COMMENTARY TO AG:PT/T104 - PRE-TREATMENT AND LOSS ON HEATING OF BITUMEN, MULTIGRADE BINDERS AND POLYMER MODIFIED BINDERS BY ROLLER MODIFIED ROLLING THIN FILM OVEN (MRTFO) TEST

PREFACE

This test method was prepared by ARRB Group on behalf of Austroads. Representatives of Austroads, ARRB Group and the Australian Asphalt Pavement Association (AAPA) have been involved in the development and review of this test method

FOREWORD

Polymer Modified Binders (PMBs) and Multigrade binders can undergo significant changes in characteristics during the process of asphalt manufacture, transport and laying. The test specified in *AS/NZS 2341.10, Method 10: Determination of the effect of heat and air on a moving film of bitumen (rolling thin film oven (RTFO) test)*, simulates the effect of the hot mixed asphalt manufacturing process on asphalt grade binders manufactured to the Standards Australia Specification AS2008. The oxidative effects at mixing temperatures (typically in the range 150°C to 170°C) on the thin binder films that occur during asphalt manufacture, transportation and placement are simulated by the RTFO test conducted at an oven temperature of 163°C over a period of 90 minutes.

Unfortunately, even at this elevated temperature, the potentially complex flow characteristics of some binders can reduce the effectiveness of the test. This occurs primarily because the flow of the binder in the conventional RTFO bottle relies on the action of gravity as the bottle rotates, in contrast with the high shear coating of the aggregate in the pugmill where the thin binder films are quickly formed with minimal influence from the flow characteristics of the binder. The use of a metal bottle and roller in this test method will reduce this problem for the more flow resistant binders resulting in a more uniform treatment across the range of bitumens, multigrades and PMBs used in asphalt applications.

To reduce the testing variability due to the range RTFO oven designs, an oven temperature calibration method that ensures a more reproducible treatment temperature between ovens sourced from different manufacturers has been adopted. This should improve the precision contribution of the RTFO pre-treatment to subsequent property determinations.

A further application for the RTFO treatment has developed from the AAPA Code of Practice for the manufacture, making and laying of SBS modified asphalt. This Code provides recommendations covering the manufacture and transportation of PMBs modified with styrene-butadiene-styrene (SBS) polymer. A recommendation of the Code is to specify a limit on the amount of volatiles evolved from a PMB when handled at specified application temperatures. In the absence of a more direct measure of binder fume, the propensity for a binder to evolve volatiles can be estimated by determining the mass loss during asphalt binder handling, as simulated in the laboratory by the RTFO test. The method described in this document provides a measure of mass loss for the standard modified RTFO treatment procedure.

SCOPE

This test method sets out the procedures for pre-treating a wide range of binders and the determination of the Loss on Heating of asphalt grade binders including bitumens, polymer modified binders and multigrade binders. The method provides a pre-treatment for binders which are to be subsequently tested in accordance with the test methods set out by Austroads for asphalt grade binders.

Further Development

There are no further plans for the development of this test method.

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1 REFERENCED DOCUMENTS

The following documents are referred to in this method:

AUSTROADS

AG:PT/T101	Method of sampling polymer modified binders, polymers and crumb rubber
AG:PT/T102	Protocol for handling polymer modified binders in the laboratory
AG:PT/T103	Pre-treatment and loss on heating of bitumen, and multigrade binders (rolling thin film oven (RTFO) test)

AS /NZS

2341.10	Method 10: Determination of the effect of heat and air on a moving film of bitumen (rolling thin film oven (RTFO) test)
2341.13	Method 13: Long-term exposure to heat and air

AS

2008	Residual bitumen for pavements, Appendix B - SAMPLING
2341	Methods of testing bitumen and related road making products
AAPA	Australian Asphalt Pavement Association Code of Practice for the manufacture, making and laying of SBS modified asphalt

2 APPARATUS

The following apparatus is required in addition to that listed in AS/NZS 2341.10 and AS/NZS 2341.13.

a. Balance - A suitable balance of 1 kg capacity, readable to 0.01 g.

b. Treatment bottles - Metal treatment bottles and rollers in accordance with the description provided in Appendix C.

c. Oven carriage modifications - In accordance with the description provided in Appendix D (Informative)

3 PROCEDURE

3.1 General

Bituminous binders are complex mixtures of petroleum products. If handled in accordance with the directions of the suppliers, there should be no significant risk. It is recommended that notices, describing the action to be taken in the event of bitumen, multigrade or PMB burns be displayed in the laboratory in the areas where these hot binders are handled. A suitable warning could be as follows:

WARNING: HOT BITUMINOUS BINDERS CAN CAUSE BURNS

The following precautions should be taken when handling hot bitumen, multigrade or PMBs:

a. Eye protection, such as safety glasses and/or face shields, shall be worn when handling hot bitumen, multigrade or PMBs.

b. Heat-resistant gloves, with close-fitting cuffs, and other suitable protective clothing, shall be worn when handling hot bitumen, multigrade or PMBs.

c. There shall be no smoking while handling hot bitumen, multigrade or PMBs.

d. While the material is still cold, loosen the lid of the sample container (invert the can and warm the lid, if necessary), or punch a hole in the lid.

e. Examine the cold sample for the presence of water. If water is thought to be present, drain most of it out, or blow with clean compressed air to evaporate the free water.

3.2 Sample preparation

PMB samples for testing shall be provided in accordance with AG:PT/T101 and AG:PT/T102. Bitumen and multigrade samples shall be provided in accordance with AS2008, Appendix B: SAMPLING. It is recommended that all binders be handled in the laboratory in accordance with AG:PT/T102.

3.3 Oven calibration

The Oven treatment temperature (163°C unless specified otherwise) is established using the method given in AS/NZS 2341.13 Long-term exposure to heat and air. This establishes the 'in-bottle' temperature at 163°C with a floating Pt_{100} probe or suitable thermometer and is less dependant on the ovens design for good testing performance. A summary of the calibration procedure is presented in Appendix A.

3.4 Pre-treatment

Where specified, this procedure shall be followed as a precursor to the determination of asphalt grade binder characteristics.

a. Sample establishment

    i. Establish a suitable sub-sample in accordance with AG:PT/T101.

ii. Heat the sample in its container with a loosely fitted cover in an oven not exceeding the treatment temperature for the minimum time necessary to ensure that the sample is completely fluid. Manually stir the sample but avoid incorporating air bubbles.

iii. Select the required number of metal treatment bottles and rollers to produce sufficient material for the characterising tests to be performed on the residue.

iv. Weigh each clean treatment bottle and roller to the nearest 0.01 g and record the mass, M_1.

v. Pour 50 $\pm$ 0.5 g of the sample into each of the treatment bottles.

vi. Allow the treatment bottles to cool to approximately room temperature (18 to 25°C).

vii. Weigh the RTFO bottle containing the binder to the nearest 0.01 g and record the mass, M_2.

b. Treatment

i. With the oven at the calibrated treatment temperature (Appendix A, see Note 1) for at least 2 hours, insert the treatment bottles into the carriage in a balanced arrangement and fill any remaining spaces with empty bottles.

ii. Leave the samples for 30 minutes without rotating the carriage or any air flowing.

iii. Start the rotation of the carriage (see Note 2) and the air flow with the flow meter indicating 4 $\pm$ 0.5 L/min. Ensure the air jet is aligned centrally with the hole in the treatment bottle.

iv. Maintain the samples in the oven for 60 min ($\pm$ 10 s), then switch off the carriage and airflow.

v. Remove the treatment bottles from the carriage and stand the samples in the oven.

c. Treated sample collection (see Note 4)

i. Weigh a collection container and stirrer to the nearest 0.01 g for each binder and record as M_3.

ii. Leave the samples to settle in the treatment bottles for 1 to 2 minutes.

iii. Taking out one bottle at a time, pour the contents from each bottle into the pre-weighed container. Drain each bottle until the treated material stops running freely (just dripping).

iv. Allow the treatment bottle and roller to cool to room temperature without further loss of treated binder.

v. Stir the material in the container briefly to ensure that the contents are mixed to yield a representative sample and allow to cool to room temperature.

vi. Weigh both the room temperature treatment bottle (+roller) and the room temperature test container to the nearest 0.01 g. Record the mass of the RTFO bottle as M_4 and that of the test container as M_5.

d. Calculation (for a single treatment bottle per sample) (see Note 3)

Calculate the loss on heating using the following formula:

$$\text{Loss on heating (\% mass)} = \frac{100 \times \left[(M_2 - M_1) - (M_5 - M_3) - (M_4 - M_1) \right]}{(M_2 - M_1)} \quad (1)$$

Where M_1 = mass of the clean treatment bottle + roller

M_2 = mass of the sample + treatment bottle + roller (cold)

M_3 = mass of the clean sample collection container

M_4 = mass of the treatment bottle + roller after draining (cold)

M_5 = mass of the collected sample + collection container (cold)

4 INFORMATION TO BE REPORTED

The following information shall be reported:

a. The loss on heating, as a percentage mass to the nearest 0.1%.

5. PRECISION

An estimate of precision for *loss on heating* was obtained on a range of PMBs by Austroads in 1998 using the standard RTFO treatment (AS2341.10). The following criteria can be used for judging the acceptability of results:

Property	Units of Precision	Repeatability (r)	Reproducibility (R)
Loss on Heating	Units of Measurement (*)	0.06	0.2

where m is the mean test result

(*) While actual results for these properties are in percent, the generic term "units of measurement" is preferred to avoid any confusion between the absolute value (percent) or percentage of the actual value.

Notes

1. The appendices supporting this test method are described as “Normative” where the appendix must be followed (same status as the standard) and “Informative” where the information is provided to assist the user but is not mandatory.

2. Variations in the rotational speed of the carriage will contribute to the performance of the test. Experience has shown that some older carriage drive motors can slow with age due to a deterioration in the control capacitor. All ovens should be checked for conformance with the carriage speed specification given in AS2341.10.

3. The procedure described in this method is for a single treatment bottle and collection container per binder. Where multiple treatment bottles are used to process a larger quantity of binder, the following equation can be used:

$$\text{Loss on Heating (\% mass)} = \frac{100 \times \left[\sum_{1}^{n} \left[M_2^n - M_1^n \right] - \left(M_5 - M_3 \right) - \sum_{1}^{n} \left[M_4^n - M_1^n \right] \right]}{\sum_{1}^{n} \left[M_2^n - M_1^n \right]} \quad (2)$$

Where M_1^n = mass of the nth clean treatment bottle

M_2^n = mass of the nth sample + treatment bottle (cold)

M_3 = mass of the clean sample collection container

M_4^n = mass of the nth treatment bottle after draining (cold)

M_5 = mass of the collected sample + collection container (cold)

4. When treating binders with the roller method a wire hook (paper clip) can be used to support the roller over the open treatment bottle to encourage complete drainage of the binder back into the bottle. The clip should be included in the various stages of the mass loss determination when used.

A worksheet for this task is presented in Appendix B

APPENDIX A
RTFO OVEN TEMPERATURE CALIBRATION (NORMATIVE)

BACKGROUND

AS/NZS 2341.13 Long-term exposure to heat and air requires the establishment of an 'in-bottle' temperature of $100 \pm 0.3°C$ with a floating Pt_{100} probe (or other appropriate thermometer). The precision required by the standard is within the capacity of the modern oven temperature controller and when used with an external calibration step against a calibrated reference thermometer will provide a low cost alternative method to that described in the standard. Currently several of the laboratories conducting this test use a more expensive Leeds and Northrup bridge for this purpose.

This RTF Oven calibration procedure relies on the stability of the Pt_{100} resistive temperature sensor to determine the internal treatment bottle temperature with the carriage in motion. This version of the method introduces several new initiatives to improve the precision of the calibration. Information on the thermal recovery characteristics of the oven can also be determined

Pt100 CONTROLLER AS A THERMOMETER

A low cost option in resistance thermometry is described in WD 97-10.1 *Temperature measurement and Control in the Laboratory*. Experience with these units in situations where their calibration has been checked against a reference thermometer supports the view that their design provides an accurate long term measure of temperature to better than 0.3°C over a wide operating range.

RTFO OVEN CALIBRATION

1 SCOPE

This Appendix sets out a method for setting the oven temperature using a platinum resistance thermometer probe. The procedure uses the standard 163°C test as the basis for the description. Where other treatment temperatures are required, the calibration should repeated.

2 APPARATUS

The following apparatus is required:

a. **Platinum resistance thermometer probe**. The construction of this probe and a suitable pivot point are shown in Fig. A1 and Fig. A2.

The probe shall consist of a straight stainless steel tube of 3 mm diameter and 0.25 mm wall thickness which shall be terminated at one end by a rounded brass cylinder (the tip), 9 mm diameter and 40 mm long.

The other end of the stainless tube shall be terminated in the pivot in a manner that is stable at the oven operating temperature (up to 180°C). The length of the probe from tip to pivot point shall be 280 mm.

A 100 Ω platinum resistance temperature sensing element (Pt_{100}), of approximately 3 mm diameter and 30 mm length , shall be fitted in the tip and connected to the pivot by insulated wires (0.3 mm diameter) to form a four lead resistance thermometer. When used with a three terminal controller the leads can be reconfigured at the controller input.

> Note: PTFE or other temperature resistant (180°C) material must be used for insulation of wires and cable.

The probe shall be calibrated at 163°C against a reference thermometer calibrated to an uncertainty of ± 0.1°C.

b. Temperature controller Capable of measuring temperature with a Pt100 platinum resistance sensor with a resolution of 0.1°C and sufficient stability to repeat an annual calibration within ± 0.2°C .

c. Reference thermometer Capable of calibrating the probe and controller system to an uncertainty of ± 0.1°C**.**

d. Oven monitoring thermometer IP 16C or ASTM 16C.

e. Resistance bridge (optional) Capable of measuring the resistance of a 100 Ω four lead resistance thermometer with a resolution of 0.01 Ω and limits of error of ± 0.01 Ω

3 PROCEDURE

The procedures shall be as follows:

a. Pre-heat the oven to 163°C according to the monitoring thermometer or previous calibration if available.

b. Place the probe and reference thermometer into a suitable calibration bath or block and determine the temperature on the controller corresponding to 163°C on the reference thermometer.

c. Place 7 empty metal treatment bottles and rollers in the carriage. Place the tip of the platinum resistance probe in the remaining bottle containing 35 mL of oil (see Figure A2). Secure the probe such that the tip of the probe is located inside the bottle about half way along. When the carriage is rotated, the probe should follow the bottle movement without fouling the jet.

> Note: The metal treatment bottle used in this calibration must be fitted with a metal cap with opening conforming to the dimensions of the standard glass bottle. This allows the free rotation of the calibration probe.

d. Lead the cable from the probe out of the oven and connect to the Temperature controller (or Resistance bridge). Ensure that sufficient cable length remains within the oven to avoid stress on the probe-cable interface during carriage rotation.

e. Insert the oven monitoring thermometer securely into its service position. Commence air flow at 4 ± 0.5 L/min and allow the oven to stabilise at a set point of 163°C.

f. Reset the oven temperature as necessary until readings on the Temperature controller (connected to the probe) or the Resistance bridge indicate that the temperature in the bottle is 163°C. Take sufficient readings over the necessary period of time and record the temperature on the monitoring thermometer and oven controller. This reading becomes the oven calibration.

> Note: Most recently manufactured treatment ovens use Pt100 based temperature controllers. The set point on the oven controller can be used in place of the monitoring thermometer.
>
> The oven should reach a stable state after 2 hours of operation. Calibration should be undertaken with the laboratory at normal operating temperature. Ideally, the temperature will be in the range 18 to 24°C and the oven will not be exposed to direct heat from the sun.

g. After the calibration, remove the platinum resistance thermometer probe and pivot assembly from the oven. Ensure that the air jet is aligned with the opening in the treatment bottle. Application of the calibration probe can disturb its position.

4 RTFO Oven temperature calibration (thermometer method)

An alternative oven calibration procedure using a thermometer in the treatment bottle is described in AS/NZS 2341.13 APPENDIX B. A calibrated thermometer (IP32C for 100°C) is secured across the face of the carriage with the bulb passing through a hole in a modified treatment bottle. The thermometer required for a higher temperature calibration must be a total immersion type of length suited for the placement shown in Fig A3. Details or this method are provided in AS/NZS 2341.13.

5 RTFO Oven temperature recovery assessment

Using the same apparatus and procedure described in section B2, the temperature recovery characteristics of the oven can be assessed. Although this is not a routine calibration exercise, users should validate the characteristics of their oven to confirm its ability to attain the test temperature from a cold (sample and bottle) start. This should be assessed for the 35 gram glass bottle standard RTFO system with all bottles carrying 35 ± 1 g of oil. No additional testing is required for the alternative metal bottles.

The fully loaded oven should achieve greater than 160°C within the 30 minutes recovery period for a 163°C calibrated treatment temperature.

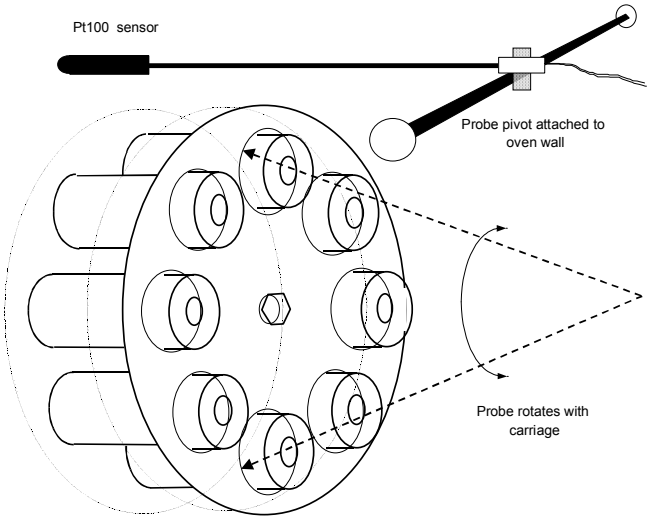

Fig. A1. Probe Construction and Oven Assembly

Note: Greater detail is provided in AS/NZS 2341.13 Appendix A. The probe as described in AS.NZS 2341.13 is recommended.

To calibrate an oven using metal bottles the bottle carrying the temperature probe must have a lid opening conforming to that of the conventional glass bottle. The other bottles can

An improved approach uses a 35 mL oil bath to improve thermal transfer to the tip of the probe. AS/New Zealand 2341.13 relies on air to achieve the heat transfer

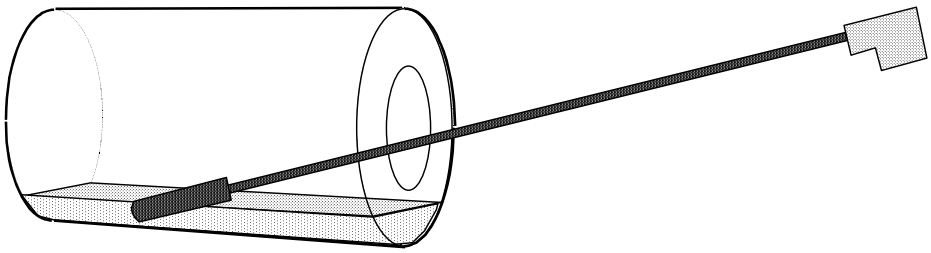

Fig. A2. Probe and oil bath

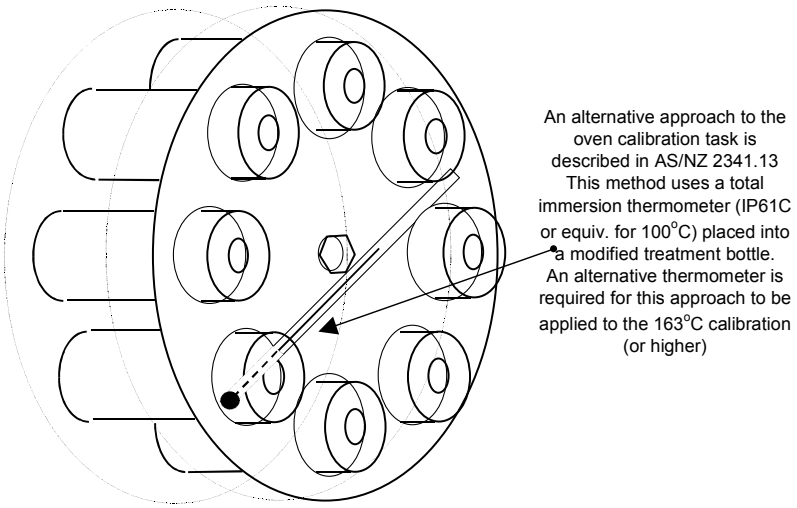

Fig. A3. Calibration thermometer (alternative approach)

APPENDIX B
LOSS ON HEATING CALCULATION (INFORMATIVE)

Initial Sample			
Mass Bottle	Bottle M_1	Bottle + Sample M_2	Sample $M_2 - M_1$
1			
2			
3			
4			
5			
6			
7			
8			
Total Mass of Sample S			

Treated Sample remaining in bottles			
Mass Bottle	Bottle M_1	Bottle + Treated sample M_4	Treated sample $M_4 - M_1$
1			
2			
3			
4			
5			
6			
7			
8			
Total mass of treated sample in bottles $T1$			
Treated Sample in collection container			
Mass collection container (M_3)			
Mass collection container + Treated sample (M_5)			
Mass of treated sample in collection container (M_5-M_3) $T2$			
Total Mass of Treated Sample $(T = T1 + T2)$			

$$\text{Loss on Heating} = \frac{(S-T)}{S} \times 100 = \quad \%$$

Note 1: All measurements in gram to 0.01g

Note 2: All bottle weighings include the mass of the roller and any roller support clip when used.

APPENDIX C
RTFO TREATMENT BOTTLE DESCRIPTION (NORMATIVE)

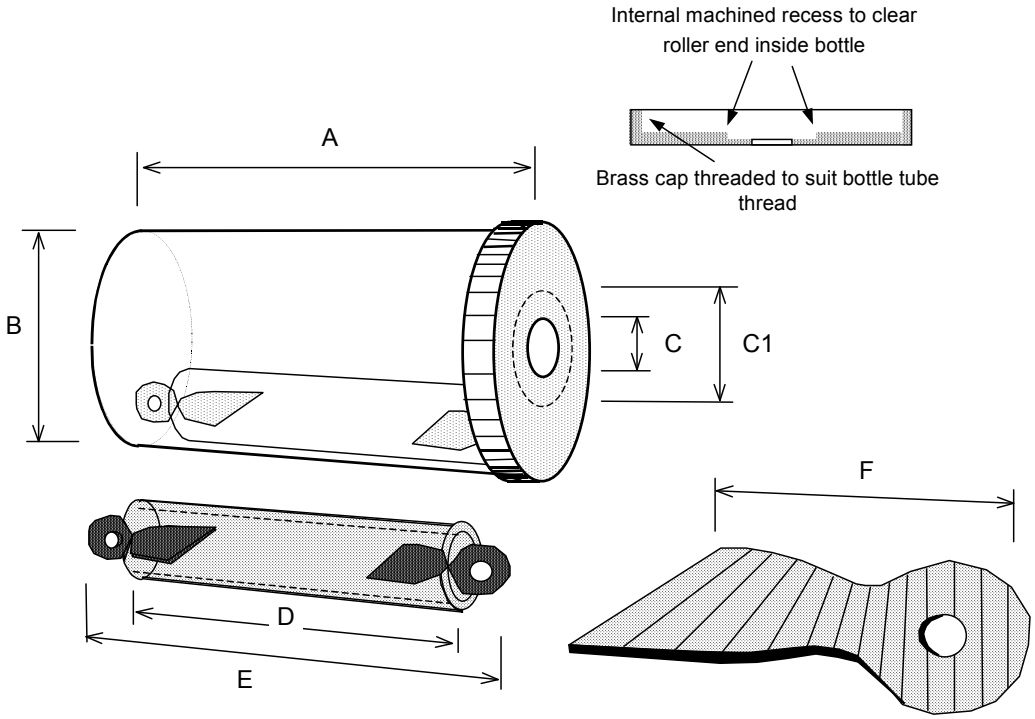

Figure C1. Metal bottle and roller assembly

Table C1 Description of items in Fig. C1

Item	Description
A	Brass tube (length) 140 ± 1 mm; Closed at base with 1.6 mm brass plate; External thread to match cap at top
B	Nominal (2.5 inch ϕ) 1.6 mm wall 63 ± 1 mm
C	Air hole in screw cap (diameter) 12 ± 0.5 mm
C1	Internal recess approximately 3 mm deep 30 ± 1 mm
D	20 mm ϕ standard brass threaded tube 120 ± 1 mm
E	Length of roller including ends 138 (+ 0) -1 mm
F	Length of 1.6 mm brass; end pieces 40 ± 2 mm with 90 degree left hand twist viewed from end of roller

APPENDIX D
CARRIAGE MODIFICATION (INFORMATIVE)

When using the heavier brass treatment bottles and rollers, a modification to the bottle retaining clips may be required to reduce the tendency for the treatment bottles to work out of the carriage.

Figure D1 describes a rearrangement of the bottle retaining clips.

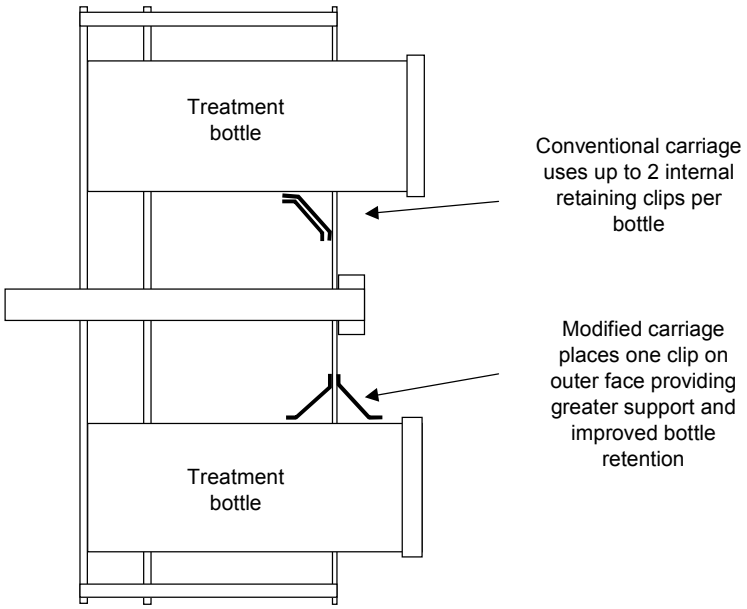

Fig. D1. RTFO bottle retaining clip modification

AMENDMENT RECORD

Amendment No.	Clauses amended	Action	Date
1	Commentary Page	New	June 2005
	Footer and header	Format	
	Applied revised test method number	Format	
	Applied new styles	Format	
2	Applied revised test method number	Substitution	March 2006
	Reformatted notes and moved to end of method	Format	

Key

Format	Change in format
Substitution	Old clause removed and replaced with new clause
New	Insertion of new clause
Removed	Old clauses removed