

What is claimed is:

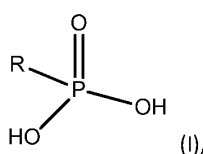
1. A process for producing a phosphorus-containing flame retardant, comprising preparing a reaction mixture, the reaction mixture comprising
 - (a) an unsubstituted or alkyl or aryl substituted phosphonic acid,
 - 5 (b) a solvent for the phosphonic acid, and
 - (c) a metal which is capable of forming a polycation, or a suitable metal compound which is represented by the formula $M_p^{(+y)}X_q$ where M is a metal, (+)y represents the charge of the metal cation, y is 2 or higher, X is an anion, and the values for p and q provide a charge balanced metal compound; and
- 10 heating the reaction mixture at a reaction temperature of 105 °C or higher for an amount of time sufficient to produce the phosphorus-containing flame retardant.
2. A process for producing a phosphorus-containing flame retardant, comprising preparing a reaction mixture, the reaction mixture comprising
 - 15 (a) an unsubstituted or alkyl or aryl substituted pyrophosphonic acid,
 - (b) a solvent for the pyrophosphonic acid, and
 - (c) a metal which is capable of forming a polycation, or a suitable metal compound which is represented by the formula $M_p^{(+y)}X_q$ where M is a metal, (+)y represents the charge of the metal cation, y is 2 or higher, X is an anion, and the values for p and q
- 20 provide a charge balanced metal compound; and
- reacting the reaction mixture at a reaction temperature of 20 °C or higher for an amount of time sufficient to produce the phosphorus-containing flame retardant.
3. The process according to claim 1 or 2, wherein the reaction mixture is prepared at a
- 25 preparation temperature below the reaction temperature.
4. The process according to claim 3, wherein the preparation temperature ranges from about 15 °C to about 40 °C.
5. The process according to any one of the preceding claims, wherein the components (a) and (b) of the reaction mixture are in the form of a solution, and preparing the reaction mixture comprises mixing the component (c) with the solution.
6. The process according to claim 1, wherein the reaction temperature is about 150 °C or
- 35 higher.

7. The process according to claim 1, wherein the reaction temperature ranges from about 140 °C to about 260 °C.
8. The process according to claim 2, wherein the reaction temperature is about 60 °C or higher.
9. The process according to claim 2, wherein the reaction temperature ranges from about 60 °C to about 240 °C.
10. The process according to any one of the preceding claims, wherein the molar ratio of component (a) to component (c) in the reaction mixture ranges from about 4:1 to about 50:1.
11. The process according to claim 1, wherein the solvent is chosen from water, sulfones, sulfoxides, halogenated hydrocarbons, aromatic hydrocarbons, and ethers.
12. The process according to claim 1, wherein the solvent comprises water.
13. The process according to claim 2, wherein the solvent is aprotic.
14. The process according to claim 1 or 2, wherein component (c) of the reaction mixture comprises a metal capable of forming a 2+, 3+ or 4+ polycation.
15. The process according to claim 1 or 2, wherein component (c) of the reaction mixture comprises a suitable metal compound which is represented by the formula $M_p^{(+y)} X_q$ where M is a metal, (+)y represents the charge of the metal cation, y is 2, 3 or 4, X is an anion, and the values for p and q provide a charge balanced metal compound.
16. The process according to claim 15, wherein y is 3.
17. The process according to claim 16, wherein M is chosen from Al, Ga, Sb, Fe, Co, B, and Bi.
18. The process according to claim 17, wherein M is Al or Fe.
19. The process according to claim 1 or 2, wherein component (c) of the reaction mixture comprises the suitable metal compound, and the suitable metal compound is chosen from a metal oxide, halide, alkoxide, hydroxide, carbonate, carboxylate, or phosphonate.

20. The process according to claim 19, wherein M in the formula $M_p^{(+y)}X_q$ is Al or Fe.

21. The process according to claim 20, wherein the suitable metal compound is chosen from
 5 alumina, aluminum trichloride, aluminum trihydroxide, aluminum isopropoxide, aluminum carbonate, aluminum acetate, iron(III) oxide, iron(III) chloride, iron(III) isopropoxide, and iron(III) acetate.

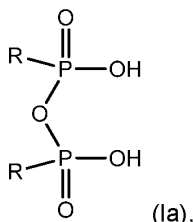
22. The process according to claim 1, wherein the unsubstituted or alkyl or aryl substituted
 10 phosphonic acid is represented by formula (I)



wherein R is H, C₁₋₁₂ alkyl, C₆₋₁₀ aryl, C₇₋₁₈ alkylaryl, or C₇₋₁₈ arylalkyl, wherein the alkyl, aryl, alkylaryl, or arylalkyl are unsubstituted or are substituted by halogen, hydroxyl, amino, C₁₋₄ alkylamino, di-C₁₋₄ alkylamino, C₁₋₄ alkoxy, carboxy or C₂₋₅ alkoxy carbonyl.

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23. The process according to claim 2, wherein the unsubstituted or alkyl or aryl substituted pyrophosphonic acid is represented by formula (Ia)



wherein R is H, C₁₋₁₂ alkyl, C₆₋₁₀ aryl, C₇₋₁₈ alkylaryl, or C₇₋₁₈ arylalkyl, wherein the alkyl, aryl, alkylaryl, or arylalkyl are unsubstituted or are substituted by halogen, hydroxyl, amino, C₁₋₄ alkylamino, di-C₁₋₄ alkylamino, C₁₋₄ alkoxy, carboxy or C₂₋₅ alkoxy carbonyl.

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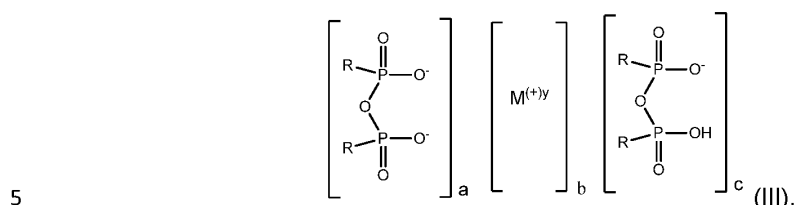
24. The process according to claim 22 or 23, wherein R is unsubstituted C₁₋₁₂ alkyl, C₆ aryl, C₇₋₁₀ alkylaryl, or C₇₋₁₀ arylalkyl.

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25. The process according to claim 24, wherein R is unsubstituted C₁₋₆ alkyl.

26. The process according to claim 26 or 27, wherein R is methyl, ethyl, propyl, isopropyl, butyl, or t-butyl.

27. A phosphorus-containing flame retardant produced according to the process of any one of claims 1 through 26, wherein the phosphorus-containing flame retardant comprises a compound of empirical formula (III)



wherein R is H, an alkyl, aryl, alkylaryl, or arylalkyl group;

M is a metal and y is 2 or 3, such that $\text{M}^{(+y)}$ is a metal cation where (+)y represents the charge formally assigned to the cation;

a, b, and c represent the ratio of the components to which they correspond relative to one another in the compound and satisfy the charge-balance equation $2(a)+c=b(y)$; and c is not zero.

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28. The phosphorus-containing flame retardant according to claim 27, wherein y is 3, a is 1, b is 1, and c is 1.

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29. A flame retardant polymer composition comprising (i) a polymer and (ii) the phosphorus-containing flame retardant according to claim 27 or 28.

30. The flame retardant polymer composition according to claim 29, wherein the polymer comprises one or more of a polyolefin homopolymer or copolymer, rubber, polyester, epoxy resin, polyurethane, polysulfone, polyimide, polyphenylene ether, styrenic polymer or copolymer, polycarbonate, acrylic polymer, polyamide, or polyacetal.

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31. The flame retardant polymer composition according to claim 30, wherein the polymer comprises one or more of a styrenic polymer or copolymer, polyolefin homopolymer or copolymer, polyester, polycarbonate, acrylic polymer, epoxy resin, polyamide, or polyurethane.

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32. The flame retardant polymer composition according to claim 31, wherein the polymer comprises a polyalkylene terephthalate, high impact polystyrene (HIPS), epoxy resin, or polyamide.

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33. The flame retardant polymer composition according to claim 32, wherein the polymer comprises a glass filled polyalkylene terephthalate, glass reinforced epoxy resin, or a glass filled polyamide.

- 5 34. The flame retardant polymer composition according to claim 32, wherein the polymer comprises a polyphthalamide.

35. The flame retardant polymer composition according to claim 32, wherein the polymer comprises polyamide 46, polyamide 6, polyamide 66, polyamide 4T, or polyamide 9T.

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36. The flame retardant polymer composition according to claim 32, wherein the polymer comprises polyamide MXD,6, polyamide 12,T, polyamide 10,T, polyamide 6,T/6,6, polyamide 6,T/D,T, polyamide 6,6/6,T/6,I, polyamide 6/6,T, or polyamide 6,T/6,I.

- 15 37. The flame retardant polymer composition according to claim 29, wherein the polymer comprises a polyphenylene ether/styrenic resin blend, acrylonitrile butadiene styrene (ABS), polyvinyl chloride/ABS blend, methacrylonitrile/ABS blend, α -methylstyrene containing ABS, polyester/ABS, polycarbonate/ABS, impact modified polyester, or impact modified polystyrene.

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38. The flame retardant polymer composition according to any one of claims 29 through 37, further comprising (iii) one or more compounds chosen from additional flame retardants, synergists, and flame retardant adjuvants.

- 25 39. The flame retardant polymer composition according to claim 38, wherein the one or more compounds are chosen from halogenated flame retardants, alkyl or aryl phosphine oxides, alkyl or aryl polyphosphine oxides, alkyl or aryl phosphates, alkyl or aryl phosphonates, alkyl or aryl phosphinates, salts of alkyl or aryl phosphinic acid, carbon black, graphite, carbon nanotubes, siloxanes, polysiloxanes, polyphenylene ether, melamine, melamine derivatives,
30 melamine condensation products, melamine salts, metal hydroxides, metal oxides, metal oxide hydrates, metal borates, metal carbonates, metal sulfates, metal phosphates, metal phosphonates, metal phosphites, metal hypophosphites, metal silicates, and mixed metal salts.

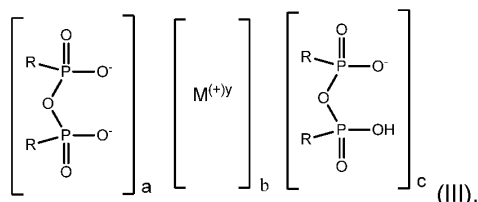
- 35 40. The flame retardant polymer composition according to claim 39, wherein the one or more compounds are chosen from aluminum tris(dialkylphosphinate), aluminum hydrogen phosphite, benzylic phosphine oxides, poly benzylic phosphine oxides, melam, melem,

- melon, melamine phosphates, melamine metal phosphates, melamine cyanurate, melamine borate, talc, clays, calcium silicate, aluminosilicate, aluminosilicate as hollow tubes, calcium carbonate, magnesium carbonate, barium sulfate, calcium sulfate, boron phosphate, calcium molybdate, exfoliated vermiculite, zinc stannate, zinc hydroxystannate, zinc sulfide, zinc
- 5 borate, zinc molybdate, zinc phosphate, magnesium oxide, magnesium hydroxide, aluminum oxide, aluminum oxide hydroxide, aluminum trihydrate, silica, tin oxide, antimony oxide (III and V), antimony (III and V) oxide hydrate, titanium oxide, zinc oxide, zinc oxide hydrate, zirconium oxide, and zirconium hydroxide.
- 10 41. The flame retardant polymer composition according to claim 40, wherein the one or more compounds are chosen from aluminum tris(dimethylphosphinate), aluminum tris(diethylphosphinate), aluminum tris(dipropylphosphinate), aluminum tris(dibutylphosphinate), methylene-diphenylphosphine oxide-substituted polyaryl ether, xylylenebis(diphenylphosphine oxide), 1,2-bis-(9,10-dihydro-9-oxy-10-
- 15 phosphaphenanthrene-10-oxide)ethane, 4,4'-bis(diphenylphosphinylmethyl)-1,1'-biphenyl, melam, melem, melon, and dimelamine zinc pyrophosphate.
42. The flame retardant polymer composition according to claim 29, further comprising one or more compounds chosen from hydrotalcite clays, metal borates, metal oxides, and metal
- 20 hydroxides.
43. The flame retardant polymer composition according to claim 42, wherein the metal of the metal borates, metal oxides, and metal hydroxides is zinc or calcium.
- 25 44. The flame retardant polymer composition according to claim 38, wherein the one or more compounds are chosen from melam, melem, melon, melamine cyanurate, melamine polyphosphate, melamine poly(metal phosphate), poly-[2,4-(piperazine-1,4-yl)-6-(morpholine-4-yl)-1,3,5-triazine]/piperazin, aluminum hypophosphite, and aluminum dialkylphosphinate.
- 30 45. The flame retardant polymer composition according to claim 38, wherein the one or more compounds are chosen from zinc borate, zinc stannate, polysiloxanes, kaolin, silica, magnesium hydroxide, zinc molybdate complex, zinc molybdate/magnesium hydroxide complex, zinc molybdate/magnesium silicate complex, calcium molybdate/zinc complex, zinc
- 35 phosphate complex, and melamine-poly(zinc phosphate).

46. The flame retardant polymer composition according to claim 38, wherein the one or more compounds comprise melam and any one or more materials selected from zinc borate, zinc stannate, zinc molybdate complex, zinc molybdate/magnesium hydroxide complex, zinc molybdate/magnesium silicate complex, calcium molybdate/zinc complex, zinc phosphate complex, and zinc oxide.

47. The flame retardant polymer composition according to claim 38, wherein the one or more compounds comprise melon and any one or more materials selected from zinc borate, zinc stannate, zinc molybdate complex, zinc molybdate/magnesium hydroxide complex, zinc molybdate/magnesium silicate complex, calcium molybdate/zinc complex, zinc phosphate complex, and zinc oxide.

48. A flame retardant material comprising a compound of empirical formula (III)



wherein R is H, an alkyl, aryl, alkylaryl, or arylalkyl group;
M is a metal and y is 2 or 3, such that $\text{M}^{(+y)}$ is a metal cation where (+)y represents the charge formally assigned to the cation;
a, b, and c represent the ratio of the components to which they correspond relative to one another in the compound, and satisfy the charge-balance equation $2(a)+c=b(y)$; and
c is not zero.

49. The flame retardant material according to claim 48, wherein y is 3, a is 1, b is 1, and c is 1.

50. The flame retardant material according to claim 49, wherein M is Al, Ga, Sb, Fe, Co, B, or Bi.

51. The flame retardant material according to claim 50, wherein M is Al or Fe.

52. The flame retardant material according to any one of claims 48 to 51, wherein R is H or alkyl.

53. The flame retardant material according to claim 52, wherein R is C₁₋₆ alkyl.
54. The flame retardant material according to claim 53, wherein R is methyl or ethyl.
- 5 55. The flame retardant material according to any one of claims 48 to 54, wherein the compound of empirical formula (III) makes up at least 75% by weight of the flame retardant material.
- 10 56. The flame retardant material according to claim 55, wherein the compound of empirical formula (III) makes up at least 90% by weight of the flame retardant material.
57. A flame retardant polymer composition comprising (i) a polymer and (ii) the flame retardant material according to any one of claims 48 through 56.
- 15 58. A process for increasing the flame resistance of a polymer, comprising incorporating a flame retardant material according to any one of claims 48 through 56 into a polymer resin, optionally with one or more additional flame retardant, synergist or flame retardant adjuvant.
- 20 59. A process for increasing the flame resistance of a polymer, comprising incorporating a phosphorus-containing flame retardant according to claim 27 or 28 into a polymer resin, optionally with one or more additional flame retardant, synergist or flame retardant adjuvant.