

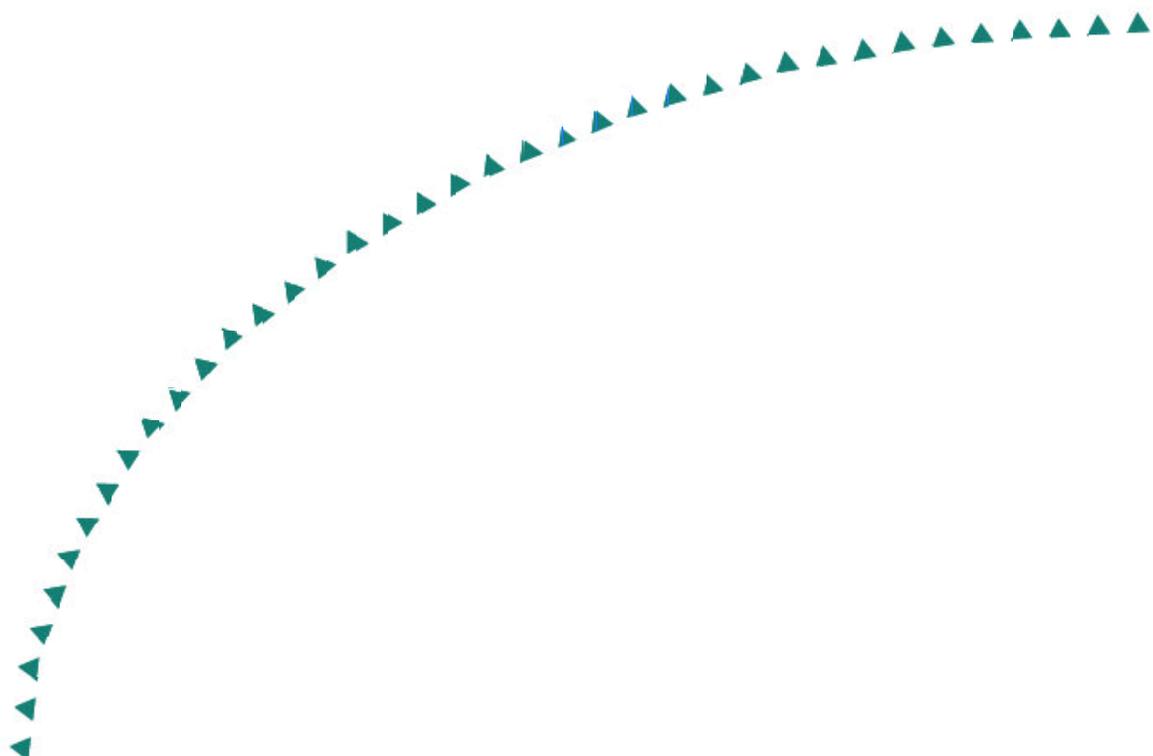
2003-35

Final Report

Selection of an Alternative Asphalt Extraction Solvent



Research



Technical Report Documentation Page

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<p>The asphalt extraction solvent n-Propyl Bromide (nPB) was selected as an alternative solvent to the currently used d-Limonene and Trichloroethylene (TCE), based on a literature search, communication with asphalt technology experts, and a laboratory investigation. With proper handling techniques and training, nPB can be substituted for d-Limonene and TCE. Extractions with nPB use less solvent per extraction, take less time, and have a shorter drying time. Exposure levels to nPB for the labs were far below the EPA recommended value.</p> <p>The use of nPB for asphalt extraction and the implementation of nPB on-site recycling would generate only non-hazardous still bottom waste. The use of nPB would decrease the hazardous waste of Mn/DOT laboratories and reduce annual solvent costs. Payback time for the purchase of solvent recovery units ranges from 3 months to 20 years, depending on the quantities of extraction waste generated per year. It is recommended that all Mn/DOT district laboratories change from d-Limonene or TCE extraction solvents to nPB and conduct on-site recycling with the purchase of solvent recovery systems, and that training be given to lab personnel on the proper handling techniques for nPB.</p>			
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SELECTION OF AN ALTERNATIVE ASPHALT EXTRACTION SOLVENT

Final Report

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EXECUTIVE SUMMARY

Flash Point testing on d-limonene asphalt extracts sampled from Minnesota Department of Transportation (Mn/DOT) laboratories indicated that the hot mix asphalt (HMA) extraction waste would be classified as hazardous. Previous flashpoint testing done for the waste profile by the hazardous waste disposal company didn't agree with those conducted on Mn/DOT's waste stream in 2000. This waste had been previously thought of as non-hazardous. The increase in hazardous waste volume would place Mn/DOT's District Materials Labs in a larger hazardous waste generator size and with it larger disposal and licensing costs and added regulation requirements.

Based on information gathered through a literature search and lab investigation, an n-propyl bromide (nPB) product was selected as an alternative asphalt extraction solvent for d-limonene.

By conducting a cost benefit analysis for on-site solvent recycling and a solvent user extraction study, it was shown that a change to nPB and on-site recycling would give Mn/DOT laboratories cost savings in hazardous waste disposal fees, shorter extraction test turn-around times and lower solvent costs.

It is recommended that all Mn/DOT District Labs change from TCE or d-limonene extraction solvents to nPB and conduct on-site recycling with the purchase of solvent recovery systems. It is also recommended that Mn/DOT Industrial Hygienist give lab personnel training on the proper handling techniques when using nPB.

INTRODUCTION

In an effort to move away from chlorinated solvents like Trichloroethylene (TCE) and 1,1,1 Trichloroethane (TCA) for chemical asphalt extractions, Mn/DOT conducted an extraction solvent study to choose an alternative solvent. In that 1994 study, a biodegradable d-Limonene based solvent was selected. The extract waste having a flash point above 140 °F then could be disposed of as a non-hazardous fuel blend. Unfortunately the flashpoint tests done for the waste profile by the hazardous waste disposal company didn't agree with those conducted on Mn/DOT's waste stream. d-Limonene asphalt extract sampled recently from Mn/DOT laboratories indicated that the extraction waste would be classified as hazardous based on a flash point lower than 140 °F. This increase in hazardous waste volume would place the district labs in a larger hazardous waste generator size and with it larger disposal and licensing costs and added regulation requirements.

The Office of Environmental Services/ Environmental Compliance and Investigation Unit requested that the Chemical Laboratory investigate replacement extraction solvents for d-Limonene. Solvent suppliers were contacted for potential replacement candidates with the requirement that the flash point exceed 140 °F. Mn/DOT's Chemical Testing Unit investigated 12 alternative solvents and all but two were eliminated from the study based on residue on heating and solubility with asphalt. A high flashpoint d-Limonene and an n-propyl bromide (nPB) solvent were further evaluated for suitability as an extraction solvent. Based on a literature search, communication with asphalt technology experts and results from our lab investigation, the n-propyl bromide product was selected as an alternative solvent for d-limonene.

A user survey and a cost benefit analysis showed that purchasing solvent recovery systems and doing on-site recycling could save both time and money. Recommendations were made to Mn/DOT's Lab Supervisors and Waste Coordinators groups on solvent recovery systems and the change to nPB for chemical extractions.

BACKGROUND

The two solvents presently used for chemical extractions at Mn/DOT are TCE and d- Limonene. TCE has been identified as a carcinogen and overexposure can cause dizziness, headaches and even death. Heating of TCE can create poisonous phosgene gas and create acid when coupled with moisture. The Clean Air Act of 1990, Volatile Organic Compounds (VOC) and Resource Conservation and Recovery Act (RCRA) regulations make the use of TCE expensive and difficult [1]. Mn/DOT's District 3 Laboratory estimate disposal costs on a 55-gallon drum of TCE waste to be about \$300.

The biodegradable d-Limonene solvent disposal costs are much less (\$50-60 per 55 gallon drum) but extracted aggregate must be washed with water and a surfactant to get rid of oily residue left by extraction process. These extra steps add time to extraction test turn-around time. With most DOT labs under a time line to get results to contractors, an alternative solvent needed to be found. Additionally it was determined by flashpoint testing by Mn/DOT's Asphalt Binder Lab that the d-Limonene extract should be classified as hazardous. The profile testing done by the hazardous waste disposal company initially showed that the extraction waste would be non-hazardous and could be disposed as a fuel blend. This change in properties of the extract waste put many of the District labs into a larger hazardous waste generator size. Moving into a larger waste generator size requires time-consuming waste management reporting and increased disposal and licensing costs.

The NCHRP 9-12 research project showed that nPB could be used as an extraction solvent without affecting asphalt cement contents and binder properties. This was determined by comparing nPB extraction test results with those of TCE and toluene. The 9-12 project panel is proposing revisions to AASHTO on TP2-Standard Test Method For Quantitative Extraction and Recovery of Asphalt Binder From Asphalt Mixtures to include nPB as an extraction solvent [2].

Research done for the Florida Department of Transportation found that Solubility and Extraction testing with different asphalts showed no statistical difference between using TCE or nPB as an extraction solvent. No difference was seen with using fresh nPB or recovered nPB on extraction results. It has been recommended that fresh nPB be used when recovering asphalt for binder testing. nPB solvent extractions took less time than the TCE extractions. Researchers concluded that nPB could be substituted for TCE as an extraction solvent without change to current test methods.

nPB is classified non-hazardous and non-carcinogenic according to hazardous waste rules unlike many of the chlorinated solvents now in use. nPB is classified as an eye and skin irritant and ordinary handling and ventilation precautions are needed to avoid headaches, dizziness and nausea [3]. This was confirmed by observing labs using nPB and User Survey Results. Some users objected to the different smell but the same response was given when Mn/DOT changed to the orange smelling d-Limonene. Poor handling techniques and using the nPB solvent outside a ventilation hood were observed during exposure monitoring by an industrial hygienist. Even with poor technique, the exposure levels for two different labs were far below EPA recommended value of 25 ppm calculated 8 hour Time Weighted Average (TWA). Lab 1 results were 1.8-ppm nPB 8 hour TWA in the subject's personal breathing zone and 2.7 ppm 8 hour TWA in the lab

area. Lab 2 results were 1.4-ppm nPB 8 hour TWA in the subject's personal breathing zone and 2.1 ppm 8 hour TWA in the lab area.

Work done by the National Center for Asphalt Technology (NCAT) at Auburn University, showed that nPB can be used as a drop-in replacement for TCE. They did however find that not all nPB solvents are created equal. One nPB solvent showed some incompatibility with polymer-modified asphalts. These incompatibilities seem to be correlated to differences in nPB purity and types of stabilizer additives in the solvents formulation. NCAT recommended that before going to a solvent replacement an evaluation should be made to ensure test results are similar to historical data. [4]

Mary Stoup Gardner, NCAT, indicated that nPB could be distilled and recycled just as TCA and TCE. Extractions run with nPB by centrifuge methods took the same time as TCE. Asphalt recovery times were the same except there was a tendency for faster rotary evaporator recoveries with nPB. She indicated that she was proposing to ASTM to add nPB as an extraction solvent used in ASTM D2174 Standard Test For Quantitative Extraction Of Bitumen From Bituminous Paving Mixtures and ASTM D5404 Standard Practice For Recovery Of Asphalt From Solution Using The Rotary Evaporator.

Tessa Beuchler from Petrofirm, an nPB supplier confirms that purity should be part of an nPB solvent specification. Keeping isopropyl bromide levels below 1000 ppm should assure higher purity levels. ASTM has added a 1000-ppm limit for isopropyl bromide to their D6368-00 Standard Specification for Vapor-Degreasing Grade and General Grade Normal-Propyl Bromide. Additionally acid levels must be monitored to assure corrosion resistance is maintained. If not, solvents in the presence of moisture may generate acids that may react with steel and aluminum metals in extractors. Acid testing kits purchased from manufacturers are used to test acid levels in recycled nPB and are used to determine how much solvent stabilizer must be added to recycled nPB to bring acid levels down. Solvent stabilizers can be purchased from solvent suppliers.

The FHWA Central Federal Land Materials Lab indicated they have used nPB with much success. They recover nPB using a solvent recovery system. In the solvent recovery process, hazardous waste testing on still bottoms was found to be non-hazardous. Comparative testing for asphalt content and sieve analysis of remaining mineral matter using nPB, TCA and ignition oven show no statistical difference in the test results. Study results showed that nPB leaves no residue, proved to be an excellent cleaner, had a strong but not objectionable odor and had a fast evaporation rate.

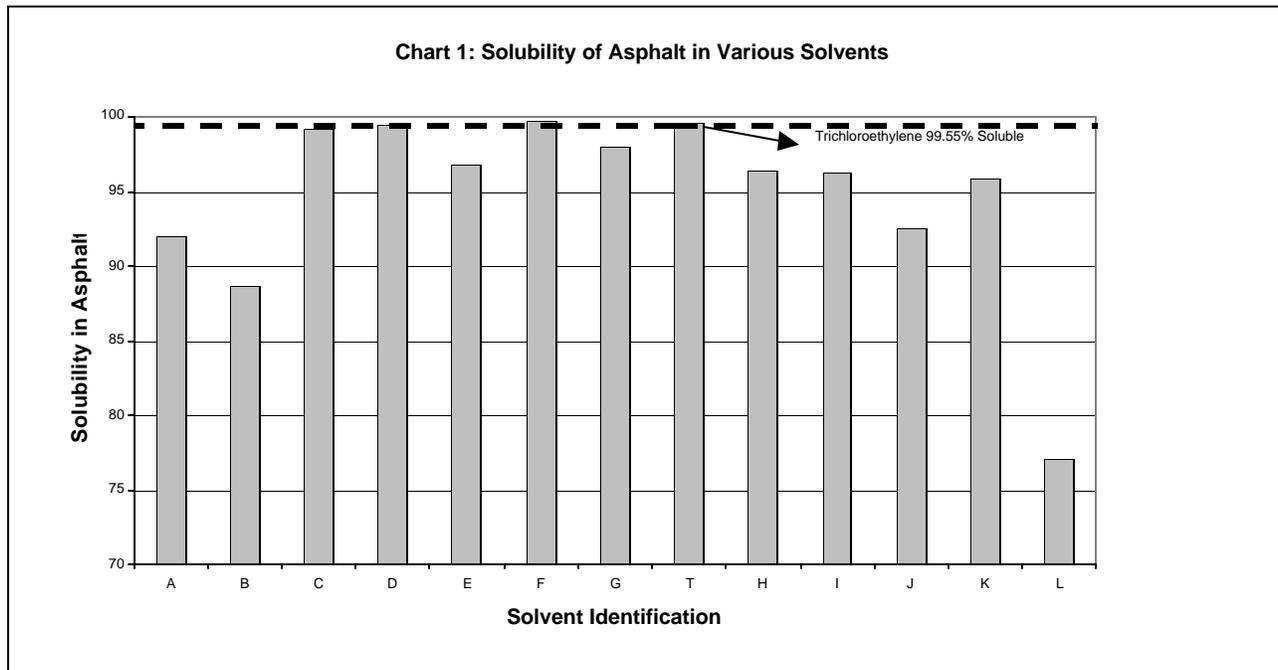
Based on human in-vitro comparative bioassays of solvents, EnviroMed Labs concluded that no significant toxicity existed for nPB and stabilized nPB at concentrations up to 400 ppm. These tests were conducted on seven key solvents side-by-side keeping time, place and test protocols as close to the same as possible. The seven solvents were ranked by their comparative relative human toxicity as follows: TCE > Perchloroethylene (PCE) > Isopropyl Bromide (iPB) > stabilized nPB/nPB > Methylene Chloride [5].

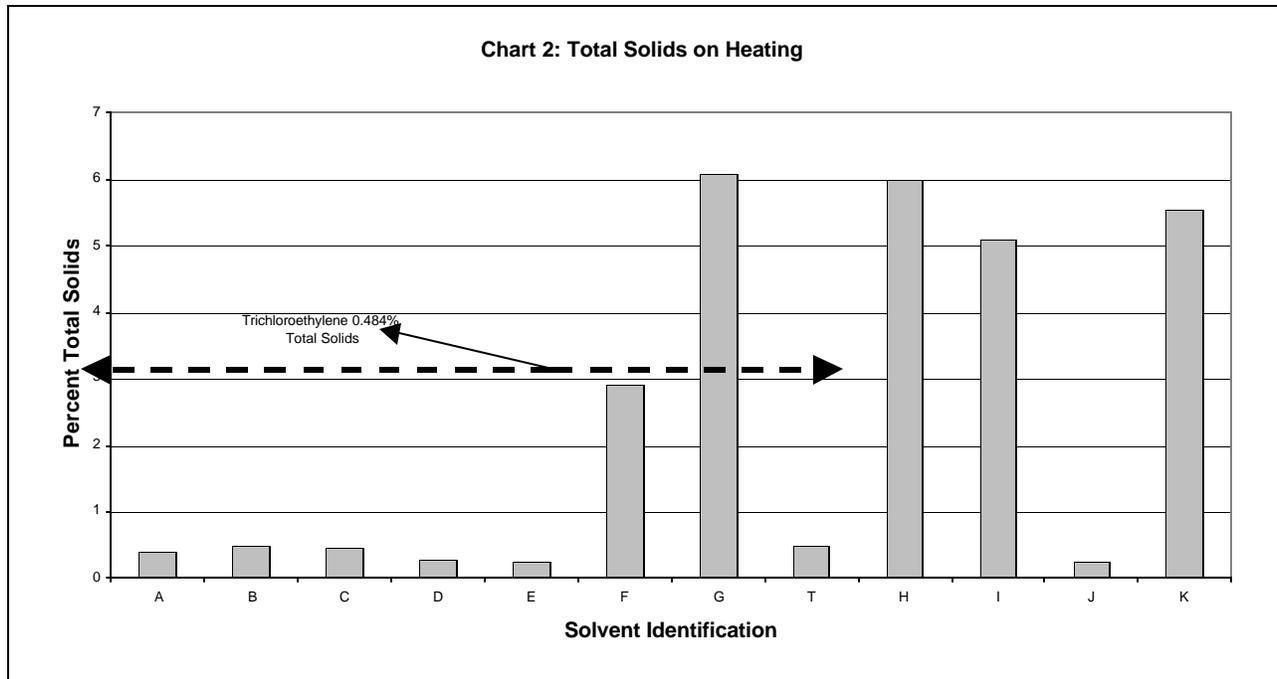
SOLVENT SELECTION PROCESS

The Office of Environmental Services polled solvent suppliers for a replacement solvent to the d-Limonene extraction solvent. 12 solvents were sent to Mn/DOT's Chemical Lab for evaluation. Product data sheets and Material Safety Data Sheets for the candidate solvents were reviewed and evaluated for hazards and compliance with environmental rules. Since TCE historically has been a very good solvent for chemical asphalt extractions it was chosen as the extraction standard to measure a replacement by [1].

Results

Charts 1 and 2 show that when combining solubility and total solids data, Solvent C and Solvent D best compare with TCE. Solvents C and D were chosen for further evaluation based on these tests. Solvent C is a high flash point d-Limonene product and Solvent D is an nPB based solvent. Table 3 shows a comparison of extractions with Solvent C and D. The extraction test with nPB (Solvent D) was faster, took less solvent and was less involved than d-Limonene extraction (Solvent C). Because the D-Limonene product leaves a greasy residue on the aggregate during the extraction process, washing with water and a surfactant is necessary. No aggregate washing was necessary with nPB. nPB is classified non-flammable and has a higher vapor pressure allowing for faster drying times.





Two 1999 Mn/DOT Interlab Round Robin samples containing 6.00% asphalt binder was extracted with nPB and compared to District lab results from these samples. Round Robin data showed that laboratories using d-Limonene solvent or TCE averaged 6.00 with a standard deviation of 0.093. The extractions with nPB gave 6.03% and 5.99% respectively. Extraction data by others confirmed that nPB could be used as a drop-in replacement for TCE using standard extraction methods. [2][3][4]. nPB was selected as the final choice based on tests, literature search & communications with asphalt technology experts and solvent suppliers.

USER EXTRACTION STUDY

In an effort to poll the state District Materials Labs on the use of nPB, a User Extraction Study was conducted. Two different bituminous mixes were sent to the District labs in replicate with instructions for extraction with nPB. One of the mixes contained 30% RAP and the other was a virgin limestone mix. The labs extracted each mix with the solvent they presently use and one with nPB. nPB solvent was distributed with the samples to each of the participating labs. Each lab was instructed to keep track of times required for testing and any difficulties encountered. A survey was also sent to all labs to get user impression with nPB. Table 1 below shows the extraction results on the two mixes by solvent type. Chart 3 shows data for extraction time - savings for all laboratories and Charts 4 and 5 show data for the individual test mixes. Extraction data for these mixes and the results of the lab survey can be found in the Appendix.

Table 1: Mn/DOT User Extraction Study Results- Solvent Comparison

	Limestone Mix		30% RAP Mix	
	Average	S.D.	Average	S.D.
d-Limonene	6.04	0.26	6.27	0.31
nPB	6.02	0.22	6.15	0.20
TCE	6.13	0.20	6.36	0.26
All	6.07	0.23	6.30	0.28

Results and Analysis for the User Extraction Study

1. Extraction time savings range from -15 minutes to 3.5 hrs
2. Quantities of solvent used for each extraction varied amongst labs – The volumes ranged from 0.75 L to 3.5 L
3. Extractions using TCE averaged 0.22 % higher AC than d-Limonene or nPB. Either TCE extracts more asphalt from the aggregate or allows more fines to pass through centrifuge filter paper.
4. Extraction results show that d-Limonene & nPB gave the same % AC
5. Testing Bias was generated by some labs not following provided study instructions or by showing preference for a certain solvent.
6. Labs presently using d-Limonene will notice a time-saving
7. Labs presently using TCE will notice disposal savings
8. Observations in two of the labs on the extraction process indicate that training in handling of solvents is necessary
9. Average nPB savings are 0.7hr or about \$11per extraction based on Mn/DOT average technician salary.

Chart 3: Total Extraction Time Savings Using nPB

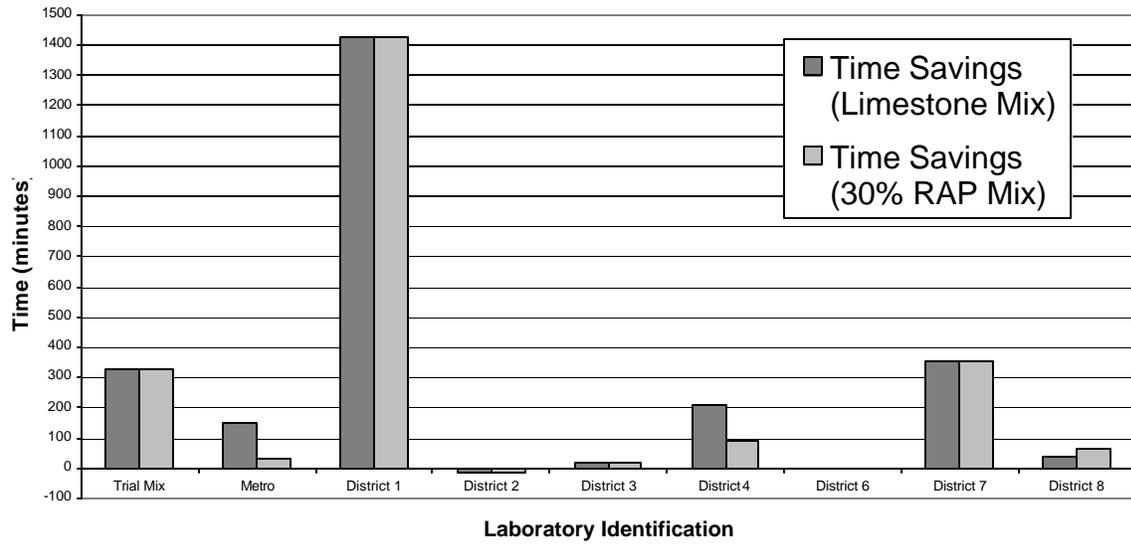
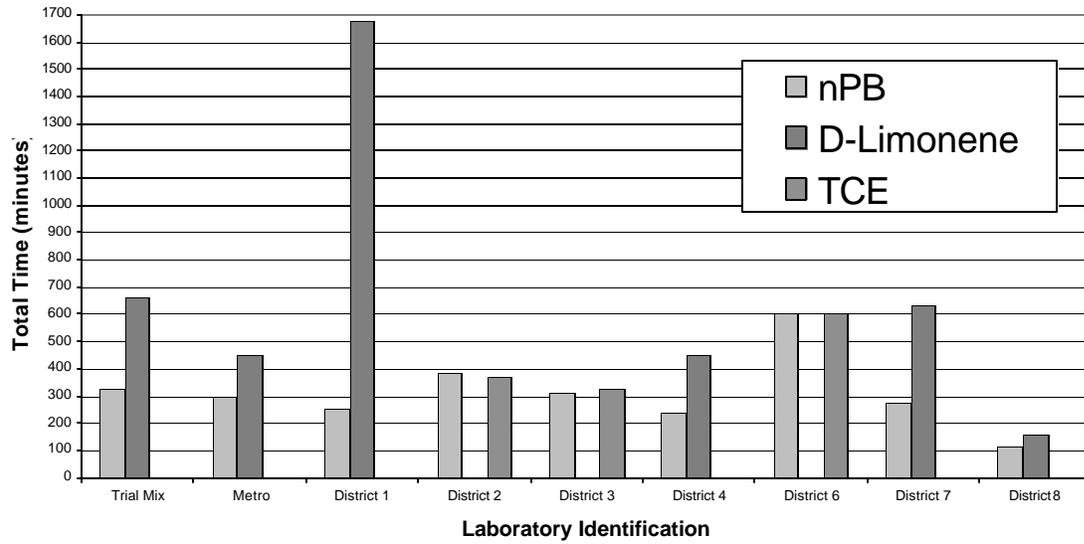
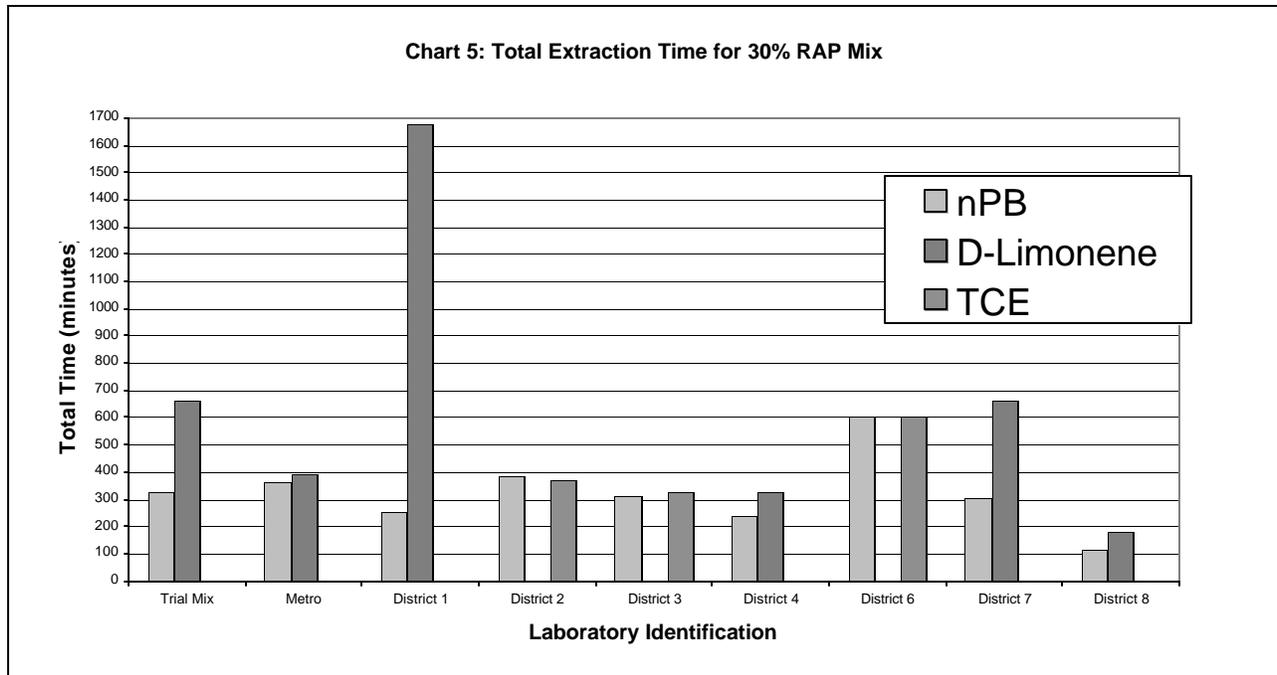


Chart 4: Total Extraction Time for Limestone Mix





Survey Conclusions :

1. The odor of the nPB didn't seem to be a problem to the users.
2. Respondents acknowledged that extractions were quicker with nPB.
3. Mixed feelings were seen on the question of recycling. Possible reasons for this are additional workload for technicians and not knowing the cost of solvent recovery systems.
4. The question on which solvent was best liked gave conflicting results considering vote to change solvent (Question 6) and quicker extraction times. Some bias was seen due to resistance to change.
5. Time and disposal savings for the most part were acknowledged.
6. The majority of the respondents voted to change, either immediately or next construction season.

COST BENEFIT ANALYSIS

Since the cost of nPB is expensive, recycling strategies were evaluated. Off-Site solvent recycling companies required very large quantities of extract to recycle. This option was eliminated since each individual lab would not generate enough waste for recyclers to take on this business. A central solvent recovery location was considered, but distance between labs can be considerable. On-site recycling appeared to be a very promising option in that solvent recovery could be done at each of the district labs.

A cost benefit analysis was conducted to determine how long it would take the labs to recover the cost of the solvent recovery unit and nPB. Individual lab waste was estimated by multiplying the number of extractions per year by 1.75 (volume of solvent needed to run each extraction). The Cost Benefit Analysis detailed in Table 8 includes cost for: solvent, waste disposal, purchase of solvent recovery unit, maintenance of solvent recovery unit and electricity. The Cost Benefit Analysis does not include costs associated with waste management at a higher generator size. In 2000 the additional cost for Minnesota Pollution Control Agency Hazardous Waste Licensing by the District 3 Laboratory to go from a Very Small Quantity Generator to Small Quantity Generator size was \$575 + \$200 Hazardous Waste Generator Tax.

Conclusions from Cost Benefit Analysis

1. Payback depending on the volume of waste generated by each lab per year
2. Payback time ranges from 3 months to 20 years
3. Dealing with safety concerns, start-up costs and procedural changes the labs could see savings in dollars and labor costs

SAFETY

Mn/DOT's Industrial Hygienist evaluated nPB potential hazards and suggested handling precautions. nPB has similar hazards and precautions as TCA and TCE. nPB is classified, as eye and skin irritant and ordinary handling and ventilation precautions are needed to avoid headaches, dizziness and nausea.

Poor handling techniques were observed during exposure monitoring by the industrial hygienist. Even with poor technique, the exposure levels for two different labs were far below EPA recommended value of 25 ppm calculated 8 hour Time Weighed Average (TWA). Lab 1 results were 1.8-ppm nPB 8 hour TWA in the subject's personal breathing zone and 2.7 ppm 8 hour TWA in the lab area. Lab 2 results were 1.4-ppm nPB 8 hour TWA in the subject's personal breathing zone and 2.1 ppm 8 hour TWA in the lab area. It is recommended that Mn/DOT's Safety Office conduct training sessions on the handling and use of nPB.

CONCLUSIONS:

1. Based on a literature search, communication with asphalt technology experts and results from our lab investigation, the n-propyl bromide (nPB) product was selected as an alternative solvent for d-limonene
2. With proper handling techniques and training nPB can be substituted for TCE and d-Limonene extraction solvent
3. Asphalt Extractions with nPB use less solvent per extraction, take less time, have shorter drying times
4. nPB Solvent Recovery Unit Payback time ranges from 3 months to 20 years depending on the quantities of extraction waste generated per year
5. Even with poor solvent handling technique in two of the labs, the exposure levels for the labs were far below EPA recommended value of 25 ppm calculated 8 hour Time Weighed Average (TWA).
6. Using nPB for asphalt extraction and by recycling nPB, the only waste generated would be a non-hazardous still bottom.
7. Using nPB for asphalt extractions would decrease Mn/DOT Laboratories hazardous waste, eliminate time-consuming reporting requirements and reduce annual solvent costs.

RECOMMENDATIONS

It is recommended that all Mn/DOT District Labs change from TCE or d-limonene extraction solvents to nPB and conduct on-site recycling with the purchase of solvent recovery systems. It is also recommended that Mn/DOT Industrial Hygienist give lab personnel training on the proper handling techniques when using nPB.

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2. McDaniel, R.S., Soleymani, H. and Anderson, R. NCHRP Report 9-12. Incorporation of Recycled Asphalt Pavement in the Superpave System.
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4. Stoup Gardner, M. and Nelson, J.W. Use of Normal Propyl Bromide Solvents for Extraction and Recovery of Asphalt Cements. NCAT Report No.2000-06, November 2000.
5. Stelljes, M. Human in Vitro Bioassays of Solvents. Clean Tech, July/August 2001

APPENDIX

Table 2: Solubility of Asphalt in Various Solvents

<u>Solvent ID</u>	<u>% Soluble</u>
A	92.07
B	88.74
C	99.20
D	99.43
E	96.77
F	99.75
G	98.04
Trichloroethylene	99.55
H	96.42
I	96.23
J	92.57
K	95.92
L	77.07

Table 3: Total Solids on Heating

<u>Solvent ID</u>	<u>% Total Solids</u>
A	0.408
B	0.489
C	0.462
D	0.262
E	0.242
F	2.923
G	6.098
Trichloroethylene	0.484
H	6.004
I	5.108
J	0.256
K	5.556

Table 4: Comparative Data on Asphalt Extractions with Solvent D and Solvent C

Solvent D

Flash point – no flash

4 washings after initial Soak
Used 1 Gallon, 2 -22oz bottles

700ml total volume from extraction

Solvent C

Flash point - 150F

7 washings after initial Soak
Used 1 gallon, 2 glass bottles

1200ml total volume from extraction

Table 5: Alternative Extraction Solvent Survey Results

Responses

1

3

5

6

0

3

2

1

6

1

2

4

7

2

0

2

5

2

1. The odor of the new solvent is:

a. better

b. worse

c. doesn't matter

2. The extraction process with the new solvent is:

a. quicker

b. slower

c. same

3. To use the new solvent I would be willing to recycle solvent in the lab.

a. yes

b. no

c. maybe

4. The solvent I like best is:

a. trichloroethylene

b. d- Limonene

C. nPB

5. I feel this new solvent would:

A. save my lab in time /extraction and disposal costs

B. would take more time and effort and is worth the change

c. be unaffected

6. My vote would be to:

a. stay with present solvent

b. make the change immediately

c. make the change next construction season

Table 6: User Extraction Study Results – Limestone Mix

Laboratory Identification	Solvent Used	Extraction Time	Drying Time	Total Time	% AC
Trial Mix Lab	nPB	210	120	330	5.85
	d-Limonene	420	240	660	5.98
	Time Savings	210	120	330	
Metro	nPB	180	120	300	6.05
	d-Limonene	300	150	450	5.80
	Time Savings	120	30	150	
District 1	nPB	75	180	255	5.73
	d-Limonene	1500	180	1680	5.80
	Time Savings	1425	0	1425	
District 2	nPB	145	240	385	6.00
	TCE	130	240	370	6.20
	Time Savings	-15	0	-15	
District 3	nPB	205	105	310	5.90
	TCE	210	120	330	5.90
	Time Savings	5	15	20	
District 4	nPB	150	90	240	5.90
	d-Limonene	270	180	450	6.10
	Time Savings	120	90	210	
District 6	nPB	480	120	600	6.14
	TCE	480	120	600	6.28
	Time Savings	0	0	0	
District 7	nPB	285	90	275	6.08
	d-Limonene	420	210	630	6.06
	Time Savings	135	120	355	
District 8	nPB	25	90	115	6.50
	d-Limonene	65	90	155	6.50
	Time Savings	40	0	40	

Table 7: User Extraction Study Results – 30% RAP Mix

Laboratory Identification	Solvent Used	Extraction Time	Drying Time	Total Time	% AC
Trial Mix Lab	nPB	210	120	330	6.05
	d-Limonene	420	240	660	6.08
	Time Savings	210	120	330	
Metro	nPB	210	150	360	6.07
	d-Limonene	270	120	390	6.12
	Time Savings	60	-30	30	
District 1	nPB	75	180	255	5.83
	d-Limonene	1500	180	1680	5.8
	Time Savings	1425	0	1425	
District 2	nPB	145	240	385	6.4
	TCE	130	240	370	6.6
	Time Savings	-15	0	-15	
District 3	nPB	205	105	310	6.3
	TCE	210	120	330	6.4
	Time Savings	5	15	20	
District 4	nPB	150	90	240	6.3
	d-Limonene	150	180	330	6.6
	Time Savings	0	90	90	
District 6	nPB	480	120	600	5.90
	TCE	480	120	600	6.09
	Time Savings	0	0	0	
District 7	nPB	285	120	305	6.2
	d-Limonene	420	240	660	6.5
	Time Savings	135	120	355	
District 8	nPB	27	90	117	6.3
	d-Limonene	77	105	182	6.5
	Time Savings	50	15	65	

Table 8: Cost Benefit Analysis for On-Site Recycling of nPB

Waste Solvent Generated:	D1	D2	D3	D4	Metro	D6	D7	D8	OMRR
Extractions Drums/Waste/year	90	199	254	35	245	22	77	20	297
=(Extractions* 1.75)/55	2.9	6.3	8.1	1.1	7.8	0.7	2.5	0.6	9.5

Capital Costs:

Recovery Unit Cost: **3422**

Installation Cost: Depends on ventilation set-up in each lab

Annual Savings in Disposal:

Current Costs:	600	1200	1600	66	480	60	120	60	600
Future Costs:	15.62	34.54	44.08	6.07	42.52	3.82	13.36	3.47	51.55
Net Saving for disposal	584.38	1165.46	1555.92	59.93	437.48	56.18	106.64	56.53	548.45

Annual Savings in Raw
Materials

3395.7	7508.27	9583.42	1320.6	9243.9	830.06	2905.21	754.6	11205.81
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Annual Operating Costs:

Operation Labor:	384	384	384	384	384	384	384	384	384
Maintenance Labor:	192	192	192	192	192	192	192	192	192
Power Costs:	67.20	67.20	67.20	67.20	67.20	67.20	67.20	67.20	67.20
Total Operating Costs:	643.20								

Net Annual Savings: **3336.88** **8030.53** **10496.14** **737.28** **9038.13** **243.04** **2368.65** **167.93** **11111.06**

Projected Payback Period:
(years) 1.0 0.4 0.3 4.6 0.4 14.1 1.4 20.4 0.3

Costs do not include Hazardous Waste License & cost associated with waste management at a higher generator size.