Applicability of the Plate Stripping Test to Asphalt

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1 Introduction

1.1 Overview

The T230 plate stripping test may be performed at different embedment temperatures and different embedment times. For example 45 minutes at 140º is more simulative of asphalt production & laying than the standard conditions; thus the resistance to water induced stripping of an aggregate coated with binder in conditions more nearly simulating asphalt production will give better assurance of field performance.

It is found that when results for a particular system at the same embedment time are plotted against temperature, the results fall on an S shaped curve. The shape of the curve is approximately the same for all materials. Systems of binder/aggregate with other variations such as additives and aggregate surface condition (including precoat) may be characterized with a single number with the units of temperature. The number may be split by simple arithmetic to report a characteristic number for each material or surface condition.

The spread of all 318 results around the common S shaped curve (master curve) allows the precision of the test to be estimated. The precision of the T230 test (AS1141.50) is reported for the first time. Greater precision and confidence in the results for asphalt may be achieved by performing the test at different temperatures and embedment times. The system proposed does not entail more work because once each material is characterized; the number may be transported with it to other systems, thus enabling them to be predicted without further laboratory work.

The adhesion of multigrade was investigated with reference to its oil exudation. Multigrade bitumen was found to have better adhesion than AS2008 bitumen types. Routine use of an exudation test is not proposed.

1.2 Background

The RTA developed T230 “Resistance to Stripping of Aggregates and Binders” (AS1141.50) over fifty years ago. The test was developed by the then DMR laboratory from a remark in an account of the similar British Immersion Tray Test (implemented by the RTA as T238) which indicated that a test like T230 could be done without actually specifying it in detail.
The test measures the adhesion at particular conditions of temperature and water soak between a particular system of binder, which may be treated with an adhesion agent and an aggregate which may be tested clean, dusty, precoated or in a damp & precoated surface condition. In the test, 30 - 35g of binder is placed in a metal tray 150mm square. Fifty aggregate particles are pressed into the binder and they are embedded for 24hr at 60ºC. The tray is then immersed in water for four days at 50ºC. At the end of which time the aggregate particles are pulled out of the binder, rated for adhesion individually on a 3 point scale (fully stripped/partly stripped/ unstripped) the fifty individual results are then combined to an overall percentage stripped.

Prior to the introduction of QA fifteen years ago, the RTA routinely performed the tests out of its own resources in its own laboratories and used the results to prequalify materials. Since that time RTA personnel have become less connected with the T230 test.

Over the last twenty or so years, the primary asphalt binder first changed from C170 to C320, QA was introduced, & now there are AR450, PMBs & multigrade which were not envisaged at the time the test was introduced. Laboratories must now be up-front about measurement uncertainty and precision; in the past it was enough for a population of experts to have a feel for such matters. The number of such experts has decreased. The test in its present form is not friendly to ranking materials and interpreting the significance of, for example, narrowly failing systems.

In response to many instances of stripped asphalt, the RTA has routinely applied the test to asphalt components, with the result that in many cases it seemed to support the greater use of adhesion agents with asphalt. The T230 test was criticised because it was perceived as being developed originally for sealing applications, specifically that the embedment time and temperature 24 hr at 60º seemed to be tuned to sealing & appeared not relevant to asphalt applications, and because of the lack of precision statistics. In recent years, perhaps with many years of drought, and perhaps with a tightening of specification R116, the RTA has relied less on T230. But the RTA needs to have available some test or process to manage stripping should it ever become a problem again. Stripping of bituminous asphalt and seals may involve mechanisms which are not addressed by the T230 methodology, for example mechanically driven ones involving pumping and pore pressures.

2 Aims of the Experimental Program

There have been deficiencies on our knowledge regarding the T230 test and its application to preventing stripping on RTA works. The following points are items which would advance the science of adhesion generally.

- A single numerical measure of adhesion. The present pass/fail at a criterion of 10% maximum stripping on T230 which may be with/without precoating or adhesion agent is not readily transportable. We need a single adhesion number to tell us how far or close we are to satisfactory adhesion and if unsatisfactory, how far to go.

- What is the expectation for a wide range of binders? We compare any new binder or aggregate or a new sample from an established source with the database, if it is significantly different, we can purposefully investigate. Time and resources are often wasted testing and retesting systems which are doomed to fail often by the specification of inappropriate conditions. Sometimes a satisfactory material could be rejected following testing under inappropriate conditions or with inappropriate criteria.

- Is there any adhesion difference between different sources of bitumen of the same AS2008 grade?
• Is there any truth in the assertion that multigrade does not need adhesion agent?

• By contrast there is a perception in some quarters that “multigrade does not pass T230” in this case it is not so much an improved T230 test that is required, rather increasing the number of practitioners with an understanding of the conditions under which C170 and C320 will pass.

• How does adhesion change with temperature? What implications does this have on sprayed sealing if the temperature is below some specified temperature and falling? If peoples’ eyes were not opened by the non-contact infra-red thermometers, then recent observations with thermal imaging are clear in demonstrating that it is almost impossible for sealing aggregate to contact binder at a temperature greater than 60°, on the other hand, sealed pavements usually reach a temperature of about 60° for an hour or so every day in Summer.

• Can T230 (done at 60° for twenty four hours) be relevant for asphalt made at, say 140° and mixed for a matter of seconds to a couple of minutes and paved perhaps within one hour?

• If an improved T230 can deliver a single numerical measure of adhesion in a binder, then it may be possible to correlate it with some other candidate measures of adhesion (e.g. syneresis, chemical functionality, asphaltene content, acid value & penetration index)

• What are the 95% confidence limits of repeatability of T230 either expressed as % stripped or as the universal, transportable measure?

• The relationship to the RTA cutback chart and integration with the RTA sprayed sealing guide is desired.

• The single numerical measure of adhesion should also be able to give a figure relevant for aggregates separately, binders separately and precoats separately. If this was indeed possible, then one could dispense with the “standard aggregates” and use the actual aggregates of a particular project.

• For certain tests a standard aggregate will still be required. The ability to define the adhesion of an aggregate with a single number would allow an aggregate no-longer available (e.g. Prospect dolerite) to be replaced with another with minimal fuss.

3 Experimental Procedure

A range of binders and aggregates was selected to be representative of the great many materials used for road construction. Of the binders, twenty six were selected, some tested under many conditions others tested less intensively. The majority of them are AS2008 binders and multigrades, some have been tested after RTFO and/or with adhesion agent added. A few special binders such as one tar, one PMB, one colourable binder and one blown bitumen are included. The database has recently been increased to include C240, AR450 and C170 bitumen currently in use in NSW and has been expanded modestly in the area of PMB especially A15E, however for reasons of space and analysis time, some of these data are not included in the present report (some new raw data of PMBs appear in the accompanying Excel).

Only two aggregates have been used so far in this program. They are well known and well contrasted; Prospect dolerite and Mugga microgranite-porphyry. Both aggregates were washed clean of dust and oven dried.
In order to study the effects of temperature on adhesion as estimated by % stripped on the test T230, only one feature of T230 was changed. At clause 5d of the method, the stones pushed into the trays of binder are conditioned for twenty four hours at 60° [or softening point +20°]. In the work reported here, a range of temperatures (for want of better guidance, following SHRP practice) separated by 6° C steps were chosen. The SHRP rule of thumb being that changes of 6° should have an effect equivalent to doubling or halving road-bitumen processes such as rutting (see section 5.4). In order to check this hypothesis, times of 1*, 2*, 4 and 8* days as well as ½, ¼, ⅛*, 1/16, 1/32* day could be chosen. In fact only the asterisked ones have actually been used so far.

The program of work was progressed through the laboratory in five parcels; this allowed the experimental design to be modified as the experiment progressed. Initially it was thought that almost all of the practical variation would be observed within 12 or 18° of 60°. By the second parcel it was clear that the breadth of the results was much greater and also that the C170 was more than 50% stripped at 60° even with Prospect dolerite. Also to test the hypothesis that the T230 test was relevant or translatable to asphalt where coating temperatures are in the order of 140° and mixing times are measured in seconds with delivery and laying perhaps within the hour, experimental conditions are needed to extend into those regions so that conclusions need not be extrapolated.

Traditionally the 60° temperature at which T230 is embedded has been achieved by a gravity convection oven (without a fan). In such an oven set to 110° a T230 plate takes 22 minutes to achieve 6° below the set point, i.e. 104°. The author had been concerned about the precision likely to be achieved with this method. The first parcel of work done by the laboratory (no embedment time less than one day) was done in two ovens, one of the old type and a modern fan-forced one. In both cases a metal plate to which a thermocouple was welded was placed under one of the plates as an additional check on temperature. It was found that especially in the natural convection oven, plenty of gaps needed to be left between and around the plates. For heating times of 3 hours and less, the natural convection oven is inadequate unless additional heat-up time is allowed (still unsatisfactory). From the third parcel of experiments only forced convection ovens were used, and by the fifth one, a purpose built oven has been used.

Although 60° is an acceptable representative maximum pavement temperature, it is well known that sealing works done in fair weather in the early afternoon may well have pavement temperatures below 40°. The effect of adhesion agent on C170 bitumen is huge, something of an order equivalent to a temperature increase of 70°C. Therefore, in order best to demonstrate this with temperatures close to those used in practice, it would be desirable to measure the adhesion agents at temperatures below 60°. The temperature 52° is quite straightforward as this is still above the water bath soak temperature of T230 (50°). 46° starts to become problematic because it is possible that the 50° soak especially as it is conducted for four days could improve adhesion. Therefore, in this case, before the 50° water soak, the trays were first soaked at room temperature for 1-3 days. The 34° experiments were conducted in the same manner but the results seemed aberrant and were not used in the data analysis. The 34° results seemed to be less stripped than the following “master curve” analysis would predict. Rather than distorting the conclusions by these data which can justly be rejected because the embedment-temperature/soak-temperature regimes can never be comparable; the data analysis is carried on without them. Nevertheless the figures are still available in section 12 for the reader to inspect. Less reliance is placed on these figures until they are investigated further, perhaps by a parallel decrease in the water soak temperature.

Another curious feature of the 34° data which continues to be observed when the experiments are repeated is that the difference in behaviour between Mugga and Prospect
which is strongly in the direction of less stripping with Prospect at temperatures 46 and above is often reversed at 34°.

As stated above, the main focus of the experimental program has been to investigate the effect of embedment time/temperature conditions on stripping, and to extend the applicability of the test up to asphalt production temperatures. A limited study of the effects of soak time was made but is not discussed further. The author had been unaware at the commencement of the experiment that the South Australian & Queensland versions of the test were conducted for much shorter embedment & soak times. Future experiments may investigate the most appropriate conditions and also to take on board potentially valuable features of the Vialit test.

Other materials used in the experiment included N422, an adhesion agent from a well known supplier. The diluted form N422/60 was not used. Hydrated lime was used in a couple of experiments because there is a belief that it can help to reduce stripping in asphalt.

Adhesion certainly changes with temperature. Viscosity changes significantly with temperature also, therefore the experimental design must be adequate to cope with the varying viscosity, and if possible to separate the viscosity/temperature characteristic of the binder from any other adhesion related property. It has been found that the most profitable way of organizing the adhesion data is in terms of temperatures and temperature shifts. Because viscosities cover an unwieldy range of values, they tend to be unfriendly to the general reader, whereas a measure such as softening point is rather more readily understandable.

Instead of softening point, the temperature at which the viscosity is 170 Pa.s has been used. This temperature is 60° for C170 bitumen. For other binders the temperature has been estimated from the 60° viscosity or consistency according to the current Austroads standards AGPT/T121 and AGPT/T111 3 plus a measurement by ARRB elastometer at a higher temperature and a shear rate of 0.3 s⁻¹ (for the binders more viscous then C170), actually a great deal of viscosity data has been collected, so that, should the equiviscous temperature (EVT) concept prove to be inadequate; capillary and elastometer data at a range of SHRP temperatures from 46 to 82° are available. The viscosity temperature data were interpolated on a log (log + 3) scale the same as the Heukelom chart.

The 170 Pa.s EVT is about 12° higher than the softening point. A simple ad hoc change was made to T230 to allow “modified binders” to be embedded at Softening point +20° this can be seen as a slightly easier concession compared with targeting a 170Pa.s EVT, nevertheless it is believed to be inferior to relating the testing conditions to the asphalt production conditions. As only a couple of PMBs are included in this report, arguments re “underlying viscosity” and consistencies do not impact on it very much. However for this work to be universally applicable an agreed technique for reporting the viscosity or EVT of PMBs is desirable.

To a very rough approximation, changes in viscosity between 52 and 100 or so degrees may be considered to double or halve for a 6° temperature change: this simple approximation is examined further in section 5.4. It is certainly broken at 34°.

4 Syneresis

Syneresis is a common phenomenon in many rubbery and soft elastic solids. All kinds of food products e.g. custard, yoghurt, junket in which casein from milk is jelled with a fair amount of water may at first set with no separate water phase, but after a while the elastic casein consolidates and water is expelled from the elastic phase forming curds and whey.
Jam, sauces &c thickened with pectin behave similarly, as does tofu. Also the watery layer found on paint. Syneresis is reduced and slowed in watery food products by thickening the watery phase with xanthan gum. Jellied sweets “lollies” are commonly covered with sugar crystal or chocolate compound or wrapped in waxed paper to prevent the adhesion promoted by exudation from adhering to the container or sticking to the fingers of the consumer.

Those young enough to remember vinyl; for example document folders and car upholstery will remember that it could sweat and stick to things like printed documents, even acting as an unwanted printing press; a few generations earlier, linoleum and rubber goods would likewise sweat. These are examples of syneresis involving consistencies much greater than foodstuffs and liquid phases other than water.

Bitumen also exhibits syneresis; there are two mechanisms whereby a gel structure can form. Blown bitumen is rubbery, having a definitely measurable elastic recovery. The gelled phase is composed of polar molecules called asphaltenes clumping together. As with water in junket, a certain amount of non-polar oily molecules can fit in the spaces between the polar molecules, but as the polar structure consolidates by making more links between polar molecules, the oily phase is expelled. In extreme cases the bitumen, especially when strained (as in a ductility experiment) has a grainy appearance; various authors term this “colloidal instability” or “Paraffinic demixing”. Although this clearly applies to high PI bitumens of PI, say 6 and does not apply to low PI bitumen say –2 it is probable that some weak aggregation of asphaltenes even occur at PI –2 and there is no clear demarcation. What we are looking for in section 7 of this report is a numerical measure of syneresis directly related to syneresis and not a surrogate like PI. Shell developed an “exudation droplet test” which became part of their Qualagon4. “Such bitumen exude an oily component which can seep into the stone, this leads to a type of adhesion which is sensitive to the presence of water” and “paraffinic demixing beyond a certain limit has an adverse effect on both adhesion and cohesion” several authors believe that if there are sufficient oils of slight polarity and “resins” rather than a dichotomy between paraffinic oils and asphaltene, the bitumen is more “balanced” and less likely to have adhesion problems. The resins being the equivalent of xanthan gum in the aqueous food systems. Cottage cheese & old fashioned yoghurt both containing no xanthan gum are crumbly and non-adhesive, in contrast to creamed cottage cheese and creamy yoghurts are adhesive and “creamy” because they do contain such gums.

The other well known gel which can form within bitumen is due to SBS. As with asphaltene structures in ordinary bitumen, the clumps of cross-linking can be “fully networked”, “gel type”, or “not fully networked” analogous to “sol type”5. Some fully networked SBS exhibit a type of syneresis manifested in laboratory testing as “pulling out of the mould” although no field failures have been conclusively traced to this phenomenon; such PMBs can be compacted more easily than their viscosity would lead one to believe, so slip due to the same phenomenon may explain it. Some PMBs other than SBS, in particular, Du Pont Elvaloy can crosslink and form gels. When an SBS PMB is first melted, it exhibits a ropy or baby-cereal-like texture until the SBS structure is broken down by further mixing and higher temperatures. Both a grainy structure (bulk) and a smooth continuous phase (coating the inner cylinder) can be seen in Fig 2 of the reference cited6.

Syneresis taking the form of oil exuded from the bitumen can be quantified by sprinkling a powder on the surface and afterwards brushing it off and extracting the oil from the powder with solvent. Although the procedure described in section 7 requires specialized TLC plates and a UV source, once set up it is simpler and less labour intensive. It can be seen that it is a variant of the powder technique.
Although junket curds are in equilibrium with the whey, the equilibrium can be shifted by putting the junket on kitchen paper or hanging it in a cheese-cloth. As the water drains out, the junket turns into various types of cheese. Bitumen does the same type of thing in contact with porous aggregates and mineral fillers in the field; it behaves similarly in the laboratory when in contact with the porous silica gel of a TLC plate.

5 Data Analysis

5.1 The Master Curve

Some typical T230 data at various temperatures are presented in Figure 5.1, it can readily be seen that the percentage stripped varies with temperature. At the standard embedment temperature, 60°C all three systems exhibit significant stripping whereas there is little stripping at 120°. The other conditions of the tests presented in Fig 5.1 are: clean, dry, unprecoated aggregates and “as received” binder with no cutter or adhesion agent, standard embedment time (24hr) and standard soaking and evaluation procedure. It is noticeable that the data for the two classes of Kurnell refinery bitumen with the same Prospect dolerite aggregate seem to form approximately one population, whereas the yellow points of the more acidic Mugga microgranite lie to the right of the dolerite points. If the Mugga points were all shifted by something like 15 or 20° to the left they would seem to merge with the other points.

![Figure 5.1: Typical T230 data reported at various embedment temperatures](image)

In Figure 5.2, one of the sets of Prospect dolerite data are shifted slightly along the abscissa axis to maximise the overlap of the two populations, then the Kurnell C320-Mugga data are shifted the same amount to the right to achieve the greatest overlap with the previous two. This procedure is continued with a total of eight binder-aggregate systems. Two aggregates are represented as well as bitumen from more than one source, C170, C320 and AR1000/320 multigrade, two RTFO treated and two with adhesion agent.
In Figure 5.2, if it was not clear in Figure 5.1, it is now readily apparent that the characteristic shape of the data is a sigmoid curve. The sigmoid shape is idealized in Figure 5.3; this is formed as a piece function of three cubic polynomials which maximise the correlation coefficients with the eight sets of data presented in Figure 5.2. The $r^2$ of the middle third of the curve is 0.89 while the left and right portions’ $r^2$ is 0.72. The curve of Figure 5.3 is the master curve.

The number of degrees any data set needs to be shifted along the axis of abscissas to achieve the greatest overlap [that is, to minimize the sum of squared residuals] with the master curve is a measure of the adhesion merit of that data-set or aggregate-binder combination.
Data have been shifted to the left and to the right to form the master curve, so the shifts may be positive, negative or zero. A high positive shift is indicative of good adhesion performance or relatively low stripping. No significance is attached to zero shift because the choice of the first data set was arbitrary.

The old-fashioned way of creating the master curve is to plot the first data set on paper and the second data set on transparent film, and then to “eyeball” both data sets into coincidence and note the shift on the x axis. Then the shifted second data set is plotted on the paper and the procedure repeated with the third set of data on the transparent film. And so on. This was in fact done, but the results reported here are not derived from such a subjective graphical procedure.

The procedure adopted was curve fitting to a piece function cubic and to maximise the correlation coefficient as each new data set was added. If the shift for each new data set had to be determined by trial and error, then the process would have been tedious. Fortunately the old-fashioned graphical estimates could be used as a guide and the maximum \( r^2 \) was always found in three trials. The master curve had stabilized sufficiently after eight sets of data, each composed of six or seven T230 results at different temperatures that one does not continually need to modify the master curve for each new set of data, particularly as several of these are composed of only three or four T230 results. The shift of any new data is referred to the master curve established by the first eight.

### 5.2 Precision of T230 and AS1141.50

The obvious way to estimate precision is by replicating some tests. Although we have tried it in six cases, it is too little to be relevant here. This type of study is more appropriate to be managed by a national committee and would be designed to estimate both repeatability and interlaboratory reproducibility.

A different procedure was used in this work. Comparison between Figures 5.2 and 5.3 reveals the fuzzy scatter of the real data of Fig.5.2, the spread of the data from the master curve is a measure of the precision of the test. The sums of the squared deviations of the individual data from the idealized master curve are the basis of estimating the precision. The data used are all the data collected, not just the eight sets which formed the master curve. For the purposes of presenting the sum of squares data, these residuals were grouped and averaged (applying Bessel’s correction \( n-1 \)) in various ways. For example, it was desired to know if the two aggregates would yield different precisions with the same group of binders, or if there was any significant difference between the precision of AS 2008 types, PMBs or binders containing adhesion agents. All this information is summarized in Table 5.1.
Table 5.1: Precision statistics of T230 estimated by deviations from the master curve

Because the population has a sigmoid form and because the % stripped are confined between 0 & 100%, normal distribution statistics do not apply to it. One could apply the transformation \( \sqrt{TR \times (100-TR)} \) as was used in the analysis of torsional and elastic recovery precision statistics\(^7\), but in this paper, a simpler approach has been taken, focused on the single long-standing requirement of RTA specifications, minimum 10% stripping.

Assuming more than 10% stripping is considered to be an unacceptable level, and aware that stripping tests are not precise, then in practice one may target some lower figure, say, 6% or maybe 0.5% and then desire to know how reliable or precise a measurement in this region was, both in case of auditing or to gain confidence that field work based on this testing would be safe from stripping. Therefore the precision in the region 0-15% stripping is of most interest, and for the purposes of this study it is assumed that the spread about the master curve will obey normal distribution statistics if the range is broken into 0-15, 16-84 and 85-100. Also because of the symmetrical shape of the master-curve and rather less data in the range 85-100 % stripping, those data have been lumped in with the 0 – 15 % data. Before doing so, a statistical test was applied to the two populations of residuals which demonstrated that they did not differ significantly.

It will be noticed that the 2\( \sigma \) estimated for the range 16-84% stripped is roughly 2 to 3 times that in the range 0-15 (or 85-100), thus confirming the initial contention that a single \( \sigma \) estimate would be invalid.

The 2\( \sigma \) for AS 2008 binders (in the range 0 - 15%) is 6.8% but the corresponding figures for PMBs and adhesion agents are 13.1 and 11.5%. Is the AS 2008 really different from the others? We can apply the “Variance Ratio” test, Fisher’s F.
In the case of the AS 2008 the variance is 11.6 estimated from 32 residuals while for Non-AS 2008 PMB, Tar. Multi & Blond binders’ variance is 42.6 estimated from 23 residuals. The variance ratio is 3.7 which is significant at the 1% level (2.55) minimum variance ratio.

In short, the precision of AS 2008 binders (6.8%) is significantly better than the other binders, and does not really justify making an average estimate of precision (11.4%).

Many people would say that a precision of 6.8% is not very good when the specification limit is 10%. The precision in the range 0-10 is likely to be better than 6.8% (because the precision 0-15 is better than the 16-84 precision) nevertheless, in order to be confident in routinely passing a 10% specification, it would be wise to target % stripping 3.2% or below; and as also noted above, the intention of the RTA specifications was to encourage adhesion conditions very well removed from the critical area.

One of the major achievements of this work is that the accuracy, confidence and reliability of T230 measurements can be increased by replicating the test at different temperatures. The present version of the test can of course be replicated at the standard 60°, and in principle more and more accurate estimates of the % stripping at 60° can be achieved. In practice it may work this way. If the test conducted at standard conditions reports, a % stripping in excess of 30 %, although it can be used to predict what conditions it will take to achieve less than 10% strip, this is no substitute for an actual measurement in the range desired. Also with certain materials, particularly PMBs, the aggregate appears to be held firmly, yet after considerable effort, the stone can be pulled away clean of binder except for perhaps a small trace which is nevertheless able to support the filter paper.

By repeating the test at a different temperature and the results more-or-less conforming to the model database presented here will give a huge boost to confidence in the whole test procedure and the results for the particular material tested, doubts about the subjective aspect of a single T230 will be dispelled if two or more fall on the master curve. It will become apparent later (if not already) that most systems without precoat or adhesion agent particularly those for asphalt applications, as well as the standard 1 day at 60° would also benefit from another test at some higher temperature than 60° for example ¾ hr at 140°. Similarly, in order to increase the precision, confidence and reliability of tests with precoats and adhesion agent, as well as the standard 1 day at 60° the merit of these systems can be brought sharply into focus with another test, say 1½ hr at 52°. It is when all this data is brought onto the one master curve and a single crisp estimate of the shift can be reported for the binder – aggregate system that its value will be appreciated.

When several measurements enter into a single reported % stripping, read off from the shifted master-curve, precision estimates given in the Table 5.1 are no longer relevant. Assuming an approximation of normal distribution statistics applies in the ranges 0 - 15, 16 - 84 and 85 - 100 taken separately, a result based on n measurements will have a precision of \( \frac{1}{\sqrt{n}} \) times the precision given in the Table 5.1.

Although T230 is currently reported as a % stripping under specified conditions, it is now becoming clear that a much friendlier method of reporting is a temperature shift. That is, for any new system, how many degrees does it need to be shifted to coincide with the master curve of Fig 5.3? If it needs to be shifted up (positive shift), it means it has relatively good adhesion likewise a downward shift means relatively poor adhesion.

When people become comfortable with temperature shifts as a means of describing adhesion properties, the question will then arise, what is the precision of a temperature shift?
Expressed as 2σ or 95% confidence & based on a single T230 plate, the precision is easily estimated by projecting the % stripped confidence interval on the master-curve and reading off the temperature.

As will be seen from the last line of Table 5.1, the confidence limit of a shift measured in the range of stripping 0-15% or 85-100% is almost three times higher than for shifts estimated from 16-84% data. It will not seem strange when you consider the different slopes of the master-curve and consider what your practical expectation would be to a question like, how much would you expect a T230 3% strip to change with a 6° change of temperature?

The value of at least one measured T 230 falling in the range 16-84 towards producing precise shift estimates is readily seen. When more than one measurement is taken, the precision of % stripped will come down from, say, 25.8% to 12.9% with four measurements; and in terms of shift down to 5°. Although the simple statistical model adopted is not able to allow calculation of a precise estimate from mixed data in both extreme and mid domains, nevertheless, both the % stripped and the shift will come down by approximately 1/√n where n is the number of T230 plates.

Without precise and reliable measures of stripping, and the ability to factor out the effect of temperature on viscosity, correlations with potential factors thought to influence adhesion such as syneresis and acid value are impossible; this is why the syneresis project funded by Western Australia a few years ago was only able to produce some tantalizing indications. It is likely that the rheological (viscosity) properties of binders will have some effect on adhesion. By reporting adhesion in units of temperature enables viscosity, which can also be reported as a temperature (EVT) to be dealt with simply and efficiently.

5.3 Effect of Embedment Time

The effect of temperature on the properties of bitumen and asphalt both in the lab and the field are well known. The same asphalt which does not rut in a shaded cutting or a cold climate will rut in a hot climate. Time affects the properties in a similar way, for example, the same asphalt which does not rut even with heavy traffic on a flat section of highway may rut in a climbing lane with somewhat less traffic travelling at ¼ speed (= four times the loading time). Bitumen which is brittle only at -15° under the eleven second loading time of the standard Fraass test may break at +10° under the 0.015 second loading time of the Fast Fraass test. As will be noted further in section 6, several researchers have been able to pool data collected at an array of conditions covering several times and temperatures. A rheological mastercurve incorporating both time and temperature data is produced, together with a key which relates temperature shifts to time shifts. Here we examine whether similar behaviour pertaining to adhesion can be demonstrated in the laboratory. In the field, it is well known that a job which initially had poor adhesion could improve after several hours or a few days, especially if there was no rain during this period.

Several binders have been tested for T230 at some embedment time other than one day, in addition to the standard one day. The times used were two days, eight days, 1/8 day = 3 hours and 1/32 day = ¾ hour.

The percentage stripping for the times other than one day was compared with the master curve for the standard one day, and the shift (that is, the temperature at which the one day curve had the same percentage stripping as the different time experiment) corresponding to the particular time has been plotted in Figure 5.4.
Figure 5.4: Mean shift °C from one day embedment as a function of Log2 embedment time, days.

The slope of the curve is such that at about two days’ embedment, a doubling or a halving of the embedment time would result in a shift of 5° whereas at about three hours embedment, doubling or halving the embedment time would result in an adhesion change equivalent to a 7.1° shift on the master curve.

If one accepts the equivalence of time and temperature e.g. as illustrated in Figure 5.4, then one could add additional points to the master curve of Figure 5.3 from data collected at times other than one day, and in so doing perhaps construct a more accurate or robust master curve. Such a use of the “superposition principle” has not yet been attempted. It should be noted that Figure 5.4 is the result of averaging a limited number of observations each of which has a significant uncertainty (see Table 5.1) much more data would be required to increase the accuracy or robustness of the master curve in this way.

5.4 Effect of Binder Viscosity

Viscosity and rheological measures in general are a poor predictor of adhesion properties. The correlation with viscosity of a set of the most reliable data on AS2008 and multigrade binders reveals hardly any correlation of curve shift with EVT. Adding adhesion agent treated binders to the data set produces another outlying curve which again does not correlate with viscosity. $R^2$ are low, about 0.02. In section 7.2 it will be demonstrated that once an independent measure which does correlate with adhesion is found, making an additional shift to equalise the effect of viscosity will improve the correlation, but as noted above, the effect of adhesion agent is so powerful that the data form two distinct populations (with and without adhesion agent) which are better analyzed separately.

It has been mentioned several times above and below that there is a simple rule of thumb that binder viscosity changes by a factor of two for a 6° change in temperature. Although it should not be assumed that adhesion should follow such a rule, the following table illustrates how good an approximation the rule of thumb is in the case of viscosity:
<table>
<thead>
<tr>
<th>Temperature Range</th>
<th>Change in Viscosity for a 6° Temperature Change</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>C170</td>
</tr>
<tr>
<td>25 - 45</td>
<td>4.14</td>
</tr>
<tr>
<td>45 - 60</td>
<td>2.55</td>
</tr>
<tr>
<td>60 - 65</td>
<td>2.17</td>
</tr>
<tr>
<td>65 - 80</td>
<td>1.95</td>
</tr>
<tr>
<td>80 - 105</td>
<td>1.83</td>
</tr>
<tr>
<td>80 - 120</td>
<td>1.57</td>
</tr>
<tr>
<td>120 - 135</td>
<td>1.39</td>
</tr>
</tbody>
</table>

Table 5.2: The effect of a 6° temperature change on the viscosity of bitumen.

If the rule of thumb was universally valid, every field would be “2”.

A corollary to the master curve hypothesis, and in support of a suggestion that adhesion as well as viscosity has time-temperature equivalence, and bearing in mind that viscosity is the quotient of stress and strain rate and thus is directly proportional to time, it should be pointed out that the 6° for a two fold viscosity change is equal to the to the 6° for a two fold time change in adhesion in Figure 5.4.

It was stated above that “viscosity ……is a poor predictor of adhesion properties” that is, in the context of the whole lot of potential binders e.g. AS2008 types, colourable, PMBs, multigrades, tar, some with adhesion agent, etc. When one has a range of differing viscosity binders from the same refinery & crude or a range of the same factory PMB variants based on the same set of ingredients then a functional relationship between adhesion and viscosity may be discerned. This is demonstrated by taking standard production C170 and C320 from the same refinery and blending them with the same oils and propane precipitate as are used to make those standard bitumens and thus make samples of C50, C170, C320 and C1200 with viscosities at 60º of 50, 170, 320 and 1200Pa.s respectively. When tested by T230 at 100º for 24 hours and with clean Prospect dolerite aggregate, the results are respectively: 0, 22, 27 and 40% stripped.

6 Relationship with ARRB research

It is beyond the scope of this paper to review the voluminous world literature on bitumen adhesion. Australian practitioners will be familiar with E.J.Dickinson’s exposition of the subject in his book[10]. In Fig. 9.3 we see a sigmoid curve relating to the wetting of aggregate by bitumen. Although it is significant that water is not involved in this experiment, nevertheless the shape is similar to the master curve of the present work discussed in section 5.1 above.

The width of Dickinson’s sigmoid curve between 15 and 85% adhesion is 1.85 log_{10} or about 70 times viscosity. The sigmoid curve of Figures 5.2 & 5.3 covers 31° between 15 and 85% adhesion. Between the temperatures 45° and 76° which are separated by that same 31° the viscosities of a C170 at those two temperatures differ by the same 1.85 log_{10} as observed by Dickinson. It should be noted that much of Dickinson’s work was done with cutbacks and below 60°. The two studies are therefore complimentary.
On p163, Fig. 7.14 of Dickinson’s book we see the equivalence of time and temperature of his rheological model. The dashed portion of the curve between 50 and 60° is equivalent to $0.66 \log_{10}$ time units or a time factor of 2.5 per 6°. The SHRP researchers Christensen and Anderson extended their temperature data to 80° (Fig.8) they actually measured similar figures to Dickinson’s dashed line. This factor of 2.5 is comparable (but not identifiable) with the factor of two in time per 6° presented at the end of section 5.4.

Figures 9.4 and 9.5 of Dickinson’s book illustrate the effect of embedment time on wetting (and thus adhesion) of aggregate.

A factor of 8 embedment time between 3.75 & 30 minutes or from 2.5 to 20 minutes yields the following % adhesion data. From the present master curve of Fig. 5.3 the temperature figure tabulated below is an estimate of the temperature shift equivalent to an 8 fold time shift:

<table>
<thead>
<tr>
<th>% Adhesion</th>
<th>Temperature Shift</th>
</tr>
</thead>
<tbody>
<tr>
<td>15 to 42.5%</td>
<td>15°</td>
</tr>
<tr>
<td>51 to 93%</td>
<td>26°</td>
</tr>
<tr>
<td>56 to 90%</td>
<td>20°</td>
</tr>
</tbody>
</table>

That is, a mean shift of 6.8° for a doubling of time (comparable with 5° to 7.1° of Fig 5.4.

The above three illustrations are meant only to demonstrate that the two studies are compatible and complimentary, similar results would be obtained if instead of reading the shifts off from Fig.5.3 the sigmoid curve of Dickinson’s Fig 9.3 was used.

Dickinson finds that adhesion agents had little effect on wetting time in dry conditions; this could be because adhesion agents’ advantages are only perceived in water-wet conditions or that the contact times he used are so short that the rate of diffusion of the adhesion agent to the aggregate surface is insufficient. Because T230 is so consistently dependent on the time and temperature conditions when dry, it is likely that the “stripping” we observe is the difference between a superficial mechanical adhesion, and true chemical adhesion.

Dickinson’s illustration of the effect of washing a quartzite aggregate (from 44 to 88% retained) corresponds with a 22° shift from the dusty state to clean and dry.

### 7 Correlation with Syneresis

The syneresis was measured by RTA test T543, developed as described in a report by the author. The test is similar to the exudation droplet test. A small blob of the binder approximately 3.5mm diameter is impressed on a silica gel TLC plate (e.g. Merck 60F254) the plate is developed for 72hr at 65° during which time it is believed the oil which exudes from the binder is absorbed by the surrounding porous plate coating. The oils being aromatic are absorptive to UV and reduce the bright fluorescence of the zinc sulphide included in the TLC plate coating. Especially looking at digital camera images magnified 5 to 10 times, one sees the glossy black bitumen blob; between the bright light-green fluorescence of the TLC plate and surrounding the black blob, a ring of a darker bluish-grey colour is seen; this is identified with the exudation or syneresis. Closer to the black bitumen blob is a dark green ring. An unequivocal interpretation of the nature of the two kinds of ring is not attempted in this paper. The raw syneresis data is presented in columns 6, 7 & 8 of section 9, they give respectively, the total syneresis, the width of the dark green ring only and the total syneresis less the dark green ring, that is, the blue-grey part of the syneresis only. In the case of the tar alone there is a small dark brown ring & the blue syneresis especially noticeable to the naked eye gradually weakens and may be greater than reported.
7.1 Method of Syneresis Data analysis

High mastercurve shift is associated with high adhesion; this is not an experimental finding, it is the convention adopted. Consider two binders/aggregate systems of the same shift and the same aggregate but of different binder viscosities.

For practical purposes, the T230 shift unmodified by viscosity is the most relevant measure of adhesion. But, to understand the chemistry of binders of different viscosity grades it may be argued that the more viscous binder would otherwise find it harder to flow around the texture of the stone and so wet it, therefore as both binders have the same shift the more viscous one must have some positive adhesive chemistry compensating for the higher viscosity. The more viscous binder has the higher EVT so if EVT is added to shift the result should be a better measure of chemical adhesivity.

Conversely, if a measure of chemical or intrinsic adhesivity (e.g. Acid Value or syneresis or whatever or some combination) was found or proposed, it would be reasonable to subtract or discount it for viscosity before a practical adhesion measure was reported.

7.2 Correlation of Syneresis with T230 Shift

Various Pearson (product moment) correlation co-efficients ($r^2$) were computed ringing the changes on these variables:-

- Prospect and Mugga aggregates computed separately.
- EVT alone.
- Shift alone.
• Shift corrected for EVT i.e. Shift + EVT
• Total syneresis
• Syneresis beyond (excluding) the dark green ring
• All 21 binders for which reliable master curves were available
• Those 16 pure binders of the above 21, that is, excluding adhesion agent and lime.

If the argument above re shift + EVT was correct, one would expect the correlation coefficient to increase significantly. If the correlation coefficient decreased, it would mean the argument had got the “sign” wrong and the analysis should be repeated recalculating with the opposite sign. If there was no significant change in the correlation coefficient it would mean that EVT was even less important that we had first thought.

The question is: what is a significant \( r^2 \)? All the computed \( r^2 \) are summarized in Table 7.1. Properly \( r^2 \) should have only positive sign; in Table 7.1 the sign given is the sense of the actual correlation, that is, positive sign means that increasing shift (better adhesion) is associated with increasing syneresis.

<table>
<thead>
<tr>
<th></th>
<th>All data, including Adhesion agent &amp; lime aggregate</th>
<th>Data excluding Adhesion agent &amp; lime aggregate</th>
</tr>
</thead>
<tbody>
<tr>
<td>Quantities correlated</td>
<td>Prospect aggregate</td>
<td>Mugga aggregate</td>
</tr>
<tr>
<td>Total syneresis with:</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Shift + EVT</td>
<td>0.0388</td>
<td>0.08</td>
</tr>
<tr>
<td>Shift only</td>
<td>-0.0002</td>
<td>0.001</td>
</tr>
<tr>
<td>EVT only</td>
<td>0.2086</td>
<td></td>
</tr>
<tr>
<td>Syneresis beyond green ring with:</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Shift + EVT</td>
<td>0.0215</td>
<td>-0.111</td>
</tr>
<tr>
<td>Shift only</td>
<td>-0.0007</td>
<td>0.0048</td>
</tr>
<tr>
<td>EVT only</td>
<td>0.1834</td>
<td></td>
</tr>
</tbody>
</table>

Table 7.1: Correlations between adhesive temperature shift and syneresis with & without viscosity correction

In order to judge whether a correlation co-efficient is significant, two model cases have been tested to the “student’s \( t \)” criterion:

\[
t = \frac{r \sqrt{N-2}}{\sqrt{1-r^2}}
\]

For the population of all binders \( N=21 \), is an \( r^2 \) of 0.2 significant?

Answer \( t = 2.2 \) (19° freedom) this is significant at the 95% confidence limit.

For the population of pure binders (without adhesion agent or lime) \( N = 16 \) is an \( r^2 \) of 0.42 significant?

Answer \( t = 3.18 \) (14° freedom) significant at better than 99% confidence.
We are now able to review the table of $r^2$ more critically. For the set of all binders the $r^2$ are rather low; only the EVT (viscosity) correlated; there is no significant correlation with the T230 shift.

When adhesion agents and lime are excluded, and considering first the Prospect aggregate and looking first at the figures from total syneresis; for EVT alone one can be 80% confident of a correlation. With the master-curve shift alone the confidence of a correlation is greater than 95% by adding the EVT to the master-curve shift the confidence rises to 99.5%.

Therefore one of the objectives of this project has been proven as well as anything is ever proven at a practical level, yes, there is a correlation between syneresis and adhesion and surprisingly for some people, the greater the syneresis, the better the adhesion. Although this is apparent from the attached tables, to some it all appears as “too much information” this simple example is illustrative:

<table>
<thead>
<tr>
<th>Binder</th>
<th>Syneresis mm</th>
<th>% Strip Prospect 64°</th>
<th>% Strip Mugga 70°</th>
</tr>
</thead>
<tbody>
<tr>
<td>Clyde C320</td>
<td>0.8</td>
<td>84</td>
<td>94</td>
</tr>
<tr>
<td>Shell Multi 1000/320</td>
<td>1.3</td>
<td>43</td>
<td>59</td>
</tr>
</tbody>
</table>

**Table 7.2:** Simple illustration of the relationship between T230 stripping and T543 syneresis

The multigrade which has the higher viscosity and the higher syneresis in fact strips less than C320. This does not prove any causal relationship between syneresis and adhesion. The binders which exhibit high syneresis may also have some other common property such as high AV which influences adhesion. The fact that the correlations do not work for adhesion agent is a demonstration that syneresis is not the only important factor; in fact when adhesion agent is present (perhaps above some threshold) it is the dominant factor. Adding adhesion agent does not increase syneresis; both syneresis and the presence of adhesion agent are positively correlated with adhesion, but they are not correlated with each other. It is now normal practice to make asphalt without adhesion agent, while ever this is the case, the results of studies without adhesion agent and the rôle of syneresis will be relevant.

Looking further at the summary table of correlation under Prospect aggregate and now considering the syneresis beyond the dark green ring, although the T230 shift is higher than with total syneresis, after correcting for EVT, the best correlation is slightly inferior to that obtained with total syneresis, these differences are at best marginally significant e.g. if one syneresis estimate changed by 0.05mm it could turn the figures around.

Looking across to the Mugga aggregate, the raw correlations of T230 shift are somewhat lower than with Prospect and hardly any different whether excluding the dark green ring. When the EVT is added there is a modest increase in $r^2$ with the total syneresis but the syneresis beyond the green jumps to the highest correlation of all. So there is an equivocal answer to the question “what is the best method of reporting syneresis, inclusive or exclusive of the dark green ring? For Prospect aggregate the answer is “total syneresis” by a narrow margin and for Mugga aggregate the answer is clearly “beyond the dark green ring”. At the end of the day maybe it doesn’t matter.

The practical conclusion to this work is that syneresis should not be used as a routine test to manage adhesion and stripping; it takes longer and is more subjective than T230 and in any case is only one of many contributing factors to adhesion, whereas T230 (especially
when the time & temperature more nearly matches field conditions) is clearly simulative of performance. The reason this work has been done is the concern in some quarters that multigrade might have more exudation or syneresis than AS2008 types (yes it does) and that the higher syneresis would cause poor adhesion (it does not), indeed, no causal relationship between adhesion and syneresis is claimed.

8 RTA Cutback Chart and Master Curve Shifts

With nil cutter and unprecoated moisture-free aggregate (Zone AB) the “Sprayed bituminous surfacing cutback chart” RTA form #382 suggests C170 bitumen will adhere at temperatures above 56°. As the type of aggregate is not mentioned, it is reasonable to assume that the chart is intended to be conservative, that is it should work for all ordinary aggregates including the more difficult ones such as the Mugga.

Does C170 bitumen adhere so well to bitumen for 24hr at 56° that it cannot be displaced by water? Answer: According to the T230 test, no.

According to T230 testing reported in this project and with data from various temperatures smoothed by reference to a master curve, the % stripping one should expect at 58° for clean dry unprecoated aggregate is:

<table>
<thead>
<tr>
<th></th>
<th>Mugga</th>
<th>Prospect</th>
</tr>
</thead>
<tbody>
<tr>
<td>Kurnell C170</td>
<td>95</td>
<td>84</td>
</tr>
<tr>
<td>Clyde C170 (pre 1998)</td>
<td>99</td>
<td>85</td>
</tr>
<tr>
<td>Clyde C170 current</td>
<td>97</td>
<td>60</td>
</tr>
</tbody>
</table>

Table 8.1: Typical stripping of unprecoated aggregates with unmodified bitumen at 58°

It is important to note that there is such a large shift between the conditions and customary criteria (<10% strip) of the T230 test and what is reasonable and achievable in practice. If the RTA cutback chart is ever queried it is usually suggested that it is too conservative, that is, that it requires too much cutter or the practitioner would only accept the lower end of the recommended range of cutter. If changes in the suggested direction were made it would make an even higher shift away from T230 conditions.

A temperature of 58° as entered into the cutter chart will obviously not be maintained for 24hr so the true shift is larger still. If the road temperature subsequently rose to 64° for an hour per day or if the seal could settle in for 8 or 16 days before the next rain then better adhesion would be achieved.

<table>
<thead>
<tr>
<th></th>
<th>Mugga</th>
<th>Prospect</th>
</tr>
</thead>
<tbody>
<tr>
<td>Kurnell C170</td>
<td>61</td>
<td>34</td>
</tr>
<tr>
<td>Clyde C170 (pre 1998)</td>
<td>97</td>
<td>64</td>
</tr>
<tr>
<td>Clyde C170 (2004)</td>
<td>89</td>
<td>13</td>
</tr>
</tbody>
</table>

Table 8.2: Illustrating improved adhesion, 8 days at 64° or 16 days at 58° or 1 day at 82°

Section 6 of the RTA “Sprayed Sealing Guide” treats 0.5% adhesion agent as normal practice. Under these conditions the stripping of all C170 even at 58° will be less than 2%. In this context there is no contrariety between T230 and the cutback chart.
In another experiment with four binders and both the Mugga and Prospect aggregates tested in both the clean and dusty state, it was found the mean shift from the dusty to the clean state was 13° with the shift of the Mugga being perhaps a little higher. Dickinson's results for a quartzite aggregate imply a shift of 22°.

In consideration of the adhesion testing of multigrade, it is true that most multigrades tested with most unprecoated aggregates at 60° will fail T230 at the 10% criterion, but so do C170 and C320. In fact, even although multigrade has 5 times the viscosity of C170 which one might expect would make it harder to achieve adhesion, it in fact adheres better than C170, on average as if C170 was at 12° higher temperature. T230 is practiced without cutter because the companion T238 addresses initial adhesion with cutter. In sealing technology, T230 is meant to be relevant for stripping in the days after any added cutter has evaporated.

What else can the RTA cutback chart tell us?

The effect of precoating. From the middle of Zone AB to the middle of Zone CD is 17°. Thus a dry precoated aggregate will give the same adhesion, on average at 17° lower temperature than the clean non-precoated aggregate (and 39° lower than the dusty unprecoated aggregate). The chart recognizes variations in precoating; the range of shift due to precoat is 11 to 22°. For precoated aggregate damp with water, the average shift is 9°. Compare this with the data recorded in section 9. No precoats have yet been measured in the present program.

The effect of cutter. Four percent cutter produces a temperature shift of about 10°. The present experimental program has not aimed at re-validating this aspect of the cutback chart. So to date only a couple of cutbacks have been included for demonstration purposes. A 4% cutback with one aggregate produced a shift of 4° and with the other aggregate, 16° an overall average of 10° shift (as predicted by the cutback chart).

9 Archive Data

Over the years and especially in the 10 years prior to 1990, the RTA/DMR amassed a large testing database on precoats and adhesion agents. Unfortunately, these were focused purely on whether a system C170 + standard aggregate + precoat or adhesion agent passed the <10% stripping criterion. Nothing is recorded about the % strip of the aggregate and bitumen system without precoat, nor is the source of the C170 recorded and, of course, no one thought to run any of the embedments at different temperatures. Nevertheless, some information relevant to the present work can be distilled out of it.

All the precoats and adhesion agents were tested with five standard aggregates. Some adhesion agents performed very well with most of the aggregates, providing little discrimination, such data are not presented here. All the rest was averaged with the following overall results of T230 % stripped:

<table>
<thead>
<tr>
<th></th>
<th>Basalt</th>
<th>Peat's Ridge</th>
<th>Dolomite</th>
<th>Prospect</th>
<th>Quartzite</th>
<th>Marrangaroo</th>
<th>River Gravel</th>
<th>Nepean</th>
<th>Microgranite</th>
<th>Mugga</th>
</tr>
</thead>
<tbody>
<tr>
<td>Adhesion Agents</td>
<td>8.4</td>
<td>19.4</td>
<td>33</td>
<td>36</td>
<td>61</td>
<td>75</td>
<td>68</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Precoats</td>
<td>2.8</td>
<td>19</td>
<td>33</td>
<td>42</td>
<td>75</td>
<td>75</td>
<td>68</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Mean</td>
<td>5.6</td>
<td>19.2</td>
<td>33</td>
<td>39</td>
<td>61</td>
<td>75</td>
<td>68</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Converted to temperature shift with master curve</td>
<td>37</td>
<td>21</td>
<td>14</td>
<td>12</td>
<td>0</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Table 9.1: Mean percentage stripped of various precoats and adhesion agents with five standard aggregates.

The difference between Prospect dolerite and Mugga microgranite is expressed as a shift of 21º. For the present work (Section 12) the differences between the two aggregates (final three columns) are separated into those binders without adhesion agent, difference between Prospect and Mugga is 28º whereas when both are tested with adhesion agent, the difference between the same aggregates is 7º.

“Surface conditions” of aggregates: the archive contains a huge amount of data of various precoats tested at the different surface conditions. An analysis of archive data of precoats reveals that relative to “clean & dry” taken as zero, dusty and saturated surface wet are both shifted -6º while saturated surface dry is shifted -4º. It is believed the archive data on adhesion agents displays meaningless differences between the surface conditions. All are pretty much the same except that both saturated surface dry and saturated surface wet are marginally high by +2º this is believed due to the fact that the saturation process is equivalent to extra washing.

Although as stated above, most archives do not record the T230 results of a control, an exception was the work requested by RTA Tamworth for six aggregates tested clean and dry required for their 1998 – 1999 sealing program. The bitumen was Caltex C170 the adhesion agent was 1% N422/60 and the precoat was Colbind C:

<table>
<thead>
<tr>
<th>Aggregate</th>
<th>WTG</th>
<th>ARDI4</th>
<th>CL1Q</th>
<th>CL14</th>
<th>D1</th>
<th>W1</th>
<th>Mean Aggregate</th>
</tr>
</thead>
<tbody>
<tr>
<td>No adhesion agent</td>
<td>No Precoat</td>
<td>85</td>
<td>87</td>
<td>42</td>
<td>35</td>
<td>78</td>
<td>4</td>
</tr>
<tr>
<td>No adhesion agent</td>
<td>Precoat</td>
<td>0</td>
<td>58</td>
<td>9</td>
<td>1</td>
<td>28</td>
<td>8</td>
</tr>
<tr>
<td>Adhesion agent</td>
<td>No Precoat</td>
<td>0</td>
<td>0</td>
<td>2</td>
<td>0</td>
<td>4</td>
<td>0</td>
</tr>
<tr>
<td>Adhesion agent</td>
<td>Precoat</td>
<td>0</td>
<td>0</td>
<td>12</td>
<td>0</td>
<td>28</td>
<td>0</td>
</tr>
</tbody>
</table>

Table 9.2: T230 % strip of six Tamworth aggregates, various combinations of adhesion agent & precoat, with control.

The mean aggregate results (admittedly computing such a mean would be of dubious validity in a planned research program) were referred to the master curve, from which the following shifts are read off:

1% N422/60 +36º
Colbind C +17º

It is comforting that the 17º shift for the precoat is exactly the same as the mean shift implicit in the RTA Cutback chart mentioned in section 8. Bearing in mind that N422/60 is 40 to 30% active compared with neat N422, the comparable data of the present work (section 9) is about 80 for 1% and 0.5% N422 but only 6 when tested with multigrade. It is interesting to note that no extra performance gain on T230 over adhesion agent alone was observed with adhesion agent in combination with precoat. Discrimination in favour of precoats is achieved with the T238 initial adhesion test.

As it has been stated above that the present work demonstrates that multigrade strips less than C170 a fact which may surprise some readers, the following additional evidence is adduced, taken from work done with a Yass aggregate. Two samples of multigrade (softening pt 52.5º) and two of C320 (softening pt 49.5º) were tested with 14mm Bald Hill aggregate by T230 with an embedment temperature of 71º. With as-received aggregate, everything
had 98% or more stripping. With clean dry aggregate the following T230 % stripped (duplicates are the two different samples) were observed:

<table>
<thead>
<tr>
<th></th>
<th>Multigrade</th>
<th>24, 14</th>
<th>% strip</th>
</tr>
</thead>
<tbody>
<tr>
<td>C320</td>
<td>88, 66</td>
<td></td>
<td>% strip</td>
</tr>
</tbody>
</table>

When read off against the master curve, the Multigrade is shifted 23° towards better adhesion than C320. In the present work, for both C320 samples compared with both multigrade samples, the multigrade is shifted on average 8° towards better adhesion with Mugga aggregate and on average 13° towards better adhesion with Prospect aggregate.

9.1 Pioneer T230 Adhesion study for Asphalt

In 1996 and again in 1998 AAPA submitted a table of data generated by Pioneer on the T230 test at temperatures 90, 130/135, 160 and a few at 60° for consideration by the RTA of the most appropriate T230 test conditions for asphalt. A paper by J. Lysenko described the experimental procedure and noting that the conditions of the test designed for use with sealing binders are “relatively severe but provide a useful insight” for sealing works. For asphalt, even conducting the embedment at Softening point + 20ºC was believed to be insufficient. A temperature of 135° was suggested (with 24hr embedment) it was found that under these conditions, all binders passed the maximum 10% strip criterion regardless of whether an adhesion agent was added. The data have been referred to the master curve and compared with RTA data in the following table:

<table>
<thead>
<tr>
<th></th>
<th>Dolerite Prospect</th>
<th>Basalt Bald Hill</th>
<th>Basalt Bass Point</th>
<th>Microgranite Mugga</th>
<th>Andesite Martins Creek</th>
<th>Conglomerate Teralba</th>
<th>Quartz Tenterfield</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>clean</td>
<td>clean</td>
<td>dusty</td>
<td>clean</td>
<td>dusty</td>
<td>as received</td>
<td></td>
</tr>
<tr>
<td>Multigrade 1000/320</td>
<td>+29</td>
<td>+30</td>
<td>+14</td>
<td>-3</td>
<td>-2</td>
<td>-30</td>
<td>-54</td>
</tr>
<tr>
<td>C320</td>
<td>+16</td>
<td>+7</td>
<td>+4</td>
<td>-7</td>
<td>-22</td>
<td>-54</td>
<td>-58</td>
</tr>
<tr>
<td>A3L, ABS, A15E</td>
<td>-12</td>
<td>no result</td>
<td>-4</td>
<td>-20</td>
<td>-30</td>
<td>-52</td>
<td>no result</td>
</tr>
</tbody>
</table>

Table 9.3: T230 Master curve shifts °C comparing five aggregates studied by Lysenko with 3 standard aggregates, this work.

A significant demonstration in the above table is that Multigrade consistently strips less than C320 and would strip the same as a C320 if its coating temperature were a value representative of the following estimates: 13, 23, 10, 4, 20, 24, 4° lower than C320 (average 14°). We also note that as expected there is a strong tendency for the basic aggregates to have a positive shift and for the acidic aggregates to have a negative shift. Also although the A3L (SBS A15E) is shifted to a somewhat lower adhesion than C320, it is only a few degrees (average 5). These data and their interpretation are recognized as a valuable contribution, the only point of disagreement would be that when choosing laboratory temperature conditions typical of asphalt production, time scales relevant to the production and laying of asphalt should also be chosen (suggest ¾ hr).

One should be careful in making detailed inferences from the above table, the RTA results are all from clean aggregate, and the surface condition of the Pioneer study is, “dusty – as received” also the RTA results are based in two sources each of C320 and multigrade. The A15E are from different suppliers. A guide to interpreting the mixed clean-dusty data is that the dusty figures should be shifted by +13° to make them comparable with the clean data. Asphalt is produced from aggregates in the dusty state, therefore it is reasonable that T230 testing for understanding the performance of asphalt should be performed in the dusty state.

The point AAPA was making at the time is that most of these binders have >>10% stripping at 60 and 90° and even at 135° some aggregates give significant stripping at the standard
twenty four hour embedment time. In view of the twenty four hour laboratory embedment
time compared with the shorter times of real asphalt production the RTA believes it would
be justified in requiring an adhesion agent or T639 testing in the case of an aggregate like the
Teralba conglomerate.

10 An Integrated System

A large number of binders have been rated with two aggregates and several aggregates have
been rated with common binders, hence it should be possible to predict the % stripping of
any of the binders with any of the aggregates at any temperature and any time of
embedment. New aggregates and binders can be included in the database by measurements
in conjunction with materials already in the database at a few temperatures. We have a
rough idea of the effect of precoats, adhesion agents and surface conditions of aggregate, in
principle this information can be increased and improved.

The single transportable number identified as desirable in section 2, for every material
involved in thermoplastic binder – aggregate adhesion has been achieved; it has the units of
temperature and is manipulated by simple addition of the component elements. To increase
the confidence in assessing a binder, instead of one aggregate, a set of standard aggregates
would be prudent. The RTA standard aggregates may not always be major-use-
representative aggregates; even in NSW, the Prospect and Penrith Quarries are nearing
exhaustion. If this work finds favour, a committee either NSW or National may wish to
define a set of standard aggregates differently.


10.1 Application to Specific Problems

A stripping problem is reported in the field. Details of prior testing, daily work records and
weather conditions both at application and failure are collected. What went wrong? And
how do we prevent it next time?

As well as collecting data as mentioned above, samples of the actual (precoated) aggregate
used should be collected as well as samples of the failed work (whether seal stones from the
side of the road or asphalt cores) should also be routine.

For seals, plot the actual conditions used on the cutback chart. How many degrees of
temperature is the plotted point on the safe or dangerous side of the chart
recommendation? If adhesion agent was used or contemplated, another point should be
plotted, shifted 50º to the right of the first one.

For seals and asphalt also note the T230 result converted to a temperature. In the case of
asphalt at least one of the T230 test condition variants must simulate asphalt production;
three quarters of an hour at 140° is recommended. Investigators of stripping failures should
also have access to similar information computed for jobs which did not strip.

10.2 Statistical Opportunities

Asset managers may note differences in performance re incidence of stripping of certain
lengths of highway or of certain work crews or contractors. What is the difference
between their work practices and materials (e.g. aggregates) used? Can the worst case be
improved?
10.3 Adhesion Agent and Cutter

The strongest tool we have to improve adhesion is the well-known fatty amine adhesion agent (except that it is ineffective in the case of multigrade, which already has superior adhesion compared with AS2008 types). The above work demonstrates shifts in the order of 70 to 90° for 0.5 to 1% adhesion agent. Unfortunately 0.1% adhesion agent does not seem to be proportionately effective, there is some threshold (related to the AV of the bitumen) which must be exceeded. Therefore adhesion agents should be tested before use. There may be a maximum level, not only because of cost but also because adhesion agent is a surfactant, too much may have an emulsifying effect. The RTA cutback chart indicates the shift to be expected from precoat, but not from adhesion agent. This is because the effect of adhesion agent is only noticed under wet conditions, as mentioned in section 6. Also the effect of adhesion agent is the exception to the principle that T230 results transformed to a temperature are additive. They may be additive in the case of AS2008 bitumen and other binders with low acid value, but the same figure (about 80°) cannot be used in the case of multigrade, where the shift is only 2° to 6°. Multigrade has a shift in adhesion approximately 7° better than C320; when RTFO treated to simulate its effect in asphalt the adhesion of multigrade improves another 8° but no gain is achieved due to RTFO when adhesion agent is present, after RTFO it is as if no adhesion agent was present.

The effect of cutter is modest, slightly less than one would expect on the basis of viscosity (EVT). One purpose of cutter in sprayed sealing is to give low viscosity at spraying temperature to achieve a wide even fan, and to achieve this at a lower temperature which will thus minimize cutter loss by evaporation before it hits the pavement. The principal purpose of cutter is to enable the aggregate to embed to half its ALD height and form a mosaic in a reasonable time (given the rolling regime and traffic), certainly before winter. Using cutter over and above the amount needed for the above requirements is risky, it often leads to bleeding problems in hotter weather and may even cause stripping because water finds it easier to push low viscosity cutback off the aggregate. Therefore, aggregates with adhesion issues should be managed with both precoats and adhesion agents and not with cutter (except in the case of multigrade where amine adhesion agent is ineffective).

10.3 Hydrated lime

When C170 bitumen is mixed with 20% hydrated lime the adhesion is perhaps 2° better in the case of the acidic Mugga aggregate, but is 12° worse with the Prospect dolerite. When tested with 1000/320 multigrade, both aggregates perform worse than they would with no lime, the Mugga 36° worse, and the Prospect 26° worse.

At the 20% level, the lime-binder is still quite pourable when hot. The viscosity and EVT was measured on the actual mixtures used and at the 20% level hardly causes any change. It is believed that any chemical reaction between the lime and the bitumen has plenty of opportunity to occur; the same cannot be said about the lime and the surface of the aggregate. It is likely that when the hot binder-lime mix is poured into the T230 trays, most of the lime would sink, if not to the bottom, at least away from the surface aggregate contact zone.

The significant softening point increases reported particularly with multigrade occur at the 1:1 filler: binder ratios commonly used in asphalt formulations.

In the opinion of the author, the chemical interaction of lime with the surface aggregate is recognised as a possibility but without much appreciation of its likely practical effect. Any improvement due to lime in T640 Lottmann type tests is put down to the stiffening effect of the lime on the mastic and hence a reduction in the rate at which water could displace mastic from the large aggregate.
11 Conclusions and Findings

A single transportable measure of adhesion has been identified. A single number can characterize separately: binders, aggregates, adhesion agents and precoats, cutters. A set of these numbers for a large and representative range of binders is available as are results for two aggregates tested intensively plus a few other aggregates. The numbers can be manipulated by simple arithmetic so that systems e.g. binder + aggregate can likewise be predicted.
The single measure has the units of temperature; therefore it is directly compatible with the RTA cutback chart. The measure is obtained from three of four T230 measurements, at different temperatures which at once halves the uncertainty of the simple T230 test and increases the confidence that something meaningful is being reported. Far from increasing cost and T230 work load, it will dramatically reduce it. It will make testing with standard aggregates and standard aggregate conditions (20 in all) seem even less necessary than previously.

All the dot points of section 2 have been addressed positively.

**Summary of Findings:**

- A measure of the repeatability of a single T230 test has been reported, together with a framework which would allow an interlaboratory study to proceed directly and confidently to measure reproducibility.

- A significant positive correlation has been found between syneresis (exudation of oil) and adhesion.

- For the first time it has been demonstrated that there are significant adhesion differences between different local sources of C170 and C320.

- There is absolutely no evidence that there is any deficiency in the adhesion of multigrade. Given the viscosity of multigrade is three times greater than C320 one might think it “fair” to expect the multigrades to perform equivalently to C320 when the multigrades were processed through the asphalt plant or tested 10° higher. This is not the case; the multigrades are more adhesive than C320 so that it is the C320 which needs to be shifted 4 to 17° warmer than the multigrade to achieve the same adhesion.

- Adhesion agents make a huge difference to the adhesion of C170 and C320 binders 70 to 90° even with 0.5% adhesion agent.

- Adhesion agents hardly make any difference to the adhesion properties of multigrade, increasing it perhaps by only 5°. However, an implication is that C170 or C320 or AR450 with adhesion agent adheres better than multigrade with or without adhesion agent.

- RTFO treatment generally increases the adhesion power of a binder by 4° even when uncorrected for the increase in viscosity which one might expect would otherwise decrease adhesion by about 6°.

- So far it has been found that all adhesion behaviour can be referred to the same mastercurve (within the precision limits recorded in Table 5.1). For % stripping of any system in the mid-range (16 – 84%) an increase in temperature of 6° will decrease stripping by 15%.

- Instead of increasing temperature the same decrease in stripping could be achieved with 2.5 % cutter. Much larger shifts can be achieved with precoat (17°) and adhesion agent (conservatively 50°). At the ends of the % stripping range e.g. 5% stripping, an increase in temperature of 6° can only be expected to reduce the stripping to 4%.

- Also in line with practical field knowledge and now for the first time quantified with precision, a doubling or halving of time for adhesion is equivalent to a 6° change in temperature.
• The above point is very relevant for hot mixed asphalt which is made at a temperature of 140° and three quarters of an hour may be considered a typical minimum time between production and the handling steps which cause some additional mixing and coating before compaction. The difference between three quarters of an hour and one day is five powers of two which is thus equivalent to 5 x 6° lower temperature. Therefore in the case of asphalt 140 – 30 = 110° would be a fair T230 embedment temperature for a 24 hour test. It is especially significant to note that only at temperatures in this range does the % stripping pass the traditional criterion of 10% maximum. At 60° all binders without adhesion agents fail. Some additional work may be required to verify the time/temperature equivalence at 110 - 140°.

• The dimensions of the S shaped adhesion master curve are equivalent to the dimensions of the adhesion curve figure 9.3 in E J Dickinson’s book. Dickinson’s study and the present work are complimentary.

• The T230 methodology does not identify any positive adhesion effect due to hydrated lime.

### 11.1 Guidelines for Measuring the “Hyper – T230” Temperature

T230 is reported as % stripping. When T230 is measured at 3 to 4 temperatures on the same system and the results referred to the master-curve one can report a single temperature shift. Until this procedure is written up as a formal test method, it may be referred to as “Hyper T230”.

In order to report a reliable Hyper T230 at least three T230 results are required at different temperatures. At least one must be in the range 3 – 15 or 85 to 97% stripped and at least one must be in the range 16 – 84% stripped. The word “striped” in this context means simply the effect or condition of a failing system on the T230 test. We have evidence from this work and elsewhere that in some cases an apparently embedded aggregate particle may be pulled from the binder tray with little binder attached, whether or not there had been a water-soak. In the real world, loss of seal aggregate or crumbling of asphalt may be due to the aggregates never coated properly in the first place, T230 methodologies are an important investigative tool, but not the only consideration.

For all binders, that is: AS2008, multigrades, PMBs, colourable and scrap rubber containing less than 6% cutter and no adhesion agent, it is suggested that the three temperatures are 64°, 88° and 100° with standard one day embedment in each case.

In the case of any binder (except multigrade and colourable) with adhesion agent it is recommended that the three conditions be:

- 58° - 1 day, 52°- 1 day and 52° 1½ hour

Aggregates can be compared by measuring with two contrasting binders whose shift with the Prospect and Mugga is already known. For example an aggregate proposed for asphalt use might be measured with Kurnell C320 and A15E at 64, 88 and 100°.

Precoats are the only materials for which the surface conditions other than clean/dusty are relevant. They are only relevantly applied when the amount of precoat needed is determined for each of the surface conditions. A dusty aggregate will require more precoat than a clean one and an aggregate saturated with water will be able to accept less precoat. The practice of deliberately dusting an aggregate with foreign dust is not recommended.
At least two sealing aggregates should be used for evaluating a precoat, at least one of these in Saturated Surface Dry condition. The sealing binders for precoat testing should be C170 and either a S35E or S45R. A single test temperature of 76° should produce satisfactory results for both the precoated and unprecoated state. This testing requirement is much less than the previous regime. If a precoat is required for a particular project where both the binder and the aggregate are known, only this combination need be tested.

The “hyper T230” temperature is the shift from the data collected as above to the master curve of Figure 5.3 computed by minimizing the sum of the squares of the deviations.

### 12 Raw Data

The data on which this paper is based are presented in the accompanying Excel file. These data are presented for the sake of completeness, it is not necessary to refer to this table to follow the paper. In the main field of the table, particularly the twenty columns of time & temperature under the blue heading, the two numbers in the field separated by a vertical mark | are respectively the percentage stripped with the Mugga aggregate followed by the percentage stripped with the Prospect aggregate.

### References

1. Main Roads, former house journal of the DMR NSW (which became the RTA) Vol. xx No 4 pp 123 – 125 June 1955.