidence limits. These limits may be too restrictive, but the techniques presented are not limited to the particular value chosen.

The calibration of the instrument must be accomplished independently of procedural errors if this **is** at all possible. The ANSI B93.28-1973 Standard avoids this question by pre-supposing that the

laboratory is fully competent in such areas. This approach is necessary to some extent as the standard contaminate (A.C. Fine Test Dust) is a particulate solid and must be suspended in a fluid for use. There are now a number of organizations marketing certified suspensions. Regardless of the source, the procedure must begin from an established base which is a solution containing a known concentration of A.C. Fine Test Dust.

The concentration to be used for calibration of the threshold levels of the counter must be such that the error due to the minimum reset time requirement is small. This is recognized in the ANSI Standard by placing the saturation procedure first. If the reset error is to be maintained at less than 1% then the value of $N_{\rm D}$ ($V_{\rm S}$ + $QT_{\rm R}$) must be less than .01. If $V_{\rm S}$ or $T_{\rm R}$ is not known, an empirical approach such as a least squares fit to EG (4-9). If the equation is formulated as

$$\frac{N_{\rm C}}{N_{\rm D}} = a e^{-bN_{\rm D}}$$
(5-1)

and repeated observations are made at various fluid concentrations, then

$$\mathbf{b} = \frac{\Sigma \mathbf{x}_{i} \ln \mathbf{y}_{i} - \frac{1}{n} (\Sigma \mathbf{x}_{i}) (\Sigma \ln \mathbf{y}_{i})}{\Sigma \mathbf{x}_{i}^{2} - \frac{1}{n} (\Sigma \mathbf{x}_{i})^{2}}$$
(5-2)

where X_i is the actual count and Y_i the ratio of observed count to actual count for the ith concentration. b is a good approximation to $(V_S + QT_R)$. The coefficient a is given by

$$a = \exp\left[\frac{\sum mY_{i}}{n} - \frac{b\Sigma X_{i}}{n}\right]$$
(5-3)

and is a measure of the calibration error and should be equal to one for a correctly calibrated particle counter. The use of this method is shown in Figure 5, based on data from a HIAC particle counter.

The flow rate used for calibration must be such that frequency response is not a significant factor. Many of the counters in use do not exhibit such a region. It may be necessary for the purpose of developing a practical standard to require only that the flow rate be held within the range .99 to 1.01. Counters with poor frequency response characteristics would then be limited to a very narrow flow rate tolerance.

It is advantageous to determine the ultimate limitations of the counting process, at least approximately, before proceeding to the final calibration. This can be accomplished by testing the null hypothesis that a series of counts of a constant concentration are distributed as the Poisson with λ equal to the mean. Such a test is given by [12]

$$\chi^{2} = \sum_{i=1}^{m} \frac{(f_{i} - e_{i})^{2}}{e_{i}}$$
(5-4)

where

 f_{i} = observed frequency, and

e_i = the expected frequency.

The null hypothesis is rejected if

$$\chi^2 \geq \chi^2_{\alpha,m-2}$$

where

m = number of terms added, and α = confidence level. This test is not very practical for use with very small samples





since the observed frequency for a particular value is almost certain to be 1. An alternative is proposed by Cox and Lewis [11] of the form

$$d = \sum_{i=1}^{k} \frac{(n_{i} - \overline{n})^{2}}{n}$$
(5-5)

where

n = the observed value and

n = the mean value of k observations.

This is essentially a test of the hypothesis that $\lambda = n$ which is precisely the question posed. d is approximately distributed as Chi Square with k - 1 degrees of freedom.

A large sample of particle count data collected by the Fluid Power Research Center was evaluated with this test. The results are summarized in Table I, and the original data are contained in the appendix. The tests are ranked in increasing order of the mean count, and it can be seen that the number of samples which do not fit the Poisson model increases rapidly at the higher levels, where saturation is probably a factor. In the cases where rejection occurs at the lower levels, such as ID No. 328C5, it is obvious that spurious counts were included at the 20 and 30 micrometer level.

If the null hypothesis is rejected the test may be altered as follows. If the principle deviation from Poisson behavior is caused by variation in volume counted, the variance of the volume, which is in fact a variation in λ , may be independent of the volume measured. Thus $\overline{n} = f(N_D, V, X)$ where X is a R.V. with a mean of zero and V is the volume counted. If volume measurement is the dominant error, it should

TABLE I

0.00	tn	MMICS	TESTS	05	TESTIO	P10	TEST20	P20 -	TESTAD	930	TES 740	DAC
603	10	HAICS	12313		123110		123120	NZ V	123130	KJU	123140	NTU
1	329B7	2587.4	1.757		3.8195		2.3514		5.7273		9.0000	*
2	32988	2751.4	9.721	*	4.0153		2.7761		5.2857		2.6667	
3	328C8	3351.6	5.343		1.2203		0.5872		1.0000		9.7647	* 1
4	328C7	3536.8	1.555		0.9795		3.6667		1.2308		0.6667	
5	328C6	3761.0	12.516	*	3.0349		5.8033		4.2222		5.3333	
6	328C 5	3806.2	6.328		4.0000		8 • 2000	*	10.3636	*	3.7143	
7	32986	3841.4	0.292		2.8218		2.2963		5.8182		4.0000	
8	328J4	4061.4	7.804	*	6.6184		3.5000		4.6250		3.0000	
9	328J3	4087.0	123.555	*	7.0153		6.8571		3.2500		1.0000	
10	328J 2	4518.4	13.138	*	1.0433		1.9420		6.3333		5.6667	
11	32985	4523.8	13.950	* '	7.4171		12.9620	*	6.5185		13.8571	*
12	32811	4611.6	4.910		5.4422		2.0000		5.0000		3.5000	
13	328J1	4884.2	9.443	*	3.5967		3.5303		3.0526		2.0000	1
14	328G1	4928.6	12.002	*	6.4031		5.8136		5.1875		11.2727	*
15	328G3	5113.8	17.465	i. *	3.6471		5.1264		3.3750		12.2857	* .
16	328G2	5116.0	28.239	*	0.3870		5.2187		2.7500		3.5.000	
17	32813	5588.4	3.865		4.4248		5.8681		3.1111	-	1.2727	
18	32812	5626.8	2.926		0.1620		3.4468		1.2000		0.4615	· · .
19	328G4	6366.0	48.503	*	2.2613		17.3902	*	8.5882	+	16.0000	*
20	32814	7157.2	18.676	· •	2.2528		15.1613	*	9.3333	*	5.3333	
21	334C 5	25268.4	72.001	* .	3.7023		2.8950		8.0000	*	8,2500	*
22	329B3	26731.8	50.685	*	16.3964	* .	6.4927		9.8394	*	14.4444	*
23	329B4	29094.2	271.623	*	38.2751	*	4.9944		9.7828	*	13.3176	*
24	329B2	29149.0	206.696	*	85.3937	*	53.6018	*	36.0676	*	36.1250	° #
25	329B1	30415.0	208.802	*	50.8339	*	21.8071	*	28.9496	*	38.3701	*
26	328C4	30952.8	193.357	*	46.5396	*	42.9070	÷.	46.9036	*	50.0036	*
27	328G5	31485.0	3.150		10.5952	*	10.0219	*	18.8423	*	19.6364	*
28	328I 5	31521.8	8.336	*	15.6651	*	21.4829	*	24.3861	+	17.6792	*
29	328 1 6	31724.2	253.155	*	25.1213	*	22.3812	* '	52.3867	*	46.7124	*
30	328G7	31993.0	2.315		4.0323		10.9198	*	7.3881		12.6836	*
31	328G6	32097.0	7.941	*	5.2009		6.3798		4.2670		6.2110	
32	334C 8	32332.2	106.773	*	6.1275		5.4041		3.5556		2.3333	•
33	32817	32545.6	113.942	*	25.6854	*	11.4016	*	18.0359	*	26.0885	*
34	328C 3	32799.8	10.900	· *	8.8920	*	26.2195	*	38.4678	*	50.6753	*
35	328.17	33320.8	4.373		7.8756	*	14.5221	+ .	36-9158	*	30.1148	*
36	32818	33483.2	192.770	*	40.2330	*	19.8845		18.6050	*	19.8304	*
37	328.15	34671.4	239.525	*	55.6834	*	28.9261	+	17.7631	*	10-6499	*
38	328.16	34821.6	82.803	*	6. 3777		4.7030		6.5303		2.1862	
39	32868	34908-4	67.713	*	18.3826	. *	8.1226	*	5.5868		8.1719	`*
40	32802	35322.2	15.650	*	22.7216	*	16.5303	*	34.5419	*	23.3919	*
41	328.18	35556.8	29.693	*	14.0220	*	9.6149	*	6.7526		10.3500	*
42	32801	36229.2	74.126	*	7.7686		25.6976	*	33.0524	*	53.5914	*
43	3340.6	41969.4	97.213	*	3, 2513		1.7706		5.4211		5.6667	
44	33407	50803.4	9.983	*	3.6414		4.9988		2.7879		6.0000	
45	33401	59995.4	48,805	*	13,3831	*	4.6624		16-4757	*	16.8702	*
46	33464	68745.0	41.838	*	9.4989	*	5.2160		7.9460	*	12.2215	*
47	33402	72436.9	44.470	*	9.4267	*	1.0205		2 . 2633		12.6742	*
11	33462	04452 0	49 973		4.7179	-	3.6542		9.0407	*	15.4639	*

TEST FOR POISSON FIT OF PARTICLE COUNT DATA

be possible to find a counting volume for which d is not sufficient to reject H_{\bullet} .

It should be noted that the methods used in most volume measuring mechanisms is such that the error is increasing for increasing flow rate. For this reason it is desirable to repeat the flow and reset error analysis after the optimum volume is selected.

The final calibration procedure specified in the A.N.S.I. Specification requires very little alteration. The grand average of a series of particle counts of a correctly calibrated counter converges stochastically on the correct value. The problem for the user is to determine when to stop adjusting the machine. The Shewhart control chart provides this information. However it may be desirable to modify the chart by using the Poisson model, for which the control limits for .90 confidence are given in Chapter IV, specifically

 $N + 0.98 \pm \sqrt{3.84N + 3.69}$

These limits replace those calculated as the setting tolerance. The range chart is replaced by the test for a Poisson distribution. The value of d must not exceed 7.779.

If the counter is properly calibrated and conforms to a Poisson process, the same procedure can be used to provide a confidence interval on counts from an unknown sample. The use of the d statistic provides a continuing measure of procedural control.

When unknown samples are evaluated, another source of error is raised particularly if the sample represents a system which incorporates a filter. The particle count distribution may not follow the log-log² model or even if it does, the slope parameter may be so large that the particles in the size range below the counting level are so many as to create a significant coincidence factor. An analysis of this phenomenon requires the evaluation of a Gamma function with a parameter λ which is in turn a function of particle size. There is no analytical method available for solving this equation but a simulation technique could be applied. Even this approach requires that the particle distribution be known. As a practical alternative it may be advisable to use the lowest size for which the counter is calibrated as a check on the next higher level so that the former is not reported nor used as a reliable count. The optimum size interval for such a practice is probably D to $D/\sqrt{2}$ although this does not preclude errors induced by a drastic change in slope below the lower count level. Although not specifically a part of the calibration and control procedure, such consideration must be given in the practical application of particle counting.

CHAPTER VI

CONCLUSIONS AND RECOMMENDATIONS

An analytical description of the particle counting process has been developed. The models postulated are fundamental and are applicable to any counter using the light reduction technique. Each of the basic sources of error in the particle counting process is evaluated and a mathematical technique is formulated to measure the degree to which that error is contributing to the total uncertainty.

There are four key elements in the calibration and control process:

- (1) The determination or estimation of the concentration saturation curve to locate a suitable operating region.
- (2) The evaluation of the extent of procedural errors affecting the count, especially the effect of volume measurement error.
- (3) The determination of sufficient convergence of the mean count with the true value to justify termination of the calibration procedure.
- (4) The continued surveillance of observed counts to detect the onset of procedural defects, saturation or coincidence error.

Recommendations for Further Development

The present American National Standard has two major weaknesses, both related to the use of the normal distribution model to develop

control limits for a Shewhart Chart. The saturation level is determined by counting at increasing concentrations until the average count falls outside the control limits. If the procedure is begun at too high a concentration, the control limits change as rapidly as the mean, and no out-of-control level is reached.

This procedure should be replaced using the same data to fit Equation (5-1). Saturation could then be defined as the point at which the ratio N_C/N_D falls below an arbitrary value.

A Shewhart Chart is also used to terminate calibration and provide continued control of the counting process. The control limits (called a setting tolerance in the ANSI standard) should be determined with Equation (4-11) with Equation (5-5) used to establish that the counting procedure is properly conducted.

The exact levels of confidence and numerical values are not purely technical questions. There are economic implications which must be considered. The selection of such values is properly a task of standards-formulating committees. A modified standard should be prepared and circulated to users for evaluation and comment. If the technique is generally acceptable and suitable parameters for the tests can be found, the document should be promulgated as a revised standard.

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APPENDIX

		ID=3	828C1		
OBS	MIC5	MICIO	MIC20	MIC30	MIC40
1	35597	6609	2312	982	462
2	36943	6661	2279	881	432
3	36231	6420	2119	874	393
4	37129	6466	2074	750	281
5	35246	6660	2061	829	346
	•	ID=3	328C2		
a a a					
OR2	M105	MICIO	MIC20	M1C30	M1C40
1	35051	6696	2270	987	486
2	35921	6729	2263	933	475
3	35432	6604	2189	918	446
4	35023	6349	2096	845	367
5	35184	6321	2 06 2	761	400
		ID=3	328C3		
085	MIC5	MICIO	MT C20	MTC30	MTC40
005	4105		NI OL O		111040
1	32295	6114	2062	869	389
2	33059	6086	1995	797	380
3	32942	5888	1920	733	299
4	32781	5977	1890	752	323
5	32922	5855	1765	637	232
		ID=3	2804		
GBS	MT C5	MICIO	MTC20	MIC30	MIC40
000			,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,		111010
1	28910	5368	1809	737	333
2	31775	5900	1979	762	362
3	31740	5812	1977	812	390
4	31629	5856	1849	740	345
5	30710	5415	1638	568	222
		ID=3	2805		
0.00		MT CLO	NICOO	N TC 20	N TC (0
082	MICS	MICIU	MIC20	M1030	M1640
1	3808	106	9	1	1
2	3779	94	5	1	1
3	3854	118	16	6	3
4	3897	108	8	3	2
5	3693	94	6	0	0

		ID=	328C6		
OBS	MIC 5	MICIO	MIC20	MIC30	MIC40
1	3610	147	10	3	1
2	3790	143	8	1	Ō
3	3850	168	11	3	0
.4	3864	145	19	5	Ō
5	3691	143	13	6	2
		ID=	32807		
OBS	MIC 5	MICIO	MIC20	MIC30	MIC40
1	3483	154	8	2	1
2	3564	155	13	.3	1
3	3524	162	17	4	2
4	3535	163	11	2	1
5	3578	148	11	2	1
		ID=	328C8	. 1880 - 1980 - 1990 - 1890 - 1990 - 1990 - 1990 - 1990 - 1990	
OBS	MIC5	MICLO	MIC20	MIC30	MIC40
1	3322	160	23	12	5
2	3323	171	20	10	7
3	345 3	166	24	8	0
4	3380	158	20	11	4
5	3280	153	22	9	1
		ID=	328G1		
OBS	MIC5	MICLO	MIC20	MIC30	MIC40
1	4778	162	16	4	2
2	5011	191	30	7	0
3	4822	207	26	10	3
4	4977	178	27	8	6
5	5055	175	19	3	0
		ID=	328G2		
OBS	MIC5	MICLO	MIC20	MIC30	MIC40
1	4859	182	17	3	1
2	4988	178	13	5	1
- 3	5336	178	14	· · · · · · · · · · · · · · · · · · ·	2
4	5202	171	14	4	0
. 5	5195	180	6	1	0

		ID=	32 8G3	، هيوه ملك حك بين جو من من من من م	
OBS	MIC5	MICIO	MIC20	MIC30	MIC40
1	4849	227	21	6	5
2	5170	212	19	2	0
3	5187	219	11	3	1
4	5209	231	22	3	1
5	5154	250	14	2	0
		ID=	328G4		
OBS	MIC 5	MICIO	MIC20	MIC30	MIC40
1	5976	274	30	8	4
2	6228	257	9	3	0
3	6386	250	15	3	0
- 4	6556	263	18	1	0
5	6684	280	10	2	0
		ID=	328G5	80 400 au an an an an an an an an	
OBS	MIC5	MIC10	MI C2 O	MIC30	MIC40
1	31630	5467	1808	756	343
2	31626	5447	1791	746	355
3	31364	5292	1711	708	343
4	31525	5204	1726	664	289
5	31280	5248	1644	619	265
		ID=	328G6		
0 B S	MIC5	MIC10	MIC20	MIC30	MIC40
1	31988	5503	1859	755	362
2	32234	5524	1842	782	364
3	32474	5638	1909	822	420
4	31901	5529	1878	786	371
5	31888	5400	1764	750	369
		I D=	328G7		
DBS	MIC5	MICIO	MIC20	MIC30	MIC40
1	31924	5563	1896	762	371
2	31878	5638	1903	786	393
3	31909	5515	1850	753	360
4	322 08	5448	1812	752	342
5	32046	5479	1730	686	304

		ID=3	328G8		
OBS	MIC5	MICIO	MI C20	MIC30	MIC40
1	33605	5752	1897	761	339
2	34888	6045	1928	803	409
3	35372	6119	2063	824	394
4	35538	61.87	1970	839	402
5	35139	6059	1945	771	376
	-	ID=:	32811	Mili alia alia alia alia alia alia alia	
		- -	_		
OBS	MIC5	MICIO	MIC20	MIC30	MIC40
. 1	4508	108	7	1	1
2	4584	145	11	4	0
3	4713	125	10	2	1
4	4612	127	6	3	2
5	4641	126	9	0	0
		ID=	328I2		
DBS	MIC 5	MICIO	MIC20	MIC30	MIC40
000	11105			111030	111010
1	5715	225	21	5	3
2	5614	228	21	4	3
3	5555	231	15	7	2
4	5666	232	23	4	2
5	5584	232	14	5	3
	· · · · · · · · · · · · · · · · · · ·	ID=	32813		
085	MIC 5	MICLO	MIC20	MIC30	MIC40
1	5553	226	23	4	3
2	5488	223	10	2	2
.3	5582	217	19	2	1
4	5667	240	22	4	3
5	5652	257	17	6	2
		ID=3	32814		
OBS	MIC5	MICLO	MIC20	MIC30	MIC40
1	6888	370	43	10	. 6
2	7146	354	29		2
2	7224	227	16	2	- 1
л 4	7124	364	27	с. К	. 2
5	7394	359	40	14	<u>د</u>
J.	1 2 / 7		TV		7

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		ID=3	2815		
OBS	MIC5	MIC10	MI C20	MIC30	MIC40
1	31207	53 84	1788	718	322
2	31566	5464	1833	776	353
3	31747	5521	1873	810	370
4	31779	53 95	1793	750	337
5	31310	5141	1617	634	270
		ID=3	2816	د حدد هاه وي خية ها كه جيه جيه بي ا	
085	MIC5	MIC10	MIC20	MIC30	MIC40
1	20234	5207	17/0	700	21.2
1	29220	5207	1749	700	513
2	32020	2010	1940	803	401
د ر	32032	2023	1002	810	202
4 5	22482	5270	1600	610	303
2	32240	5570	1000	019	204
		ID=3	2817	ی میلو طاقه کنتر میند متنو هاید خانه کانه ا	-
OBS	MIC5	MIC10	MIC20	MIC30	MIC40
1	30847	5248	1711	652	279
2	33121	5681	1858	756	370
3	33108	5671	1884	793	389
4	32922	5681	1802	736	338
5	32730	5498	1753	680	297
		ID=3	2818		
OBS	MIC5	MICLO	MIC20	MIC30	MIC40
· 1	31222	5185	1701	724	342
2	341.28	5769	1886	758	336
2	342.09	5755	1930	823	403
4	33894	562.0	1788	690	342
5	33963	5616	1751	678	287
		ID=3	28J1		
0.00	MICE	NICIO	NTC20	M1C20	MICAO
0 B 2	MICS	MICIU	MICZU	MICOU	M1640
1	4722	259	27	7	3
2	4841	224	18	5	2
3	5003	251	28	11	5
4	4921	250	30	9	2
5	4934	231	29	6	د .

OBS MIC5 MIC10 MIC20 MIC30 MIC40 1 4487 191 16 5 3 2 4507 173 11 1 0 3 4726 188 11 0 0 4 4464 187 16 3 2 5 4408 185 15 3 1			ID=	328J2		
1 4487 191 16 5 3 2 4507 173 11 1 0 0 3 4726 188 11 0 0 4 4464 187 16 3 2 5 4408 185 15 3 1	OBS	MIC 5	MIC10	MIC20	MIC30	MIC40
1 4437 191 10 3 3 2 4507 173 11 1 0 0 4 4464 187 16 3 2 5 4402 185 15 3 1	,	1407	1.01	17	r	2
2 4507 173 11 1 0 3 4726 188 11 0 0 4 4464 187 16 3 2 5 4408 185 15 3 1 ID=328J3 OBS MIC 5 MIC10 MIC20 MIC30 MIC40 1 3775 170 19 3 1 2 4691 218 17 0 0 3 3893 210 11 2 1	1	4487	191	10	2	3
3 4726 188 11 0 0 4 4464 187 16 3 2 5 4402 185 15 3 1	2	4507	173	11	- 1	0
4 4464 187 16 3 2 5 4408 185 15 3 1 ID=328J3 OBS MIC 5 MIC10 MIC20 MIC30 MIC40 1 3779 170 19 3 1 2 4691 218 17 0 0 3 3893 210 11 2 1 4 4071 193 7 1 1 5 4001 192 16 2 1	5	4126	188	11	0	0
5 4402 185 15 3 1 ID=328J3 OBS MIC 5 MIC 10 MIC 20 MIC 30 MIC 40 1 3775 170 19 3 1 2 4691 218 17 0 0 3 3893 210 11 2 1 4 4071 193 7 1 1 5 4001 192 16 2 1	4	4464	187	16	3	2
	5	4408	185	15	3	1
OBS MIC 5 MIC10 MIC20 MIC30 MIC40 1 3779 170 19 3 1 2 4691 218 17 0 0 3 3893 210 11 2 1 4 4071 193 7 1 1 5 4001 192 16 2 1			ID=	328J3	و هذه هيد جي فقة ولو جيه الي من و	
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4 35488 6759 2365 1051 514 5 35533 6750 2323 928 464 ID=328J6 ID=328J6 0BS MIC5 MIC10 MIC20 MIC30 MIC40 1 33345 6483 2279 997 470 2 34882 6736 2317 1003 493 3 35410 6627 2197 931 484 4 35163 6510 2213 928 471 5 35308 6633 2289 1006 509	3	35798	6658	2265	973	479
5 35533 6750 2323 928 464 ID=328J6 ID=328J6 0BS MIC5 MIC10 MIC20 MIC30 MIC40 1 33345 6483 2279 997 470 2 34882 6736 2317 1003 493 3 35410 6627 2197 931 484 4 35163 6510 2213 928 471 5 35308 6633 2289 1006 509	4	35488	6759	2365	1051	514
ID=328J6 OBS MIC5 MIC10 MIC20 MIC30 MIC40 1 33345 6483 2279 997 470 2 34882 6736 2317 1003 493 3 35410 6627 2197 931 484 4 35163 6510 2213 928 471 5 35308 6633 2289 1006 509	5	35533	6750	2323	928	464
OBS MI C5 MI C10 MI C20 MI C30 MI C40 1 33345 6483 2279 997 470 2 34882 6736 2317 1003 493 3 35410 6627 2197 931 484 4 35163 6510 2213 928 471 5 35308 6633 2289 1006 509			ID=	328J6		
133345648322799974702348826736231710034933354106627219793148443516365102213928471535308663322891006509	OBS	MI C5	MICIO	MIC20	MIC30	MIC40
2348826736231710034933354106627219793148443516365102213928471535308663322891006509	1	33345	6483	2279	997	470
3 35410 6627 2197 931 484 4 35163 6510 2213 928 471 5 35308 6633 2289 1006 509	2	348 82	6736	2317	1003	493
4 35163 6510 2213 928 471 5 35308 6633 2289 1006 509	3	35410	6627	2197	931	484
5 35308 6633 2289 1006 509	4	35163	6510	2213	928	471
	5	35308	6633	2289	1006	509

		ID=3	28J7		
OBS	MIC5	MIC10	MIC20	MIC30	MIC40
1	33090	6251	2157	983	514
2	33443	6339	2189	1000	506
້າ	334 01	6444	2185	961	401
4	32147	6195	1077	919	271
т '5	33523	6175	2110	912	571
2	12752	0115	2110	015	504
		ID=3	28J8		
OBS	MIC5	MIC10	MIC20	MIC30	MIC40
1	34654	6701	2263	958	470
2	35681	6943	2398	1038	538
3	35909	7138	2455	1068	550
4	35842	6892	2310	1025	486
5	35698	6937	2378	1000	482
					•
		ID=3	2981		
08.5	MT C5	MICIO	MIC20	MIC30	MT C40
000	111.00				
1	28238	4895	1704	666	300
2	30783	5428	1782	707	318
3	31516	5549	1773	679	331
4	30850	5152	1637	631	285
5	30688	5147	1554	531	198
		ID=3	2982		
OBS	MIC5	MICIO	MIC20	MIC30	MIC40
1	27723	4749	1443	576	248
2	29980	5316	1699	670	302
3	30804	5350	1710	612	239
4	28611	4793	1401	476	171
5	28627	4685	1632	626	240
		10-3	2002	A	
		10-5	2903		
OBS	MI C5	MICIO	MIC20	MIC30	MIC40
1	25839	4720	1485	552	225
2	26743	4825	1533	554	247
3	26595	4878	1506	552	238
4	27342	5089	1543	576	239
5	27140	4782	1419	480	176

E D	D.	r	D	A C	T	IC	1 6	COUNT	D AT A

	•	ID=3	329B4		
OBS	MIC5	MIC10	MIC20	MIC30	MIC40
1	26832	5023	1609	617	278
2	29204	5389	1693	670	305
3	29742	5500	1709	595	255
4	29065	5270	1613	564	229
5	30628	5611	1650	611	246
		I D=:	329B5	و میں طابع بروں بروہ جی اوراد ہیں مراد مار	
OBS	MIC 5	MICIO	MIC20	MIC30	MIC40
1	4329	171	17	10	8
2	4471	1 71	18	4	3
3	4603	168	10	5	2
4	4577	159	8	2	0
5	463 9	206	26	6	1
		ID=	32986		
OBS	MIC5	MIC10	MIC20	MIC30	MIC40
1	3815	149	10	2	1
2	3843	135	13	4	2
3	3858	124	7	0	ō
4	3853	143	11	1	0
5	3838	145	13	4	2
		ID=	32987		
085	MTC 5	MICIO	MICZO	MICOO	MICAO
003	HICJ	HICIO	HICZU	H 1000	
1	2635	122	18	5	2
2	2605	132	18	8	5
3	2547	150	13	4	3
4	2579	137	13	4	0
5	2571	124	12	1	0
		ID=	32988		
OBS	MIC 5	MIC10	MIC20	MIC30	MIC40
1	2612	123	11	2	2
.2	2748	118	11	0	0
3	2793	139	15	4	2
4	2817	146	12	3	2
5	2787	129	18	5	3

		ID=3	34C1		
			1000 - 1000 - 1000 - 1000 - 1000 - 1000 - 1000 - 1000 - 1000 - 1000 - 1000 - 1000 - 1000 - 1000 - 1000 - 1000 -		
OBS	MIC5	MICIO	MIC20	MIC30	MIC40
,	50107	(9 / /	2071	202	
1	28491	0844	2041	892	440
2	60397	7160	2124	877	398
3	60366	1222	2064	804	369
4	60604	6976	2029	782	365
5	60113	6975	1992	761	341
		ID=3	3402		
000	MICE	41010	NICOO	MT COO	MICLO
082	MICS	MICIU	MIC20	MICSU	M1 C40
1	70918	7776	2184	870	437
2	73155	8091	2197	825	406
3	73035	8114	2134	817	352
4	72639	7922	2174	819	358
		ID=3	34C3		
OBS	M1 C5	MICLO	MIC20	MIC30	MIC40
1	83421	9206	2334	873	422
2	83629	91 37	2317	908	442
3	84324	9361	2380	928	422
4	85886	9387	2265	842	374
5	85009	9268	2278	821	347
		ID=3	3404	-	
OBS	MIC5	MICLO	MIC20	MIC30	MIC40
1	67290	8725	2277	895	462
$\overline{2}$	69417	9113	2394	884	41.8
3	68745	8912	2334	856	382
4	69159	9030	2300	832	407
5	69114	8940	2253	793	372
		ID=3	34C5		
0 B S	MIC5	MIC10	MIC20	MIC30	MIC40
1	24996	826	45	. 9	4
2	24462	776	48	7	3
3	25030	783	37	1	0
4	25635	840	50	5	1
5	26219	806	39	3	0

		ID=3	34C6		
OBS	MIC5	MIC10	MIC20	MIC30	MIC40
1	40632	1624	100	2	0
2	41210	1644	95	9	3
3	42389	1707	108	10	0
4	42551	1705	109	9	1
5	43065	1663	111	8	2
		ID=3	34C7		
OBS	MIC5	MIC10	MIC20	MIC30	MIC40
1	50221	2474	191	14	1
2	51080	2507	156	16	1
3	50770	2413	166	8	0
4	50846	2428	161	14	3
5	51101	2521	157	14	0
		ID=3	340 8		
OBS	MIC5	MICIO	MIC20	MIC30	MIC40
1	31706	2111	153	12	1
2	31020	2055	186	12	1
3	32740	2180	159	6	2
4	33114	2154	182	8	0
5	33081	2197	159	7	2

VITA

Gary Allen Roberts Candidate for the Degree of

Master of Science

Thesis: PARTICLE COUNTING: AN ANALYSIS OF THE PROBLEM OF CALIBRATION AND UNCERTAINTY

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