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Confidential report for:

Campden BRI

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Report on:

Investigation of a Foil Pouch

Work performed by Campden BRI (Chipping Campden) Limited
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[DC: R-AC-9-5-15: 03/18 (27) : R/KAH]



SAMPLE DETAILS

Complaint Reference: 1254841
Purchase Order No.: JD12378GUY897

SAMPLE INFORMATION

Date sample(s) received : 23rd March 2018
Packaging : Original product packaging
Storage conditions : Ambient temperature
Date(s) sample(s) examined : 27th March – 11th April 2018

METHODS AND REFERENCES

Analyst reference : ZL
Method reference(s) : TES-AC-192 : Light Stereomicroscopy
TES-AC-193 : Compound Light Microscopy
TES-AC-198 : Scanning Electron Microscopy and X-ray
Microanalysis
TES-AC-385 : Fourier Transform Infrared Spectroscopy
Deviations from the
method reference(s) : None

INTRODUCTION

A foil pouch was received by the microscopy section for investigation of flexion creases to determine if the pouch integrity had been compromised. The client also requested the determination of the number of layers, thickness of these layers and material identification of the layers. The pouch and an example of a flexion crease were photographed as received and can be referred to in Plates 1 and 2.

The client had provided a specification for comparison against the foil pouch. The specification stated that the pouch should be comprised of four layers; one layer of polyethylene (PE) 40µm, one layer of aluminium 20µm, one layer of polyethylene (PE) 20µm and one layer of polyethylene terephthalate (PET) 40µm (plus adhesives).

RESULTS

Flexion Creases

The flexion creases were examined visually and under a stereo-microscope. There were no obvious signs of delamination at this stage.

Sections through the flexion creases were taken for imaging and examination using a scanning electron microscope, here it was possible to see delamination between one of the external layers and the next internal layer (Plate 3).

Number and Thickness of Layers

Multiple cryostat sections were taken of the pouch for examination under a compound microscope to determine the number of layers. Here it was possible to see that the sections were comprised of four layers (labelled 1-4, Plate 4), three of which appeared to be polymer based and a single optically dense layer, suggestive of metal.

Multiple measurements of the layers were taken and an average measurement was calculated per layer. An average measurement was used as measurements can vary slightly between sections, due to sample preparation, natural raw material variation and adhesive layers present. The average measurements were layer one 35µm, layer two 19µm, layer three 19µm and layer four 36µm. The number of layers in the pouch matched that of the specification, and the average measurements taken were close to the specification.

Identification of Layers

Further sections of the pouch were taken for imaging using FT-IR microscopy and identification of the layers using FT-IR spectroscopy. The layers were identified as follows; layer one polyethylene, layer two unidentified (suspected metallic layer), layer three polyethylene and layer four polyethylene terephthalate. These identifications matched those of the specification provided.

X-ray mapping was performed on further sections of the pouch for identification of layer two (suspected metallic layer); this layer was identified as aluminium (Al).

A summary of results can be referred to in Table 1.

Table 1

	<u>Layer one</u>	<u>Layer two</u>	<u>Layer three</u>	<u>Layer four</u>	<u>Plate/Figure</u>
Complaint pouch	Polyethylene 35µm	Aluminium 19µm	Polyethylene 19µm	Polyethylene terephthalate 36µm	Plate 4 Figures 1-3
Specification	Polyethylene 40µm	Aluminium 20µm	Polyethylene 20µm	Polyethylene terephthalate 40µm	N/A

CONCLUSIONS

It was concluded that the complaint pouch was comprised of four layers; polyethylene 35µm, aluminium 19µm, polyethylene 19µm and polyethylene terephthalate 36µm. The identification of the materials in the pouch matched those in the specification provided by the client.

The measurements of the pouch were very similar to those in the specification. The measurements did differ slightly, however measurements would be expected to show some variation due to sample preparation, natural raw material variation and adhesive layers present.

The flexion creases examined showed delamination between the outer layer of polyethylene and the inner aluminium layer. The specific cause for the delamination could not be confirmed.

Sample

PLATE 1 Shows the selected pouch photographed as received against a millimetre-squared background



PLATE 2 Shows an example of a flexion crease seen on the selected pouch viewed under a stereo microscope.



PLATE 3 Shows an example of a cross section through a flexion crease viewed side on using a scanning electron microscope. Note the delamination (highlighted) between the external layer and the next internal layer

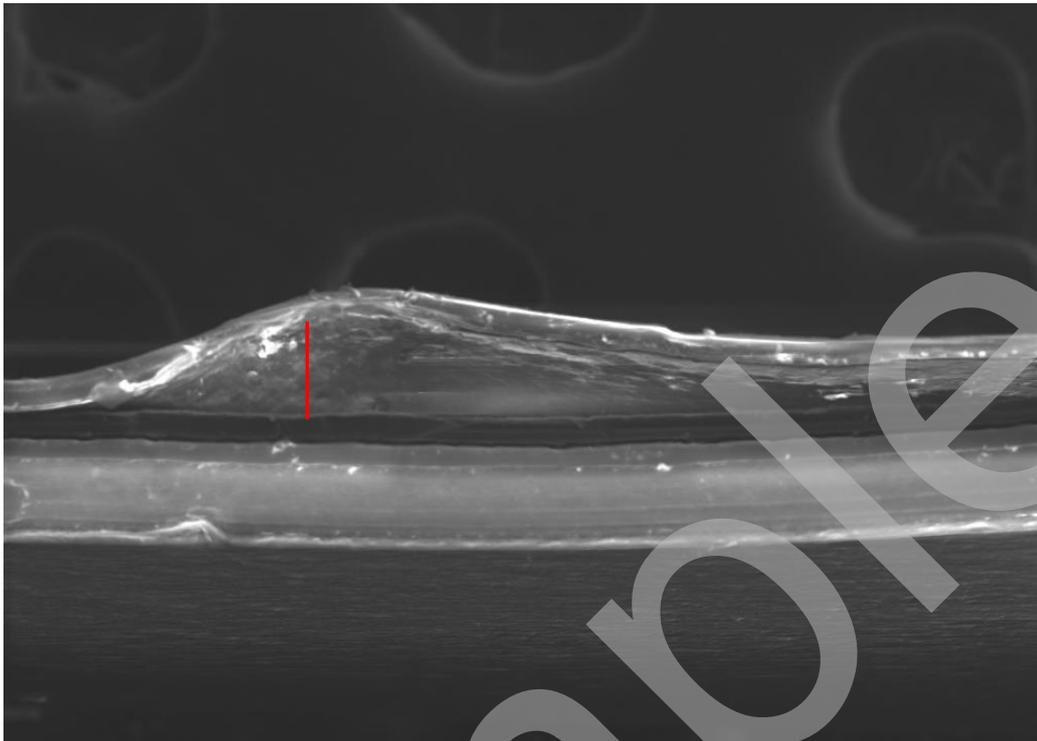
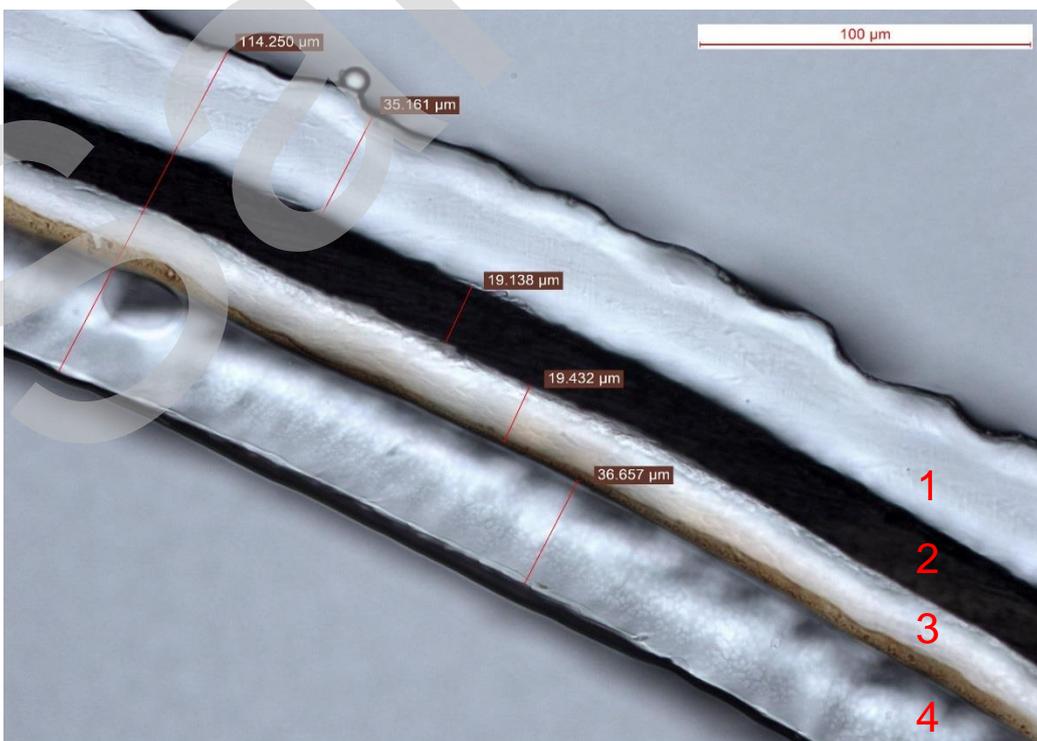
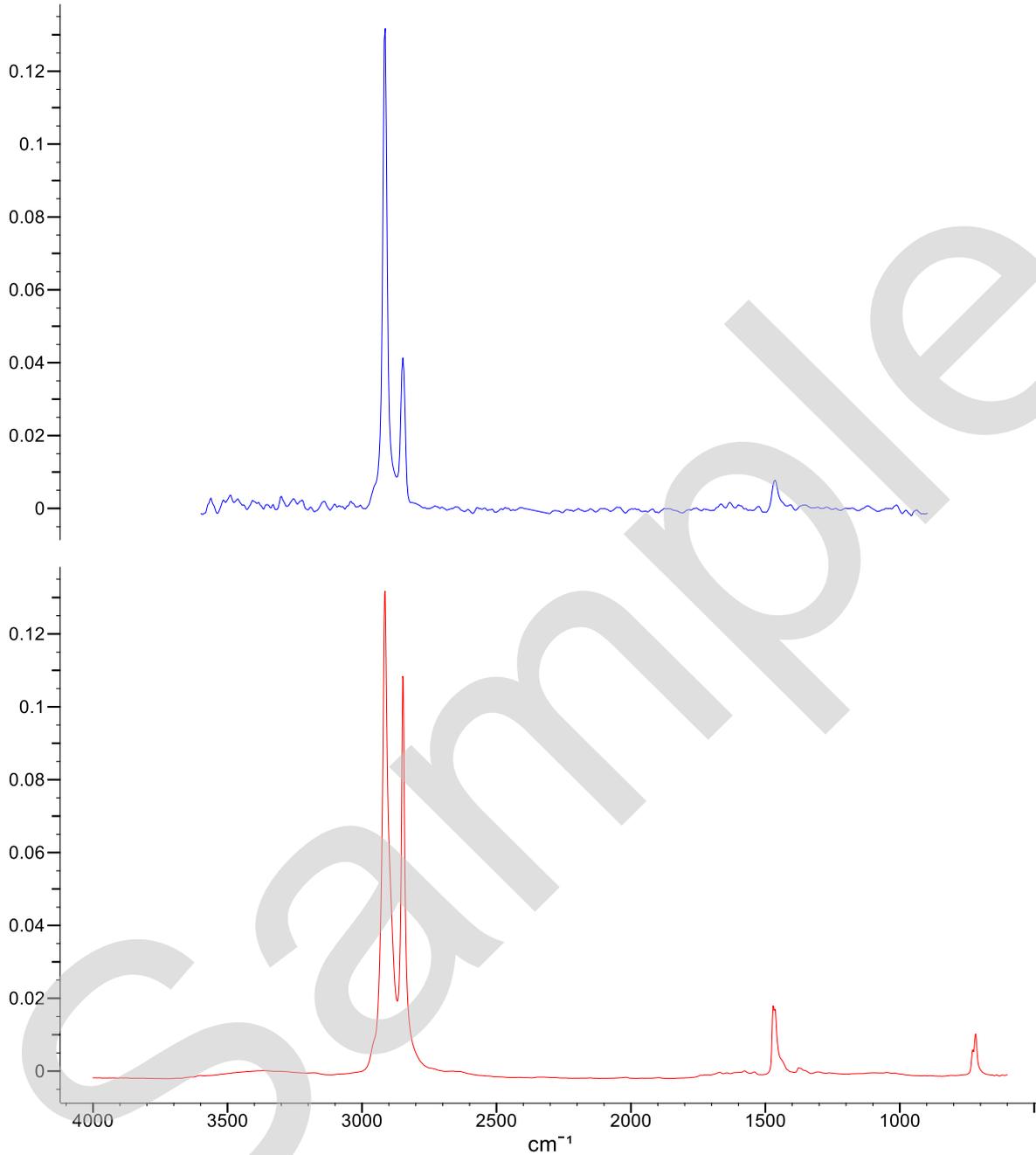


PLATE 4 Shows an example of a section taken from the pouch viewed under a compound microscope. Note the presence of four layers (labelled 1-4) with added measurements. The optical dense layer (layer two) is suggestive of a metallic layer

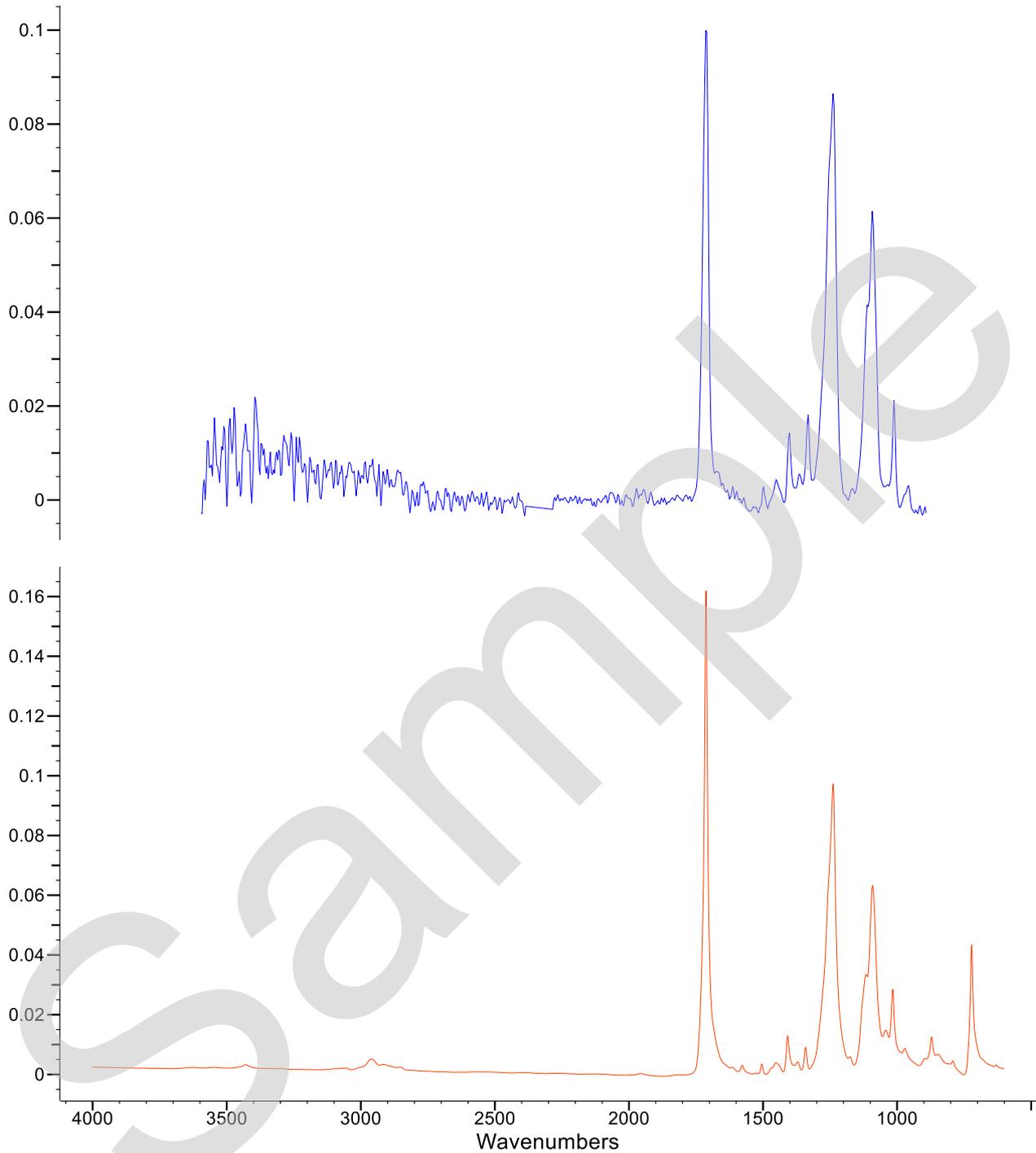


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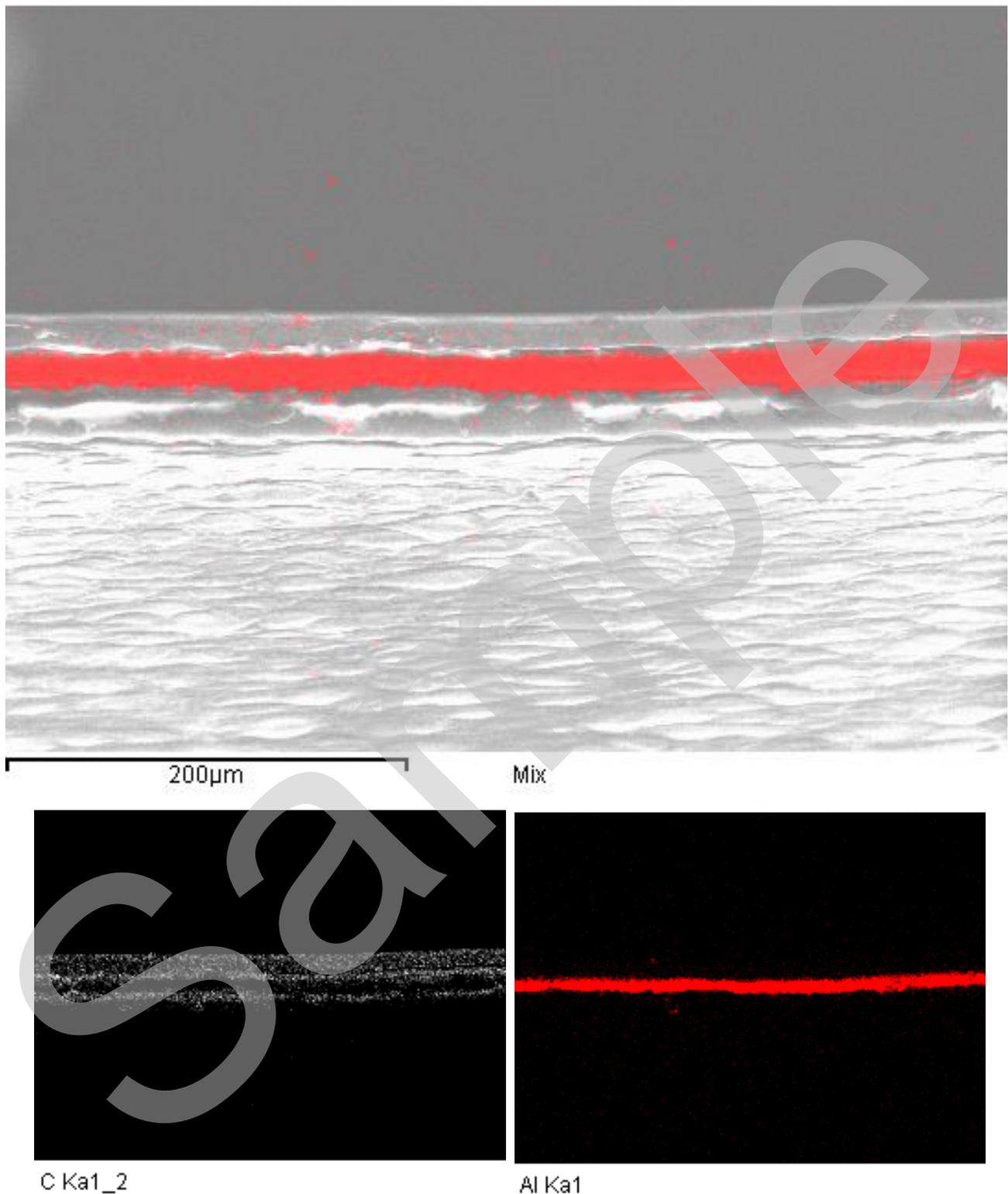
Top Spectrum: Representative spectrum for layers 1 and 3
Bottom Spectrum: Polyethylene

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Top Spectrum: Layer 4
Bottom Spectrum: Polyethylene terephthalate (PET)

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The top image is an electron micrograph of a section of the selected pouch viewed edge on. The below images are scans of the pouch showing the presence and distribution of the elements (brighter white areas and Al highlighted red). Note the aluminium (Al) is localised to a single layer which corresponded to layer two (highlighted in red over the top electron micrograph image). The carbon (C) present would correspond to the polymer layers.