

Confidential Technical Report 45981

Date: 9th March 2006



*Registered Office:
Shawbury Shrewsbury
Shropshire SY4 4NR
United Kingdom*

T: +44 (0)1939 250383

F: +44 (0)1939 251118

E: info@rapra.net

W: www.rapra.net

PROJECT NO: AN0043

EXAMINATION OF THE SUITABILITY OF A POLYURETHANE SPRAY COATING FOR CONTACT WITH FOOD

LINE-X PROTECTIVE COATINGS LIMITED,
PO Box 244,
Barnstaple,
North Devon
EX31 2ZR

For the attention of: Mr Neil Whitton
e-mail: neil@line-x.co.uk

Joanna Gidlow

John Sidwell
Authors

Keith Scott
Business Manager
Polymer Analysis

EXAMINATION OF THE SUITABILITY OF A POLYURETHANE SPRAY COATING FOR CONTACT WITH FOOD

1. INTRODUCTION

A sample of polyurethane spray coating designated XS100 "Foodsafe", for walls and other surfaces in food preparation or storage rooms was supplied for testing to confirm its suitability for potential occasional contact with food. Test material sprayed on to aluminium foil was received on the 6th February 2006 and given the Rapra internal code RTL8374. Information was supplied that the polyurethane utilized methylene bis di-isocyanate (MDI) in the polymerization of the coating.

All food contact materials used in Europe are covered by the revised Framework Regulation 2004/1935/EC. This states that any material or article intended to come into contact directly or indirectly with food must be sufficiently inert to preclude substances from being transferred to food in quantities large enough to endanger human health or to bring about an unacceptable change in the composition of the food or deterioration in its organoleptic properties.

To confirm suitability of the coating for contact with food, the coating has been tested to the requirements of the Plastics Directive 2002/72/EC, Plastic materials and articles in contact with food.

2. CONCLUSIONS

Levels of overall migration from the coating into the food simulants distilled water and the alternative fatty food simulant isooctane under the test condition of 24 hours at 40°C are within the migration limit for plastics of 10 mg/dm². No transfer of colour to the colourless oil simulant HB307 was observed. The level of specific migration of the aromatic amine methylene diamine from the coating into distilled water was less than the migration limit of 10µg/kg. Therefore, from the analytical data obtained, the coating has been found to be suitable for contact with food in its intended application.

3. EXPERIMENTAL

Levels of overall migration

The level of overall migration into the following simulants was determined under the test condition of 24 hours at 40°C.

- a) Distilled water.
- b) The alternative fatty food simulant 95% ethanol.
- c) The alternative fatty food simulant isooctane.

The total immersion method was employed. Duplicate testing was undertaken on cut portions of sample of known surface area ~1 dm². Blank simulants were also examined. The level of overall migration was determined gravimetrically.

The nature of material extracted with 95% ethanol was examined by infrared spectroscopy.

Colour extractability

A portion of coating was immersed in colourless food oil simulant HB307 for 24 hours at 40°C. After this time period, the oil was examined for colour transfer.

Specific migration of restricted aromatic amines

The level of extractable MDA was determined using the technique of combined liquid chromatography/ mass spectrometry. A series of reference standards containing 2,2, 2,4 and 4,4 MDA isomers were prepared at concentrations of 1, 5, 10, 15, 20 and 50µg/L. Migration of MDA into the distilled water simulant was examined. (MDA can be formed by the reaction of any residual MDI with water). The sample and standard preparation was undertaken between the 7th and 8th February 2006. LC-MS Conditions employed are detailed in Table 1.

Table 1 LC-MS Conditions

Parameter	Set Point
Liquid Chromatograph	Agilent 1100 Series
Injection	100µl
Flow rate	0.400ml/min
Chromatographic column	Phenomenex Aqua C18 150mm x 2.00mm 3µ 125A compartment @ 30°C
Mobile Phase	Solvent A : 5mM Ammonium acetate (aq) Solvent B : Acetonitrile 8-11% B over 5 minutes. 11-90% B 5 to 20 minutes. Post time 8 minutes Total runtime 28 minutes
UV Wavelength	Signal A: 240-nm BW = 8; Reference 360-nm BW= 100 Signal B: 254-nm BW = 8; Reference 360-nm BW=100
Mass Spectrometer (MS)	Agilent Ion Trap
Ionisation	Atmospheric Pressure Ionisation- Electrospray (API- ES)
Spray chamber Conditions	Dry temp = 350°C Nebuliser = 40psi Drying gas = N2 @ 10L/min HV Capillary = 3500V
MS Trap Conditions	Scan 150-250m/z Averages = 5 Spectra Target Mass = 199 m/z (+ve ESI) Compound Stability = 100% Trap Drive Level = 100% Accumulation Time = 300000 µs Max. Accu Time = 300000 µs ICC Target = 30000 Charge Control = On Multiplier Voltage = 1830 Volt Dynode = 7KV

The analysis was undertaken on 8th February 2006.

4 RESULTS

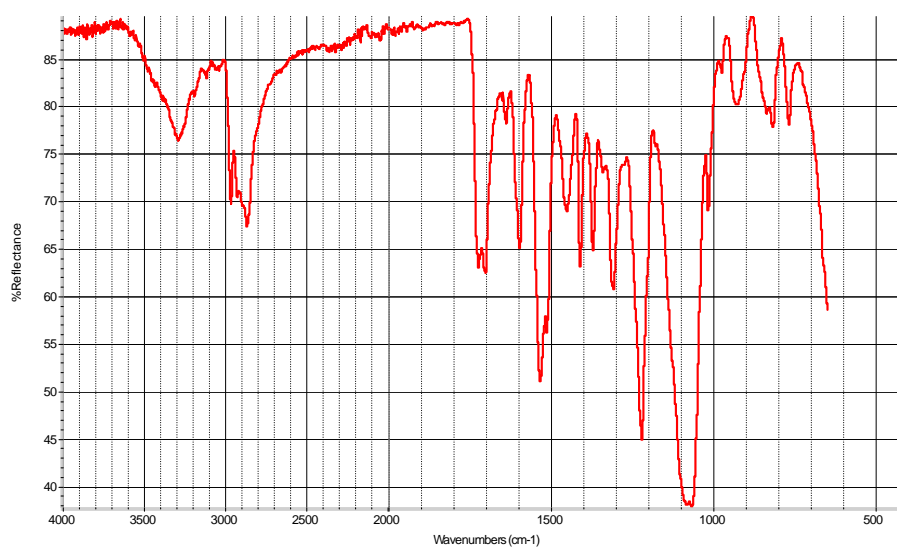
4.1 Overall Migration

Levels of overall migration were as follows;

Simulant	Migration (mg/dm ²)
Distilled Water	3.6, 3.7
Isooctane	1.5, 1.3
95% ethanol	161.7, 155.9

The level of migration into distilled water and the alternative fatty food simulant isooctane was less than the migration limit for plastics of 10mg/dm². The coating showed significant solubility in 95% ethanol indicating that this was an inappropriate alternative fatty food simulant. The extracted material in 95% ethanol was identified as being low molecular weight polyurethane by infrared spectroscopy.

AN0043. Golden Gate IR of 95% Ethanol test, 24hrs at 40°C.HF
AV16587



Rapra Technology Ltd

Figure 1 Infrared spectrum of 95% ethanol extracted material

4.2 Colour extraction

No extraction of pigment was observed after contact with the colourless food oil. This indicates that the pigment is not oil soluble.

4.3 Specific Migration of aromatic amines

The positive electrospray ionisation (ESI) chromatogram (m/z 199) obtained for the sample is shown in Figure 2. The positive ESI chromatogram obtained for a 10 µg/L standard of 2,2' 2,4' and 4,4' MDA is shown in Figure 3.

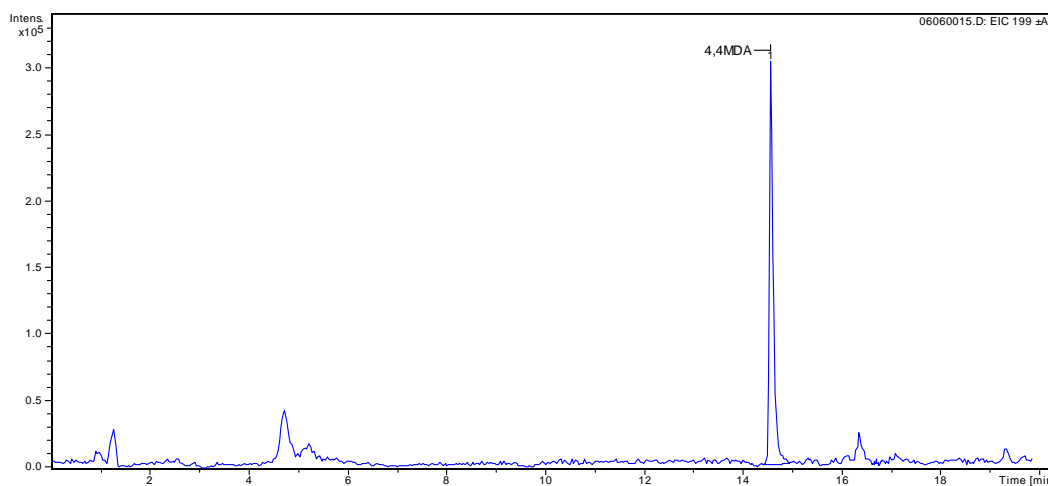


Figure 2 - Specific ion chromatogram of the water extract from the PU sample
(Note Intensity x10⁵)

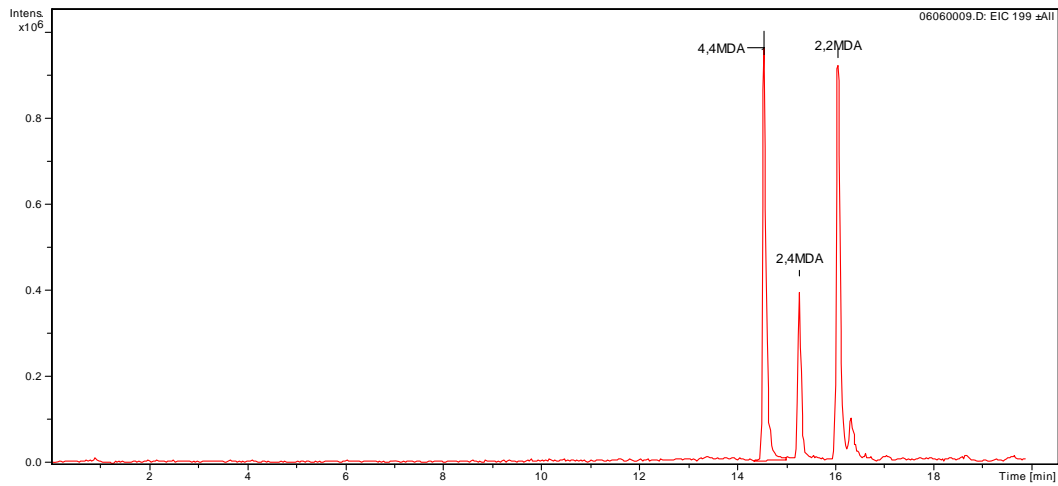


Figure 3 - 10µg/litre (10ppb) mixed standard containing 2,2' 2,4' and 4, 4' MDA in deionised water – specific ion chromatogram.
 Note intensity x 10⁶

Calibration data for 4,4' MDA in deionised water is shown in Figure 4.

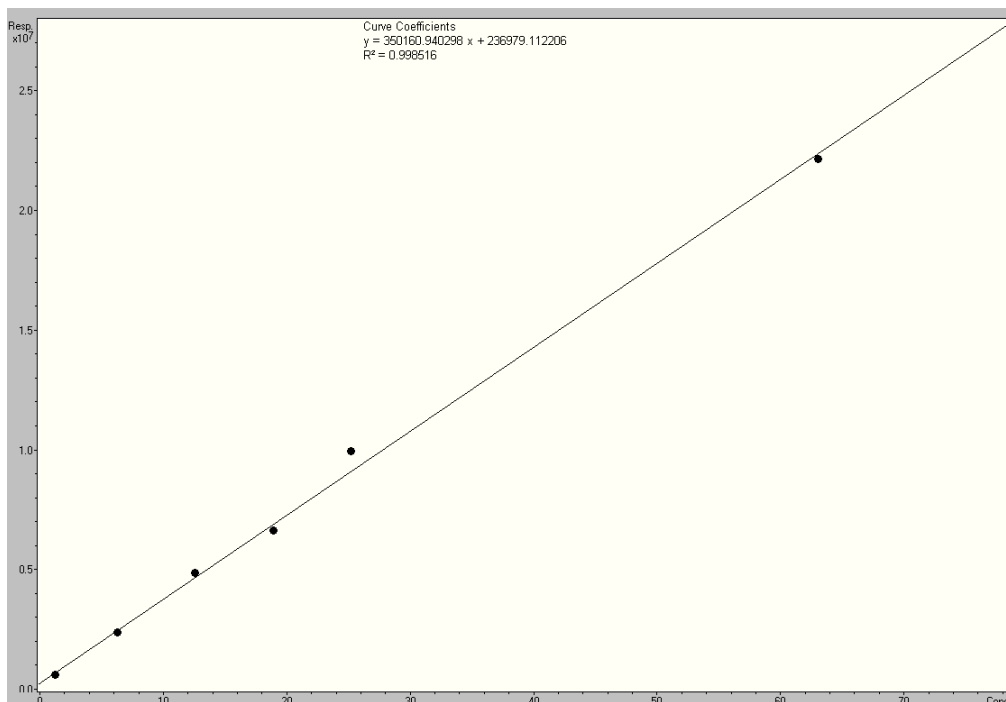


Figure 4 Calibration graph for standards of 4,4' MDA, 1, 5, 10, 15, 20 and 50 µg/litre (ppb).

The levels of 4,4' MDA found in the water extracts are shown in Table 2. No other MDA isomers were detected.

Table 2

Sample	MDA content of extracts			
	(µg/litre)	(µg/100ml)	(µg/dm ²)	(µg/6dm ²)
Sample RTL 8374 (1)	4.2	0.42	0.47	2.8
Sample RTL 8374 (2)	3.9	0.39	0.43	2.6

The specific migration limit for aromatic amines is 10µg/kg for 6dm² in contact with 1kg food (not detectable at this limit). The level of extracted MDA from the coating (mean 2.7µg/kg) after 24 hours contact with distilled water is below this limit.

**RAPRA TECHNOLOGY LIMITED UNITED KINGDOM
CONDITIONS OF BUSINESS**

FORMATION OF CONTRACT

- 1.1 All quotations are made and all orders are accepted subject to the following conditions. All other terms, conditions or warranties whatsoever are excluded from any contract between the parties unless expressly accepted by Rapra Technology Limited ("Rapra") in writing.
- 1.2 Quotations shall be available for acceptance for a maximum period of 30 days from the dates thereof and may be withdrawn by Rapra within such period at any time by written or oral notice. "Work" shall mean the work and services that Rapra agree to provide in the quotation.
- 1.3 If any statement or representation has been made to the Client by Rapra, or its employees upon which the Client relies (other than in the documents enclosed with Rapra's quotation) then the Client must set out that statement or representation in a document to be attached to the return copy of the quotation and in any such case Rapra may accept or reject the same and/or submit a new quotation.
- 1.4 The supply of materials, products or information by the Client pursuant to the quotation shall constitute acceptance of these conditions where acceptance has not previously been communicated by the Client to Rapra.

PRICES

- 2.1 All prices are, unless otherwise stated, quoted net exclusive of VAT.
- 2.2 All requests for variations or addition to the Work must be made by the Client in writing. In the event of any variation or addition being so requested and agreed to by Rapra, Rapra shall be entitled to make an adjustment to the contract price fairly reflecting such variation or addition.

PAYMENT

- 3.1 Unless otherwise agreed by Rapra in writing the terms of payment shall be 30 days from receipt of invoice by the Client, which shall be deemed to be two working days after posting. Rapra may submit interim invoices in respect of each stage of Work completed for the Client.
- 3.2 No disputes arising under the contract nor delays beyond the reasonable control of Rapra shall interfere with prompt payment in full by the Client.
- 3.3 In the event of default in payment by the Client Rapra shall be entitled at its option to treat the whole contract as repudiated by the Client or to suspend all further work on any contract or contracts between Rapra and the Client without notice and to charge interest on any amount outstanding at the rate of 2% per annum above the Base Rate of National Westminster Bank plc in force at the time when payment was due.

COMPLETION

- 4.1 Time for completion of Work is given as accurately as possible but is not guaranteed. The Client shall have no right to damages or to cancel the order for failure for any cause to meet any time stated for completion of Work.
- 4.2 Any estimate of the date of completion of Work shall in every case be dependent upon prompt receipt of all necessary information, instructions or approvals from the Client. Variations or additions to the Work requested by the Client may result in delay in completion.

CANCELLATION

5. Either party may cancel the contract on 30 days written notice to the other on condition that all costs and expenses incurred by Rapra up to the time of cancellation and, where cancellation is at the insistence of the Client, all loss of profits and other loss or damage resulting to Rapra by reason of such cancellation, will be paid forthwith by the Client to Rapra.

LIABILITY

- 6.1 Rapra undertakes that it will indemnify and keep the Client indemnified against all liabilities, costs and expenses in respect of claims in relation to death or injury to persons or damage to tangible property to the extent that such death, injury, loss or damage is attributable to the negligent acts or omissions of Rapra, its officers, employees, agents or sub-contractors.
- 6.2 Save where Rapra is shown to have failed to exercise reasonable care in the performance of the Work and such failure results in death or personal injury, Rapra shall not be liable in respect of claims arising by reason of death or personal injury. Further, under no circumstances whatsoever shall Rapra be liable for consequential loss, loss of profits, damage to property or wasted expenditure.
- 6.3 Rapra's liability, whether in respect of one claim or the aggregate of various claims other than claims for death or personal injury due to negligence on the part of Rapra shall not exceed £500,000 in any Year and the Client agrees to insure adequately to cover claims in excess of such amount.

CONFIDENTIAL INFORMATION AND INDUSTRIAL PROPERTY RIGHTS

- 7.1 All data, information and reports are produced for the benefit of the addressee only. Rapra accepts no liability arising from unauthorised use of such information or reports by a third party.
- 7.2 The Client shall not reproduce or abstract for the purpose of advertising or otherwise any report or other information on the Work or use the name of Rapra either expressly or by implication in any of its advertising or sales promotional material without the prior written consent of the Company Secretary of Rapra.
- 7.3 All drawings, documents, confidential records, computer software and other information supplied by Rapra are supplied on the express understanding that copyright is reserved to Rapra and that the Client will not, without the written consent of Rapra, either give away, loan, exhibit or sell any such drawings, documents, records, software or other information or extracts therefrom or copies thereof or use them in any way except in connection with the Work in respect of which they are issued.
- 7.4 All Intellectual Property Rights belonging to or otherwise in the control of either party prior to entering into the contract shall remain the property of the party owning such Intellectual Property Rights.
- 7.5 All title and ownership of, or relating to, any intellectual property, including, but not limited to ideas, inventions, discoveries, creations, improvements or any other property subject to patent protection or intellectual property rights as developed or resulting from work under this agreement, shall directly or indirectly be solely owned by Rapra Technology Ltd, unless otherwise agreed to in writing by all participating parties.
- 7.6 In the event that Rapra Technology Ltd does not wish to apply for or maintain patent protection for any invention owned by it in accordance with clause 7 herein, it will on request assign its rights in respect of that patent to the client but in any event Rapra Technology Ltd shall be granted a royalty free, irrevocable, non-exclusive, world-wide right to use such intellectual Property Rights assigned under this condition 7.6.
- 7.7 Rapra Technology Ltd will on request grant rights to the client for exploitation or patenting of the ideas, inventions, discoveries, creations, improvements arising from the work, in the client's traditional or defined new areas of business. In all other areas, rights remain vested with Rapra Technology Ltd.

SAMPLES

8. Rapra retains the right to return or dispose of the samples at the customers cost after a period of 6 months unless otherwise agreed with the client. Storage of the samples beyond the initial 6 month period will be charged for, invoiced in advance for the agreed period (minimum additional 6 months).

CUSTOMER'S INFORMATION

- 9.1 The Client shall be solely responsible for ensuring that all drawings, information, advice and recommendations given to Rapra, either directly or indirectly by the Client or by the Client's agents, servants, consultants or advisers, are accurate and sufficient for completion of the Work. Examination or consideration by Rapra of such drawings, information, advice or recommendations shall in no way limit the Client's responsibility hereunder unless Rapra specifically agrees in writing to accept responsibility.
- 9.2 Rapra shall not disclose to any third party any knowledge or information relating to the Work which is, on receipt by Rapra, marked 'confidential' by the Client unless and until such information becomes public knowledge.

INSOLVENCY

10. If either party shall become bankrupt or under the provisions of Section 123 of the Insolvency Act 1986 is deemed to be unable to pay its debts or compounds with creditors or in the event of a resolution being passed or proceedings commenced for its administration or liquidation (other than for a voluntary winding up for the purposes of reconstruction or amalgamation) or if a Receiver or Manager is appointed of all or any part of its assets or undertaking, the other party shall be entitled to cancel the contract in whole or in part by notice in writing without prejudice to any other right or remedy accrued or accruing to that party.

FORCE MAJEURE

11. In the event of the performance of any obligation accepted by Rapra being prevented, delayed, or in any way interfered with by direction of government, war, industrial dispute, strike, breakdown of machinery or plant, accident, fire or by any other cause beyond Rapra's control Rapra may at its option suspend performance or cancel its obligations under the contract without liability for any damage or consequential loss resulting therefrom, such suspension or cancellation being without prejudice to Rapra's right to recover all sums owing to it in respect of works performed and costs incurred prior to the date of suspension or cancellation.

ASSIGNMENT

12. This Contract is personal to the parties and may not be assigned or transferred without the prior written consent of the other party.

LEGAL

13. The contract shall be governed and interpreted exclusively according to the Law of England and shall be subject to the jurisdiction of the English Courts only.