

**TOWARDS MORE SUSTAINABLE PRACTICE IN
BITUMINOUS PRODUCTS LABORATORIES**

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Abstract

A challenge facing Southern Africa is the development of an improved environment in laboratories carrying out tests on bituminous products, whereby the use of toxic solvents is eliminated. This entails the replacement of harmful substances currently in general use as solvents in standard test methods as well as cleaning procedures. In pursuance of this goal, the accuracy of adapted test methods should not be compromised and test results should continue to meet international standards. Improved practices should also be associated with improved solvent recycling procedures with zero emission requirements.

This paper details the current practices in South Africa with regard to the types of solvents in use. Current waste management practices are listed.

A brief review of current international developments in solvent replacement/elimination is presented. The review findings and statistics on current South African practices are used as a basis for the formulation of recommendations for achieving enhanced safety of test procedures that are also less harmful to the environment.

The review of current practices is based on a recent investigation carried out for Sabita (South African Bituminous Association) to formulate a guide to practice in bituminous product laboratories, namely Sabita Manual 29.

1 INTRODUCTION

Solvents in a bituminous/asphalt laboratory emanate from the use of reagents for specific test methods and from the need for cleaning agents for glassware and equipment. Samples under test may also contain solvents, e.g. cut-back bitumen contains paraffin.

Specific test methods used in South African bituminous/asphalt laboratories where solvents are used as reagents are listed as follows:

- The determination of the binder content of a hot mix asphalt (HMA) sample
- The recovery of the binder of a HMA sample
- The determination of the solubility of bitumen
- The xylene/heptane spot test
- The Dean and Starke test for the determination of binder content of bitumen emulsions or water content of bituminous mixtures
- The determination of the softening point of bitumen where the softening point is expected to exceed 80°C.

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There are many disadvantages associated with the use of solvents in a bituminous laboratory:

- Health and Safety Hazards – Solvents represent significant health risks as well as explosion/fire risks.
- Environmental Hazards – Solvents generally have a high carbon footprint. In addition, chlorinated solvents have been linked to damage of the ozone layer, and their use has become increasingly restricted under the Montreal Protocol.
- Cost Implications – The costs of solvents have increased significantly in the past number of years, as costs are generally related to the cost of crude oil. Beside factors such as supply and demand, stricter environmental and safety regulations have resulted in more costly manufacturing and disposal/ recycling processes.

2 RECOMMENDED APPROACH TO MANAGING SOLVENT USE

Best practice for any bituminous laboratory would be to eliminate the use of solvents for all test methods, as well as for cleaning purposes. Advances in laboratory equipment over the last decade have made this a viable option for some test methods such as the determination of binder content, as well as for certain cleaning requirements. However, it is not always possible to eliminate the use of solvents. Under such circumstances, the following approach is recommended:

- The substitution of more hazardous solvents with less hazardous solvents – A number of test method developments over the last decade have allowed for the validation of existing test methods by replacing unsafe solvents such as trichloroethylene (TCE) with “safer” solvent combinations such as toluene/ethanol (85/15). However, no solvent is completely safe and there will always be some laboratory operators who would be sensitized to such solvents, whether through a medical or genetic predisposition.
- The substitution of hazardous solvents with one of the new generation “non-toxic”, environmentally friendly solvents, e.g. limonene from oils of orange peels is marketed as a safe and biodegradable solvent.
- Where it is not practical to replace a hazardous solvent, it is important, depending on the toxicity of the solvent, as well as legislative requirements, that the laboratory personnel undertake the required measures to minimize risk exposure.

Risk management for solvent handling would entail:

- A set of updated general laboratory safety rules to minimize the exposure of a laboratory operator to any solvent, as well as minimize the risk of fire and explosion. These general safety rules include details regarding personal protective equipment (PPE), safety training, laboratory signage, maintenance and validation of safety equipment such as fire extinguishers, fume cupboards and extractor fans, etc.
- Management and interpretation of Material and Safety Data Sheets (MSDS).
- Compliance with the legislative requirements of South Africa in terms of the Occupation Health and Safety Act of 1993 (with Amendments), the General Safety Regulations of 1986 as per Government Gazette Notice R1031, as well as the Hazardous Chemical Substances Regulations of 1995 as per Government Gazette Notice R1179.

Overall, when working with solvents in a bituminous / asphalt laboratory, a balance needs to be attained between the safety and environmental requirements, technical accuracy and cost effectiveness.

3 MANAGING SOLVENT USE FOR INDIVIDUAL TEST PROCEDURES

3.1 The Determination of Binder Content and Recovery of Binder From (HMA)

Most solvent used for testing in a bituminous/asphalt laboratory can be attributed to the determination of binder content. The recovery of the binder is an extension of the determination of binder content, but the choice of solvent becomes more limited, because an additional variable, namely the effect of the solvent on the recovered binder properties, comes into consideration.

3.1.1 Historical Developments

Extraction and recovery of bitumen have been practiced in some form or other since the early twentieth century. In 1903 DOW (Abson, 1933) extracted hot mix asphalt with carbon disulphide (CS₂) and recovered the binder using simple distillation. Although many solvents and recovery methods were used and developed after this, none gained widespread acceptance until the use of benzene as solvent along with the development of the Abson method in the 1930's. However, once the toxicity of benzene (carcinogenic) was understood, it was replaced in the 1950's and 1960's by a range of mostly chlorinated solvents still widely used today. These are:

- Trichloroethylene (TCE)
- 1,1,1-trichloroethane (TCA)
- Methylene chloride (or dichloromethane (DCM))
- Chloroform (or trichloromethane (TCM))
- Carbon tetrachloride
- Toluene (methylbenzene)

As mentioned previously, chlorinated solvents have been restricted under the Montreal Protocol and the US Clean Air Act. More recently, they have been found to be carcinogenic.

3.1.2 More Recent Developments

The late 1990's and early 2000's have seen the development of new and safer solvent systems and an improvement in the effectiveness of existing solvent systems. It has also seen the development of the ignition oven for the determination of binder contents of HMA mixes – a revolutionary step towards solvent-free testing.

The ignition oven test (formalized in AASHTO T308) employs the principle of a decrease in mass of HMA, after combustion of the binder, to determine the binder content. The lack of solvents, time savings and SHE considerations make this a most cost effective method. Correction factors are required to for:

- Aggregates mass loss of up to 0.4% (Brown *et al*, 1996) during the ignition process
- Temperature compensation, determined using a blank.
- In some exceptional circumstances it may be necessary to obtain a correction factor for the hot mix asphalt by comparing the ignition result with a specially manufactured standard sample of known binder content.

Proficiency testing conducted in the USA has found that ignition oven testing leads to greater repeatability and accuracy than solvent extraction testing (Brown *et al*, 1996). Coefficients of variation of less than 2% are obtained.

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Although growing acceptance of the ignition oven has resulted in a marked decline in the need for solvent, there are still circumstances where solvent extractions are required:

- In cases where aggregates degrade substantially during ignition oven testing and the aggregate after ignition testing cannot be used for a grading determination.
- In cases where HMA samples are removed from a site some time after construction and no samples are available to determine the appropriate correction factors.
- In circumstances where the binder of the HMA needs to be recovered and characterised.

In the search for safer and more environmentally friendly solvents, a number of new solvents have been developed, or alternatively, such solvents have been introduced into the bitumen/asphalt industry from other sectors in the economy.

Solvent developments include:

- D-limonene - This is a hydrocarbon (cyclic terpene) obtained by distilling the oil from orange peels (a by-product from the orange juice processing industry). It is a natural product used in a variety of applications, including the cosmetics, food and pharmaceutical industry. Limited amounts can even be disposed of via the water drainage system. With a flash point of 50°C, D-limonene has a higher fire/explosion risk than the other new solvents that have entered the market (even a higher risk than diesel with a flash point of 75°C). Although used for binder content determination, it cannot be used for binder recovery.
- Florasolvs Solvent Range - These consist of a range of products ranging from ethoxylated macadamia nut oil to ethoxylated jojoba oil, which not only serve as solvents, but also as non-ionic surfactants (ie soaps / cleaning agents). They have high boiling points / flash points, resulting in lower fire/explosion-risk materials. Although used for binder content determination, it cannot be used for binder recovery.

N-propyl bromide solvents (nPB) - Various studies (Clark, 1998) have shown that nPB is a safe solvent in comparison with chlorinated solvents. nPB has been reported (Collins-Garcia *et al*, 2000) to work well technically when comparing the properties of the recovered binder with those of the original binder. The use of nPB also leads to a significant reduction in extraction and recovery times (30-40% less). These advantages have resulted in a number of states in the USA adopting nPB as their solvent of choice for the determination of binder content as well as binder recovery. However, dissenting voices maintain that the nPB appears to be safe because a lack of research into side-effects have resulted in an illusion of safety. More recently, the Californian Department of Health Services and the Alaskan Department of Labor, amongst others, (www.cypenv.info, www.tekmarltd.com/Company_News.asp, labor.state.ak.us/lss/forms/bromopropane-flyer.pdf, 2011) has raised a health hazard alert regarding the use of nPB in the asphalt industry, whereby nPB is suspected of damaging the reproductive system. Until further clarity is obtained, the authors recommend that nPB's not be used at this stage.

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- A number of patented safety solvents characterized by low toxicity, fire and explosion risk – These include, but are not limited to:
 - Oil-Flo (water-soluble) from TITAN Laboratories
 - Bind-off from Chemical Solutions, Inc.
 - X-it from Pantheon Chemical
- In recent years, ethanol blending with existing solvents has led to an improvement in the overall effectiveness of the blends in extracting binder from HMA, resulting in an overall improvement in the accuracy and repeatability of the properties of the recovered binder. Generally, the addition of 15% ethanol (Cipione *et al*, 1991) has been found to be most effective.

3.1.3 The Technical Performance of Solvent Replacements

In order to replace an existing solvent, it is necessary to evaluate the technical performance of the replacement.

The technical performance of a particular solvent can be defined in terms of:

- Accuracy - How close are the properties of the recovered binder compared with the properties of the in-situ binder prior to recovery or how complete is the binder content determination?
- Repeatability – How do the recovered binder properties or binder content values vary with repeat determinations?

The technical performance of a replacement solvent is often determined by comparing the properties of a binder recovered with that solvent to the properties of the same binder recovered by TCE (considered by some to be an asphalt industry standard in the USA). In South Africa, benzene has been used as the comparative standard.

The technical performance of any particular solvent may vary according to:

- The type and age of the binder
- The type of aggregate
- The property used to characterize the recovered binder
- The extraction and recovery method used. Researchers (Burr *et al*, 1993) have, for example, established that the repeatability for the ASTM D1856 (Absorbance distillation recovery at atmospheric pressure) is significantly superior to ASTM D5404 (Distillation recovery under reduced pressure using the Rotavapor Apparatus).

Poor solvent technical performance (as well as poor coefficients of variation) may be attributed to errors that stem from four main areas:

- The hardening effect of the solvent on the recovered binder -
- Incomplete removal of the solvent from the recovered binder
- Incomplete removal of all the binder from the aggregate
- Incomplete separation of aggregate fines from the binder

Binder hardening is a factor that affects the technical performance of all solvents (Burr *et al*, 1991; Peterson *et al*, 1994). It can be defined as the process whereby a binder dissolved in a solvent exhibits stiffer properties after recovery from that solvent compared to its properties before it was dissolved in that solvent. It can result in an increase of 5 – 300% in the properties depicting the stiffness of a recovered binder,

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depending on the conditions of extraction and recovery, the solvent used, the binder-aggregate system as well as the specific property being measured to depict binder stiffness. For example, properties such as rotational viscosity values and complex shear values determined by a dynamic shear rheometer may show much larger increases than properties such as Ring and Ball softening point or penetration.

Factors that affect the extent of hardening include:

- Solvent type – Researchers (Burr *et al*, 1991; Abson *et al* (1960); Abu-Elgheit *et al*, 1969) have found that the hardening effect for commonly used solvents are:
carbon tetrachloride >> other chlorinated solvents > toluene > benzene
The addition of ethanol to toluene to improve its solvency power results in an increase in the hardening effect of toluene, but not to such an extent that it exceeds the hardening effect of the chlorinated solvents.
- Time – A longer contact time between the binder and the solvent results in a greater hardening effect (Abson, 1933; Burr *et al*, 1991; Noureldin *et al*, 1978).
- Temperature – Although binder hardening occurs at room temperature (Burr *et al*, 1991; Abson *et al*, 1960), a higher temperature during contact between the binder and the solvent results in a greater hardening effect (Burr *et al*, 1991; Cipione *et al*, 1991; Noureldin *et al*, 1978).
- Light – The absence of light during contact between the binder and the solvent reduces the hardening effect (Burr *et al*, 1991; Bussow, 1936). RILEM Technical Committee TC 5611 has recommended that extracted binder solution be kept in the dark until the recovery is completed.
- Oxygen – A lower partial pressure of oxygen during contact between the binder and the solvent reduces the hardening effect (Burr *et al*, 1991).
- Different binder-aggregate types exhibit different extents of binder hardening for the same extraction and recovery conditions (Burr *et al*, 1991).

A number of researchers (Stroup-Gardiner *et al*, 1994; Stroup-Gardiner *et al*, 2000; Cipione *et al*, 1991; Peterson *et al*, 1999) have shown that the binder is never completely removed from the aggregate, regardless of the solvent used for extraction. Incomplete extraction causes the binder content of hot mix asphalt to be underestimated by 0.1 to 1.0 % (Burr *et al*, 1991; Stroup-Gardiner *et al*, 1994; Peterson *et al*, 1999). The binder retained by the aggregate (after extraction) can be highly oxidized (aged). Incorporation of this fraction into the recovered binder can have a significant effect on the recovered binder properties (Cipione *et al*, 1991; Stroup-Gardiner *et al*, 1994).

3.1.4 Case studies

Peterson *et al* (1999) studied the effects of solvent on recovered binder properties using a modified AASHTO T319 / Rotavapor (ASTM D5405) as extraction/recovery process. They could find no differences between the technical performances for TCE, nPB and toluene/ethanol using $G^*/\sin\delta$ as the defining parameter. The intra-laboratory coefficient of variation for $G^*/\sin\delta$ was 26%. Such a relatively high value limited the validity of any conclusions drawn from the study.

Collins-Garcia *et al* (2000 x2) studied the effects of solvent on recovered binder properties using a modified Reflux (ASTM D2172 (Method B)) / Rotavapor (ASTM D5405) as extraction/recovery process. They could find no differences between the technical performances for TCE and nPB using Brookefield viscosity at 60 °C and penetration as the defining parameters.

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Stroup-Gardiner *et al* (2000) studied the effects of solvent on recovered binder properties using the centrifuge method (ASTM D2172 (Method A)) / Rotavapor (ASTM D5405) as extraction/recovery process. They used six combinations of different grades of binders (including a SBS-modified binder) and aggregates. They could find no differences between the technical performances for TCE and four nPB's (Lenium, Leksol, Hypersolve, Ensolv) using $G^*/\text{Sin } \delta$ and BBR Stiffness as the defining parameters. When the Hypersolve solvent was used, the SBS polymer separated from the binder in solution. Intra-laboratory coefficient of variation for $G^*/\text{Sin } \delta$ varied from 25% to 65%, severely compromising the validity of conclusions drawn from the study.

Van Assen (1997) studied the effects of solvent on recovered binder properties using the centrifuge method (ASTM D2172 (Method A)) / Abson distillation recovery (ASTM D1856) Results indicated that for South African binders and aggregates, good technical performance was obtained when using benzene and toluene as solvents. However, results for very aged asphalt tended to display poorer repeatability. Chlorinated solvents displayed poorer technical performance.

3.2 The Determination of the Solubility of Bitumen

The solubility of bitumen is defined by a specific solvent as determined by a particular national/state standard or specification. The solvent can be changed by re-defining the specification and/or test method through an appointed national committee that oversees standards and specifications for the bitumen industry.

Technically, this is easily done and researchers⁸ have successfully replaced various solvents specified in ASTM D2042.

3.3 The Xylene/Heptane Spot Test (AASHTO T102)

No evidence for attempts at solvent replacement has been found in the literature. However, the relatively low toxicity of the solvents (xylene, heptane) and the small volumes of solvent used in this test, make the task of finding solvent replacements a low priority.

3.4 The Determination of the Binder Content of Emulsions (ASTM D244)

No evidence for attempts at solvent replacement has been found in the literature. However, the relative toxicity of the solvents (xylene, toluene) is low compared to chlorinated solvents and the associated risks are small, especially when recommended safety procedures are followed.

3.5 The Determination of the Softening Point of Binder (where the softening point is expected to exceed 80°C - ASTM D36)

No evidence for attempts at solvent replacement has been found in the literature. The relatively toxicity of the solvent (glycerine) is low and the associated risks are small, especially when the recommended safety procedures are followed. Furthermore, this is an international test method, and any changes to the method would necessarily be driven internationally.

4 Cleaning

In many laboratories, the volume of solvents used for cleaning exceeds that used in laboratory testing.

4.1 New Developments

The development of high temperature ovens for cleaning of glassware and equipment has led to a decreased dependency by laboratories on solvents. This has led to a decrease in laboratory operation costs and decreased exposure of laboratory workers to the hazards of solvents. An example of such ovens is the PYRO-CLEAN series of ovens from M&L Testing Equipment (Inc).

4.2 Solvent Cleaning

Typical solvents used for cleaning include:

- Paraffin
- Diesel
- Toluene
- Xylene
- Hexane

These solvents vary in their toxicity and risk of fire/explosion. Their use should be accompanied by the required safety precautions.

Alternative 'safety solvents' as adopted for test methods, have been adopted by many laboratories internationally for cleaning purposes. As previously mentioned these include:

- D-limonene
- Florasolvs Solvents
- N-propyl bromide solvents (nPB's)
- Oil-Flo
- Bind-off
- X-it

5 SOLVENT USE IN SOUTH AFRICA

Current solvent use in South Africa is summarized in Table 1.

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Table 1: Summary of solvent use in South Africa

Laboratory	Determination of Binder Content	Binder Recovery	Method of Binder Recovery	Other Test Methods	Cleaning	Disposal of waste Solvent
CSIR	Toluene	Benzene	Abson Method	As per test method Requirements	Toluene	Initial recycling, Residue disposed via commercial waste disposal company
Matrolab	Toluene	N/a	N/a	As per test method Requirements	Toluene	Initial recycling, Residue disposed via commercial waste disposal company
Roadlab	Toluene	Toluene	Rotary Evaporator	As per test method Requirements	Toluene	Initial recycling, Residue disposed via commercial waste disposal company
Soillab	Toluene	TCE	Rotary Evaporator	As per test method Requirements	Toluene	Initial recycling, Residue disposed via commercial waste disposal company
SRT	Toluene	TCE	Rotary Evaporator	As per test method Requirements	Toluene	Initial recycling.

For binder recovery, all laboratories follow a TMH 1-type extraction process. The waste handling procedures of the commercial waste companies are unknown, but it is highly doubtful that they would be zero emission processes.

The COV for tests carried out by the various laboratories on recovered binder has not been determined.

6 CONCLUSIONS AND RECOMMENDATIONS

To summarise our approach to solvent management:

- Eliminate a solvent if it is practical to do so. The ignition oven should be made mandatory for bitumen / asphalt laboratories.
- Replace dangerous / environmentally unfriendly solvents.
- Safe Handling of a hazardous solvent that is not practical to replace.

In circumstances where the binder must be recovered from the hot mix asphalt or where the ignition oven would not be suitable for binder content determination, the use of solvent will still be required. However, the use of benzene and chlorinated solvents are no longer acceptable and should be phased out in South Africa. This recommendation is justifiable in terms of:

- Health and safety considerations (benzene and the chlorinated solvents are all carcinogens)
- Environmental considerations
- International legislative requirements (Montreal Protocol)

Any number of solvents can be proposed as replacement for those currently used nationally, but such a replacement/s must conform to the SHE / legislative requirements of South Africa as well as the requirements for technical performance as defined in chapter 3.1.3.

Furthermore, for enhanced repeatability, only one specific solvent should be prescribed in South Africa. It is the author's recommendation that toluene/ethanol should be evaluated for South African conditions, based on:

- The acceptable technical performance of this solvent combination as seen in the literature review (Chapter 3.1.4).
- The lower health and safety risks associated with this solvent

In order to assess the technical performance of a replacement solvent, it is recommended that one single recovery/distillation procedure be prescribed for South Africa. Possible choices include the SUPERPAVE-developed method as described in AASHTO T319, or alternatively, ASTM D5404 (rotary evaporator method), but amended to include a 2 flask system for lower binder hardening.

Based on international experience as reported in the literature (Stroup-Gardiner, 2000), any replacement solvent should be evaluated in order to verify its technical performance. This evaluation should include:

- The recovery of a number of binders of known properties from different refineries (prior to mixing), as well as a number of polymer-modified binders of known properties, using the replacement solvent in conjunction with reference solvents such as benzene and TCE. This would establish the technical performance of the proposed solvent in the absence of any aggregate effects. It is recommended that un-aged binder as well as long-term aged binder be used to determine the effects of binder ageing on the recovery process.
- A repeat of the process above, but using relatively older pavement samples encompassing at least six of the more common aggregate types and at least one aggregate predicted to give problems (eg absorptive limestone). In this way aggregate effects can be established. For comparison, pyridine can be used to establish the "true" binder content of the mixes.

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- The identification of those test values obtained from the recovered binders that would best reflect the performance characteristics of the binder prior to recovery (performance characteristics such as stiffness, elasticity, adhesion/cohesion).
- The determination of the effect of background lighting and time constraints at the various stages of extraction / recovery.

It is important that sufficient experimental repetitions be carried out in order to establish the statistical repeatability with confidence. The repeatability would have a direct effect on the quality of the conclusions drawn from the work.

In conclusion, the authors believe that the current status quo in South Africa with regards to the determination of binder content, as well as to the recovery of binders cannot be maintained. We are trailing Europe and the USA in the formulation of a cohesive national binder recovery philosophy.

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KEY WORDS

Binder Recovery, Binder Content, Solvent, Asphalt Laboratory