The Performance of Adhesive Joints

1993-1996

A research programme
funded by the Department of Trade and Industry
MTS Adhesives Project 5
Measurements For Optimising Adhesives Processing

Task 2
Optimisation of Key Process Parameters

Report 6
Case Study
Packaging Application 2: Full Report

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PIRA International

This report forms part of the deliverable for Task 2

Reports for Task 2

Report 2; Optimisation of Key Process Parameters - Summary Report
Report 3; Case Study: Precision Mechanical Assembly in the Business Machines Industry
Report 4; Case Study: Packaging Applications 1 & 2: Summary Report
Report 5; Case Study: Packaging Application 1: Full Report
Report 6; Case Study: Packaging Application 2: Full Report
Report 7; Case Study: Access Flooring Application
Report 8; Case Study: Construction Application - Steel Plate Bonding
Report 9; Optimisation of Key Process Parameters - Draft Code of Best Practice
Second Packaging Application - Adhesive Lamination

Collaborator - Sidlaw Packaging - Colodense

1 Introduction
During the period 1993-1996, a series of projects to look at 'The Performance of Adhesive Joints' has been running. This was a research programme funded by the Department of Trade and Industry under the Measurement, Technology and Standards budget.

The projects in the programme were led or supported by AEA Technology, the Centre for Adhesive Technology (CAT) and the National Physical Laboratory. The five project areas were:
1) Measurement of basic mechanical properties of adhesives for design use.
2) Failure modes and criteria.
3) Environmental durability of adhesive bonds.
4) Characterisation of surface condition.
5) Measurements for optimising adhesive processing.

Projects 1 and 5 were led by the National Physical Laboratory and projects 2, 3 and 4 were led by AEA Technology.

Pira International were involved in project 5 as a contractor to the National Physical Laboratory. The input was to assess the bond performance of two packaging applications during processing, and whether experimental design on line could help the adhesive performance and was feasible.

2 Objective
To assess the benefit or suitability of using experimental design based on Taguchi methodology to adhesive performance under process conditions.

3 Background
The first packaging adhesives application (cardboard tray erection) was in essence a very straightforward, low cost, low technical demand type of
application. For the second packaging application it was therefore felt that a more technically demanding system should be investigated. One such area which has widespread applications in the packaging industry is the area of film lamination. Laminated films are now used for many applications including crisp and snack packs, biscuit wrappers, cheese wrappers etc, where the various component parts of the film provide a separate function. For example, a metallised film which is laminated to a polypropylene film provides light protection, protection from gas and moisture exchange, a good background for printing and a shiny upmarket appearance.

In order to look for a suitable application a number of companies were approached by means of a mailshot.

4 Companies approached
1. Omega Developments
2. Lawson Mardon Group - Workington *
3. Cobelplast *
4. Ademso Seal
5. Bonar Flexible Packaging
6. Bonar Teich Flexibles
7. Brand Packaging
8. Broadoak Flexible Packaging
9. CMB Sunuco Composites
10. Courtaulds Films and Packaging
11. Sidlaw Packaging Colodense *
12. DRG Medical Packaging
13. Foilwraps Flexible Packaging Ltd
14. Grace WR Ltd
15. Kensulat Plc
16. Lawson Mardon Flexible, Liverpool
17. Lawson Mardon Flexible, Midsummer Norton
18. MacLean's Printed Packaging
19. Moore and Buckle (Flexible Packaging) Ltd
20. P&W Printers Ltd
21. Parkside Flexible Packaging Ltd
22. Presspahn Ltd
23. Protecta Print Ltd
Companies approached (continued)

24. Rexham Ltd
25. Rhinopac Ltd
26. Sonoco Packaging Tapes Ltd
27. Lawson Mardon Star
28. Camvac (Europe) Ltd
29. 4P Packaging (UK) Ltd
30. Nielsen, Otto Plc
31. Planned Packaging
32. Printpak Europe Ltd
33. Raackman UK Ltd
34. Stanley Smith and Co Plastics Ltd
35. Walki Pak Ltd
36. Dolphin Packaging Materials Ltd
37. Haendler and Naterman (UK) Ltd
38. South Wales Sack & Bag Co Ltd
39. Action Packaging Co Ltd/Ecoform/Sudpack
40. British Sisalkraft Ltd
41. Coles, Walter and Co Ltd
42. Danapak UK Ltd
43. Hoechst UK Ltd
44. Printpak Europe
45. Sidlaw (Venus) Flexible Packaging Ltd
46. Britannia Packaging Ltd
47. Industrial Textiles and Plastics Ltd
48. Klöckner Pentaplast Ltd
49. Röhm Ltd
50. Tripac (UK) Ltd
51. Akerlund and Rausing Ltd
52. Bartec Paper and Packaging Ltd
53. Crest Flexible Packaging Ltd
54. Impak Agencies
55. Van Der Bergh and Partners Ltd
56. Wolff Walstrode
57. CMB Technology
58. George Payne & Co Ltd
Of these 58 companies only those marked with an asterisk (*) replied either asking for further information, or volunteering to take part. Of those requesting further information, visits were made to explain the rationale and the methodology to be used to:

- Sidlaw Packaging Colodense
- Protecta Print
- CMB Technology

Presentations were made to each of the above three companies regarding the Taguchi Methodology to be used. Also an insight into each company's production was gained. After these initial consultations and presentations it was felt that Protecta Print were not involved in the required type of lamination; CMB Technology were very interested but declined to participate in the project. Sidlaw Packaging Colodense were very keen to participate in the project and they had a suitable packaging application. Hence it was decided to use Sidlaw Packaging Colodense as the second packaging application.

### 5 System to be studied

The laminated film system studied was a metallised polypropylene film laminated using solvent based adhesive to a reverse printed polypropylene film.

This type of film is widely used in snack and biscuit type packaging, hence represents a large usage.

### 6 Reasons for choice of system

- Adhesive bond strength should achieve 200 g/25 mm strip using a machine-direction peel test.

- Look for bond strength which will strip the metal from its polypropylene film.

- More often found that it is adhesive to metal surface failure.

- The low bond strengths manifest themselves at the customers, which results in material complaints and re-making of orders.
• Problem of low bond strength is intermittent, may get several reels of acceptable bond strength, then the problem will occur although no changes on machine have been made.

• Neither of the films are treated prior to lamination as it is believed that surface energies are acceptable.

7 Lamination process

The metallised polypropylene is laminated to the reverse printed polypropylene such that the print is sandwiched between the two layers.

Sidlaw Packaging Colodense have two laminating machines which are basically very similar, although L2 has a narrower deckle and can be run at higher speeds than L1.

The first unwind, at one end of the machine, will contain one of the webs, in this example it would be the metallised film. The set then passes through a corona discharge or boost unit which may be used to alter the surface energy of the film surface to which the adhesive will be applied. The web then passes through a number of tension rollers into the adhesive application system.

The adhesive is applied to the first web by gravure cylinder. The adhesive already being used in the example studied was a two-part prepolymer adhesive which is alcohol dilutable from ICI Speciality Chemicals called Novacote NC 120 ASL + 111B. It is claimed to provide excellent clarity, high green tack, high final bonds and long pan life unaffected by moisture reaction due to high humidities. The two component system is mixed at the recommended levels and diluted to a given viscosity with ethyl acetate.

The adhesive is then pumped into the tray where an excess is picked up by the gravure roll which is then motorc d using a doctor blade set at the lightest pressure possible in order to minimise blade and cylinder wear, yet remove the excess adhesive. The amount of adhesive applied is therefore controlled by adhesive viscosity, gravure roll type and temperature. The adhesive is transferred to the web by pressure contact. Generally the corona discharge
would be used such that the surface energy of the film gave an acceptable result using a 38 dyne pen.

The web then passes through the drying tunnel in order to drive off the solvents present in the adhesive. The tunnel has two sections which are set with the first zone cooler than the second zone (eg 75°C/95°C). The solvent is generally exhausted to the outside. The draws at the end of the tunnel and on the reel unwind unit control the reel/film tension.

The second web comes from a second unwind unit. The two webs are then brought together and aligned before passing into the nip. The nip where the two webs are joined is normally held at 40-45°C, but may be taken as high as 50-55°C. After the nip the joined web passes over a cool roller and to the rewind unit. The tension and pressure on the rewind unit are altered depending on type of product, so that air is expelled from the reel and a uniform tension holds the reel steady (ie no telescoping or slippage of reel). The cool roller just before the rewind helps to prevent stretching of the film.

Films are supplied by various manufacturers. The bond strength can be affected by viscosity effects of the adhesive, cylinder wear, cylinder fill-in and humidity effects on adhesive flow. Bond strength can also be affected by the surface type and treatment and nip contamination. Tension control is critical as if the webs are not parallel then feathering can occur. Problems can also occur if the printed surface has high levels of retained solvents as these may affect the clarity of the adhesive and ink bonding strength relative to adhesive bonding strength.
8 Testing process

The testing of the bond is carried out by machine-direction peel tests with no cross-direction profile being observed. The samples are taken after lamination but tested by the following shift, hence the time before testing can be variable.

Generally full bond strength occurs only after 24 hours and hence often the strips are incubated for 1 hour at 60°C, this incubation time can also be variable. The strips are cut 25mm wide and 15cm long and in order to start dissolving the adhesive the end 2cm are placed in THF (tetrahydrofuran) so that the peel test can be performed. This immersion in THF has no fixed time, and although only the first 2cm are immersed in THF, the use of test tubes means that a longer length of the strip may be exposed to effects by solvent fumes from the THF.

Once the peel has been initiated, the samples are separated on a tensile tester (Lloyd) at 200 mm/min speed, with fixed load cells. The data is logged by computer and the mean and peak forces calculated. Normally only mean results are taken for control purposes. A trace of the peel can be obtained. It is noted where the peel occurs, ie adhesive layer, metal from film, print from film or whether film tear occurs in which case the bond strength would be greater than the film strength. For accurate results the laminated strips must be loaded straight into the tensile tester.

Other tests which may be performed include solvent retention by gas chromatogram, seal strength using an impulse heat sealer and appearance against colour standard.
For the product selected retained solvents the levels are less than 10 mg/m² for esters and less than 20 mg/m² for alcohols. Seal strength tests should not show delamination and the appearance must be clean and glossy.

If there is a problem with test results, they are repeated after 24 hours storage.

9 Initial brainstorming session
This included all types of personnel concerned with the lamination process together with outside observers or facilitators.

Firstly it was decided that nothing should be ignored during this session, hence the discussions were based on the use of the following 'fishbone' diagram.

And the process diagram was simplified to

```
<table>
<thead>
<tr>
<th>Problem</th>
</tr>
</thead>
<tbody>
<tr>
<td>People</td>
</tr>
<tr>
<td>Machines</td>
</tr>
<tr>
<td>Materials</td>
</tr>
<tr>
<td>Environment</td>
</tr>
<tr>
<td>Methods</td>
</tr>
</tbody>
</table>
```

Unwind Unit 1

Adhesive Applicator

Drying

Unwind Unit 2

Nip

Rewind

From this the following were raised at parameters which could affect final bond strength.
Unwind unit 1
- Nature of metallised surface - surface properties
- Film grade/supplier

Adhesive application unit
- Outside storage
- Two component ratio - mixing
- Type of adhesive
- Amount of adhesive
- Consistency of mixing/accuracy
- Viscosity (related to amount)
- Cylinder pattern/condition
- Backing roll pattern

Drying
- Temperature
- Web speed
- Solvent type
- Solvent extraction
- Retained solvent

Unwind Unit 2
- Retained solvent in the ink
- Ink type/weight
- Temperature of preheat roller/web speed

Nip
- Pressure
- Roller surface condition (rubber)
- Temperature
- Speed
- Chill roll temperature
Rewind
- Storage
- Temperature of factory
- Humidity

Testing
- Sample preparation time
- Temperature

NB Variations in test procedures were not included in the experiment.

10 **Initial selection of most important parameters**
This was carried out by a democratic voting process involving the eight people most accustomed to the lamination system or experimental system.

The selected parameters were as follows:
- mixing ratios/accuracy of adhesive mixing
- type of adhesive/solvent type
- amount of adhesive
- drying temperature and profile/retained solvent
- metallised surface properties
- retained solvent in ink
- Nip temperature
- Storage of roll
- Web speed

It should be noted that some factors were combined as it was believed they were related and it would be unable to separate them.

Other factors were eliminated so that the experiment contained a manageable number of parameters for study.
11 Initial setting of factor levels

In order to assess whether a parameter has an effect or not, it is necessary to set each one at more than one level, e.g. high and low.

<table>
<thead>
<tr>
<th>Metal surface properties</th>
<th>Level 1</th>
<th>Level 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Adhesive mixing ratio</td>
<td>10:1</td>
<td>10:2</td>
</tr>
<tr>
<td>Type of adhesive **</td>
<td>Current</td>
<td>New</td>
</tr>
<tr>
<td>(details of adhesives are appended)</td>
<td>Novacote 120 ASL/111B</td>
<td>Holdens 10-2525/3/1025-26/3</td>
</tr>
<tr>
<td>Nip temperature</td>
<td>40</td>
<td>50</td>
</tr>
<tr>
<td>* Retained solvent in ink</td>
<td>Normal</td>
<td>High</td>
</tr>
<tr>
<td></td>
<td>&lt;15 mg/m²</td>
<td>&gt;30 mg/m²</td>
</tr>
<tr>
<td>Dry temperature/profile</td>
<td>60/90</td>
<td>70/110</td>
</tr>
<tr>
<td>Amount of adhesive</td>
<td>New 130</td>
<td>Old 130+</td>
</tr>
<tr>
<td>Temperature of finished roll/chill roll</td>
<td>With cold water</td>
<td>No cold water</td>
</tr>
<tr>
<td>Web speed m/min</td>
<td>130</td>
<td>180</td>
</tr>
</tbody>
</table>

* This parameter was eliminated as it was not possible to attain the higher level.

+ Actually used a 140 cylinder

** Adhesive data sheets are in Appendix 1

In a perfect world the parameters would be truly randomised to ensure that the experiment was completely unbiased. However due to the nature of the laminating process it was clear that some changes would be difficult or too time consuming. Hence Sidlaw were asked to decide which parameters would be difficult to change.

These were identified as adhesive type and adhesive amount. Hence the experimental plan was designed to take account of this and the minimum number of changes for each of these parameters was incorporated.
12 **Additional work on metallised surface properties (Appendix 2)**

The additional work was based on a comparison of metallised oriented polypropylene compared to metallised polyester. This was because bond strength problems have been encountered with metallised oriented polypropylene but not with metallised polyester.

12.1 **Surface energy**

The surface energy was looked at by use of contact angles and the following results were found.

<table>
<thead>
<tr>
<th>Surface Treatment</th>
<th>Mean</th>
<th>Range</th>
</tr>
</thead>
<tbody>
<tr>
<td>Metallised OPP</td>
<td>82</td>
<td>77-85</td>
</tr>
<tr>
<td>Metallised polyester</td>
<td>35</td>
<td>34-37</td>
</tr>
</tbody>
</table>

Hence for the original films the metallised polyester was much more wettable than the metallised OPP.

It was then decided to carry out various treatments to the metallised surface of the OPP and then recheck the contact angle.

<table>
<thead>
<tr>
<th>Surface Treatment</th>
<th>Mean</th>
<th>Range</th>
</tr>
</thead>
<tbody>
<tr>
<td>Metallised OPP + corona treatment</td>
<td>78</td>
<td>71-83</td>
</tr>
<tr>
<td>+ methylated spirit wipe</td>
<td>72</td>
<td>67-77</td>
</tr>
<tr>
<td>+ acetone wipe</td>
<td>83</td>
<td>75-85</td>
</tr>
<tr>
<td>+ dilute acid wipe</td>
<td>75</td>
<td>71-80</td>
</tr>
<tr>
<td>+ corona + dilute acid wipe</td>
<td>59</td>
<td>54-64</td>
</tr>
</tbody>
</table>

Hence it was possible to improve the wettability of the metallised surface by use of corona treatment and a dilute acid wipe. However this would not be practicable in the production process.

In addition, alternative supplies of metallised films were assessed.

<table>
<thead>
<tr>
<th>Surface Treatment</th>
<th>Mean</th>
<th>Range</th>
</tr>
</thead>
<tbody>
<tr>
<td>Metallised polyester</td>
<td>(1)</td>
<td>82</td>
</tr>
<tr>
<td></td>
<td>(2)</td>
<td>43</td>
</tr>
<tr>
<td>Metallised OPP</td>
<td>(1)</td>
<td>89</td>
</tr>
<tr>
<td></td>
<td>(2)</td>
<td>81</td>
</tr>
</tbody>
</table>
Hence for all suppliers used the metallised OPP was less wettable than the metallised polyester with the exception of one sample of metallised polyester which was found to be old stock.

The metallised surface of the films supplied by Sidlaw Packaging were also evaluated using special surface tension testing inks which give measurements in dynes/cm.

Metallised OPP had a surface tension of below 34 dynes/cm.
Metallised polyester had a surface tension of 48 dynes/cm.

The metallised OPP is significantly less wettable than the metallised polyester.

12.2 Infra-red analysis

Infra-red analysis was performed by use of Attenuated Total Reflectance (ATR) on both surfaces of the metallised oriented polypropylene and metallised polyester. (See IR spectra attached).

Spectrum (1) showed the ATR of the polypropylene side of the film, the trace being typical of oriented polypropylene, with no evidence of any copolymers or additives. An ATR of the metallised surface (2) showed a very basic trace with no real peaks visible as expected for an aluminium surface.

For the metallised polyester, an ATR spectrum of the polyester surface (3), showed the film to be a typical polyester. As with the metallised polypropylene, the metallised surface (4), showed a flat trace with no significant peaks (4).

The metallised surfaces were then washed off with acetone and an infra-red spectrum taken of the wash off. For both the metallised polypropylene and metallised polyester there was nothing on the metallised surface which was removed by an acetone wash. Hence it is unlikely that a lacquer had been applied to the metallised surface.
12.3 **Surface examination by scanning electron microscope**

The metallised surface of both the polypropylene and polyester films was looked at under high magnification using an electron microscope. It was found that the metallised polypropylene showed a smooth metal surface with no cracking present, although small "lumps" were observed (1-2 μm diameter) which were integral to the coating. A cut edge examination showed the presence of the polypropylene film and a thickish layer of aluminium.

Examination of the metallised polyester showed the surface to be smooth with the presence of hairline cracks. Examination of the edge showed the polyester film and a thinner layer of aluminium.

Use of elemental surface analysis showed that the metallised polypropylene showed a large carbon peak, with smaller aluminium and oxygen peaks. The metallised polyester showed larger aluminium and oxygen peaks and a small carbon peak.

Discussion with metallisers revealed that the same grade of aluminium is used for both polypropylene and polyester and is normally carried out in the same chambers. Films are not supplied with protective coatings, so the carbon content must originate from the film itself. The lumps seen in the metallised polypropylene may be anti-block. The presence of aluminium is as expected and oxygen may be present due to oxidation of the surface which occurs over time.

The aluminium used for metallisation is normally 99.5% pure, with the largest impurity (iron) being left as a residue after metallisation. The aluminium normally has a particle size of 200-300Å with a control of ± 5% on the mean size, although the variation can be as much as ± 10-15%.

It is believed that oriented polypropylene is more difficult to metallise because it is rougher than polyester and because species migration from the oriented polypropylene occurs under the vacuum/temperature conditions in the metallisation chamber. Migration of these species is believed to cause the lower wetting capability. However these species are bound to the body of the material and hence are not free to be washed off by solvent.
The only practical way of improving the wettability of the metallised surface during the production process would be to corona discharge (boost) treat the surface immediately prior to adhesive application.

13 Final selection of parameters and levels for experiment

The following parameters and levels were chosen:

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Level 1</th>
<th>Level 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Metal surface properties</td>
<td>Untreated</td>
<td>Boost treated</td>
</tr>
<tr>
<td>2. Adhesive mixing ratio</td>
<td>10:1</td>
<td>10:2</td>
</tr>
<tr>
<td>3. Adhesive type</td>
<td>Current</td>
<td>New</td>
</tr>
<tr>
<td></td>
<td>Novacote 120 ASL/111B</td>
<td>Holdens 10-2525/3/10-2526/3</td>
</tr>
<tr>
<td>4. Nip temperature °C</td>
<td>40</td>
<td>50</td>
</tr>
<tr>
<td>5. Drying temp/profile °C</td>
<td>60/90</td>
<td>70/110</td>
</tr>
<tr>
<td>6. Amount of adhesive lines/inch</td>
<td>100</td>
<td>140</td>
</tr>
<tr>
<td>7. Temperature of finished roll/chill roll</td>
<td>With cold water</td>
<td>No cold water</td>
</tr>
<tr>
<td>8. Web speed m/min</td>
<td>130</td>
<td>180</td>
</tr>
</tbody>
</table>

The retained ink solvent was dropped due to the high level being unattainable. The old 130 cylinder was replaced by use of a 140 cylinder.

It was decided that the adhesive type should not be randomised and that the amount of adhesive was kept to the minimum number of changes to aid runnability.

These parameters were then organised into an experimental plan by Xyratex.
### Experimental plan

<table>
<thead>
<tr>
<th>Run</th>
<th>Adhesive type</th>
<th>Adhesive Amount</th>
<th>Metal Surface Properties</th>
<th>Adhesive Mix Ratio</th>
<th>Nip Temp °C</th>
<th>Drying Temp and Profile °C</th>
<th>Chill Roll</th>
<th>Web Speed m/m</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Holdens</td>
<td>130</td>
<td>Boost</td>
<td>10:1</td>
<td>40</td>
<td>70/110</td>
<td>Cold water</td>
<td>180</td>
</tr>
<tr>
<td>2</td>
<td>Holdens</td>
<td>130</td>
<td>Untreated</td>
<td>10:2</td>
<td>50</td>
<td>60/90</td>
<td>Cold water</td>
<td>180</td>
</tr>
<tr>
<td>3</td>
<td>Holdens</td>
<td>130</td>
<td>Untreated</td>
<td>10:1</td>
<td>50</td>
<td>70/110</td>
<td>No cold water</td>
<td>130</td>
</tr>
<tr>
<td>4</td>
<td>Holdens</td>
<td>130</td>
<td>Boost</td>
<td>10:2</td>
<td>40</td>
<td>60/90</td>
<td>No cold water</td>
<td>130</td>
</tr>
<tr>
<td>5</td>
<td>Holdens</td>
<td>140</td>
<td>Untreated</td>
<td>10:1</td>
<td>40</td>
<td>60/90</td>
<td>No cold water</td>
<td>180</td>
</tr>
<tr>
<td>6</td>
<td>Holdens</td>
<td>140</td>
<td>Boost</td>
<td>10:2</td>
<td>50</td>
<td>70/110</td>
<td>No cold water</td>
<td>180</td>
</tr>
<tr>
<td>7</td>
<td>Holdens</td>
<td>140</td>
<td>Boost</td>
<td>10:1</td>
<td>50</td>
<td>60/90</td>
<td>Cold water</td>
<td>130</td>
</tr>
<tr>
<td>8</td>
<td>Holdens</td>
<td>140</td>
<td>Untreated</td>
<td>10:2</td>
<td>40</td>
<td>70/110</td>
<td>Cold water</td>
<td>130</td>
</tr>
<tr>
<td>9</td>
<td>Novacote</td>
<td>130</td>
<td>Boost</td>
<td>10:1</td>
<td>50</td>
<td>60/90</td>
<td>No cold water</td>
<td>180</td>
</tr>
<tr>
<td>10</td>
<td>Novacote</td>
<td>130</td>
<td>Boost</td>
<td>10:2</td>
<td>50</td>
<td>70/110</td>
<td>Cold water</td>
<td>130</td>
</tr>
<tr>
<td>11</td>
<td>Novacote</td>
<td>130</td>
<td>Untreated</td>
<td>10:1</td>
<td>40</td>
<td>60/90</td>
<td>Cold water</td>
<td>130</td>
</tr>
<tr>
<td>12</td>
<td>Novacote</td>
<td>130</td>
<td>Untreated</td>
<td>10:2</td>
<td>40</td>
<td>70/110</td>
<td>No cold water</td>
<td>180</td>
</tr>
<tr>
<td>13</td>
<td>Novacote</td>
<td>140</td>
<td>Untreated</td>
<td>10:2</td>
<td>50</td>
<td>60/90</td>
<td>No cold water</td>
<td>130</td>
</tr>
<tr>
<td>14</td>
<td>Novacote</td>
<td>140</td>
<td>Boost</td>
<td>10:1</td>
<td>40</td>
<td>70/110</td>
<td>No cold water</td>
<td>130</td>
</tr>
<tr>
<td>15</td>
<td>Novacote</td>
<td>140</td>
<td>Untreated</td>
<td>10:1</td>
<td>50</td>
<td>70/110</td>
<td>Cold water</td>
<td>180</td>
</tr>
<tr>
<td>16</td>
<td>Novacote</td>
<td>140</td>
<td>Boost</td>
<td>10:2</td>
<td>40</td>
<td>60/90</td>
<td>Cold water</td>
<td>180</td>
</tr>
</tbody>
</table>
Notes

- The headings are coded from the parameters for experiment.

- The experiment must be run in the order indicated by run column.

- If any changes to parameters or level descriptions, the above can be modified.

- It can be useful when running the experiment, to have each run description of the parameter settings on a separate sheet of paper. This can make it easier/clearer for operational personnel to ensure that the particular settings are followed.

- All data collected from the experiment, must be recorded so that it can be referenced back to the run number and its parameter settings. The adoption of the scheme above, means that this sheet could also be used for data collection.

- During the running of the experiment, it is important to note things which change or are unusual happenings (not covered specifically in the parameter changes).

- Sample sizes will be evaluated using background current information.

15 Setting of sample sizes (Appendix 3)

The background bond strength data showed that for the metallised oriented polypropylene/reverse printed polypropylene laminate the bond strengths gave a mean of 261 g/25 mm with a range from 112 g/25 mm to 366 g/25 mm. The target is 200 g/25 mm or above.

The preferred bond strength would be a mean of 350 g/25 mm with a minimum of 275 g/25 mm.
In addition to the bond strengths, there is a requirement for the level of retained solvents. The acceptable levels are below 10 mg/m² for esters and below 20 mg/m² for alcohols.

There is also a heat seal requirements for this type of product whereby it would be preferable to obtain film tear heat seal bonds rather than delamination.

From all this data, the sample size would need to be ten samples per trial. The experiment should be run so that for each trial is represented by a period of time or quantity of parts. The ten samples should be taken as two samples at five positions through the trial, with the time/quantity sequence of the sample being retained.

16 Experiment running

It was realised that the Novacote and Holdens' adhesives had different mixing ratios. The Novacote had a mixing ratio of 10:1 and 10:2, whilst the Holdens' had a mixing ratio of 60:1 and 60:2. Technically if these ratios were applied to the experiment matrix the number of trials should increase to 32. It was found that not enough reverse printed polypropylene film had been produced to cover 32 trials. Hence the adhesive mix ratio was treated as a low and high variant thus curtailing the experiment to the original 16 trials.

It was also requested that the running order was changed slightly to improve ease of production runnability. Hence the final experimental plan was as below.
<table>
<thead>
<tr>
<th>Run No</th>
<th>Adhesive Type</th>
<th>Adhesive Amount</th>
<th>Metallised Surface Properties</th>
<th>Adhesive Mix Ratio</th>
<th>Nip Temp °C</th>
<th>Drying Temp Profile °C</th>
<th>Temp of Reel/Chill Roll</th>
<th>Web Speed</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Holdens</td>
<td>130</td>
<td>Boost</td>
<td>60:1 (L)</td>
<td>40</td>
<td>70/110</td>
<td>Cold water</td>
<td>180</td>
</tr>
<tr>
<td>3</td>
<td>Holdens</td>
<td>130</td>
<td>Untreated</td>
<td>60:1 (L)</td>
<td>50</td>
<td>70/110</td>
<td>No cold water</td>
<td>130</td>
</tr>
<tr>
<td>2</td>
<td>Holdens</td>
<td>130</td>
<td>Untreated</td>
<td>60:2 (H)</td>
<td>50</td>
<td>60/90</td>
<td>Cold water</td>
<td>180</td>
</tr>
<tr>
<td>4</td>
<td>Holdens</td>
<td>130</td>
<td>Boost</td>
<td>60:2 (H)</td>
<td>40</td>
<td>60/90</td>
<td>No cold water</td>
<td>130</td>
</tr>
<tr>
<td>8</td>
<td>Holdens</td>
<td>140</td>
<td>Untreated</td>
<td>60:2 (H)</td>
<td>40</td>
<td>70/110</td>
<td>Cold water</td>
<td>130</td>
</tr>
<tr>
<td>6</td>
<td>Holdens</td>
<td>140</td>
<td>Boost</td>
<td>60:2 (H)</td>
<td>50</td>
<td>70/110</td>
<td>No cold water</td>
<td>180</td>
</tr>
<tr>
<td>5</td>
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<td>140</td>
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<td>60:1 (L)</td>
<td>40</td>
<td>60/90</td>
<td>No cold water</td>
<td>180</td>
</tr>
<tr>
<td>7</td>
<td>Holdens</td>
<td>140</td>
<td>Boost</td>
<td>60:1 (L)</td>
<td>50</td>
<td>60/90</td>
<td>Cold water</td>
<td>130</td>
</tr>
<tr>
<td>16</td>
<td>Novacote</td>
<td>140</td>
<td>Boost</td>
<td>10:2 (H)</td>
<td>40</td>
<td>60/90</td>
<td>Cold water</td>
<td>180</td>
</tr>
<tr>
<td>13</td>
<td>Novacote</td>
<td>140</td>
<td>Untreated</td>
<td>10:2 (H)</td>
<td>50</td>
<td>60/90</td>
<td>No cold water</td>
<td>130</td>
</tr>
<tr>
<td>14</td>
<td>Novacote</td>
<td>140</td>
<td>Boost</td>
<td>10:1 (L)</td>
<td>40</td>
<td>70/110</td>
<td>No cold water</td>
<td>130</td>
</tr>
<tr>
<td>15</td>
<td>Novacote</td>
<td>140</td>
<td>Untreated</td>
<td>10:1 (L)</td>
<td>50</td>
<td>70/110</td>
<td>Cold water</td>
<td>180</td>
</tr>
<tr>
<td>9</td>
<td>Novacote</td>
<td>130</td>
<td>Boost</td>
<td>10:1 (L)</td>
<td>50</td>
<td>60/90</td>
<td>No cold water</td>
<td>180</td>
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<tr>
<td>11</td>
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<td>130</td>
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<td>Cold water</td>
<td>130</td>
</tr>
<tr>
<td>12</td>
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<td>70/110</td>
<td>No cold water</td>
<td>180</td>
</tr>
<tr>
<td>10</td>
<td>Novacote</td>
<td>130</td>
<td>Boost</td>
<td>10:2 (H)</td>
<td>60</td>
<td>70/110</td>
<td>Cold water</td>
<td>130</td>
</tr>
</tbody>
</table>
The experiment was therefore run according to this plan, in the order presented, run numbers were used only for the analysis.

The following points were noted during the trial.

- Boost was set to 40 amps
- Reels were rewound and tested after 24 hours
- Samples taken at start, middle and end of each run
- 400 metres of each trial were run
- Adhesives were diluted to same viscosity with ethyl acetate
- All cleaning of cylinders etc was done with ethyl acetate
- Film tension for all trials was 10 kg
- Nothing of any significance was noted during the trial (ie according to operators the films/lamination ran as usual).
# Results of trial

## a) Bond strengths (g/25 mm)

<table>
<thead>
<tr>
<th>Running Test</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
<th>7</th>
<th>8</th>
<th>9</th>
<th>10</th>
<th>11</th>
<th>12</th>
<th>13</th>
<th>14</th>
<th>15</th>
<th>16</th>
<th>Mean</th>
<th>Sd</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>283+</td>
<td>405+</td>
<td>440+</td>
<td>392+</td>
<td>312+</td>
<td>405+</td>
<td>376+</td>
<td>312+</td>
<td>197</td>
<td>225</td>
<td>221</td>
<td>150</td>
<td>219</td>
<td>218</td>
<td>186</td>
<td>229</td>
<td></td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>266+</td>
<td>399+</td>
<td>191+</td>
<td>521+</td>
<td>343+</td>
<td>353+</td>
<td>307+</td>
<td>278+</td>
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<td>164</td>
<td>239</td>
<td>176</td>
<td>266</td>
<td>287</td>
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<td></td>
</tr>
<tr>
<td>3</td>
<td>468+</td>
<td>385+</td>
<td>474+</td>
<td>422+</td>
<td>243+</td>
<td>468+</td>
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<td>370+</td>
<td>185</td>
<td>231+</td>
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<td>205</td>
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<td>224</td>
<td></td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>408+</td>
<td>376+</td>
<td>370+</td>
<td>338+</td>
<td>382+</td>
<td>428+</td>
<td>318+</td>
<td>422+</td>
<td>161</td>
<td>221</td>
<td>161</td>
<td>188</td>
<td>199</td>
<td>234</td>
<td>256</td>
<td>255</td>
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<tr>
<td>5</td>
<td>364+</td>
<td>486+</td>
<td>457+</td>
<td>226+</td>
<td>353+</td>
<td>486+</td>
<td>423+</td>
<td>497+</td>
<td>240</td>
<td>101+</td>
<td>238</td>
<td>173+</td>
<td>242</td>
<td>205</td>
<td>266+</td>
<td>267</td>
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<td></td>
</tr>
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<td>6</td>
<td>283+</td>
<td>388+</td>
<td>205+</td>
<td>571+</td>
<td>300+</td>
<td>345+</td>
<td>324+</td>
<td>206+</td>
<td>171</td>
<td>168</td>
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<td></td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>301+</td>
<td>187+</td>
<td>428+</td>
<td>445+</td>
<td>364+</td>
<td>347+</td>
<td>393+</td>
<td>145</td>
<td>215</td>
<td>196</td>
<td>90+</td>
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<td>434+</td>
<td>405+</td>
<td>364+</td>
<td>278+</td>
<td>154</td>
<td>202</td>
<td>159</td>
<td>237</td>
<td>218</td>
<td>244</td>
<td>272+</td>
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</tr>
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<td>9</td>
<td>474+</td>
<td>468+</td>
<td>405+</td>
<td>422+</td>
<td>416+</td>
<td>283+</td>
<td>598+</td>
<td>237+</td>
<td>326</td>
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<td>174</td>
<td>151+</td>
<td>215</td>
<td>144+</td>
<td></td>
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<td></td>
</tr>
<tr>
<td>10</td>
<td>416+</td>
<td>480+</td>
<td>630+</td>
<td>388+</td>
<td>561+</td>
<td>364+</td>
<td>563+</td>
<td>463+</td>
<td>233</td>
<td>243</td>
<td>228</td>
<td>125+</td>
<td>199</td>
<td>189</td>
<td>228</td>
<td>208</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Mean</td>
<td>364</td>
<td>396</td>
<td>399</td>
<td>411</td>
<td>374</td>
<td>388</td>
<td>373</td>
<td>346</td>
<td>201</td>
<td>196</td>
<td>203</td>
<td>173</td>
<td>201</td>
<td>189</td>
<td>234</td>
<td>229</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sd</td>
<td>77</td>
<td>85</td>
<td>120</td>
<td>94</td>
<td>85</td>
<td>62</td>
<td>101</td>
<td>96</td>
<td>54</td>
<td>43</td>
<td>30</td>
<td>45</td>
<td>41</td>
<td>35</td>
<td>33</td>
<td>46</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

+ denotes film tear, i.e., material tested to destruction.
b) Retained solvents

<table>
<thead>
<tr>
<th>Run No.</th>
<th>Ethyl Acetate mg/m²</th>
<th>Ethanol mg/m²</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>13.9</td>
<td>0.7</td>
</tr>
<tr>
<td>2</td>
<td>37.1</td>
<td>0.8</td>
</tr>
<tr>
<td>3</td>
<td>4.0</td>
<td>0.5</td>
</tr>
<tr>
<td>4</td>
<td>18.7</td>
<td>0.8</td>
</tr>
<tr>
<td>5</td>
<td>20.1</td>
<td>0.8</td>
</tr>
<tr>
<td>6</td>
<td>0.8</td>
<td>0.6</td>
</tr>
<tr>
<td>7</td>
<td>7.7</td>
<td>0.7</td>
</tr>
<tr>
<td>8</td>
<td>0.7</td>
<td>0.6</td>
</tr>
<tr>
<td>9</td>
<td>0.8</td>
<td>11.5</td>
</tr>
<tr>
<td>10</td>
<td>0.6</td>
<td>5.8</td>
</tr>
<tr>
<td>11</td>
<td>0.6</td>
<td>6.4</td>
</tr>
<tr>
<td>12</td>
<td>0.6</td>
<td>6.9</td>
</tr>
<tr>
<td>13</td>
<td>0.7</td>
<td>5.0</td>
</tr>
<tr>
<td>14</td>
<td>0.7</td>
<td>6.6</td>
</tr>
<tr>
<td>15</td>
<td>0.7</td>
<td>5.4</td>
</tr>
<tr>
<td>16</td>
<td>0.7</td>
<td>5.0</td>
</tr>
</tbody>
</table>

Hence runs 1, 2, 4 and 5 are out of specification for retained solvents (limit is 10 mg/m² for esters).

All runs were acceptable for retained ethanol (limit 20 mg/m²)

c) Heat seal

Trials 1-8 all gave film tear heat seal bonds, well inside the specifications.

Trials 9-16 all delaminated on the heat seal test, however, the bonds achieved would be adequate for the end use of the product.
14 Standard production conditions
Normally the following production conditions were used with this product:
- Adhesive type: Novacote NC 120 ASL + 111B
- Adhesive amount: 130 cylinder
- Metal surface properties: untreated
- Adhesive mix: 10:1
- Nip temperature: 45°C
- Drying profile: 70/90°C
- Chill roll temperature: cold
- Web speed: 150-160 m/min

19 Analysis of results (Appendix 4)
The parameters used during the trial were as below, with the two levels for each given, the parameters have been coded for the analysis as (A-H).

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Level 1</th>
<th>Level 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>A  Adhesive type</td>
<td>Novacote</td>
<td>Holdens</td>
</tr>
<tr>
<td>B  Adhesive amount</td>
<td>New 130</td>
<td>140</td>
</tr>
<tr>
<td>C  Metal surface properties</td>
<td>Untreated</td>
<td>Boost</td>
</tr>
<tr>
<td>D  Adhesive mix Ratio</td>
<td>60:1</td>
<td>60:2 Holdens</td>
</tr>
<tr>
<td></td>
<td>10:1</td>
<td>10:2 Novacote</td>
</tr>
<tr>
<td>E  Nip temperature</td>
<td>40</td>
<td>50</td>
</tr>
<tr>
<td>F  Drying profile</td>
<td>60/90</td>
<td>70/110</td>
</tr>
<tr>
<td>G  Chill roll temperature</td>
<td>Cold</td>
<td>No cold</td>
</tr>
<tr>
<td>H  Web speed</td>
<td>130</td>
<td>180</td>
</tr>
</tbody>
</table>

19.1 Use of CpK
The CpK (Current Process Capability) is the measure of the goodness of a process, it is an index of quality given by

\[
CpK = \frac{\text{mean} - \text{specification}}{3 \times \text{standard deviation}}
\]
There is a relationship between the CpK and the likelihood of producing defects:

- \( \text{CpK} = 0 \)  \( \text{defect level} = 50\% \)
- \( \text{CpK} = 1 \)  \( \text{defect level} = 0.27\% \) (2700 ppm)
- \( \text{CpK} = 1.5 \)  \( \text{defect level} = 3 \text{ ppm} \)

Hence as the CpK increases the defect level decreases. The CpK can be negative, which indicates a defect level of above 50%.

If the CpK is good, the process is inherently good and it is possible to reduce the amount of product testing. If the CpK is lower then a greater level of testing is required to remove bad results.

If a specification has both upper and lower limits than there are two CpK values:

1. \( \text{CpK} = \left[ \frac{\text{upper limit} - \text{mean}}{3 \times \text{standard deviation}} \right] \)

2. \( \text{CpK} = \left[ \frac{\text{mean} - \text{lower limit}}{3 \times \text{standard deviation}} \right] \)

Note: CpK is only applicable to measured processes (that is it cannot be used for subjective tests such as good/bad, pass/fail).

For this experiment the CpK of each run varied between -0.2 and 1.01, so there was a large variation in the goodness of the process.
19.2 Summary of results - whole experiment

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Mean</th>
<th>Standard deviation</th>
<th>Best setting</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>Significant - Holdens</td>
<td>Significant - Novacote</td>
<td>Holdens</td>
</tr>
<tr>
<td>B</td>
<td>Significant - New 130</td>
<td>Not significant</td>
<td>New 130</td>
</tr>
<tr>
<td>C</td>
<td>No significant</td>
<td>Not significant</td>
<td>Cost/productivity</td>
</tr>
<tr>
<td>D</td>
<td>See later analysis</td>
<td>→</td>
<td>→</td>
</tr>
<tr>
<td>E</td>
<td>Not significant</td>
<td>Significant (40)</td>
<td>40</td>
</tr>
<tr>
<td>F</td>
<td>Not significant</td>
<td>Significant (70/110)</td>
<td>70/110</td>
</tr>
<tr>
<td>G</td>
<td>Not significant</td>
<td>Not significant</td>
<td>Cost/productivity</td>
</tr>
<tr>
<td>H</td>
<td>Not significant</td>
<td>Not significant</td>
<td>Cost/productivity</td>
</tr>
</tbody>
</table>

From this it can be seen that Holdens and adhesive amount have a significant effect on the mean bond strength. However Novacote, Nip temperature and drying profile have a significant effect on the standard deviation.

19.3 Subset specific to Holdens

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Mean</th>
<th>Standard deviation</th>
<th>Best setting</th>
</tr>
</thead>
<tbody>
<tr>
<td>B</td>
<td>Not Sig? (New 130)</td>
<td>Sig? (140)</td>
<td>Trade off</td>
</tr>
<tr>
<td>C</td>
<td>Not sig</td>
<td>Sig? (boost)</td>
<td>Boost? Cost?</td>
</tr>
<tr>
<td>D</td>
<td>Not sig 60:2</td>
<td>Not sig</td>
<td>Cost/productivity 60:2?</td>
</tr>
<tr>
<td>E</td>
<td>Not sig</td>
<td>Not sig</td>
<td>Cost/productivity</td>
</tr>
<tr>
<td>F</td>
<td>Not sig</td>
<td>Sig 70/110</td>
<td>70/110</td>
</tr>
<tr>
<td>G</td>
<td>Not sig? no cold</td>
<td>Not sig</td>
<td>Cost/productivity? no cold</td>
</tr>
<tr>
<td>H</td>
<td>Not sig</td>
<td>Not sig</td>
<td>Cost/productivity</td>
</tr>
</tbody>
</table>

All results are taken on the length of the line between the two levels, in this case those marked ? have a tendency towards better variability.
19.4 **Steps used in the analysis (see Appendices)**
Firstly, an exploratory data analysis was performed whereby the data is put into "picture form".

19.5 **Diagram 1**
This is termed a box whisker plot where the following is represented

\[
\begin{align*}
X & \leftarrow \text{largest result} \\
\text{box} & = \text{middle 50\% of the 10 results} \\
\text{line} & = \text{middle result of the 50\%} \\
\bullet & = \text{mean of whole set of results} \\
X & \leftarrow \text{lowest result}
\end{align*}
\]

It is obvious from this plot that Holdens has a greater mean and variability than Novacole.

Hence the effect of the variability has to be taken out.

19.6 **Diagram 2**
To remove variability need to look at the log values of the mean and standard deviation, ie the data has undergone a transformation.

Then plot log standard deviation against log mean and look to see if the pattern is regular or random. In this case there is a relationship given by

\[
Y = -1.0926 + 1.1745 \times X
\]

constant = approx. 1, hence data has to be logged for transformation
19.7 Diagram 3
Re-examine the box plot using the log data. This shows that variability is reduced but the mean stays the same. All data is now in transformed form.

19.8 Diagram 4
This is called a 'Response Graph'. For the mean data, there is a central line and two boundary lines, one either side of the central line.

If the two results for a parameter are within the boundaries, then the result is not significant. If the two results are outside the boundaries the result is significant. If the results are on the boundaries then the result is tending towards significance.

Hence the conditions are that A is very significant, with B and D being somewhat significant.

19.9 Analysis of variance
What are the factors that have the biggest influence on explaining variability.

The experiments account for 67% of all variation, with parameter A explaining 63% of the variability.

Hence A = 0%  
B = 3%  ) percentage chance of being not significant 
C = 2%  )
19.10 Diagram 6
  This is the Response Graph for the standard deviation. Here there are no boundaries as there is only one result per sample set. Hence the length of line shows the level of significance.

  - E and F are most significant
  - A and G are next most significant
  - Remaining parameters are not significant

19.11 For Holdens only analysis
  For the mean the response graph shows that none of the parameters are significant. There is a tendency for a higher mean with New 130, 60:2 and No Cold.

  For the standard deviation there are no really significant factors, the longer lines are shown for 70/110, 140, Boost which all give a tendency towards better (lower) variability.

  The main question is would these effects move to a situation with the mean and variability such that the CpK has moved to give a low level of defects?

  As the original CpK valves were not above 1.0, there is something else that is affecting variability, even when set to the process conditions. This could be the testing procedure on process parameters which have not been studied.
19.12 Other analysis
The main analysis has used only bond strength data, although it was noted that some runs gave high retained solvents. These were runs 1, 2, 4 and 5.

<table>
<thead>
<tr>
<th></th>
<th>Run 1</th>
<th>Run 2</th>
<th>Run 4</th>
<th>Run 5</th>
<th>Summary</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>Holdens</td>
<td>Holdens</td>
<td>Holdens</td>
<td>Holdens</td>
<td>4 x Holdens</td>
</tr>
<tr>
<td>B</td>
<td>New 130</td>
<td>New 130</td>
<td>New 130</td>
<td>140</td>
<td>3 x New 130</td>
</tr>
<tr>
<td>C</td>
<td>Boost</td>
<td>Untreated</td>
<td>Boost</td>
<td>Untreated</td>
<td>-</td>
</tr>
<tr>
<td>D</td>
<td>60:1</td>
<td>60:2</td>
<td>60:2</td>
<td>60:1</td>
<td>-</td>
</tr>
<tr>
<td>E</td>
<td>40</td>
<td>50</td>
<td>40</td>
<td>40</td>
<td>3 x 40</td>
</tr>
<tr>
<td>F</td>
<td>70/110</td>
<td>60/90</td>
<td>60/90</td>
<td>60/90</td>
<td>3 x 60/90</td>
</tr>
<tr>
<td>G</td>
<td>Cold</td>
<td>Cold</td>
<td>No cold</td>
<td>No cold</td>
<td>-</td>
</tr>
<tr>
<td>H</td>
<td>180</td>
<td>180</td>
<td>130</td>
<td>180</td>
<td>3 x 180</td>
</tr>
</tbody>
</table>

This summary shows that a New 130, 40° nip temperature, 60/90 drying profile and 180 web speed have the possibility to give higher retained solvents so it is recommended these are avoided.

Sidlaw have changed to Holdens, as bond strengths with Novacote had reduced even further, they have had no problems with retained solvents and bond strengths have to date shown material failure.

19.13 Proposed confirmatory trial

<table>
<thead>
<tr>
<th></th>
<th>Proposed</th>
<th>Current Use</th>
</tr>
</thead>
<tbody>
<tr>
<td>Adhesive type</td>
<td>Holdens</td>
<td>Holdens</td>
</tr>
<tr>
<td>Adhesive amount</td>
<td>140</td>
<td>140</td>
</tr>
<tr>
<td>Metal props</td>
<td>Boost</td>
<td>No treatment</td>
</tr>
<tr>
<td>Adhesive mix ratio</td>
<td>60:1</td>
<td>60:1</td>
</tr>
<tr>
<td>Nip temperature</td>
<td>45</td>
<td>45</td>
</tr>
<tr>
<td>Drying profile</td>
<td>70/110</td>
<td>70/90</td>
</tr>
<tr>
<td>Chill roll temperature</td>
<td>Cold</td>
<td>Chill roll off</td>
</tr>
<tr>
<td>Web speed</td>
<td>170</td>
<td>170</td>
</tr>
</tbody>
</table>
There are reasons why some parameters have been kept to certain values, eg chill roll cold as this helps with better reel-up of the material.

The proposed trial should give optimum conditions for both mean and standard deviation.

19.14 Other comments
There was still a large amount of variability even when the lamination process was set to the same conditions. It is most probable that this extra variability is accounted for by the testing conditions and raw material variations. The bond strength is actually a combination of raw materials, process controls and measurement system.

For the confirmatory trials it is important that everything else is performed as in the original trials, ie normal test conditions. A second set of tests will be performed by a second tester ensuring that sample sizes, timings are all measured as carefully as possible.

All data/solvent retention results/bond strengths should be kept in time order to see if data varies through a production run. A number of reels should be run, with five samples per reel tested. Unfortunately due to the destructive nature of the testing, it is not possible to find out the reproducibility of the testing equipment.
20 Confirmation trials (Appendix 5)

20.1 Prediction for Sidlaw experiment (Appendix 5)

The analysis was performed on the transformed data (In). The prediction equation for the confirmation trial is

\[
\text{Mean} = 5.6017 + 0.313 - 0.039 - 0.045
\]

'A' effect 'B' effect 'D' effect

Holdens 140 60:1

Hence Mean = 5.8307 on In scale.

The confidence interval around this value is based on:

\[
\text{CI} = \sqrt{F_{0.05}; 1; \nu_e \left( \frac{1}{\nu_{\text{eff}}} + \frac{1}{r} \right)}
\]

where \( \nu_e \) = degrees of freedom for error

\( \nu_e = \text{mean square error} \)

\( \nu_{\text{eff}} = \frac{N}{1 + \text{d.f. on mean prediction}} \)

\( N \) = total sample size in experiment

\( r \) = number of repeats in confirmation trial

For this experiment

\( \alpha \) = 90% confidence (0.90)

\( \nu_e = 144 \) from ANOVA

\( \nu_e = 0.05746 \)

\( r = 10 \) trials for confirmation run

\( \therefore F_{0.09}; 1; 144 = 2.71 \)

\( \nu_{\text{eff}} = 160 = 40 \)

\( 1 + 3 \)
Cl is ± 0.1395 around the prediction.

\[ \text{\text{: : Cl is } \pm \sqrt{2.72 \times 0.05746 \left( \frac{1}{40} + \frac{1}{10} \right)} \]

When converted back to original scale.

90% Cl interval is between 296 and 391.

20.2 Results of confirmatory trials
The first confirmatory trial showed mean bond strengths in the range 80-118 g/25mm, which was well below the target of 200g/25mm.

It was noted that the failure was not due to the adhesive bond failing, but was due to the metal stripping, gold print split or a mixture of both. Hence the measurement was one of material failure rather than bond strengths.

The bond strengths were measured by two testers using the same piece of equipment, in order to ascertain the effect of tester on bond strength results. It was found that the difference between tests was not statistically significant and hence it is possible to use different testers without affecting the results.

Due to the problem of material failure, the confirmatory trial had to be repeated. It was identified that the materials used during the first confirmatory trial were not from the same suppliers as the original trial. This leads to the suggestion that material supplier could have been another area for study.

The second confirmatory trial was run using the same material suppliers as the original trial work. In this case mean bond strengths of 498 and 452g/25mm were found. These results are
above the predicted results and were significantly higher than the original trial results. Although the results are above the 200g/25mm target, there is a big discrepancy between these results and the original results and indicate that other changes may have occurred.

These changes could include variation in the adhesive or materials through to variation in the test method. As there is a difference between the original trial and the confirmation trial it indicates that the process is not fully understood. Whilst the results found are good, and above the specification, if the process is not fully understood, the results could have been poorer.

It is therefore recommended that the process is further investigated in order to fully understand it.

21 Conclusions

The use of Taguchi methodology could be applied to film lamination.

The experiments identified that the adhesive type hand the most significant effect on bond strength, with the use of a ‘New 130’ applicator roll also being significant. The variation in bond strength was reduced by using a nip temperature of 40°C and a drying profile of 70/110°C. For the whole experiment the Novacote adhesive gave a lower mean bond strength but lower variability.

If only the Holdens adhesive was analysed, then a mixing ratio of 60:2 was slightly better for mean bond strength and a new 130 applicator roll. The variability was reduced by use of a 140 applicator roll, boost treatment of the metallised film and a drying profile of 70/110°C.
In all cases the web speed, temperature of chill roll, nip temperature and boost treatment did not have a significant effect on the mean bond strength. However these could have a large effect on costs or productivity.

As the confirmatory trials did not agree with the prediction it indicates that the process is not fully understood and that further investigations should be carried out in order to fully understand it.

S C Tilbury (Miss)
Senior Consultant
Packaging Technology Group

D A Shires
Chief Consultant
Packaging Technology Group
Appendix 1

Adhesives - chemical data sheets
NOVACOTE NC 120 ASL + 111 B

Technical Data Sheet

Description: NOVACOTE NC 120 ASL + 111 B is an alcohol-dilutable two-part prepolymer adhesive for the lamination of film to film, paper and metallized structures. It provides excellent clarity, high green tack, high final bonds, long pan life unaffected by moisture reaction due to high humidities. NOVACOTE NC 120 ASL differs from NC 120 A, in that it contains modifiers intended to reduce the scavenging of slip additive from polyolefine films.

Specifications:

<table>
<thead>
<tr>
<th></th>
<th>NOVACOTE 120 ASL</th>
<th>111 B</th>
</tr>
</thead>
<tbody>
<tr>
<td>Solids</td>
<td>70 +/- 1 %</td>
<td>100 %</td>
</tr>
<tr>
<td>Viscosity</td>
<td>9000-12000 cps</td>
<td>10000-16000 cps</td>
</tr>
<tr>
<td>Density</td>
<td>0.96 gms/cc</td>
<td>1.16 gms/cc</td>
</tr>
<tr>
<td>Storage Life</td>
<td>min. 6 months</td>
<td>min. 6 months</td>
</tr>
<tr>
<td>Mixing Ratio</td>
<td>100 : 10</td>
<td></td>
</tr>
</tbody>
</table>

F & DA/BGA
Status: The individual components of NOVACOTE NC 120 ASL and 111 B are listed and comply with F.D.A. Regulations 21 CFR 175.105.

Product Performance: NOVACOTE NC 120 ASL + 111 B has outstanding machinability and wettabiliy on a wide range of laminations. These include polymer-coated or uncoated, printed and metallized films like Cellophane, oriented polypropylene, polyester, Nylon polyethylene and paper. For polyester, polypropylene and polyethylene a corona discharge treatment is required for best wetting/adhesion.
Mixing instructions:

Application of NOVACOTE NC 120 ASL + 111 B recommended at 30 to 35 % solids by diluting to the required running viscosity as follows:

<table>
<thead>
<tr>
<th></th>
<th>30 % solids</th>
<th>35 % solids</th>
</tr>
</thead>
<tbody>
<tr>
<td>kg NOVACOTE NC 120 ASL</td>
<td>100</td>
<td>100</td>
</tr>
<tr>
<td>kg NOVACOTE NC 111 B</td>
<td>10</td>
<td>10</td>
</tr>
<tr>
<td>kg Diluent</td>
<td>157</td>
<td>119</td>
</tr>
<tr>
<td>kg Total</td>
<td>267</td>
<td>229</td>
</tr>
</tbody>
</table>

Viscosity (No. 4 Ford Cup) 16 sec 25 sec

Prepare the mix by adding NOVACOTE NC 111 B to 120ASL, blend thoroughly, then adjust to final solids and viscosity.

Diluents:

Recommended diluting solvents are specific grades of denatured ethanol, isopropanol and mixtures of alcohol/water (90:10) also in blends with hexane up to 1:1. MEK as denaturant in ethanol could cause odour problems. Other solvents like ethyl acetate or acetone also a mixture of acetone/water (90:10) may be used. Cleaning solvents are alcohols, esters or ketones.

Pan Life:

NOVACOTE NC 120 ASL + 111 B has excellent dynamic pan stability because there is no interreaction possible due to moisture influence. Pot life of the mixed adhesive at 35 % solids should be three days or more.

Drying:

Oven temperatures are usually in the range of 00 to 90 °C, a web temperature of about 65 °C is adequate.

Combining:

Usually a nip temperature of 50 to 65 °C is sufficient. When combining two polymer coated webs, warmer nips may improve clarity and bond strengths.
Curing time: The bonds off machine are normally greater than 200 gms/15 mm. The curing reaction starts immediate after lamination. Further processing is possible 20 hours after lamination, maximum crosslinking is reached after 5 to 7 days depending on substrate and machine conditions.

Storage: NOVACOTE NC 120 ASL + 111 B are inflammable and should be stored in a cool place.

07/89
Material Safety Data Sheet

Company: NOVACOTE International
2000 Hemery Schlachtkurgrale 62
Tel. 040/8531030

Product name: NOVACOTE NC 120 ABL

1.1 Chemical characteristics: Polyaminepolyurethane 70% solids in Ethanol

1.2 Physical constitution: liquid
1.3 Colour: waterwhite - light amber
1.4 Smell: Ethanol

2. Physical and safety data
2.1 Change in state:
Solidification temperature: not applicable
Initial boiling point: 78 °C

2.2 Density: 0,98 g/cm³ at 25°C

2.3 Vapour pressure: 77 hPa at 25°C

2.4 Viscosity: 7000 - 12000 mPa.s at 25°C

2.5 Solubility in water: not evaluated

2.6 pH-Value: not evaluated

2.7 Flash point: 412 °C

2.8 Ignition temperature: 425 °C

2.9 Explosive limits: lower 3,5 upper 15,0 Vol-%

2.10 Thermal decomposition: no decomposition if applied as specified

2.11 Hazardous decomposition products:
in case of burning oxides of carbon and nitrogen, vapors of isocyanates and traces of hydrocyanic acid.

2.12 Dangerous reactions: none

2.13 Additional informations: none

3. Transport

GVVSee/IMDG-Code: 3.2
UN-Nr.: 1133
MFAG 305
EmS: 3-05

GBVE/CGVS:
K1 3 Z1 5b
R1B/ADR: K1 3 Z1 5b

ICAO/IATA-DGR: 3.2 1133 11

Additional information: MFAG - classification is based on the solvent different from the recommendation of IMDG

4. German Regulations

Verordnung über brennbare Flüssigkeiten (VBF): A1
MAK-Wert: 1000 ppm Spitzenbegrenzung Kat.IV (Ethanol)
TA-Luft: Klasse III (Ethanol)

Gefahrstoffverordnung:
Symbol: F
Description of danger: highly flammable

R 11: Highly flammable
S 71: Keep container tightly closed
S 16: Keep away from sources if ignition - No smoking

(continued)
3. Precautions, storage and handling

3.1 Technical precautions
Keep container tightly closed, store in a cool dry place. Keep away from sources of ignition.
Take care for adequate ventilation during application and storage. Minimize formation of aerosols.

3.2 Personal protective equipment
Respiratory protection: If necessary use face mask with fume/dust filter
Skin protection: If necessary use protective gloves/face protection
Eye protection: If necessary use protective goggles.

3.3 Personal hygiene
Keep away from food, drink and animal feeding stuffs. Keep working clothes separate.
Take all contaminated clothing off immediately.

3.4 Fire and explosion protection
Keep away from sources of ignition - No smoking. Take precaution against static discharge.

3.5 Waste disposal
Dispose in accordance with local and national environmental regulations.

6. Measures for accidents and fire

6.1 Spillage or leakage
Ventilate area, remove all sources of ignition, clean up with absorbent material (sand, sand dust, kieselguhr)
Do not pour in sewerage system.
Act in accordance with waste disposal regulations.

6.2 Fire extinguishing media
Water fog, extinguishing powder, extinguishing foam, carbon dioxide
Not suitable:
In case of firefighting wear self-containing breathing equipment.

6.3 First aid
After contact with skin, wash immediately with plenty of water and soap
In case of contact with eyes, rinse immediately with plenty of water and seek medical advice.
If swallowed do not induce vomiting. Keep patient rested, seek immediately medical advice.

6.4 Additional informations:

7. Details of toxicology

Inhalation: In high concentrations irritation to mucous membranes and narcosis possible.
Reactions and coordination may be affected.
Skin contact: Repeated and prolonged skin contact may cause irritation and dermatitis.
Eye contact: Irritation
Ingestion: Very small quantities may cause serious injuries of health.

8. Details upon ecology

Do not allow to get into inshore waters, sewer or soil.

9. Further references
The attached M.S.D.S. is issued by Novacote International of Germany, to supplement the safety information given, the U.K. Occupational Exposure Limits which have been established by the Health and Safety Commission for certain ingredients, which may appear on the Novacote M.S.D.S., are listed below:

<table>
<thead>
<tr>
<th>Ingredient</th>
<th>Long Term Exposure Limit (8-hour TWA)</th>
<th>Short Term Exposure Limit (10 Minute TWA)</th>
<th>Notes</th>
</tr>
</thead>
<tbody>
<tr>
<td>Isocyanates</td>
<td>0.02 mgm⁻¹</td>
<td>0.07 mgm⁻¹</td>
<td>(M)</td>
</tr>
<tr>
<td>Ethyl Acetate</td>
<td>400 ppm</td>
<td>-</td>
<td>(O)</td>
</tr>
<tr>
<td>Ethanol</td>
<td>1000 ppm</td>
<td>-</td>
<td>(O)</td>
</tr>
</tbody>
</table>

(M) Maximum Exposure Limit

(O) Occupational Exposure Standard

TWA - Time Weighted Average

These Occupational Exposure Limits should be observed; for further information see HSE Guidance Note EH40
1. Chemical characteristics: Reaction product of Bisphenol A and Epichlorhydrin
100% Solids  CAS-Nr. 25068-38-6

1.2 Physical constitution: liquid
1.3 Colour: yellowish
1.4 Smell: odourless

2. Physical and safety data

2.1 Change in state:
Solidification temperature: < -15 °C
Initial boiling point: not applicable °C

2.2 Density:
1.16 g/cm³ at 25°C
2.3 Vapour pressure:
not evaluated hPa at 25°C
2.4 Viscosity:
10000 - 16000 mPa.s at 25°C
2.5 Solubility in water:
not evaluated
2.6 pH-Value:
not applicable
2.7 Flash point:
+252 °C
2.8 Ignition temperature:
> 300 °C
2.9 Explosive limits: not applicable
2.10 Thermal decomposition:
no decomposition if applied as specified
2.11 Hazardous decomposition products:
none
2.12 Dangerous reactions:
with basic substances
2.13 Additional informations:
high temperatures for a long period decompose the resin

3. Transport

GS/See/IMDG-Code: --
UN-Nr.: ----
MFAC ----
E3S: ----

4. German Regulations

EG-No. 603-074-00-8

Gefahrstoffverordnung:

Symbol: Xi Description of danger: irritant
R 36/38: Irritating to eyes and skin
R 43: May cause sensitization by skin contact
S 28: After contact with skin, wash immediately with plenty of water and soap
S 37/39: Wear suitable gloves and eye/face protection

(continued)
5. Precautions, storage and handling

5.1 Technical precautions
   Keep container tightly closed, store in a cool dry place. Keep away from sources of ignition.
   Take care for adequate ventilation during application and storage. Minimize formation of aerosols.

5.2 Personal protective equipment
   Respiratory protection: —
   Skin protection: Use protective gloves/face protection
   Eye protection: Use protective goggles.

5.3 Personal hygiene
   Keep away from food, drink and animal feeding stuffs. Keep working clothes separate.
   Take all contaminated clothing off immediately.

5.4 Fire and explosion protection
   —

5.5 Waste disposal
   Dispose in accordance with local and national environmental regulations.

6. Measures for accidents and fire

6.1 Spillage or leakage
   Clean up with absorbent material (sand, sawdust, kieselguhr)
   Do not pour in sewage system.
   Act in accordance with waste disposal regulations.

6.2 Fire extinguishing media
   Extinguishing powder, extinguishing foam, carbon dioxide
   Not suitable: Water
   In case of firefighting wear self-contained breathing equipment.

6.3 First aid
   After contact with skin, wash immediately with plenty of water and soap
   In case of contact with eyes, rinse immediately with plenty of water and seek medical advice.
   If swallowed do not induce vomiting. Keep patient rested, seek immediately medical advice.

6.4 Additional informations:

7. Details of toxicology
   Inhalation: If you feel unwell, look for fresh air, seek medical advice.
   Skin contact: Repeated and prolonged skin contact may cause irritation and dermatitis.
   Eye contact: Irritation
   Ingestion: Very small quantities may cause serious injuries of health.

8. Details upon ecology
   Do not allow to get into inshore waters, sewer or soil.

9. Further references
SUPPLEMENTARY INFORMATION TO NOVACOTE MATERIAL SAFETY DATA SHEETS

The attached M.S.D.S. is issued by Novacote International of Germany. To supplement the safety information given, the TWA Occupational Exposure Limits, which have been established by the Health and Safety Commission for certain ingredients, which may appear on the Novacote M.S.D.S., are listed below:

<table>
<thead>
<tr>
<th>Ingredient</th>
<th>Long Term Exposure Limit (8-hour TWA)</th>
<th>Short Term Exposure Limit (10 Minute TWA)</th>
<th>Notes</th>
</tr>
</thead>
<tbody>
<tr>
<td>Isocyanates</td>
<td>0.02 mg/m³</td>
<td>0.07 mg/m³</td>
<td>(M)</td>
</tr>
<tr>
<td>Ethyl Acetate</td>
<td>400 ppm</td>
<td>-</td>
<td>(C)</td>
</tr>
<tr>
<td>Ethanol</td>
<td>1000 ppm</td>
<td>-</td>
<td>(C)</td>
</tr>
</tbody>
</table>

M: Maximum Exposure Limit
C: Occupational Exposure Standard
TWA - Time Weighted Average

These Occupational Exposure Limits should be observed; for further information see HSE Guidance Note EH40.
DESCRIPTION: 10-2525/3/10-2526/3 is a two component curing polyurethane laminating adhesive designed for combinations of plastic films, foils, paper and metallised substrates.

COMPONENTS: 10-2525/3 Adhesive 10-2526/3 Coreactant

TYPICAL USES: For use where high bonds on aluminium foil and transparent laminations are required and for metallised substrates. Boilable on transparent laminates.

Typical laminations for 10-2525/3/10-2526/3 are:

- Aluminium foil to treated LDPE
- Polyester to aluminium foil
- Polyester to treated LDPE
- Metallised polyester to treated LDPE
- Metallised polypropylene to treated LDPE
- Polyamide to treated LDPE
- Oriented polypropylene to treated LDPE

TYPICAL PHYSICAL PROPERTIES:

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Solids content</td>
<td>70% ± 2%</td>
</tr>
<tr>
<td>Viscosity</td>
<td>1000-2500 cps</td>
</tr>
<tr>
<td>Density</td>
<td>1.14 ± 0.01</td>
</tr>
<tr>
<td>Flash Point</td>
<td>Below 22°C</td>
</tr>
<tr>
<td>Appearance</td>
<td>Hazy to clear amber solution</td>
</tr>
<tr>
<td>Base Solvent</td>
<td>Ethyl acetate</td>
</tr>
</tbody>
</table>

DILUENTS: Ethyl Acetate, Methyl Ethyl Ketone or Acetone

Use urethane grade solvents only.
MIXING RATIO: 

10-2525/3 69 kilogrammes
10-2526/3 1 kilogramme

The adhesive is supplied in pre-weighed containers to give correct mixing ratio.

MIXED SOLIDS: 

The mixed solid content of the undiluted adhesive is typically 70.4%.

MIXING INSTRUCTIONS: Mix as above and dilute to machine viscosity using a recommended diluent. Dilute to a known solids content rather than a viscosity.

APPLICATION

Typical application viscosities of 10-2525/3/10-2526/3 with Ethyl Acetate as a diluent are:

<table>
<thead>
<tr>
<th>% Solids</th>
<th>Viscosity DIN 4 150 at 20</th>
</tr>
</thead>
<tbody>
<tr>
<td>30</td>
<td>13&quot;</td>
</tr>
<tr>
<td>35</td>
<td>15&quot;</td>
</tr>
<tr>
<td>40</td>
<td>18&quot;</td>
</tr>
<tr>
<td>45</td>
<td>23&quot;</td>
</tr>
</tbody>
</table>

MIXED POT LIFE: 

We recommend that only sufficient adhesive is mixed for an 8 hour period of use.

LAMINATING

PROCEDURE: The adhesive should be applied to the most heat and solvent resistant and least extensible of the laminating films.

APPLICATION: Gravure cylinder or smooth roll may be used.

COATING WEIGHT: 1.5 - 3.5 g/m².

Clear laminates are generally satisfactory with the lower coating weights, however, where there are printed areas or for aluminium foil laminates, the slightly higher coating weights are normally used.

DRYING: Forced air drying sufficient to remove the solvents completely.

COMBINING: The coated web should be combined to the second web using a heated nip roller. Temperatures of 50°C - 90°C are recommended as high as is practical.
FURTHER PROCESSING: The adhesive gives good initial/green bonds which cure to enable further processing and slitting within 24 hours. Maximum properties will develop in 5 - 10 days.

INKS: All lamination inks should be checked for suitability for a particular end use. Ink Alcohol solvents, if retained, will destroy the reactivity of the adhesive and cause undercure. Care must be taken to fully dry the inks, particularly where ink coverage is high or multi-layer.

F.D.A. STATUS: The individual components of 10-2525/3/10-2526/3 are listed in the Code of Federal Regulations, Section 175 - 105 Adhesives.

B.G.A. STATUS: The individual components of 10-2525/3/10-2526/3 are listed in Section XXVIII of the B.G.A. Regulations "Crosslinked Polyurethanes as adhesives for packaging materials".

STORAGE: 10-2525/3 is the water/moisture sensitive component.

Store under dry conditions at 5 - 30°C.

SHELF LIFE: When stored in the original unopened containers at the recommended storage conditions, these products will be suitable for use for up to six months from the date of shipment.
IDENTIFICATION OF PREPARATION AND OF COMPANY

PRODUCT NUMBER: 10-2525/3

PRODUCT NAME: ADHESIVE

Supplied by: Holden Flexpack Ltd., Bordesley Green Road, Birmingham, B9 4TQ, ENGLAND.

Emergency Telephone: Birmingham (021) 766 6600 DAYTIME
(021) 766 6691 NIGHTTIME

INTENDED USE

This product is formulated for the factory coating of flexible packaging substrates, only after reference to the supplier's Technical Information. Suitability for other uses must be referred to the supplier.

2. COMPOSITION/INFORMATION ON INGREDIENTS

Certain ingredients, having recognised health effects, may be present at concentrations above the exemption limits (0.1% for toxic materials and 1% for all others). These are:

<table>
<thead>
<tr>
<th>CAS No.</th>
<th>HAZARDOUS INGREDIENTS</th>
<th>% SYMBOLS HEALTH R PHRASES</th>
</tr>
</thead>
<tbody>
<tr>
<td>101-68-3</td>
<td>DIPHENYL METHANE DIISOCYANATE</td>
<td>&lt; 2.5 Xn</td>
</tr>
<tr>
<td>141-78-6</td>
<td>ETHYL ACETATE</td>
<td>25-50 F</td>
</tr>
</tbody>
</table>

Note: The text for R phrase codes shown above (if any) is given in section 16.

HER INGREDIENTS:

POLYURETHANE RESIN (NON-VOL)

HAZARDS IDENTIFICATION

This product has been assessed under the Dangerous Preparations Directive (88/379/EEC) and is classified as follows:

- Xn HARMFUL
- F HIGHLY FLAMMABLE

R20 Harmful by inhalation.
R36/37/38 Irritating to eyes, respiratory system and skin.
R42 May cause sensitisation by inhalation.
P93 Contains Isocyanates. See information supplied by the manufacturer.

Information on Occupational Exposure Limits is given in Section 8.
4. FIRST-AID MEASURES

In all cases of doubt, or where symptoms persist, seek medical attention.

INHALATION: Remove to fresh air, keep patient warm and at rest. If breathing is irregular or stopped administer artificial respiration. Give nothing by mouth. If unconscious place in the recovery position. Seek medical advice.

EYE CONTACT: Irrigate copiously with clean, fresh water for at least 10 minutes, holding lids apart. Seek medical advice.

SKIN CONTACT: Remove contaminated clothing, wash skin thoroughly with soap and water, or use a proprietary skin cleanser. Do not use solvents or thinners. Seek medical advice if symptoms persist.

INGESTION: If accidentally swallowed, DO NOT INDUCE VOMITING. Keep at rest and obtain medical attention.

5. FIRE-FIGHTING MEASURES

Extinguishing media: Recommended - alcohol resistant foam, CO2, powders. Not to be used - waterjet.

Recommendations: Fire will produce dense black smoke. Exposure to decomposition products may cause a health hazard. Firefighters should wear self-contained breathing apparatus.

Closed containers exposed to fire should be cooled with water. Do not allow run-off from fire-fighting to enter drains or water-courses.

6. ACCIDENTAL RELEASE MEASURES

Exclude sources of ignition and ventilate the area. Avoid breathing vapours. Refer to protective measures listed in section 8. Contain and collect spillage with non-combustible absorbent materials, eg sand, earth, vermiculite or diatomaceous earth and place in a metal container, fill with water and leave open for 24 hours to neutralise, before sealing for disposal according to local regulations (see section 13). Isocyanate decontaminant Ethanol 50%, water 45%, Ammonia 5%. Do not allow to enter drains or water courses. Clear preferably with a detergent; avoid use of solvents.

If the product contaminates lakes, rivers or sewages, inform appropriate authorities in accordance with local regulations.

7. HANDLING AND STORAGE

Handling CONDITIONS: Avoid contact with the skin and eyes. Smoking, eating and drinking should be prohibited in the work area.

Vapours are heavier than air and may spread along floors. Vapours may also form explosive mixtures with air. Prevent the creation of flammable or explosive concentrations of vapour in air and avoid vapour concentration higher than the occupational exposure limits. Additionally, the product should only be used in areas from which all naked lights and other sources of ignition have been excluded. Electrical equipment should be protected to the appropriate standard.

Reparations may charge electrostatically: always use earthing leads when transferring from one container to another. Operators should wear antistatic footwear and clothing, and floors should be of the conducting type.
STORAGE CONDITIONS: Store in a cool, dry, well ventilated place and keep container tightly closed.

Products with a flash point of 32 degrees Celsius or below must be stored in accordance with the Highly Flammable Liquids and Liquefied Petroleum Gases Regulations 1972.

8. EXPOSURE CONTROLS/PERSONAL PROTECTION

HAZARDOUS INGREDIENT

<table>
<thead>
<tr>
<th>DIPHENYLMETHANE DIISOCYANATE</th>
<th>LTEL (8hr TWA) ppm</th>
<th>STEL (10min) ppm</th>
<th>Time (mins)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>400.00</td>
<td>1400.00</td>
<td>0.07</td>
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<tr>
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<tr>
<td></td>
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<td></td>
<td>0 OES</td>
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</table>

OEL - Occupational Exposure Limits (HSE Guidance Note EH40)  
OES - Occupational Exposure Standard  
MEL - Maximum Exposure Limit  
SEL - Manufacturer's recommended Limit  
TEL - Short term Exposure Limit  
STEL - Long-term Exposure Limit. TWA - Time weighted Average  
Risk of absorption through unbroken skin  
Further guidance on OELs and assessment of occupational exposure to harmful materials (including mixed exposures) is given in HSE Guidance Note EH40.

RESPIRATORY PROTECTION: Avoid the inhalation of vapour, particulates and mist. Where reasonably practicable, this should be achieved by the use of local exhaust ventilation and good general ventilation. If this is not sufficient to maintain concentrations of particulates and solvent vapour below the occupational exposure limit, respiratory protection must be worn.

The selection of respiratory equipment should be in accordance with BS 4275. Recommendations for the selection, use and maintenance of Respiratory Equipment, and the current certificates of approval are issued annually by the Health and Safety Executive.

If isocyanate-based products are applied by high speed roller, the aerosol mist droplets produced are mainly of respirable size and inhalation must therefore be avoided. Air-fed respiratory equipment must be worn even when good ventilation is provided. Steps must be taken to ensure that persons nearby who may be unconnected with the application operation are not affected.

Isocyanate-based products are applied by high speed roller, the aerosol mist droplets produced are mainly of respirable size and inhalation must therefore be avoided. Air-fed respiratory equipment must be worn even when good ventilation is provided. Steps must be taken to ensure that persons nearby who may be unconnected with the application operation are not affected.

WHERE OF THIS PRODUCT AT ROOM TEMPERATURE PPOSES LITTLE HAZARD, HOWEVER IF THE PRODUCT IS HEATED, ISOCYANATE VAPOUR MAY BE PRODUCED AND ADDITIONAL CARE MUST BE TAKEN TO ENSURE THE OEL IS NOT EXCEEDED.

NDD PROTECTION: Wear suitable gloves for protection against materials in section 2.

YE PROTECTION: Wear suitable eye protection to BS 2092.

RIN PROTECTION: Wear appropriate protective clothing. Barrier creams may help to protect exposed areas of skin but are not a substitute for full physical protection.
All the above precautions also apply to dry sanding and thermal decomposition eg welding or flame cutting of the dried product, which will give rise to dense smoke and/or fumes.

9. PHYSICAL AND CHEMICAL PROPERTIES

Physical State: Liquid
Specific gravity: 1.136
Flash point: -18 < 21 deg. Celsius
Water miscible: No information
pH: No information
Explosion limits: Lower - approx. 0.8% Upper - no information

10. STABILITY AND REACTIVITY

CONDITIONS TO AVOID: Extremes of temperature.
To prevent the creation of flammable concentrations of vapour in air, good natural ventilation, and if necessary, local exhaust ventilation, should be provided. The accumulation of dry overspray, contaminated rags, etc may result in spontaneous combustion. Good housekeeping standards plus the regular and safe removal of waste materials will minimise the risk.

MATERIALS TO AVOID: Keep away from oxidising agents, strongly alkaline and strongly acidic materials in order to avoid exothermic reactions.

HAZARDOUS DECOMPOSITION PRODUCTS: When exposed to high temperatures may produce hazardous decomposition products such as carbon monoxide and dioxide smoke and oxides of nitrogen.

11. TOXICOLOGICAL INFORMATION

There is no experimental data available on the product itself. However, it has been assessed according to the Preparations Directive (88/379/EEC) and classified for toxicological hazards. See Section 15 for these details including associated risk and safety phrases.

Exposure of vapour are irritating to eyes and respiratory system. Excessive concentrations may produce effects on the central nervous system including drowsiness. In extreme cases loss of consciousness may result. Long term exposure to vapour concentrations in excess of quoted OELs may result in adverse health effects. Splashes entering the eye will cause discomfort and possible damage. Prolonged contact with the skin may have a defatting effect which may lead to skin irritation and in some cases dermatitis.

Moisture-cured, two-pack and blocked isocyanate containing products may cause irritation and/or sensitisation to the respiratory system leading to an asthmatic condition, wheeziness and tightness of the chest. Persons with a history of chronic or recurrent respiratory disease should not be employed in any process in which these products are used.

12. ECOLOGICAL INFORMATION

There is no specific data available on the product itself. The product should not be allowed to enter drains or watercourses. Products classified as Marine Pollutants are indicated as such under section 1A - Transport.
13. DISPOSAL CONSIDERATIONS

Waste material and containers must be treated as a fire hazard and disposed in accordance with the general requirements of the Control of Pollution Act 1974.

The 1990 Environmental Protection Act introduces new controls on the disposal or depositing of waste. Due regard should be given to the "duty of care" required under the regulations and the guidance given under this act.

4. TRANSPORT INFORMATION

N number: 1133 Packing group: II
DR/RID Class: 3 Item: 5b
MDG Class: 3.2 MFAG: 330 EMS: 3-05

Hazard label(s): FLAMMABLE LIQUID

5. REGULATORY INFORMATION

A.D SUBS.: DIPHENYLMETHANE DIISOCYANATE

HAZARD CLASSIFICATION:
Xn HARMFUL
F HIGHLY FLAMMABLE

Warning label phrases:

R11 Highly flammable.
R20 Harmful by inhalation.
R36/37/38 Irritating to eyes, respiratory system and skin.
R42 May cause sensitisation by inhalation.
P93 Contains Isocyanates. See information supplied by the manufacturer.
S23 Do not breathe vapour/spray.
S26 In case of contact with eyes, rinse immediately with plenty of water and seek medical advice.
S51 Use only in well ventilated areas.

6. OTHER INFORMATION

Text for R Phrases shown in section 2 describing each ingredient:

R20 Harmful by inhalation.
R36/37/38 Irritating to eyes, respiratory system and skin.
R42 May cause sensitisation by inhalation.

Information given in this data sheet is required pursuant to EC Directive 379/EEC.


Guidance on the use of Isocyanate-based products is given in HSE Guidance leaflets MS8 "Isocyanates - Medical Surveillance" and EH16 "Isocyanates - Toxicity, Precautions". Information is also given in the Printing Industry Advisory Committee publication "Safety in the use of Isocyanate Pre-polymers the Printing and Packaging Industries".
1. IDENTIFICATION OF PREPARATION AND OF COMPANY

PRODUCT NUMBER: 10-2526/3

PRODUCT NAME: ADHESIVE

Supplied by: Holden Flexpack Ltd., Bordesley Green Road, Birmingham, B9 4TQ, ENGLAND.
Emergency Telephone: Birmingham (021) 766 6600 DAYTIME

INTENDED USE

This product is formulated for the factory coating of flexible packaging substrates, only after reference to the supplier's Technical Information. Suitability for other uses must be referred to the supplier.

2. COMPOSITION/INFORMATION ON INGREDIENTS

Certain ingredients, having recognised health effects, may be present at concentrations above the exemption limits (0.1% for toxic materials and 1% for all others). These are:

<table>
<thead>
<tr>
<th>CAS No.</th>
<th>HAZARDOUS INGREDIENTS</th>
<th>%</th>
<th>SYMBOLS HEALTH R PHRASE</th>
</tr>
</thead>
<tbody>
<tr>
<td>111-46-6</td>
<td>DIETHYLENE GLYCOL</td>
<td>50-100</td>
<td></td>
</tr>
</tbody>
</table>

Note: The text for R phrase codes shown above (if any) is given in section 1.

OTHER INGREDIENTS:

None

3. HAZARDS IDENTIFICATION

This product has been assessed under the Dangerous Preparations Directive (88/379/EEC) and is classified as follows:

: NOT CLASSIFIED

Information on Occupational Exposure Limits is given in Section 8.

FIRST-AID MEASURES

In all cases of doubt, or where symptoms persist, seek medical attention.
**INHALATION:** Remove to fresh air, keep patient warm and at rest. If breathing is irregular or stopped administer artificial respiration. Give nothing by mouth. If unconscious place in the recovery position. Seek medical advice.

**EYE CONTACT:** Irrigate copiously with clean, fresh water for at least 10 minutes, holding lids apart. Seek medical advice.

**KIN CONTACT:** Remove contaminated clothing, wash skin thoroughly with soap and water, or use a proprietary skin cleanser. Do not use solvents or thinners. Seek medical advice if symptoms persist.

**INGESTION:** If accidentally swallowed, DO NOT INDUCE VOMITING. Keep at rest and obtain medical attention.

5. **FIRE-FIGHTING MEASURES**

Extinguishing media: Recommended - alcohol resistant foam, CO2, powders. Not to be used - waterjet.

Recommendations: Fire will produce dense black smoke. Exposure to decomposition products may cause a health hazard. Firefighters should wear self-contained breathing apparatus. Closed containers exposed to fire should be cooled with water. Do not allow run-off from fire-fighting to enter drains or water-courses.

5. **ACCIDENTAL RELEASE MEASURES**

Exclude sources of ignition and ventilate the area. Avoid breathing vapours. Refer to protective measures listed in section 8. Contain and collect spillage with non-combustible absorbent materials, eg sand, earth, vermiculite, diatomaceous earth, and place in container for disposal according to local regulations (see section 13). Do not allow to enter drains or watercourses. Clean preferably with a detergent; avoid use of solvents. If the product contaminates lakes, rivers or sewages, inform appropriate authorities in accordance with local regulations.

6. **HANDLING AND STORAGE**

**STORAGE CONDITIONS:** Avoid contact with the skin and eyes. Smoking, eating and drinking should be prohibited in the work area.

**STORAGE CONDITIONS:** Store in a cool, dry, well ventilated place and keep container tightly closed.

Water-borne products must be protected from frost and extremes of temperature.

7. **EXPOSURE CONTROLS/PERSONAL PROTECTION**

<table>
<thead>
<tr>
<th>DANGEROUS INGREDIENT</th>
<th>LTEL (8hr TWA) ppm</th>
<th>LTEL (8hr TWA) mg/m³</th>
<th>STEL (10m) ppm</th>
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<th>Time (mins)</th>
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<tr>
<td>ETHYLENE GLYCOL</td>
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<td>100.00</td>
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<td></td>
<td></td>
<td>OES</td>
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</table>

**OES** - Occupational Exposure Standard

**EH40** - Occupational Exposure Limit

**HSE** - Manufacturer's recommended Limit
**Long-term Exposure Limit (TEL)** - TWA - Time weighted Average
**Short term Exposure Limit (STEL)** - Risk of absorption through unbroken skin

Further guidance on OELs and assessment of occupational exposure to harmful materials (including mixed exposures) is given in HSE Guidance Note EH40.

**Respiratory Protection:** Avoid the inhalation of vapour, particulates and spray mist. Where reasonably practicable, this should be achieved by the use of local exhaust ventilation and good general ventilation. If this is not sufficient to maintain concentrations of particulates and solvent vapour below the occupational exposure limit, respiratory protection must be worn.

The selection of respiratory equipment should be in accordance with BS 4275. Recommendations for the selection, use and maintenance of Respiratory Equipment, and the current certificates of approval are issued annually by the Health and Safety Executive.

**Hand Protection:** Wear suitable gloves for protection against materials in Section 2.

**Eye Protection:** Wear suitable eye protection to BS 2092.

**Skin Protection:** Wear appropriate protective clothing. Barrier creams may help to protect exposed areas of skin but are not a substitute for full body protection.

All the above precautions also apply to dry sanding and thermal decomposition or welding or flame cutting of the dried product, which will give rise to gases and/or fumes.

**Physical and Chemical Properties**

- **Physical State:** Liquid
- **Specific Gravity:** 1.100
- **Flash Point:** > 100 deg. Celsius
- **Water Miscible:** No information
- **pH:** No information

**Stability and Reactivity**

- **Conditions to Avoid:** Extremes of temperature.
- **Materials to Avoid:** Keep away from oxidising agents, strongly alkaline and strongly acidic materials in order to avoid exothermic reactions.

**Hazardous Decomposition Products:** When exposed to high temperatures may produce hazardous decomposition products such as carbon monoxide and dioxide, smoke and oxides of nitrogen.

**Toxicological Information**

There is no experimental data available on the product itself. However, it has been assessed according to the Preparations Directive (88/379/EEC) and classified for toxicological hazards. See Section 15 for these details, including associated risk and safety phrases.

Exposure to vapour may irritate the eyes and respiratory system. Excessive concentrations may produce effects on the central nervous system including drowsiness. In extreme cases loss of consciousness may result. Long
term exposure to vapour concentrations in excess of quoted OELs may result in adverse health effects. Splashes entering the eye will cause discomfort or possible damage. Prolonged contact with the skin may have a defatting effect which may lead to skin irritation and in some cases dermatitis.

12. ECOLOGICAL INFORMATION

There is no specific data available on the product itself. The product should not be allowed to enter drains or watercourses. Products classified as Marine Pollutants are indicated as such under section 14 - Transport.

13. DISPOSAL CONSIDERATIONS

Waste material and containers must be treated as a fire hazard and disposed in accordance with the general requirements of the Control of Pollution Act 1974. The 1990 Environmental Protection Act introduces new controls on the disposal or depositing of waste. Due regard should be given to the "duty of care" required under the regulations and the guidance given under this act.

14. TRANSPORT INFORMATION

UN number: 0000  Packing group: EX
ADR/RID Class: EXEMPT  Item: EXEMPT
IMDG Class: EXEMPT  MFAG: EXEMPT  EMS: EXEMPT

5. REGULATORY INFORMATION

NAMED SUBS.: Nil
HAZARD CLASSIFICATION: NOT CLASSIFIED

6. OTHER INFORMATION

Text for R Phrases shown in section 2 describing each ingredient:


The information on this sheet is not a specification; it does not guarantee specific properties. The information is intended to provide general guidance as to health and safety based upon our knowledge of the handling, storage and use of the product. It is not applicable to unusual or non-standard uses of the product nor where instructions and recommendations are not followed.

For further information please contact:
Appendix 2

Additional work on metallised surface properties
Appendix 3

Background bond strength data
File: INTERPLY.DAT
INTERPLY BOND STRENGTHS IN g/25mm
INTERPLY RESULTS FOR OPP/MET OPP LAMINATES

Samples: 59
Mean: 261.458
Std. Dev.: 55.59
Process Cap: +/- 3s

3 s Limits: (94.687, 428.23)
Spec. Limits: (200 , )
Est x outside: (13.45 , .0000)

Mean
LSL
-3σ
+3σ
100 200 300 400
0 5 10 15 20
Appendix 4

Full analysis of experimental data
<table>
<thead>
<tr>
<th>FACTOR</th>
<th>MEAN</th>
<th>SDEV</th>
<th>'BEST' SETTING</th>
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<td>(HOLDENS)</td>
<td>(NEW 130)</td>
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## HOLDENS - SUMMARY

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## EXPERIMENTAL WORKSHEET FOR SIDLAW PACKAGING

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<th>DRY PROF</th>
<th>CHILL ROUT</th>
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<th>BOND STRENGTH</th>
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### NOTES IN TRIAL 2/2/95

1. 140 CYLINDER USED INSTEAD OF OLD 130
2. BOOST WAS SET AT 40 AMPS
3. REELS TO BE REWOUND AND TESTED AFTER 24 HRS
4. SAMPLES TAKEN AT START MIDDLE AND END OF EACH RUN
5. 400 METRES OF SAMPLE WERE RUN FOR EACH RUN
6. ADHESIVES DILUTED TO SAME VISCOSITY WITH ETHYL ACETATE
7. ALL CLEANING DONE WITH ETHYL ACETATE
8. FILM TENSION ALL RUNS 10 KG
9. NOTHING DIFFERENT NOTED DURING RUNNING OF TRIAL

### NOTES ON RESULTS

1. INDICATES THAT WEB HAS BROKEN RATHER THAN SEPARATING → TESTED TO DESTRUCTION
2. TRIALS 1-8 ALL GAVE FILM TEAR HEATSEAL BONDS WELL WITHIN SPECIFICATION
3. TRIALS 9-16 ALL DELAMINATED ON HEATSEAL TEST HOWEVER BONDS WOULD BE ADEQUATE
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SIDLAW PACKAGING EXPERIMENT

STRENGTH

RUN NUMBER

1  2  3  4  5  6  7  8  9  10  11  12  13  14  15  16
LOG MEANS V. LOG SDEVS PLOT
SCATTER PLOT, SSZ=16

Y = -1.0926 + 1.1745 \times X

CONCLUSION
Transformation necessary.
Take logs of data.
Figure 3: Log of data

SIDLAW

LN of each test set

TEST NUMBER
ANALYSIS OF FACTORIAL EXPERIMENT: DTISIDEMF

ANALYSIS OF MEANS

\[ \mu = 5.6017, \sigma = 0.23971 \]

CONCLUSION

A extremely significant
B/D somewhat
Others not significant
ANALYSIS OF FACTORIAL EXPERIMENT: DTISIDLAW

FACTOR INTERACTIONS

\[ \mu = 0.23426, \sigma = 0 \]

CONCLUSIONS

E/F MOST SIGNIFICANT
A/G NEXT MOST SIGNIFICANT
OTHERS NOT SIGNIFICANT.
ANALYSIS OF FACTORIAL EXPERIMENT: DTISIDHOLD

ANALYSIS OF MEANS
\[ \mu = 5.9145, \sigma = 0.25449 \]

CONCLUSION (MEANS):

NOTHING SIGNIFICANT WITHIN HOLDENS MATERIAL
Appendix 5

Bond strength for confirmatory trials, also showing tester variation and remarks from Sidlaw Packaging - Colodense
## CONFIRMATORY TRIAL AT SIDLAW PACKAGING 26/6/95

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| MEAN        | 118           | 140                   | 120      | 202             |          |
| STD DEV     | 53            | 56                    | 50       | 76              |          |

| 2           | 1             | 98                    | 107       | G               | 72       | 88         | G            |
| 2           | 124           | 165                   | M         | 142            | 140      | M/G         |
| 3           | 88            | 111                   | M         | 106            | 128      | M          |
| 4           | 91            | 111                   | M/G       | 101            | 134      | M          |
| 5           | 106           | 128                   | M         | 69             | 88       | G          |
| 6           | 103           | 128                   | M         | 128            | 128      | M          |
| 7           | 76            | 105                   | G         | 128            | 128      | M          |
| 8           | 116           | 169                   | M         | 128            | 128      | M          |
| 9           | 138           | 157                   | M         | 128            | 128      | M          |
| 10          | 79            | 111                   | M/G       | 128            | 128      | M          |

| MEAN        | 102           | 131                   | 98       | 116             |          |
| STD DEV     | 20            | 29                    | 30       | 26              |          |

| 3           | 1             | 67                    | 111       | M               | 71       | 75         | M            |
| 2           | 93            | 111                   | M         | 56             | 70       | G          |
| 3           | 78            | 105                   | M         | 57             | 70       | G          |
| 4           | 93            | 109                   | M         | 131            | 152      | M          |
| 6           | 106           | 117                   | M         | 81             | 99       | M/G         |
| 6           | 110           | 134                   | M         | 134            | 134      | M/G         |
| 7           | 80            | 93                    | M/G       | 93             | 93       | M/G         |
| 8           | 68            | 88                    | M         | 88             | 88       | M          |
| 9           | 133           | 157                   | M         | 133            | 157      | M          |
| 10          | 87            | 99                    | M/G       | 133            | 157      | M          |

| MEAN        | 92            | 112                   | 90       | 113             |          |
| STD DEV     | 20            | 20                    | 30       | 48              |          |

| 4           | 1             | 100                   | 123       | G               | 54       | 54         | G            |
| 2           | 107           | 128                   | M         | 62             | 70       | M          |
| 3           | 90            | 117                   | M         | 134            | 169      | M/G         |
| 4           | 72            | 140                   | M/G       | 88             | 123      | M          |
| 5           | 117           | 140                   | M         | 129            | 158      | M/G         |
| 6           | 112           | 158                   | M         | 158            | 158      | M/G         |
| 7           | 110           | 123                   | M/G       | 123            | 123      | M/G         |
| 8           | 128           | 161                   | M         | 128            | 161      | M/G         |
| 9           | 128           | 146                   | M         | 128            | 146      | M/G         |
| 10          | 129           | 146                   | M/G       | 129            | 146      | M/G         |

<p>| MEAN        | 111           | 137                   | 93       | 117             |          |
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<th>MEAN</th>
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Fax Transmission

To: Susan Tilbury  
Company: PIRA International  
Fax number: 01372 802245  
cc: Graham Hobbs  
From: Ged Sweeney  
Date: April 18, 1996  
No. of pages incl. this one: 2 (two)

If you do not receive all pages, please contact:
++ 44 (0) 117 975 3200 (Tele)  
++ 44 (0) 117 975 3301 (Fax)

Subject: Confirmatory Trials on Metallised OPP

Following the confirmatory trial on March 28 where 20μm metallised OPP was laminated to underside printed 20μm OPP, the following results were obtained:

<table>
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<tr>
<th>Sample</th>
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<th>Interply</th>
<th>Seal Strength</th>
<th>Interply</th>
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<td>Mean</td>
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<td>498 FT</td>
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</table>

Results are in g/25mm. Seal strengths made at 130°C/14psi/1 sec. Interply results are obtained in the same way as described in your previous report. Hope they are of use!
April 18, 1996

Dear Susan

Further to our recent conversations I am sending you this note highlighting the benefits of the lamination trials, we believe we obtained.

The work we carried out enabled us to focus on the problem of variable interply strengths on the metallised OPP//clear OPP laminates. Another useful aspect of the work was that it showed that certain production variables did not have the effect on interply strength that intuitively we would have expected.

The trial procedure was useful because it should be possible for us to utilise the procedure to make improvements in other areas of our manufacturing process. I found it a useful exercise in obtaining co-operation between the laminator operators and the production and technical management teams. However, most importantly we have found a process from which we obtain improved interply strengths on OPP//metallised OPP laminates.

Yours sincerely

Ged Sweeney - Technical and Quality Assistant - Sidlaw Packaging, Bristol