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Polycarbonates having a good wettability

The invention provides polycarbonates as a substrate material for the production of transparent injection mouldings, in particular for the production of injection mouldings to be coated, and mouldings obtainable from the polycarbonates according to the invention. Mouldings can be e.g. transparent sheets, lenses, optical storage media or carriers for optical storage media or also articles from the field of automotive glazing, such as e.g. light-diffusing panes. The invention provides in particular optical storage media or carriers for optical storage media, such as e.g. writable optical data stores, which have a good capacity for being coated and wetting capacity and are suitable e.g. for application of dyestuffs from solution, in particular from non-polar media. Furthermore, the optical injection mouldings of the polycarbonates according to the invention have a relatively low tendency to soil.

Transparent injection mouldings are of importance above all in the field of glazing and of storage media.

Optical data recording materials are increasingly being used as a variable recording and/or archiving medium for large quantities of data. Examples of this type of optical data stores are CD, super-audio CD, CD-R, CD-RW, DVD, DVD-R, DVD+R, DVD-RW, DVD+RW and BD.

Transparent thermoplastics, such as, for example, polycarbonate, polymethyl methacrylate and chemical modifications thereof, are typically employed for optical storage media. Polycarbonate as a substrate material is suitable in particular for optical disks which can be written to once and read several times and also for optical disks which can be written to several times, and for the production of mouldings from the field of automotive glazing, such as e.g. light-diffusing panes. This thermoplastic has an excellent mechanical stability, is not very susceptible to changes in dimension and is distinguished by a high transparency and impact strength.



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Polycarbonate prepared by the phase interface process can be used for the production of optical data stores of the formats described above, such as e.g. for compact disks (CD) or digital versatile disks (DVD). These disks often have the property of building up a high electrical field during their production in the injection moulding process. During production of the optical data store, this high field strength on the substrate leads e.g. to attraction of dust from the environment or to sticking of the injection-moulded articles, such as e.g. of the disks, to one another, which reduces the quality of the finished injection-moulded articles and makes the injection moulding process difficult.

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It is furthermore known that the electrostatic charging, in particular of disks (for optical data carriers), leads to a deficient wettability above all with non-polar media, such as e.g. a non-polar dyestuff, or with a dyestuff application from solvents, such as e.g. dibutyl ether, ethylcyclohexane, tetrafluoropropanol, cyclohexane, methylcyclohexane or octafluoropropanol. A high electrical field on the surface of the substrate during dyestuff application to writable data stores thus causes, for example, an irregular coating with dyestuff and therefore leads to defects in the information layer.

20 The extent of the electrostatic charging of a substrate material can be quantified e.g. by measurement of the electrical field at a particular distance from the substrate surface.

In the case of an optical data store in which e.g. a dyestuff component is applied to the surface in a spin coating process, a low absolute electrical field strength is necessary in order to guarantee uniform application of the writable layer and to ensure a trouble-free production process.

25 Furthermore, a high electrostatic field causes losses in yield in respect of the substrate material due to the facts described above. This can lead to a halt to the particular production step and is associated with high costs.

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In order to solve this problem of a high static charging, several set-ups have been pursued. In general, antistatics are added to the substrate material as additives. Antistatic polycarbonate compositions are described e.g. in JP 62 207 358-A. Here, phosphoric acid derivatives, inter alia, are added to the polycarbonate as antistatics.

5 EP 0922 728 describes various antistatics, such as polyalkylene glycol derivatives, ethoxylated sorbitan monolaurate, polysiloxane derivatives, phosphine oxides and distearylhydroxyamine, which are employed individually or as mixtures. The Japanese Application JP 62 207 358 describes esters of phosphorous acid as additives. US Patent 5,668,202 describes sulfonic acid derivatives. In WO 00/50

10 488, 3,5-di-tert-butylphenol is employed as a chain terminator in the phase interface process. This chain terminator leads to a lower static charging of the corresponding substrate material compared with conventional chain terminators. JP 62 207 358-A describes polyethylene derivatives and polypropylene derivatives as additives for polycarbonate.

15 However, the additives described can also have an adverse effect on the properties of the substrate material, since they tend to escape from the material, i.e. to ... the ... the surface. This is indeed a desirable effect for the antistatic properties, but can lead to formation of a deposit or defective copying. Moreover, the content of

20 oligomers in the polycarbonate can also lead to a poorer level of mechanical properties and to a lowering of the glass transition temperature. Furthermore, these additives may cause side reactions. The subsequent "end-capping" of polycarbonate which has been obtained from the transesterification process is expensive and the results achieved are not optimum. Introduction of new end groups into the material

25 is associated with high costs.

There is thus the object of providing a composition or a substrate material which meets the requirements of a field strength on the substrate surface which is as low as possible, and avoids the disadvantages described above.

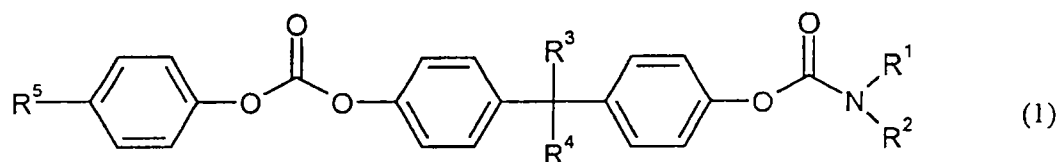
30 Surprisingly, the object has been achieved by employing for the production of optical data stores those materials in particular which contain as few incorrect

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structures, in particular as few carbamate compounds of specific structure, as possible in the low molecular weight content which are able to accumulate in a solvent extract.

- 5 A certain content of carbamate compounds in the substrate material can be caused by addition of additives, by contamination of precursors or by the preparation process itself.

10 The present invention provides polycarbonates as substrate materials, which, measured in the acetone extract by chromatography by means of HPLC, 0.2 to 300 ppm, preferably 0.2 to 250 ppm, particularly preferably 0.2 to 200 ppm, one or more compounds chosen from the formula (I)



wherein

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R^1 and R^2 independently of one another denote hydrogen or $\text{C}_1\text{-C}_{12}$ -alkyl, preferably methyl, ethyl, propyl, isopropyl or butyl, or

20 R^1 and R^2 together denote $\text{C}_4\text{-C}_{12}$ -alkylidene, preferably $\text{C}_4\text{-C}_8$ -alkylidene, particularly preferably $\text{C}_4\text{-C}_5$ -alkylidene,

R^3 and R^4 independently of one another represent hydrogen, $\text{C}_1\text{-C}_{12}$ -alkyl, preferably $\text{C}_1\text{-C}_8$ -alkyl, or phenyl, or R^3 and R^4 with the carbon atom to which they are bonded form cyclohexyl or trimethylcyclohexyl, and

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R^5 denotes hydrogen, $\text{C}_1\text{-C}_{12}$ -alkyl, $\text{C}_5\text{-C}_{12}$ -cycloalkyl, phenyl or cumyl, preferably hydrogen, tert-butyl or cumyl.

The polycarbonates according to the invention show, after processing to an injection-moulded article, preferably an optical disk, a low electrostatic charging. This is important in particular for the production of optical storage media.

- 5 The polycarbonates/substrate materials according to the invention can be prepared by choosing suitable process parameters:

10 The content of compounds of the formula 1 or 4 can be influenced by several factors. For example, the purity of the educts and auxiliary substances is important. Furthermore, process parameters such as the molar ratio of bisphenol and phosgene employed, temperatures during the reaction, reaction and dwell times, may be decisive. For the person skilled in the art, the object is to control the process such that the limits according to the invention of the carbamate content in the substrate material are not exceeded.

- 15 A suitable choice of process parameters in order to obtain the desired substrate material can appear as follows:

20 While the excess of phosgene used, based on the total of bisphenols employed, is between 3 and 100 mol%, preferably between 5 and 50 mol%, in conventional continuous polycarbonate synthesis, the substrate material according to the invention is prepared with phosgene excesses of 5 to 20 mol%, preferably 8 to 17 mol%. In this procedure, the pH of the aqueous phase is kept in the alkaline range, preferably between 8.5 and 12, during and after metering in of the phosgene via subsequent
25 metering in of sodium hydroxide solution once or several times or corresponding subsequent metering in of bisphenolate solution, while it is adjusted to 10 to 14 after addition of the catalyst. The temperature during the phosgenation is 0 °C to 40 °C, preferably 5 °C to 36 °C.

- 30 The polycarbonates according to the invention are prepared by the phase interface process. This process for the synthesis of polycarbonate is described in many cases in the literature; reference may be made by way of example to H. Schnell, Chemistry

and Physics of Polycarbonates, Polymer Reviews, vol. 9, Interscience Publishers, New York 1964 p. 33 et seq., to Polymer Reviews, vol. 10, "Condensation Polymers by Interfacial and Solution Methods", Paul W. Morgan, Interscience Publishers, New York 1965, chap. VIII, p. 325, to Dres. U. Grigo, K. Kircher and P. R. Müller,
5 "Polycarbonate" in Becker/Braun, Kunststoff-Handbuch, volume 3/1, Polycarbonate, Polyacetale, Polyester, Cellulose-ester, Carl Hanser Verlag Munich, Vienna 1992, p. 118-145 and to EP-A 0 517 044.

According to this process, the phosgenation of a disodium salt of a bisphenol (or of a
10 mixture of various bisphenols) which has been initially introduced into an aqueous-alkaline solution (or suspension) is carried out in the presence of an inert organic solvent or solvent mixture which forms a second phase. The oligocarbonates formed, which are chiefly present in the organic phase, are subjected to a
15 condensation reaction with the aid of suitable catalysts to give high molecular weight polycarbonates dissolved in the organic phase. Finally, the organic phase is separated off and the polycarbonate is isolated therefrom by various working up steps.

Dihydroxyaryl compounds which are suitable for the preparation of polycarbonates
20 are those of the formula (2)

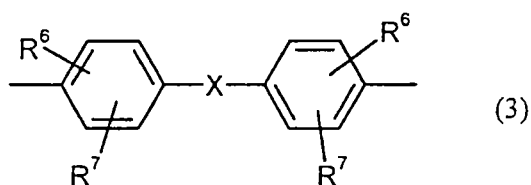


in which
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Z is an aromatic radical having 6 to 30 C atoms, which can contain one or more aromatic nuclei, can be substituted and can contain aliphatic or cycloaliphatic radicals or alkylaryls or heteroatoms as bridge members.

30 Preferably, Z in formula (2) represents a radical of the formula (3)

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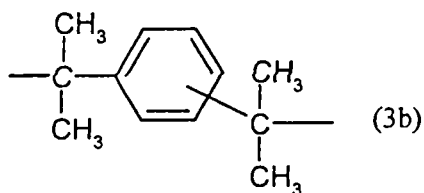
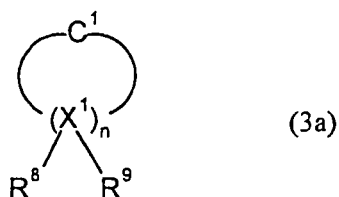
in which

5 R^6 and R^7 independently of one another represent H, C_1 - C_{18} -alkyl, C_1 - C_{18} -alkoxy, halogen, such as Cl or Br, or in each case optionally substituted aryl or aralkyl, preferably H or C_1 - C_{12} -alkyl, particularly preferably H or C_1 - C_8 -alkyl, and very particularly preferably H or methyl, and

10 X represents a single bond, $-SO_2-$, $-CO-$, $-O-$, $-S-$, C_1 - to C_6 -alkylene, C_2 - to C_5 -alkylidene or C_5 - to C_6 -cycloalkylidene, which can be substituted by C_1 - to C_6 -alkyl, preferably methyl or ethyl, and furthermore C_6 - to C_{12} -arylene, which can optionally be fused with further aromatic rings containing heteroatoms.

15 Preferably, X represents a single bond, C_1 to C_5 -alkylene, C_2 to C_5 -alkylidene, C_5 to C_6 -cycloalkylidene, $-O-$, $-SO-$, $-CO-$, $-S-$, $-SO_2-$

or a radical of the formula (3a) or (3b)



20 wherein

R^8 and R^9 can be chosen individually for each X^1 and independently of one another denote hydrogen or C_1 to C_6 -alkyl, preferably hydrogen, methyl or ethyl,

X^1 denotes carbon and

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n denotes an integer from 4 to 7, preferably 4 or 5, with the proviso that on at least one atom X^1 , R^8 and R^9 are simultaneously alkyl.

Examples of dihydroxyaryl compounds are: dihydroxybenzenes,
10 dihydroxydiphenyls, bis-(hydroxyphenyl)-alkanes, bis-(hydroxyphenyl)-cycloalkanes, bis-(hydroxyphenyl)-aryls, bis-(hydroxyphenyl) ethers, bis-(hydroxyphenyl) ketones, bis-(hydroxyphenyl) sulfides, bis-(hydroxyphenyl) sulfones, bis-(hydroxyphenyl) sulfoxides, 1,1'-bis-(hydroxyphenyl)-diisopropylbenzenes and compounds thereof which are alkylated on the nucleus and
15 halogenated on the nucleus.

Diphenols which are suitable for the preparation of the polycarbonates to be used according to the invention are, for example, hydroquinone, resorcinol, dihydroxydiphenyl, bis-(hydroxyphenyl)-alkanes, bis-(hydroxyphenyl)-
20 cycloalkanes, bis-(hydroxyphenyl) sulfides, bis-(hydroxyphenyl) ethers, bis-(hydroxyphenyl) ketones, bis-(hydroxyphenyl) sulfones, bis-(hydroxyphenyl) sulfoxides, α,α' -bis-(hydroxyphenyl)-diisopropylbenzenes and compounds thereof which are alkylated on the nucleus and halogenated on the nucleus.

Preferred diphenols are 4,4'-dihydroxydiphenyl, 2,2-bis-(4-hydroxyphenyl)-1-phenyl-propane, 1,1-bis-(4-hydroxyphenyl)-phenyl-ethane, 2,2-bis-(4-hydroxyphenyl)propane, 2,4-bis-(4-hydroxyphenyl)-2-methylbutane, 1,3-bis-[2-(4-hydroxyphenyl)-2-propyl]benzene (bisphenol M), 2,2-bis-(3-methyl-4-hydroxyphenyl)-propane, bis-(3,5-dimethyl-4-hydroxyphenyl)-methane, 2,2-bis-
25 (3,5-dimethyl-4-hydroxyphenyl)-propane, bis-(3,5-dimethyl-4-hydroxyphenyl) sulfone, 2,4-bis-(3,5-dimethyl-4-hydroxyphenyl)-2-methylbutane, 1,3-bis-[2-(3,5-

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dimethyl-4-hydroxyphenyl)-2-propyl]-benzene and 1,1-bis-(4-hydroxyphenyl)-3,3,5-trimethylcyclohexane (bisphenol TMC).

Particularly preferred diphenols are 4,4'-dihydroxydiphenyl, 1,1-bis-(4-hydroxyphenyl)-phenyl-ethane, 2,2-bis-(4-hydroxyphenyl)-propane, 2,2-bis-(3,5-dimethyl-4-hydroxyphenyl)-propane, 1,1-bis-(4-hydroxyphenyl)-cyclohexane and 1,1-bis-(4-hydroxyphenyl)-3,3,5-trimethylcyclohexane (bisphenol TMC).

These and further suitable diphenols are described e.g. in US-A 2 999 835, 3 148 172, 2 991 273, 3 271 367, 4 982 014 and 2 999 846, in the German Offenlegungsschriften 1 570 703, 2 063 050, 2 036 052, 2 211 956 and 3 832 396, French Patent Specification 1 561 518, in the monograph "H. Schnell, Chemistry and Physics of Polycarbonates, Interscience Publishers, New York 1964, p. 28 et seq.; p. 102 et seq.", and in "D. G. Legrand, J. T. Bendler, Handbook of Polycarbonate Science and Technology, Marcel Dekker New York 2000, p. 72 et seq."

In the case of the homopolycarbonates, only one diphenol is employed, and in the case of copolycarbonates two or more diphenols are employed. The diphenols used, like all the other chemicals and auxiliary substances added to the synthesis, may be contaminated with the impurities originating from their own synthesis, handling and storage. However, it is desirable to work with raw materials which are as pure as possible.

The monofunctional chain terminators required for regulation of the molecular weight, such as phenol or alkylphenols, in particular phenol, p-tert-butylphenol, iso-octylphenol, cumylphenol, chlorocarbonic acid esters thereof or acid chlorides of monocarboxylic acids or mixtures of these chain terminators, either are fed to the reaction with the bisphenolate or the bisphenolates, or are added at any desired point in time of the synthesis, as long as phosgene or chlorocarbonic acid end groups are still present in the reaction mixture or, in the case of the acid chlorides and chlorocarbonic acid esters as chain terminators, as long as sufficient phenolic end

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groups of the polymers forming are available. Preferably, however, the chain terminator or terminators are added after the phosgenation, at a position or at a point in time when phosgene is no longer present, but the catalyst has not yet been metered in, or they are metered in before the catalyst, together with the catalyst or parallel thereto.

In the same manner, any branching agents or branching agent mixtures to be used are added to the synthesis, but conventionally before the chain terminators. Trisphenols, quaternary phenols or acid chlorides of tri- or tetracarboxylic acids are conventionally used, or also mixtures of the polyphenols or of the acid chlorides.

Some of the compounds having three or more than three phenolic hydroxyl groups which can be used are, for example,

phloroglucinol,

4,6-dimethyl-2,4,6-tri-(4-hydroxyphenyl)-hept-2-ene,

4,6-dimethyl-2,4,6-tri-(4-hydroxyphenyl)-heptane,

1,3,5-tri-(4-hydroxyphenyl)-benzene,

1,1,1-tri-(4-hydroxyphenyl)-ethane,

tri-(4-hydroxyphenyl)-phenylmethane,

2,2-bis-[4,4-bis-(4-hydroxyphenyl)-cyclohexyl]-propane,

2,4-bis-(4-hydroxyphenyl-isopropyl)-phenol and

tetra-(4-hydroxyphenyl)-methane.

Some of the other trifunctional compounds are 2,4-dihydroxybenzoic acid, trimesic acid, cyanuric chloride and 3,3-bis-(3-methyl-4-hydroxyphenyl)-2-oxo-2,3-dihydroindole.

- 5 Preferred branching agents are 3,3-bis-(3-methyl-4-hydroxyphenyl)-2-oxo-2,3-dihydroindole and 1,1,1-tri-(4-hydroxyphenyl)-ethane.

The catalysts used in the phase interface synthesis are tertiary amines, in particular triethylamine, tributylamine, trioctylamine, N-ethylpiperidine, N-methylpiperidine
10 and N-i/n-propylpiperidine; quaternary ammonium salts, such as tetrabutylammonium / tributylbenzylammonium / tetraethylammonium hydroxide / chloride / bromide / hydrogen sulfate / tetrafluoroborate; and the phosphonium compounds corresponding to the ammonium compounds. These compounds are described in the literature as typical phase interface catalysts, commercially
15 obtainable and familiar to the person skilled in the art. The catalysts can be added to the synthesis individually, in a mixture or also side by side and successively, optionally also before the phosgenation, although meterings after the introduction of phosgene are preferred, unless an onium compound or mixtures of onium compounds are used as catalysts, in which case addition before the metering in of
20 phosgene is preferred. The metering of the catalyst or catalysts can be carried out in bulk, in an inert solvent, preferably that of the polycarbonate synthesis, or also as an aqueous solution, and in the case of the tertiary amines then as ammonium salts thereof with acids, preferably mineral acids, in particular hydrochloric acid. If several catalysts are used or part amounts of the total amount of catalyst are metered
25 in, different methods of metering can of course also be carried out at different places or at different times. The total amount of catalysts used is between 0.001 to 10 mol%, based on the moles of bisphenols employed, preferably 0.01 to 8 mol%, particularly preferably 0.05 to 5 mol%.

30 The conventional additives for polycarbonates can also be added to the polycarbonates according to the invention in the conventional amounts. The addition of additives serves to prolong the duration of use or the colour (stabilizers),

to simplify processing (e.g. mould release agents, flow auxiliaries, antistatics) or to adapt the polymer properties to particular stresses (impact modifiers, such as rubbers; flameproofing agents, colorants, glass fibres).

5 These additives can be added to the polymer melt individually or in any desired mixtures or several different mixtures, and in particular directly on isolation of the polymer or after melting of granules in a so-called compounding step. In this context, the additives or mixtures thereof can be added to the polymer melt as a solid, i.e. as a powder, or as a melt. Another type of metering is the use of
10 masterbatches or mixtures of masterbatches of the additives or additive mixtures.

Suitable additives are described, for example, in "Additives for Plastics Handbook, John Murphy, Elsevier, Oxford 1999", and in "Plastics Additives Handbook, Hans Zweifel, Hanser, Munich 2001".

15 Preferred heat stabilizers are, for example, organic phosphites, phosphonates and phosphanes, usually those in which the organic radicals consist entirely or partly of optionally substituted aromatic radicals. UV stabilizers which are employed are e.g. substituted benzotriazoles. These and other stabilizers can be used individually or in
20 combinations and added to the polymer in the forms mentioned.

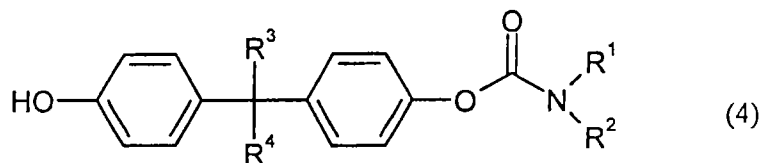
Furthermore, processing auxiliaries, such as mould release agents, usually derivatives of long-chain fatty acids, can be added. Pentaerythritol tetrastearate and glycerol monostearate e.g. are preferred. They are employed by themselves or in a
25 mixture, preferably in an amount of 0.02 to 1 wt.%, based on the weight of the composition.

Suitable flame-retardant additives are phosphate esters, i.e. triphenyl phosphate, resorcinol diphosphoric acid esters, bromine-containing compounds, such as
30 brominated phosphoric acid esters, brominated oligocarbonates and polycarbonates, and preferably salts of fluorinated organic sulfonic acids.

Suitable impact modifiers are, for example, graft polymers comprising one or more graft bases chosen from at least one polybutadiene rubber, acrylate rubber (preferably ethyl or butyl acrylate rubber), ethylene/propylene rubbers, and grafting monomers chosen from at least one monomer from the group consisting of styrene, acrylonitrile or alkyl methacrylate (preferably methyl methacrylate), or interpenetrating siloxane and acrylate networks with grafted-on methyl methacrylate or styrene/acrylonitrile.

Furthermore, colorants, such as organic dyestuffs or pigments or inorganic pigments or IR absorbers, can be added, individually, in a mixture or also in combination with stabilizers, glass fibres, glass (hollow) beads or inorganic fillers.

The polycarbonates according to the invention can moreover comprise carbamate compounds of the formula (4)



wherein R^1 , R^2 , R^3 and R^4 have the meaning given for formula (1).

The content of carbamate compounds of the formula (4) is measured by means of HPLC in the acetone extract after alkaline hydrolysis with sodium hydroxide solution and is in general 0.2 to 500 ppm, preferably 0.2 to 400 ppm, particularly preferably 0.2 to 300 ppm (cf. also the examples).

The present Application furthermore provides the extrudates and mouldings obtainable from the polycarbonates according to the invention, in particular those for use in the field of transparent products, very particularly in the field of optical uses, such as e.g. sheets, multi-wall sheets, glazing, diffuser panes, lamp covers or optical data stores, such as audio-CD, CD-R(W), DVD, DVD-R(W) and minidisks in their various only readable or once-writable and optionally also repeatedly writable embodiments.

The present invention furthermore provides the use of the polycarbonates according to the invention for the production of extrudates and mouldings.

5 Further uses are, for example, but without limiting the subject matter of the present invention:

1. Safety panes, which are known to be required in many areas of buildings,
vehicles and aircraft, and also as visors of helmets.
- 10 2. Films
3. Blow-moulded articles (see also US-A 2 964 794), for example 1 to 5 gallon
water bottles.
- 15 4. Transparent sheets, such as solid sheets or, in particular, hollow chamber
sheets, for example for covering buildings such as railway stations,
greenhouses and lighting installations.
- 20 5. Optical data stores, such as audio CDs, CD-R(W)s, DVDs, DVD-R(W)s,
minidisks and the follow-up developments
6. Traffic light housings or traffic signs
- 25 7. Foams having an open or closed, optionally printable surface
8. Threads and wires (see also DE-A 11 37 167)
9. Lighting uses, optionally using glass fibres for uses in the field of translucent
30 products

10. Translucent formulations with a content of barium sulfate and/or titanium dioxide and/or zirconium dioxide or organic polymeric acrylate rubbers (EP-A 0 634 445; EP-A 0 269 324) for the production of transparent and light-scattering mouldings.
- 5
11. Precision injection mouldings, such as holders, e.g. lens holders; polycarbonates are optionally used here with glass fibres and an optionally additional content of 1-10 wt.% molybdenum disulfide (based on the total moulding composition).
- 10
12. Optical equipment components, in particular lenses for photographic and film cameras (DE-A 27 01 173).
13. Light transmission carriers, in particular light conductor cables (EP-A 0 089 801) and illumination cover strips
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14. Electrical insulating materials for electrical conductors and for plug housings and plug connectors as well as capacitors.
- 20
15. Mobile telephone housings.
16. Network interface devices
17. Carrier materials for organic photoconductors
- 25
18. Lamps, headlamps, light-diffusing panes or internal lenses.
19. Medical uses, such as oxygenators and dialyzers.
- 30
20. Foodstuffs uses, such as bottles, utensils and chocolate moulds.

21. Uses in the automobile field, such as glazing or, in the form of blends with ABS, as bumpers.
22. Sports articles, such as slalom poles, ski boot buckles.
- 5 23. Household articles, such as kitchen sinks, wash basins, letterboxes
24. Housings, such as electrical distribution boxes
- 10 25. Housings for electrical equipment, such as toothbrushes, hairdryers, coffee machines and machine tools, such as drilling, milling and planing machines and saws
26. Washing machine portholes
- 15 27. Safety glasses, sunglasses, corrective glasses and their lenses.
28. Lamp covers
- 20 29. Packaging films
30. Chip boxes, chip supports, boxes for Si wafers
31. Other uses, such as fattening stable doors or animal cages.
- 25

Examples

The method for measurement of the carbamate content in the acetone extract is described in the following:

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The concentration of the carbamate bisphenol A oligomer of formula (1) in the acetone extract is determined as follows:

50 mg extract are dissolved in 50 ml acetonitrile. 10 μ l of the solution are injected
10 into the HPLC. Detection is carried out with a diode array detector (DAD),
fluorescence detector (FLD) or by mass spectrometry (MS) as desired. Calibration
is carried out by the external standard method (multiple point calibration).

The method for measurement of the field strength on the corresponding injection
15 moulded article, this being an optical disk, is as follows:

For measurement of the electrical field strength, a field meter (EMF 581230) from
Eltec is used. Immediately after the end of the injection moulding process, the
moulding (disk) is removed and deposited via a robot arm. During this procedure,
20 the disk must not come into contact with metal, since otherwise the measurement is
impaired. Furthermore, any ionizers present must be switched off.

The field meter is positioned above the disk at a distance of 100 mm from the
horizontal disk surface. The centre of the field meter is positioned such that its
25 projection on the disk currently to be measured lies 39 mm outside the centre of the
disk. The disk is not moved during this procedure. The field is measured in this
way in a period of 3 to 10 seconds after conclusion of the injection moulding
process.

30 The measuring apparatus is connected to an x/y recorder, on which the particular
values are printed out. Each disk measured is thus assigned a particular integral
value of the electrical field. To limit the amount of data, 100 measurements are

carried out after the start of the process, i.e. the corresponding electrical field of the first 100 disks is recorded. After in each case 60 minutes, a further 100 measurements are performed. After the 4th measurement series, i.e. after approx. 3 hours, the measurement is stopped.

5

When carrying out the measurement it is to be ensured that the atmospheric humidity during the measurement is 30 to 60 %, preferably 35 to 50 %, and the room temperature is 25 to 28 °C.

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In this method, the electrical field on the surface of the optical disk is measured by means of a probe directly after the injection moulding process. A disk is then considered difficult to write on if the electrical field exceeds a value of 18 kV/m.

Example 1

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Preparation of 1-(4-tert-butylphenyloxycarbonyloxy)-1'-(piperidinecarboxylic acid) 4,4'-isopropylidenediphenyl ester

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9.30 g (0.025 mol) isopropylidenediphenyl bischlorocarbonate are initially introduced into 150 ml methylene chloride under argon and the mixture is cooled to 0 °C. 48.49 g (0.428 mol) N-ethylpiperidine are dissolved in 20 ml methylene chloride and the solution is added dropwise to the bischlorocarbonate solution at 0 °C. 3.76 g (0.025 mol) tert-butylphenol, dissolved in 10 ml methylene chloride, are then added dropwise to this solution at 0 °C. The mixture is allowed to warm to room temperature and is stirred for 3 hours. Thereafter, the solvent is removed in vacuo. The residue is boiled up in 500 ml toluene and filtered off hot. On cooling, crystals precipitate out in the mother liquor. The mother liquor is filtered and concentrated (95 °C, 25 mbar). 13.2 g of a highly viscous red oil are obtained. This oil is dissolved in 100 ml ethyl acetate and, after addition of 10 g silica gel (silica gel 60; 0.04-0.063 µm, Merck 109385/Lt: 948 785 203), the mixture is concentrated and introduced on to a silica gel column (column Ø 5 cm, filling height approx.

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25 cm). After chromatography with a solvent mixture of n-hexane/ethyl acetate: (9:1), 2.3 g of a vitreous solid are obtained.

¹H-NMR (400 MHz, CDCl₃) δ = 7.4-7.38 (m, 2 H), 7.28-7.23 (m, 2 H), 7.22-7.13 (m, 6 H), 7.03-6.98 (m, 2 H), 3.65-3.45 (m, 4 H), 1.70-1.55 (m, 6 H), 1.66 (s, 6 H), 1.32 (s, 9 H).

Example 2

10 Preparation of 1-(4-tert-butylphenoxy)carbonyloxy)-1'-(4,4'-isopropylidene-
diphenyl) N,N-diethylcarbamate

5.0 g (0.013 mol) isopropylidenediphenyl bischlorocarbonate are initially introduced into 100 ml methylene chloride at 0 °C under argon. 4.29 g (0.042 mol) triethylamine, dissolved in 30 ml methylene chloride, are added dropwise to this solution at 0 °C. 2.02 g (0.013 mol) tert-butylphenol, dissolved in 30 ml methylene chloride, are then added dropwise. The mixture is allowed to warm to room temperature and is stirred for 3 hours. Thereafter, the solvent is removed in vacuo. The residue is boiled up in 500 ml toluene and filtered off hot.

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The solvent is removed in vacuo. The crude product is chromatographed on silica gel (h: 16 cm, Ø 5 cm, mobile phase n-hexane/EE 9:1).

2.1 g of a yellow highly viscous resin are obtained.

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¹H-NMR (400 MHz, CDCl₃) δ = 7.45-7.38 (m, 2 H), 7.28-7.15 (m, 8 H), 7.05-6.98 (m, 2 H), 3.50-3.30 (m, 4 H), 1.67 (s, 6 H), 1.32 (s, 9 H), 1.28-1.15 (m, 6 H).

Example 3**Preparation of piperidinecarboxylic acid 4-[1-(4-hydroxyphenyl)-1-methylethyl]-phenyl ester**

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0.5 g 1-(4-tert-butylphenoxy)carbonyloxy)-1'-(piperidinecarboxylic acid) 4,4'-isopropylidenediphenyl ester are dissolved in 20 g THF, 0.5 g 32 % strength sodium hydroxide solution and 5 g water are added and hydrolysis is carried out over night (min. 15 hours), while shaking.

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Working up:

The aqueous phase of the THF solution is separated off and the org. phase is concentrated. The residue is taken up in diethyl ether and the mixture is washed several times with water. The organic phase is dried over magnesium sulfate, the drying agent is filtered off and the solvent is removed in vacuo. 1.46 g of crude product are obtained and this product is chromatographed on silica gel (silica gel 60; 0.04-0.063 μm ; Merck 109385/Lt: 948 785 203) with a solvent mixture of hexane/ethyl acetate (9:1) (column $\varnothing$ 5 cm, filling height approx. 25 cm). In the subsequent course, hexane/ethyl acetate (5:1) is used as the solvent mixture. 1.0 g of a white solid is obtained.

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Example 4**Preparation of diethylcarbamate acid 4-[1-(4-hydroxyphenyl)-1-methylethyl]-phenyl ester**

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0.5 g 1-(4-tert-butylphenoxy)carbonyloxy)-1'-(4,4'-isopropylidenediphenyl) N,N-diethylcarbamate are dissolved in 20 g THF, 0.5 g 32 % strength sodium hydroxide

solution and 5 g water are added and the mixture is hydrolysed over night (min. 15 hours), while shaking.

Working up:

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The aqueous phase of the THF solution is separated off and the org. phase is concentrated. The residue is taken up in diethyl ether and the mixture is washed several times with water. The organic phase is dried over magnesium sulfate, the drying agent is filtered off and the solvent is removed in vacuo. The crude product
10 is chromatographed on silica gel (silica gel 60; 0.04-0.063 μm ; Merck 109385/Lt: 948 785 203) with a solvent mixture of hexane/ethyl acetate (9:1) (column $\varnothing$ 3 cm, filling height approx. 25 cm). In the subsequent course, hexane/ethyl acetate (1:1) is used as the solvent mixture. 0.29 g of a white solid is obtained.

15 $^1\text{H-NMR}$ (400 MHz CDCl_3) δ = 7.26-7.22 (m, 2 H), 7.12-7.08 (m, 2 H), 7.04-6.98 (m, 2 H), 6.72-6.68 (m, 2 H), 3.55-3.35 (m, 4 H), 1.67 (s, 6 H), 1.35-1.15 (m, 6 H).

Example 5

20 The preparation of the polycarbonate is carried out by the known phase interface process. It is operated by a continuous process.

The bisphenolate solution (bisphenol A; alkali content 2.12 mol NaOH/mol BPA) at 750 kg/h (14.93 wt.%), the solvent (methylene chloride/chlorobenzene 1:1) at
25 646 kg/h and the phosgene at 56.4 kg/h are fed into the reactor and reacted. The temperature in the reactor is 35 °C. Sodium hydroxide solution (32 wt.%) at 9.97 kg/h is furthermore metered in. In the course of the condensation reaction a second amount of sodium hydroxide solution (32 wt.%) at 29.27 kg/h and a solution
30 of chain terminators (11.7 wt.% tert-butylphenol in methylene chloride/chlorobenzene 1:1) at 34.18 kg/h are metered in. Thereafter, N-ethylpiperidine, dissolved in methylene chloride/chlorobenzene (1:1, 2.95 wt.% N-ethylpiperidine) at 33.0 kg/h is fed in as the catalyst. The phases are separated

and the organic phase is washed once with dilute hydrochloric acid and five times with water. The polycarbonate solution is then concentrated, concentrated further in an evaporation vessel, and the polymer melt is spun off via a devolatilization extruder and granulated.

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300 g of the polycarbonate prepared in this manner (see Table 1) are subjected to a Soxhlet extraction with 500 ml acetone (Fluka, ACS for UV spectroscopy). The extract content is approx. 5 g. 50 mg of the extract are dissolved in 50 ml acetonitrile, and 10 μ l of this solution are injected into the HPLC. Detection is carried out by MS. Calibration is carried out by the external standard method (multiple point calibration) using the reference substance from Example 1.

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The content of carbamate compounds of Example 1 in this polycarbonate sample is 160 mg/kg (160 ppm).

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Example 6

Comparison example

The preparation of the polycarbonate is carried out as described in Example 5. However, the bisphenolate solution (bisphenol A) at 750 kg/h (14.93 wt.%), the solvent (methylene chloride/chlorobenzene 1:1) at 646 kg/h and the phosgene at 58.25 kg/h are fed into the reactor. Sodium hydroxide solution (32 wt.%) at 12.34 kg/h is furthermore also metered in. The second amount of sodium hydroxide solution is 36.20 kg/h; the amount of chain terminator is 34.18 kg/h at the concentrations stated in Example 5. The amount of catalyst is 33 kg/h. Working up is carried out as described in Example 5.

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300 g of this polycarbonate (see Table 1) are subjected to a Soxhlet extraction with 500 ml acetone (Fluka ACS for UV spectroscopy). The extract content is approx. 5 g. 50 mg of the extract are dissolved in 50 ml acetonitrile, and 10 μ l of this solution are injected into the HPLC. Detection is carried out by MS. Calibration is

carried out by the external standard method (multiple point calibration) using the reference substance from Example 1.

The content of carbamate compounds of Example 1 in this polycarbonate sample is
5 600 mg/kg (600 ppm).

Example 7

The preparation of the polycarbonate is carried out by the known phase interface
10 process. It is operated by a continuous process.

The bisphenolate solution (bisphenol A; alkali content 2.12 mol NaOH/mol BPA) at
750 kg/h (14.93 wt.%), the solvent (methylene chloride/chlorobenzene 1:1) at
646 kg/h and the phosgene at 56.4 kg/h are fed into the reactor and reacted. The
15 temperature in the reactor is 35 °C. Sodium hydroxide solution (32 wt.%) at
9.97 kg/h is furthermore metered in. In the course of the condensation reaction a
second amount of sodium hydroxide solution (32 wt.%) at 29.27 kg/h and a solution
of chain terminators (11.7 wt.% tert-butylphenol in methylene
chloride/chlorobenzene 1:1) at 34.18 kg/h are metered in. Thereafter,
20 N-ethylpiperidine, dissolved in methylene chloride/chlorobenzene (1:1, 2.95 wt.%
N-ethylpiperidine) at 33.0 kg/h is fed in as the catalyst. The phases are separated
and the organic phase is washed once with dilute hydrochloric acid and five times
with water. The polycarbonate solution is then concentrated, concentrated further in
an evaporation vessel, and the polymer melt is spun off via a devolatilization
25 extruder and granulated.

300 g of the polycarbonate prepared in this manner (see Table 1) are subjected to a
Soxhlet extraction with 500 ml acetone (Fluka ACS for UV spectroscopy). Approx.
5 g of extract are obtained. 50 mg of the extract are dissolved in 2 g THF, 0.19 mg
30 32 % strength sodium hydroxide solution and 0.5 g water are added and hydrolysis
is carried out over night (min. 15 h), while shaking. After the hydrolysis, the

solution is acidified with hydrochloric acid and topped up to 5 ml with THF. 15 μ l of the solution are injected into the HPLC. Detection is carried out by FLD.

Calibration is carried out by the external standard method (multiple point calibration) using the reference substance from Example 3.

The content of carbamate compounds of Example 3 in this polycarbonate sample is 220 mg/kg (220 ppm).

10 **Example 8**

Comparison example

The preparation of the polycarbonate is carried out as described in Example 7. However, the bisphenolate solution (bisphenol A) at 750 kg/h (14.93 wt.%), the solvent (methylene chloride/chlorobenzene 1:1) at 646 kg/h and the phosgene at 58.25 kg/h are fed into the reactor. Sodium hydroxide solution (32 wt.%) at 12.34 kg/h is furthermore also metered in. The second amount of sodium hydroxide solution is 36.20 kg/h; the amount of chain terminator is 34.18 kg/h at the concentrations stated in Example 5. The amount of catalyst is 33 kg/h. Working up is carried out as described in Example 7.

300 g of this polycarbonate (see Table 1) are subjected to a Soxhlet extraction with 500 ml acetone (Fluka, ACS for UV spectroscopy, Germany). The extract content is approx. 5 g. 50 mg of the extract are dissolved in 2 g THF, 0.19 g 32 % strength sodium hydroxide solution and 0.5 g water are added and hydrolysis is carried out over night (min. 15 h), while shaking. After the hydrolysis, the solution is acidified with hydrochloric acid and topped up to 5 ml with THF. 15 μ l are injected into the HPLC. Detection is carried out by FLD.

Calibration is carried out by the external standard method (multiple point calibration) using the reference substance from Example 3.

The content of carbamate compounds of Example 3 in this polycarbonate sample is 1,200 mg/kg (1,200 ppm).

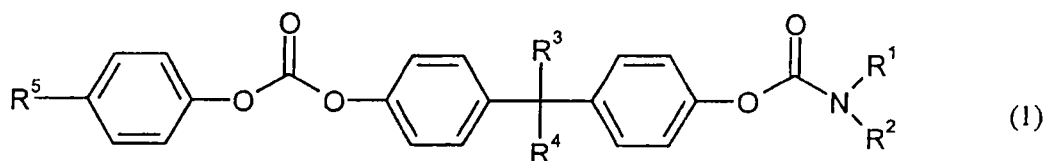
5 **Table 1**

Ex. no.	Molecular weight	Tg [°C]	Carbamate derivative of Example 3 after hydrolysis [mg/kg]	Carbamate derivative of Example 1 [mg/kg]	E field on disks after 3 h [KV/m]
5	17,500	145	-	160	< 18
6 (comp.)	17,500	145	-	600	> 18
7	17,500	145	220	-	< 18
8 (comp.)	17,700	145	1,200	-	> 18

As can be seen from the table, the polycarbonate according to the invention shows carbamate concentrations in the desired range and the associated good electrostatic properties.

Patent claims

1. Polycarbonate, characterized in that the content of carbamate derivatives of the formula (1), measured in the acetone extract of these polycarbonate resins, is 0.2 to 300 ppm



wherein

- 10 R^1 and R^2 independently of one another denote hydrogen or C_1 - C_{12} -alkyl, or

R^1 and R^2 together denote C_4 - C_{12} -alkylidene,

- 15 R^3 and R^4 independently of one another represent hydrogen, C_1 - C_{12} -alkyl or phenyl, or R^3 and R^4 with the carbon atom to which they are bonded form cyclohexyl or trimethylcyclohexyl, and

R^5 denotes hydrogen, C_1 - C_{12} -alkyl, C_5 - C_{12} -cycloalkyl, phenyl or cumyl.

- 20 2. Polycarbonate according to claim 1, comprising compounds of the formula (1) in an amount of 0.2 to 250 ppm.

3. A polycarbonate according to claim 1, wherein the content of compounds of the formula (1) in the acetone extract is 0.2 to 200 ppm.

- 25 4. Polycarbonate according to claim 1, wherein, in the formula (1)

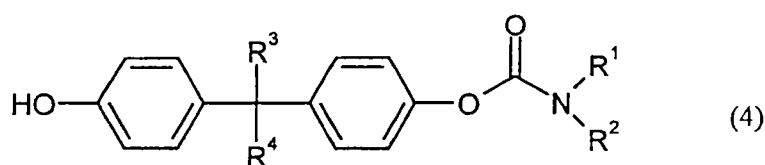
R^1 and R^2 independently of one another represent hydrogen, methyl, ethyl, propyl or butyl, or

R^1 and R^2 together represent C_4 - C_5 -alkylidene,

R^3 and R^4 independently of one another represent hydrogen, C_1 - C_8 -alkyl or phenyl, or R^3 and R^4 with the carbon atom to which they are bonded form cyclohexyl or trimethylcyclohexyl, and

R^5 denotes hydrogen, tert-butyl or cumyl.

5. Polycarbonate according to claim 1 as a substrate material.
6. Polycarbonate according to claim 1, wherein the electrostatic field, measured on injection mouldings produced therefrom at a distance of 100 mm, is not more than 18 kV/m.
7. Use of polycarbonate according to claim 1 for the production of injection mouldings.
8. Injection mouldings comprising polycarbonate according to claim 1, wherein the electrostatic field, measured at a distance of 100 mm, is not more than 18 kV/m.
9. Carrier for an optical disk, comprising polycarbonate according to claim 1.
10. Optical data storage medium comprising polycarbonate according to claim 1.
11. Polycarbonate, characterized in that the content of carbamate compounds of the formula (4)



wherein

R^1 and R^2 independently of one another denote hydrogen or C_1 - C_{12} -alkyl, or

5 R^1 and R^2 together denote C_4 - C_{12} -alkylidene and

R^3 and R^4 independently of one another represent hydrogen, C_1 - C_{12} -alkyl or phenyl, or R^3 and R^4 with the carbon atom to which they are bonded form cyclohexyl or trimethylcyclohexyl,

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measured in the acetone extract, after alkaline hydrolysis with sodium hydroxide solution, by means of HPLC is 0.2 to 500 ppm.