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BDX-613-1905 (Rev.)

INVESTIGATION OF METHYLPENTENE POLYMER (TPX)
MOLDING RESIN

By C. L. Walter
Department 814

Published March 1978

Final Report

Prepared for the United States Department of Energy
Under Contract Number EY-76-C-04-0613.

**Kansas City
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By C. L. Walter
Department 814

Published March 1978

✓ Final Report
C. L. Walter, Project Leader

Project Team:
F. N. Larsen

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BDX-613-1905 (Rev.), UNCLASSIFIED Final Report, Published
March 1978

Prepared by C. L. Walter, D/814

A polymethylpentene polymer (TPX) produced by Mitsui Petrochemical Industries, Ltd., Japan, was evaluated and compared to a TPX previously obtained from Imperial Chemical Industries, Ltd., England, who no longer produce TPX and have sold their manufacturing process for this polymer to Mitsui. The rheological, chemical, and physical properties of the materials were compared and the two materials were found to be essentially identical.

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SUMMARY

A polymethylpentene polymer (TPX), produced by Mitsui Petrochemical Industries, Ltd., Japan, was evaluated and compared to a previously used TPX from Imperial Chemical Industries, Ltd., England (ICI). ICI no longer produces TPX and have sold their manufacturing process for TPX to Mitsui Industries. Therefore, an investigation of the new Mitsui TPX was needed. Two grades of the Mitsui material and one from ICI were chemically characterized and their physical properties were evaluated.

Results of the elemental and ash analyses, outgassing products, and dynamic shear modulus testing clearly indicated the composition of Mitsui TPX grades RT-18 and RT-20 to be essentially the same as ICI's RT-18 TPX. In addition, molecular weight determinations and thermal analyses show no significant differences between Mitsui and ICI TPXs.

The flexural modulus of RT-20 is significantly lower than that of RT-18. Although the tensile strength is also lower, the ultimate elongation of RT-20 is higher than RT-18. The RT-20 is more flexible than RT-18 even without a higher additive level. This indicates RT-20 may be a copolymer. Solvent stress analyses did not detect any difference in stress-crack resistance between RT-18 and RT-20.

This study has shown that the chemical and physical properties of the Mitsui RT-18 are similar enough to ICI RT-18 that the two materials may be used interchangeably for the production of parts.

DISCUSSION

SCOPE AND PURPOSE

Poly (4-methylpentene-1), a rigid stereoregular polymer, designated TPX, will be injection molded to produce parts in the future. Prior to 1975, TPX was a product of the Imperial Chemical Industries, Ltd., England. The ICI polymer has been evaluated at Bendix Kansas City.¹ ICI stopped production of TPX in 1975 and the manufacturing rights were sold to Mitsui Petrochemical Industries Ltd., Japan, who prepared their own TPX.

The Mitsui TPX was evaluated to ensure that the quality and the chemical and physical properties are the same as the TPX previously obtained from ICI. Two grades of Mitsui TPX (RT-18 and RT-20) were compared to the ICI RT-18. The three materials were chemically characterized and their physical properties were evaluated. Also, two lots of Mitsui RT-20 were compared for lot-to-lot variations.

PRIOR WORK

As a part of a prior endeavor, ICI's TPX was evaluated and reported upon.¹ The material was found to be easily processed over a wide range of molding conditions. The chemical characteristics were investigated and the physical properties measured. High and anisotropic mold shrinkage was noted as being a potential drawback to molding precision parts.

ACTIVITY

The evaluation of the Mitsui TPX consisted of two parts. First, a chemical characterization study compared the Mitsui RT-18 and RT-20 with the previously evaluated ICI RT-18. Then, an injection molding and physical properties study of the same three materials was performed.

Molecular weight, elemental analyses, ash content, thermal analyses, outgassing products, solvent extraction for additives, melt flow rate, and dynamic shear modulus tests were performed to chemically characterize the polymer. The tensile strength, ultimate elongation, impact strength, flexural strength, flexural modulus, specific gravity, mold shrinkage, and heat deflection temperature were determined to appraise the physical properties of TPX.

In addition, a second lot of Mitsui RT-20 was purchased to evaluate lot-to-lot variations in both chemical and physical properties. The RT-20 is reported by the vendor to be a stress-crack resistant TPX grade as compared to the general purpose grade RT-18. Therefore, a stress-crack analysis study was performed on both the Mitsui RT-18 and RT-20.

Chemical Characterization

Elemental Analysis and Ash Content

The analysis for carbon, hydrogen, and nitrogen gave comparable values for all three materials. The theoretical or calculated values for polymethylpentene are 86.0, 14.0, and 0 percent for carbon, hydrogen, and nitrogen, respectively (Table 1).

Hydrogen values were slightly low as might be expected, since some residual vinyl groups ($-\text{CH}=\text{CH}-$) are present as confirmed by infrared spectra, and some replacement of hydrogen by oxygen due to slight oxidation may have occurred. The small nitrogen content found is not significant.

Chlorine and phosphorous are present but only in the 10 to 20 ppm range and are not significant. Sulfur is the major impurity at about 100 ppm and is caused by a common olefin polymer antioxidant dilaurylthiodipropionate (DLTP). At this level of sulfur the antioxidant is present at 0.1 to 0.2 percent.

The ash content for all four samples was consistent at the 40 to 50 ppm level indicating very clean material. The ash analysis shows only zinc as the major metallic impurity together with a few ppm of aluminum. These two elements are undoubtedly present as a result of a coordination catalyst system used for the TPX polymerization reaction.

The results of these elemental analyses and ash content clearly indicate the Mitsui material is identical to the ICI material and the levels of impurities are consistent between the different lots and grades and are very low.

Molecular Weight and Melt Flow Rate

The molecular weight and molecular weight distribution of the four materials were obtained by gel permeation chromatography. The melt flow rate tests were performed using a standard melt indexer at a temperature of 260°C and 5 kg load (Table 2). The vendor reports the nominal melt flow rate is 26 g/10 min for both RT-18 and RT-20.

The molecular weight for the four materials was essentially identical with the number average molecular weight ($\bar{M}_n$) ranging between 108,000 and 117,000. The weight average molecular weight ($\bar{M}_w$) varied from 490,000 to 610,000 and the dispersity or molecular weight distribution (MWD) was between 4.2 and 5.6. These values reflect a normal range expected for this olefin polymerization and are not significantly different. This relatively narrow molecular weight range is also reflected in the closeness of values reported for the melt flow rate (22.3 to 23.9 g/10 min). The Mitsui process appears to be well in control and no significant differences in molecular weight can be found between lots or grades.

Table 1. Elemental Analysis and Ash Content

Element	ICI TPX	Mitsui TPX*	
	RT-18	RT-18	RT-20-1
Elemental Analysis			
Carbon (Percent)	85.4	85.8	85.8
Hydrogen (Percent)	13.3	12.9	13.1
Nitrogen (Percent)	0.1	0.2	0.1
Chlorine (ppm)	<10	20	<10
Sulfur (ppm)	95	106	113
Phosphorous (ppm)	21	21	20
Ash Content (ppm)			
Total Ash	36	45	53
Zinc	Major component of ash		
Copper	0.02	0.05	0.07
Titanium	0.3	1	1
Silicon	0.2	0.4	0.2
Manganese	0.006	0.007	0.007
Magnesium	0.006	0.02	0.02
Chromium	0.01	0.02	0.02
Iron	0.09	0.13	0.19
Aluminum	1.4	2.8	3.1
Calcium	0.08	0.08	0.1

*The total ash content of RT-20-2 was 38 ppm.

Table 2. Molecular Weight and Melt Flow Rate

Definition	ICI TPX	Mitsui TPX		
	RT-18	RT-18	RT-20-1	RT-20-2
Melt Flow Rate (g/10 min)	23.0	22.3	23.5	23.9
Molecular Weight				
Number Average ($\bar{M}_n$)	108,200	111,500	116,600	115,900
Weight Average ($\bar{M}_w$)	610,000	562,800	606,600	488,700
Distribution (MWD)	5.6	5.0	5.2	4.2

Additive Identification

Solvent extraction was used to identify the additive and the amount of low molecular weight material present. A 16-hour Soxhlet extraction of a 20 gram sample, using acetone as the reflux solvent, gave values of weight percent extractable material of 0.36 for ICI RT-18, 0.28 for Mitsui RT-18, and 0.22 for Mitsui RT-20-1. An infrared spectrum of the extract confirmed the material to be DLTP.

Dilaurylthiodipropionate

DLTP is a solid with a very low vapor pressure, a molecular weight of 514, and a melting point of 34° to 40°C. It is added to the TPX to retard oxidation of the hydrocarbon during normal processing. The ICI RT-18 sample also indicated the presence of an amide (probably a stearamide slip agent) and some glycerol. These components were not found in the extractables from the Mitsui samples. The additive level estimated from the sulfur analysis (0.1 to 0.2 percent) is in reasonably good agreement with the percent extractables at 0.2 to 0.3 percent. There is undoubtedly some hydrocarbon material present in the extractions but the amounts are only a few tenths of one percent, which is very low for polyolefin polymers. Acetone will not dissolve high molecular weight polyolefin but will dissolve low molecular weight hydrocarbons below about 1000 molecular weight.

With the exception of the DLTP antioxidant, these TPX materials are relatively free of low molecular weight materials. There should be no problems with migration of low molecular weight material or from the covalent-bonded sulfur present in these materials from an aging or compatibility viewpoint.

Outgassing Characteristics

Mass spectrometry outgassing on bulk samples of the TPX material was performed using a direct probe-solids inlet system and scanning a temperature range of ambient to 300°C. The following data describe outgassing for the three materials.

ICI RT-18 TPX outgassing is trace throughout the range of 35° through 105°C. There is a trace of DLTP fragment $\text{S}-(\text{CH}_2\text{CH}_2\text{COOH})_2$ in this range. The outgassing increases at 110°C and peaks at 205°C. The outgassing gradually decreases on through 300°C. The major constituent from 110°C through 250°C is the DLTP. Trace stearic acid shows up around 205°C. There is some trace TPX pattern around 280°C.

Mitsui RT-18 TPX outgassing is trace from 30° through 105°C. The outgassing drops to nearly nothing at 105°C. The outgassed material is trace DLTP sulfide fragment. Outgassing slowly increases starting at 110°C, peaks around 250°C, and then decreases on through 300°C. The major constituent is the DLTP with the trace stearic acid.

Mitsui RT-20 TPX outgassing profile is basically identical to the preceding two TPX samples. There is trace outgassing from 45° through 105°C. Stearic acid is quite apparent over this temperature range, more so than for the previous two samples. There are also some DLTP fragments over this temperature range. Outgassing once again begins to increase after 110°C, peaking at around 250°C, and then begins to decrease through 300°C. Once again the DLTP is the major component from 110° through 300°C. Stearic acid is still detectable over this range.

There is essentially no outgassing of volatile components up to 100°C for these materials. From 110° to 250°C, the additive DLTP is fragmented and volatilizes. There is also evidence of stearic acid, probably from a stearamide slip agent, in all three materials, with apparently a significantly larger amount in the RT-20 grade. This may be the difference between the RT-18 and RT-20 grades. Stress-crack resistance is claimed for RT-20. However, it must be remembered that the mass spectrometer is a very sensitive detection system (parts per million and less) and is used primarily for a qualitative identification of volatile components. The levels of antioxidant and slip agent are still quite low and should present no problems in compatibility or migratory species (Table 3).

Thermal Analysis

The following methods were used to determine the thermal stability of the three materials. The results are given in Table 4.

Differential Scanning Calorimetry (DSC) was used to determine the melting point (T_m) and the onset of decomposition (T_d). Samples were tested in air and nitrogen atmospheres.

Thermogravimetric Analysis (TGA) was used to determine weight loss as a function of temperature with primary interest in the onset of decomposition (T_d). Samples were run in air and nitrogen atmospheres. Isothermal TGAs were also run to measure weight loss as a function of time at 150°C.

Thermomechanical Analysis (TMA) was used to determine the glass transition temperature (T_g) and the softening or melting point by using a loaded quartz penetrating probe and noting the change in probe displacement (ΔL) as a function of temperature. Samples were only tested in ambient air.

Table 3. Outgassing Characteristics

Test Temperature (°C)	ICI RT-18	Mitsui RT-18	Mitsui RT-20
30° to 110°C	Trace DLTP	Trace DLTP	Trace DLTP Stearic Acid
115° to 205°C	Increased DLTP		DLTP Trace Stearic Acid
210° to 250°C	DLTP Trace Stearic Acid	DLTP Trace Stearic Acid	Increased DLTP Trace Stearic Acid
260° to 300°C	Trace TPX	Decreased DLTP Stearic Acid	Increased DLTP

The DSC data show a melting point of 220° to 232°C which is not affected by the change in atmosphere. The same values are also indicated from the TMA data. There appears to be only slight differences between the Mitsui RT-18 (230° to 232°C) and the Mitsui RT-20 and ICI RT-18 (222° to 228°C). These differences could be attributed to differences in the amount of crystallinity reflected in perhaps different cooling rates from processing.

The decomposition temperatures from DSC and TGA are very consistent. With DSC, Td values at 255° to 258°C are slightly lower in air than the TGA Td values at 270° to 275°C. This is because the DSC will show the oxidation exotherm at a lower temperature than the temperature where the TGA indicates weight loss. In the nitrogen atmosphere, the situation is reversed with the Td from TGA at 320° to 340°C while the DSC values are 387° to 397°C. This is due to the difference in sensitivity of the two techniques to carbon-carbon rupture from thermal energy. In any case, the data show no significant differences in the materials. It should be noted the TMA gives evidence for a secondary transition (change in volume) at about 20° to 30°C.

Isothermal TGA shows that exposure to air or nitrogen at 150°C gives an insignificant amount of weight loss (less than 0.3 percent) up to 4 hours. The materials appear to be similar under these conditions. In summary, thermal analysis gives evidence of excellent thermal stability for these materials and no significant differences exist between the grades or manufacturers.

Table 4. Differential Scanning Calorimetry, Thermogravimetric Analysis, and Thermomechanical Analysis

Property	ICI	Mitsui	
	RT-18	RT-18	RT-20-1
DSC in Air			
T _m (°C)	228	232	222
T _d (°C)	258	255	255
DSC in N ₂			
T _m (°C)	228	232	220
T _d (°C)	387	394	397
TGA in Air			
T _d (°C)	270	275	275
TGA in N ₂			
T _d (°C)	320	340	340
TMA in Air			
T _g (°C)	28	31	18
T _m (°C)	223	230	223
TGA (Isothermal)*			
Weight loss in air (Percent)	<0.3	<0.2	<0.1
Weight loss in N ₂ (Percent)	<0.2	<0.1	<0.2

*Four hours at 150°C

Dynamic Mechanical Properties

The dynamic shear modulus of the three materials was determined using a Rheometrics Mechanical Spectrometer in the fixed torsional pendulum mode. The data at selected temperatures from -40° to 100°C for the elastic or storage shear modulus (G'), the viscous or loss shear modulus (G''), and the loss tangent (G''/G') are given in Table 5. These data show no real significant differences. The differences that do exist can probably be related to the variation in crystallinity. A plot of the storage shear modulus is shown for the ICI and Mitsui TPXs in Figure 1.

Table 5. Dynamic Shear Modulus

Test Temperature (°C)	ICI RT-18 (MPa)	Mitsui	
		RT-18 (MPa)	RT-20-1 (MPa)
Storage Shear Modulus (G')			
-40	966	975	902
0	852	855	773
40	145	186	111
100	32	53	32
Loss Shear Modulus (G'')			
-40	22	14	21
0	35	30	40
40	40	48	21
100	11	9.2	6
Loss Tangent (G''/G')			
-40	0.023	0.014	0.023
0	0.041	0.035	0.052
40	0.276	0.258	0.189
100	0.344	0.174	0.188

Physical Properties Study

Test Specimen Preparation

This portion of the study consisted of injection molding physical test specimens from Mitsui RT-18 and RT-20 along with ICIs RT-18 retained from a previous investigation of TPX (PDO 6984844). The molding conditions used for this study were determined from the previous ICI TPX endeavor and are representative conditions needed to produce quality parts. The significant molding parameters for molding the four lots of TPX are melt temperature of 280°C, mold temperature of 76°C, inject pressure of 31 MN/m², inject speed of 1 second fill time, and clamp pressure of 2.2 MN.

The test specimens were molded in a six-cavity mold (GenK-2590) using a Lester 2.2 MN injection molding press. The specimens were subjected to the following tests.

Figure 1. Dynamic Shear Modulus Versus Temperature

The 102-mm-diameter, 3-mm-thick, edge-grated disks were used for visual evaluation and mold shrinkage in parallel and perpendicular directions to material flow.

The 230-mm, end-gated, tensile test bars were used for tensile strength and ultimate elongation tests.

The 127-mm by 13-mm by 3-mm end-gated bars were used for Izod impact strength, flexural strength, flexural modulus tests, and solvent stress analysis.

The 127-mm by 13-mm by 6-mm bars were used for Izod impact strength, heat deflection temperature, and specific gravity tests.

Specimen Testing

Tensile strength, ultimate elongation, Izod impact strength, flexural strength, flexural modulus, specific gravity, and heat deflection temperature were tested using ASTM test procedures. The mold shrinkage measurements were obtained using an optical comparator and the factor (Fms) was determined from the formula

$$Fms = \text{mold diameter} - \text{specimen diameter} / \text{mold diameter}.$$

Warp and appearance tests were performed visually.

The physical properties obtained from the various tests performed on ICI RT-18, Mitsui RT-18, and the two lots of Mitsui RT-20 are shown in Table 6.

Physical Properties Data Analysis

When comparing the physical properties (Table 6) of ICI and Mitsui RT-18 TPX, it is evident there is little difference between these two materials. For most parameters the difference is less than 5 percent; however, where a difference does exist the Mitsui TPX has the higher value. This indicates Mitsui's new process is equal to ICI's. In addition to the above data, no differences in clarity, surface finish, or appearance were noted among the three materials or from lot-to-lot.

It should be reiterated that the mold shrinkage for TPX is anisotropic. The mold shrinkage factors are quite large, with approximately twice the shrinkage in the direction of material flow versus shrinkage perpendicular to material flow. This is opposite to the usual mold shrinkage of thermoplastic resins. In addition, the mold shrinkage factors for RT-20 are lower than those of RT-18.

There was no measurable warpage in any of the 102-mm diameter discs; TPX does not appear to warp from differential mold shrinkage. TPX will warp in thin flat sections when the two mold surfaces are at different temperatures.

The major difference found between RT-18 and RT-20 was flexural modulus. It is about 40 percent lower for RT-18. However, elongation is higher and the tensile strength is lower for RT-20. These differences are also reflected in the vendor's literature, indicating RT-20 is a more flexible polymer than RT-18. Because no differences in additives were found, coupled with higher weight average molecular weight and lower number average molecular weight, RT-20 could very possibly be a copolymer. A further investigation in this area is needed before a conclusion can be drawn.

Table 6. Physical Properties

ASTM Test	Property	ICI	Mitsui		
		RT-18	RT-18	RT-20-1	RT-20-2
D-638	Tensile Strength (MPa)	24.6	25.5	23.1	22.3
D-638	Ultimate Elongation (Percent)	29	31	41	55
D-790	Flexural Strength (MPa)	33.3	36.0	25.8	24.6
D-790	Flexural Modulus (GPa)	1.03	1.10	0.690	0.695
D-792	Specific Gravity (g/cm ²)	0.83	0.83	0.83	0.83
D-256	3 mm Izod Impact (J/m)	36.4	39.0	36.4	---
D-256	6 mm Izod Impact (J/m)	26.0	26.0	21.4	20.8
	Mold Shrinkage				
	Parallel to flow (mm/mm)	0.0225	0.0246	0.0175	0.0181
	Perpendicular to flow (mm/mm)	0.0125	0.0124	0.0077	0.0072
D-648	Heat Deflection Temperature (°C)	48	51	54	56

Lot-to-Lot Analysis

The lot-to-lot study was performed on two lots of RT-20. The data in Table 6 show a lot-to-lot variation of about 5 percent for tensile and flexural strengths and 30 percent variation in elongation; however, the other parameters have very minor variations. It was found in the previous study that the ultimate elongation was highly variable, both within a group of specimens molded under the same set of conditions and when molding parameters changed. Erratic elongation measurements in TPX could be caused by the material being notch sensitive. If it is notch sensitive, sample preparation and specimen quality are more important. TPX is also brittle; it will shatter when struck with a sharp blow. Therefore, the difference in these two lots is reasonable.

Stress Crack Analysis

A series of solvent stress studies did not show any advantage of RT-20 over RT-18 in stress-crack resistance. The comparative studies utilized tetrahydrofuran, toluene, benzene, chloroform, trichloroethane, methanol, and isopropanol. The 3-mm by 13-mm by 127-mm test specimens were deflected 25 mm to produce a 3 percent strain level and were exposed to the various solvents for a minimum of 60 minutes.

Moldability

The four lots of TPX were conditioned and injection molded as nearly alike as possible; no differences were noted in processing these lots. TPX is not difficult to mold; however, care must be taken in mold design to compensate for the differential mold shrinkage factors.

ACCOMPLISHMENTS

Mitsui RT-18 TPX was evaluated and the chemical and physical properties were determined to be similar enough to the ICI RT-18 that they may be used interchangeably. Molecular weight data and the elemental analyses indicate the two vendors' products are essentially identical. The additive identification and outgassing studies amplify that Mitsui's polymer manufacturing process probably is identical to ICI's. The physical properties add further proof to the similarity of the two companies' molding resins.

Significant chemical differences between Mitsui's RT-18 and RT-20 could not be found to substantiate the reported stress-crack resistance of the RT-20. However, differences in elongation and flexural modulus do exist between RT-18 and RT-20, indicating that RT-20 could be a copolymer; additional work would be required to verify the chemical structures.

FUTURE WORK

An investigation of the secondary transition phenomenon observed at ambient temperature as revealed by the dynamic shear modulus and thermomechanical analysis testing is being planned. It would be helpful to understand the origin of this transition from a polymer microstructure viewpoint and to know the variables such as mold temperature and annealing time on the magnitude of the loss of mechanical and physical properties. Further investigation of the polymeric structural differences between RT-18 and RT-20 should be made.

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A polymethylpentene polymer (TPX) produced by Mitsui Petrochemical Industries, Ltd., Japan, was evaluated and compared to a TPX previously obtained from Imperial Chemical Industries, Ltd., England, who no longer produce TPX and have sold their manufacturing process for this polymer to Mitsui. The rheological, chemical, and physical properties of the materials were compared and the two materials were found to be essentially identical.