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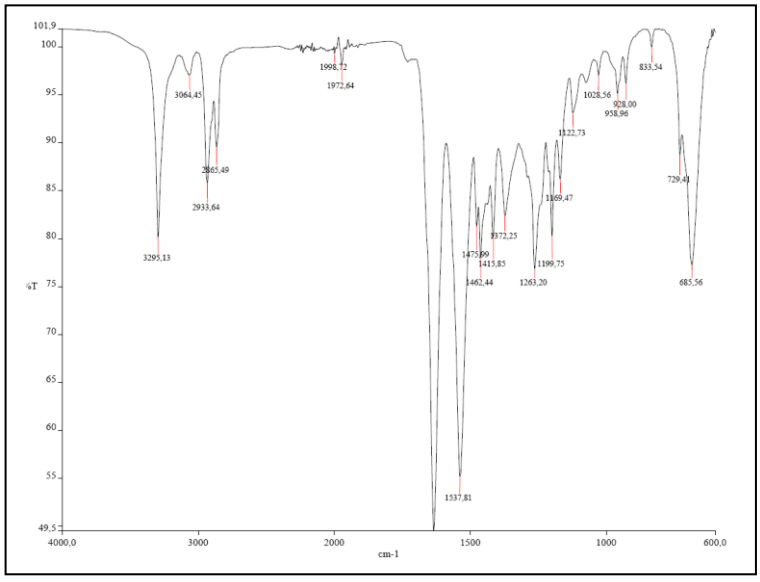
Report of an interlaboratory comparison from the European Reference Laboratory for Food Contact Materials:

ILC002 2013 –Identification of Polymeric Materials



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Report of the interlaboratory comparison

ILC002 2013 - Comparative testing (PT) on the Identification of Polymeric Materials of Unknown Nature

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1. Summary

Interlaboratory comparisons (ILCs) are an important element of laboratory quality assurance and allow individual laboratories to compare their analytical results with those from other laboratories while providing them objective standards to perform against. One of the core duties of the European Reference Laboratory is to organize such ILCs, as stipulated in Regulation (EC) No 882/2004 of the European Parliament and of the Council [1]. The European Reference Laboratory for Food Contact Material (EURL-FCM) organized in 2013 a number of ILCs for its network of appointed National Reference Laboratories (NRLs).The scope of the 2013 ILCs were agreed in a plenary meeting of June 2012. The ILC 002 2013 was directed towards evaluation of the laboratory performance on the identification of polymeric materials with unknown nature. This report present the work and results obtained in this exercise which targeted 12 monolayer materials. The results of the identification of such materials were fully satisfactory. In addition a scoping exercise was conducted on multilayer foils, which highlighted needs for guidance for the separation and identification of layers within multilayer materials.

2. Introduction

The range of plastics polymers encountered for food contact materials is vast. The correct identification of a plastic material is a critical first step for enforcement purposes. Several recent Regulations such as Regulation EC No 284/2011 on polyamide and melamine articles [2] or regulation 321/2011 restricting Bisphenol A [3] use in plastic infant feeding bottles required the ability to correctly determine the polymer type.

Materials that have traditionally been used in food packaging and food contact materials include glass, metals, paper and paperboards, and plastics [4]. Today's food packages often combine several materials to exploit each material's functional properties. Multiple types of plastics are being used as materials for packaging food, including polyolefin, polyester, polyvinyl chloride, polyvinylidene chloride, polystyrene, polyamide, and ethylene vinyl alcohol, among other. Although more than 30 types of plastics have been used as packaging materials [5], polyolefins and polyesters are the most common.

Polyolefins represent a collective term for polyethylene (PE) and polypropylene (PP), the 2 most widely used plastics in food packaging, and other less popular olefins polymers. Both, PE and PP possess a combination of properties, including flexibility, strength, lightness, stability, moisture and chemical resistance, and easy processability, and are well suited for recycling and reuse.

- PE is made by addition polymerization of ethylene. There are two categories of PE: high density (HDPE) and low density (LDPE). HDPE is stiff, strong, though, resistant to chemicals and moisture, permeable to gas and easy to process. It is used to make for example bottles, boxes, tubes and bags. LDPE is flexible, strong, tough, easy to seal and resistant to moisture. Since LDPE is transparent, it is used in film applications and in applications were heat sealing is necessary. Uses for this polymer include bread and frozen food bags, flexible lids and squeezable bottles.
- PP is harder, denser and more transparent than PE; it has a good resistance to chemicals and is effective as moisture barrier. Its high melting point (160 ºC) makes it suitable for applications where thermal resistance is required, such as hot fill and microwaveable packaging. Uses of PP include yogurt containers and margarine tubes. When used with an oxygen barrier such as ethylene vinyl alcohol or polyvinylidene chloride, PP provides the strength and moisture barrier for catsup and salad dressing bottles.

Polyesters represent a family of polymers including polyethylene terephthalate (PET or PETE), polycarbonate (PC), and polyethylene naphthalene (PEN). They are condensation polymers

formed from ester monomers that result from the reaction between carboxylic acid and alcohol. The most commonly used polyester in food packaging is PET.

- PET provides a good barrier to gases (oxygen and carbon dioxide) and moisture. It also has good resistance to heat, mineral oils, solvents, and acids, but not to alkali. The main reasons of its popularity are its glass-like transparency, adequate gas barrier for retention of carbonation, light weight. Uses of PET include beverages and mineral water, containers (bottles, jars, tubes), semi rigid sheets for thermoforming (trays and blisters), and thin oriented films (bags and snack food wrappers).
- PC is formed by polymerization of a sodium salt of bisphenol A with carbonyl dichloride. Clear, heat resistant and durable, it can be used for large returnable/refillable water bottles and or articles that can be sterilized.
- PEN is a condensation polymer of dimethyl naphthalene dicarboxylate and ethylene glycol. It is a newer member of the polyester family with high performance because of its high glass transition temperature. It possesses high barrier properties to carbon dioxide, oxygen and moisture. In addition it presents high thermal resistance allowing hot refills, rewashing, and reuse. This polymer can be used for bottles (but is more expensive than PET).

Polyvinyl chloride (PVC) is an addition polymer of vinyl chloride. It is heavy, stiff, ductile and a medium-strong, amorphous, transparent material. It has excellent resistance to chemicals (acid and alkali), grease, and oil. Its uses include bottles and packaging films. PVC can be transformed into materials with wide range of flexibility with the addition of plasticizers such as adipates, citrates, and phosphates.

Polyvinylidene chloride (PVdC) is an addition polymer of vinylidene chloride. It is heat sealable and serves as barrier to water, gases, and oil. It is used in flexible packaging as a monolayer film, as coating or part of a co-extruded product. Uses of PVdC include packaging of poultry, cured meats, cheese, snack foods, tea, and coffee. It is also used in hot filling, retorting, low temperature storage and modified atmosphere.

Polystyrene (PS), is an addition polymer of styrene. It is clear, hard, and brittle. It can be mono-extruded, co-extruded with other plastics, injection molded or foamed to produce a range of products. Foaming produces an opaque, rigid, lightweight material with impact protection and thermal insulation properties. Uses of PS include protective packaging such as egg cartons, containers, disposable plastic tableware, lids, cups, plates, bottles and food trays.

Polyamide (PA), commonly known as Nylon (DuPont), are formed by condensation reaction between diamide and diacid; they are polymers in which the repeating units are held together by amide links. Different types of polyamides are characterized by the number of carbons in the originating monomer. For example, PA6 (Nylon 6) has 6 carbons and is typically used in packaging. Nylon also offers good chemical resistance, toughness and low gas permeability,

Ethylene vinyl alcohol (EVOH) is a copolymer of ethylene and vinyl alcohol. It is an excellent barrier to oil, fat and oxygen. However, EVOH is moisture sensitive and is thus mostly used in multi-layered co-extruded films in situation where is not in direct contact with the food.

Melamine, (1,3,5-Triazine-2,4,6-triamine) is a high-production volume chemical that can be used in a variety of food contact materials. It is a common monomer in the manufacturing of plastic materials (melamine–formaldehyde plastics) for food contact. Melamine–formaldehyde is well suited for tableware products such as cups, bowls, plates or utensils because of its hardness, heat resistance and general stability during repeated use.

3. Scope

The scope of this ILC was a Proficiency Testing in which the participants had to identify correctly the polymer type for a range of plastics that may be used as food contact material plastics. The exercise consisted in the analysis of 12 samples (monolayers). In addition there was a scoping trial on 3 samples that were multilayers. Participants were invited to report their result in a table indicating the technique used and justifying their answers.

4. Time frame

Fifteen samples overall of different plastic types were prepared by the EURL-FCM in May 2013. Invitation letters were sent to the laboratories on 17th May 2013 (Annex 1).
The samples –wrapped and codified- were dispatched to the participants on 20th May 2013 together with a letter (Annex 2), a letter of confirmation of receipt (Annex 3), Instructions for compilation of results (Annex 4). The participants were asked to fill in a letter of confirmation of the receipt of the samples (Annex 3), and were also asked to complete the Table Template for compilation of Results (Annex 5). The deadline for reporting was set to 25st of June 2013.

5. Test materials

The samples are given in table 1.

Table 1 – Test materials for the polymer identification exercise

CODE	IDENTIFICATION
PI A1	Low Density Polyethylene (LDPE) (Monolayer)
PI B2	High Density Polyethylene (HDPE) (Monolayer)
PI C3	Polypropylene (PP) (Monolayer)
PI D4	Polyethylene terephthalate Film (PET) (Monolayer)
PI E5	Polyamide 6 Film (PA 6) (Monolayer)
PI F6	Polyvinyl chloride (PVC) (Monolayer)
PI H7	Polyvinyl chloride (PVC) (Monolayer)
PI I8	Polyethylene terephthalate (PET) (Monolayer)
PI J9	Polystyrene (PS) (Monolayer)
PI K10	Polyvinylidene chloride (PVDC) (Monolayer)
PI M12	PET/Al-foil/PA/PE (Multilayer)+ PE (Monolayer)
PI N13	PE/PET-Al/PE (Multilayer)+ PE (Monolayer)
PI O14	PE/PET-Al/PE (Multilayer)+ PE (Monolayer)
PI P15	Polycarbonate (PC) (Monolayer)
PI Q16	Melamine (Monolayer)

Figure 1 shows the photos of each multilayer, M12, N13 and O14. The multilayer itself (shiny metal layer) and the extra layers can be seen.

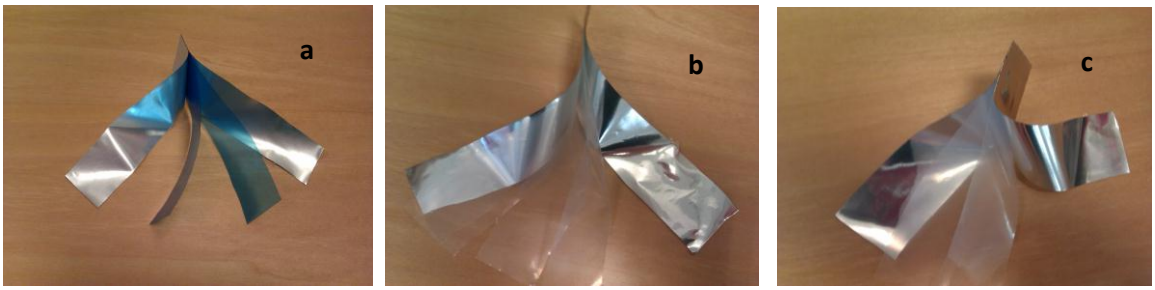


Figure 1 – Photos of the multilayers samples: a) M12; b) N13; c) O14

The sample kits were dispatched to the participants as follows:

- Test materials (each material individually wrapped in aluminum foil and labelled).
- Letters:
- Letter of confirmation of receipt (Sample Receipt ILC021 2013);
- Letter with instructions for the compilation of results (Instructions Excel ILC002 2013);
- Table to complete with the results

6. Instructions to participants

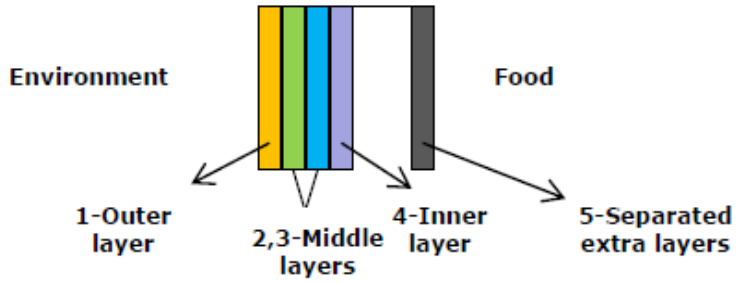
Instructions were given to all participants in the letters that accompanied the sample (Annex 4). Laboratories were asked to report with sufficient specific information for each sample. Participants were free to use their own methods and technique that they considered the most appropriate for the task.

7. Evaluation of Results

The standard ISO/IEC 17043:2010 [6] contains in section B.3.2 - performance for qualitative and semi-quantitative results- descriptions to assess qualitative data (also called categorical data) by comparing the participants' result with assigned values. If they are identical, then the performance is "acceptable". If they are not identical, then expert judgment is needed to determine whether the results are fit for their intended purposes. In some situations, the proficiency testing provider may review the results from participants and determine that the proficiency testing item was not suitable for evaluation, or that the assigned value was not correct. For semi-quantitative (ordinal) results the techniques used for qualitative data are appropriate and numbers are given e.g. 1=Poor; 2=Unsatisfactory; 3=Satisfactory; 4=Good; 5=Very good. For these results it is more appropriate to use specific statistics designed for ordinal data [6,7] rather than common summary statistics. A principle applied is to list the distribution of results from all participants (or produce a graph), along with the number or percentage of results in each category, and to provide summary parameters, such as the mode (most common responses) and range (lowest and higher response). It may also be appropriate to evaluate results as acceptable based on closeness to the assigned value. In some situations, it may be appropriate to evaluate performance based on percentiles, (e.g. unacceptable would be the 5% of results farthest from the mode or farthest from the assigned value).

7.1 Scores

For this exercise the results had to be evaluated in a qualitative manner; thus it was necessary to give scores to each answer as previously described.
For monolayers, the score "very good" was deemed an absolutely correct answer and for this answer the score was 2 points. An "acceptable" score meant that the laboratories where able to identify the plastic, so the results were considered as positive, but the answer was not the most specific and for this answer the score was 1 point. A "poor" score was when a participant failed in the identification of the sample and 0 points were given..
For multilayers, it was a pilot exercise of much higher difficulty. The highest score was 5 points, 1 point for each layer, but for the middle layer or layers 2 points if the identification was correct. Therefore, the scores for multilayer were 1/2/1+1 for outer layer/middle layers/inner layer+ separated extra layers.



The scores "very good", "good" and "poor" were also applied to multilayers as follows:

- Very good: Score>4
- Acceptable: 4≤ score ≤ 2.5
- Poor: Score <2.5

7.1.1 Normalization of the results.

The score obtained for each sample was divided by the maximum score in order to have values from 0 to 1, for mono- and multilayers. Therefore, monolayers scores were divided by 2 while scores from multilayers were divided by 5.

7.1.2 Binary data and fixed threshold

The excel function IF(VALUE>=threshold,1,0) was used to describe the results under binary data (0/1) and to evaluate the performance of the participants with a certain threshold for determine whether the answer was correct or not. In this exercise a threshold of 0.7 was used. In this way, scores higher or the equal to 0.7 (70%) obtained 1 point, whereas those below obtained 0 points. This was a much more severe analysis but useful for the evaluation.
It should be noted that there are no protocols currently existing to tackle this type of qualitative data of complex nature (identification, rather than a binary positive/negative choice). Therefore the methods that were developed and presented in this report are the first of their kind and experimental in nature.

8. Results

8.1 Preliminary results from EURL-FCM

Before sending the samples to the participants, the EURL-FCM analyzed in blind the samples in order to check whether it was possible to identify them correctly and evaluate the difficulty of the test.
It should be noted that a number of materials that were in this exercise used came from a RTD project ((EU AIR Research Programme CT94-1025), which had been kindly donated to the Joint Research Centre upon completion of the project. These materials thus also had been subjected to identifications which had been reported[1].
For the identification, the techniques used were based on FTIR measurements using a Perkin Elmer-Spectrum One-FTIR Spectrometer, using attenuated total reflection technique in the region between 400 and 4000 cm^{-1} at room temperature.
Both monolayer and multilayers samples were analyzed directly, however multilayer metallic layers were treated with different solvents in order to separate the different layers.

[1] Safety and quality of food contact materials. Part 1: Evaluation of analytical strategies to introduce migration testing into good manufacturing practice. (2002) Feigenbaum, A.; Scholler, D.; Bouquant, J.; Ferrier, D.; Franz, R.; Lillemark, L.; Riquet, A. M.; Petersen, Jens Højslev; van Lierop, B.; Yagoubi, N., Food Additives and Contaminants, Vol. 19, No. 2, 2002, p. 184-201.

8.1.1 Analysis of Monolayers

Figures 2 and 3 show the spectra of samples A1 and B2, respectively. The main stretching vibrations for polyethylene appear at 2927 and 2848 cm^{-1}. The main bending mode of the -CH2 is located between 1475 cm^{-1} and 1463 cm^{-1}, in the case of HDPE these bands are located at 1472 and 1463 cm^{-1}; while for LDPE at 1464 cm^{-1}. It is difficult to differentiate both polyethylenes by FTIR. The main difference with LDPE is seen in 1380 cm^{-1} peak. For LDPE, the band region 1400- 1330 cm^{-1} consists in three peaks, while that for HDPE consists in two peaks. A peak at 1377 cm^{-1} is assigned to –CH3 group terminating the short and long-chain branching. If no individual peak is observed at 1377 cm^{-1}, then the material is HDPE. In the sample it could be seen that HDPE spectra presented a peak at 1368 and no peak at 1377 cm^{-1}. The LDPE sample presented three peaks in that region (shoulders) with the most intense is at 378 cm^{-1} [8].

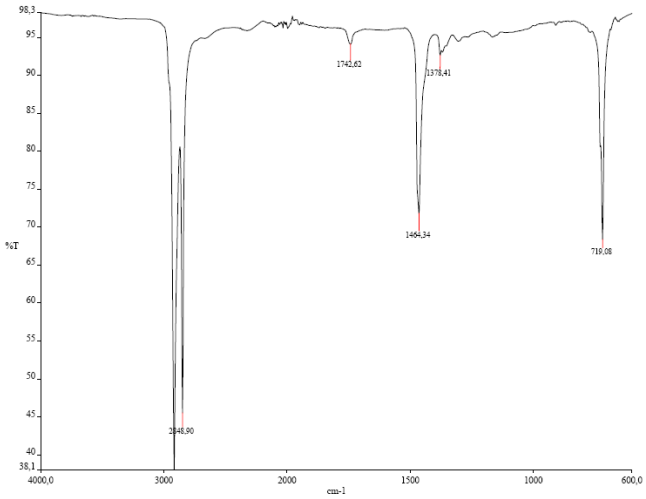


Figure 2 – FTIR spectrum of PI A1: LDPE –Low Density Polyethylene

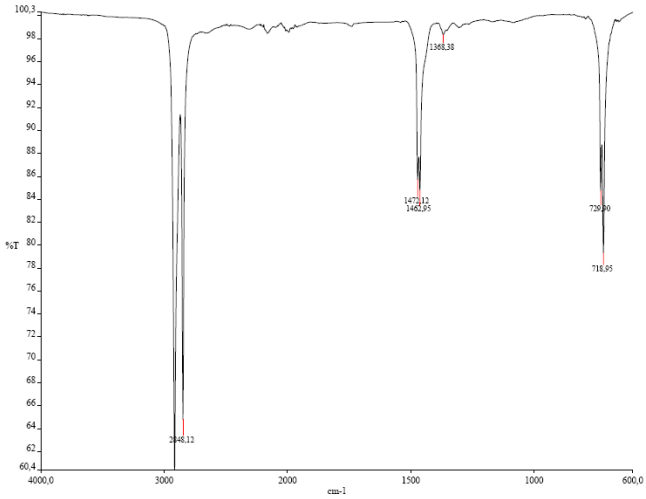


Figure 3 - FTIR spectrum of PI B2: HDPE – High Density Polyethylene

Figure 4 shows the spectrum of sample C3. It is possible to define this polymer as polypropylene. The FTIR spectrum of polypropylene presents a shoulder at 2875 cm^{-1}, and the asymmetric and symmetric in-plane C–H (–CH3) at 1455 and a shoulder at 1358 cm^{-1} confirms that it is a polypropylene. The peak at 1376 cm^{-1} is assigned to –CH3 group.

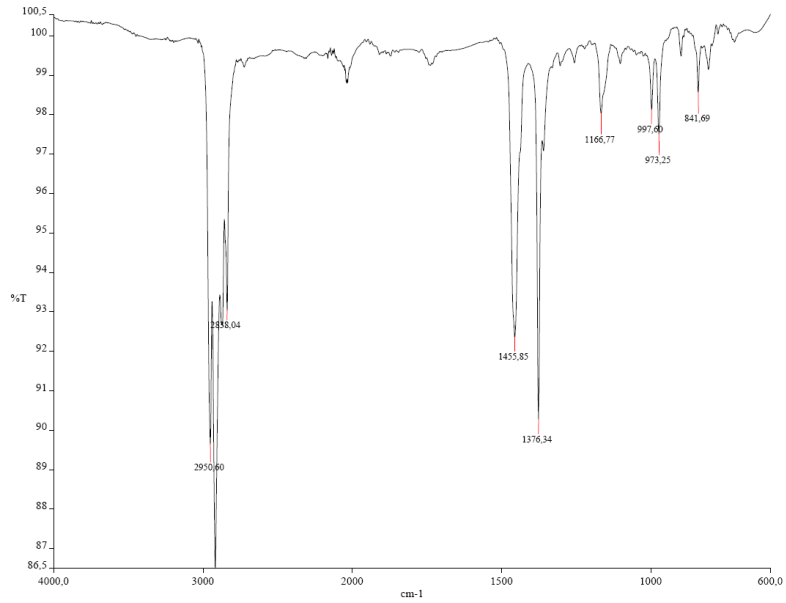


Figure 4 - FTIR spectrum of PI C3: PP – Polypropylene

Figure 5 shows the FTIR spectrum of sample D4, a polyethylene terephthalate – PET- sample, with its main characteristics bands listed in Table 2.

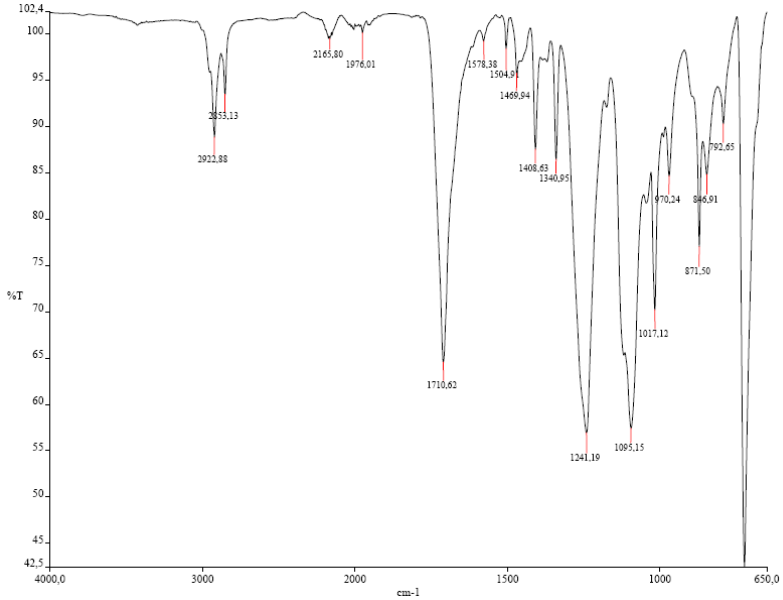


Figure 5 - FTIR spectrum of PI D4: PET film (polyethylene terephthalate)

Table 2 – Assignment of main Vibrations of PET

Wavenumber (cm^{-1})	Assignment
723	Out of plane of benzene group
845	CH_2 rocking of glycol
870	Out of plane of benzene group
970	C-O stretching of glycol
1017	In-plane vibration of benzene
Broad band 1090	Mainly due to ester C=O stretching
Broad band 1230	Mainly due to ester C=O stretching
Sharp 1710	C=O stretching (conjugated with the ring)
1340	CH_2 wagging of glycol
1370	CH_2 wagging of glycol
1408	Aromatic skeletal stretching band. Ring in-plane deformation

The spectrum of a polyamide sample is showed in Figure 6 and corresponds to sample E5. The characteristics bands in polyamides are at 3295 cm^{-1} the stretching of NH; at 3072 cm^{-1} due to the first overtone of amide II; the symmetrical and asymmetrical stretching of CH2 at 2865 and 2933 cm^{-1}, respectively; the band at 1640 cm^{-1} corresponds to the stretching of the amide carbonyl and 1537 cm^{-1} due to the amide II (NH) in plane bending + CN stretching [8].

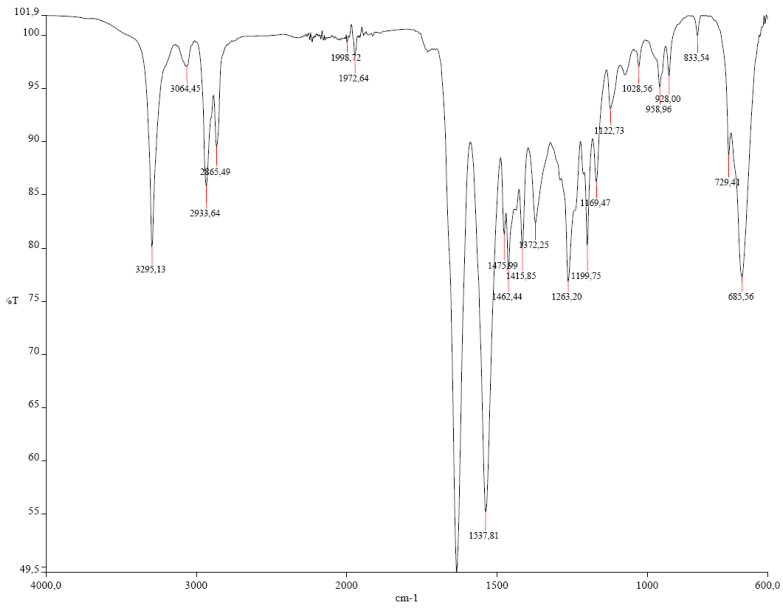


Figure 6 - FTIR spectrum of PI E5: PA film – PA6 (Polyamide 6)

In order to determine the type of polyamide the melting temperature was determined. For this, Differential Scanning Calorimetry analysis was performed using a DSC-TA Q20, at a heating rate of 20ºC/min from 30 ºC up to 280 ºC under nitrogen atmosphere. Figure 7 shows the thermogram of sample E5. The endothermic peak correspond to the melting process of the

polymer, and the maximum of the peak corresponds to the melting temperature (Tm) corresponding to the Tm of PA6 at 220 ºC [9].

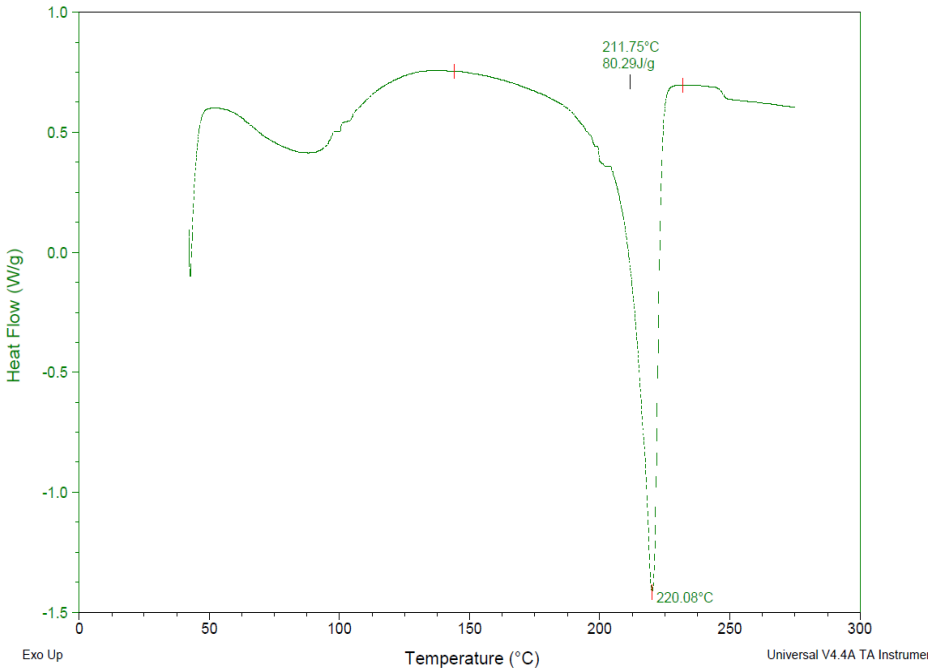


Figure 7 – DSC thermogram of sample E5

Sample F6 and H7 were both pieces of PVC bottles of different colors. Figure 8 and 9 show their spectra, respectively. The spectra for this material show bands at 2970, 2910 and 2843 cm^{-1} corresponding to –CH stretches, 1424 and 1439 cm^{-1}, 1424 cm^{-1}, due to CH3 rocking, 966 cm^{-1} skeletal vibrations and 700, 615 cm^{-1} due to C-Cl stretches and skeletal vibrations [8;10]. The peak at 1730 cm^{-1} could be due the presence of additives.

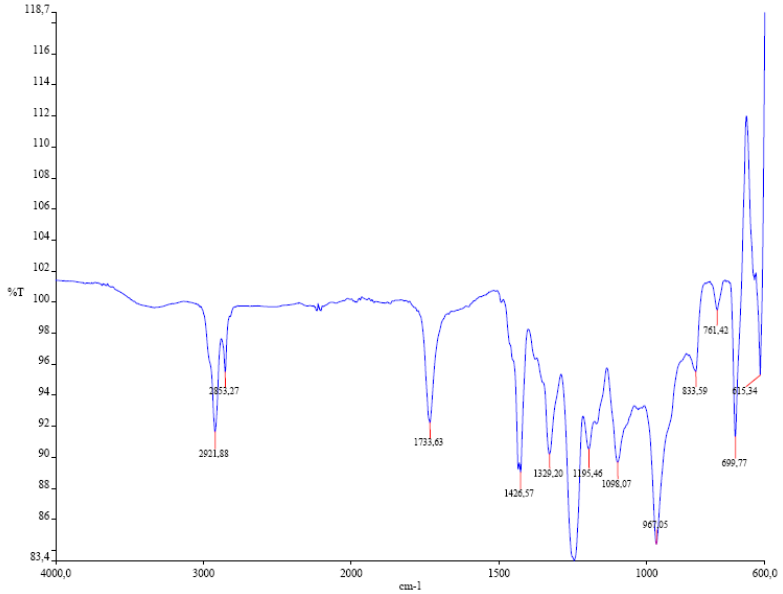


Figure 8 – Spectrum of sample PI F6: PVC bottle (Polyvinyl chloride)

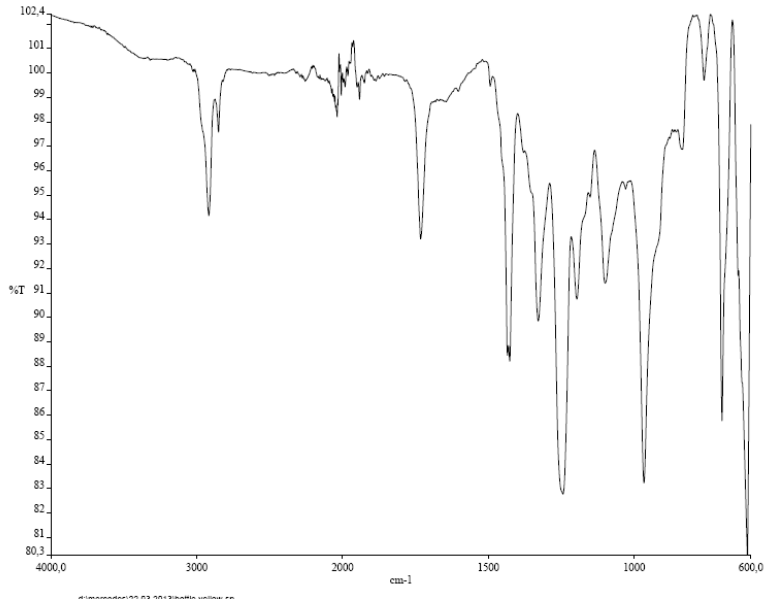


Figure 9 – Spectrum of sample PI H7: PVC bottle (Polyvinyl chloride)

The spectrum of sample I8 is showed in Figure 10. The same bands of sample D4 are observed indicating that the material is film of PET.

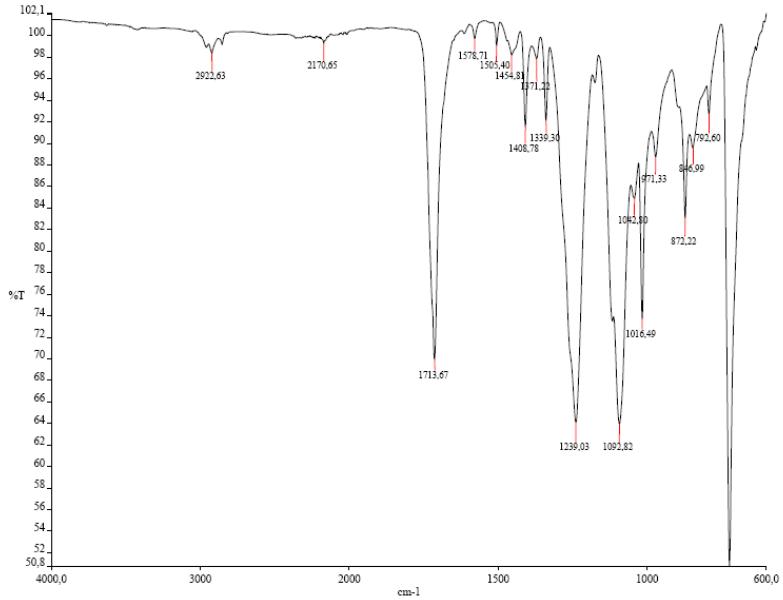


Figure 10 – FTIR spectrum of PI I8: PET bottle

Sample J9 was Polystyrene and its spectrum is showed in Figure 11. The normal absorption bands are the C-H stretching between 3110 cm^{-1} and 3000 cm^{-1} and the characteristic ring vibration bands at approximately 1601 and 1493 cm^{-1}. The spectrum also presents the band of monosubstitution at 1741 cm^{-1} and the out-of-plane deformation for monosubstitution 740 cm^{-1} and 700 cm^{-1} [8].

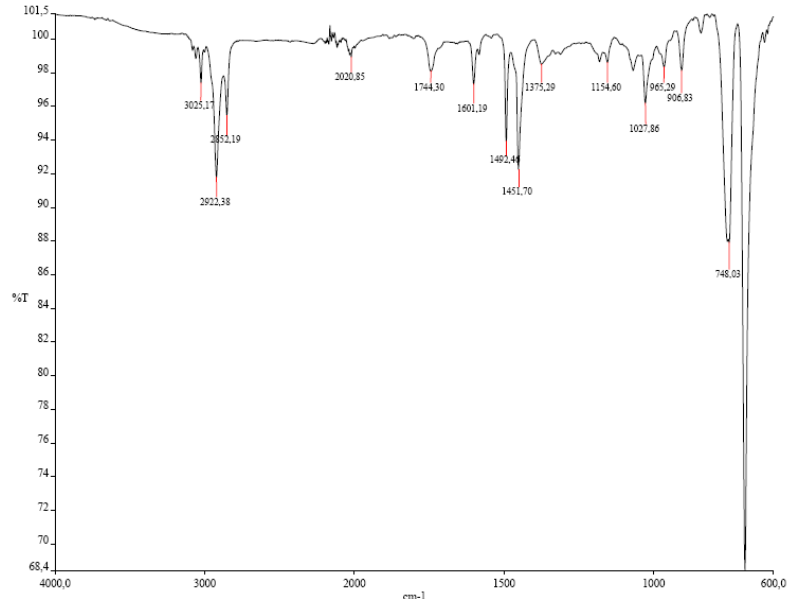


Figure 11 – Spectrum of sample PI J9: PS - Polystyrene

PVDC common peaks take the form of a doublet at 1068 and 1044 cm^{-1} and a band at 1405 cm^{-1} due to the CH2 bending. The C-Cl2 stretching vibrations are situated at 600 and 655 cm^{-1}. These bands were observed in the spectrum of sample K10 in Figure 12a, indicating that this material was PVdC. However, there was a peak at 1740 cm^{-1} corresponding to the C=O stretching, this band appeared due to the presence of additives, such as plasticizers. In order to check this, the film was immersed in acetonitrile during 24 h. In Figure 12b is the spectrum after extraction. It can be seen that the band corresponding to the C=O decreased in intensity due to the extraction of the additive by the acetonitrile.

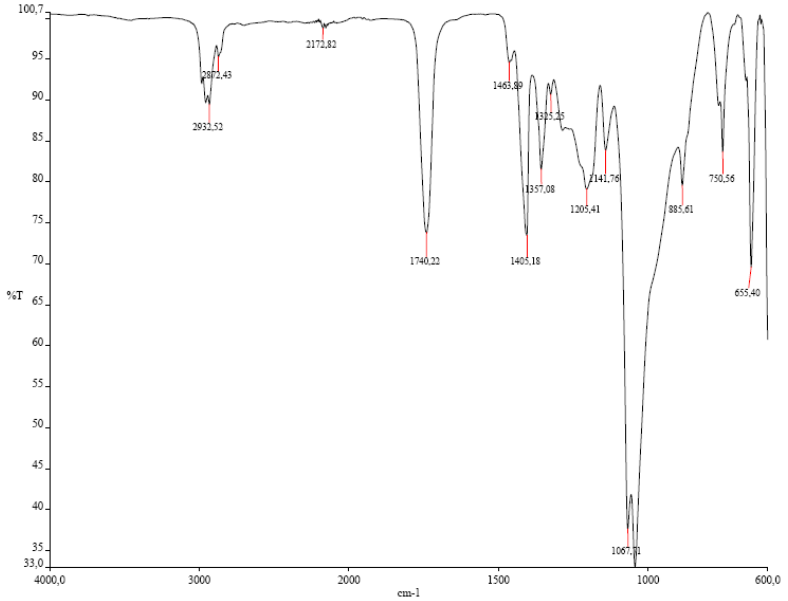


Figure 12a – FTIR spectrum of PI K10: PVdC - Polyvinylidene chloride

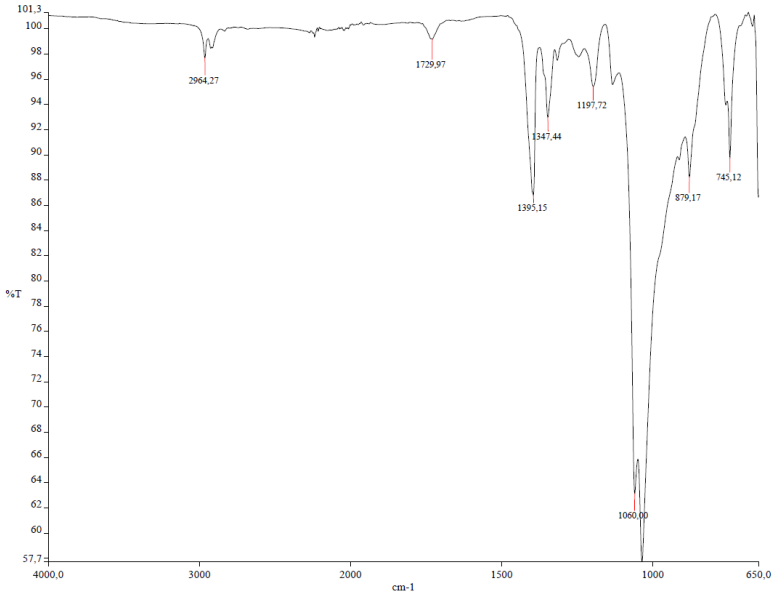


Figure 12b – FTIR spectrum of PI K10 after the extraction with acetonitrile

The spectrum in Figure 13 corresponds to sample P15 and the characteristic bands are from polycarbonate, which are listed in table 3.

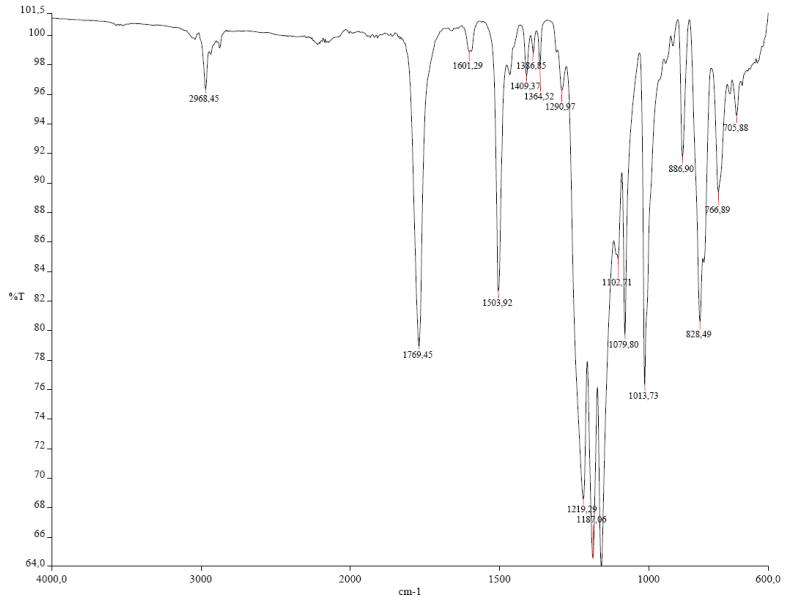


Figure 13 – FTIR spectrum of PI P15: PC - Polycarbonate

Table 3 – Assignment of main vibrations of PC

Wavenumber (cm^{-1})	Assignment
828	Out of plane deformation of (C-H)
1014	Stretching (sim) (O-C-O)
1080	Bending of (C-C-C)
1504	Ring stretching (C-C)
1770	C=O stretching
2970	Stretching (asymm) Methyl group (C-H)

Sample Q16 was Melamine and its spectrum is showed in Figure 14. The melamine spectrum shows absorbance in the region between 3330 cm^{-1} and 3190 cm^{-1} which corresponds to the stretching vibration of secondary amines. The bang at 1530 cm^{-1} corresponds to the C=N ring vibration, meanwhile the band at 1370 cm^{-1} correspond to the methylene C-H bending vibration. The band at 1230 cm^{-1} corresponds to the aliphatic methylene C-H bending vibration. The absorbance band at 1120 cm^{-1} corresponds to the C-O stretching vibration [11].

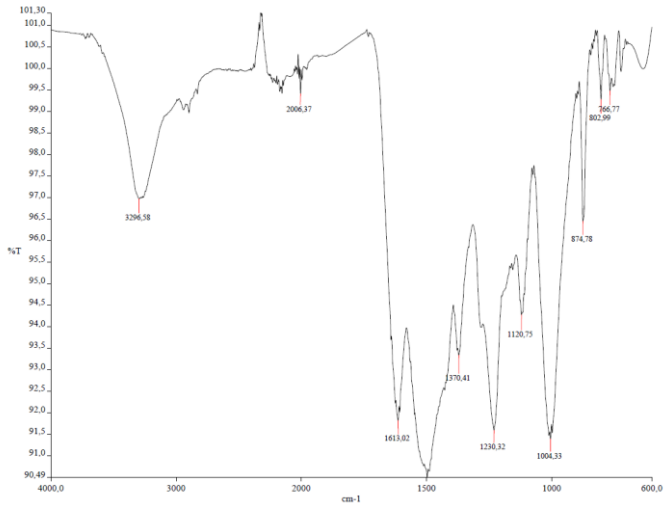


Figure 14 – FTIR spectrum of PI Q16 - Melamine

8.1.2 Analysis of Multilayers

Figure 15 shows two schematics of multilayer samples (M12, N13 and O14). They were composed by the main packaging layer (metallized) that was the multilayer itself ("external multilayer") and separated layers composed by a monolayer polymer. The external multilayer was analyzed directly to characterize both external sides, afterwards several treatments were applied in order to separate each layer. Solvents used for the layers separation as follows:

- Hydrogen chloride (HCl): dissolves the aluminum
- Xylene (hot): dissolves the polyethylene. Also, it is possible to use toluene for this purpose.
- Formic acid: dissolves polyamide.

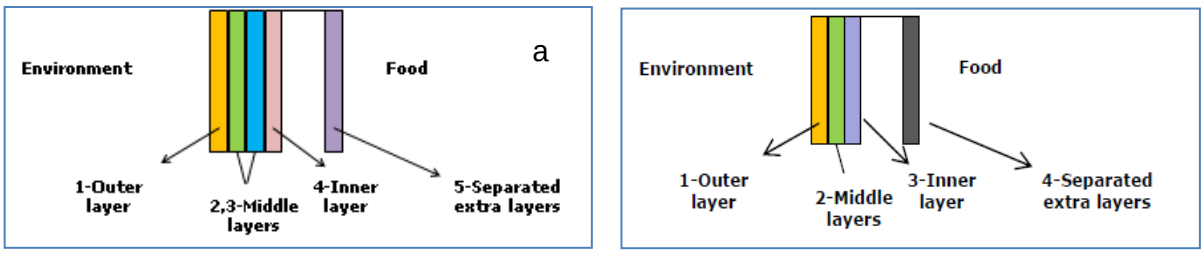


Figure 15 – a) Schematics of the multilayer M12; b) Schematics of the multilayer N13 and O14.

PI M12 - PET/Al-foil/PA/PE (Multilayer) + PE (Monolayer)

The layers of the multilayer film were separated with concentrated HCl, boiling xylene and formic acid as illustrated in the following figures.

1) No treatment
The figures below represent the sample M12 without any treatment

PET/Al-foil/PA/PE+PE

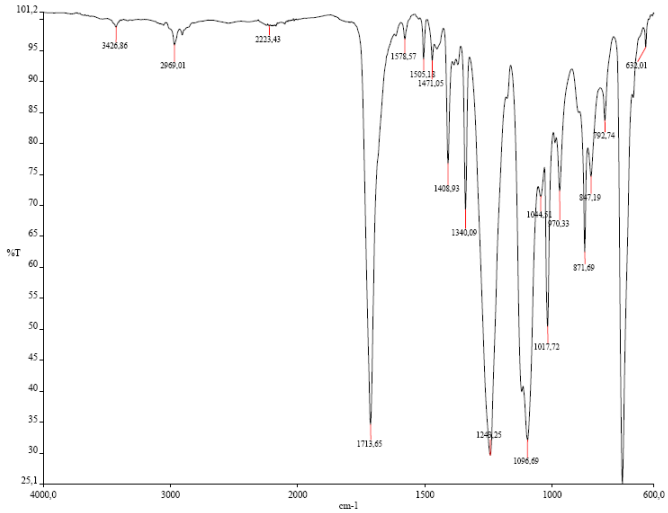


Figure 16 – Spectra of the outer layer (Layer 1 in Figure 15a) in M12

PET/Al-foil/PA/PE+PE

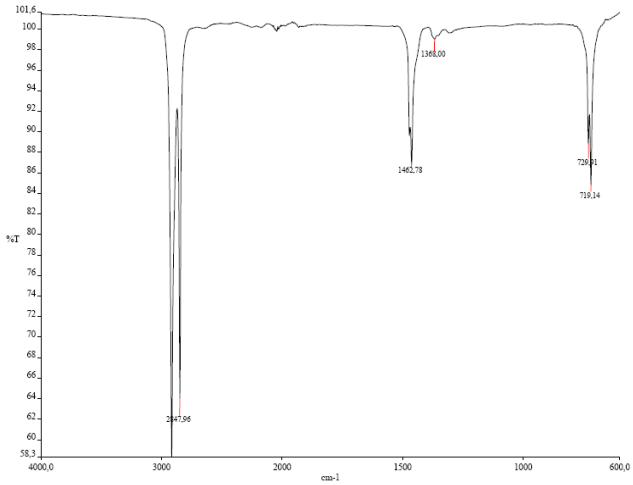


Figure 17 – Spectra of the inner layer (Layer 4 in Figure 15a) in M12

PET/Al-foil/PA/PE+PE

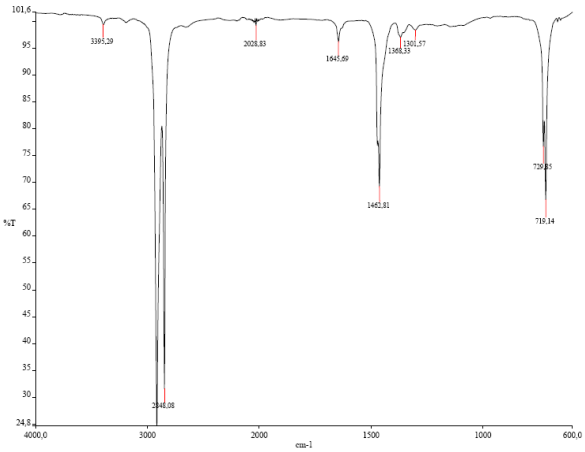


Figure 18 – Spectra of the separated layer (Layer 5 in Figure 15a) in M12

2) Immersion in xylene and heating during 10 minutes

By immersing the multilayer in hot xylene, the PE starts to be dissolved and this treatment allows scratching the PE to remove it from the surface. It becomes possible to identify the PA layer (spectrum in Figure 19) from the inner layers.

PET/Al-foil/PA/PE+PE

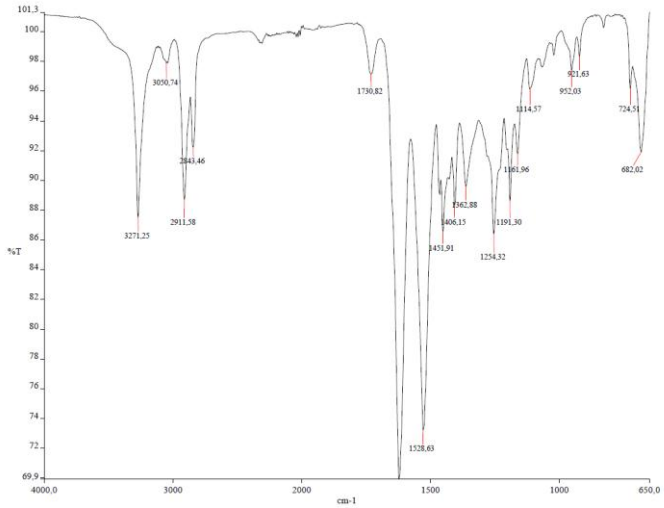


Figure 19 – Spectra of the middle layer (Layer 3 in Figure 15a) in M12

3) Immersion in formic acid

PA is soluble in formic acid and also, by this treatment is easy to scratch the PET. Therefore, the aluminum foil is separated (Layer 2 in Figure 15a in M12).

PI N13 and PI O14 - PE/PET-Al/PE (Multilayer) + PE (Monolayer)

1) No treatment
The figures below represent the samples N13 and O14 without any treatment

PE/PET-Al/PE+PE

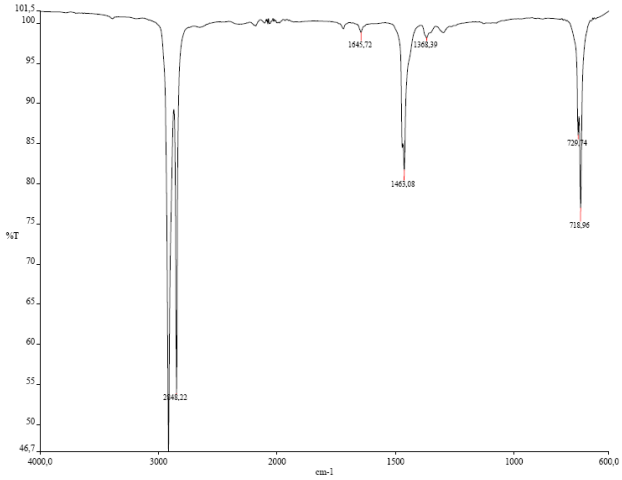


Figure 20 - Spectra of the outer layer (Layer 1 in Figure 15b) in N13 and O14

PE/PET-Al/PE+PE

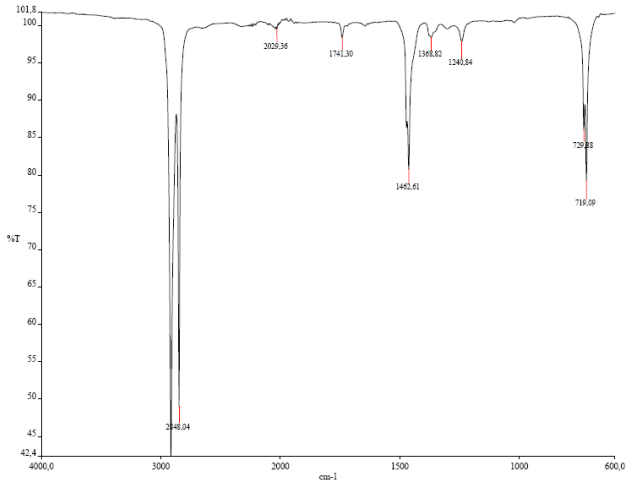


Figure 21 - Spectra of the inner layer (Layer 3 in Figure 15b) in N13 and O14

PE/PET-Al/PE+PE

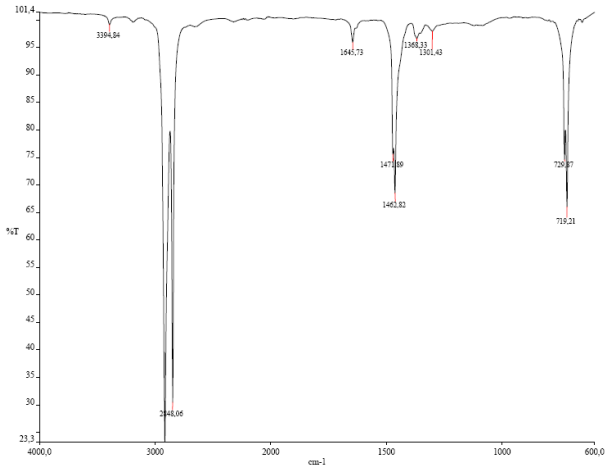


Figure 22 - Spectra of the separated layers (Layer 4 in Figure 15b) in N13 and O14

2) Immersion in HCl until all the Aluminum is dissolved: PE/PET-Al/PE+PE

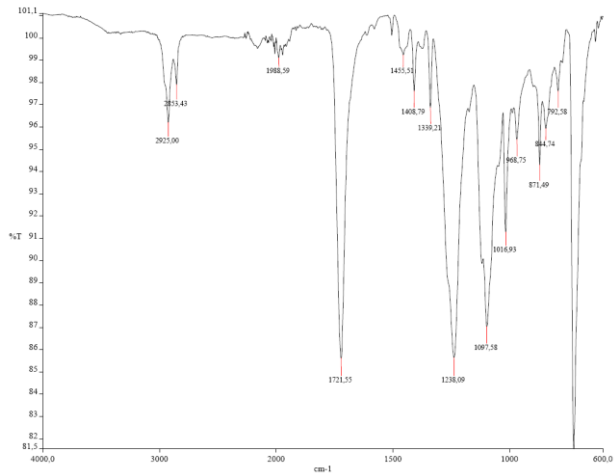


Figure 23 - Spectra of the separated layers (Layer 4 in Figure 15b) in N13 and O14

8.2 Results from Participants

There were 30 NRLs invited and 26 which sent results. Therefore, the evaluation of the results was performed on the 26 sets of data received.
The NRLs used several techniques for the polymer identification listed as follows:

- FTIR
- Pyrolysis
- Pyrolysis-GC-MS
- Flame test
- Melting range
- Solubility in acetone
- Reaction with pH paper
- Flotation test
- Differential Scanning Calorimetry (DSC)
- Beilstein test
- Combination of them.

Among these techniques, FTIR was the principal one used for the identification.

8.2.1 – Evaluation of laboratories performance

The evaluation of the performance of the laboratories for the identification of plastics materials was carried out giving scores to the answers, as mentioned in Section 7.1. For monolayers, the scoring range was between 0 and 2 and for multilayers from 0 to 5. The instructions for the compilation of results recommended to be specific with the answers. This specificity was more important for samples A1, B1, C3 and E5. The necessity for this specificity was based on the fact that those materials, although belonging to the same family, may present different properties such as crystallinity, ductility, types of additives in their formulation, melting temperature, type of processing, among others. Since crystallinity influences migration processes and this crystallinity is different between polyolefins HDPE, LDPE, PP, the migration of additives would not be the same in these materials. Thus, for laboratories which gave as answer only "polyolefin" for sample A1, B2 and C3, it was not considered correct. However, when laboratories were able to identify A1 or B2 as "polyethylene" (more specific) or E5 as "polyamide", these answers were considered correct (1 point).

The multilayers that were sent were composed by a multilayer (with at least three main polymer layers), aluminum, and layers separated from the multilayer itself. In the case of sample M12, the extra layer was 1 blue film (Figure 1a), while for sample N13 and O14, extra layers were transparent (Figure 1 b,c). The participants were asked to identify all the layers, but for the evaluation as correct answer, the identification of the 3 main layers from the multilayer (4 points) and the extra layer (1 point) was necessary. If the participants were not able to identify the polymer in the inner part of the multilayer, but identified the aluminum, they obtained 0.5 points for this identification (note: the identification of the middle layer gave 2 points). Table 4 summarizes the results for monolayers and Table 5 results from multilayers. The results should be considered as separate since the scope of the exercise was for monolayers and that the multilayers were only an exploratory exercise (due to its much higher inherent difficulty).
The scores obtained for each sample was divided by the maximum score in each task, in monolayer by 2, while in multilayer by 5. By this normalization for monolayer and multilayers, scores were between 0 and 1.

Table 4 - Scoring and normalization for monolayers - Normalization: Score/2

	PI A1		PI B2		PI C3		PI D4		PI E5		PI F6		PI H7		PI I8		PI J9		PI K10		PI P15		PI Q16	
Lab Code	Score	Norm	Score	Norm	Score	Norm	Score	Norm	Score	Norm	Score	Norm	Score	Norm	Score	Norm	Score	Norm	Score	Norm	Score	Norm	Score	Norm
LC0003	1	0.5	1	0.5	2	1	2	1	2	1	1	0.5	1	0.5	2	1	2	1	1	0.5	2	1	0	0
LC0004	2	1	2	1	2	1	2	1	1	0.5	2	1	2	1	2	1	2	1	0	0	2	1	2	1
LC0005	2	1	1	0.5	2	1	2	1	2	1	2	1	2	1	2	1	2	1	0	0	0	0	2	1
LC0006	1	0.5	1	0.5	2	1	2	1	1	0.5	2	1	2	1	0	0	2	1	2	1	2	1	2	1
LC0010	1	0.5	1	0.5	2	1	2	1	2	1	2	1	2	1	2	1	2	1	0	0	2	1	0	0
LC0011	1	0.5	2	1	2	1	0	0	1	0.5	2	1	2	1	0	0	2	1	0	0	2	1	2	1
LC0013	2	1	2	1	2	1	2	1	2	1	2	1	2	1	2	1	2	1	2	1	2	1	2	1
LC0016	1	0.5	2	1	2	1	2	1	1	0.5	2	1	2	1	2	1	2	1	2	1	2	1	2	1
LC0017	1	0.5	2	1	2	1	2	1	2	1	2	1	2	1	2	1	2	1	2	1	2	1	2	1
LC0018	2	1	2	1	2	1	2	1	1	0.5	1	0.5	1	0.5	0	0	2	1	2	1	2	1	2	1
LC0020	1	0.5	1	0.5	2	1	2	1	1	0.5	2	1	2	1	2	1	2	1	0	0	2	1	2	1
LC0025	2	1	2	1	2	1	2	1	2	1	2	1	2	1	2	1	2	1	2	1	2	1	2	1
LC0028	1	0.5	1	0.5	0	0	2	1	1	0.5	2	1	2	1	2	1	2	1	0	0	2	1	2	1
LC0031	2	1	1	0.5	2	1	2	1	2	1	2	1	0	0	0	0	2	1	0	0	1	0.5	2	1
LC0037	2	1	2	1	2	1	2	1	2	1	2	1	2	1	2	1	2	1	2	1	2	1	2	1
LC0040	2	1	2	1	2	1	2	1	2	1	2	1	2	1	2	1	2	1	2	1	2	1	2	1
LC0041	1	0.5	1	0.5	2	1	2	1	1	0.5	2	1	2	1	2	1	2	1	0	0	2	1	2	1
LC0043	1	0.5	1	0.5	0	0	2	1	1	0.5	2	1	2	1	2	1	2	1	0	0	2	1	2	1
LC0044	1	0.5	2	1	2	1	2	1	1	0.5	2	1	2	1	2	1	2	1	2	1	2	1	2	1
LC0047	1	0.5	1	0.5	2	1	2	1	2	1	2	1	2	1	2	1	2	1	2	1	2	1	2	1
LC0048	1	0.5	1	0.5	2	1	2	1	1	0.5	2	1	2	1	2	1	2	1	1	0.5	2	1	2	1
LC0049	2	1	2	1	2	1	0	0	1	0.5	0	0	0	0	2	1	2	1	0	0	0	0	2	1
LC0050	1	0.5	2	1	2	1	2	1	1	0.5	2	1	2	1	2	1	2	1	2	1	2	1	2	1
LC0052	1	0.5	1	0.5	2	1	2	1	1	0.5	2	1	2	1	2	1	2	1	2	1	2	1	2	1
LC0055	2	1	2	1	2	1	2	1	2	1	2	1	2	1	2	1	2	1	2	1	2	1	2	1
LC0056	2	1	2	1	2	1	0	0	1	0.5	2	1	2	1	2	1	2	1	0	0	2	1	2	1

Table 5. Scoring and normalization for multilayers - Normalization: Score/5

	PI M12		PI N13		PI O14	
Lab Code	Score	Norm	Score	Norm	Score	Norm
LC0003	2	0.4	2	0.4	2	0.4
LC0004	3.5	0.7	5	1	5	1
LC0005	3.5	0.7	3.5	0.7	3.5	0.7
LC0006	3.5	0.7	3.5	0.7	3.5	0.7
LC0010	3.5	0.7	5	1	5	1
LC0011	1	0.2	2	0.4	2	0.4
LC0013	3.5	0.7	3.5	0.7	3.5	0.7
LC0016	1	0.2	1	0.2	1	0.2
LC0017	3	0.6	3	0.6	3	0.6
LC0018	3.5	0.7	3.5	0.7	3.5	0.7
LC0020	1	0.2	0	0	0	0
LC0025	5	1	5	1	5	1
LC0028	3.5	0.7	3.5	0.7	3.5	0.7
LC0031	2	0.4	2	0.4	2	0.4
LC0037	5	1	5	1	5	1
LC0040	3.5	0.7	3.5	0.7	3.5	0.7
LC0041	2	0.4	3	0.6	3	0.6
LC0043	3.5	0.7	3.5	0.7	3.5	0.7
LC0044	3	0.6	3	0.6	3	0.6
LC0047	3.5	0.7	5	1	5	1
LC0048	3	0.6	3	0.6	3	0.6
LC0049	2.5	0.5	2.5	0.5	0	0
LC0050	5	1	5	1	5	1
LC0052	3.5	0.7	3	0.6	3	0.6
LC0055	3.5	0.7	3.5	0.7	3.5	0.7
LC0056	0	0	0	0	0	0

Performance of the NRLs in each sample

The evaluation of the performance of the laboratories in each sample gives an idea of the difficulty of the test. Figure 24 shows graphically the results for monolayers and Figure 25 multilayer.

Figure 24 shows the overall performance for monolayers, and Figure 25 for multilayers. It is possible to observe that monolayers were in general much easier than multilayers, as expected. The percentages of fails in monolayers were very low. Sample K10 was a very difficult one, and many laboratories were not able to identify it. Sample A1, B1 and E5 were considered an easier task, with the percentage of acceptable very high. This was due to the lack of specificity required in the answers. Samples A1 and B2 were also in the range of acceptable answers even if lacking the outmost specification. The same case was for sample E5, which the participants did not in general identify very specifically (i.e. type of polyamide). The acceptable answers were correct but these would obtain half of the maximum score in the design used a stricter score attribution. The case of sample K10 is different; it was a difficult sample to identify correctly.

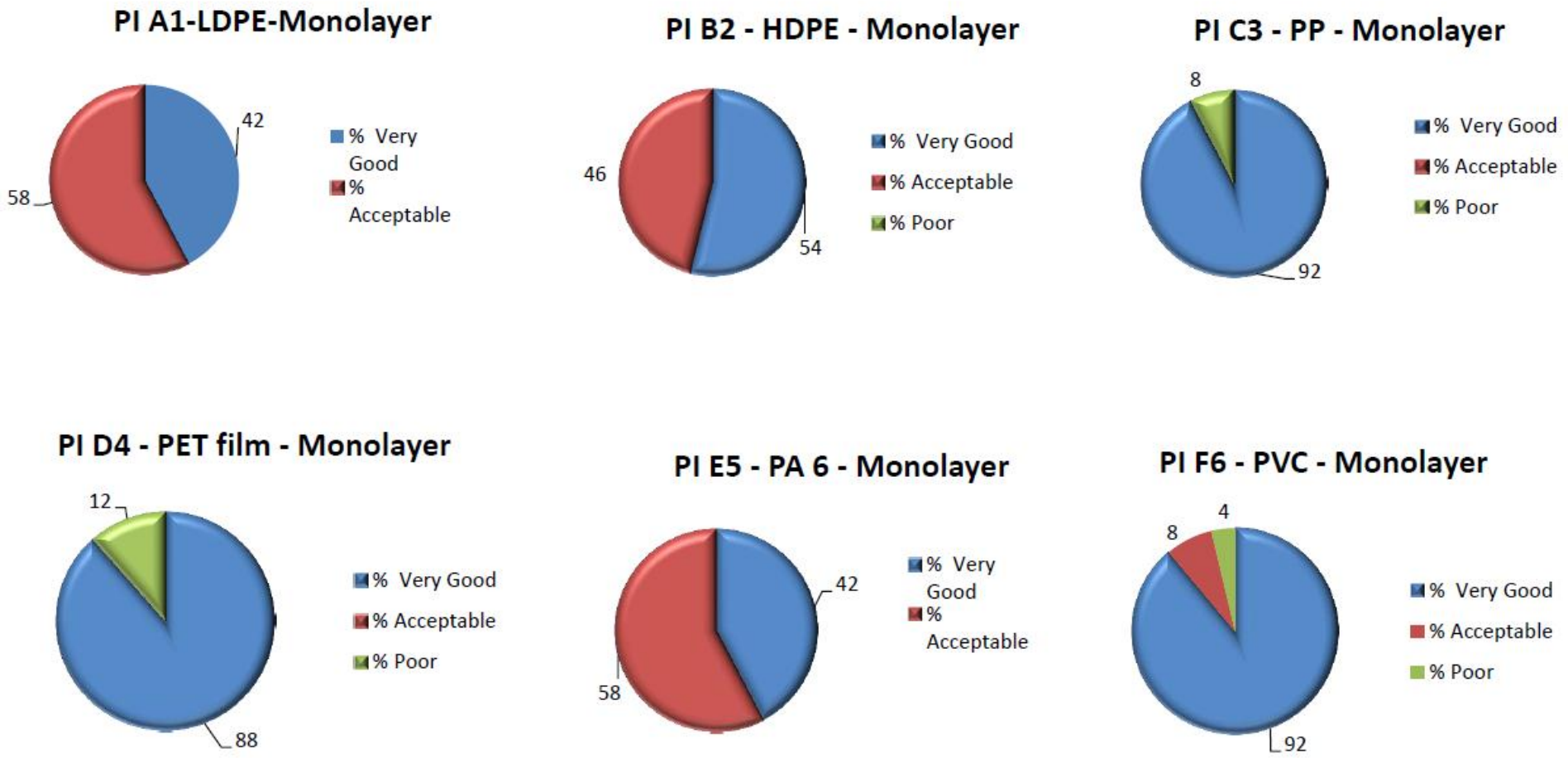


Figure 24 - Performance of the NRLs in Monolayers

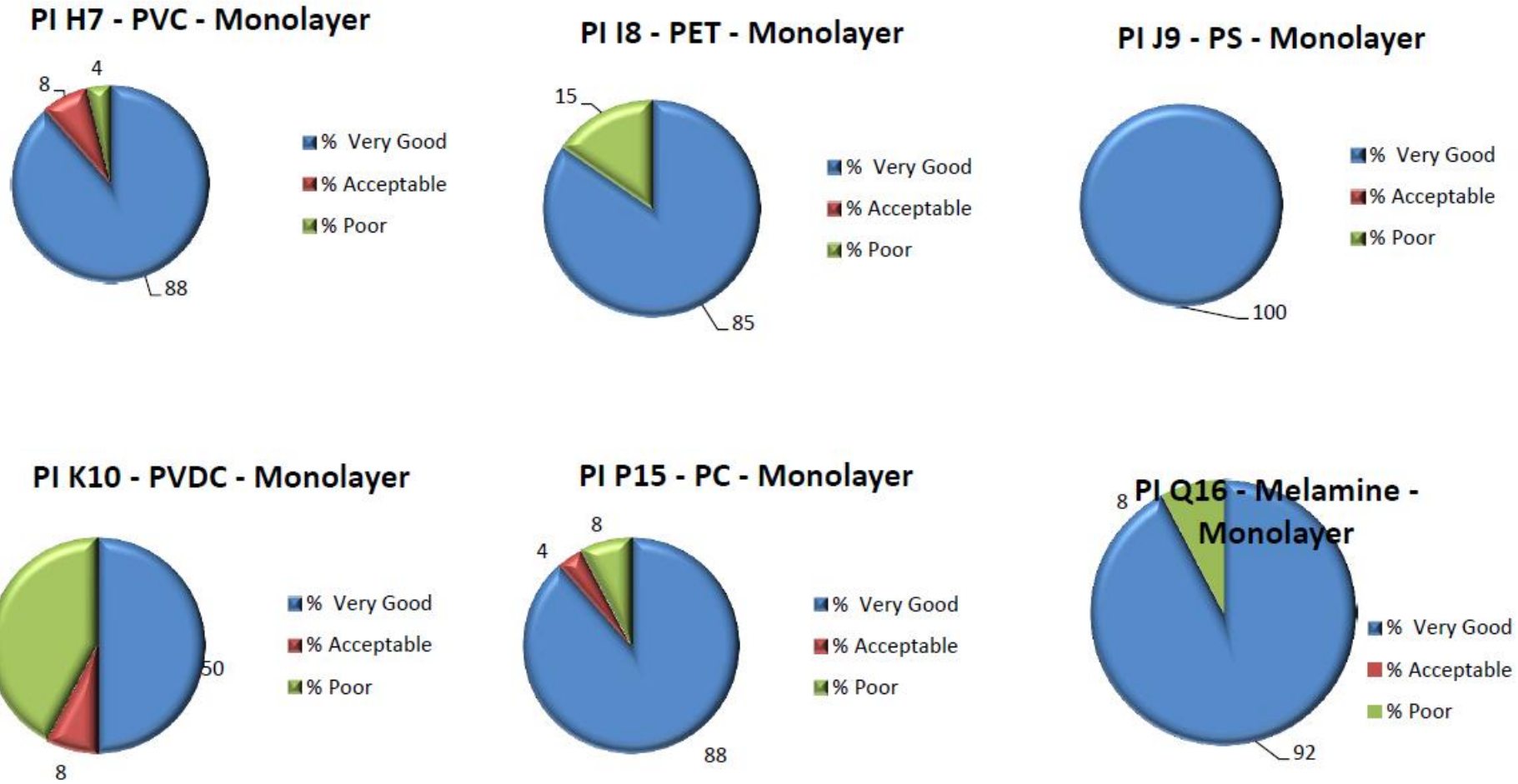


Figure 24 (Cont.) - Performance of the NRLs in Monolayers

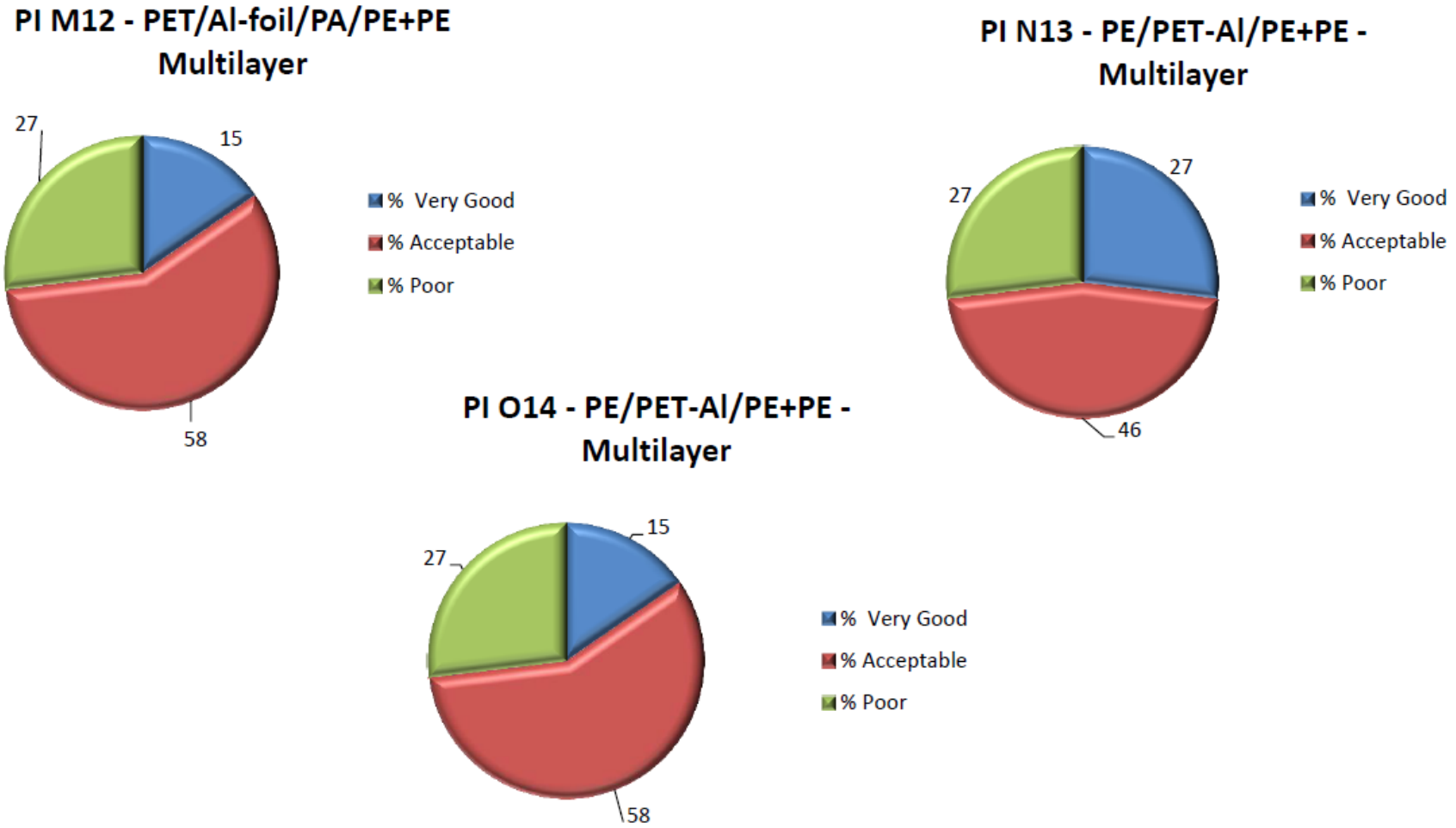


Figure 25 - Performance of the NRLs in Multilayers

Performance of the NRLs for the identification exercise (all samples)

Table 6 summarizes the total scores obtained in the exercise by each laboratory. This was calculated by the average of the normalized scores indicated in Table 4 and 5 for each laboratory. This "Total score" represented the performance of each laboratory in the entire exercise.

Table 6 - Performance of the NRLs for the identification exercise- Total score

Lab Code	Total Score (%)
LC0003	65
LC0004	88
LC0005	77
LC0006	77
LC0009	85
LC0010	78
LC0011	60
LC0013	94
LC0016	77
LC0017	89
LC0018	77
LC0020	65
LC0025	100
LC0028	71
LC0031	61
LC0037	100
LC0040	94
LC0041	74
LC0043	71
LC0044	85
LC0047	91
LC0048	79
LC0049	50
LC0050	93
LC0052	83
LC0055	94
LC0056	63

The evaluation of the performance is based on percentiles: "satisfactory" results, when the total score was higher or the same as 70%; then "questionable", when the total score was lower than 70 but higher or the same as 40%; and "unsatisfactory", when the total score was lower than 40%. In this scheme, the evaluation found that 21 laboratories out of 26 obtained satisfactory results and none of them obtained unsatisfactory.

8.2.2 Evaluation by binary data and fixed threshold

As described previously, the excel tool IF(VALUE>=threshold,1,0) was used in order to transform the results into binary data and evaluate the performance of the laboratories in a more severe way taking into account whether the answers were perfectly correct or not.
The threshold used was 0.7. In other words, samples that were 100% correct for monolayers (those that obtained a "very good", 2 points or 1 point in the normalization) obtained 1, those that did not obtained 0 points.
Regarding multilayers, it was necessary to identify at least three layers in the entire system (3.5 points or 0.7 in the normalization) to obtain 1 point, if not 0 points were assigned.
Table 7 shows the results obtained by this approach for all the samples and for all laboratories. The last column of Table 7 shows the performance of each participant evaluated under the threshold 0.7. On the other hand, the very last row (1-Average) represents the difficulty of the test; while the row above it (Average) the level of success of the laboratories in each sample.

Table 7 – Threshold 0.7 for correct answers

Lab Code	PI A1	PI B2	PI C3	PI D4	PI E5	PI F6	PI H7	PI I8	PI J9	PI K10	PI M12	PI N13	PI O14	PI P15	PI Q16	Average
LC0003	0	0	1	1	1	0	0	1	1	0	0	0	0	1	0	0.40
LC0004	1	1	1	1	0	1	1	1	1	0	1	1	1	1	1	0.87
LC0005	1	0	1	1	1	1	1	1	1	0	1	1	1	0	1	0.80
LC0006	0	0	1	1	0	1	1	0	1	1	1	1	1	1	1	0.73
LC0010	0	0	1	1	1	1	1	1	1	0	1	1	1	1	0	0.73
LC0011	0	1	1	0	0	1	1	0	1	0	0	0	0	1	1	0.47
LC0013	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1.00
LC0016	0	1	1	1	0	1	1	1	1	1	0	0	0	1	1	0.67
LC0017	0	1	1	1	1	1	1	1	1	1	0	0	0	1	1	0.73
LC0018	1	1	1	1	0	0	0	0	1	1	1	1	1	1	1	0.73
LC0020	0	0	1	1	0	1	1	1	1	0	0	0	0	1	1	0.53
LC0025	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1.00
LC0028	0	0	0	1	0	1	1	1	1	0	1	1	1	1	1	0.67
LC0031	1	0	1	1	1	1	0	0	1	0	0	0	0	0	1	0.47
LC0037	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1.00
LC0040	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1.00
LC0041	0	0	1	1	0	1	1	1	1	0	0	0	0	1	1	0.53
LC0043	0	0	0	1	0	1	1	1	1	0	1	1	1	1	1	0.67
LC0044	0	1	1	1	0	1	1	1	1	1	0	0	0	1	1	0.67
LC0047	0	0	1	1	1	1	1	1	1	1	1	1	1	1	1	0.87
LC0048	0	0	1	1	0	1	1	1	1	0	0	0	0	1	1	0.53
LC0049	1	1	1	0	0	0	0	1	1	0	0	0	0	0	1	0.40
LC0050	0	1	1	1	0	1	1	1	1	1	1	1	1	1	1	0.87
LC0052	0	0	1	1	0	1	1	1	1	1	1	0	0	1	1	0.67
LC0055	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1.00
LC0056	1	1	1	0	0	1	1	1	1	0	0	0	0	1	1	0.60
Average	0.42	0.54	0.92	0.88	0.42	0.88	0.85	0.85	1.00	0.50	0.58	0.54	0.54	0.88	0.92	
1-Average	0.58	0.46	0.08	0.12	0.58	0.12	0.15	0.15	0.00	0.50	0.42	0.46	0.46	0.12	0.08	

9. Conclusions

Figure 26 represents the difficulty of each sample. In this plot, sample A1, B1 and E5 were found to be difficult samples; however, the difficulty in this case was the level of specificity. The participants were not able to define A1 as LDPE, B2 as HDPE and E5 as PA6. On the other hand, samples K10 and the multilayers were difficult to identify. The middle layer was especially not identified in most cases. For multilayers some laboratories did not realize that the main layer (metallized one) was a multilayer itself. This is normal since it was a very unusual sample.

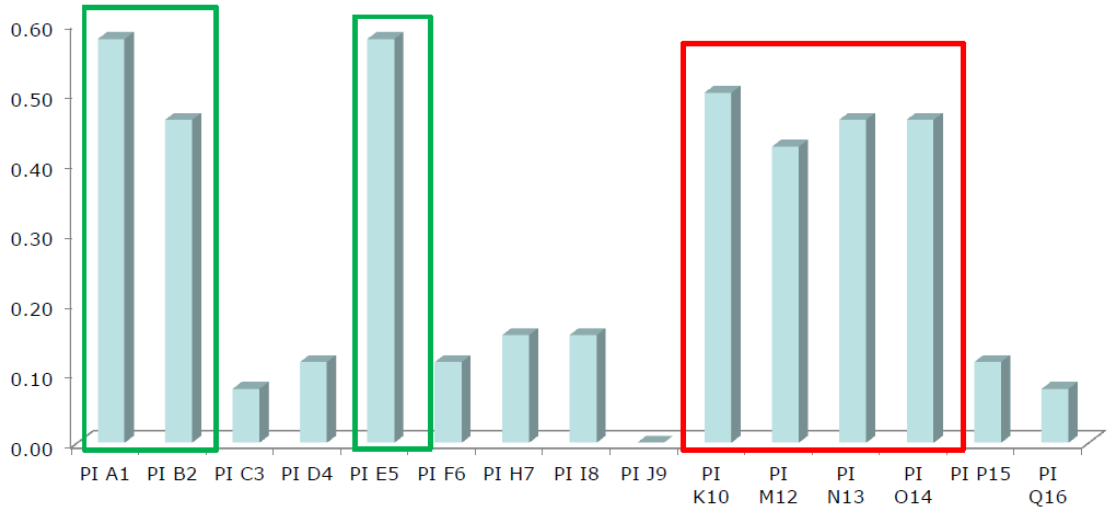


Figure 26 – Difficulty of the test

Figure 27 represents the performance of each laboratory in the entire test under a threshold of 0.7. In this case, a division in three parts was performed to evaluate the final results as: satisfactory, questionable and unsatisfactory. The evaluation was the same as for the overall performance but not in percentage: "satisfactory" results, when the total score was higher or the same as 0.7; then "questionable", when the total score was lower than 0.7 but higher or the same as 0.4; and "unsatisfactory", when the total score was lower than 0.4. By using this approach 12 participants out of 26 obtained satisfactory results and no unsatisfactory results were obtained by none of the laboratories.

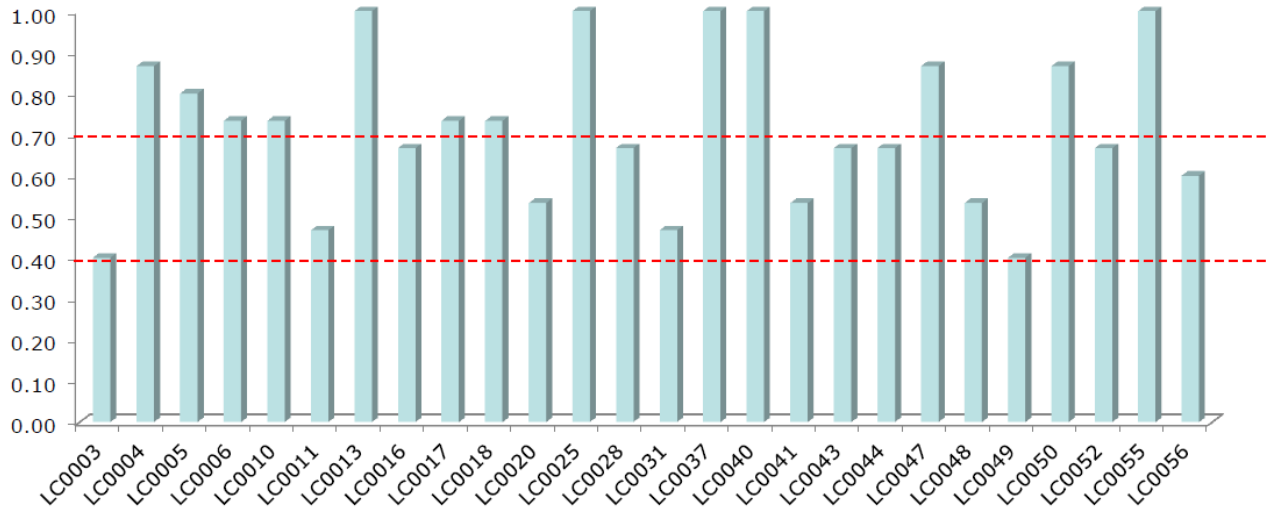


Figure 27 – Performance of the participants

The overall evaluation was as follows:

- 21/26 participants obtain satisfactory results (81%)
- The 15 samples were satisfactory identified
- None of the laboratories obtained unsatisfactory results

Using the stricter method of setting a threshold of 0.7 to be counted as a correct

- 8/15 samples were satisfactory identified (53%)
- 14/26 participants obtained satisfactory results (54%)
- None of the laboratories obtained unsatisfactory results

The main difficulty found was the separation and identification of the multilayers. The final conclusion is that the performance of the laboratories for identification blind samples was satisfactory.

10. Acknowledgements

The NRLs participating in this exercise - listed below - are kindly acknowledged.

AUSTRIA	Austrian Agency for Health and Food Safety (AGES), Wien
BELGIUM	Institute of Public Health, ISSP-LP, Bruxelles
CYPRUS	Laboratory for Control of Food Contact Materials and Control of Toys Ministry of Health, State General Laboratory (SGL), Nicosia
CZECH	NIPH- NRL for Food Contact Materials and for Articles for children under 3 years old, National Institute of Public Health (SZU'), Praha
DENMARK	Department of Food Chemistry, Danish Veterinary and Food Administration, Lystrup
DENMARK	Danish Veterinary and Food Administration Laboratory Århus, Lystrup
ESTONIA	Health Protection Inspectorate - Central Laboratory of Chemistry, Tallinn
FINLAND	Finnish Customs Laboratory, Espoo
FRANCE	Centre for Energy Material and Packaging - Laboratoire National d'Essais, Trappes Cedex
FRANCE	SCL Laboratoire de Bordeaux-Pessac, Pessac
GERMANY	Bundesinstitut für Risikobewertung (BFR) (Federal Institute for Risk Assessment), Berlin
GREECE	General Chemical State Laboratory, D' Chemical Service of Athens, Section Laboratory of Articles and Materials in Contact with Foodstuffs, Athens
HUNGARY	Central Agricultural Office, Food and Feed Safety Directorate, Food Toxicology National Reference Laboratory, Budapest
IRELAND	Public Analyst Laboratory - Sir Patrick Duns Hospital, Dublin
ITALY	Istituto Superiore di Sanità, Laboratorio Esposizione e rischio da materiali, c/o Dipartimento ambiente e connessa prevenzione primaria, Roma
LITHUANIA	National Public Health Surveillance Laboratory, Laboratory of Chemistry, Vilnius
LUXEMBOURG	Laboratoire National de Santé, Division du Controle des Denrées Alimentaires, Luxembourg
POLAND	Laboratory of Department of Food and Consumer Articles Research, National Institute of Hygene, Warsaw
PORTUGAL	ESB-SE (Portuguese Catholic University - Biotechnology College – Packaging Department), Porto
SLOVAKIA	National Reference Centre and Laboratory for material and articles intended to come into contact with food, Regional Public Health Authority In Poprad (RUVZ), Poprad
SLOVENIA	National Institute of Public Health of Republic of Slovenia, Department of Sanitary Chemistry, Ljubijana

SPAIN	Centro Nacional de Alimentación, Agencia Espanola de Seguridad Alimentaria y Nutrición (AESAN), Madrid
SWEDEN	National Food Administration, Food Standards Devision, Uppsala
THE NETHERLANDS	Food and Consumer Product Safety Authority (VWA), Ministry of Economic Affairs, Agriculture and Innovation, Groningen
UNITED KINGDOM	Central Science Laboratory, York
SWITZERLAND	Official Food Control Authority of the Canton of Zurich

11. References

[1]	Regulation (EC) No 882/2004 of the European Parliament and of the Council of 29 April 2004 on official controls performed to ensure the verification of compliance with feed and food law, animal health and animal welfare rules.
[2]	Regulation EC No 284/2011of 22 March 2011 laying down specific conditions and detailed procedures for the import of polyamide and melamine plastic kitchenware originating in or consigned from the People's Republic of China and Hong Kong Special Administrative Region, China.
[3]	Commission Implementing Regulation No 321/2011 of 1 April 2011 amending Regulation (EU) No 10/2011 as regards the restriction of use of Bisphenol A in plastic infant feeding bottles
[4]	Marsh, K., Bugusu, B Food packaging-Roles, Materials and environment issues. 2007. Journal of Food Science 72 (3): 39-55.
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[6]	ISO/IEC 17043:2010 Conformity assessment - General requirements for proficiency testing
[7]	Selection, use and interpretation of proficiency testing (PT) schemes. Eurochem. Edited by Ian Mann and Brian Brookman. Second edition, 2011.
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[9]	Antron.Technical Boulletin "Differences between type 6,6 and Type 6 Nylon" 2013, available in: http://antron.net/na/pdfs/literature/K02510_N66vsN6_Tech_Bulletin_06_18_13.pdf)
[10]	S. Ramesha, Koay Hang Leen, K. Kumutha, A.K. Arof. FTIR studies of PVC/PMMA blend basedpolymer electrolytes. Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy 66 (4–5) 2007:1237–1242.
[11]	Merline, D. Vukusic, S. Abdala, A.A. melamine formaldehide: curing studies and reaction mechanism. Polymer Journal (2012), 1-7.

11. Annexes

Annex 1. Invitation letter to laboratories ILC002 2013
Annex 2. Letters accompanying the sample ILC002 2013
Annex 3. Letter of confirmation of receipt of ILC002 2013
Annex 4. Instructions for compilation of the results of ILC002 2013
Annex 5. Table template for the compilation of the results

EUROPEAN COMMISSION
GENERAL DIRECTORATE JRC
JOINT RESEARCH CENTRE
Institute for Health and Consumer Protection – IHCP
Chemical Assessment and testing

Annex 1. Invitation letter to the laboratories to ILC001 2013

EUROPEAN COMMISSION
GENERAL DIRECTORATE JRC
JOINT RESEARCH CENTRE
Institute for Health and Consumer Protection – IHCP
Unit Chemical Assessment and Testing

Ispra May 17th, 2013

Dear Madam, Sir

Comparative trial ILC 2013 - 002 from EURL FOOD CONTACT MATERIALS "Identification of the nature of plastics polymers. "

On behalf of the EURL for food contact materials, I would like to invite you to participate in a comparative trial/interlaboratory comparison (ILC) exercise for the identification of the nature of polymers which is due to start by end of May. As agreed in the EURL-NRL FCM plenary, the scope of this ILC exercise is a Proficiency testing, the laboratories are free to use their own methods.
I would like to remind you that it is a duty for you as an NRL-FCM to participate in the ILCs organised by the EURL-FCM since the work programme is decided with your agreement.
This exercise is the first one of a more qualitative nature, so it is limited to NRLs.
We have pre-registered everyone, which means we will send test kits to all of you. We however need to receive the **reception of samples** for our own administrative purposes to: Mercedes Peltzer (mercedes-Ana.PELTZER@ec.europa.eu)**.**

The samples will be sent to you early next week. You will find additional information in the kit sent and on the form "shipment test". You will also receive more detailed instructions for the compilation of the results. The deadline for submission of results is **25 June.**

If you have any question, please contact Mercedes Peltzer.

Sincerely yours,

Catherine Simoneau

Dr. Catherine Simoneau
Operating Manager, Community Reference Laboratory for Food Contact Materials
European Commission, DG-Joint Research Centre
Institute for Health and Consumer Protection
Unit Chemical Assessment and Testing, T.P. 260
Ispra Va 21020 Italy

Cc: P. Aguar (JRC), D. Rembges (JRC)
A. Schaefer, B. Schupp (SANCO)

Annex 2. Shipping kit letter accompanying the samples to ILC002 2013

Ispra, May 17th, 2013

Shipping kit for interlaboratory comparative testing 2013 002: IDENTIFICATION OF POLYMERIC MATERIALS"

Shipping kit - samples

- PI A1
- PI B2
- PI C3
- PI D4
- PI E5
- PI F6
- PI H7
- PI I8
- PI J9
- PI K10
- PI M12
- PI N13
- PI O14
- PI P15

Among the samples there are monolayers and multilayer materials.

Shipping kit - documentation

- Letter of confirmation of receipt ILC 002 2013
- Letter with instructions to perform the identification of the polymer samples and compilation of the results for interlaboratory comparative testing EURL-FCM ILC002 2013.
- Print copy of word file "ILC 002 2013 Table for Results.doc" to fill in with the results compilation
- Electronic word file "ILC 002 2013 Table for Results.doc" will be sent by email.

Storage

- The samples should be stored at room temperature

Annex 3. Letter of confirmation of receipt of the sample to ILC001 2013

Ispra, May 17th, 2013

PARTICIPATION TO EURL-FCM ILC002 2013
POLYMER IDENTIFICATION

CONFIRMATION OF RECEIPT OF THE SAMPLES

Please return this form to confirm that the sample package has arrived. In case the package is damaged, please state this on the form and contact us immediately.

Your Name:	
Organization:	
E-mail:	
Phone:	

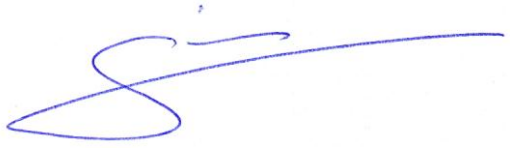
Any remarks …….

Date arrival package …….

Signature …….

Kindly send back this form to: Mercedes A. Peltzer (Mercedes-Ana.PELTZER@ec.europa.eu)

Sincerely yours,



Catherine Simoneau

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Chemical Assessment and testing

Annex 4. Instructions for compilation of the results ILC 002 2013

Ispra, May 17th, 2013

Instructions for the compilation of the results for interlaboratory comparative testing 2013_002: IDENTIFICATION OF POLYMERIC MATERIALS

DEADLINE: June 25th 2013

Instructions

1. Analyse and identify the samples given by the EURL-FCM.
2. The analysis and identification must be done by using any analytical technique that it is considered appropriate for the exercise.
3. Identify the nature of the polymer with sufficient level of specificity, i.e: high density polyethylene rather than polyolefin.
4. For multilayer, identify each of the layers.
5. Justify your answers.

Results requested:

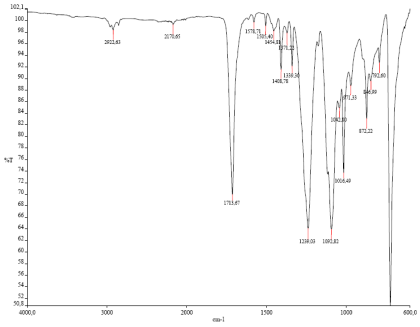
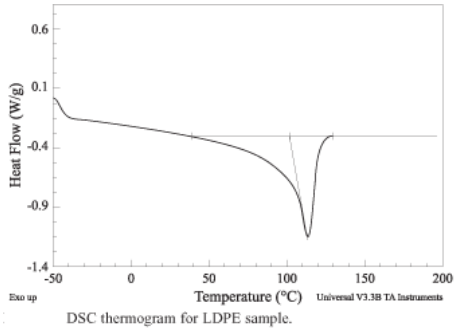
When reporting the results you must indicate:

- The type of sample: monolayer or multilayer
- The nature/identity of the polymer
- The technique used for the identification
- The parameters used to identify each polymer, i.e: melting point, FTIR bands, etc.
- You must fill in **Table 1** given as separate document.
- If you need to explain with more details the results that you found, it is possible to use the space provided for "Notes"-

Please fill in Table 1, as shown in the example, with the results obtained and send it back by e-mail to Mercedes A. Peltzer (Mercedes-Ana.PELTZER@ec.europa.eu) by June 25th 2013.

Example:

Table1 – Results obtained in the polymer identification

<table>
<tr><th>CODE</th><th>Polymer</th><th>Technique used for the identification</th><th>Parameters used for the identification</th></tr>
<tr><td>PI Z5</td><td>PET
Monolayer</td><td>FTIR</td><td>Characteristic bands from PET were observed:
<table>
<tr><th>Wavenumber (cm^{-1})</th><th>Assignment</th></tr>
<tr><td>723</td><td>Out of plane of benzene group</td></tr>
<tr><td>845</td><td>CH_2 rocking of glycol</td></tr>
<tr><td>870</td><td>Out of plane of benzene group</td></tr>
<tr><td>970</td><td>C-O stretching of glycol</td></tr>
<tr><td>1017</td><td>In-plane vibration of benzene</td></tr>
<tr><td>Broad band 1090</td><td>Mainly due to ester C=O stretching</td></tr>
<tr><td>Broad band 1230</td><td>Mainly due to ester C=O stretching</td></tr>
<tr><td>Sharp 1710</td><td>C=O stretching (conjugated with the ring)</td></tr>
<tr><td>1340</td><td>CH_2 wagging of glycol</td></tr>
<tr><td>1370</td><td>CH_2 wagging of glycol</td></tr>
<tr><td>1408</td><td>Aromatic skeletal stretching band. Ring in-plane deformation</td></tr>
</table>
 </td></tr>
<tr><td>PI X1</td><td>LDPE</td><td>DSC</td><td>Determination of thermal parameters: T_m ºC


DSC thermogram for LDPE sample.
T_m: 113ºC</td></tr>
<tr><td>PI R3</td><td>Multilayer
PP/PET-metallized/PP</td><td>FTIR</td><td>Separation of the layers by different chemicals and analysis of the characteristic bands.
FTIR spectrum of polypropylene presents a shoulder at 2875 cm^{-1}, and the asymmetric and symmetric in-plane C–H (–CH_3) at 1455 and a shoulder at 1358 cm^{-1} confirm that it is polypropylene. The peak at 1376 cm^{-1} is assigned to –CH_3 group.

Al was dissolved in HCl, the layer were separated and the PET layer was characterized.</td></tr>
</table>

If you have any question, please contact Mercedes A. Peltzer (Mercedes-Ana.PELTZER@ec.europa.eu)

Annex 5.Table template for the compilation of the results in non-electronic format

Ispra, May 17th, 2013

Table template for the results of the EURL-FCM ILC002 2013

(See instructions on separate document)

CODE	Polymer	Technique used for the identification	Parameters used for the identification
PI A1			
PI B2			
PI C3			
PI D4			
PI E5			
PI F6			
PI H7			
PI I8			
PI J9			
PI K10			
PI M12			
PI N13			
PI O14			
PI P15			
PI Q16			

Notes:

European Commission
EUR 26467 – Joint Research Centre – Institute for Health and Consumer Protection

Title: Report of an Interlaboratory Comparison from the European Reference Laboratory for Food Contact Materials: ILC002 2013 –Identification of Polymeric Materials

Author(s): Mercedes Ana Peltzer and Catherine Simoneau

Luxembourg: Publications Office of the European Union

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Abstract
Interlaboratory comparisons (ILCs) are an important element of laboratory quality assurance which allows individual laboratories to compare their analytical results with those from other laboratories while providing them objective standards to perform against. One of the core duties of the European Reference Laboratories is to organise such ILCs, as stipulated in Regulation (EC) No 882/2004 of the European Parliament and of the Council [1].

In accordance with the above requirements the European Reference Laboratory for Food Contact Material (EURL-FCM) organised in 2013 a number of ILCs for its network of appointed National Reference Laboratories (NRLs).The scopes of the 2013 ILCs were agreed in a plenary meeting of June 2012 which established the work programme of the agreed exercises for the year 2013. The ILC 002 2013 was directed towards evaluation of the laboratory performance on the identification of polymeric materials with unknown nature. This report present the work and results obtained in this exercise which targeted 12 monolayer materials. The results of the identification of such materials were fully satisfactory. In addition a scoping exercise was conducted on multilayer foils, which highlighted needs for guidance for the separation and identification of layers within multilayer materials.

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Key policy areas include: environment and climate change; energy and transport; agriculture and food security; health and consumer protection; information society and digital agenda; safety and security including nuclear; all supported through a cross-cutting and multi-disciplinary approach.

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