

Asphalt Extraction and Recovery Procedures

An Introduction

Presentation for OAPC Fall Seminar

By

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Acknowledgements

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- Any errors or opinions in this slide deck are those of today's presenter and do not necessarily represent the views of the Asphalt Institute.

Outline

- Context
- Purpose of Asphalt Extraction and Recovery Procedures
- Extraction Procedures Overview
- Recovery Procedures Overview
- Considerations When Interpreting Results
- Summary



Context

- Emerging interest in recovered asphalt cement binder properties
 - Asphalt cement extracted from loose asphalt mix or pavement samples
 - Asphalt cement recovered
 - Results used for QA acceptance of asphalt cement
- Value in understanding
 - Available extraction and recovery procedures
 - Interpretation of test results
 - How do recovered binder properties compare to supplied binder supplied and what factors affect results?

Purpose of Extraction-Recovery Procedures

- Extraction
 - Quantify asphalt content in mixture
 - Determine aggregate gradation of mixture
 - Volumetric mix design, QA, forensic investigation
- Recovery
 - Test asphalt binder properties
 - Asphalt binder grade selection for mixtures incorporating RAP (i.e. blending charts)
 - Forensic investigation
 - QA Acceptance of PGAC? (emerging)

Solvent Extraction Procedures

- “Quantitative of Bitumen from Bituminous Paving Mixtures” (ASTM D2172-17)
 - Method A – **Centrifuge** (most commonly used)
 - Method B – **Reflux** (completely contained)
 - Method C – **Vacuum**
 - Method D – **Extraction Kettle**
- Ignition Oven (ASTMD6307-16)
 - Only determines asphalt cement content
 - Asphalt binder is burned off and cannot be subsequently tested



Method A – Centrifuge Extraction ASTM D2172 Extraction Procedures



Centrifuge Extraction Apparatus

Method B – Reflux Extractor

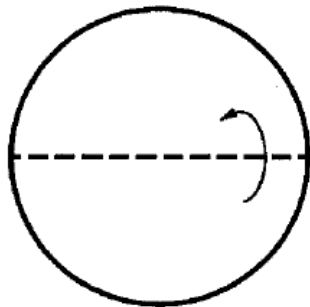
ASTM D2172 Extraction Procedures



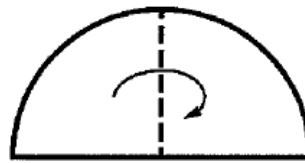
Reflux Extraction Apparatus

Method C – Vacuum Extractor

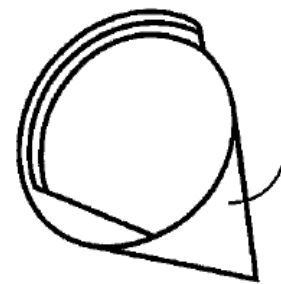
ASTM D2172 Extraction Procedures



First fold



Second fold



Open to a cone



Vacuum Extraction Apparatus

Method D – Extraction Kettle

ASTM D2172 Extraction Procedures

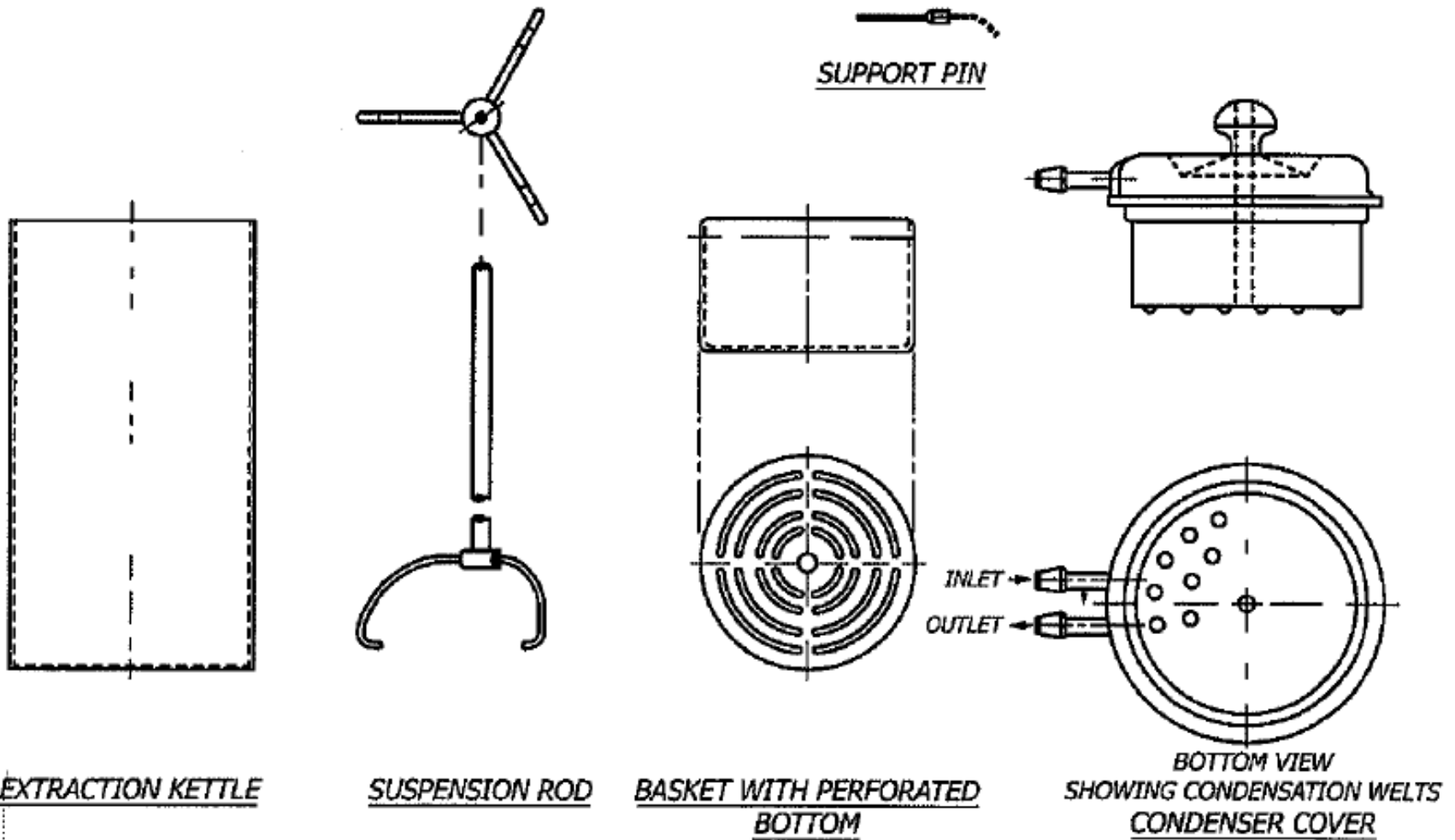


FIG. 6 Extractor Unit (Test Method D)

Mix Component Analysis After Asphalt Extraction

- Determine asphalt content
 - Final mass of sample and amount of asphalt binder extracted used to calculate **asphalt content**
- Testing extracted aggregate
 - Gradation
 - Consensus properties



ASTM D2172 Extraction Procedures

- Section 3: “Summary of Test Methods”
 - The asphalt mixture is extracted with trichloroethylene (TCE), normal propyl bromide (nPb), or methylene chloride using the extraction equipment applicable to the particular method.
 - The asphalt binder content is calculated by difference from the mass of extracted aggregate, moisture content, if determined, and mineral matter in the effluent (binder-solvent solution).
 - The bitumen content is expressed as a mass percent of moisture-free mixtures.

ASTM D2172 Extraction Procedures

- Section 8.2: “Preparation of Test Specimens”
 - 8.2.1
 - Separate sample by hand spatula or trowel, then split and reduce sample to required testing size in accordance to AASHTO R47.
 - If sample is not able to be separated or split, place sample in a large, flat pan and warm to $110 \pm 5^{\circ}\text{C}$, only heating mixture until it is pliable enough to separate.
 - Split or quarter the material until the mass of material required for test is obtained and determine the mass of the sample

ASTM D2172 Extraction Procedures

- Section 8.2: “Preparation of Test Specimens”
 - 8.2.1 (con’t)
 - Note 5: In some cases, polymer modified mixtures need to be warmed at temperatures higher than 110°C [230°F] in order to split or quarter the mix. In all cases, the minimum temperature for the minimum time needed to split the mixture should be used so that any aging to the asphalt binder is minimized.
 - 8.2.2
 - The size of the test sample shall be governed by the nominal maximum aggregate size of the mixture and shall conform to the mass requirement shown in Table 1
 - Based on Nominal Maximum Aggregate Size (NMAS) of mix

ASTM D2172 Extraction Procedures

- Section 8.2: “Preparation of Test Specimens”
 - 8.2.3
 - If the sample was obtained from the field and contains moisture, oven dry the HMA sample to a constant mass at a temperature of $110 \pm 5^{\circ}\text{C}$ or determine the moisture content of the sample according to Test Method D1461, so that the measured mass loss can be corrected for moisture.
 - ASTM D1461, “Moisture or Volatile Distillates in Bituminous Paving Mixtures”.

Precision and Bias

ASTM D2172 Extraction Procedures

- Section 15: “Precision and Bias”
 - 15.1 Mixtures with Aggregate Water Absorption Capacities of Less than 1.25% (Note 12):

Test and Type Index	Standard Deviation (1s) ^A	Acceptable Range of Two Test Results (d2s) ^A
Single Operator Precision (Note 14)		
Method A (Centrifuge)	0.21	0.59
Method B, C, D (Reflux)	0.19	0.54
Method E (Vacuum)	0.21	0.59
Multilaboratory Precision (Note 14)		
Method A (Centrifuge)	0.22	0.62
Method B, C, D (Reflux)	0.23	0.65
Method E (Vacuum)	0.22	0.59

- ^AThese numbers represent, respectively, the (1s%) and (d2s%) limits as described in Practice C670.

Precision and Bias

ASTM D2172 Extraction Procedures

- Section 15: “Precision and Bias” (con’t)
 - 15.1 Mixtures with Aggregate Water Absorption Capacities of Less than 1.25% (Note 12):
 - Note 12: 1974-1985 AMRL data
 - AC Grades: AC-10, AC-15, AC-20, AR-2000, AR-4000, AR-8000
 - Gradations: NMAS of $\frac{3}{4}$ - inch stone or smaller
 - Water absorption consistently below 1.25%
 - Note 15: No statistical difference between solvents
 - Therefore precision statements includes data obtained using: benzene, trichloroethane, trichloroethylene, methylene chloride

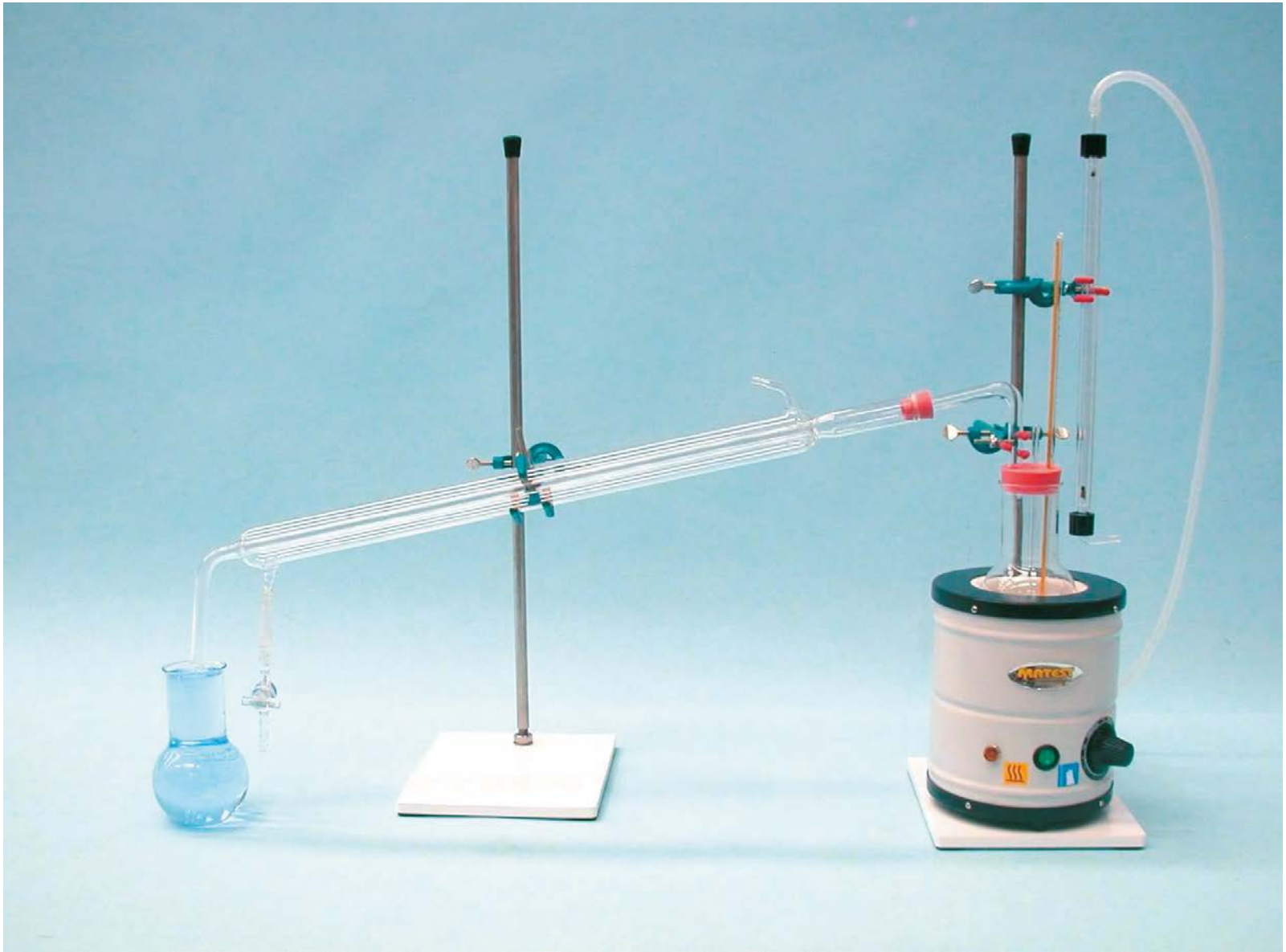
Asphalt Recovery Procedures

- Performed after or with asphalt extraction
- ASTM D5404-17
 - “Recovery of Asphalt from Solution Using the Rotary Evaporator”
- ASTM D1856-15
 - “Recovery of Asphalt from Solution by Abson Method”
- ASTM D7906-14
 - “Recovery of Asphalt from Solution Using Toluene and the Rotary Evaporator Method”

Rotary Evaporator



ASTM D1856 Absorbance Recovery



Asphalt Recovery Procedures

- AASHTO T319
 - “Quantitative Extraction and Recovery of Asphalt Binder from Hot Mix Asphalt (HMA)”
 - Modification of SHRP Procedure
 - Tumbles solvent and mix together
 - Performs extraction and recovery



Historical Recovery Procedures – Predating Abson

- Dow (1903)
 - Mix heated in oven at 300°F for 30 minutes
 - Extraction with carbon disulfide (CS₂) and simple distillation
 - Change in asphalt consistency from heating/aging measured by penetrometer
- Bateman and Delp (1927)
 - Centrifuge extraction with CS₂ and vacuum distillation

ASTM D1856 Abson Recovery

- ASTM D1856: “Recovery of Asphalt from Solution by Abson Method”
 - Simple distillation
 - Addition of carbon dioxide (CO₂) to recovery flask near end of the process
- Abson Method
 - Introduced in 1933
 - Gene Abson (Asphalt Institute Roll of Honor, 1965)

ASTM D1856 Abson Recovery

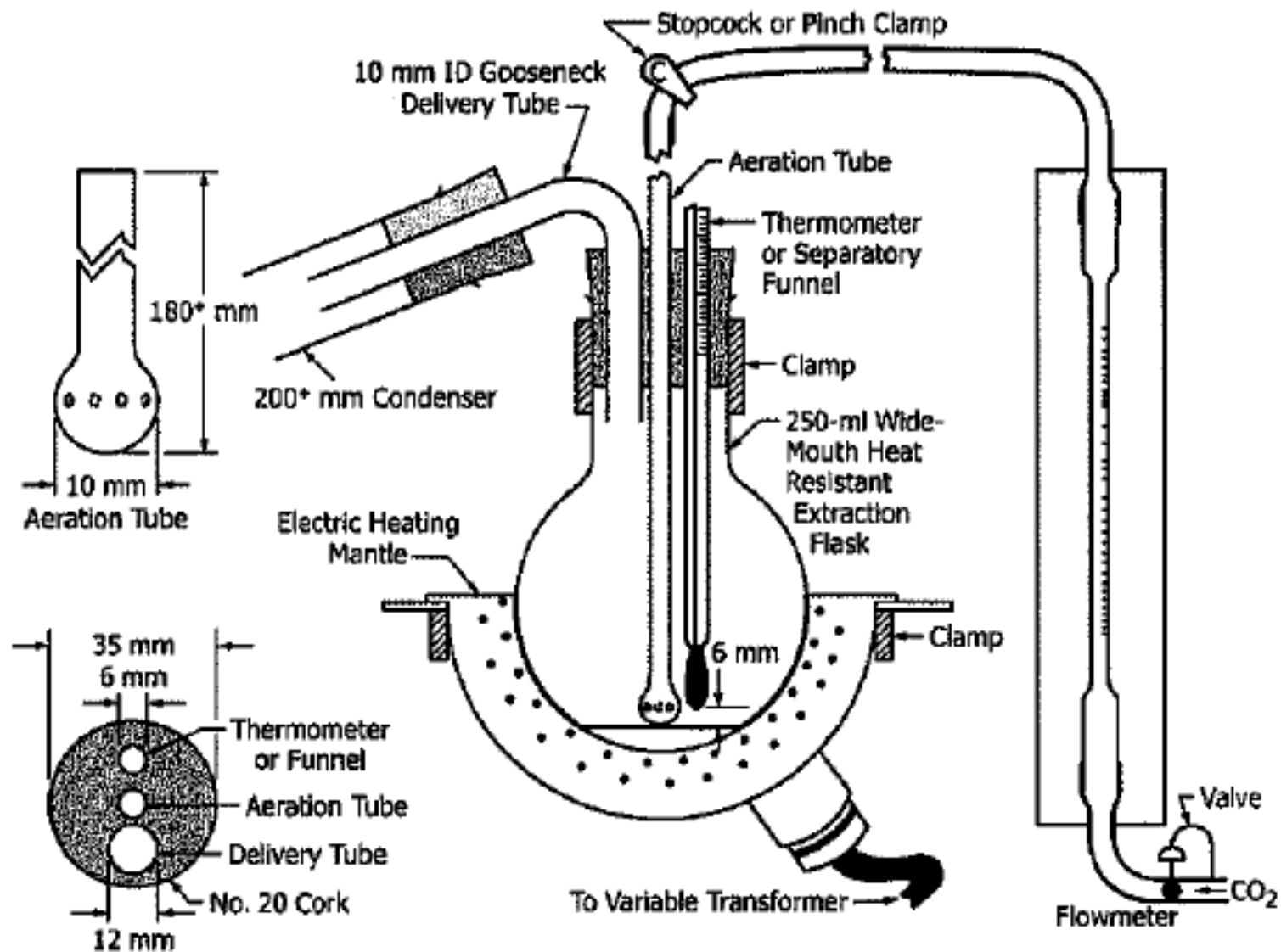


FIG. 2 Distillation Assembly for Bitumen Recovery

ASTM D1856 Abson Recovery

- Section 6: “Reagents and Materials”
 - Solvents
 - Trichloroethylene (TCE)
 - Normal Propyl Bromide (nPb)
 - Methylene Chloride
 - Note 8: It is recommended that for each new supply of the solvent a blank should be run on an asphalt of known properties

ASTM D1856 Abson Recovery

- Section 8: “Sample”
 - 8.1
 - The sample shall consist of the solution from a previous extraction by Method A of Test Methods D2172 of asphalt-aggregate mixture of sufficient quantity to result in 75 to 100g of recovered bitumen.
 - More or less may be recovered but the properties of the recovered bitumen may not be the same
 - Important that all asphalt in the mixture be extracted as there could be selective solvency
 - Harder, more viscous components of asphalt might be left in mixture if extraction not carried out to completion

ASTM D1856 Abson Recovery

- Section 8: “Sample”
 - 8.4
 - Protect against exposure to air, particularly in loose mix condition, due to hardening
 - Preferably store in airtight containers at temperature below 0°C [32°F] until ready for testing
 - Heat to maximum temperature of 110°C [230°F] for minimum time to obtain workability, but no longer than 30 minutes
 - Allow samples stored at low temperatures to reach room temperature before placing in oven

ASTM D1856 Absorbance Recovery

- Section 9: “Procedure”
 - 9.1
 - Entire procedure from start of extraction to final recovery, must be completed within 8 hours.
 - 9.2
 - Centrifuge extract from previous extraction
 - Batch process
 - 30 minutes at 770x gravity in bottles/tubes
 - Continuous extraction
 - Charge extract solution at maximum 150 ml/min rate
 - While operating at minimum centrifugal force of 3000x gravity

ASTM D1856 Absorbance Recovery

- Section 9: “Procedure” (con’t)
 - 9.3
 - Concentrate solution to ~200 ml by primary distillation using flask large enough to hold all of solution from extraction
 - Transfer into the 250 ml distillation flask



ASTM D1856 Abson Recovery

- Section 9: “Procedure” (con’t)
 - 9.3
 - Start distillation by heating flask
 - Continue until temperature reaches 135°C [275°F]
 - Lower aeration tube into sample at bottom of flask
 - Introduce CO₂ at slow rate (100 ml/min)
 - When sample temperature increases to 157-160°C [315-320°F] increase CO₂ flow to 900 ml/min

ASTM D1856 Absorption Recovery

- Section 9: “Procedure” (con’t)
 - 9.3
 - Maintain flow rate for 10 minutes while maintaining temperature at 160 – 166°C [320 – 330°F]
 - Maintain flow rate until 5 minutes after dripping ceases
 - In no case shall flow of CO₂ be less than 15 minutes
 - Discontinue flow rate and heat
 - 9.4
 - For viscous binder (Penetration < 30 dmm @25°C)
 - Maintain CO₂ flow and temperature for 20 – 22 minutes

ASTM D1856 Abson Recovery

- Section 9: “Procedure” (con’t)
 - 9.5
 - Ash content (ASTM D2939) > 1% may affect accuracy of measured properties
 - Note 1: Determination of weight of recovered asphalt serve as check that all solvent was removed when compared to weight of asphalt extracted
 - Note 1 (con’t): Correct weight of recovered asphalt for ash content

ASTM D1856 Abson Recovery

- Section 10: “Precision and Bias”
 - 10.1
 - Data from 1974 to 1992
 - Grades of Asphalt
 - AC-5, AC-10, AC-15, AC-20, AC-30, AC-40
 - AR-2000, AR-4000
 - Precision estimates
 - Penetration at 25°C 29 to 181 dmm
 - Kinematic viscosity at 135°C 200 to 720 cSt
 - Viscosity at 60°C 520 to 5320 P

ASTM D1856 Abson Recovery

- Section 10: “Precision and Bias” (con’t)
 - 10.1.2 Multi-laboratory Precision

Test Index	Single Operator Within Lab d2s% (Percent of Mean)	Multi-Laboratory d2s% (Percent of Mean)
Penetration at 25°C, dmm	30	58
Kinematic Viscosity at 135°C, cSt	26	46
Viscosity at 60°C, P	51	93

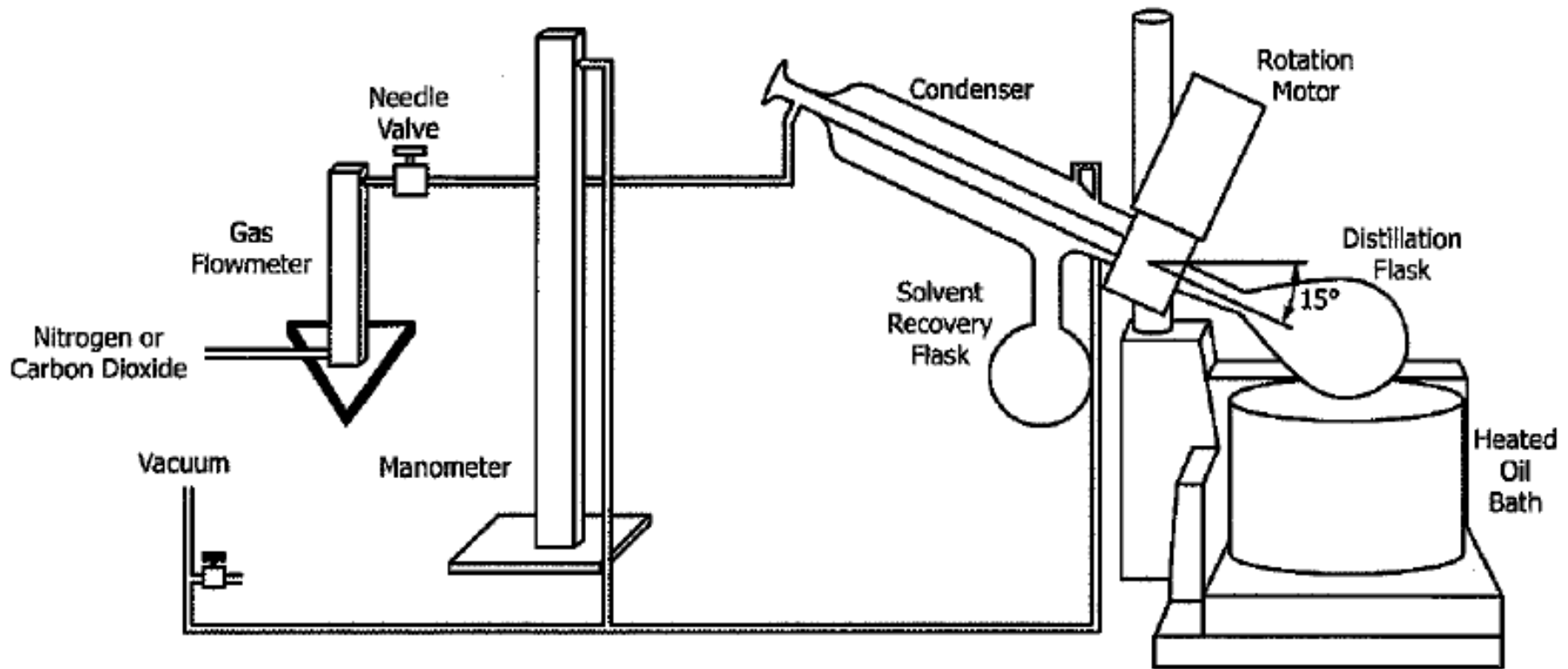
- Interpretation

- Lab 1 and Lab 2 would be within acceptable reproducibility if Lab 1 result for recovered Penetration was 56 dmm and lab 2 result was 101 dmm

ASTM D5404 Rotary Evaporator Recovery

- ASTM D5404: “Recovery of Asphalt from Solution Using the Rotary Evaporator”
 - Solution from extraction is distilled by partially immersing the rotating distillation flask of the rotary evaporator in a heated oil bath while the solution is subjected to a partial vacuum and a flow of nitrogen or carbon dioxide (CO₂) gas
 - The recovered asphalt can be subjected to testing as required
 - Procedure became popular in the 1970’s

ASTM D5404 Rotary Evaporator Recovery



NOTE 1—It is important that the needle valve is located as shown instead of being placed ahead of the flowmeter.

FIG. 1 Rotary Evaporator and Recovery System

ASTM D5404 Rotary Evaporator Recovery

- Section 6: “Reagents and Materials”
 - Solvents
 - Trichloroethylene (TCE)
 - Normal Propyl Bromide (nPb)
 - Methylene Chloride
 - It is recommended that for each new supply of solvent a blank should be run on an asphalt of known properties
- Section 8: “Sample Preparation”
 - In accordance with ASTM D1856 (Abson Recovery)

ASTM D5404 Rotary Evaporator Recovery

- Section 9: “Procedure”
 - 9.1
 - Heat oil bath to $140 \pm 3^{\circ}\text{C}$ [$285 \pm 5^{\circ}\text{F}$]
 - Circulate cold water through condenser
 - 9.2
 - Apply vacuum of 5.3 ± 0.7 kPa [40 ± 5 mm Hg] below atmospheric pressure
 - Draw ~600 mL asphalt solution into distillation flask

ASTM D5404 Rotary Evaporator Recovery

- Section 9: “Procedure”
 - 9.2
 - Start gas flow (N₂ or CO₂) at 500 mL/min
 - Note 4: Vacuum, and N₂ or CO₂ flows may need to be adjusted based on location.
 - Low flow values may be necessary at beginning to prevent back flow into vacuum system. Can also minimize back flow by regulating depth of immersion of distillation flask

ASTM D5404 Rotary Evaporator Recovery

- Section 9: “Procedure” (con’t)
 - 9.2
 - Start rotation of distillation flask at 40 RPM
 - Immerse distillation flask in oil bath at depth required to achieve controlled solvent evaporation rate
 - i.e. Steady controlled stream of condensed solvent collected in recovery flask
 - 9.3
 - When solution in distillation flask is low enough so more may be added, stop N₂ or CO₂ gas flow.
 - Add remaining asphalt solution and readjust gas flow
 - Note 5: Can use continuous flow such that liquid in distillation flask is maintained at 600 mL.
 - Start gas flow AFTER all solution has entered flask

ASTM D5404 Rotary Evaporator Recovery

- Section 9: “Procedure” (con’t)
 - 9.4
 - When most of solvent has been distilled and no obvious distillation is occurring, immerse distillation flask to maximum recommended depth (40 mm)
 - Note 6: Immerse up to 2 minutes before applying vacuum
 - Slowly apply vacuum of 80 ± 0.7 kPa [$600 \pm$ mm Hg] below atmosphere
 - Increase N_2 or CO_2 gas flow to 600 mL/min and rotation rate of distillation flask to 45 RPM

ASTM D5404 Rotary Evaporator Recovery

- Section 9: “Procedure” (con’t)
 - 9.4
 - Hold or reduce vacuum if foaming or bubbling occurs
 - When foaming stops apply maximum vacuum for 10 ± 1 min
 - Note 8: May need to increase oil bath temperature (150 – 155°C) to maintain constant sample temperature

ASTM D5404 Rotary Evaporator Recovery

- Section 9: “Procedure” (con’t)
 - 9.5
 - Remove distillation flask and wipe clean of oil at end of 10 minute period
 - Pour asphalt into container
 - If necessary for asphalt to flow, invert flask and heat in oven at $165 \pm 5^{\circ}\text{C}$ for 10-15 minutes
 - Do not allow asphalt to be distributed over interior surface of flask
 - 9.6
 - Note 9: Ash content $> 1\%$ may affect accuracy of measured properties


ASTM D5404 Rotary Evaporator Recovery

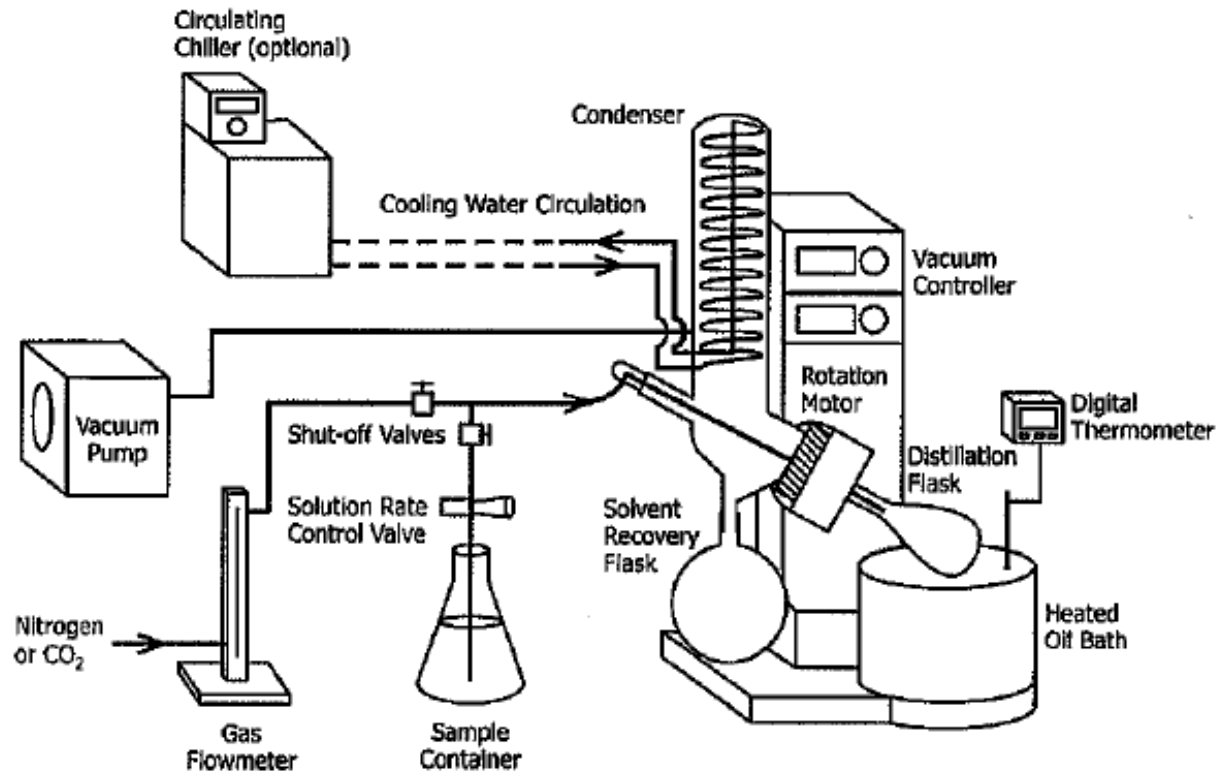
- Section 9: “Procedure” (con’t)
 - 9.6
 - Note 10: Precision
 - Data from 1992 – 1996
 - Grades – no information provided
 - Inter-laboratory precision (1s%)
 - Penetration (16 – 30%)
 - Kinematic viscosity at 135°C (10 – 22%)
 - Viscosity at 60°C (16 – 45%)
 - Ranges sufficiently larger than reported for testing original asphalts.

ASTM D7906-14 Rotary Evaporator Recovery - Toluene

- Summary is same as ASTM D5404 (Rotary Evaporator)
 - TCE boiling point = 87°C
 - nPb boiling point = 71°C
 - methylene chloride = 39.8°C
- Modification of ASTM D5404 allows use of toluene solvent
 - Toluene has a higher boiling point (110.6°C)
 - Requires procedure modification
- Asphalt Institute (AI) advanced at ASTM
 - Approved in 2014

ASTM D7906-14 Rotary Evaporator Recovery - Toluene

 D7906 - 14



NOTE 1—The vacuum pump and vacuum controller are recommended for maintaining the required vacuum in 5.1. The diagram displays a Büchi model rotary evaporator.

FIG. 1 Rotary Evaporator and Recovery System

ASTM D7906-14 Rotary Evaporator Recovery - Toluene

- Section 6: “Reagents and Materials”
 - Toluene Solvent
 - ACS Grade that does not contain 1,2-epoxybutane as stabilizer
 - 1,2-epoxybutane can cause with asphalts containing acids
- Section 8: “Sample Preparation”
 - Same as for Abson, Rotavapor

ASTM D7906-14 Rotary Evaporator Recovery - Toluene

- Section 10: “Procedure”
 - 10.1.1
 - Heat oil bath to 150°C
 - Circulate cold water through condenser
 - 10.1.2
 - Draw 200-300 mL solution into distillation flask
 - Amount may be increased as long as no back flow occurs into condenser

ASTM D7906-14 Rotary Evaporator

Recovery - Toluene

- Section 10: “Procedure”
 - 10.1.3
 - Start rotation of distillation flask at ~40 RPM
 - Note 8: May need to reduce to 30 RPM for stiff materials
 - Slower spin = increased surface area exposure to solvent
 - 10.1.4
 - Maintain 200 – 300 mL of solution in distillation flask until all solution has been added
 - Continuous feed solution or feed in batches every 3-5 min
 - Never allow solution to go below 200 mL
 - Avoid overheating / premature aging

ASTM D7906-14 Rotary Evaporator

Recovery - Toluene

- Section 10: “Procedure”
 - 10.1.3
 - When most of solvent has been removed (i.e. drip from condenser is 1 drop per 15 sec)
 - Slowly increase vacuum to 6.7 ± 0.7 kPa
 - Hold or release vacuum if foaming/bubbling occurs
 - When foaming subsides, apply max vacuum
 - 10.2.2
 - After condensate drip rate returns to 1 drop per 15 sec
 - Turn on N₂ or CO₂ gas flow rate at 600 L/min
 - Maintain gas purge and maximum vacuum for 45 ± 2 min
 - Note 12: Increase in oil bath temperature generally required to maintain sample temperature

ASTM D7906-14 Rotary Evaporator

Recovery - Toluene

- Section 10: “Procedure”
 - 10.2.3
 - Removed distillation flask and wipe clean after 45 minute period from previous step (gas purge/max vacuum)
 - Pour recovered binder into sample container
 - May heat inverted flask in oven at $165 \pm 1^{\circ}\text{C}$ for 10-15 min
 - Note 13: Higher temp / longer time may be required for stiffer binders
 - 180°C for RAP
 - 200°C for RAS
 - Do not exceed 200°C

ASTM D7906-14 Rotary Evaporator Recovery - Toluene

- Precision has not yet been published
- Internal Ruggedness Evaluation conducted

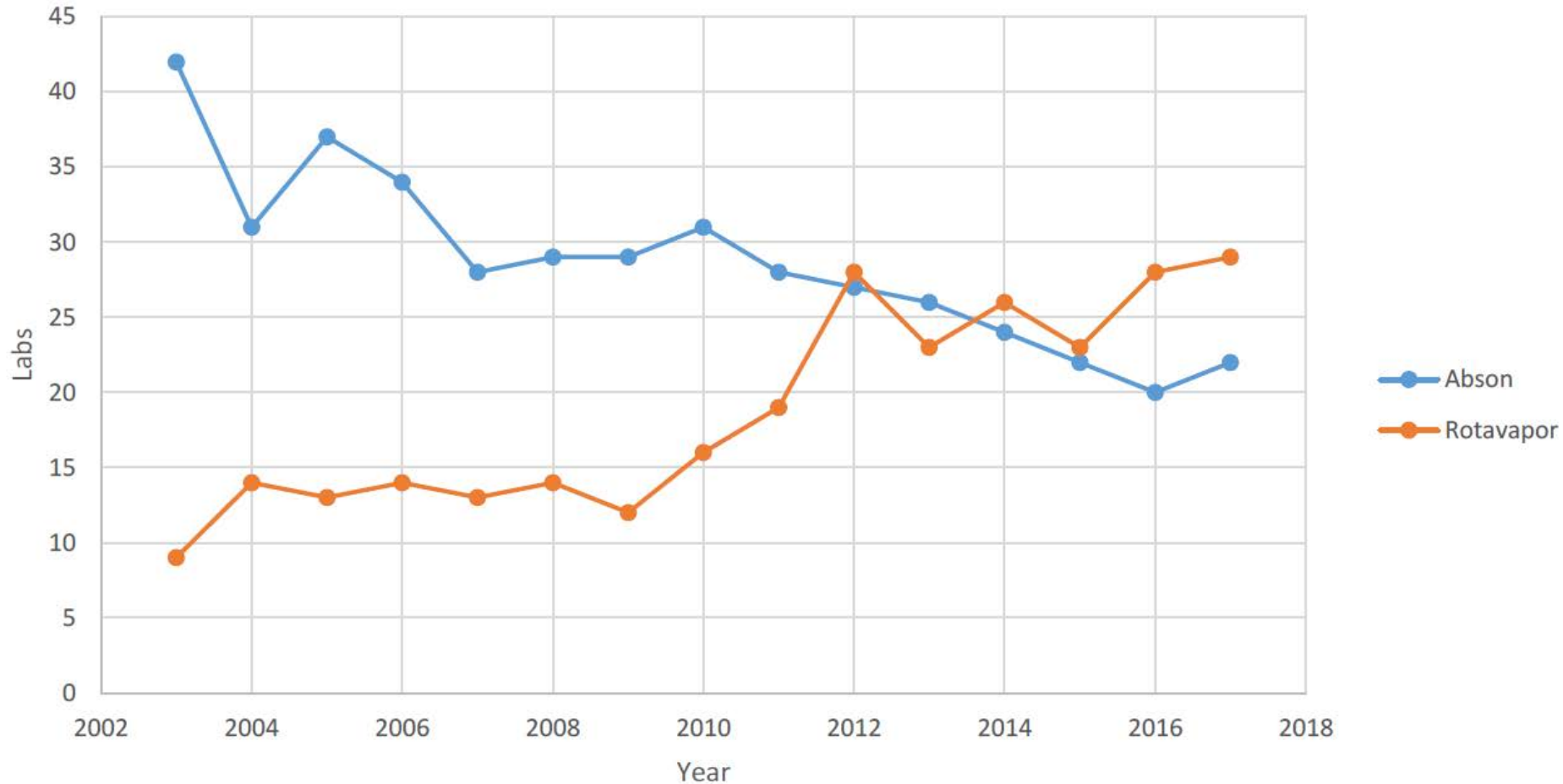
AASHTO T-319 Quantitative Extraction and Recovery of Asphalt Binder from HMA

- Comprehensive procedure
 - Modified version of SHRP procedure
 - Tumbles mix and solvent together
 - Extracts binder from mix
 - Removes fine aggregates
 - Recovery using modified Rotavapor procedure
- Advantage
 - Less solvent hardening
- Disadvantage
 - Smaller mix sample (less recovered binder)

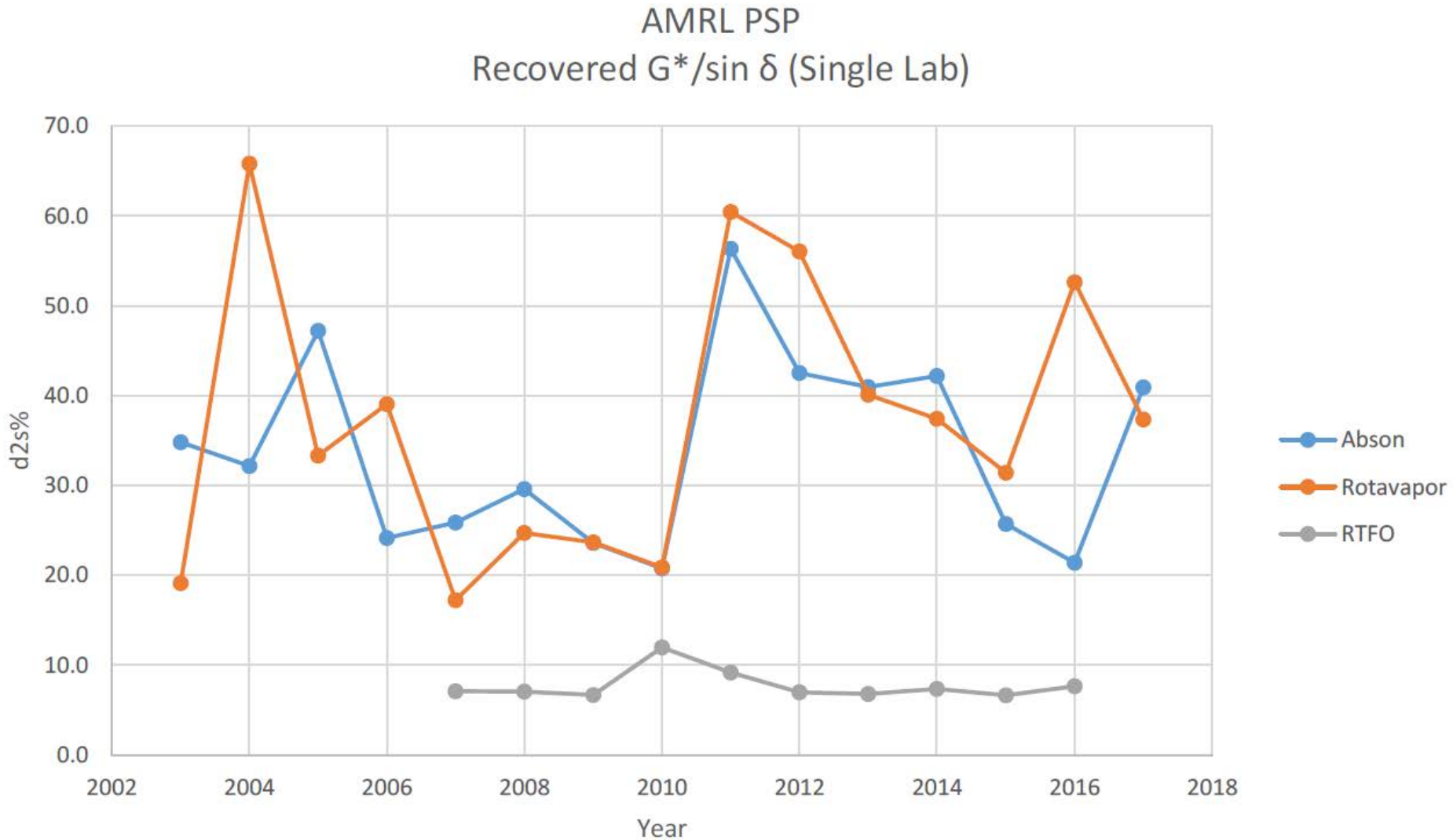


AMRL Proficiency Sample Program Analysis

AMRL PSP
Recovered $G^*/\sin \delta$

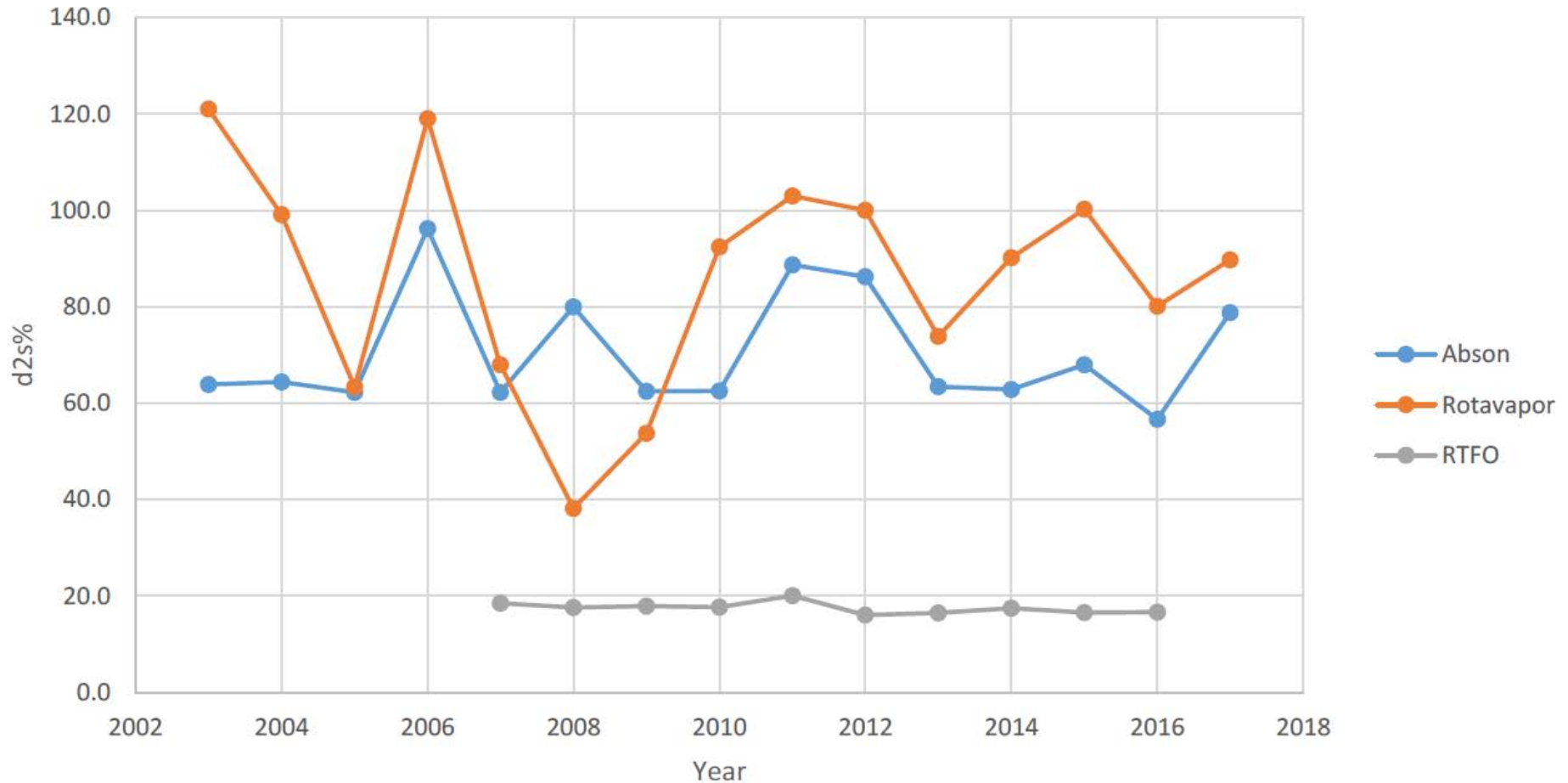


AMRL Proficiency Sample Program Analysis



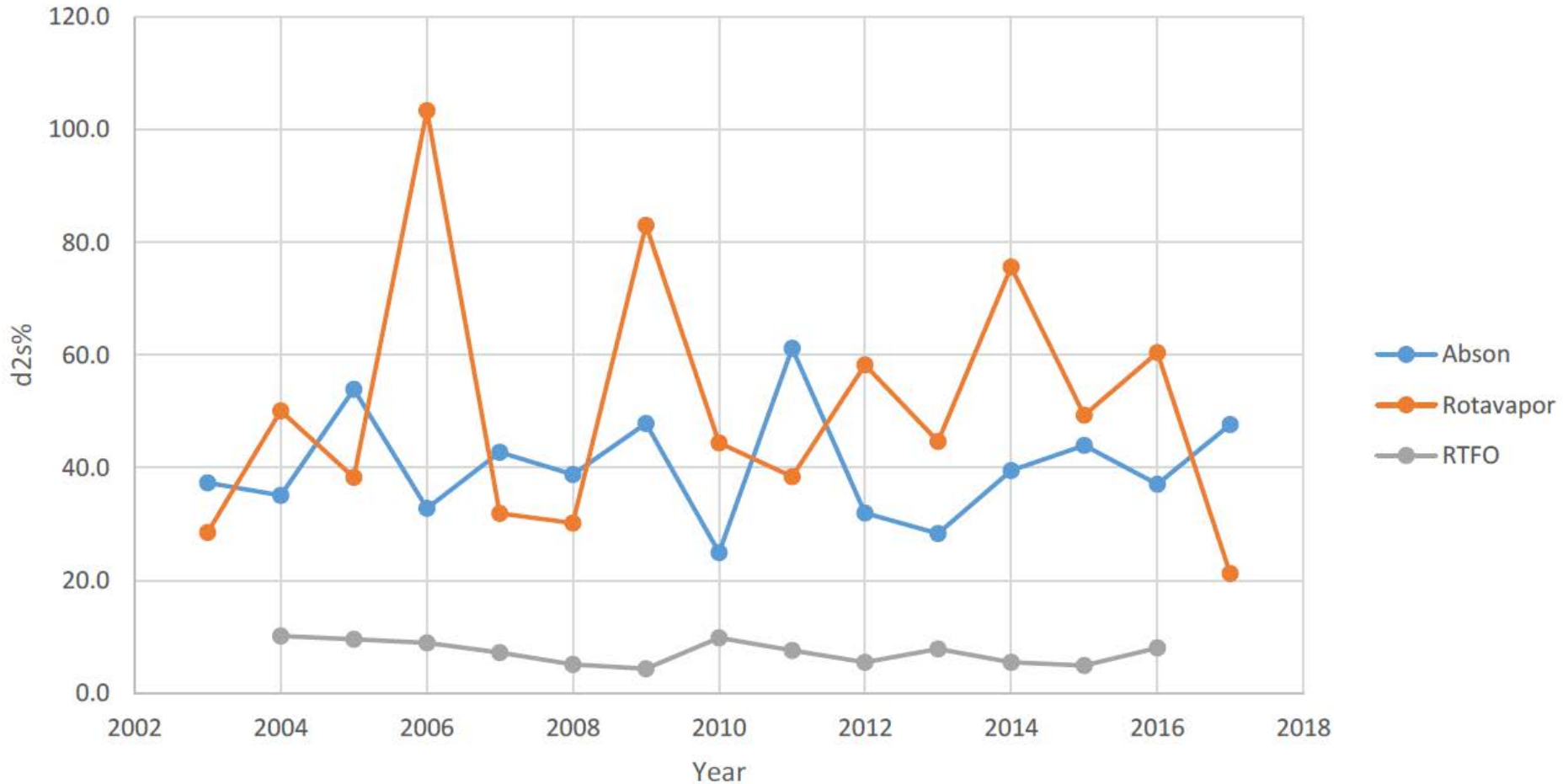
AMRL Proficiency Sample Program Analysis

AMRL PSP
Recovered $G^*/\sin \delta$ (Multi-Lab)



AMRL Proficiency Sample Program Analysis

AMRL PSP
Recovered Absolute Viscosity (Single Lab)



NCHRP 9-12 Research

Table 17. Average High Temperature Stiffness and Critical Temperature of Extracted RAP Binders

Extraction	Centr.	Centr.	SHRP	SHRP	SHRP	Centr.	Centr.	SHRP	SHRP
Recovery	Abson	Rotavap	Rotavap	Rotovap	Rotavap	Abson	Rotavap	Rotavap	Rotavap
Solvent	TCE	Tol/Eth	Tol/Eth	NPB	TCE	TCE	Tol/Eth	Tol/Eth	NPB
G*/sin δ	KY	KY	KY	KY	KY	FL	FL	FL	FL
64C	6.34	33.16	24.43	26.74	21.47	3.00	9.29	7.33	4.22
COV-64	69%	12%	11%	26.2%	14%	48%	20%	5%	20.6%
70C	2.98	13.84	10.13	11.12	9.02	1.43	3.96	3.18	1.85
COV-70	64%	11%	12%	24.0%	12%	44%	21%	5%	20.1%
76C	1.42	5.81	4.36	4.77	3.92	0.70	1.77	1.44	0.85
COV-76	60%	12%	13%	20.5%	10%	38%	20%	5%	23.0%
T _c	77.8	88.1	86.4	87.0	85.8	72.4	80.2	78.8	74.7
COV-T _c	5.6%	1.0%	1.2%	1.1%	0.5%	4.7%	1.8%	0.4%	2.3%

- Centrifuge-Abson-TCE treatment
 - Lowest G*/sin δ and poorest repeatability
 - Confirms previous SHRP research
 - Abson has poor repeatability susceptible to incomplete solvent removal
 - More apparent with harder RAP (harder KY RAP has greater delta between Abson and Rotavapor than softer FL RAP)

NCHRP 9-12 Research

- Rotavapor recovery treatments have similar precision with COV much lower than Abson (5-20% vs. 38-69%)
- Centrifuge-Rotavapor-Toluene/Ethanol
 - Highest $G^*/\sin\delta$ for both RAP sources owing to two possibilities:
 - 1) More fines removed from effluent from SHRP extraction procedure vs. centrifuge extraction (excess fines could stiffen binder)
 - 2) Additional hardening occurring with standard Rotavapor vs. SHRP Rotavapor procedure
 - Modified procedure uses lower temperature (may help minimize hardening) and higher vacuum

NCHRP 9-12 Research

- Toluene-ethanol vs. n-propyl bromide
 - Statistically equivalent results with the with centrifuge-Rotavapor extraction-recovery procedure

- Reference
 - NCRHP 9-12 Recommended Use of Reclaimed Asphalt Pavement in the Superpave Mix Design Method (2000).

SHRP Research

- Volatile loss from unaged asphalt binders during solvent removal can produce 7-10% hardening
 - Not apparent with RTFO-aged binders
- Same asphalt binders show 10-40% hardening after exposure to TCE and solvent removal
 - Extended time at elevated temperature (such as reflux extraction) can produce significant hardening
- Reference
 - Burr, B.L., R.R Davison, C.J. Glover, and J.A. Bullin. “Solvent Removal from Asphalt”, Transportation Research Record 1269, 1990.

SHRP Research

- Standard Abson procedure can leave enough solvent in binder to produce significant softening
 - Especially true for larger recovered quantities and hardened asphalt binders
- Rotavapor procedure appears to be less consistent and reproducible than the Abson procedure but may have advantages for solvent removal
- Reference
 - Burr, B.L., R.R Davison, C.J. Glover, and J.A. Bullin. “Solvent Removal from Asphalt”, Transportation Research Record 1269, 1990.

Summary

- Removal of solvent is critical during recovery procedures
 - Absorption standard procedure may not perform as well, particularly for hardened asphalt binders
- Variability in recovered asphalt binder properties is approximately 3x greater than the variability of a non-recovered asphalt binder
- Variability appears to be a function of recovery, not the specific recovery procedure
- Technique/skill matters and impacts results



QUESTIONS & DISCUSSION



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