CLAIMS

1. A process for producing a phosphorus-containing flame retardant, comprising reacting at a reaction temperature a mixture comprising a metal or suitable metal compound and a stoichiometric excess relative to the metal or suitable metal compound of an unsubstituted or alkyl or aryl substituted phosphonic acid, wherein:

-the metal is capable of forming a polycation, preferably a 2+, 3+ or 4+ polycation, or the suitable metal compound 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;

-the molar ratio of the unsubstituted or alkyl or aryl substituted phosphonic acid to the metal or suitable metal compound in the mixture is higher than 4:1;

-the reaction temperature is 105 °C or higher, preferably 150 °C or higher, most preferably in the range from 140 °C to 260 °C; and

-the unsubstituted or alkyl or aryl substituted phosphonic acid is in a molten state at the reaction temperature.

2. A process for producing a phosphorus-containing flame retardant, comprising reacting at a reaction temperature a mixture comprising a metal or suitable metal compound and a stoichiometric excess relative to the metal or suitable metal compound of an unsubstituted or alkyl or aryl substituted pyrophosphonic acid, wherein:

-the metal is capable of forming a polycation, preferably a 2+, 3+ or 4+ polycation, or the suitable metal compound 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;

-the molar ratio of the unsubstituted or alkyl or aryl substituted pyrophosphonic acid to the metal or suitable metal compound in the mixture is higher than 2:1; and

-the unsubstituted or alkyl or aryl substituted pyrophosphonic acid is in a molten state at the reaction temperature.

3. The process according to claim 2, wherein the reaction temperature is 40 °C or higher, preferably from 60 °C to 260 °C.

4. The process according to claim 1, wherein the molar ratio is 8:1 or higher, preferably from 10:1 to 50:1.

5. The process according to any one of claims 2 and 3, wherein the molar ratio is 4:1 or higher, preferably from 5:1 to 25:1.

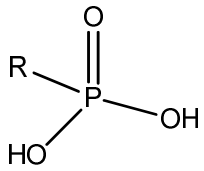
6. The process according to claim 1 or 2, wherein the mixture comprises a suitable metal compound which is represented by the formula $M_p^{(+)y} X_q$ where M is a metal, (+)y represents the charge of the metal cation, y is 2, 3 or 4, X is an anion, and the values for p and q provide a charge balanced metal compound, preferably wherein the suitable metal compound is chosen from a metal oxide, halide, alkoxide, hydroxide, carbonate, carboxylate, or phosphonate.

7. The process according to claim 6, wherein y is 3.

8. The process according to claim 7, wherein M is chosen from Al, Ga, Sb, Fe, Co, B, and Bi, preferably Al or Fe.

9. The process according to claim 8, wherein the suitable metal compound is chosen from alumina, aluminum trichloride, aluminum trihydroxide, aluminum isopropoxide, aluminum carbonate, aluminum acetate, iron(III) oxide, iron(III) chloride, iron(III) isopropoxide, and iron(III) acetate.

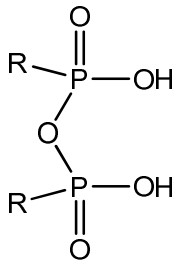
10. The process according to claim 1, wherein the unsubstituted or alkyl or aryl substituted phosphonic acid is represented by formula (I)



(I),

wherein R is H, C_{1-12} alkyl, C_{6-10} aryl, C_{7-18} alkylaryl, or C_{7-18} arylalkyl, wherein the alkyl, aryl, alkylaryl, or arylalkyl are unsubstituted or are substituted by halogen, hydroxyl, amino, C_{1-4} alkylamino, di-C_{1-4} alkylamino, C_{1-4} alkoxy, carboxy or C_{2-5} alkoxycarbonyl, preferably unsubstituted C_{1-12} alkyl, C_6 aryl, C_{7-10} alkylaryl, or C_{7-10} arylalkyl, more preferably unsubstituted C_{1-6} alkyl and most preferably methyl, ethyl, propyl, isopropyl, butyl or t-butyl.

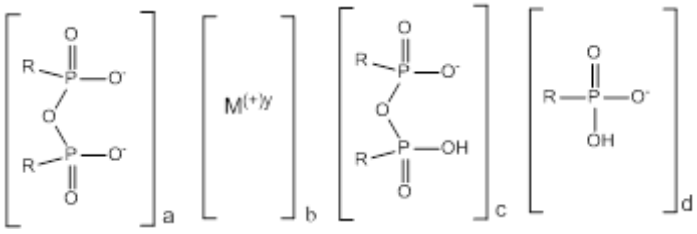
11. The process according to claim 2, wherein the unsubstituted or alkyl or aryl substituted pyrophosphonic acid is represented by formula (Ia)



(Ia),

wherein R is H, C_{1-12} alkyl, C_{6-10} aryl, C_{7-18} alkylaryl, or C_{7-18} arylalkyl, wherein the alkyl, aryl, alkylaryl, or arylalkyl are unsubstituted or are substituted by halogen, hydroxyl, amino, C_{1-4} alkylamino, di-C_{1-4} alkylamino, C_{1-4} alkoxy, carboxy or C_{2-5} alkoxycarbonyl, preferably unsubstituted C_{1-12} alkyl, C_6 aryl, C_{7-10} alkylaryl, or C_{7-10} arylalkyl, more preferably unsubstituted C_{1-6} alkyl and most preferably methyl, ethyl, propyl, isopropyl, butyl or t-butyl.

12. A crystalline phosphorus-containing flame retardant produced according to the process of any one of claims 1 through 11 directly in the form of a powder or small particles, wherein said flame retardant is a compound or mixture of compounds of empirical formula (II)



(II)

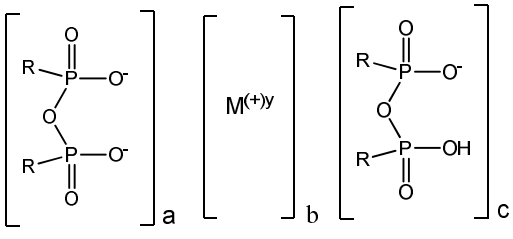
wherein R is H, an alkyl, aryl, alkylaryl, or arylalkyl group;

M is a metal and y is a number from 2 to 5, such that $M^{(+)y}$ is a metal cation where (+)y represents the charge formally assigned to the cation;

a, b, c, and d 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+d=b(y); and

and c is not zero.

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



(III),

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

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

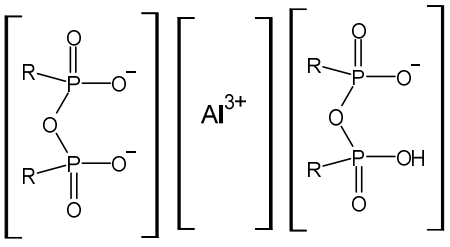
a, b, and c represent the ratio of the components to which they correspond relative to one another in the compound and satisfy the charge-balance equation 2(a)+c=b(y); and
c is not zero; and
wherein the compound of empirical formula (III) makes up at least a majority by weight of the flame retardant material.

14. The flame retardant material according to claim 13, wherein y is 3, a is 1, b is 1, and c is 1.

15. The flame retardant material according to claim 14, wherein M is chosen from Al, Ga, Sb, Fe, Co, B, and Bi, preferably Al, Fe, Ga, Sb, or B.

16. The flame retardant material according to any one of claims 13-15, wherein R is H, C_{1-12} alkyl, C_{6-10} aryl, C_{7-18} alkylaryl, or C_{7-18} arylalkyl, wherein the alkyl, aryl, alkylaryl, or arylalkyl are unsubstituted or are substituted by halogen, hydroxyl, amino, C_{1-4} alkylamino, di-C_{1-4} alkylamino, C_{1-4} alkoxy, carboxy or C_{2-5} alkoxycarbonyl, preferably unsubstituted C_{1-12} alkyl, C_6 aryl, C_{7-10} alkylaryl, or C_{7-10} arylalkyl, more preferably unsubstituted C_{1-6} alkyl, most preferably methyl, ethyl, propyl, isopropyl, butyl or t-butyl.

17. The flame retardant material according to claim 13, wherein the compound of empirical formula (III) is a compound of empirical formula (IIIa)



(IIIa)

wherein R is H or alkyl, preferably unsubstituted C_{1-6} alkyl, most preferably methyl or ethyl.

18. The flame retardant material according to any one of claims 13-17, wherein the compound of empirical formula (III) or (IIIa) makes up at least 75% by weight, preferably at least 90% by weight, of the flame retardant material.

19. A flame retardant polymer composition comprising (i) a polymer and (ii) the flame retardant material according to any one of claims 13-18.

20. The flame retardant polymer composition according to claim 19, 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, preferably polyalkylene terephthalate or glass filled polyalkylene terephthalate, high impact polystyrene (HIPS), epoxy resin or glass reinforced epoxy resin, or polyamide or glass filled polyamide, preferably a polyamide selected from polyamide 46, polyamide 6, polyamide 66, polyamide 4T, polyamide 9T, 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, or a polyphthalamide.

21. The flame retardant polymer composition according to claim 19, 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.

22. The flame retardant polymer composition according to any one of claims 19 through 21, further comprising (iii) one or more compounds chosen from additional flame retardants, synergists, and flame retardant adjuvants.

23. The flame retardant polymer composition according to claim 22, 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, 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, preferably 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 borate, zinc molybdate, zinc phosphate, magnesium oxide, magnesium hydroxide, aluminum oxide, aluminum oxide

hydroxide, aluminum trihydrate, silica, tin oxide, antimony oxide (Ill and V), antimony (III and V) oxide hydrate, titanium oxide, zinc oxide, zinc oxide hydrate, zirconium oxide, and zirconium hydroxide, or preferably 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-phosphaphenanthrene-10-oxide)ethane, 4,4′-bis(diphenylphosphinylmethyl)-1,1′-biphenyl, melam, melem, melon, and dimelamine zinc pyrophosphate.

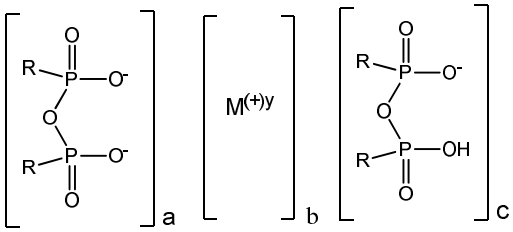
24. The flame retardant polymer composition according to claim 22, wherein the one or more compounds are chosen from melam, melem, melon, melamine cyanurate, melamine polyphosphate, melamine poly(metal phosphate), poly-[2,4-(piperazine-1,4-yl)-6-(morpholine-4-yl)-1,3,5-triazine]/piperazin, aluminum hypophosphite, and aluminum dialkylphosphinate, or 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 phosphate complex, and melamine-poly(zinc phosphate).

25. The flame retardant polymer composition according to claim 22, 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.

26. The flame retardant polymer composition according to claim 22, 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.

27. A process for increasing the flame resistance of a polymer, comprising incorporating the flame retardant material according to any one of claims 13 through 18 into a polymer resin, optionally with one or more additional flame retardant, synergist or flame retardant adjuvant.

28. A crystalline compound of empirical formula (III)



(III),

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

M is a metal and y is 2 or 3, such that $M^{(+)y}$ 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.

29. The crystalline compound of claim 28, wherein y is 3.

30. The crystalline compound of claim 28, wherein M is aluminum or iron.

31. The crystalline compound of any one of claims 28-30, wherein R is unsubstituted C_{1-6} alkyl.

32. The crystalline compound of claim 31, wherein R is methyl or ethyl.