

# DETERMINING THE COMPOSITION OF ROOFING MEMBRANES MANUFACTURED WITH ATACTIC POLYPROPYLENE (APP)

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**B**itumens modified with plastomeric or elastomeric polymers are increasingly used in waterproofing materials.

The design of the composition of a roofing or waterproofing sheet with a binary system of bitumen and atactic polypropylene (APP) has gained importance mainly because this polymer—a by-product of the manufacture of isotactic polypropylene—is economically very competitive.

The variability of the chemical composition of APP as well as the impurity of the material (the presence of isotactic polypropylene), its copolymerization with polyethylene and the addition of mineral substances make it difficult not only to develop a composition for roofing but also to perform control analyses on the finished goods.

This paper describes a method of determining the composition of a polymer-bitumen made with APP (e.g., manufacturing batch), as well as the analytical characterization of the constitution of a roofing sheet or waterproofing layer (e.g., bridge waterproofing). An operating procedure based on the insolubility of some constituents and on the solubility and quantitative separation of other components makes it possible, using infrared spectrography, to determine the full composition of a waterproofing or roofing sheet.

Results obtained on various samples show the narrow dispersion of the values found, which is due to the heterogeneity of the material during the constitution of the sample for analysis.

## KEYWORDS

Analysis, atactic polypropylene, bitumen, composition, determination, isotactic polypropylene, mineral filler, modified bitumen, roofing membrane, waterproofing sheet.

## INTRODUCTION

Be it in the road or construction field, roofing membranes containing bitumens modified with plastomeric and elastomeric polymers are increasingly used. Among the composition designs of roofing sheets the ones with a binary system (bitumen plus APP) have gained importance in Europe because they offer good resistance to heat as well as to climatic aging.

The main goal when modifying bitumens with polymers, particularly APP, is to broaden their temperature interval by making them more resistant to bending (to increase their softening point) and more resistant to breaking (lowering their weakness to cold).

For users of this type of material the real value and steadiness of the roofing membrane is judged by its performance when being laid, and in service.

On the other hand, more or less conventional laboratory trials also enable one to determine the physico-chemical behavior of the constituents and provide information on their durability through aging tests. The results of such trials are taken into account when choosing substances with modified bitumen.

It appears that the identification of the chemical composition of the substances would enable us to better complete and understand the trials performed by judging and interpreting the experiments to improve the material used.

On a practical side, it might be useful to carry out the analysis of the quality control tests made during the manufacturing process as well as on the finished goods that will also lead to a better analysis of the product.

## CHARACTERIZATION OF POLYPROPYLENE<sup>1,2</sup>

A rough summary of the characteristics and properties of polypropylene will explain the process followed to develop the correct method of determining this polymer.

Different polymers arise during the polymerization process of polypropylene.

### Isotactic Polypropylene (Figure 1)

All the CH<sub>3</sub> groups find themselves on the same side of the polymeric chain. This regularity favors apparition of the crystalline zones and confers good mechanical properties as well as high melting point (172°C).

This polymer resists organic solvents and takes solvents (the boiling point of which lies above 100°C) such as xylene, decahydronaphtalene, tetrahydronaphtalene to be partly dissolved.

### Atactic Polypropylene

Atactic Polypropylene (Figure 2) is a by-product obtained during the production of isotactic polypropylene and is also currently manufactured as a primary polymer in the United States.

The side chains are randomly distributed resulting in poor molecular symmetry and little or no order or cristallinity. This amorphous polymer has poor mechanical properties and has few uses in the plastic industry. It is soluble in hot xylene and heptane but precipitates partly again at room temperature. Methyl alcohol cannot dissolve it.

**Syndiotactic Polypropylene** (Figure 3)

Syndiotactic polypropylene is only of scientific interest at present.

Small links of isotactic polypropylene form alternately on each side of the chain. This type of polymer is also amorphous. Its properties are close to those of APP.

**METHOD OF ANALYSIS—APPLICATION FIELDS**

The process developed is based on selective solubility and insolubility of the different constituents of a roofing membrane.

They are separated one after the other with solvents like xylene, normal heptane and a mix of diethyl ether—methyl alcohol (80/20 in volume).

The reinforcement and mineral filler are not soluble in these solvents and are separated at the beginning of the determination.

During the analysis the bitumen is broken up into asphaltenes and malthenes.

These operations are carried out with usual laboratory equipment except for the final infrared spectrographic exam.<sup>5</sup>

The general analytic sketch is presented in Figure 4.

This method is used on:

- A mix of bitumen and APP (main binder).
- A mix of bitumen with APP and mineral filler (during the manufacturing process).
- The finished product (roofing membrane).

The values obtained during the analysis are interpreted and reveal the composition of the analyzed substance.

**OPERATING METHODS USED TO DETERMINE THE COMPOSITION OF BITUMEN-POLYPROPYLENE MIXES****Determining the Insoluble Part in Xylene**

Introduce a sample of roughly 2 g weighed to within 0.1 mg (mass m1) in a 250-ml tarred erlenmeyer (number 1: mass m2). Add 50 ml of xylene (pro analysis) and heat one hour in a reflux refrigerant with magnetic shaking (\*).

Let everything cool down for 30 minutes and filter on a SS n°597 filter previously tarred in a filter-weigher (mass m3).

Collect the filtrate in a 250-ml tarred erlenmeyer (number 2: mass m4).

Rinse the erlenmeyer carefully with xylene and wash the filter with successive portions of 10 ml xylene. Dry the erlenmeyer and the filter in an oven at  $120 \pm 5^\circ\text{C}$  and weigh them after complete cooling down (erlenmeyer number 1: mass m5; filter-weigher containing the insoluble part in xylene: mass m6).

Put the filter with this insoluble part in an extractor (Figure 5)<sup>4</sup> set above an erlenmeyer containing 50 ml xylene. Keep it one hour at boiling point. Dry and weigh the filter once again (mass m7).

Calcinate the filter during one hour in oven at  $500^\circ\text{C}$  in a tarred crucible (mass m8) and weigh after cooling with desiccator (\*\*) (mass m9).

**Notes**

- When the sample to be analyzed is a roofing sheet it can be put in an extractor (Figure 5) and treated with boil-

ing xylene in a reflux refrigerant to separate the reinforcement. The latter is weighed after drying in an oven at  $120 \pm 5^\circ\text{C}$  and cooled down (mass M). After 30 minutes of cooling, the analysis can be carried on as described in the operating method. To reach a more precise weight, it is advisable to take a larger piece of roofing sheet.

- The remnants of the calcination process can be further analyzed and can undergo a second calcination process at  $900^\circ\text{C}$ . If the mineral filler is constituted by calcium carbonate, the calcination residue at  $900^\circ\text{C}$  is CaO. This content, multiplied by 1.7 to express the final result in  $\text{CaCO}_3$ , must correspond to the value obtained for the calcination residue at  $500^\circ\text{C}$ . If the mineral filler is not only made of calcium carbonate, the final result expressed in percent will be higher than the one obtained earlier. One must then go on separating and add 10 ml of hydrochloric acid 1/3 in the crucible (preferately platinum), boil for a few minutes, filter on an ashless filter, wash with boiling water and eliminate the chlorides (check with a solution of silver nitrate). Then, place the filter in the same crucible, dry, calcinate at  $900^\circ\text{C}$  and proceed with the weighing (mass m10). This insoluble residue in chlorodic acid is generally made of a siliceous particles ( $\text{SiO}_2$ ).

**Determining the Insoluble Part in Normal Heptane**

Distill the filtrate contained in erlenmeyer n°2 in a rotating evaporator to eliminate the xylene completely. This operation must be achieved at  $130^\circ\text{C}$  with an initial vacuum of 400 mbar brought at 10 mbar in the end of the distillation.

Add 50 ml of normal heptane and boil for one hour in a reflux refrigerant. Let cool down for half an hour, filter on SS n°597 filter previously tarred in a filter-weigher (mass m12). Collect the filtrate in a tarred erlenmeyer of 250 ml (number 3: mass m11).

After rinsing the erlenmeyer, dry it in an oven at  $105 \pm 5^\circ\text{C}$ , wash the precipitate with normal heptane added in small portions and dry in an oven at  $105 \pm 5^\circ\text{C}$ . Weigh the erlenmeyer and the filter-weigher after cooling (erlenmeyer n°2: mass m13) (filter-weigher: mass m14).

**Determining the Insoluble Part in Ether-Alcohol Blend**

Distill the filtrate contained in erlenmeyer number 3 in a rotating evaporator at  $100^\circ\text{C}$  under a vacuum of 400 mbar brought to 10 mbar in the end of the operation. Then treat the residue in 50 ml mix made of 80 percent diethyl ether and 20 percent methyl alcohol.

Slightly warm the erlenmeyer in a reflux refrigerant to dissolve the malthenes and place it in a thermostatic box for one hour at a low temperature ( $-20^\circ\text{C}$ ). Separate with a double precipitation: dry and tarre a filter-weigher containing two SS n°597 filters (mass m15) and filter on one of the filters. Collect the filtrate in a tarred erlenmeyer of 250 ml (number 4: mass m16).

Wash the filter with some ether-alcohol blend and repeat the operation described above. Once you have finished washing, dry the erlenmeyer and the filter-weigher and weigh them (erlenmeyer number 3: mass m17, filter-weigher with the two filters containing the precipitate: mass m18).

**Determining the Soluble Part in Ether-Alcohol Blend**

Distill the filtrate of erlenmeyer number 4 in a rotating evaporator at 30°C in the beginning of the distillation and progressively bring the temperature to 90°C, increasing the vacuum from 900 to 200 mbar at the end of the distillation. Weigh the erlenmeyer at the end of the operation and after cooling (mass m19).

Put the erlenmeyer in the oven at 105 ± 5°C for a few minutes and take some substance that you spread in a layer on a NaCl window used in infrared spectrography.

Develop the infrared spectrum (Figure 6) after controlling the transmission at 1376 cm<sup>-1</sup>. This should be comprised between 15 and 20 percent. If this is not the case, adapt the thickness of the film consequently. Calculate the absorbance at 974 and 1376 cm<sup>-1</sup> make the ratio and input the value obtained on the calibration curve set up below to determine the content of APP in the soluble fraction in the ether-alcohol mix (percent IR).

**Determining the Content of APP in the Soluble Fraction in the Ether-Alcohol Mix by Infrared Spectroscopy**

*Preparation of the Titrated Solution of Atactic Polypropylene*—Introduce more or less 7.5 g of APP clippings in an erlenmeyer of 500 ml and add 250 ml of normal heptane. Boil one hour in a reflux refrigerant and let cool at room temperature. Filtrate the solution quickly on a folded filter and collect the filtrate in a flask of 500 ml. Wash the precipitate with some normal heptane and complete to the volume. After homogenization, take three aliquote fractions of 10 ml, put them in three beakers of 50 ml previously tarred (m1, m2, m3). Evaporate the solvent in an oven at 105 ± 5°C. Weigh them after cooling and go on drying until constant mass (m1', m2', m3'). The content of the solution of APP is then deducted: (m1' - m1)/10 = g PPA/ml.

*Preparation of Standard Mixes*—Take five erlenmeyers of 250 ml, introduce samples of roughly 2 g of straight-run petroleum bitumen, add 50 ml of normal heptane and boil one hour in a reflux refrigerant. Let cool for 30 minutes then filtrate the solutions on SS n°597 filters and collect the filtrates in tarred erlenmeyers of 250 ml. Wash the filters with some normal heptane and distill the filtrates individually, at 100°C, in a rotary evaporator in a vacuum of 40 mbar brought to 10 mbar for 10 minutes at the end of the distillation.

After cooling, weigh the erlenmeyers and deduce the quantities of malthenes you have collected. Calculate, starting from the titre of the APP solution, for the quantity of malthenes contained in each erlenmeyer, the volumes of titrated PPA solution corresponding approximately to 5 percent, 7.5 percent, 10 percent, 12.5 percent and 15 percent of PPA:

Example preparation of a standard of 15 percent PPA:

- Titre of the atactic polypropylene solution 0.0169 g/ml.
- Quantity of collected malthenes 1.9013 g.
- Weight of PPA + malthenes (15 percent + 85 percent) 2.2368 g.
- Weight of PPA (2.2368 g - 1.9013 g) 0.3355 g.
- Volume of the titrated solution to be added 19.85 ml.

This volume rounded up to 20 ml is added to the malthenes. The content of APP then reaches in this case 15.09 percent. The normal heptane introduced in the erlenmeyer is eliminated through distillation as described earlier.

*Setting Up the Calibration Curve*—When all the normal heptane is eliminated, take one drop of the mix contained in the erlenmeyer with a stirring rod. Put it on a window in NaCl, spread it to make an even film. Put the sample in the measuring beam of the infrared spectrograph and one NaCl window in the reference beam.

Develop the infrared spectrum after having controlled its transmission to be measured at 1376 cm<sup>-1</sup>. It should be comprised between 15 and 20 percent. If this is not the case, adapt the thickness of the film consequently. Calculate the absorbance at 974 and 1376 cm<sup>-1</sup> make the ratio: absorbance 974 cm<sup>-1</sup>/absorbance 1376 cm<sup>-1</sup>. Put this value on the graph and make the calibration curve according to the content in APP.

**Calculations**

The sample being represented under mass m1, the content of the different fractions it contains can be calculated as follows:

- Reinforcement:  $C1 = \frac{M}{m1} \times 100$

- Insoluble part in xylene (after cooling):

$$C2 = \frac{(m5 - m2) + (m6 - m3)}{m1} \times 100$$

- Soluble part in xylene (under reflux):

$$C3 = \frac{(m6 - m7)}{m1} \times 100$$

- Mineral substances:  $C4 = \frac{(m9 - m8)}{m1} \times 100$

- Insoluble part normal heptane:

$$C5 = \frac{(m13 - m4) + (m14 - m12)}{m1} \times 100$$

- Insoluble part in ether-alcohol mix:

$$C6 = \frac{(m17 - m11) + (m18 - m15)}{m1} \times 100$$

- Soluble part in ether-alcohol mix:

$$C7 = \frac{(m19 - m16)}{m1} \times 100$$

- APP content of the soluble part in ether-alcohol mix:

$$C8 = \% \text{ IR} \times (T7 / 100)$$

**Interpretation**

The composition of the mix, expressed in percent of the mass of the sample, is deduced from the analytic values obtained when determining the different parts which constitute this mix:

- Reinforcement C1
- Mineral filler C4
- Isotactic polypropylene C2 - (C3 + C4)
- Atactic polypropylene C3 + C6 + C8
- Bitumen C5 + (C7 - C8)

As far as the waterproofing sheets are concerned, in order to get the composition of the bituminous mix used for their manufacturing, that is excluding the reinforcement, one has to divide the values obtained here above with the following factor:  $(100 - C1) \times 0.01$ .

### ANALYZED MIXES

The analyses performed on 12 samples of bitumen-polymer mixes are divided up as follows:

- Four mixes manufactured in the laboratory (CRR 1 to 4).
- Five industrial mixes containing mineral fillers from three different manufacturers (CRR 5 to 7, CRR 10 and 11).
- Three roofing membranes of unknown composition sold by two commercial companies (CRR 8, 9 and 12).

Table 1 sums up the theoretical composition of these mixes given by the producers.

### RESULTS OF THE TRIALS

#### Bitumen-Polymer and Bitumen-Polymer-Mineral Filler Mixes

Table 2 presents the individual values obtained when applying the operating method. It can be noticed that the results of these determinations are very satisfactory, given the complexity of the analysis requiring a great deal of manipulations, each of which introduces its error.

It is commonly admitted that the determination of asphaltenes is spoiled by 10 percent error in relative value<sup>4,6</sup> whereas one can notice that the values given for the insoluble part in normal heptane (see Table 2) are generally below this tolerance.

It seems that some dispersions observed on samples CRR 7, 10 and 11 should be due to the heterogeneity of the different samples taken.

The interpretation of the elements obtained can be found in Table 3 showing the contents of the different constituents (mineral filler, bitumen, atactic and isotactic polypropylene) calculated starting from the average analytical values according to the operating method.

The composition of the bitumen-polymer mixes offer a good correspondence with the theoretical compositions shown in Table 1. The sum of their components is very close to 100 percent.

#### Roofing Membranes

The results of the analyses performed in three samples of roofing sheets form the subject of the Table 4 (which contains the individual values and the calculated averages). The results are also satisfactory for this type of sample.

The insoluble part in xylene was calcinated under 500°C and under 900°C. The final residue was treated in 1/3 hydrochloric acid according to the remark in the operating method.

The sum of the calcium carbonate with the silica gives values that are much above the ones found with calcination at 500°C. When the mineral filler is made of limestone there is a noticeable agreement among the results.

This difference is due to the fact that the silicious part contains salts that are dissolved when attacked by hydrochloric acid and when calcinated at 900°C, the silica loses its constitution or crystallization water. This extra control is

therefore only advisable when the mineral filler is made of limestone. The results of the analyses are interpreted in Table 5.

### CONCLUSIONS

When combined with bitumen, APP is very difficult to detect and all the more so difficult to determine directly via infrared spectrography.

To determine the composition of a bitumen modified by APP or the composition of a roofing membrane made of a bitumen-atactic polypropylene mix, it is necessary to resort to analytic methods of separation comprising gravimetry, pyrolysis and infrared spectrography.

In the first stage, the inert parts of the membrane (reinforcement, mineral filler, isotactic polypropylene) are separated after a selective dissolution in xylene. A calcination at 500°C of the insoluble part distinguishes the mineral filler from the isotactic polypropylene. A second calcination at 900°C will finally reveal the calcareous nature of the mineral filler.

The separation of the asphaltenes from the bitumen with normal heptane concentrates the APP in the maltenic part and facilitates the precipitation of the APP by the mix of diethyl ether-methyl alcohol and the determination of the APP still soluble by the infrared spectrography.

The results of the different parts are representative of the material analyzed and give back its composition correctly.

The differences noticed in the results are mostly due to the heterogeneity of the analyzed sample.

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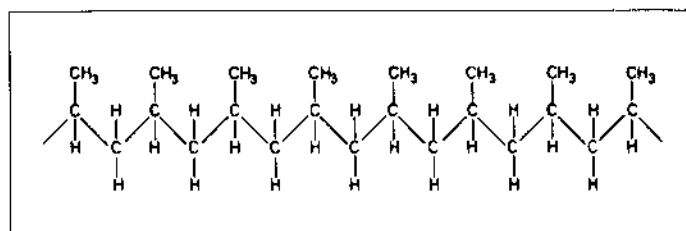


Figure 1 Isotactic polypropylene structure of the polypropylene.

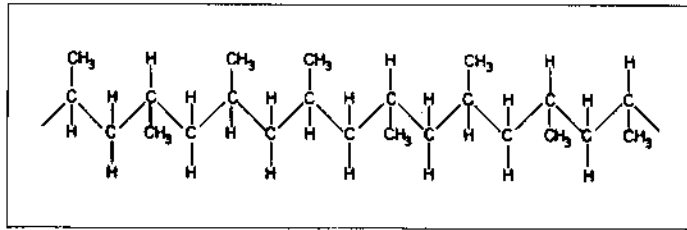


Figure 2 Atactic structure of the polypropylene.

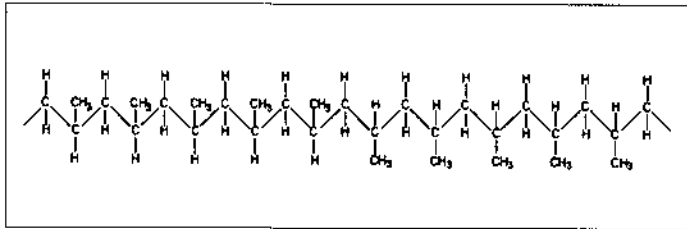


Figure 3 Syndiotactic structure of polypropylene.

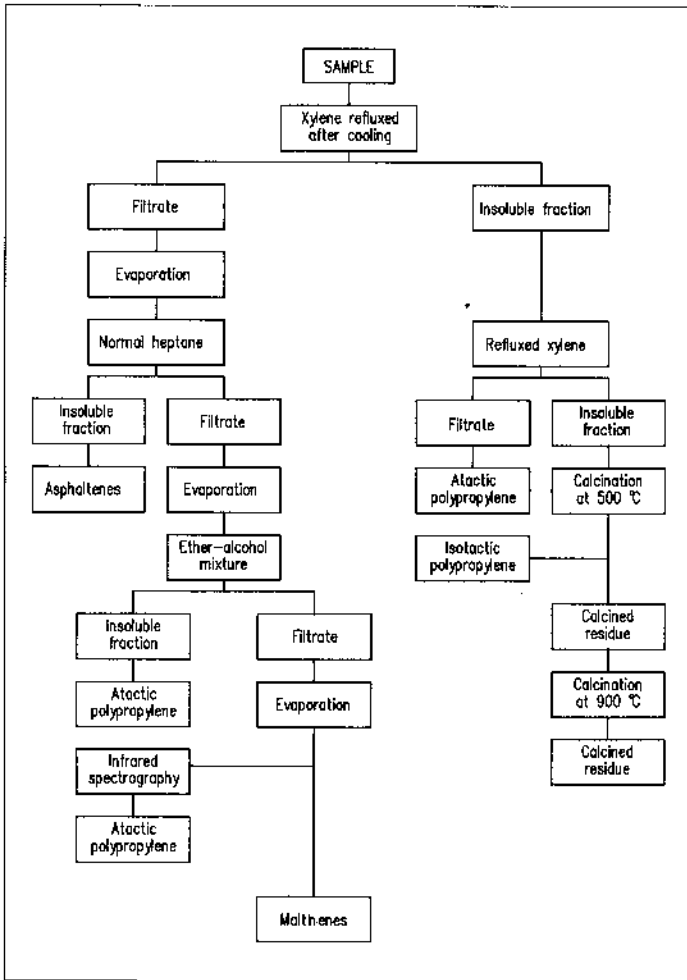


Figure 4 Schematic flow diagram of the procedure for determining atactic and isotactic polypropylene in bituminous mixes.

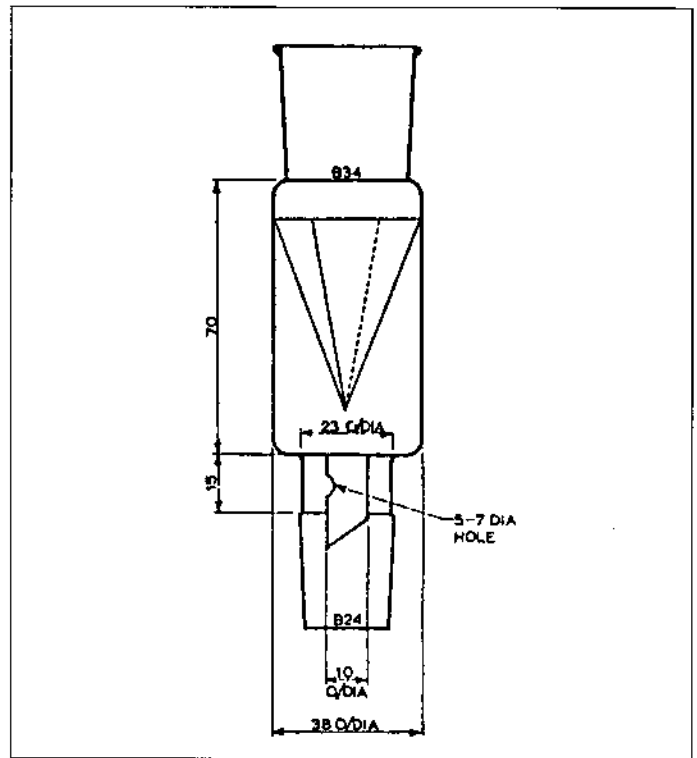


Figure 5 Extractor.

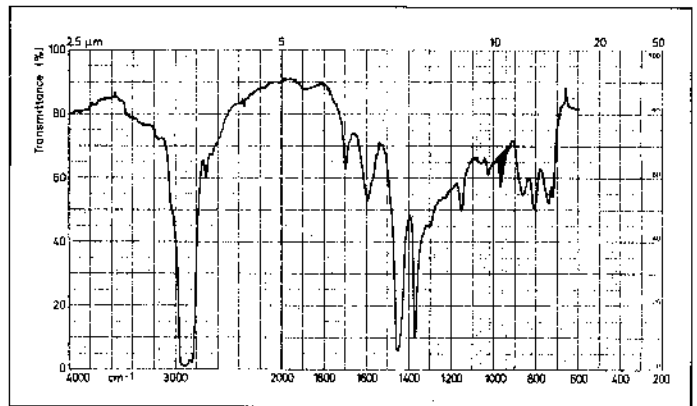


Figure 6 Spectrum of the malthenic fraction of a bitumen containing atactic polypropylene.

## Legend of the columns:

- Column 1: insoluble part in xylene after cooling  
 Column 2: soluble part of 1 after treatment in xylene under reflux  
 Column 3: residue of calcination at 500°C  
 Column 4: insoluble part in normal heptane  
 Column 5: Insoluble part in ether-alcohol mix  
 Column 6: soluble part ether-alcohol mix  
 Column 7: soluble APP in ether-alcohol (measured by IR)  
 Column 8: APP content (column 7) related to the sample

N°	1 %	2 %	3 %	4 %	5 %	6 %	7 %	8 %
CRR 1	-	-	-	10.3	20.5	71.0	13.3	9.4
	-	-	-	10.7	18.5	71.8	13.5	9.7
	-	-	-	10.7	20.9	70.4	15.0	10.0
Average	-	-	-	10.6	20.0	71.1	13.9	9.9
CRR 2	-	-	-	11.6	19.9	69.2	14.5	10.0
	-	-	-	11.6	19.7	69.4	15.5	10.7
	-	-	-	11.4	19.1	70.0	15.5	10.9
Average	-	-	-	11.5	19.6	69.5	15.2	10.5
CRR 3	-	-	-	11.4	23.6	65.9	10.7	7.0
	-	-	-	12.1	22.9	65.9	-	6.1
	-	-	-	11.8	23.9	65.3	8.6	5.6
Average	-	-	-	11.8	23.5	65.7	9.7	6.2
CRR 4	4.4	-	-	15.3	-	-	-	-
	5.1	-	-	15.0	-	-	-	-
	4.7	-	-	14.8	-	-	-	-
	6.0	-	-	15.1	-	-	-	-
	5.3	-	-	14.9	-	-	-	-
	4.7	-	-	15.0	-	-	-	-
Average	5.0	-	-	15.0	-	-	-	-
CRR 5	15.7	4.4	9.9	10.1	14.1	59.9	11.1	6.7
	15.7	3.7	10.2	9.9	14.2	60.6	11.4	6.9
	15.4	4.2	10.1	10.3	14.1	63.7	14.2	9.0
Average	15.6	4.1	10.0	10.1	14.1	61.4	12.2	7.5
CRR 6	15.5	3.4	10.8	10.8	14.2	58.8	11.3	6.6
	15.7	4.1	10.6	9.5	12.1	61.9	11.7	7.2
	15.5	3.6	10.2	10.4	12.3	62.9	13.4	8.4
Average	15.6	3.7	10.5	10.2	12.9	61.2	12.1	7.4
CRR 7	26.1	4.4	21.2	9.1	13.3	51.8	11.0	5.7
	26.6	4.1	21.9	8.4	13.7	51.7	11.1	5.7
	26.5	4.4	21.5	8.4	14.1	51.6	9.6	5.0
	25.1	3.4	20.6	8.8	14.7	51.0	15.0	7.7
	26.3	3.5	21.3	7.5	14.5	51.1	13.1	6.7
	25.0	4.2	19.9	8.6	14.3	51.8	10.3	5.3
	24.2	3.7	19.2	10.5	14.7	51.4	11.3	5.8
	24.5	3.8	19.5	9.1	14.7	51.7	11.7	6.1
	26.3	4.5	20.9	8.6	13.8	51.7	9.8	5.1
Average	25.6	4.0	20.7	8.8	14.2	51.5	11.4	5.9
CRR 10	7.3	7.4	0	8.1	13.2	70.8	11.9	8.4
	8.0	7.7	0	8.8	14.1	69.5	11.4	7.9
	6.8	7.1	0	7.6	14.2	71.6	10.9	7.8
	6.6	6.6	0	8.1	13.9	72.3	11.1	8.0
	7.1	7.1	0	7.0	14.1	72.1	11.5	8.3
	6.8	6.8	0	7.7	15.5	70.3	10.9	7.7
Average	7.1	7.1	0	7.9	14.2	71.1	11.3	8.0
CRR 11	17.6	6.8	10.7	4.8	12.7	62.9	12.3	7.7
	15.9	6.0	9.9	5.3	12.0	67.2	12.8	8.6
	16.1	6.8	9.3	3.9	13.3	66.8	12.0	8.0
	16.5	6.6	9.9	4.1	11.1	68.4	13.7	9.3
Average	16.5	6.6	9.9	4.5	12.3	66.3	12.7	8.4

Table 2 Composition of the bitumen-polymer mixes (per analysis, in percent).

N°	Mineral filler (%)	Bitumen (%)	APP (%)	IPP (%)	Total (%)
CRR 1	-	71.8	29.9	0.0	101.7
CRR 2	-	70.5	30.1	0.0	100.6
CRR 3	-	71.3	29.7	0.0	101.0
CRR 4	-	95.0	-	5.0	100.0
CRR 5	10.0	64.0	25.8	1.5	101.3
CRR 6	10.5	64.0	24.0	1.4	99.9
CRR 7	20.7	54.4	24.1	0.9	100.1
CRR 10	-	70.9	29.3	0.0	100.2
CRR 11	9.9	62.4	27.3	0.0	99.6

Table 3 Composition of bitumen-polymer (per analysis).

Legend of the columns:

Column 1 : reinforcement

Column 2 : insoluble part in xylene after cooling

Column 3 : soluble part of 2 after treatment in xylene under reflux

Column 4 : residue of calcination at 500°C

Column 5 : residue of calcination at 900°C

Column 6 : insoluble part of the calcination residue at 900°C in HCl 1/3

Column 7 : insoluble part in normal heptane (asphaltenes)

Column 8 : insoluble part in ether-alcohol mix

Column 9 : soluble part in ether-alcohol mix (malthenes + APP)

Column 10 : content in soluble APP in ether-alcohol mix (IR spectrography), related to the sample

N°	1 (%)	2 (%)	3 (%)	4 (%)	5 (%)	6 (%)	7 (%)	8 (%)	9 (%)	10 (%)
CRR 8	6.1	16.9	4.5	10.8	7.5	2.6	9.3	13.6	52.2	3.2
	5.5	15.9	3.7	10.0	6.7	1.9	9.1	14.1	53.2	3.5
	5.5	16.7	3.9	10.8	7.4	2.5	9.0	13.3	52.9	2.7
	5.5	15.8	4.7	10.9	7.4	2.4	10.2	13.1	55.9	4.1
	5.5	14.8	4.0	10.9	7.3	2.2	11.9	13.2	54.8	5.4
	5.8	16.0	4.9	11.0	7.4	2.4	9.5	13.4	55.7	5.1
	5.8	16.7	4.6	10.5	7.0	2.1	9.2	11.0	60.3	6.5
	6.0	16.2	5.3	10.3	6.9	2.0	10.1	10.8	58.3	6.3
	6.4	15.8	5.2	10.2	6.7	1.8	10.5	10.7	58.4	6.7
	5.5	14.8	4.2	10.1	6.6	1.6	8.6	13.6	57.8	6.4
	5.8	15.5	4.5	10.7	7.2	2.3	8.5	13.5	56.3	4.9
Average	5.8	15.9	4.5	10.5	7.1	2.2	9.5	12.8	56.0	4.9
CRR 9	4.2	14.3	6.1	7.6	7.4	7.1	12.5	11.7	58.2	3.9
	6.4	14.2	4.0	8.2	8.0	7.7	13.2	11.3	56.8	5.0
	5.0	14.3	4.8	8.0	7.8	7.5	12.7	11.3	57.0	4.4
Average	5.2	14.3	5.0	7.9	7.7	7.4	12.8	11.4	57.4	3.3
CRR 12	5.3	24.8	3.4	21.4	18.4	11.5	13.5	7.0	50.1	5.2
	4.7	26.1	3.5	22.7	19.6	12.9	12.6	8.5	48.8	5.1
	5.3	24.8	3.3	21.4	18.6	12.4	-	-	-	-
	5.1	25.7	3.6	22.1	19.7	13.2	10.9	7.0	51.4	5.3
	5.3	25.6	3.8	21.8	19.4	13.5	11.9	6.6	49.8	4.8
	5.8	24.1	2.9	21.2	18.6	13.1	13.6	6.8	49.4	4.9
Average	5.2	25.2	3.4	21.8	19.1	12.8	12.5	7.2	49.9	5.1

Table 4 Composition of the roofing sheets (per analysis).

1. Sample					
N°	Reinforcement	Mineral filler	Bitumen	Polypropylene	
	(%)	(%)	(%)	Atactic (%)	Isotactic (%)
CRR 8	6.1	10.8	58.3	21.3	1.6
	5.5	10.0	58.8	21.3	2.2
	5.5	10.8	59.2	19.9	2.0
	5.5	10.9	62.0	21.9	0.2
	5.5	10.9	61.3	22.6	0.0
	5.8	11.0	60.1	23.4	0.1
	5.8	10.5	63.0	22.1	1.6
	6.0	10.3	62.1	22.4	0.6
	6.4	10.2	62.2	22.6	0.4
	5.5	10.1	60.0	24.2	0.5
	5.8	10.7	59.9	22.9	0.3
	5.6	10.5	60.6	22.7	0.4
Average	5.8	10.5	60.6	22.3	.09
Standard deviation	0.3	0.3	1.5	0.8	1.1
CRR 9	4.2	7.6	66.8	21.7	0.6
	6.4	8.2	65.0	20.3	2.0
	5.0	8.0	65.3	20.5	1.5
Average	5.2	7.9	65.7	20.8	1.4
CRR 12	5.3	21.4	58.4	15.6	0
	4.7	22.7	56.3	17.1	0
	5.3	21.4	-	-	0
	5.1	22.1	57.0	15.9	0
	5.3	21.8	56.9	15.2	0
5.8	21.2	58.1	14.6	0	
Average	5.2	21.8	57.3	15.7	0
2. Composition of the mixes used (reinforcement deducted)					
N°	Mineral filler	Bitumen	Polypropylene		Total (%)
	(%)	(%)	Atactic (%)	Isotactic (%)	
CRR 8	11.2	64.3	23.7	1.2	100.4
CRR 9	8.3	69.3	21.8	1.5	100.9
CRR 12	23.0	60.5	16.5	0	100.0

Table 5 Composition of the roofing sheets (interpretation of the results).