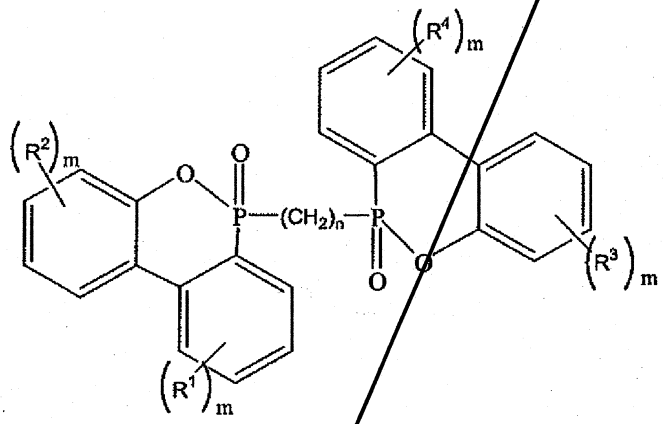
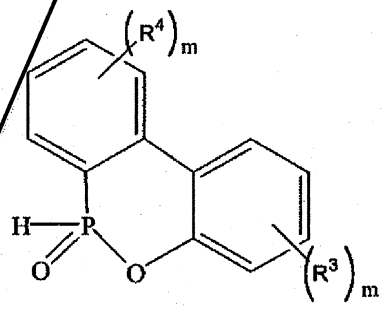
CLAIMS

1. A process for preparing the compound of Formula I:



Formula I

wherein each R^1, R^2, R^3 and R^4 are independently hydrogen, C_1-C_{15} alkyl, C_6-C_{12} aryl, C_7-C_{15} aralkyl or C_7-C_{15} alkaryl; or R^1 and R^2 or R^3 and R^4 taken together can form a saturated or unsaturated cyclic ring, wherein said saturated or unsaturated cyclic ring may be optional substituted by a C_1-C_6 alkyl; each m is independently 1, 2, 3 or 4; and n is 2 to about 18; comprising reacting a compound of Formula A:



Formula A

wherein R^3, R^4 and m are defined above;

with a diol compound of Formula B in the presence of a catalyst, optionally a solvent, and optionally an entrainer;

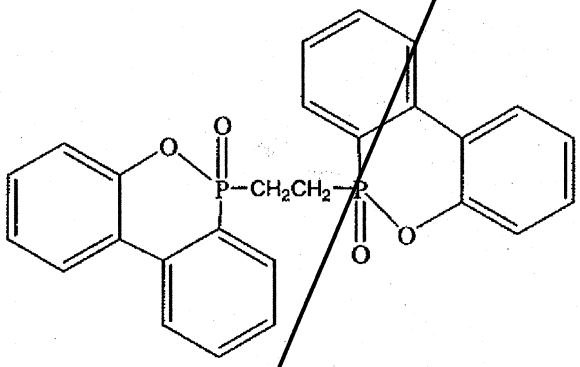
$$HO-(CH_2)_n-OH$$

Formula B

wherein n is defined above.

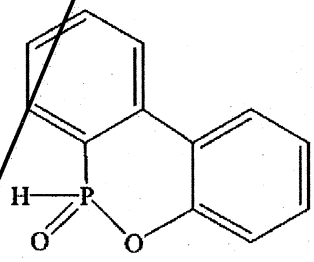
2. The process of Claim 1, wherein n is 2 to 6 and R^1, R^2, R^3 and R^4 are hydrogen.

3. A process for preparing the compound of Formula II:



Formula II

comprising reacting a compound of Formula C:



Formula C

with ethylene glycol in the presence of a catalyst, optionally a solvent and optionally an entrainer.

4. The process of Claims 1 or 3, wherein the entrainer is present.

5. The process of Claims 1 or 3, wherein the solvent is present.

6. The process of Claims 1 or 3, wherein both the solvent and the entrainer are present.

7. The process of Claims 1 or 3 wherein said catalyst is an alky halide, alkali halides, alkaline earth metal halides, transition metals and their halides or acid catalysts.

8. The process of Claims 1 or 3, wherein said catalyst is sodium iodide, lithium bromide, lithium chloride, potassium iodide, potassium bromide, lithium iodide, C_1-C_6 alkyl iodide, C1-C_6 alkyl bromide, 2-iodoethanol, 2-bromoethanol, 2-chloroethanol, 3-iodopropanol, 3-bromopropanol, ferric bromide, ferrous chloride, ferrous bromide, manganous halide, copper powder, nickel halide, cobalt chloride, cesium bromide, palladium chloride, sulfuric acid, aryl sulfonic acid, alkyl sulfonic acid, arylalkyl sulfonic acid, hydrochloric acid, hydrobromic acid, hydrofluoric acid, oxalic acid, perchloric acid, trifluoromethane sulfonic acid, fluorosulfonic acid, nitric acid, aluminum chloride, diethyl aluminum chloride, triethylaluminum/hydrogen chloride, ferric chloride, zinc chloride, antimony trichloride, stannic chloride, boron trifluoride, acidic zeolites, acidic clays, polymeric sulfonic acids, or mixtures thereof.

9. The process of Claim 5, wherein the solvent is a high boiling point solvent, wherein the high boiling point solvent has a boiling point greater than about 150°C at 1 atmosphere and melting point greater than about -100°C.

10. The process of Claim 9, wherein the high boiling point solvent is diphenylmethane, diphenylethane, diphenylpropane, biphenyl, decahydronaphthalene, cyclohexylbenzene, 1,3-diisopropylbenzene, dimethyl sulfoxide (DMSO), dimethyl formamide (DMF), dimethylacetamide (DMAC), ethylene glycol dimethyl ether, ethylene glycol diethyl ether or mixtures thereof.

11. The process of Claim 5, wherein the process is conducted greater than about 1 atmospheric pressure and the solvent is a low boiling point solvent having a boiling point less than about 150°C at 1 atmosphere.

12. The process of Claim 11, wherein the solvent is heptane, hexane, petroleum ether, methylcyclohexane; toluene, xylene, mesitylene, ethyl benzene, tetrahydrofuran, 1,4-dioxane, acetonitrile or mixtures thereof.

13. The process of Claims 1 or 3, wherein the temperature of the reaction ranges from about 100°C to about 250°C.

14. The process of Claims 1 or 3, wherein the process is carried out in a continuous mode.

15. The process of Claims 1 or 3, wherein the process is carried out in a batch mode.

16. The process of Claims 1 or 3, wherein the a product of the reaction is water and wherein the water and the diol compound of Formula B or ethylene glycol are continuously recycled back to reactor.

17. The process of Claim 6, wherein the process is conducted at the normal pressure and the entrainer is continuously recycled back to reactor.

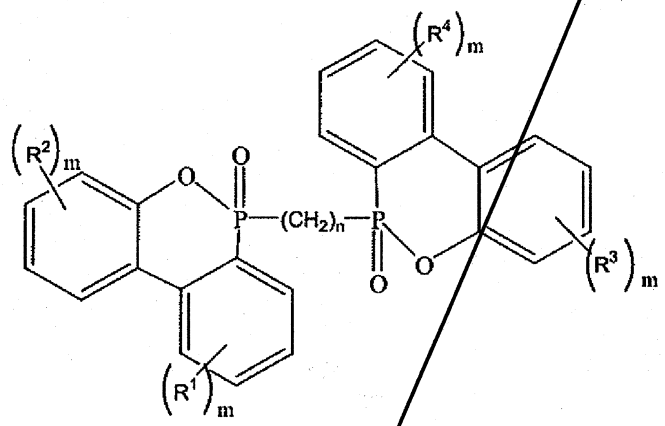
18. The process of Claims 1 or 3, wherein the entrainer is a low boiling point solvent, wherein the low boiling point solvent has a boiling point lower than about 150°C.

19. The process of Claim 18, wherein the low boiling point solvent is pentane, hexane, heptane, octane, cyclohexane, methylcyclohexane, toluene, xylenes, ethylbenzene, isopropylbenzene or mixtures thereof.

20. The process of Claim 1 or 3, wherein the catalyst is an acid catalyst.

21. The process of Claim 20, wherein said acid catalyst is sulfuric acid, aryl sulfonic acid, alkyl sulfonic acid, aralkyl sulfonic acid, hydrochloric acid, hydrobromic acid, hydrofluoric acid, oxalic acid, perchloric acid, trifluoromethane sulfonic acid, fluorosulfonic acid, nitric acid, phosphoric acid, phosphonic acids, phosphinic acids aluminum chloride, diethyl aluminum chloride, triethylaluminum/hydrogen chloride, ferric chloride, zinc chloride, antimony trichloride, stannic chloride, boron trifluoride, acidic zeolites, acidic clays, polymeric sulfonic acids, or mixtures thereof.

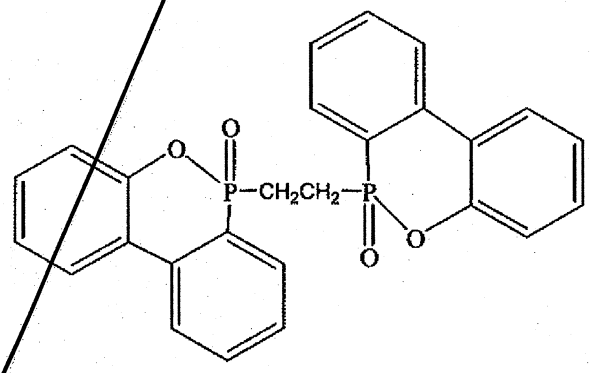
21. The compound of Formula I:



Formula I

wherein each R^1, R^2, R^3 and R^4 are independently hydrogen, C_1-C_{15} alkyl, C_6-C_{12} aryl, C_7-C_{15} aralkyl or C_7-C_{15} alkaryl; or R^1 and R^2 or R^3 and R^4 taken together can form a saturated or unsaturated cyclic ring, wherein said saturated or unsaturated cyclic ring may be optional substituted by a C_1-C_6 alkyl; each m is independently 1, 2, 3 or 4; and n is 2 to about 18: produced by the process of Claim 1.

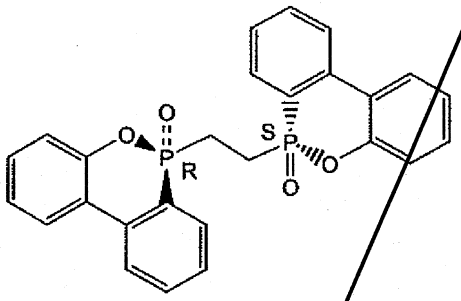
23. The compound of Formula II:



Formula II

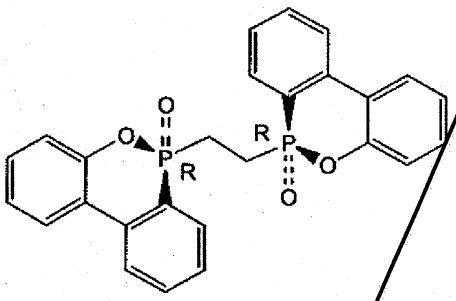
produced by the process of Claim 3.

24. A composition comprising the diastereomer of Formula IIa:

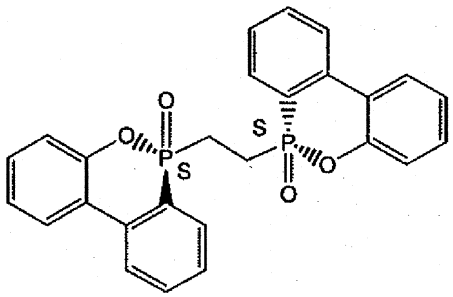


Formula IIa

25. The composition of Claim 24, further comprising enantiomers having the following Formulas:



Formula IIb



Formula IIc

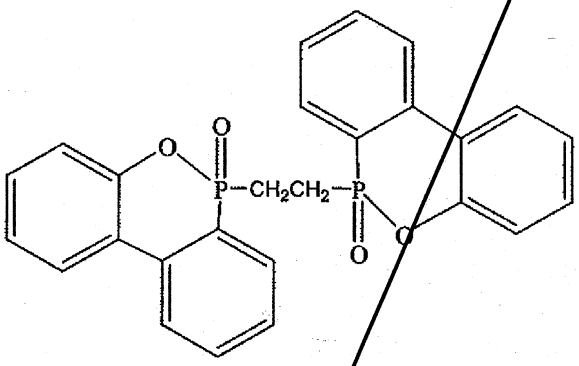
26. The composition of Claim 24, wherein a plurality of the compounds in the composition is the diastereomer of Formula IIa.

27. The composition of Claim 24 having an Isomer Ratio of greater than 0.5 utilizing the 31P NMR method.

28. The composition of Claim 24, having an Isomer Ratio of greater than 0.8 utilizing the 31P NMR method.

29. The composition of Claim 24, having an Isomer Ratio of greater than 0.95 utilizing the 31P NMR method.

30. A method for producing a higher melting point composition comprising the compound of Formula II:



Formula II

comprising contacting a composition containing lower amounts of high melting point isomers of Formula II, with alcohols, water, or mixtures thereof in the presence of an acid catalyst, thereby producing a composition containing larger amounts of higher melting point isomers of Formula II.

31. The composition of Claim 30, wherein said acid catalyst is sulfuric acid, aryl sulfonic acid, alkyl sulfonic acid, aralkyl sulfonic acid, hydrochloric acid, hydrobromic acid, hydrofluoric acid, oxalic acid, perchloric acid, trifluoromethane sulfonic acid, fluorosulfonic acid, nitric acid, phosphoric acid, phosphonic acids, phosphinic acids aluminum chloride, diethyl aluminum chloride, triethylaluminum/hydrogen chloride, ferric chloride, zinc chloride, antimony trichloride, stannic chloride, boron trifluoride, acidic zeolites, acidic clays, polymeric sulfonic acids, or mixtures thereof.

32. A flame retardant polymer composition comprising a polymer and the compound of Claim 24.

33. The composition of Claim 32, wherein said polymer is polyolefins, polyesters, polyethers, polyketones, polyamides, polyvinylchlorides, natural and synthetic rubbers, polyurethanes, polystyrenes, poly(meth)acrylates, phenolic resins, polybenzoxazine, polyacetals, polyacrylonitriles, polybutadienes, polystyrenes, polyimides, polyamideimides, polyetherimides, polyphenylsulfides, polyphenylene oxide, polycarbonates, cellulose, cellulose derivatives, cyanate esters, polyphenylene esters, polybutadiene resins, butadiene-styrene resins, butadiene-divinylbenzene-styrene resins, epoxy-modified polybutadiene

resins, acrylic or vinyl acetate adhesives, carboxyl-terminated butadiene-acrylonitrile copolymers, phenylene ethers, maleic anhydride-grafted butadiene-styrene copolymers, maleic anhydride-modified 4-methyl-1pentene resins, maleated 1-butene-ethylene copolymers, resins derived from vinylbenzyl ether compounds, epoxy resins or mixtures thereof.

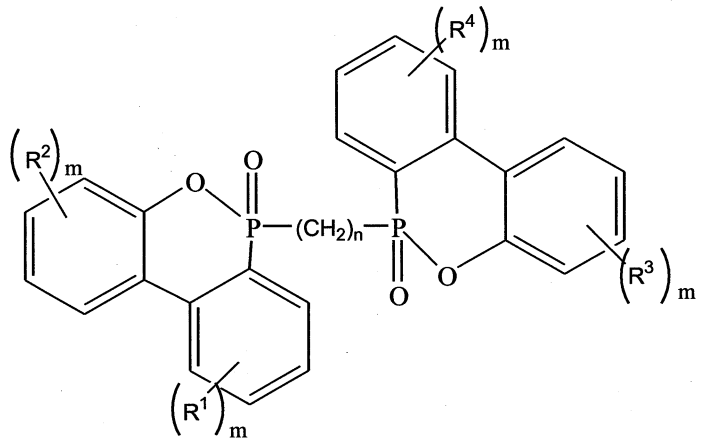
34. The composition of Claim 32, wherein said polymer is polyolefins, polyesters, phenolic resins, polyamides, polyurethanes, polystyrene, epoxy resins or mixtures thereof.

35. The composition of Claim 32, further comprising a melamine polyphosphate.

36. The composition of Claim 32, further comprising silica.

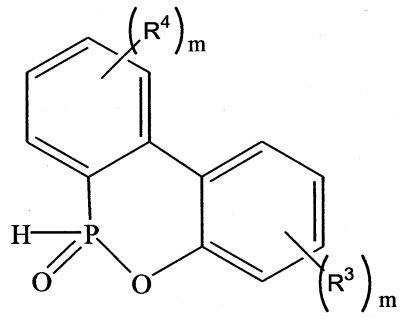
CLAIMS

1. A process for preparing the compound of Formula I:



Formula I

wherein each R^1, R^2, R^3 and R^4 are independently hydrogen, C_1-C_{15} alkyl, C_6-C_{12} aryl, C_7-C_{15} aralkyl or C_7-C_{15} alkaryl; or R^1 and R^2 or R^3 and R^4 taken together can form a saturated or unsaturated cyclic ring, wherein said saturated or unsaturated cyclic ring may be optional substituted by a C_1-C_6 alkyl; each m is independently 1, 2, 3 or 4; and n is 2 to 18; comprising reacting a compound of Formula A:



Formula A

wherein R^3, R^4 and m are defined above;

with a diol compound of Formula B in the presence of a catalyst, optionally a solvent, and optionally an entrainer;

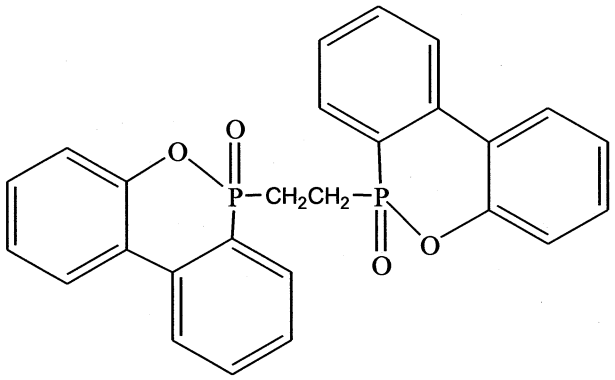
$$HO-(CH_2)_n-OH$$

Formula B

wherein n is defined above.

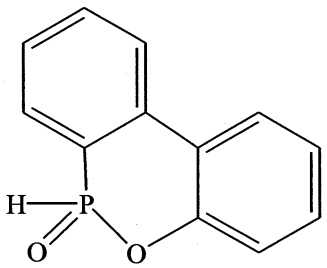
2. The process of Claim 1, wherein n is 2 to 6 and R^1, R^2, R^3 and R^4 are hydrogen.

3. A process for preparing the compound of Formula II:



Formula II

comprising reacting a compound of Formula C:



Formula C

with ethylene glycol in the presence of a catalyst, optionally a solvent and optionally an entrainer.

4. The process of Claims 1 or 3, wherein the entrainer is present.

5. The process of Claims 1 or 3, wherein the solvent is present.

6. The process of Claims 1 or 3, wherein both the solvent and the entrainer are present.

7. The process of Claims 1 or 3 wherein said catalyst is an alky halide, alkali halides, alkaline earth metal halides, transition metals and their halides or acid catalysts.

8. The process of Claims 1 or 3, wherein said catalyst is sodium iodide, lithium bromide, lithium chloride, potassium iodide, potassium bromide, lithium iodide, C_1-C_6 alkyl iodide, C1-C_6 alkyl bromide, 2-iodoethanol, 2-bromoethanol, 2-chloroethanol, 3-iodopropanol, 3-bromopropanol, ferric bromide, ferrous chloride, ferrous bromide, manganous halide, copper powder, nickel halide, cobalt chloride, cesium bromide, palladium chloride, sulfuric acid, aryl sulfonic acid, alkyl sulfonic acid, arylalkyl sulfonic acid, hydrochloric acid, hydrobromic acid, hydrofluoric acid, oxalic acid, perchloric acid, trifluoromethane sulfonic acid, fluorosulfonic acid, nitric acid, aluminum chloride, diethyl aluminum chloride, triethylaluminum/hydrogen chloride, ferric chloride, zinc chloride, antimony trichloride, stannic chloride, boron trifluoride, acidic zeolites, acidic clays, polymeric sulfonic acids, or mixtures thereof.

9. The process of Claim 5, wherein the solvent is a high boiling point solvent, wherein the high boiling point solvent has a boiling point greater than 150°C at 1.03 kg/cm^2 and melting point greater than -100°C.

10. The process of Claim 9, wherein the high boiling point solvent is diphenylmethane, diphenylethane, diphenylpropane, biphenyl, decahydronaphthalene, cyclohexylbenzene, 1,3-diisopropylbenzene, dimethyl sulfoxide (DMSO), dimethyl formamide (DMF), dimethylacetamide (DMAC), ethylene glycol dimethyl ether, ethylene glycol diethyl ether or mixtures thereof.

11. The process of Claim 5, wherein the process is conducted greater than 1 atmospheric pressure and the solvent is a low boiling point solvent having a boiling point less than 150°C at 1.03 kg/cm^2.

12. The process of Claim 11, wherein the solvent is heptane, hexane, petroleum ether, methylcyclohexane; toluene, xylene, mesitylene, ethyl benzene, tetrahydrofuran, 1,4-dioxane, acetonitrile or mixtures thereof.

13. The process of Claims 1 or 3, wherein the temperature of the reaction ranges from 100°C to 250°C.

14. The process of Claims 1 or 3, wherein the process is carried out in a continuous mode.

15. The process of Claims 1 or 3, wherein the process is carried out in a batch mode.

16. The process of Claims 1 or 3, wherein the a product of the reaction is water and wherein the water and the diol compound of Formula B or ethylene glycol are continuously recycled back to reactor.

17. The process of Claim 6, wherein the process is conducted at the normal pressure and the entrainer is continuously recycled back to reactor.

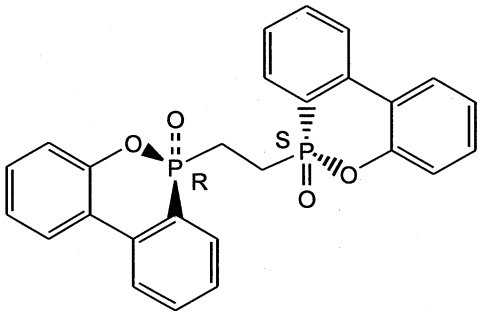
18. The process of Claims 1 or 3, wherein the entrainer is a low boiling point solvent, wherein the low boiling point solvent has a boiling point lower than 150°C.

19. The process of Claim 18, wherein the low boiling point solvent is pentane, hexane, heptane, octane, cyclohexane, methylcyclohexane, toluene, xylenes, ethylbenzene, isopropylbenzene or mixtures thereof.

20. The process of Claim 1 or 3, wherein the catalyst is an acid catalyst.

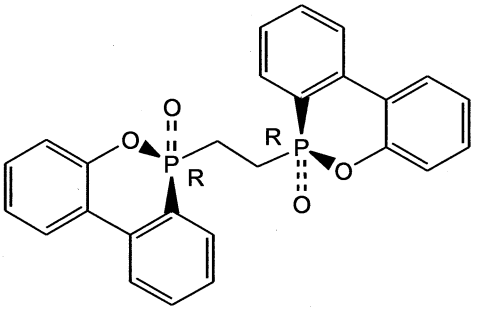
21. The process of Claim 20, wherein said acid catalyst is sulfuric acid, aryl sulfonic acid, alkyl sulfonic acid, aralkyl sulfonic acid, hydrochloric acid, hydrobromic acid, hydrofluoric acid, oxalic acid, perchloric acid, trifluoromethane sulfonic acid, fluorosulfonic acid, nitric acid, phosphoric acid, phosphonic acids, phosphinic acids aluminum chloride, diethyl aluminum chloride, triethylaluminum/hydrogen chloride, ferric chloride, zinc chloride, antimony trichloride, stannic chloride, boron trifluoride, acidic zeolites, acidic clays, polymeric sulfonic acids, or mixtures thereof.

22. A composition comprising the high melting point isomer of Formula IIa having the Formula:

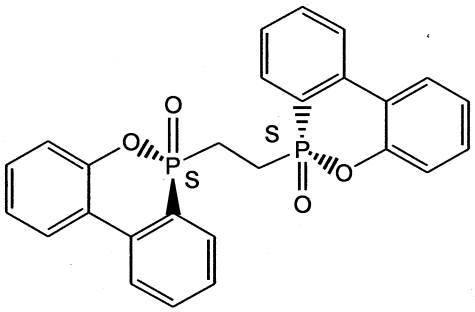


Formula IIa

and the low melting point isomers of Formula IIb and IIc having the Formulas:



Formula IIb



Formula IIc

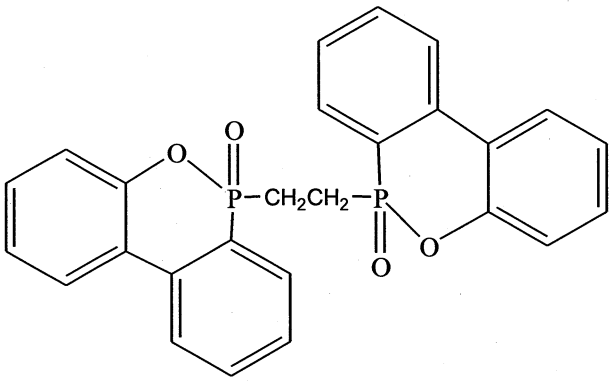
wherein the composition has an Isomer Ratio of greater than 0.5; wherein the Isomer Ratio is Ah/(Ah+Al); wherein Ah is the area of the high melting point isomer peak and Al is the area of low melting point isomers peaks utilizing 31P NMR.

23. The composition of Claim 22, having an Isomer Ratio of greater than 0.8 utilizing the 31P NMR method.

24. The composition of Claim 22, having an Isomer Ratio of greater than 0.95 utilizing the 31P NMR method.

25. The composition of Claim 22, having an Isomer Ratio of greater than 0.98 utilizing the 31P NMR method.

26. A method for producing a higher melting point composition comprising the compound of Formula II:



Formula II

comprising contacting a composition containing lower amounts of high melting point isomers of Formula II, with alcohols, water, or mixtures thereof in the presence of an acid catalyst, thereby producing a composition containing larger amounts of higher melting point isomers of Formula II.

27. The composition of Claim 26, wherein said acid catalyst is sulfuric acid, aryl sulfonic acid, alkyl sulfonic acid, aralkyl sulfonic acid, hydrochloric acid, hydrobromic acid, hydrofluoric acid, oxalic acid, perchloric acid, trifluoromethane sulfonic acid, fluorosulfonic acid, nitric acid, phosphoric acid, phosphonic acids, phosphinic acids aluminum chloride, diethyl aluminum chloride, triethylaluminum/hydrogen chloride, ferric chloride, zinc chloride, antimony trichloride, stannic chloride, boron trifluoride, acidic zeolites, acidic clays, polymeric sulfonic acids, or mixtures thereof.

28. A flame retardant polymer composition comprising a polymer and the compound of Claim 22.

29. The composition of Claim 28, wherein said polymer is polyolefins, polyesters, polyethers, polyketones, polyamides, polyvinylchlorides, natural and synthetic rubbers, polyurethanes, polystyrenes, poly(meth)acrylates, phenolic resins, polybenzoxazine, polyacetals, polyacrylonitriles, polybutadienes, polystyrenes, polyimides, polyamideimides, polyetherimides, polyphenylsulfides, polyphenylene oxide, polycarbonates, cellulose, cellulose derivatives, cyanate esters, polyphenylene esters, polybutadiene resins, butadiene-styrene resins, butadiene-divinylbenzene-styrene resins, epoxy-modified polybutadiene

resins, acrylic or vinyl acetate adhesives, carboxyl-terminated butadiene-acrylonitrile copolymers, phenylene ethers, maleic anhydride-grafted butadiene-styrene copolymers, maleic anhydride-modified 4-methyl-1pentene resins, maleated 1-butene-ethylene copolymers, resins derived from vinylbenzyl ether compounds, epoxy resins or mixtures thereof.

30. The composition of Claim 28, wherein said polymer is polyolefins, polyesters, phenolic resins, polyamides, polyurethanes, polystyrene, epoxy resins or mixtures thereof.

31. The composition of Claim 28, further comprising a melamine polyphosphate.

32. The composition of Claim 28, further comprising silica.