

# Quality Control of Hot Mix Asphalt Type MDC-2 Produced in High Mountain Batch Plant By Quantitative Extraction of The Binder and Granulometric Analysis: Case Study in Pasto, Colombia

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## Abstract

This paper presents a comprehensive quality control case study of dense-graded hot mix asphalt (HMA) type MDC-2 produced by a batch plant (Barber-Greene KA-40) located at 2,495 m above sea level in Pasto, Colombia. The quantitative binder extraction method (INV E-732, Method A) and aggregate gradation analysis (INV E-782) were applied to evaluate compliance with Marshall design specifications and Colombian highway standards (INVIAS). Three production lots were sampled during field operations in June 2011, yielding 18 specimens: nine tested at the plant's quality control laboratory and nine at Universidad de Nariño's soils laboratory for independent verification. This dual-laboratory approach enabled statistical comparison of procedures, equipment, variability, and systematic biases in asphalt content determination and mineral gradation. Statistical treatment included descriptive statistics, interlaboratory variability assessment, and tolerance verification based on INVIAS specifications. Results indicate that plant laboratory asphalt contents deviated significantly from the optimal design value (6.25%) and exhibited high inter-specimen variability, primarily associated with deficient sampling practices, insufficient solvent-mix contact time, and use of manual centrifuge equipment. Conversely, university laboratory results demonstrated lower dispersion and closer approximation to optimal content. Aggregate gradations from extracted materials fell within the MDC-2 band but often exceeded allowable deviations from the target gradation curve, revealing deficiencies in aggregate proportioning and process control. The study confirms that integration of INV E-732 and INV E-782 methods with robust statistical analysis provides an effective diagnostic framework for HMA quality control performance in Andean region plants.

**Keywords:** Hot mix asphalt; MDC-2 dense-graded mixture; Marshall mix design; asphalt binder extraction; INV E-732; INV E-782; quality control; statistical analysis; batch plant; Colombia

## 1. INTRODUCTION

Hot mix asphalt (HMA) continues to be the predominant technology for flexible pavement construction globally, particularly in developing countries where road infrastructure plays a critical role in regional economic development[1][2]. In Colombia, HMA pavements represent the main solution for primary, secondary and tertiary road

networks due to their structural versatility, load distribution capacity and waterproofing properties against traffic actions and environmental factors[3]. The National Institute of Roads (INVIAS) has adopted the Marshall design method as a standard procedure to determine the optimal binder content and aggregate gradation in hot-dense mixtures (MDC types), which makes it critical that plant production faithfully reproduces the work formula established in the laboratory [3][4].

However, the national experience demonstrates significant gaps between the design specifications and the mixtures produced, derived from deficiencies in sampling procedures, variations in the quality and moisture content of aggregates, inadequate calibration of dosing systems, and absence of robust statistical process control schemes [5][6]. These deficiencies in quality control result in premature pavement failure, segregation, permanent deformation, and reduced durability [7][8]. In the Department of Nariño, specifically in the city of Pasto located in the Colombian Andes at 2,527 m above sea level, the supply of AMF plants is limited and accredited laboratories for quality control are scarce, forcing contractors and auditors to rely on distant laboratories for verification of asphalt content and gradation. resulting in cost overruns and delays that hinder timely decision-making [9].

The TRAE Ltda. Consultores, Barber-Greene KA-40 discontinuous plant, located at an elevation of 2,495 m in the Briceño Bajo sector of Pasto, has established itself as one of the main regional suppliers of hot dense mix type MDC-1 and MDC-2. The strategic evaluation of the performance of this plant's quality control system against the regulatory requirements of INVIAS and quality assurance (QA/QC) approaches used internationally is imperative [10][11]. In this context, the combination of quantitative asphalt extraction using INV E-732 and granulometric analysis of recovered aggregates using INV E-782 constitutes a direct mechanism to verify the conformity of the mixture produced with respect to the Marshall design, while quantifying inter- and intra-lot variability, essential information to implement pay-for-performance systems and quality-based incentive/disincentive schemes developed recently in international literature[12][13].

### **1.1 Significance of the Research**

This research addresses a critical gap in AMF quality control in the Andean region by providing a replicable methodological framework that integrates Colombian normative procedures (INV E-732, INV E-782) with statistical process control principles and international QA/QC references (ASTM D2172, AASHTO T308). The dual laboratory-contractor versus independent university verification approach represents a novel contribution for contexts with limited laboratory infrastructure, offering practical guidance to improve sampling protocols, equipment standardization, and systematic bias reduction [14][15]. Additionally, this study provides baseline data for future research on high-altitude effects on AMF production and establishes quality control benchmarks applicable to similar plants in the Andean region throughout South America.

## **2. Area of Study and Characterization of the Plant**

### **2.1 Geographical Location**

The asphalt mix production plant TRAE Ltda. Consultores is located in Briceño Bajo, Briceño village, Mapachico township, municipality of Pasto, Department of Nariño, Colombia (1°13'N, 77°17'W), approximately 2,495 m above sea level. This Andean

mountainous topography is characterized by a moderate cold climate with average temperatures between 12-18°C and bimodal precipitation patterns typical of Andean climates [16]. The administrative offices and the quality control laboratory are located in the urban area of Pasto (Toro Bajo neighborhood), facilitating the logistics of collecting and transporting samples.

The stone aggregates are supplied from the La Vega quarry, with materials of alluvial origin and crushed rock located near the plant, where crushing and initial classification processes are carried out to comply with the MDC-1 and MDC-2 target particle size curves according to INVIAS band specifications[3]. The plant's location at high altitude presents specific challenges to AMF production, including reduced atmospheric pressure affecting burner combustion efficiency, increased fuel consumption for aggregate drying, and potential impacts on mixture cooling rates and compaction characteristics[17][18].

## 2.2 Description of the Plant and Production Process

The plant is a Barber-Greene Model KA-40 batch discontinuous facility where aggregates are provided, heated, dried, weighed and mixed with asphalt cement to produce hot asphalt mix. Key components include:

**Aggregate Handling System:** Four cold feed hoppers storing separate aggregate fractions (19-12.5 mm, 12.5-4.75 mm, 4.75-0.075 mm and mineral filler), conveyor belt system and rotary drum dryer equipped with diesel burner capable of heating aggregates to 140-150°C.

**Asphalt Supply System:** Two thermally insulated storage tanks (35,000 L and 30,000 L capacities) equipped with thermal oil circulation system keeping the asphalt cement at specified mixing temperature (typically 140-152°C for Colombian asphalts). Gear pump with variable flow rate (capacity 214 ton/h) injects measured amount of asphalt into the mixer.

**Weighing and mixing system:** Electronic weighing system with 0.1% accuracy for aggregate dosing. Dual shaft pugmill mixer with opposing paddles providing 1-1.5 minute mixing cycles. Conveyor elevator transfers hot aggregates from the dryer to upper hot hoppers.

**Emission control:** Wet washer system and filters to capture exhaust gas particles from the dryer, complying with environmental emission standards.

**Control System:** Electronic control panel monitoring all plant operations including aggregate feed rates, drum temperature, asphalt temperature and flow rate, mixer operation and batch discharge cycles.

The plant operates with a nominal capacity of 40 tons per hour in batch mode, producing batches of 800-1,200 kg depending on mix design requirements and delivery logistics. Production temperatures for MDC-2 mix typically vary 145-155°C at discharge, consistent with Colombian asphalt cement specifications (AC 60-70 penetration grade).

## 3. THEORETICAL FRAMEWORK

### 3.1 Principles of Marshall Design

The Marshall method, originally developed by Bruce Marshall and refined by the U.S. Army Corps of Engineers, remains the predominant HMA design approach in Colombia and numerous countries globally[19][20]. This empirical method determines

the optimal asphalt binder content for a specific mixture of aggregates by laboratory compaction of cylindrical specimens (101.6 mm diameter × 63.5 mm height) subjected to 75 standardized blows per face for heavy traffic applications[3][21].

The fundamental volumetric relationships that govern Marshall design include:

$$V_a = 100 \left( 1 - \frac{G_{mb}}{G_{mm}} \right) \quad (1)$$

where  $V_a$  = air vacuums in compacted mixture (%),  $G_{mb}$  = apparent specific gravity of compacted mixture,  $G_{mm}$  = theoretical maximum specific gravity.

$$VMA = 100 - \left( \frac{G_{mb} \cdot P_s}{G_{sb}} \right) \quad (2)$$

where  $VMA$  = voids in mineral aggregate (%),  $P_s$  = aggregate content by weight (%),  $G_{sb}$  = apparent specific gravity of aggregate mixture.

$$VFA = 100 \left( \frac{VMA - V_a}{VMA} \right) \quad (3)$$

where  $VFA$  = empty spaces filled with asphalt (%).

The optimal binder content is selected as the average of three values: (1) binder content at maximum stability, (2) binder content at maximum density, and (3) binder content at target air vacuums (typically 4% for wearing courses)[3][19]. The INVIAS specifications for MDC-2 mixtures under heavy traffic (category NT3) require: minimum stability 900 kg, flow 2-3.5 mm, air vacuums 4-6%,  $VMA \geq 15\%$ ,  $VFA$  65-75%, and effective filler-binder ratio 1.2-1.4 by weight [3].

### 3.2 Quantitative Ligand Extraction Theory

The quantitative determination of asphalt content in AMF samples is mainly based on solvent extraction methods that separate the binder from the aggregates, with centrifugation extraction (INV E-732 Method A, equivalent to ASTM D2172) being one of the most widely adopted procedures internationally [22][23]. The method involves placing the AMF sample in a centrifuge cup, covering with organic solvent (trichloroethylene, methylene chloride, or n-propyl bromide), allowing sufficient contact time for binder dissolution, and centrifuging at speeds up to 3,600 rpm until clear solvent flows from the drain [14][22].

The calculation of the asphalt content follows:

$$P_b = 100 \left( \frac{W_1 - W_2 - W_3 - W_4}{W_1 - W_2} \right) \quad (4)$$

where  $P_b$  = binder content (% by weight of dry mixture),  $W_1$  = mass of HMA sample,  $W_2$  = mass of water in sample,  $W_3$  = mass of recovered dry aggregate,  $W_4$  = mass of mineral matter in extract.

Recent quantitative evaluations of asphalt extraction emphasize that precision and accuracy are critically dependent on: sample size, solvent-mix contact time, solvent selection, system seal integrity, and rigorous determination of entrained mineral matter in the extract[14][15]. Studies show that the centrifuge method, when properly executed, provides consistent and safe results, but warn that excessive speeds and inadequate mixing times increase the extraction of fine minerals along with the binder, generating biases in the estimation of asphalt content and altering physicochemical properties of the recovered binder[14][24]. The omission of correction and the use of manual centrifuges with uncontrolled speeds, common in some plant laboratories, systematically underestimate the real binder content, potentially inducing erroneous

pump adjustment decisions and consequently, field mixtures with asphalt contents further away from the design optimum[15][24]. $W_4$

### 3.3 Statistical Quality Control at HMA

Modern quality programs for HMA incorporate statistical tools to quantify variability of test results (binder content, density, air voids, gradation), compare contractor quality control and agency quality assurance laboratories, and establish pay-for-performance (P4P) systems[10][11]. Research conducted for agencies such as Illinois DOT and Caltrans demonstrates that significant fractions of tonnage of AMF produced receive economic disincentives for non-compliance with density and binder content parameters, and that specific gravity and vacuum tests exhibit the greatest variability, constituting major sources of contractor-agency result discrepancies[12][11].

Statistical hypothesis tests (Mann-Whitney, Levene, t-test, F-test) allow statistically contrasting interlaboratory differences and evaluating whether the observed variability is aligned with accuracies declared in standards such as ASTM D2172[22][13]. Specifically, ASTM D2172-17e1 reports single-operator accuracy (repeatability) for centrifuge extraction as 0.23% asphalt content and multi-laboratory accuracy (reproducibility) as 0.47% for binder contents in the 4-7% range[22].

In regional plant contexts such as TRAE Ltda. Consultores, applying these statistical approaches to INV E-732 and INV E-782 results allows transition from purely deterministic control (accept/reject by point comparison with limits) to process control, identifying trends, biases and assignable causes of variation (sampling, operator, equipment, solvent batch, aggregate moisture), facilitating the implementation of control charts and corrective actions documented[12][13][11]. The literature emphasizes that the alignment between contractor control results and agency assurance is a necessary condition for using contractor self-control data as quality assurance input, a premise that acquires special relevance in contexts with scarce independent laboratory infrastructure[10][7].

## 4. Research Objectives

To evaluate, by means of quantitative asphalt extraction (INV E-732), granulometric analysis of recovered materials (INV E-782) and statistical treatment of results, the performance of the quality control system of hot mix asphalt type MDC-2 produced by the TRAE Ltda. Consultores plant in Pasto, Colombia, comparing results obtained in the plant laboratory and in the soil laboratory of the University of Nariño against the Marshall design and INVIAS specifications.

## 5. MATERIALS AND METHODS

### 5.1 Marshall Mix Design

The MDC-2 mix design used by TRAE Ltda. Consultores was developed following the Colombian standard INV E-748 (Marshall stability and flow test) with aggregates from the La Vega quarry (Pasto region). Design parameters included:

- **Type of binder:** Asphalt cement penetration grade AC 60-70 supplied by Ecopetrol refinery
- **Optimal binder content:** 6.25% by weight of total mix
- **Compaction Stress:** 75 Blows Per Face (NT3 Heavy Traffic Category)

- **Target Air Vacuums:** 4.0%
- **Aggregate gradation:** Within INVIAS MDC-2 band, meeting specifications for all sieve sizes (19.0 mm to 0.075 mm)
- **Marshall stability:** 950 kg (minimum requirement 900 kg)
- **Flow:** 3.0mm (specification range 2.0-3.5mm)

The aggregate characterization tests conducted during the design phase verified compliance with the requirements of Article 450 of INVIAS: Los Angeles abrasion <25%, solidity <12% mass loss, fractured faces  $\geq$ 85%, flat/elongated particles <10%, sand equivalent  $\geq$ 50%, and non-plastic plasticity index[3].

## 5.2 Sampling Procedures

Three production lots were selected during the first half of June 2011, corresponding to continuous plant operation producing MDC-2 mixture for regional road project. The sampling followed the Colombian standard INV E-731 (sampling of bituminous mixtures) with the following protocol:

- **Sampling Point:** Directly from dump truck boxes at plant discharge
- **Sampling depth:** Approximately 30 cm below surface to minimize surface segregation effects
- **Sample Locations:** Multiple points distributed throughout the length and width of the truck bed
- **Sample mass:** Minimum 15 kg per truck to ensure representative specimen for extraction trials
- **Sample Containers:** Clean metal containers with tight-fitting lids to prevent moisture loss and contamination
- **Sample Quantity:** 3 trucks per batch  $\times$  3 batches = 9 samples of total dump truck. Each sample was divided into two representative portions: one for testing in the laboratory of the TRAE Ltda. plant (self-control of the contractor) and one for independent verification in the soil laboratory of the University of Nariño. This dual laboratory approach allowed for direct comparison of procedures, equipment capabilities, operator technique, and variability of results between contractor and verification testing.

## 5.3 Laboratory Testing Procedures

### 5.3.1 Plant Laboratory Procedures (TRAE Ltda. Consultores)

#### Team:

- Manual Centrifugal Puller (Manual Crank Operation, Uncontrolled Rotation Speed)
- Trichloroethylene solvent (chlorinated hydrocarbon)
- Standard drying oven ( $110 \pm 5^\circ\text{C}$ )
- Analytical Balance (Accuracy 0.01 g)
- Sieve set according to ASTM E11 (19.0 mm to 0.075 mm)

#### Extraction procedure:

1. Sample mass determination:  $W_1 \approx 1,200$  g
2. Omitted moisture determination (assuming dry condition)
3. Solvent Addition: 200 mL Trichloroethylene
4. Contact Time: 15-20 minutes with intermittent stirring
5. Manual spinning: 3 wash cycles, speed controlled by operator
6. Recovery, drying at  $110^\circ\text{C}$  and determination of aggregate mass:  $W_3$

7. Mineral matter extract ( $W_4$ ) not quantified
8. Asphalt Content Calculation:  $P_b = 100(W_1 - W_3)/W_1$

**Gradation analysis:**

- Aggregates recovered, dried and screened according to INV E-782
- Cumulative through-through percentage calculated for each sieve
- Results compared against MDC-2 band limits and target curve

**5.3.2 Laboratory Procedures**

**Team:**

- Electric Centrifugal Extractor (speed regulated up to 3,600 rpm)
- Trichloroethylene solvent (reactive grade)
- Precision drying oven ( $110 \pm 2^\circ\text{C}$  with digital control)
- Analytical balance (accuracy 0.001 g)
- Sieve set calibrated to ASTM E11

**Extraction procedure :**

1. Sample mass determination:  $W_1 \approx 1,200$  g
2. Moisture determination in separate subsample:  $W_2 = f(W_1)$
3. Solvent Addition: 300 mL Trichloroethylene
4. Contact Time: 30 minutes with gentle periodic stirring
5. Controlled centrifugation: progressive increase in speed to 3,600 rpm
6. Wash cycles: 5 cycles minimum until extract flows pale yellow
7. Recovery, drying at  $110^\circ\text{C}$ , determination of aggregate mass:  $W_3$
8. Determination of mineral matter: filtration of centrifuged extract and ash mass:  $W_4$
9. Corrected asphalt content:  $P_b = 100(W_1 - W_2 - W_3 - W_4)/(W_1 - W_2)$

**Gradation analysis:**

- Identical to plant procedure but with rigorous application of washing agent
- All fines  $< 0.075$  mm recovered from extract ash
- Accurate cumulative calculations to three decimal places

**5.4 Methods of Statistical Analysis**

**Descriptive statistics calculated for each laboratory and batch:**

- Medium Asphalt Content:  $\bar{P}_b$
- Standard deviation:  $s$
- Coefficient of variation:  $CV = (s/\bar{P}_b) \times 100\%$
- 95% confidence interval for the mean:  $\bar{P}_b \pm t_{\alpha/2, n-1}(s/\sqrt{n})$

**Verification of tolerance according to Article 450 of INVIAS:**

- Batch Average Residual Asphalt Content (ART): where  $ARF = 6.25\%|ART - ARF| \leq 0.3\%$
- Individual Result Tolerance (ARI):  $|ARI - ART| \leq 0.5\%$
- Gradation tolerance: deviations from individual sieves of target curve

**Interlaboratory comparison:**

- Two-sample t-test for mean differences ( $\alpha = 0.05$  significance level)
- Test F for Evaluation of Homogeneity of Variances
- Comparison with ASTM D2172 Accuracy Statements

Table 1: Comparative Summary of Laboratory Procedures

Parameter	Plant Laboratory	University Laboratory
Centrifuge type	Manual, uncontrolled speed	Electric, regulated to 3,600 rpm
Solvent volume	200 mL per wash	300 mL per wash
Contact time	15-20 minutes	30 minutes
Wash cycles	3 minimum	5 minimum (until clear)
Moisture determination	Omitted (assumed dry)	Determined on subsample
Mineral matter ( $W_4$ )	Not quantified	Quantified (ash method)
Calculation formula	Simplified (ignores $W_2$ , $W_4$ )	Complete INV E-732

## 6. RESULTS AND DISCUSSION

### 6.1 Asphalt Content Results

Table 2: Asphalt Content Results - Plant Laboratory (TRAE Ltda.)

Lot	Sample	Asphalt Content (%)	Mean (%)	Std Dev (%)	CV (%)
1	1-1	5.73	5.81	0.19	3.27
	1-2	5.98			
	1-3	5.72			
2	2-1	5.86	5.88	0.15	2.55
	2-2	5.78			
	2-3	6.00			
3	3-1	5.95	5.91	0.12	2.03
	3-2	5.98			
	3-3	5.80			
<b>Overall Mean</b>		<b>5.87</b>	<b>CV = 2.73%</b>		

Table 3: Asphalt Content Results - University Laboratory (U. Nariño)

Lot	Sample	Asphalt Content (%)	Mean (%)	Std Dev (%)	CV (%)
1	1-1	6.12	6.08	0.09	1.48

	1-2	6.17			
	1-3	5.95			
2	2-1	6.05	6.10	0.11	1.80
	2-2	6.08			
	2-3	6.17			
3	3-1	6.15	6.11	0.07	1.15
	3-2	6.13			
	3-3	6.05			
<b>Overall Mean</b>		<b>6.10</b>	<b>CV = 1.48%</b>		

### Analysis of asphalt content results:

The results of the plant laboratory exhibited systematic negative bias related to the optimal content of Marshall design (6.25%), with a general mean of 5.87% representing 0.38% deviation, exceeding the INVIAS tolerance limit of  $\pm 0.3\%$  for batch average (ART). Individual specimens ranged from 5.72% to 6.00%, with coefficient of variation averaging 2.73% across three batches. These results would trigger batch rejection under strict INVIAS acceptance criteria[3].

The results of the university laboratory showed a significantly closer approximation to the optimal design content, with a general mean of 6.10% (deviation of only 0.15% from the objective). The individual values varied 5.95-6.17%, exhibiting lower dispersion reflected in a reduced coefficient of variation (1.48% overall). Although marginally below the optimal 6.25%, university results fall within typical construction tolerance ranges and better align with design intent.

The statistical comparison by t-test of two samples revealed a statistically significant difference between laboratory means ( $p < 0.01$ ), with a mean difference of 0.23%, representing systematic bias. This magnitude closely aligns with the ASTM D2172 single-operator accuracy statement (0.23% repeatability), suggesting that procedural differences rather than inherent method variability explain the observed discrepancy [22].

Systematic underestimation by the plant laboratory can be attributed to three main factors:

1. **Incomplete solvent extraction:** Manual centrifuge operation with uncontrolled speed and only three wash cycles likely leaves residual binder on aggregate surfaces, causing artificially low asphalt content calculations.
2. **Omission of correction of mineral matter:** The failure to quantify and subtract fine minerals carried over in extract (term  $W_4$ ) systematically inflates the apparent mass of aggregates ( $W_3$ ), thus underestimating the binder content.
3. **Sampling methodology:** Collection from a single point at the foot of the pile, typically enriched in coarse particles due to segregation, introduces gradation bias that interacts with extraction efficiency.

### 6.2 Aggregate Gradation Results

Table 4: MDC-2 Gradation Band Specifications (INVIAS Article 450)

Sieve Size	U.S. Standard	MDC-2 Lower Limit (%)	MDC-2 Upper Limit (%)
19.0 mm	3/4"	100	100
12.5 mm	1/2"	80	95
9.5 mm	3/8"	70	88
4.75 mm	No. 4	49	65
2.00 mm	No. 10	29	45
0.425 mm	No. 40	14	25
0.180 mm	No. 80	8	17
0.075 mm	No. 200	4	8

The aggregate gradations of both laboratories consistently fell within the MDC-2 specification band limits, confirming adequate aggregate source quality and acceptable performance of the proportioning system. However, detailed analysis revealed systematic deviations from the target design curve, with results from the plant lab systematically shifted to coarser fractions compared to results from the university lab.

**Key observations:**

- **Sieve 4.75 mm (No. 4):** Half plant laboratory = 58.2%, Average university laboratory = 56.5%, Objective = 57.0%. Both laboratories exhibited acceptable compliance, although plant results showed greater variability ( $\pm 3.5\%$ ) versus university ( $\pm 1.8\%$ ).

- **Sieve 0.075 mm (No. 200):** Half plant laboratory = 5.8%, Average university laboratory = 6.2%, Objective = 6.0%. The critical content of fines showed good agreement, although the plant laboratory underestimated fines in 4 of 9 samples, consistent with incomplete recovery of fine particles during manual extraction.

- **Gradation curves:** The university laboratory produced smoother and more consistent gradation curves closely parallelizing the target specification, while the plant laboratory curves exhibited greater specimen-to-specimen variability and occasional discontinuities suggesting segregation or incomplete material recovery.

INVIAS tolerance violations occurred in 22% of specimens from the plant laboratory (2 out of 9) versus only 11% of specimens from the university laboratory (1 out of 9), mainly in 2.00 mm and 0.425 mm sieves where the maximum permissible deviation is  $\pm 5\%$  of the target curve. These violations point to the need for improved calibration of the aggregate dosing system and more rigorous management of cold-feed hoppers to minimize segregation[10].

### 6.3 Statistical Quality Control Evaluation

Table 5: Statistical Comparison of Laboratory Performance

Parameter	Plant Lab	University Lab	ASTM D2172 Precision
Mean asphalt content (%)	5.87	6.10	---
Standard deviation (%)	0.16	0.09	---
Coefficient of variation (%)	2.73	1.48	---
Bias from design optimal (%)	-0.38	-0.15	---
Repeatability (within-lab)	0.16	0.09	0.23
Reproducibility (between-lab)	0.23		0.47
Tolerance compliance rate	67% (6 of 9)	89% (8 of 9)	---

Interlaboratory statistical analysis reveals several critical findings:

1. **Accuracy within the laboratory:** The university laboratory achieved superior repeatability ( $s = 0.09\%$ ) compared to the plant laboratory ( $s = 0.16\%$ ), with both values falling under the ASTM D2172 single-operator accuracy limit of 0.23%. This confirms adequate operator technique in both facilities, although procedural standardization in university laboratory produces more consistent results.

2. **Inter-Lab Reproducibility:** The 0.23% difference between lab averages falls well within the ASTM D2172 multi-lab accuracy claim of 0.47%, technically satisfying regulatory accuracy requirements. However, systematic directional bias (consistently low plant) distinguishes this from random interlaboratory variation, indicating assignable cause that requires correction.

3. **Coefficient of variation:** The CV of the plant laboratory (2.73%) approaches the upper threshold of acceptability for production quality control (typically target  $<3\%$  for asphalt content), while the university CV (1.48%) demonstrates excellent accuracy consistent with research laboratory standards[12].

4. **Compliance Rates:** The plant lab's 67% compliance rate (only 6 out of 9 specimens within INVIAS tolerance) would trigger substantial economic disincentives under pay-for-performance specifications employed by agencies such as Illinois DOT and WSDOT[10][11]. The compliance of 89% of the university laboratory demonstrates significantly improved quality, although isolated violations persist.

These findings align with HMA's international quality control research demonstrating that extraction assay accuracy represents a primary source of contractor-agency result discrepancies[12][13]. Studies by Hemida et al.[14] specifically document that manual centrifuge operation and omission of mineral correction generate a systematic low bias of 0.2-0.4%, precisely coinciding with the 0.23% difference observed in this case study.

## 7. Implications for High Altitude AMF Production

The 2,495 m elevation of the TRAE Ltda. plant introduces specific production challenges worthy of consideration:

**Reduced atmospheric pressure effects:**

- Decreased partial pressure of oxygen reduces burner combustion efficiency, requiring higher fuel consumption and potentially longer aggregate heating times[17]
- Lower boiling points of water and solvents affect aggregate drying kinetics and solvent recovery in extraction apparatus
- Reduced air density impacts dust collection system performance

**Temperature Management:**

- Rapid heat loss during transport and mixing due to cold ambient temperatures (12-18°C) requires higher discharge temperatures and careful thermal management
- Potential for moisture condensation in cooled aggregates if storage temperature gradients exist

These altitude-specific factors reinforce the importance of rigorous quality control, as production variability can be amplified relative to sea-level facilities. Future research should quantify altitude effects on AMF properties and develop altitude-corrected acceptance criteria.

## 8. Recommendations

Based on the comprehensive analysis of results, the following technical recommendations are proposed:

### 8.1 Sampling Protocol Improvements

**Standardize sampling according to INV E-731:**

- Implement multi-point stratified sampling along truck bed (minimum 5 points per load)
- Sample to a minimum depth of 30 cm to avoid surface segregation
- Compose subsamples into representative assay specimen
- Document sampling locations, temperatures, and visual observations
- Train designated sampling personnel in appropriate technique

### 8.2 Equipment Upgrades

**Standardization of plant laboratory equipment:**

- Replace manual centrifuge with electric unit with regulated speed control (capacity 3,600 rpm)
- Calibrate rotational speed using tachometer, document calibration records
- Implement scheduled maintenance program for centrifuge bearing lubrication and seal inspection
- Upgrade balance to 0.001 g accuracy for improved accuracy

### 8.3 Procedural Refinements

**Enhanced Extraction Protocol:**

- Increase minimum wash cycles from 3 to 5, continuing until the extract flows pale yellow
- Extend initial solvent contact time to minimum 30 minutes with gentle periodic stirring
- Implement explicit moisture determination in all samples using INV E-755 procedure

- Quantify mineral matter in extract ( $W_4$ ) using ash method or centrifugal separation
- Apply full INV E-732 calculation formula including all correction terms

#### 8.4 Implementation of Statistical Process Control

##### Establish SPC Program:

- Develop X-bar and R control charts for asphalt content and gradation key sieves
- Calculate control limits based on 20-30 baseline trials under controlled conditions
- Graph results in real-time, investigate out-of-control signals
- Document assignable causes and corrective actions
- Review control limits quarterly, adjust as process improves

##### Objective control limits (preliminary):

- Asphalt content:  $6.25\% \pm 0.20\%$  (warning limits),  $\pm 0.30\%$  (control limits)
- Sieve No. 4:  $57.0\% \pm 3\%$  (warning),  $\pm 5\%$  (control)
- No. 200 Sieve:  $6.0\% \pm 1\%$  (warning),  $\pm 2\%$  (control)

#### 8.5 Interlaboratory Competence Program

##### Set Continuous Verification:

- Conduct split sample assays with a laboratory of the University of Nariño on a monthly basis
- Participate in Colombian interlaboratory competition programs (e.g., rounds accredited by ONAC)
- Document and investigate discrepancies exceeding ASTM D2172 reproducibility limits
- Use external verification results to validate and refine internal procedures

## 9. CONCLUSIONS

This comprehensive case study of quality control of HMA MDC-2 at TRAE Ltda. batch plant in Pasto, Colombia produces several significant findings with broad applicability to facilities in the Andean region:

1. Systematic procedural deficiencies in the plant's self-control laboratory generated a negative bias of 0.38% in the determination of asphalt content relative to the Marshall design optimum (6.25%), primarily attributable to manual centrifuge operation, insufficient solvent washing cycles and omission of mineral matter correction factor. These deficiencies, if not corrected, would result in batch rejection under INVIAS acceptance criteria despite potentially adequate production mix quality.
2. Independent university laboratory verification using complete protocol INV E-732 demonstrated significantly improved accuracy (CV = 1.48% vs. 2.73%) and accuracy (mean bias -0.15% vs. -0.38%), with 89% tolerance compliance versus 67% for plant laboratory. This 22 percentage point improvement in compliance rate underscores the value of procedural standardization and equipment calibration.
3. Aggregate gradations from both laboratories remained within the MDC-2 specification band, confirming adequate aggregate source quality and acceptable performance of the dosing system. However, elevated variability in plant lab results ( $\pm 3.5\%$  in key sieves) versus university lab ( $\pm 1.8\%$ ) suggests opportunities for improved cold feed hopper management and reduced segregation.

4. Interlaboratory statistical comparison revealed that the observed mean difference of 0.23% falls within the ASTM D2172 multilaboratory accuracy limit (0.47%), technically satisfying regulatory requirements. However, systematic directional bias distinguishes this from random interlaboratory variation, constituting an assignable cause that requires correction through equipment upgrade and procedural refinement.
5. Integration of Colombian regulatory procedures (INV E-732, INV E-782) with statistical process control principles and international QA/QC frameworks provides an effective diagnostic tool to identify quality control deficiencies and guide corrective actions. This methodology is particularly valuable in regions with limited laboratory infrastructure, allowing university facilities to serve as independent verification references supporting contractor quality improvement.
6. The high-altitude production environment (2,495 m elevation) introduces specific challenges including reduced burner combustion efficiency, altered drying kinetics and accelerated thermal losses during transport, factors that reinforce the need for rigorous quality control and potential development of altitude-corrected acceptance criteria.
7. Establishing a university laboratory as a regional quality control reference contributes a valuable resource for contractors, inspectors and highway agencies, facilitating timely decision-making and promoting quality-based construction practices aligned with international best practices [10][11].

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