Calibration and Reliability of a Nuclear Asphalt Content Gauge

Final Report
for
MLR-87-2

April 1987

Highway Division
Iowa Department of Transportation

Disclaimer

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by

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ABSTRACT

Based on results of an evaluation performed during the winter of 1985-86, six Troxler 3241-B Asphalt Content Gauges were purchased for District use in monitoring project asphalt contents. Use of these gauges will help reduce the need for chemical based extractions. Effective use of the gauges depends on the accurate preparation and transfer of project mix calibrations from the Central Lab to the Districts.

The objective of this project was to evaluate the precision and accuracy of a gauge in determining asphalt contents and to develop a mix calibration transfer procedure for implementation during the 1987 construction. The first part of the study was accomplished by preparing mix calibrations in the Central Lab gauge and taking multiple measurements of a sample with known asphalt content. The second part was accomplished by preparing transfer pans, obtaining count data on the pans using each gauge, and transferring calibrations from one gauge to another through the use of calibration transfer equations. The transferred calibrations were tested by measuring samples with a known asphalt content.

The study established that the Troxler 3241-B Asphalt Content Gauge yields results of acceptable accuracy and precision as evidenced by a standard deviation of 0.04% asphalt content on multiple measurements of the same sample. The calibration transfer procedure proved feasible and resulted in the calibration transfer portion of Materials I.M. 335 - Method of Test For Determining the Asphalt Content of Bituminous Mixtures by the Nuclear Method.

Calibration and Reliability of a Nuclear Asphalt Content Gauge

INTRODUCTION

Testing performed by the Central Materials Lab Bituminous Section during the winter of 1985-86 (MLR-85-11) investigated the accuracy of the Troxler 3241-B Nuclear Asphalt Content Gauge in determining asphalt cement (AC) content of mixes produced with different asphalt sources and grades and with different aggregate sources and blends. It was concluded from the study that the Troxler 3241-B Gauge provides a rapid, safe method of determining bitumen content with precision well within limits specified in ASTM D2172-81 (Quantitative Extraction of Bitumen from Bituminous Paving Mixtures).

Based on the results of this initial study, the Troxler 3241-B Asphalt Content Gauge will be used by the Central Lab and the Districts during the 1987 construction season to determine the AC content of bituminous paving mixtures. Use of these gauges, along with the shift from extracted to cold feed gradations for project gradation control, will help reduce the need for extraction procedures involving hazardous solvents.

PROBLEM STATEMENT

For the 1987 construction season, the Central Lab mix design procedure will include, for each mix, the development of a two or three point nuclear gauge calibration curve defined by slope, intercept, calibration temperature (deg. F.), and sample weight. The mix calibration data will be used in District asphalt content gauges for project monitoring purposes.

When a calibration developed in one gauge is transferred to another gauge for sample or calibration testing, the calibration slope and intercept must be appropriately adjusted to compensate for differences in measurement characteristics inherent to each individual gauge and its operating environment. Effective Iowa DOT use of the Troxler 3241-B gauges will depend upon the reliable transfer of calibration data from one gauge to another and on the ability of the gauges to repeatedly determine AC contents based on a particular calibration.

OBJECTIVE

The objective of this project is twofold: (1) To establish that the 3241-B gauges can repeatedly determine AC content of a mix with acceptable accuracy based on the specific calibration of that mix, (2) to develop an accurate and reliable mix calibration transfer procedure which can be implemented for the 1987 construction season.

PROCEDURE

Part 1: A three pan (4.0%, 5.0%, and 6.0%) mix calibration was determined in the lab gauge. This calibration was then tested in the lab gauge at different times and in different locations, with multiple measurements of the same sample taken to determine accuracy and repeatability of the results produced by recommended calibration and measurement procedures.

Part II: The concept behind calibration transfers is that changes in calibration slope and intercept from one gauge to another on the same mix are proportional to the difference in counts from one gauge to another on the same transfer pans.

The calibration transfer concept is illustrated by the following equations as adapted from the Troxler Instruction Manual.

1. $S_{field} = S_{cal} \dfrac{(C_{lab2} - C_{lab1})}{(C_{field2} - C_{field1})}$

and

2. $I_{field} = (S_{cal} * C_{lab2}) - (S_{field} * C_{field2}) + I_{cal}$

Where:

S_{cal} = Calibration slope determined by lab gauge
I_{cal} = Calibration intercept determined by lab gauge

S_{field} = New calibration slope to be used in field gauge
I_{field} = New calibration intercept to be used in field gauge

C_{lab1} = Lab gauge transfer pan counts on lower AC content
C_{lab2} = Lab gauge transfer pan counts on higher AC content

To use these two equations, twenty one-minute counts are taken and averaged for each transfer pan with the gauge in the stability test mode. The transfer pans each contained 7100 grams of asphalt concrete mix prepared with the same aggregate proportions and having asphalt contents of 4.0, 5.0, 6.0, 7.0, and 8.0 percent. Each pan was sealed with the same amount (approximately 60 grams) of epoxy to prevent moisture absorption which would be detected as asphalt cement by the gauge. Stability test mode counts were taken on each of the five pans by all seven gauges at the same location in the lab to establish the "count" data necessary to test the calibration transfer procedure.

With the transfer pan counts established, a 100% gravel mix was then prepared and a calibration established using 5.0%, 6.0% and 7.0% AC contents. The calibration was transferred to other gauges using the above equations, and a sample with known asphalt content was measured in the gauge. This was repeated with different gauges and under varying conditions to establish the validity of the calibration transfer procedure, and to investigate the use of various transfer pan combinations.

RESULTS

Repeatability and Accuracy:

On December 12, 1986, the following three pan mix calibration was determined in the lab gauge.

Slope x 1000	= 3.97
Intercept	= -4.34
Temperature	= 250°F
Background Count	= 1647

The following day, to test repeatability and accuracy of the gauge and of the above calibration, twenty four-minute measure counts were taken of a sample of the 5.0% mix used in the calibration. The calibration data was entered, and a sixteen-minute background count of 1650 was determined prior to taking the measure counts. Gauge location for measure counts and for mix calibration remained the same. Twenty measurements of the 5% sample produced the following results.

Table 1

% AC	% AC	
5.00	5.00	
5.02	4.91	
5.01	4.95	
5.00	5.00	
4.93	4.96	
5.01	4.97	Ave. = 4.98%
5.00	4.99	Std. Dev. = 0.04
4.93	5.02	
4.98	4.97	
4.94	5.02	

The gauge was then moved to a new location in the north end of the Bituminous Section and the calibration data re-entered. The background count from the previous location (1650) was entered to determine the effect of using an incorrect background count on the accuracy of sample measurements. Five four-minute counts were taken on a 5.0% sample with the following results:

Table 2

% AC	
4.72	
4.72	Ave. = 4.69%
4.70	Std. Dev. = 0.03
4.67	
4.64	

At this same location, a new sixteen-minute background count was taken (1575), and ten four-minute counts were obtained on the same 5.0% mix sample.

Table 3

% AC	
4.95	5.00
5.00	4.99
5.00	4.98
5.01	4.97
4.98	4.94

Ave. = 4.98%
Std. Dev. = 0.02

On December 15, 1986, the gauge was again set up in the north end of the Bituminous section. The original three pan calibration data was entered and a new background count taken. The 5.0%, 6.0%, and 7.0% samples used in the original calibration were measured using four-minute counts and yielding the following percentages:

Table 4

Actual %	Measured %
5.0 pan	4.98
6.0 pan	5.99
7.0 pan	7.00 & 7.03

Calibration Transfers:

Gauges from the Central Lab, District 4, and District 3 were used in the Central Lab to test the calibration transfer procedure. Transfer pan counts by these three gauges, taken in the Central Lab are as follows:

Table 5
Measure Counts

Transfer Pan	Lab-Gauge	Dist. 3 Gauge	Dist. 4 Gauge
1 (4.0%)	2233	1999	2016
2 (5.0%)	2462	2197	2221
3 (6.0%)	2716	2427	2453
4 (7.0%)	2995	2671	2702
5 (8.0%)	3286	2927	2951

A new mix of 100% gravel at 5.0%, 6.0%, and 7.0% asphalt content was used in testing the transfer procedure. Because of an error in mixing, the intended 6.0% mix was actually less than 6.0% by an undetermined amount.(1) The following calibration data was developed in the lab gauge for this mix based on sixteen-minute calibration counts.

<u>100 % Gravel Mix</u>

Slope x 1000	= 4.68
Intercept	= -5.75
Background Count	= 1646
Temperature	= 215°F
Sample Weight	= 6900 grams

The transfer procedure was attempted using the above calibration and the District 4 gauge. A background count of 1503 was determined on the District 4 gauge and the calibration transfer equations were used to determine a corrected slope and intercept for use in measuring the 6.0% sample. For this 6.0% sample, counts on the 5.0% and 7.0% transfer pans were used for the calculations.

$$S_{Dist.4} x1000 = 4.68 \times \frac{(2995-2462)}{(2702-2221)} = \underline{5.19}$$

$$I_{Dist.4} = (.00468x2995)-(.00519x2702) + (-5.75) = -\underline{5.76}$$

(1) When this batch was removed from the mixer, it was noticed that the paddle and scraper hadn't been lowered to the bottom of the bowl during mixing, thus resulting in a non-homogeneous batch. The gauge sample was later assumed to contain less asphalt than the intended 6.0% because of the lower content indicated by the nuclear gauge.

The new calibration of Sx1000 = 5.19 and I = -5.76 was manually entered into the District 4 gauge. A four-minute measure count taken on the 6.0% gravel sample indicated an asphalt content of 5.92%.

This procedure was repeated using the 5.0% gravel mix in place of the 6.0% mix. The 4.0% and 6.0% transfer pan counts were used to adjust the lab calibration slope to 5.16 and intercept to -5.70. The 5.0% gravel mix, based on a four-minute measure count using the adjusted calibration, was read at 5.11% in the District 4 gauge.

Repeating this procedure a third time using the 7.0% gravel mix sample and calibrations based on the 6.0% and 8.0% transfer pans, the new mix calibration was Sx1000 = 5.36 and I = -6.19. A four-minute measure count indicated 7.13% asphalt.

To determine the effect of using transfer calibrations based on different transfer pan combinations, all three mix percentages were measured in the District 4 gauge using the transfer pan combinations indicated below.

Combination 1

Adjusted Slopex1000 = 5.19
Adjusted Intercept = -5.76

Transfer Pans Used	AC Content (%) Actual	Measured
	5.0	4.97
5.0% & 7.0%	6.0	5.89
	7.0	7.04

Combination 2

Adjusted Slopex1000 = 5.16
Adjusted Intercept = -5.70

Transfer Pans Used	AC Content (%) Actual	AC Content (%) Measured
	5.0	5.00
4.0% & 6.0%	6.0	5.95
	7.0	7.03

Combination 3

Adjusted Slopex1000 = 5.36
Adjusted Intercept = -6.19

Transfer Pans Used	AC Content (%) Actual	AC Content (%) Measured
	5.0	4.98
6.0% & 8.0%	6.0	5.89
	7.0	7.07

Combination 4

Adjusted Slopex1000 = 5.27
Adjusted Intercept = -5.92

Transfer Pans Used	AC Content (%) Actual	AC Content (%) Measured
	5.0	5.09
4.0% & 8.0%	6.0	5.93
	7.0	7.11

An actual field test of the transfer procedure was conducted on February 4-5, 1987, in the District 4 Materials Lab. A mix was calibrated in the Central Lab and samples of the mix were taken to District 4 along with the five transfer pans. Twenty one-minute counts were recorded and averaged in the statistical test mode for each transfer pan with the gauge in its anticipated operating location.

Table 6

Transfer Pan	District 4 Measure Counts
1 (4.0%)	1998
2 (5.0%)	2213
3 (6.0%)	2442
4 (7.0%)	2687
5 (8.0%)	2943

An adjusted slope and intercept was calculated for the District 4 gauge based on transfer pan counts and mix samples with known AC contents of 5.0%, 6.0%, and 7.0% were measured using the adjusted calibration. The measured asphalt contents, using the District 4 gauge and based on the 4.0% and 8.0% transfer pans were 5.04%, 5.95%, and 7.01% respectively.

This procedure was repeated using the District 4 gauge to produce a calibration on the same mix, then transferring the calibration back to the Central Lab gauge where samples of the mix were tested. Back in the Central lab, the 5.0%, 6.0%, and 7.0% samples were measured at 4.97%, 6.04%, and 6.99% respectively, using calibration adjustments based on the 4.0% and 8.0% transfer pans.

A final focus of calibration transfers investigated the measurement of mixes when the gauge is in a location other than where the transfer pans were measured. This situation would occur if the transfer pans are measured in the District Lab and the gauge is then moved to the field for on-site testing. To simulate this situation, the District 3 gauge was used to measure transfer pans and to obtain a background count at a particular location. The gauge was then moved to a new lab location for sample measurements. The 5.0%, 6.0%, and 7.0% gravel mix was again used to test calibration transfers. Transfer pan counts on the Central Lab and District 3 gauges can be found in Table 5. The background count was 1446 at the calibration location and 1388 at the measurement location. The original lab calibration (Sx1000 = 4.68, I = -5.75, Bkg. = 1646, T = 215°F) in the lab gauge for the gravel mix was used in this trial.

To calculate the adjusted calibration for this situation, a "B" factor is introduced into the intercept adjustment calculation. The "B" factor is the difference in background counts between the District location and the field measurement location.

The lab gauge calibration was adjusted for District 3 gauge use at a plant site, based on the 4.0% and 8.0% transfer pans, as follows:

$$S_{Dist.3} = 4.68 \; \frac{(3286-2233)}{(2927-1999)} = \underline{5.31}$$

$$I_{Dist.3} = (.00468x3286)-(.00531x(2927-B)) + (-5.75)$$

Where: B = 1646-1388 = 58

$$I_{Dist.3} = \underline{-5.61}$$

The adjusted slope and intercept were manually entered into the District 3 gauge, and four-minute measure counts were taken on the three gravel mix samples, yielding the following results:

Table 7

Actual AC %	Measured AC %
5.0	4.98
6.0	5.87
7.0	7.04

DISCUSSION AND SUMMARY

The first four series of measurements established that gauge results are repeatable within an acceptable tolerance as evidenced by standard deviations of 0.04, 0.03, and 0.02 on multiple tests of the same samples. This work also demonstrated that a calibration will give accurate results when used in the same gauge on a day-to-day basis, provided that a new background count is taken at the beginning of each workday and when measurements are taken in locations other than where the calibration was performed. When an incorrect background count was used, the series of five measure counts averaged 0.31% less than the actual AC content, thus illustrating the importance of using the correct background count.

The investigation of the calibration transfer procedure indicates that it will be feasible to develop mix calibrations in the Central Lab as part of the mix design process, and transfer them to District gauges for monitoring project asphalt contents with an acceptable degree of accuracy and repeatability. Under ideal laboratory conditions, the worst case measurement was 0.13% higher than the actual asphalt content. This compares favorably with results expected from current extraction procedures.

The selection of transfer pans used in calibration adjustment did not appear to have a significant effect on asphalt content measurements. This is due to the amount of care used in preparing the transfer pan mixes as indicated by the five pan correlation factor of 0.9992 (Figure 1). The Troxler 3241-B Instruction Manual recommends use of transfer pans with asphalt contents nearest the intended content of the mix being samples; however, with such a high correlation on the five pans, use of the high and low pans only should yield acceptable calibration adjustments.

When analyzing the calibration transfer data, it must be kept in mind that the 6.0% gravel sample was improperly mixed and was actually somewhat less than 6.0%. This would explain why measurements on the 6.0% sample consistently measured around 5.9% (four measurements averaged 5.915%). The correlation factor for the 100% gravel mix (Slopex1000 = 4.68, Intercept = -5.75) was 0.9974. The Troxler Manual states that a calibration should be considered invalid if the correlation factor is less than 0.995, so even though the calibration was valid, it was nearing the point where it would be considered unacceptable. The correlation factor of the calibration used in the District 3 field test was 0.9996, and excellent results were obtained using the 4.0% and 8.0% transfer pans. These figures demonstrate that for the transfer procedure to be successful, the need for properly prepared calibration mixes, transfer pans, and production samples cannot be overstressed.

The final point indicated by the research is that if there is a need to have the gauge at a plant or field site for a special reason, use of the "B" factor in the intercept calibration adjustment appears to make this workable when using calibrations produced in the Central Lab. More research on this particular procedure should be conducted; however, to verify its reliability. Even if a procedure can be developed, field use of these gauges should be kept to a minimum since they were not designed to withstand frequent handling and the types of conditions they would be exposed to in field use.

RECOMMENDATIONS:

Results from this study were used as a basis for the calibration transfer section of Materials I.M. 335 - "Method of Test For Determining the Asphalt Content of Bituminous Mixtures by the Nuclear Method" issued January 1987. The Troxler 3241-B asphalt content gauge will be used by the District Materials Offices to monitor project asphalt contents and help reduce the number of chemical extractions performed.

Until more experience is gained in the use of the gauge and the procedures developed for its use, it would be beneficial for the Districts to assure themselves that the transferred calibrations are correct by obtaining cold feed gradation material, mixing a sample of known asphalt content and measuring it in the gauge. If the measured asphalt content is off by more than several tenths of a percent, the calibration, as well as the technician's testing and sampling methods, should be reviewed.

Further research should be conducted investigating the use of this gauge for determining asphalt content of RAP samples, and for use of Marshal samples for asphalt content determination.

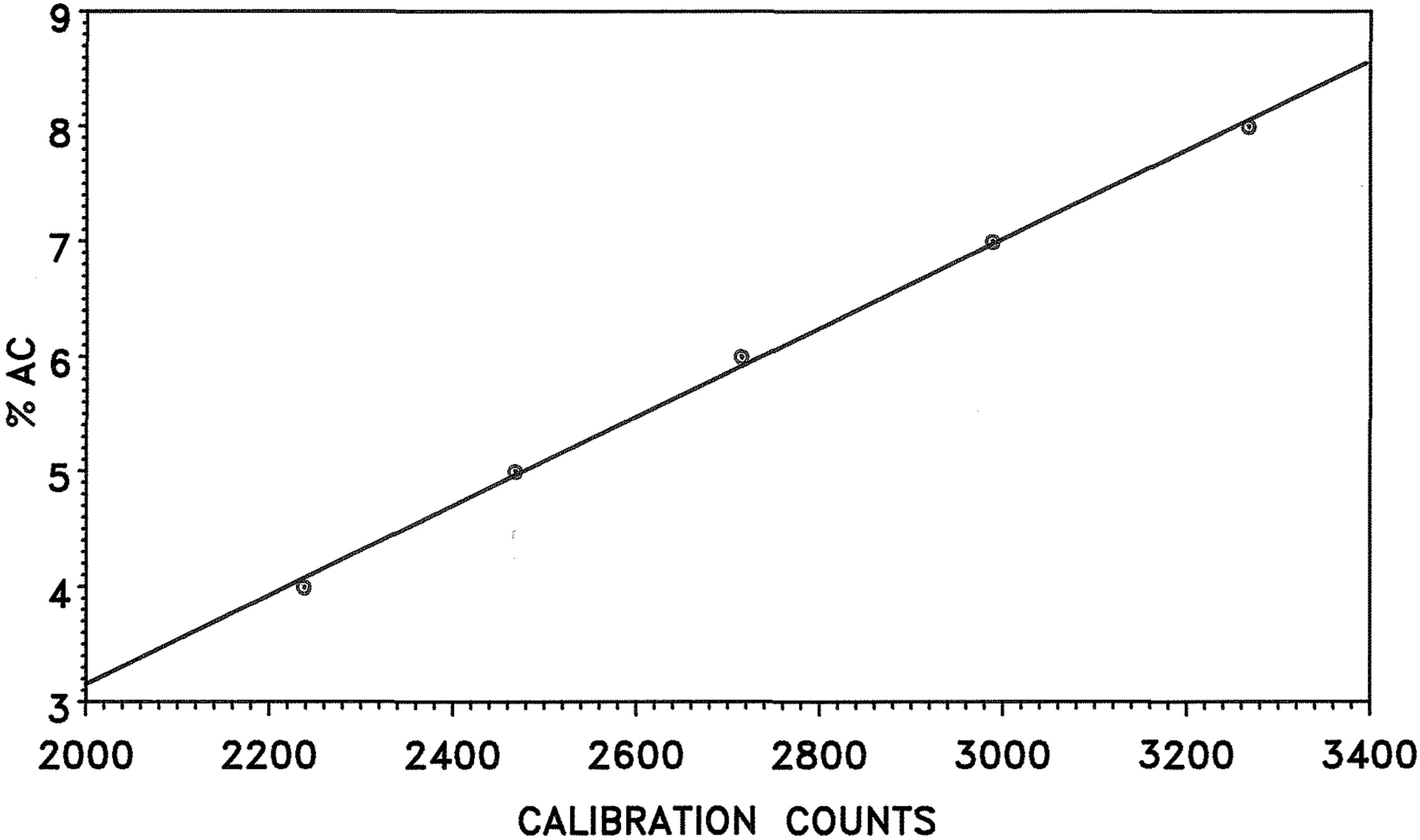
FIGURE 1

TRANSFER PAN CALIBRATION CORRELATION – SEALED

1 – 16 MINUTE CALIBRATION COUNT

%AC = 0.00387*(COUNTS) – 4.59

12/04/1986 C.C.=0.9992



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