Melt index

Melt index, I_2, was tested at 190 °C with a 2.16 kg weight according to ASTM D1238 (1999) and is reported herein as gram per 10 minutes (g/10 min).

Dynamic Mechanical Spectroscopy (DMS) Frequency Sweep

Resin samples were compression molded into 3 mm thick x 1 inch circular plaques at 350°F for 5 minutes under 10342 kilopascal (1500 psi) pressure in air. The compression molded samples were removed from the press and allowed to cool at ambient temperatures in air.

A constant temperature frequency sweep was performed using an Advanced Rheometric Expansion System ("ARES"), available from TA Instruments, equipped with 25 mm parallel plates, under a nitrogen purge. The sample was placed on the plate and allowed to melt for five minutes at 190°C. The plates were then closed to 2 mm gap, the sample trimmed, and the test started following a five minute delay, to allow for temperature equilibration. The tests were performed at 190 °C over a frequency range of 0.1 to 100 rad/s. The strain amplitude was constant at 10%. The stress response was analyzed in terms of amplitude and phase, from which the storage modulus (G'), loss modulus (G"), complex modulus (G*), dynamic viscosity η^*, and tan (δ) or tan(delta) were calculated.

Conventional GPC $M_{w\text{-}gpc}$ determination

To obtain $M_{w\text{-}gpc}$ values, the chromatographic system used was either a Polymer Laboratories Model PL-210™ or a Polymer Laboratories Model PL-220™. The column and carousel compartments were operated at 140 °C. Three Polymer Laboratories 10-μm Mixed-B columns were used employing 1,2,4-trichlorobenzene as solvent. The samples were prepared at a concentration of 0.1 g of polymer in 50 ml of solvent. The solvent used to prepare the samples contained 200 ppm of the antioxidant butylated hydroxytoluene (BHT) (i.e., 2,6-bis(1,1-dimethylethyl)-4-methylphenol). Samples were prepared by agitating lightly for 4 hours at 160 °C. The injection volume was 100 microliters and the flow rate was 1.0 ml/min. Calibration of the GPC columns was performed with twenty one narrow molecular weight distribution polystyrene standards, available from Varian, Inc. The polystyrene standard peak molecular weights were converted to polyethylene molecular weights using Equation 2:

$$M_{polyethylene}=A(M_{polystyrene})^B \quad \text{(Eq. 2)}$$

where M_x is the molecular weight of compound x, A is 0.4316 and B is 1.0. A third order polynomial is determined to build the logarithmic molecular weight calibration as a function of

elution volume. Polyethylene equivalent molecular weight calculations were performed using Viscotek TriSEC™ software Version 3.0. The precision of the weight-average molecular weight M_w was less than 2.6 %.

EPDM Composition

EPDM Composition was measured by FTIR as follows:

a. Ethylene content was measured according to ASTM D3900-05;
b. ENB (ethylidenenorborene) content was measured according to ASTM D6047-99; and
c. Propylene content (as % by weight) was calculated as 100 wt % – (Ethylene + ENB) where ethylene and ENB are expressed as weight percent.

Inventive Example 1 and Comparative Example 1

The composition of the procatalyst slurry used in Inventive Example 1 is shown in Table 1 and that of the procatalyst solution used in Comparative Example 1 is shown in Table 2. Such procatalyst preparations were each used in 3.79 liter (one gallon) batch polymerization reactors. The polymerization reactor conditions are given in

Table 1

Reactive and inert components		
Component	Inventive Example 1	Comparative Example 1
Solvent (g)	1329.5	1329.7
Ethylene (g)	107.2	105.7
Hydrogen (millimole)	20.1	20.0
Octene (g)	251.4	251.7
Max ethylene flow (g/min)	14.6	17.0
Reactor measurements made		
Catalyst efficiency (mass polymer/mass zirconium)	1102754	1132531
Initial Pressure (kilopascal (psi))	2961 (429.5)	2972 (431.1)
Temperature at start (°C)	190.0	189.9
Exotherm (°C)	6.00	6.30
Time of reaction (min)	10.0	10.0

In each of Inventive Example 1 and Comparative Example 1, the solvent used in batch polymerization reactor was Isopar™ E and the following procedure was followed: Solvent, Isopar E, was added to the 3.79 liter (one gallon) polymerization batch reactor, followed by addition of 1-octene and hydrogen with agitation at 1000 rpm; the reactor was heated to the start temperature and ethylene was added to achieve the initial pressure; the procatalyst slurry (for Inventive Example 1) or procatalyst solution (for Comparative Example 1) was prepared under a dry nitrogen atmosphere, injected into a sample loop, and the loop contents were then pumped into the reactor with additional high pressure Isopar E. As polymerization progressed, the temperature was maintained at the Initial Temperature and the Initial Pressure was maintained by adding ethylene to the batch reactor. Polymerization was permitted to proceed for ten minutes, at which time the ethylene flow was stopped and the reactor contents were emptied into a catch pot in which most of the ethylene and solvent flashed-off. The ethylene/octene copolymers were removed from the catch pot and the residual solvent removed from the copolymers in a vacuum oven (100°C for 24 hrs). The resins obtained from for each of Inventive Example 1 and Comparative Example 1 were analyzed and the results are listed in Table 4-7.

Table 8

	Inventive Example 2	**Comparative Example 2**
First Reactor Procatalyst Flow (g/hr (lbs/hr))	43 (0.95)	295 (0.65)
First Reactor Procatalyst Concentration (ppm)	7.0	7.0
First Reactor Cocatalyst Flow (g/hr (lbs/hr))	567 (1.25)	390 (0.86)
First Reactor Cocatalyst Concentration(ppm)	78	78
Second Reactor Procatalyst Concentration (ppm)	7	7
Second Reactor Cocatalyst Concentration (ppm)	78	78
Second Reactor Monomer Conversion (%)	67.5	64.8
Polymer made in First Reactor (%)	57	66
Production Rate (kg/hr (lb/Hr))	21.23 (46.8)	20.59 (45.4)
First Reactor Polymer (Wt %) (molten solids)	14.6	17.1
Second Reactor Polymer (Wt%) (molten solids)	13.0	12.6

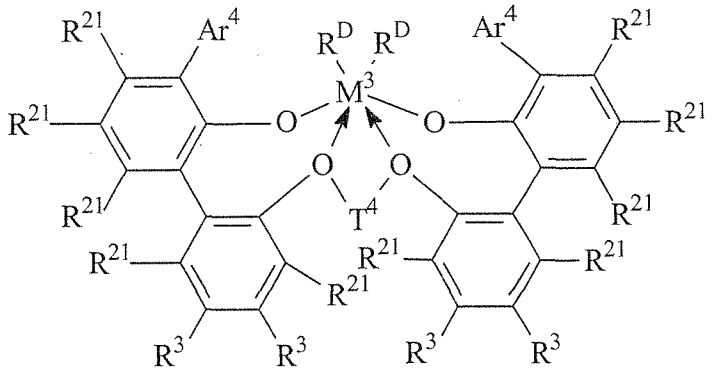
Table 9

	Inventive Example 2	**Comparative Example 2**
First Reactor Temperature, °C	141.84	142.42
First Reactor Pressure, kilopascal (psig)	3617 (524.56)	3623 (525.44)
First Reactor feed solvent flow, (kg/hr (lbs/hr))	69.13 (152.40)	69.2 (152.55)
First Reactor feed hydrogen flow, (standard cubic centimeters per minute (SCCM))	739.97	740.45
First Reactor ethylene flow, (kg/hr (lbs/hr))	9.66 (21.29)	9.68 (21.35)
First Reactor propylene flow, (kg/hr (lbs/hr))	5.09 (11.23)	5.35 (11.80)
First Reactor ENB flow, (g/hr (lbs/hr))	213 (0.47)	181 (0.40)
First Reactor feed total inlet flows, (kg/hr (lbs/hr))	81.74 (180.20)	81.42 (179.50)
Second Reactor feed hydrogen flow, (SCCM)	900.13	902.95
Second Reactor ethylene flow, (kg/hr (lbs/hr))	9.07 (19.99)	9.08 (20.01)
Second Reactor propylene flow, (kg/hr (lbs/hr))	6.35 (14.00)	5.03 (11.08)
Second Reactor ENB flow, (g/hr (lbs/hr))	172 (0.38)	113 (0.25)
Second Reactor total inlet flows, (kg/hr (lbs/hr))	162.35 (357.91)	159.72 (352.12)
Total Monomer Conversion, [%]	83.20	82.5
Second Reactor Monomer Conversion, [%]	67.53	69.55

The copolymer resins obtained from Inventive Example 2 and Comparative Example 2 were analyzed and the results are given in Table 10. The GPC traces (RI detector) of the copolymer produced in each of Inventive Example 2 and Comparative Example 2 are shown in Fig. 2. DMS (Viscosity) data for Inventive Example 2 and Comparative Example 2 is shown graphically in Fig. 3.

We claim:

1. A procatalyst carrier system comprising one or more paraffinic solvents, one or more paraffin-insoluble procatalysts, and optionally one or more cocatalysts wherein the carrier system is in the form of a slurry; wherein the paraffin-insoluble procatalysts are not supported on inorganic materials and wherein the one or more paraffin-insoluble procatalysts are selected from the group of compounds according to the following formula:



wherein M^3 is Ti, Hf or Zr, preferably Zr; Ar^4 independently each occurrence is a substituted C_{9-20} aryl group, wherein the substituents, independently each occurrence, are selected from the group consisting of alkyl; cycloalkyl; and aryl groups; and halo-, trihydrocarbylsilyl- and halohydrocarbyl- substituted derivatives thereof, with the proviso that at least one substituent lacks co-planarity with the aryl group to which it is attached; T^4 independently each occurrence is a C_{2-20} alkylene, cycloalkylene or cycloalkenylene group, or an inertly substituted derivative thereof; R^{21} independently each occurrence is hydrogen, halo, hydrocarbyl, trihydrocarbylsilyl, trihydrocarbylsilylhydrocarbyl, alkoxy or di(hydrocarbyl)amino group of up to 50 atoms not counting hydrogen; R^3 independently each occurrence is hydrogen, halo, hydrocarbyl, trihydrocarbylsilyl, trihydrocarbylsilylhydrocarbyl, alkoxy or amino of up to 50 atoms not counting hydrogen, or two R^3 groups on the same arylene ring together or an R^3 and an R^{21} group on the same or different arylene ring together form a divalent ligand group attached to the arylene group in two positions or join two different arylene rings together; and R^D, independently each occurrence is halo or a hydrocarbyl or trihydrocarbylsilyl group of up to 20 atoms not counting hydrogen, or 2 R^D groups together are a hydrocarbylene, hydrocarbadiyl, diene, or poly(hydrocarbyl)silylene group.

2. The procatalyst carrier system according to Claim 1 wherein the one or more paraffin-insoluble procatalysts are selected from the group of transition metal organo-metallic compounds

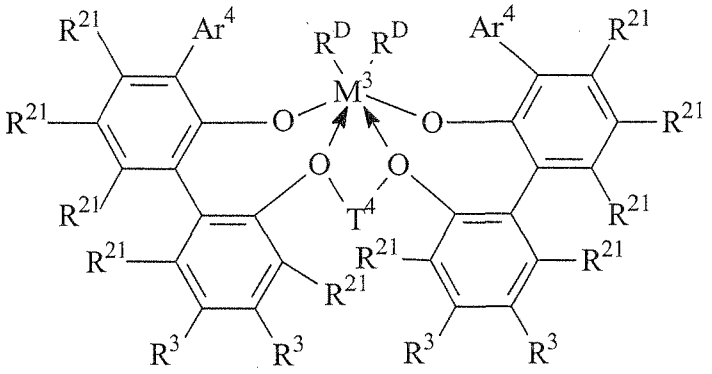
adding the one or more procatalysts to a sufficient quantity of paraffinic solvent to form a slurry of the one or more procatalysts in the paraffinic solvent;

introducing one or more first cocatalysts into a polymerization reactor; and

introducing the slurry into the polymerization reactor;

wherein the paraffin-insoluble procatalysts are not supported on inorganic materials and

wherein the one or more paraffin-insoluble procatalyst is selected from the group of compounds according to the following formula:



wherein M^3 is Ti, Hf or Zr, preferably Zr; Ar^4 independently each occurrence is a substituted C_{9-20} aryl group, wherein the substituents, independently each occurrence, are selected from the group consisting of alkyl; cycloalkyl; and aryl groups; and halo-, trihydrocarbylsilyl- and halohydrocarbyl- substituted derivatives thereof, with the proviso that at least one substituent lacks co-planarity with the aryl group to which it is attached; T^4 independently each occurrence is a C_{2-20} alkylene, cycloalkylene or cycloalkenylene group, or an inertly substituted derivative thereof; R^{21} independently each occurrence is hydrogen, halo, hydrocarbyl, trihydrocarbylsilyl, trihydrocarbylsilylhydrocarbyl, alkoxy or di(hydrocarbyl)amino group of up to 50 atoms not counting hydrogen; R^3 independently each occurrence is hydrogen, halo, hydrocarbyl, trihydrocarbylsilyl, trihydrocarbylsilylhydrocarbyl, alkoxy or amino of up to 50 atoms not counting hydrogen, or two R^3 groups on the same arylene ring together or an R^3 and an R^{21} group on the same or different arylene ring together form a divalent ligand group attached to the arylene group in two positions or join two different arylene rings together; and R^D, independently each occurrence is halo or a hydrocarbyl or trihydrocarbylsilyl group of up to 20 atoms not counting hydrogen, or 2 R^D groups together are a hydrocarbylene, hydrocarbadiyl, diene, or poly(hydrocarbyl)silylene group.

6. The process according to Claim 5 wherein the one or more organometallic procatalysts are selected from the group of transition metal organometallic compounds which catalyze the