Solvent Removal from Asphalt

B. L. Burr, R. R. Davison, C. J. Glover, and J. A. Bullin

Asphalt recoveries by the Abson and Roto-vap methods were performed at various temperatures and for several asphalt viscosities. Solvent (trichloroethylene, TCE) concentrations after recovery were measured by gel permeation chromatography. Asphalt viscosities and residual solvent concentrations during solvent removal were determined for tank, oven-aged, and solvent-exposed asphalt to evaluate the effectiveness of the procedures and operating parameters. Small amounts of solvent cause significant decreases in viscosity, and present recovery methods do not remove solvent adequately. In general, recovery rates increase strongly with temperature in both methods. High viscosities and larger asphalt samples hinder solvent removal rates in the Abson method. Asphalt hardens significantly on extended exposure to TCE at both 200°F and 80°F. This hardening also occurs during solvent removal processes, but removal at a reduced temperature through use of a vacuum in the early stages can inhibit it.

To obtain asphalt properties that are representative of the binder in situ properties, procedures must be effective in removing the binders from the aggregate without changing or aging the asphalts. The solvents used for the extraction must be adequately removed from the asphalt binder, so as not to distort the physical properties of the binder that are subsequently measured. Problems have been reported with all aspects of the asphalt extraction and recovery process, including incomplete asphalt extraction, solvent hardening of the extracted material, loss of volatiles during recovery, and incomplete solvent removal. Incomplete solvent removal, which seems a simple problem but which has flawed many studies involving extracted asphalt, is emphasized. Typical residual concentrations of solvent can significantly distort the physical properties of asphalt.

HISTORY

Extraction and recovery of asphalt have been practiced in some form since the turn of the century. In 1903, as discussed by Abson (1), Dow extracted with carbon disulfide (CS_2) and recovered using simple distillation. In 1927, Bateman and Delp (2) centrifuge-extracted with CS_2 and removed the solvent by vacuum distillation. Soxhlet-type reflux extractions using CS_2 were common in this period. Several other methods were developed through 1930, but none gained lasting acceptance (1,3).

Typically today, solvent is removed after extraction by the Abson method introduced in 1933 or by rotary evaporation. These two methods share equal popularity. The literature shows six instances of rotary evaporation and eight of the

Department of Chemical Engineering and Texas Transportation Institute, Texas A&M University, College Station, Tex. 77843-3122.

Abson since the mid-1970s. Before that time, the Abson method dominated.

The Abson method involves recovering benzene-extracted asphalt at 300°F to 325°F with the aid of bubbling carbon dioxide (CO_2). Abson tested seven asphalts ranging in penetration from 175 to 26. After being mixed with benzene, the asphalts were recovered to within 3 percent of original penetrations. The method was designated ASTM D1856 and is still the recommended method (1). Rotary evaporation methods became common in the mid-1970s. ASTM is considering a standard method for recovery using this apparatus.

Through the years, several solvents have been used for extracting asphalt. CS_2 was commonly used initially, but it was phased out because of its high volatility and flammability. Benzene became its primary replacement after Abson's method appeared. In the 1950s and 1960s, chlorinated solvents became popular. The most common were trichloroethylene (TCE), 1,1,1-trichloroethane, and methylene chloride. In 1960, Abson tested several of these and found TCE to be as effective as benzene (4). Adding about 10 percent ethanol or methanol to benzene removed more asphalt from the aggregate (5). This practice has become quite popular among many researchers. Because benzene has been proven carcinogenic, its use has been phased out, and TCE has been the primary replacement.

Although Abson (1) showed that solvent can be completely removed using his method, many researchers have had problems caused by residual solvent that often they do not even realize.

For example, in 1963, Lottman et al. (6) modified the extraction-recovery procedure because he was experiencing excess hardening (probably because of solvent aging). He replaced the CO_2 distribution coil with a smaller one. This change lowered the hardening to 2 percent on test samples. However, when studying hot-mix samples he discovered viscosities lower than the originals, or negative hardening, during the mix process. In the discussion of Lottman's paper, Rostler suggested that negative hardening was probably caused by residual solvent in the sample.

A classic example of how incomplete solvent removal can ruin a massive research project was given by Carey and Paul (7) in a study of factors affecting asphalt in the extraction-recovery process. The project's goals were to study the effect of time in TCE for aged and unaged asphalts, the effect of reduced asphalt concentration during recovery, intra- and interoperator variations, the effect of solvent during primary distillation, and the effect of fines in aggregate mix on recovered asphalt properties. Carey and Paul performed Abson recoveries after 54 similar AC–30 samples and 54 artificially aged samples had spent different lengths of time in TCE. Using ANOVA, a statistical package, they concluded that the time in a solvent before recovery directly affected viscosity. This

result had been shown by other researchers. However, 22 of the 54 AC–30 samples had lower viscosities after solvent aging and recovery. The aged samples (30,000 to 200,000 poise) softened 40 out of 54 times.

Cary and Paul noted this softening but gave no explanation for it (7). Residual solvent may have caused softening and added enough scatter to the data that the variables of interest had negligible effects in comparison. This effect probably masked most of the effects of solvent aging also. Larger asphalt samples had lower viscosities following recovery, indicating incomplete solvent removal.

In 1983, the Pacific Coast Users Group tested the Abson, Roto-vap, and two other recovery methods (8). On four different asphalts having viscosities from 4,000 to 50,000 poise, they found that no method outperformed the others. The Abson method had the lowest reproducibility rating and failed to remove the solvent adequately from two of the four asphalts tested. Consequently, the residues' viscosities were about 75 percent of the original. The other methods caused excessive hardening on the other two asphalts tested. Viscosity increases were as high as 50 percent.

Other authors have noticed or had data that indicated the presence of solvent removal problems. Roberts and Gotolski (9), Sandvig and Kovalt (10), and Gietz and Lamb (11) noted viscosities on recovered road samples that sometimes decreased with time on the road. This effect may actually occur, but decreasing viscosities are probably caused by an extraction-recovery problem. Noureldin and Manke (12) reported work in which recovered asphalt had been hardened by the recovery process. They lowered the Abson time and temperature and almost certainly compensated for solvent hardening by leaving residual solvent in the asphalt.

The problem of incomplete solvent removal is not as simple as it sounds. First, direct verification of solvent removal is rarely performed. Usually, an asphalt of known properties is mixed with solvent, recovered, and tested again. The deviations from original properties indicate the effectiveness of the recovery for that asphalt, which is assumed to be the same for similar asphalts. Petersen et al. (13) mention using infrared to detect solvent in asphalt. In 1936, Bussow (14) used odor and loss of heating as criteria for residual solvent concentration. Secondly, the mechanism of solvent evaporation may not be well understood. In a discussion of a paper by Hagen et al. (15), Petersen states, "When dealing with low concentrations of solvents left in residues, boiling point is no longer a major factor and diffusion and molecular associations between asphalt and solvent become controlling factors for solvent removal."

EXPERIMENTAL PROCEDURES

In order to determine the effectiveness of the existing recovery methods, examine the procedures' responses to changes in process variables, and establish new conditions that would guarantee complete solvent removal, a method of directly analyzing solvent concentrations in asphalts had to be found. TCE was chosen because of its common use. Recoveries were performed using the Abson and Roto-vap methods at various temperatures and asphalt sample sizes, on a wide range of asphalt viscosities. An in situ sampling method was developed so that samples could be taken at several different times during a recovery. To show the effect of solvent, the viscosities of solvent-contaminated asphalts were compared to their original viscosities in terms of a hardening index (ratio of the viscosity of recovered asphalt to that of the original asphalt).

The general procedure was to dissolve the asphaltic material in TCE and recover either by the Abson or Roto-vap method. Sample size, temperature, and asphalt viscosity were varied. During the recoveries, small samples of asphalt were withdrawn and analyzed for solvent content, so that the solvent concentration versus time for each set of conditions could be determined.

The tendency of solvent to harden asphalt was also studied. The asphalt-solvent solution was allowed to stand for different lengths of time before recovery at room temperature and at 200°F. A few Roto-vap recoveries were made under vacuum to achieve a low recovery temperature and less hardening.

Three asphalt viscosities were used. A 2,000-poise tank asphalt was used and was hardened to 20,000 and 200,000 poise. Recovery conditions were 325°F, 340°F, and 380°F with the Abson procedure; 280°F, 280°F at reduced pressure; and 325°F, 350°F with the Roto-vap method. Sample sizes were 50, 75, and 100 g for the Abson method and 50 g for the Roto-vap.

Most data were for 50-g samples. Although contrary to the Abson specification of 70 to 100 g, an inconsistency exists in the Abson specification. The equipment for extraction by the specified Method A of ASTM D2172 will generally allow a sample size of about 1 kg. With most mixes, this sample size yields only 50 to 60 g of asphalt. In general, as shown later, larger sample sizes only aggravate the deficiencies of the standard Abson procedure.

TCE Analysis by Gel Permeation Chromatography

The residual concentration of TCE during solvent removal can be measured easily by gel permeation chromatography (GPC). An earlier study of extracted pavements indicated that TCE elutes as a single peak with no interference from other materials of small molecular size in the sample. A distinct peak of small molecular size appeared on the chromatograms. No such peak had been seen on tank or oven-aged samples.

The GPC analyses were performed on an IBM Model LC-9533 high-pressure liquid chromatograph. A 100-μL sample [7 weight-percent asphalt in tetrahydrofuran (THF)] was injected into the carrier solvent flowing at 1 mL/min through two Polymer Laboratories (PL) columns in series containing PL gel material of 500-Å (Column 1) and 50-Å (Column 2) pore sizes. The details are described by Donaldson et al. (16).

Standard blends of TCE in THF were made for calibration of the solvent analysis. Dilutions in THF equivalent to those that occur in GPC analysis of 5, 2, 1, 0.5, 0.1, and 0.05 weight-percent TCE in asphalt were analyzed by GPC; the relative peak areas related linearly to the TCE concentrations with an r^2 value of 0.997. Analyses of TCE in asphalt samples were performed similarly. Solvent concentrations were determined using the calibration. Residual solvent concentrations in recovered asphalt ranged from 0 to 2.5 percent.

Asphalt Viscosity

Asphalt viscosities, measured according to ASTM D2171, were obtained at 140°F in Cannon-Manning viscometers. All samples were melted and mixed well before viscosity measurement, because asphalt tends to stratify during cooling. Samples taken near the top while cool and hard tended to have much higher TCE concentrations because of this stratification.

Fourier Transform Infrared Analysis

A Nicolet Fourier transform infrared (FT-IR) spectrometer was used to analyze the functional chemistry of samples. Some of the hot mixes left a mysterious oily film on the flask sides after recovery. This film was run on the FT-IR because of the small sample size required. An attenuated total reflectance method worked well (*17*). The samples were applied as thin films onto a special zinc selenide prism.

Sampling

An in situ sampling technique was devised. It was first thought that asphalt could be drawn into a 1-mL disposable pipette during the recovery. The asphalt would then solidify and could be stored until time for GPC sample preparation. However, the asphalt had to be melted and drained into a scintillation vial. During this process, solvent was apparently lost. Consequently, this method yielded solvent concentrations that were low and imprecise. To remedy this, 8-in. pieces of ⅛-in.-ID Nalgene autoclavable tubing were substituted for the glass pipettes. After sampling, these tubes were cut lengthwise at room temperature to allow easy removal of the asphalt for GPC sample preparation. This method seemed to be more precise than any alternative.

Materials

The asphalts were derived mainly from locally sampled Exxon AC-20. Hardened samples were produced by bubbling oxygen into 450°F Exxon AC-20 for from 3 to 8 hr. The two 300-g samples of hardened asphalt had viscosities of 20,000 and 200,000 poise. After each recovery, the asphalt was remixed with solvent for use in another recovery experiment. The small changes in properties caused by reusing material were insignificant in their effect. Recovery data on several extracted hot mixes were also taken. The recovery solvent, TCE, was recycled after each run. No serious changes in the asphalt's or solvent's properties were detected.

Abson Method

The Abson method (ASTM D-1856-79) removes solvent by distillation with the aid of a CO_2 purge. About 150 mL of a concentrated solution of asphalt and solvent, containing 50 to 100 g of asphalt, are charged to a 250-mL widemouth, round-bottom flask. The flask is electrically heated. The liquid boils and is condensed and collected in a receiving flask. The CO_2 flow is set at 100 mL/min once the temperature reaches 275°F. When the temperature is 315°F, the flow is increased to 900 mL/min. The temperature is then maintained between 320°F and 330°F for 15 min, or 5 min after the last drop has fallen, whichever is longer. In this study, the last drop had fallen within 10 min in every case but one, and that was only a few seconds over. In the case of material having a penetration less than 30, the method specifies 20 to 22 min. Therefore, the Abson time would be 15 min for all but the 200,000-poise material, and 20 to 22 min for these runs.

Instructions set forth in ASTM D1856 were adhered to strictly except for varying sample size, time, and temperature to study these parameters. The CO_2 flow was accurately metered using a Gilmont R–012 flow meter. A variac and heating mantle supplied heat to the flask of asphalt and solvent. Temperature control was found to be difficult from the beginning. Because of a lag in the response of the asphalt temperatures to heater action, significant excursions outside the specified limits were difficult to avoid. Consequently, an analysis of the heater system with respect to temperature control was performed to obtain the sequence of heater settings that would give good control at 320°F, 340°F, or 380°F, as required. Manual control of the variac using these setting profiles made temperature control easier and more accurate.

Roto-vap Method

The Roto-vap method, which uses a rotary evaporator to mix the solution and a vacuum and vent gas to vaporize and sweep away solvent, has a distinct advantage over the Abson method in that there is no need for a primary distillation of the solution down to 150 mL before proceeding with the recovery. Large volumes of solution can be handled in this apparatus. Recoveries were run on mixtures of 100 mL of TCE and 50 g of asphalt to save time and solvent.

No formally approved Roto-vap recovery procedure is available, but a tentative ASTM method and another used by the Pacific Coast User's Group (*9*) appeared identical and well studied. These methods formed the basis for the procedure.

Initially, the oil bath is heated to 280°F. A 1,000-mL round-bottom flask containing 150 mL of concentrated solution is attached to the Roto-vap. CO_2 is metered at 500 mL/min through a tube that touches the solution's surface. The flask rotates at 45 rpm in the oil bath. When the bulk of the solvent has been distilled, a 600-mm (Hg) vacuum is applied and the CO_2 flow is increased to 600 mL/min. These conditions are held for 15 min. In an alternate method studied, no vacuum is applied, but the CO_2 flow is increased to 900 mL/min.

Asphalt sampling was similar to that for the Abson method. The 1,000-mL flask was modified with the addition of a 24/40 ground-glass fitting approximately 45 degrees from the flask's neck. The connection was closed with a ground-glass cap. During the recovery, the flask rotation was stopped, the cap was removed, the Nalgene sample tube was inserted into the hole, and the sample was pulled using a pipetter. Immediately afterward, the cap was replaced and rotation resumed. This process took approximately 20 sec to perform when there was no vacuum and about 35 sec if a vacuum was used.

RESULTS AND DISCUSSION

Extraction and Solvent Removal Effects on Viscosity

Figure 1 shows the viscosity relative to initial viscosity (the hardening index) of asphalt during solvent removal processes for three situations. The tank (AC–20) and oven-aged (rolling thin-film oven test, RTFOT) asphalts were well dissolved in TCE at room temperature, and solvent removal was initiated within 1 hr. The refluxed sample was a mixture of approximately 25 weight-percent tank asphalt (AC–20) in TCE that was refluxed for 4 hr at 200°F before initiating solvent removal. This reflux procedure simulated the conditions of hot extraction methods such as ASTM D2172, Method B.

These data indicated several effects of the extraction and recovery procedures on asphalt viscosity. The first factor was the considerable softening of asphalt by even small amounts of residual solvent. Even 0.5 percent of residual solvent can produce viscosities that are 50 percent low. Second, there are different degrees of residual hardening on reaching zero-solvent concentration for different asphalts and situations. The tank asphalts typically harden 5 to 10 percent above their original values. This effect probably results from a loss of volatile components during recovery. The RTFOT samples return to their original viscosities, because their volatiles were removed in the oven-aging test. Asphalts exposed to hot reflux (simulating a reflux extraction procedure) may exhibit a 20 to 40 percent increase of viscosity, indicating significant solvent hardening beyond that attributable to volatiles lost.

Figure 2 shows that the solvent-hardening phenomenon also occurs at room temperature after extended exposure times. A series of Roto-vap solvent removals (asphalt recoveries) were conducted for mixtures of 7 weight-percent asphalt in TCE. Before recovery, however, the mixtures were allowed to incubate at room temperature for the times shown in Figure 2. For short room-temperature incubation times, hardening caused by volatiles loss and solvent aging in the hot-recovery Roto-vap method was from 14 to 18 percent. For extended times, hardening was in excess of 40 percent. For comparison, a sample was recovered immediately after dissolution using

FIGURE 2   Hardening of asphalt in TCE at room temperature for extended periods of incubation time prior to hot recovery. Also shown is the aging during a cool recovery process with short incubation time.
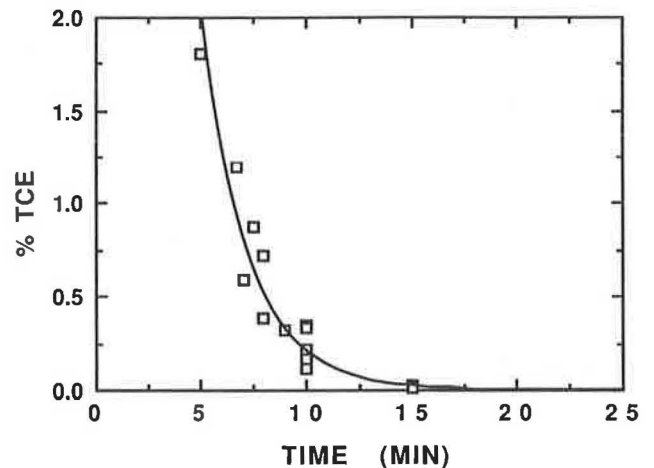
FIGURE 3   Residual solvent concentration versus Abson recovery time for a tank (AC–20) asphalt.

a vacuum to obtain a reduced solution temperature of approximately 100°F. This short, room-temperature exposure produced about 10 percent hardening, close to that caused by volatiles loss only.

Abson Solvent Removal Method

Several recoveries at strict Abson conditions consistently resulted in significant TCE presence after the time required by the method. The experiments also showed the reproducibility of the Abson recovery and sampling methods used (Figure 3). Approximately 0.19 percent of the TCE remained in the asphalt after the Abson time on recoveries of 2,000-poise asphalt. From Figure 1, this value implies about a 10 percent decrease in viscosity. If the recovery procedure is calibrated with no TCE analysis technique and using tank asphalts, inadequate operating parameters are established
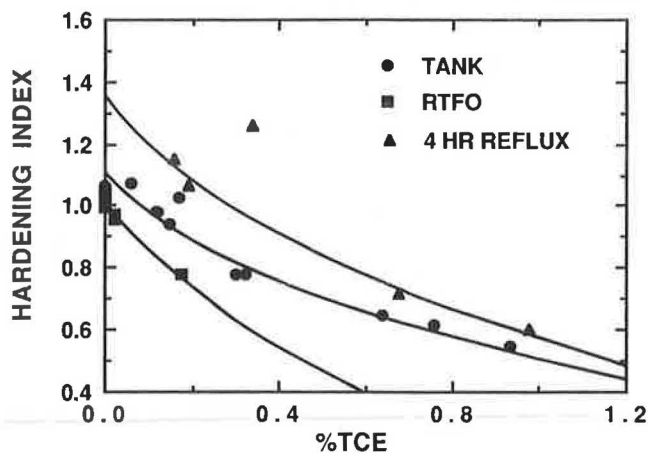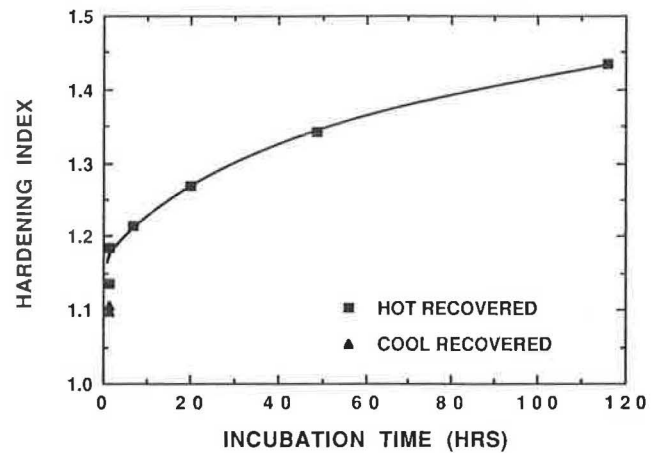

FIGURE 1   Removal of solvent and resulting changes in asphalt viscosity. The tank and oven-aged asphalts were dissolved in solvent and immediately recovered with no incubation time in the solvent.

because tank asphalts show no viscosity decrease even though their residual TCE levels are about 0.1 to 0.2 percent, because the presence of solvent is offset by a loss of volatiles.

Recovery temperature and asphalt viscosity noticeably affect the Abson method's ability to remove TCE. Figures 4–6 show the TCE concentration profiles for recoveries of 2,000-, 20,000-, and 200,000-poise asphalts each at 325°F, 340°F, and 380°F. Figures 7–9 show the same data for each asphalt grade. These data show, without fail, that removal is achieved sooner for lower-viscosity and higher-temperature material. The poor removal at high viscosities at 325°F illustrates the need to modify the existing conditions. The viscosity of the 20,000-poise material was lowered nearly 30 percent at completion of the prescribed method.

Changes in recovery temperature and asphalt viscosity should affect the method's performance. As the solvent is removed, the solution behaves like a pure molten asphalt. The high viscosities of these materials lower the mobility of the solvent, because of increased diffusion resistance. This resistance slows the solvent in reaching the liquid-vapor interface where evaporation occurs. So, although equilibrium may be maintained
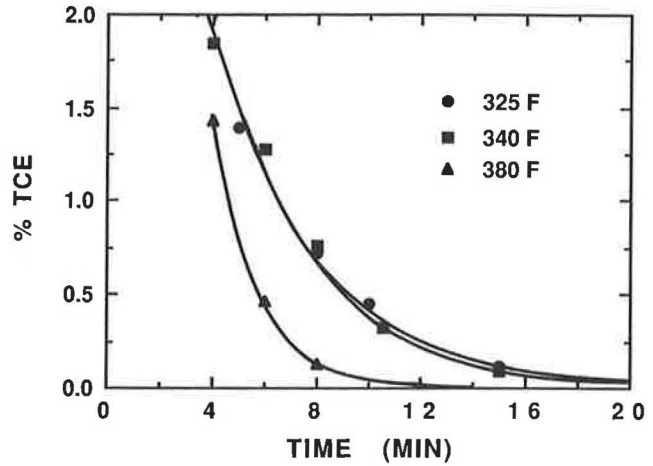
FIGURE 6   Residual solvent concentrations versus Abson recovery time at three temperatures for a 200,000-poise asphalt.
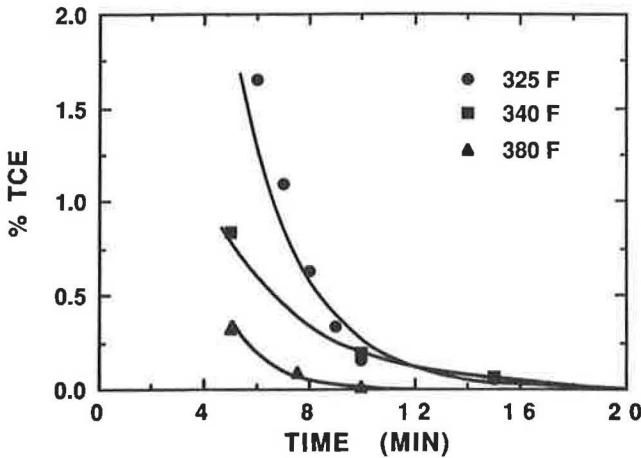
FIGURE 4   Residual solvent concentrations versus Abson recovery time at three temperatures for a tank (AC–20) asphalt.
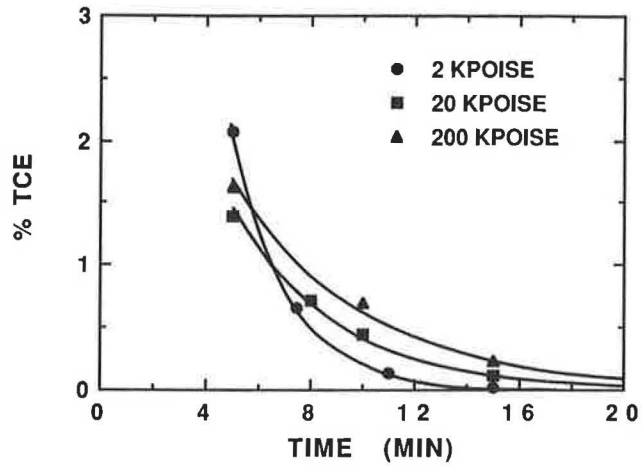
FIGURE 7   Residual solvent concentrations versus Abson recovery time for three asphalt viscosities at 325°F.

FIGURE 5   Residual solvent concentrations versus Abson recovery time at three temperatures for a 20,000-poise asphalt.

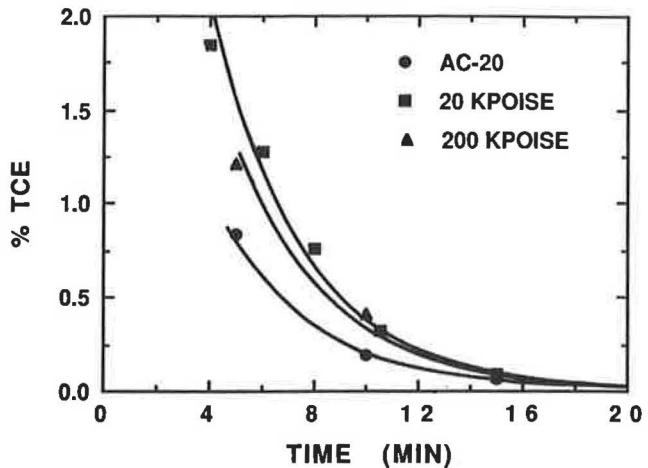FIGURE 8   Residual solvent concentrations versus Abson recovery time for three asphalt viscosities at 340°F.
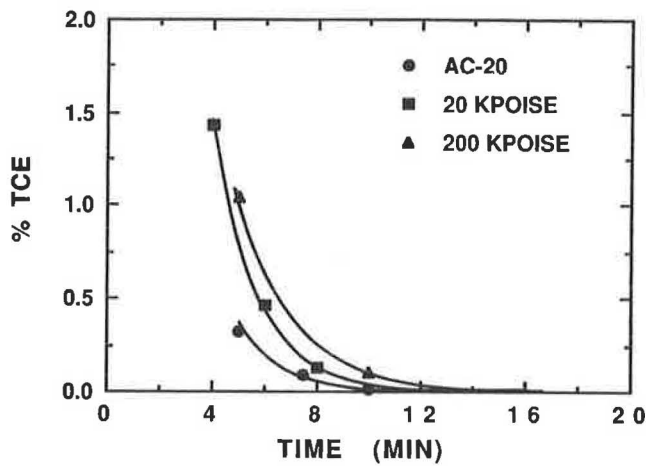
FIGURE 9   Residual solvent concentrations versus Abson recovery time for three asphalt viscosities at 380°F.
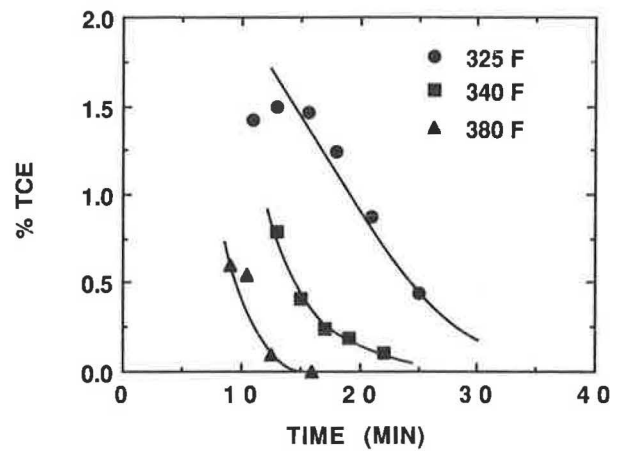

FIGURE 11   Residual solvent concentrations versus Abson recovery time at three temperatures for a 100-g sample of 200,000-poise asphalt.
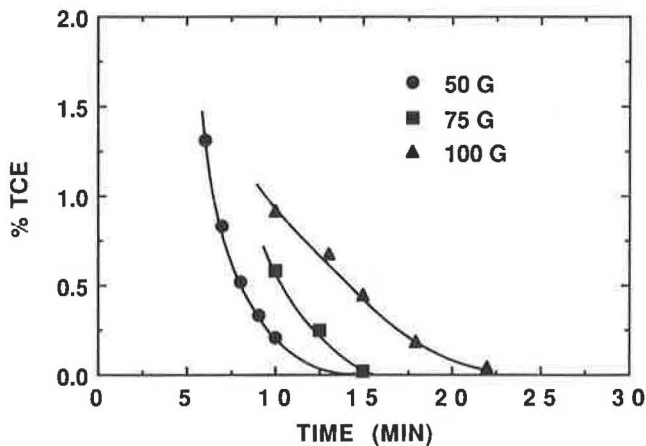

FIGURE 10   Residual solvent concentrations versus Abson recovery time for three sample sizes for 2,000-poise asphalt at 325°F.
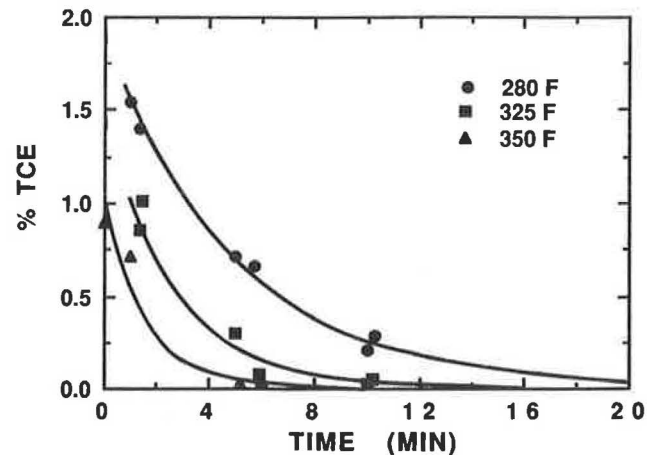

FIGURE 12   Residual solvent concentrations versus Roto-vap recovery time at three temperatures for an AC–20 asphalt.

at the asphalt surface, significant quantities of solvent remain in the bulk asphalt, its removal rate being limited by diffusion. Furthermore, higher-viscosity asphalts have lower diffusion rates, resulting in increased difficulty in removing solvent. Also, the higher asphalt viscosities reduce the mixing effectiveness of the CO_2 purge.

Figure 10 shows that increasing sample size detrimentally affects solvent removal. The Abson procedure (ASTM D1856-79), while allowing recovered asphalt quantities greater or less than the specified 75 to 100 g, states that this may affect the properties of the recovered material, in which case, 75 to 100 g should be recovered. This specification would appear to be incorrect. Figure 11 shows how poorly the Abson procedure removes solvent at higher sample sizes (except at 380°F).

Roto-vap Solvent Removal Method

Figures 12–14 show Roto-vap method residual solvent profiles for the AC–20, 20,000-poise, and 200,000-poise materials, respectively, at three removal temperatures. Zero time

is that of the last-observed drop from the solvent condenser. Unless otherwise indicated, the Roto-vap method used was that without vacuum (i.e., with a CO_2 purge only). The profiles show pronounced temperature dependence, but, surprisingly, no discernible viscosity dependence. Also, the use of a gas purge alone seems to be as effective as that with a vacuum at 280°F (Figures 11 and 12). In most cases, the 280°F recoveries, vacuum or gas purged, left significant solvent residues after the method's suggested time of 15 min past the last sign of condensation. However, the 325°F and 350°F runs showed complete removal after 15 min.

The Roto-vap method appears to be less reproducible than the Abson method. Two separate recoveries of 20-kpoise asphalt at 325°F gave drastically different solvent removal profiles, one having complete removal in less than 2 min. This difference may have been caused by the difficulty in determining when the last sign of condensate disappears, though other factors are likely to contribute.

The use of a vacuum during the initial recovery seems to reduce solvent hardening. Two AC–20 recoveries were performed using a high vacuum, so that the boiling temperature
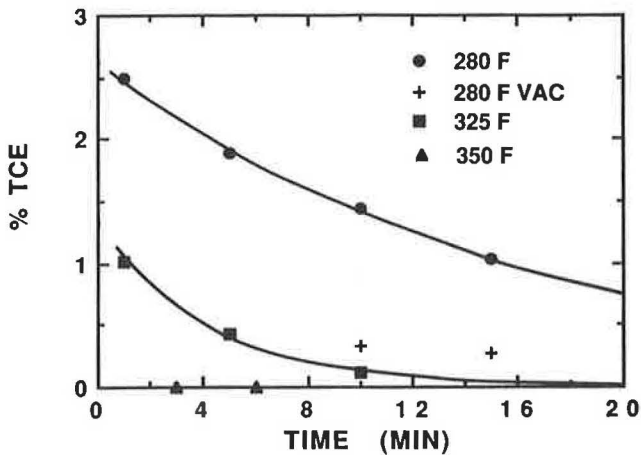
FIGURE 13   Residual solvent concentrations versus Roto-vap recovery time at three temperatures for a 20,000-poise (aged) asphalt.
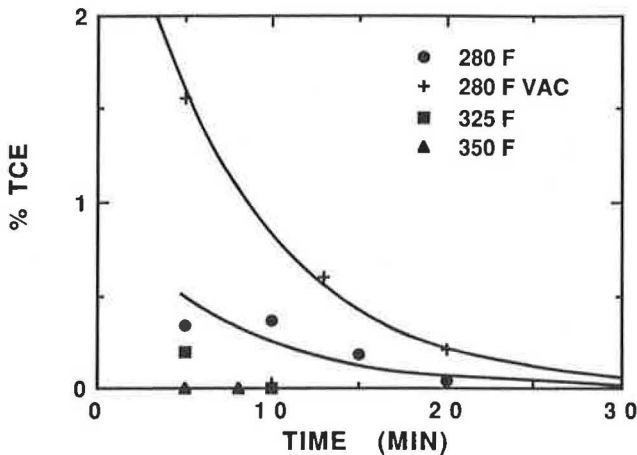

FIGURE 14   Residual solvent concentration versus Roto-vap recovery time at three temperatures for a 200,000-poise (aged) asphalt.
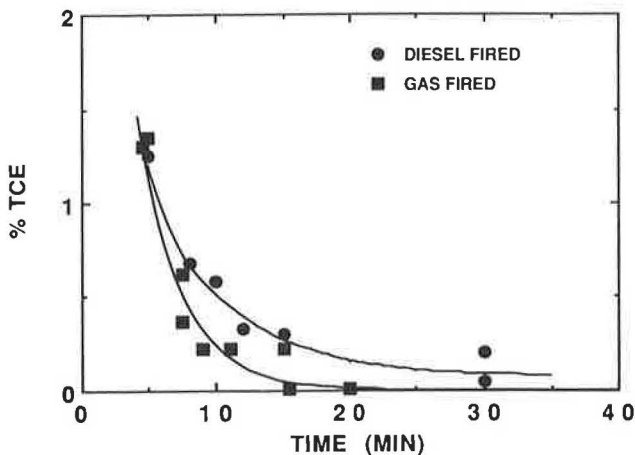

FIGURE 15   Residual solvent concentration versus Abson recovery time for two hot-mix extractions.

was less than 100°F. Two others were run using no vacuum, and boiling points were about 200°F. Figure 2 shows that the vacuum-recovered asphalts hardened about 10 percent, which is barely above the hardening typically caused by volatiles loss. The hot-recovered samples hardened about 16 percent.

Hot-Mix Recoveries and Volatiles Loss

Several extracted hot mixes were recovered (Abson method) and found to behave somewhat differently than tank or oven-aged material. Figure 15 shows a solvent removal comparison between diesel- and gas-fired plants. Hot mixes from batch and natural gas-fired plants showed normal solvent removal behavior when conducted at 320°F. However, diesel-fired drum plant mixes, even though recovered at 340°F for 30 min, contained significant levels of material eluting where TCE does on the GPC.

The diesel-fueled mixes were also different, in that they evolved a long-lasting fog and deposited an oily residue on the recovery flask's surfaces. These asphalts were all mixed at drum plants fired by diesel or fuel oil. Infrared analyses of the oily film showed it to be composed of highly oxidized hydrocarbons of small molecular weight (Figures 16a and 16b), possibly from partially oxidized fuel oil. Other asphalts mixed at batch or natural gas-fired plants did not exhibit this phenomenon.

The presence of this unusual and slightly volatile material may have given a false indication of residual solvent concentration and may account for the excessive solvent removal times of these hot mixes. A hot mix containing this material was extracted with THF in a sequence of cold, batch extractions using an amount of solvent that gave an overall concentration suitable for direct GPC analysis. Because no solvent removal was performed, all the volatile impurities remained in solution and were detected on the GPC. Several new peaks and valleys appeared near the TCE elution time, leaving open the possibility that the apparent TCE peak in the hot-mix recoveries was caused by residual oxidized fuel oil. Although the relative amount of this material in the asphalt was not determined, its significance to highway performance and to relating measured chemical properties to performance is potentially great and bears further study.

SUMMARY

The volatiles loss from virgin or unaged tank asphalts during solvent removal can produce 7 to 10 percent hardening of the original asphalt viscosity. RTFOT asphalts do not exhibit this hardening, apparently because of the loss of volatiles during the aging.

The same asphalts show hardening of from 10 to 40 percent (including that caused by volatiles loss) on contact with TCE and subsequent solvent removal. Short times and moderate temperatures for incubation of the asphalt with solvent produce little hardening; extended times at elevated temperatures (such as during reflux solvent extraction) can produce significant hardening.

Experiments were also conducted on the Abson and Roto-vap solvent removal methods for the purpose of evaluating their effectiveness in removing solvents. The Abson method, taken to its standard recovery time, can leave enough solvent
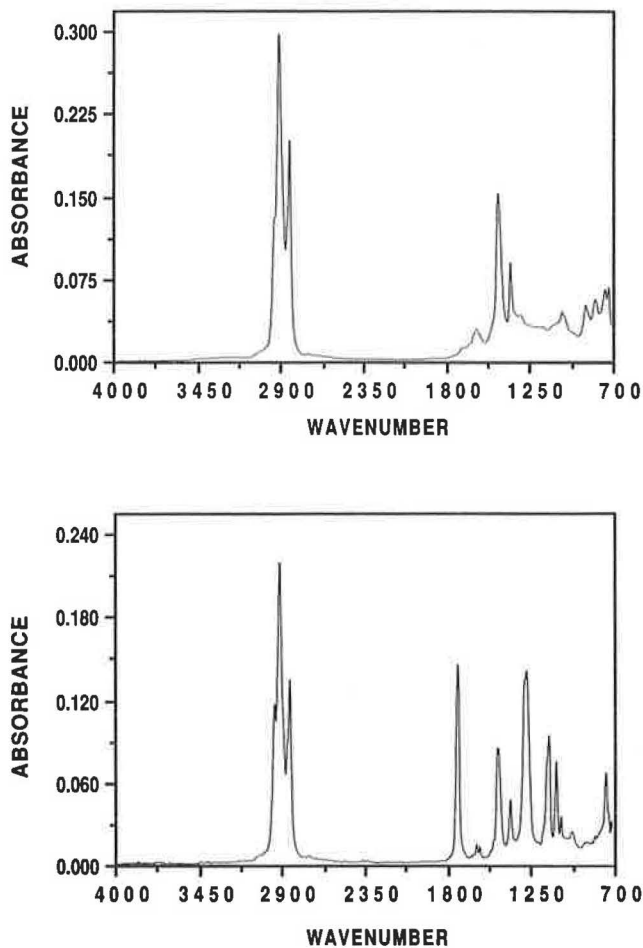
FIGURE 16 FT-IR spectra for the recovered hot-mix asphalt (top); for the volatile material recovered from the diesel-fired hot mix (bottom).

to produce significant softening, especially for larger quantities of recovered material and for hardened asphalts such as those obtained from aged pavement cores. Increasing the temperature of the solvent removal and the recovery time can reduce this residual solvent concentration, although the previously mentioned solvent-hardening effects must be considered. The Roto-vap method appears to be less consistent and less reproducible than the Abson method, but it may have some advantages for solvent removal.

For the Abson procedure at 325°F, a minimum recovery time (after the last drop) for 50-g samples is about 25 min. For 100-g samples, 380°F will remove the solvent within 20 min. For the Roto-vap procedure, 15 min past the last drop was adequate at 325°F.

A complicating factor in hot-mix and pavement core recoveries appears to be the possibility of fuel contamination from the hot-mix plant. Evidence of such contamination was found in some drum hot mixes, but the extent and significance of this possible contamination has yet to be determined.

ACKNOWLEDGMENTS

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