STATE OF VERMONT
AGENCY OF TRANSPORTATION
MATERIALS DIVISION

INTERIM REPORT

STATISTICAL STUDIES OF LOSS OF FINES IN THE EXTRACTION OF BITUMINOUS MIXTURES

REPORT NO. 78-10

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ABSTRACT

The presence of fine particulate matter in hot mix affects the analysis for asphalt content. This study was undertaken to determine how uniformly the analysis is affected and to compare the deviations from the job aim of the slip % asphalt and the extracted % asphalt with and without correction for fines. A comparison of laboratory and field analyses was also undertaken.

The results show that the fines content of the extracts is not uniform throughout a job and that the slip value is the closest approximation to the job aim. It was also shown that there is no experimentally significant difference between the % asphalt without correction for fines, as determined in the laboratory and in the field.

INTRODUCTION

Environmental concerns regarding particulate emission into the atmosphere have led to restrictions in the operation of hot-mix plants. The use of a bag-house to collect these emissions and return them to the mix has resulted in questionable effects on both the mix and the testing thereof. Two problems have been noted: 1 - Inconsistant mixes due to "slugs" of fines being introduced occasionally; and 2 - Loss of fines through the extraction filter in testing, resulting in erroneous values for % asphalt.

The difficulty with inconsistent mixes is mechanical in nature. There appears to be no economically feasible alternative for disposal of the collected fines other than by returning them to the mix. Since the feed rate of aggregates to a plant is not always uniform, variations in the collection of fines force the producer to dispose of them inconsistently. He must rid the bag-house of all fines at the end of each production sequence or suffer the possibility of damage to the system via moisture.

The loss of fines in testing has caused more concern in recent years, due in part to quality assurance specifications which contain provisions for penalty payment. Fine particles escaping the filter of a centrifuge extractor are recorded as loss of asphalt. The computed asphalt content is therefore higher than the actual asphalt content of the mix.

During the 1977 paving season, a correction for fines was established for each mix produced by recovering fines by centrifugation of the collected extract. This was done for several samples at the beginning of a project and repeated periodically throughout the course of the contract. Inconsistent results caused the validity of a constant correction factor for an entire job to be questioned. This report attempts to deal with that question by statistical analysis of the fines recovered from all samples taken during a job.

Other questions examined are: 1 - How closely the corrected value of % asphalt agrees with the value on the slip and with the job aim; and 2 - How closely analyses done by different operators using different extractors agree with each other.

PROCEDURE

A series of 445 hot-mix samples was extracted with xylene according to AASHTO T164, Method A. Of these, 35 split samples were taken in 1976 and extracted under both laboratory and field conditions. The remaining 410 were field samples taken in 1977.

Fines were determined on 88 of the 1977 samples by centrifugation. These comprised all of the samples from the Addison-Bridport job (25) plus three samples each from several other jobs.

The data were analysed statistically by established procedures. The "t" test was used to determine whether a mean differed significantly from a given value, and the "χ^2" test was used to determine how well a set of points fit a predetermined curve.

RESULTS

1. COMPARISON OF LABORATORY AND FIELD ANALYSIS

Values for asphalt content without correction for fines of the 35 split samples taken in 1976 were obtained in both the laboratory and the field. Table I gives the frequency distribution of the difference between laboratory and field results. Figure 1 is a graph of the frequency distribution with the entire number within an interval plotted at the midpoint. Both show a peak around 0 with a slight skew to the right, i.e., the laboratory results appear to be a little higher than the field results.

The difference between laboratory and field values averaged + 0.08% with a standard deviation of 0.19%. Application of the "t" test showed that this is a mathematically significant deviation from zero at the 98% confidence level. However, the difference is experimentally insignificant, as the most probable error in a single measurement due to weighings is 0.10% to 0.15% (see Appendix), making the combined most probable error in the difference 0.20 to 0.30% due to errors in weighing alone. This does not include possible mechanical losses. According to AASHTO T194, the reproducibility of the procedure is 0.56% asphalt content.

Table II gives the frequency of the absolute deviation and the cumulative % as a function of absolute deviation. Over 90% of the values lie within the most probable error due to weighing for the combined measurements. This would imply that there is no experimentally significant difference between results obtained in the laboratory and those obtained in the field. It would further imply that any differences obtained are due to inherent limitations in the procedure, rather than differences among operators or instruments.

TABLE I

FREQUENCY DISTRIBUTION OF DIFFERENCE BETWEEN
LABORATORY AND FIELD RESULTS
(SPLIT SAMPLES)

Lab-Field Results			Number
-.30	-	-.21	2
-.20	-	-.11	4
-.10	-	-.01	8
.00	-	.09	8
.10	-	.19	4
.20	-	.29	5
.30	-	.39	3
.40	-	.49	2
.50	-	.59	2

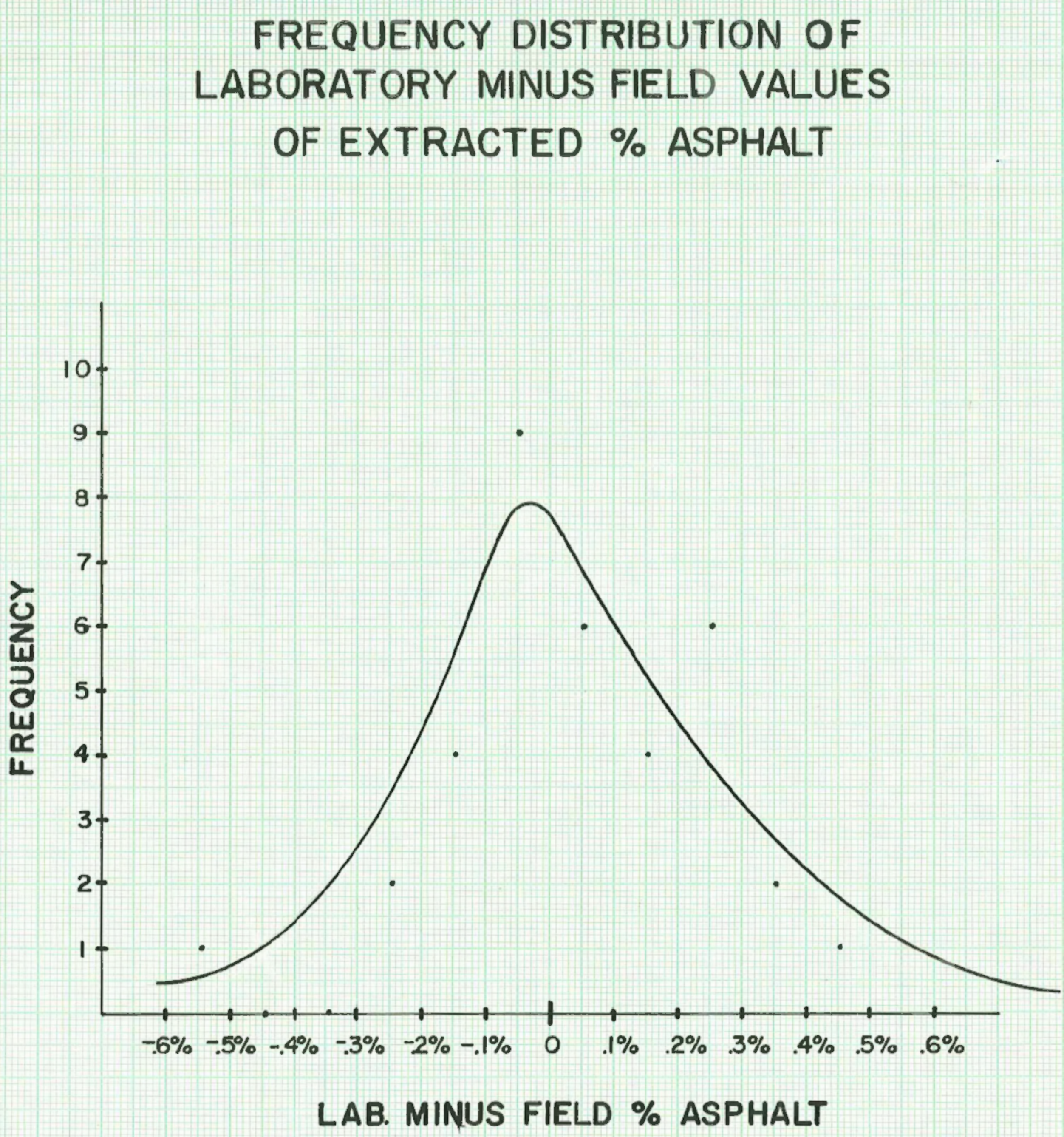
FREQUENCY DISTRIBUTION OF
LABORATORY MINUS FIELD VALUES
OF EXTRACTED % ASPHALT
FREQUENCY
10
9
8
7
6
5
4
3
2
1
-.6% -.5% -.4% -.3% -.2% -.1% 0 .1% .2% .3% .4% .5% .6%
LAB. MINUS FIELD % ASPHALT

FIG. I

TABLE II

ABSOLUTE VALUE OF LABORATORY - FIELD % ASPHALT

Deviation	Number	Cumulative Number	Cumulative %
0 - .04	8	8	22.9
.05 - .09	7	15	42.9
.10 - .14	3	18	51.4
.15 - .19	5	23	65.7
.20 - .24	2	25	71.4
.25 - .29	5	30	85.7
.30 - .34	2	32	91.4
.35 - .39	1	33	94.3
.40 - .44	1	34	97.1
.45 - .49	0	34	97.1
.50 - .54	1	35	100.0

2. ASSIGNMENT OF A CONSTANT % FINES FOR AN ENTIRE JOB

The percentage of fines was determined by centrifugation on extracts from all 25 samples taken from the 1977 Addison-Bridport job. A mean value of 0.56% with a standard deviation of 0.17% was obtained. Individual values ranged from 0.29% to 1.01%. Table III gives the frequency distribution and Figure 2 a plot of the frequency distribution of % fines obtained. For clarity, all values within each interval of 0.1 are lumped at the midpoint. It may be seen that the frequency distribution has a peak in the interval of 0.40 to 0.49 and that it is slightly skewed to the right, causing the mean to be higher than the mode.

On the basis of the first few samples, a constant correction factor of 0.6% was assigned to the project, a value quite close to the final average. In spite of the range of 0.72%, all % fines are within the acceptable tolerance of 0.4% when a factor of either 0.6% or 0.7% is used. (The value below 0.3 is 0.29, which would round upward.)

The corrected asphalt content, based on actual determination of % fines, was compared with the values obtained using constant correction factors of 0.4%, 0.5%, 0.6%, 0.7% and 0.8%. The results are given in Table IV. The average of the absolute value of the deviation from the job aim was 0.31%, 0.24%, 0.21%, 0.20% and 0.22% using factors of 0.4%, 0.5%, 0.6%, 0.7% and 0.8% respectively. The average absolute value of the deviation from the job aim of the individually corrected values was 0.16. The differences between the deviation from job aim for the individually corrected values and those obtained with factors

TABLE III

FREQUENCY DISTRIBUTION OF % FINES
IN EXTRACTS FROM ADDISON-BRIDPORT JOB

% Fines	Number
.00 - .09	0
.10 - .19	0
.20 - .29	1
.30 - .39	2
.40 - .49	8
.50 - .59	5
.60 - .69	5
.70 - .79	2
.80 - .89	1
.90 - .99	1
1.00 - 1.09	1

FREQUENCY DISTRIBUTION OF % FINES IN SAMPLES FROM 1977 ADDISON-BRIDPORT PROJECT

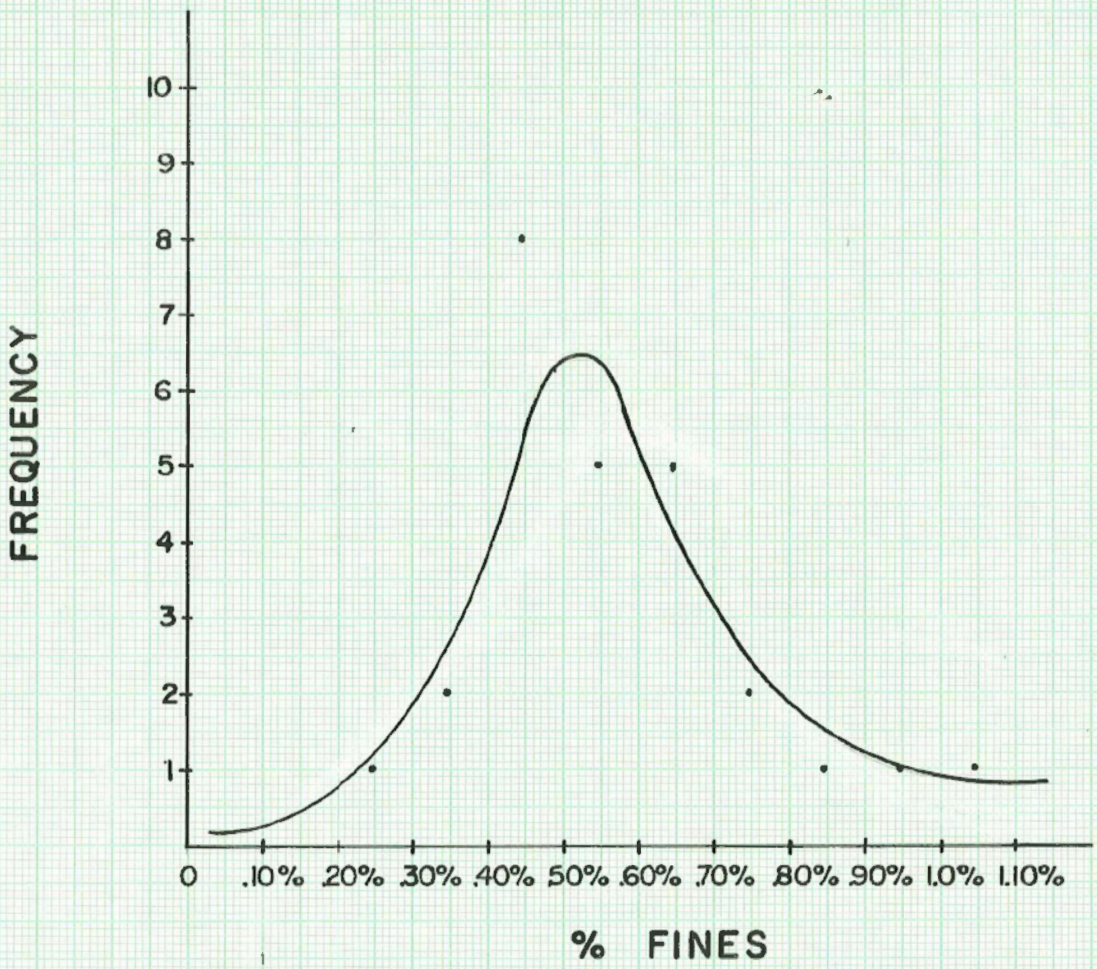

FIG. 2

TABLE IV

COMPARISON OF DEVIATIONS FROM JOB AIM OF ASPHALT CONTENT (%) OBTAINED BY INDIVIDUAL ANALYSIS VS. VARIOUS CORRECTION FACTORS

Individual Analysis	Factors				
Extract - % Fines - Job Aim	Extract - 0.4 -Job Aim	Extract - 0.5 -Job Aim	Extract - 0.6 -Job Aim	Extract - 0.7 -Job Aim	Extract - 0.8 -Job Aim
.43*	1.04*	.94*	.84*	.74*	.64*
.31	.52*	.42*	.32	.22	.12
-.11	- .03	- .13	- .23	- .33	- .43*
.16	.56*	.46*	.36	.26	.16
.17	.48*	.38	.28	.18	.08
.00	.53*	.43*	.33	.23	.13
-.02	- .02	- .12	- .22	- .32	- .42*
.27	.43*	.33	.23	.13	.03
.34	.33	.23	.13	.03	- .07
.22	.21	.11	.01	- .09	- .19
.12	.22	.12	.02	- .08	- .18
.17	.36	.26	.16	.06	- .04
.36	.65*	.55*	.45*	.35	.25
.03	.29	.19	.09	- .01	- .11
.20	.29	.19	.09	- .01	- .11
-.06	.09	- .01	- .11	- .21	- .31
.28	.17	.07	- .03	- .13	- .23
-.06	.27	.17	.07	- .03	- .13
.02	.06	- .04	- .14	- .24	- .34
.20	.30	.20	.10	.00	- .10
-.06	- .09	- .19	- .29	- .39	- .49*
-.02	- .02	- .12	- .22	- .32	- .42*
.07	.13	.03	- .07	- .17	- .27
.28	.49*	.39	.29	.19	.09
-.03	.13	.03	- .07	- .17	- .27
Average of absolute value of deviation					
.16	.31	.20	.21	.20	.22

*Values greater than 0.4 from job aim (apparent failures) of 0.5%, 0.6%, 0.7% and 0.8% are, at best, of only marginal significance. It is not possible to distinguish between the values of the factor on this basis.

Another point to examine is the possible introduction of spurious failures, i.e., how many samples that would have passed had a constant factor not been used would fail when the factor is used. The starred values in Table IV are those samples that would be rejected because they are more than 0.4% away from the job aim. One sample failed when individually corrected values were used. In addition to that 7, 4, 1, 0, and 4 spurious failures occurred with factors of 0.4%, 0.5%, 0.6%, 0.7% and 0.8%. On this basis, it would appear as though a factor of 0.7% would be the best factor to fit the data.

Yet how can a factor of 0.7% be considered representative of all samples taken when only 5 of 25 samples are at or above 0.7%, when the mean is 0.6%, and the mode is 0.4%? Furthermore, given the actual distribution of samples, the average of the first few samples was far more likely to have been 0.5% than 0.6%, which would have resulted in four spurious failures and an unknown number of spurious passes.

One may conclude that assigning a constant % fines on the basis of the first few samples would result in incorrectly passing or failing a significant number of samples, and that the assignment of a single meaningful factor is not possible even in retrospect.

3. COMPARISON OF UNCORRECTED % ASPHALT AND SLIP VALUES WITH JOB AIM

Analysis of 410 samples for which the uncorrected % asphalt, slip % asphalt, and job aim are available shows that the values obtained by extraction without correction for fines are significantly different from the job aim. This is an expected result due to the known fines content of the extract. The differences between extracted % asphalt and job aim average 0.20% with a standard deviation of 0.31%. Application of the test shows this average to be different from 0 at the 99.5% confidence level.

Table V gives the frequency distribution of the difference between extracted % asphalt and job aim. Figure 3 is a plot of the frequency distribution in which all values within an interval are plotted at the midpoint. Both the table and the figure show that the data are somewhat skewed to the right, presumably due to the fines content. Application of the "χ^2" test shows that a Gaussian distribution about the mean can be rejected at the 99.5% confidence level, which implies that the differences are not due to random experimental error in the extraction process. This is also consistent with the bias due to fines content. The extracted value of the asphalt content, without correction, is therefore, not a good approximation to the job aim.

The difference between slip value and job aim averaged -0.02% with a standard deviation of 0.057%. The "t" test shows that the average is different from 0 at the 99.5% confidence level. The frequency distribution of the values of slip - job aim is given in Table VI, and a plot of this distribution is given in Figure 4. The values are peaked very sharply about 0. Therefore, for greater clarity, the data are presented as a cumulative deviation from 0 in units of 0.01 in Table VII. It may be seen that almost 95% of the differences are within $\pm$ .10 of 0. Application of the "χ^2" test shows that a Gaussian distribution can be rejected at the 99.5% confidence level because of the sharp peak about zero and the skew to the left. This would imply that deviations from zero are not due to random experimental fluctuations and that the slip value can be set or controlled to agree with the pre-set job aim to a high degree of precision.

TABLE V

FREQUENCY DISTRIBUTION OF EXTRACTED AC-JOB AIM

Range			Frequency
- .90	-	- .81	1
- .80	-	- .71	0
- .70	-	- .61	0
- .60	-	- .51	1
- .50	-	- .41	2
- .40	-	- .31	1
- .30	-	- .21	13
- .20	-	- .11	34
- .10	-	- .01	56
.00	-	.09	62
.10	-	.19	62
.20	-	.29	44
.30	-	.39	35
.40	-	.49	24
.50	-	.59	24
.60	-	.69	23
.70	-	.79	11
.80	-	.89	4
.90	-	.99	8
1.00	-	1.09	2
1.10	-	1.19	1
1.20	-	1.29	0
1.30	-	1.39	0
1.40	-	1.49	2

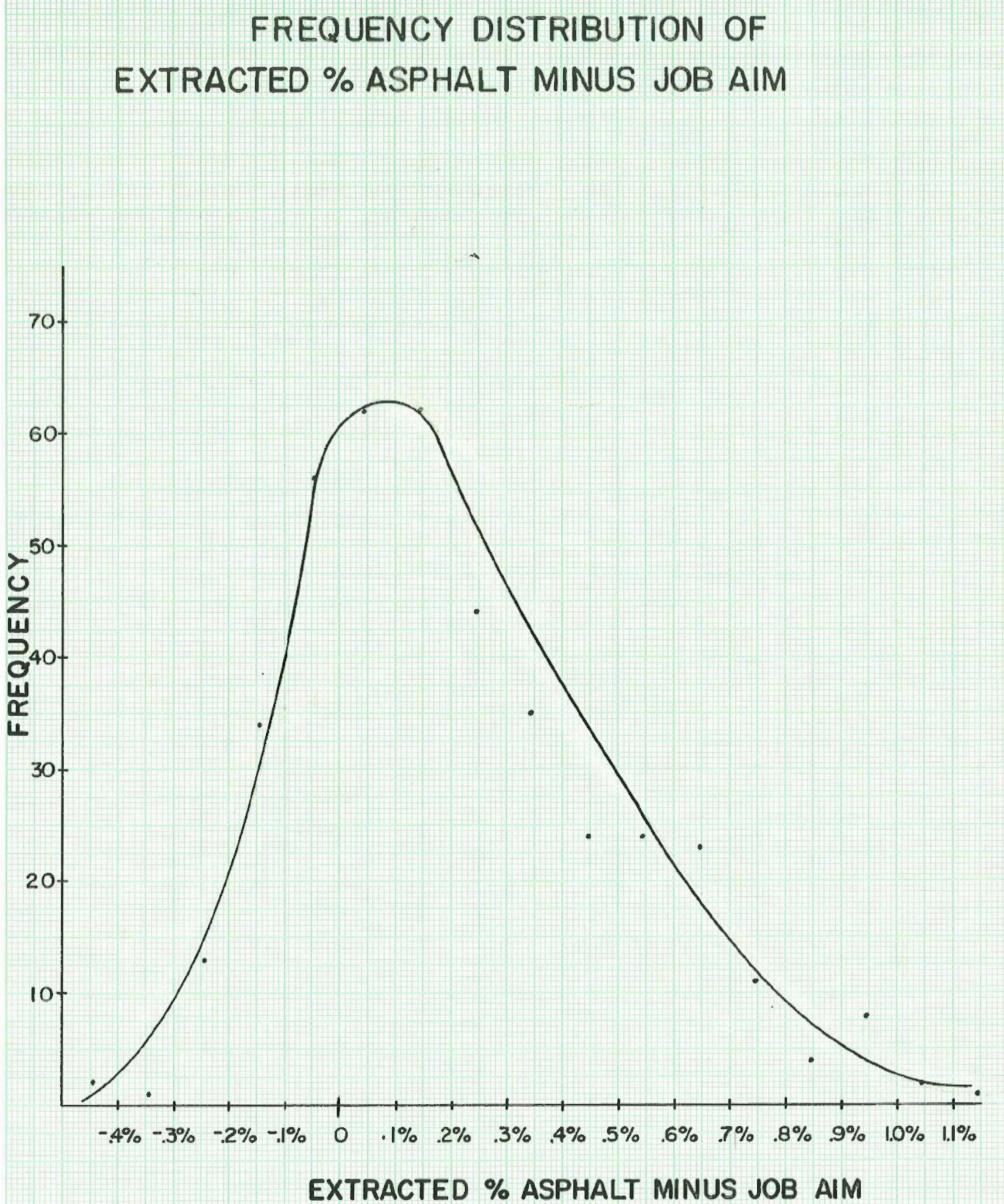
FREQUENCY DISTRIBUTION OF
EXTRACTED % ASPHALT MINUS JOB AIM
FREQUENCY
70
60
50
40
30
20
10
-.4% -.3% -.2% -.1% 0 .1% .2% .3% .4% .5% .6% .7% .8% .9% 1.0% 1.1%
EXTRACTED % ASPHALT MINUS JOB AIM

FIG. 3

TABLE VI

FREQUENCY DISTRIBUTION OF SLIP % ASPHALT - JOB AIM

Range	Number
- .66 - -.51	1
- .50 - -.41	0
- .40 - -.31	1
- .30 - -.21	4
- .20 - -.11	17
- .10 - -.01	220
.00 - .09	163
.10 - .19	4

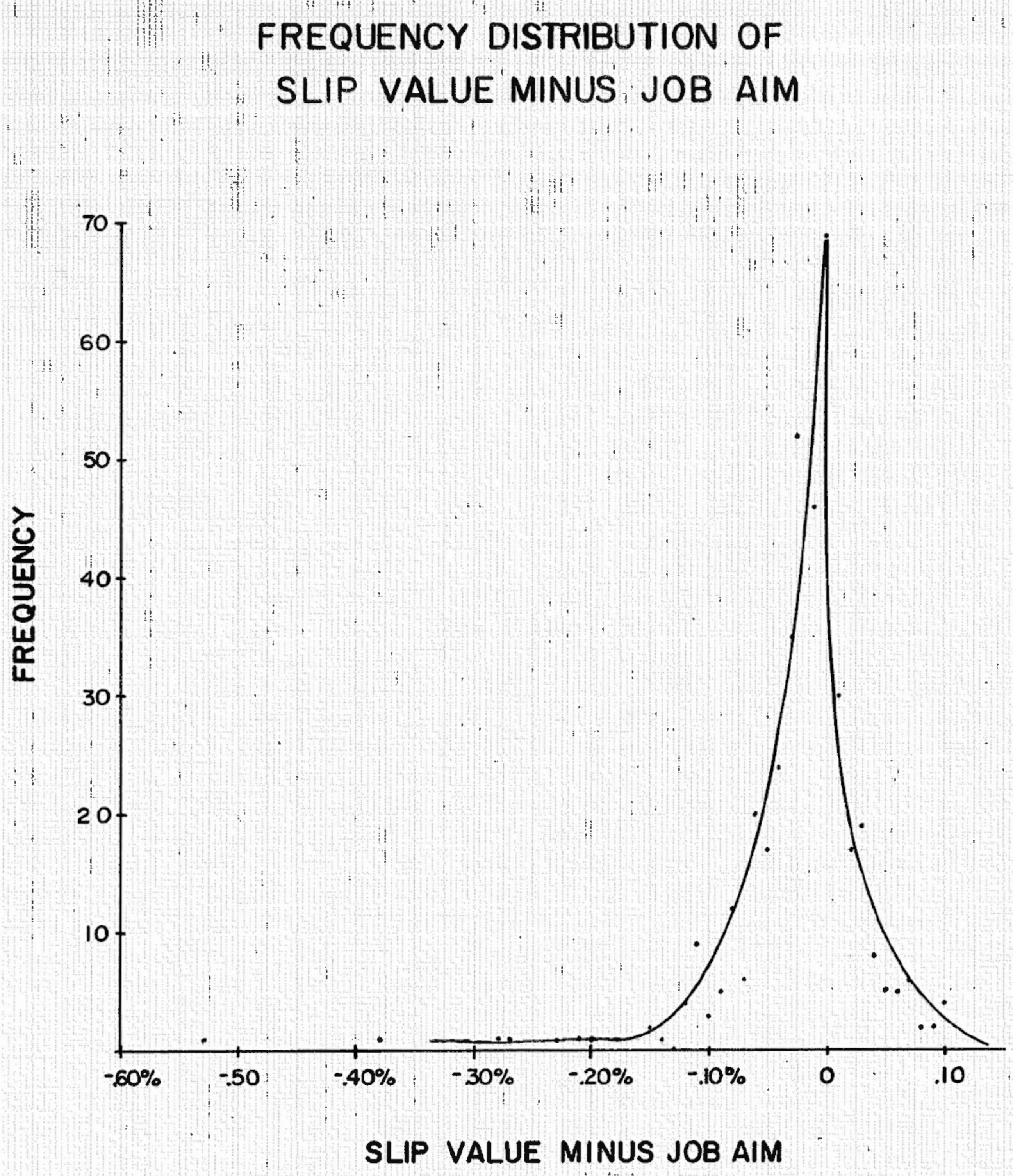
FREQUENCY DISTRIBUTION OF
SLIP VALUE MINUS JOB AIM
FREQUENCY
70
60
50
40
30
20
10
-.60%
-.50
-.40%
-.30%
-.20%
-.10%
0
.10
SLIP VALUE MINUS JOB AIM

FIG. 4

TABLE VII

DEVIATION FROM 0 OF SLIP % ASPHALT -JOB AIM

Deviation	Number	%	Cumulative Number	Cumulative %
0	69	16.8	69	16.8
± .01	76	18.5	145	35.3
± .02	69	16.8	214	52.1
± .03	54	13.2	268	65.3
± .04	32	7.8	300	73.1
± .05	22	5.4	322	78.5
± .06	25	6.1	347	84.6
± .07	12	2.9	359	87.5
± .08	14	3.4	373	90.9
± .09	7	1.7	380	92.6
± .10	7	1.7	387	94.3
> ± .10	23	5.6	410	99.9*

*Deviation from 100% due to round-off error.

4. COMPARISON OF CORRECTED % ASPHALT WITH JOB AIM

Percentage of fines was determined by centrifugation for 88 of the 410 samples discussed in the previous section. These samples represented all of those taken at the Addison-Bridport job (25) and three each from several other jobs. In the previous section, it has been shown that the slip value is very close to the job aim, and that the value obtained by extraction without correction for fines is not a good approximation to the job aim. This section compares the closeness to the job aim of the corrected values with that of the extracted and slip values.

The difference between corrected value and job aim averaged $\pm$ 0.06% with a standard deviation of 0.19%. Application of the "t" test shows this difference to be significant at the 99% level. The "χ^2" test shows that a normal distribution about the mean can not be rejected. However, the sample size is too small and the value of "χ^2" too large for the normal distribution to be accepted at a high confidence level.

Examination of a plot of the frequency distribution (Figure 5) bears this out. The graph shows a slight skew to the right, i.e., the corrected values tend to be a little higher than the job aim. This is consistent with the fact that all major experimental errors in the determination of fines tend to make the corrected % asphalt high due to loss of fines in the extraction apparatus and incomplete removal of fines from the extract by centrifugation.

The data were examined to determine whether the corrected values yield a better approximation to the job aim than the slip value.

FREQUENCY DISTRIBUTION OF CORRECTED % ASPHALT MINUS JOB AIM

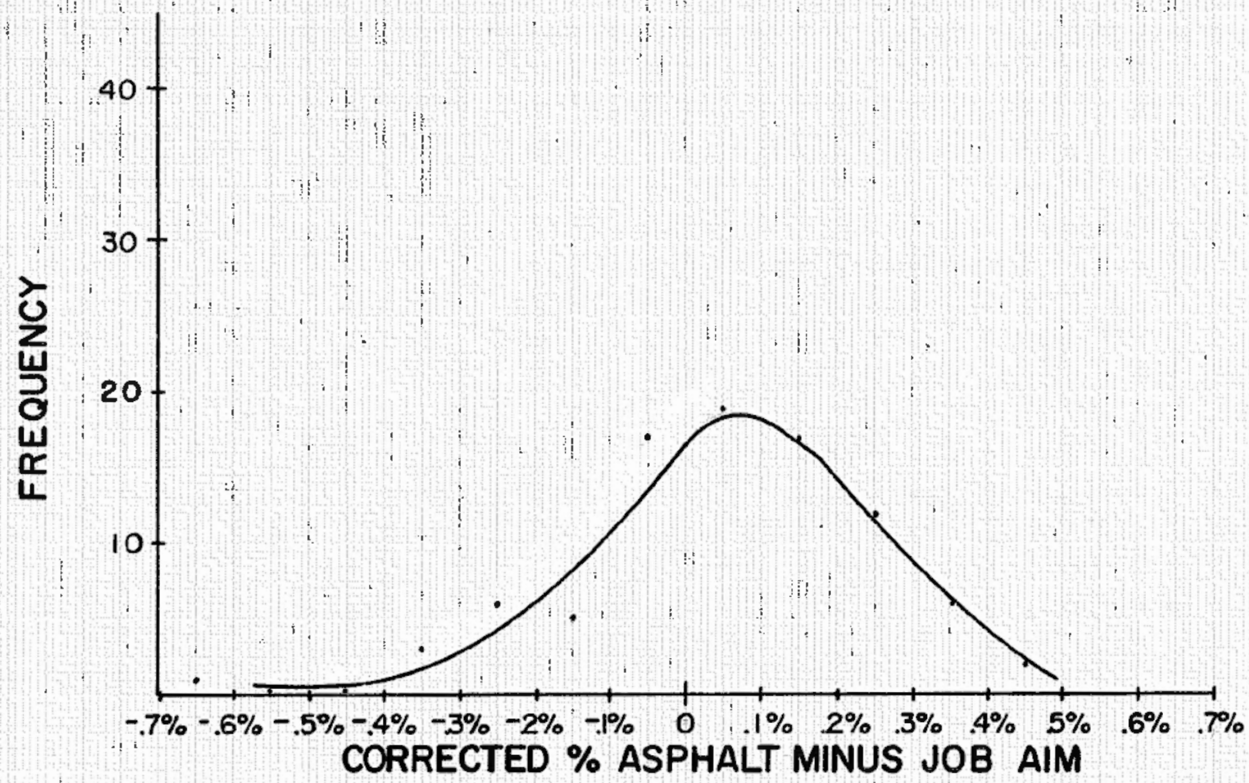

FIG 5

Table VIII gives the number of samples within a given absolute value range of the job aim for the extracted, corrected, and slip values. It also gives the cumulative number and cumulative percentage within a given level of deviation from the job aim. It may be seen that the corrected values are closer to the job aim than the uncorrected values, but that the slip values are far closer than either. One may conclude that the slip value is the closest available approximation to the job aim.

TABLE VIII

DEVIATION AND CUMULATIVE DEVIATION FROM JOB AIM
OF EXTRACTED % ASPHALT, CORRECTED % ASPHALT, AND SLIP % ASPHALT

Absolute Value of Deviation	Number Ext.-Aim	Number Corr.-Aim	Number Slip-Aim	Cum. # Extr.-Aim	Cum. # Corr.-Aim	Cum. # Slip Aim
.00 - .04	1 (1.1)	16 (18.2)	55 (62.5)	1 (1.1)	16 (18.2)	55 (62.5)
.05 - .09	4 (4.5)	19 (21.5)	24 (27.3)	5 (5.7)	35 (39.8)	79 (89.8)
.10 - .14	4 (4.5)	11 (12.5)	6 (6.8)	9 (10.2)	46 (52.3)	85 (96.6)
.15 - .19	8 (9.1)	10 (11.4)	1 (1.1)	17 (19.3)	56 (63.6)	86 (97.7)
.20 - .24	7 (8.0)	11 (12.5)	1 (1.1)	24 (27.3)	67 (76.1)	87 (98.9)
.25 - .29	5 (5.7)	9 (10.2)	0	29 (33.0)	76 (86.4)	87 (98.9)
.30 - .34	7 (8.0)	5 (5.7)	0	36 (40.9)	81 (92.0)	87 (98.9)
.35 - .39	8 (9.1)	4 (4.5)	1 (1.1)	44 (50.0)	85 (96.6)	88 (100.0)
.40 - .44	4 (4.5)	1 (1.1)		48 (54.5)	86 (97.7)	
.45 - .49	5 (5.7)	1 (1.1)		53 (60.2)	87 (98.9)	
.50 - .54	4 (4.5)	0		57 (64.8)	87 (98.9)	
.55 - .59	4 (4.5)	0		61 (69.3)	87 (98.9)	
.60 - .64	6 (6.8)	1 (1.1)		67 (76.1)	88 (100.0)	
.65 - .69	6 (6.8)			73 (83.0)		
.70 - .74	5 (5.7)			78 (88.6)		
.75 - .79	1 (1.1)			79 (89.8)		
.80 - .84	0			79 (89.8)		
.85 - .89	3 (3.4)			82 (93.2)		
.90 - .94	3 (3.4)			85 (96.6)		
.95 - .99	1 (1.1)			86 (97.7)		
1.00 and up	2 (2.3)			88 (100.0)		

CONCLUSIONS AND RECOMMENDATIONS

1. It has been determined that there is no experimentally significant difference between values of extracted % asphalt obtained in the laboratory and in the field. The results are, however, indicative that slightly higher values are obtained in the laboratory. Variations are due to the procedure and not to the multiplicity of instruments and operators.

2. It has also been determined that the assignment of a single value to represent the % fines of all the extracts from a job is of questionable validity. This conclusion is based on data from a single job. Data from a number of other jobs would be needed in order to draw more definitive conclusions.

3. Comparison of the extracted, individually corrected, and slip values for asphalt content has shown that the slip value is by far the closest approximation to the job aim. The corrected values are not as close as the slip values, but are much closer than the uncorrected values.

It is therefore recommended that:

1. The practice of assigning a constant % fines for an entire job be discontinued,

2. the slip % asphalt be adopted as the most nearly correct value,

3. the asphalt content be spot-checked by determining % fines and subtracting from extracted % asphalt, and

4. investigation be initiated into a more accurate procedure for determining asphalt content.

APPENDIX

CALCULATION OF MOST PROBABLE ERROR

If a quantity, y, is calculated as the result of experimental measurements X_1, X_2, .., then the most probable error in y is given by:

$$\delta y = \left[\Sigma \left(\frac{\partial y}{\partial x_i} \right)^2 \delta x_i^2 \right]^{1/2},$$

where δy is the most probable error,

X_i is an experimental measurement, and the summation is carried out over all experimental measurements appearing in the equation for y.

The extracted % asphalt (AC) is given by:

$$\% \text{ AC} = \frac{(W-R)}{W} \times 100,$$

where W is the weight of sample and R is the weight of the residue after extraction.

After taking derivatives and simplifying the most probable error is given by:

$$\delta \% AC = \frac{R}{W} \left[\left(\frac{\delta W}{W} \right)^2 + \left(\frac{\delta R}{R} \right)^2 \right]^{1/2} \times 100 .$$

Both W and R are 1 gm, the readibility of the balance used.

Typical values for R and W give a most probable error of 0.10 to 0.15% in the extracted asphalt content due to the weighings in the extraction procedure, before correction for fines. This does not include possible mechanical losses in the filter or extractor or other errors.