

Polycarbonates with Good Wettability

The present invention provides polycarbonates as a substrate material for the production of transparent injection-moulded parts, in particular for the production of injection-moulded parts to be coated, as well as moulded parts obtainable from the polycarbonates according to the invention. Moulded parts may for example include transparent sheets, lenses, optical storage media and carriers for optical storage media, or also articles from the automotive glazings sector, such as for example headlight diffusers. The invention provides in particular optical storage media and carriers for optical storage media, such as for example writeable optical data loggers that exhibit a good coatability and wettability and that are suitable for example for the application of colourants from solution, in particular from non-polar media. In addition the optical injection-moulded parts made from the polycarbonates according to the invention have a lesser tendency to become contaminated.

Transparent injection-moulded parts are important in particular in the glazings and storage media sectors.

Optical data recording materials are increasingly used as variable recording and/or archiving medium for large amounts of data. Examples of this type of optical data loggers are CDs, superaudio-CDs, CD-Rs, CD-RWs, DVDs, DVD-Rs, DVD+Rs, DVD-RWs, DVD+RWs and BDs.

Transparent thermoplastic materials such as for example polycarbonate, polymethyl methacrylate and chemical modifications thereof are typically used for optical storage media. Polycarbonate as substrate material is suitable in particular for write-once and read-many optical discs as well as for write-many optical discs, and also for the production of moulded parts in the automotive glazings sector, such as for example headlamp diffusers. This thermoplastic material has an excellent mechanical stability, is only slightly susceptible to dimensional changes, and is characterised by a high transparency and impact resistance.

Polycarbonate produced by the phase interface (interfacial) process may be used for the production of optical data loggers of the aforescribed formats, such as for

example compact discs (CDs) or digital versatile discs (DVDs). These discs often have the property that they build up a high electrostatic field during their production in the injection-moulding process. This high electrostatic field strength on the substrate leads during the production of the optical data loggers to for example the attraction of dust from the surroundings or causes the injection-moulded articles to stick together, for example disks to stick to one another, which reduces the quality of the finished injection-moulded articles and complicates the injection-moulding process.

It is furthermore known that the electrostatic charge (in the form of electrostatic fields) in particular of discs (for optical data carriers) leads to an inadequate wettability in particular with non-polar media, such as for example a non-polar colourant or application of a colourant from solvents, such as for example dibutyl ether, ethylcyclohexane, tetrafluoropropanol, cyclohexane, methylcyclohexane or octafluoropropanol. Thus, a high electrical field on the surface of the substrate during the application of a colourant in the case of writeable data loggers causes for example an irregular coating with the colourant and thus leads to defects in the information layer.

The degree of the electrostatic charging of a substrate material may be quantified for example by measuring the electric field at a certain distance from the substrate surface.

In the case of an optical data logger in which a writeable substrate is applied to the surface by a spin-coating process, a low absolute electric field strength is necessary in order to guarantee the uniform application of the writeable layer and ensure a problem-free production process.

In addition a high electrostatic field causes yield losses with regard to the substrate material on account of the aforescribed factors. This can lead to a stoppage of the respective production step and is associated with high costs.

Many approaches have been followed in order to solve this problem of a high electrostatic charge. In general antistatics are added as additives to the substrate material. Antistatic polycarbonate compositions are described for example in JP 62 207 358-A. Here, *inter alia*, phosphoric acid derivatives are added as antistatics to the polycarbonate. EP 0922 728-A describes various antistatics such as polyalkylene glycol derivatives, ethoxylated sorbitan monolaurate, polysiloxane derivatives, phosphine oxides, as well as distearylhydroxyamine, which are used individually or as mixtures. Japanese application JP 62 207 358 describes esters of phosphorous acid as additives. Sulfonic acid derivatives are described in US patent 5,668,202 . In WO 00/50 488 3,5-di-tert.-butylphenol is used as chain terminator in the phase interface process. This chain terminator leads to a low static charge of the corresponding substrate material compared to conventional chain terminators. JP 62 207 358-A describes polyethylene and polypropylene derivatives as additives for polycarbonate.

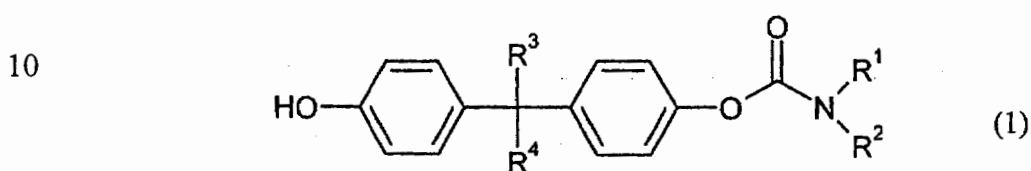
The aforescribed additives may however also have a disadvantageous effect on the properties of the substrate material, since they tend to leach out from the material. Although this is a desirable effect as regards the antistatic properties, it may lead to the formation of a coating or defective moulding. Furthermore, the content of oligomers in the polycarbonate may also lead to poorer mechanical properties and to a reduction of the glass transition temperature. In addition these additives can cause secondary reactions. The subsequent "endcapping" of polycarbonate that has been obtained by the transesterification process is complicated and the results achieved are not optimal. Moreover, high costs are involved in introducing new terminal groups into the material.

The object therefore exists of providing a composition and a substrate material that satisfies the requirements of as low a field strength as possible on the substrate surface and that avoids the aforescribed disadvantages.

This object has surprisingly been achieved if in particular such materials that contain as few defective structures as possible, in particular that contain as few special structure carbamate compounds as possible, are used for the production of optical

data loggers. A certain amount of carbamate compounds in the substrate material may arise due to the addition of additives, impurities in precursors, or due to the production process itself.

- 5 The present invention provides polycarbonates that after alkaline hydrolysis with sodium hydroxide followed by chromatography by means of high pressure liquid chromatography (HPLC) contain 0.01 to 150 ppm, preferably 0.01 to 100 ppm and particularly preferably 0.01 to 50 ppm, of compounds of the formula (1)



wherein

- 15 R^1 and R^2 independently of one another denote hydrogen or C_1 - C_{12} -alkyl, preferably methyl, ethyl, propyl, isopropyl or butyl, or R^1 and R^2 together denote C_4 - C_{12} -alkylidene, preferably C_4 - C_8 -alkylidene, particularly preferably C_4 - C_5 -alkylidene,
- 20 R^3 and R^4 independently of one another denote hydrogen, C_1 - C_{12} -alkyl, preferably C_1 - C_8 -alkyl, or phenyl, or R^3 and R^4 together with the carbon atom to which they are bonded form cyclohexyl or trimethylcyclohexyl.

- 25 Injection-moulded articles, preferably optical discs, obtainable from the polycarbonates according to the invention exhibit a low electrostatic charge after processing to form an injection-moulded article. This is important in particular for the production of optical storage media.

- 30 The polycarbonates/substrate materials according to the invention can be produced by choosing suitable process parameters.

The content of compounds of the formula 1 can be influenced by several factors. For example, the purity of the educts and auxiliary substances is important. In

addition process parameters such as the molar ratio of bisphenol and phosgene that are used, temperatures during the reaction, and the reaction and residence times may be decisive. For the person skilled in the art the aim is therefore to control the process in such a way that the limits according to the invention of the carbamate content in the substrate material are not exceeded. The described measurement of the carbamate content of the formula 1 is for the person skilled in the art a suitable way of controlling the process.

An appropriate choice of process parameters in order to obtain the substrate material may appear as follows:

The production of the substrate material may take place in a continuous phase interface process. Whereas the excess of phosgene that is used, referred to the sum of the bisphenols used, is between 3 and 100 mole %, preferably between 5 and 50 mole % in conventional continuous polycarbonate syntheses, the substrate material according to the invention is produced with phosgene excesses of 5 to 20 mole %, preferably 8 to 17 mole %. At the same time the pH value of the aqueous phase during and after the phosgene addition is maintained in the alkaline range, preferably between 8.5 and 12, by single or repeated subsequent addition of sodium hydroxide or appropriate subsequent addition of bisphenolate solution, while after the addition of catalyst the pH is adjusted to 10 to 14. The temperature during the phosgenation is 0° to 40°C, preferably 5° to 36°C.

The production of the polycarbonates according to the invention is carried out by the interfacial process. This process for polycarbonate synthesis is described copiously in the literature; reference may be made for example to H. Schnell, Chemistry and Physics of Polycarbonates, Polymer Reviews, Vol. 9, Interscience Publishers, New York, 1964, p. 33 ff., to Polymer Reviews, Vol. 10, "Condensation Polymers by Interfacial and Solution Methods", Paul W. Morgan, Interscience Publishers, New York, 1965, Chapter VIII, p. 325, to Drs. U. Grigo, K. Kircher and P.R. Müller, "Polycarbonate" in Becker/Braun, Kunststoff-Handbuch, Vol. 3/1, Polycarbonate, Polyacetale, Polyester, Cellulose-ester, Karl Hanser Verlag, Munich, Vienna, 1992, pp. 118-145 as well as to EP-A 0 517 044.

According to this process the phosgenation of a disodium salt of a bisphenol (or a mixture of various bisphenols) in 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 that are formed, and which are present mainly in the organic phase, are condensed with the aid of suitable catalysts to form high molecular weight polycarbonates dissolved in the organic phase. The organic phase is finally separated and the polycarbonate is isolated therefrom by various working up steps.

Suitable dihydroxyaryl compounds for the production of polycarbonates are those of the formula (2)

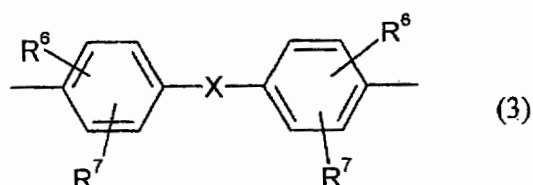


in which

Z denotes an aromatic radical with 6 to 30 C atoms, which may contain one or more aromatic nuclei, may be substituted, and may contain aliphatic or cycloaliphatic radicals and/or alkylaryls or heteroatoms as bridging members.

Preferably Z in formula (2) denotes a radical of the formula (3)

in which

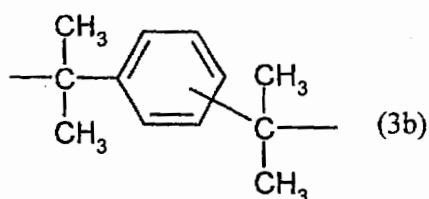
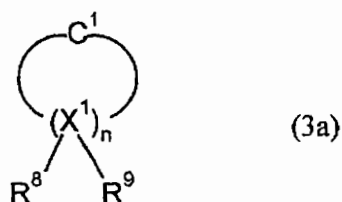


R^6 and R^7 independently of one another denote H, $\text{C}_1\text{-C}_{18}$ -alkyl, $\text{C}_1\text{-C}_{18}$ -alkoxy, halogen such as Cl or Br, or in each case optionally substituted aryl or aralkyl, preferably H or $\text{C}_1\text{-C}_{12}$ -alkyl, particularly preferably H or $\text{C}_1\text{-C}_8$ -alkyl, and most particularly preferably H or methyl, and

X denotes a single bond, -SO₂-, -CO-, -O-, -S-, C₁- to C₆-alkylene, C₂- to C₅-alkylidene or C₅- to C₆-cycloalkylidene, which may be substituted with C₁- to C₆-alkyl, preferably methyl or ethyl, and may also denote C₆-C₁₂-arylene, which may optionally be condensed with further aromatic rings containing heteroatoms.

Preferably X denotes a single bond, C₁- to C₅-alkylene, C₂- to C₅-alkylidene, C₅- to C₆-cycloalkylidene, -O-, -SO-, -CO-, -S-, -SO₂-,

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



in which

R⁸ and R⁹ denote for each X¹, which may be individually selectable, independently of one another hydrogen or C₁ to C₆-alkyl, preferably hydrogen, methyl or ethyl, and

X¹ denotes carbon and

n is a whole number from 4 to 7, preferably 4 or 5, with the proviso that on at least one atom X¹, R⁸ and R⁹ are simultaneously alkyl.

Examples of dihydroxyaryl compounds are : dihydroxybenzenes, 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, as well as their nuclear-alkylated and nuclear-halogenated compounds.

Suitable diphenols for the production of the polycarbonates to be used according to the invention are for example hydroquinone, resorcinol, dihydroxydiphenyl, bis-(hydroxyphenyl)-alkanes, bis(hydroxyphenyl)-cycloalkanes, bis-(hydroxyphenyl)-sulfides, bis-(hydroxyphenyl)-ethers, bis-(hydroxyphenyl)-ketones, bis-(hydroxyphenyl)-sulfones, bis-(hydroxyphenyl)-sulfoxides, α - α' -bis-(hydroxyphenyl)-diisopropylbenzenes, as well as their alkylated, nuclear-alkylated and nuclear-halogenated compounds.

Preferred diphenols are 4,4'-dihydroxydiphenyl, 2,2-bis-(4-hydroxyphenyl)-1-phenylpropane, 1,1-bis-(4-hydroxyphenyl)-phenylethane, 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-(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-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)-phenylethane, 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 for example in: US-A 2 999 835, 3 148 172, 2 991 273, 3 271 367, 4 982 014 and 2 999 846, in German laid-open

specifications 1 570 703, 2 063 050, 2 036 052, 2 211 956 and 3 832 396, in French patent specification 1 561 518, in the monograph by H. Schnell "Chemistry and Physics of Polycarbonates", Interscience Publishers, New York 1964, pp. 28 ff.; pp. 102 ff.; and in D.G. Legrand, J.T. Bendler "Handbook of Polycarbonate Science and Technology", Marcel Dekker, New York, 2000, pp. 72 ff.

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

The monofunctional chain terminators that are necessary to regulate the molecular weight, such as phenol or alkylphenols, in particular phenol, p-tert.-butylphenol, isooctylphenol, cumylphenol, their chlorocarbonic acid esters or acid chlorides of monocarboxylic acids or mixtures of these chain terminators, are either added together with the bisphenolate or bisphenolates to the reaction or alternatively are added at any arbitrary time during the synthesis, so long as phosgene or chlorocarbonic acid terminal groups are still present in the reaction mixture or, in the case of acid chlorides and chlorocarbonic acid esters as chain terminators, so long as sufficient phenolic terminal groups of the polymer being formed are available. Preferably however the chain terminator or terminators is/are added after the phosgenation at a site or at a time at which phosgene is no longer present but the catalyst has not yet been metered in, or the chain terminator(s) is/are metered in before, together with or in parallel with the catalyst.

In the same way branching agents or mixtures of branching agents that are possibly used are added to the synthesis, but usually before the chain terminators. Normally trisphenols, quaternary phenols or acid chlorides of tricarboxylic acids or tetracarboxylic acids are used, or also mixtures of the polyphenols or acid chlorides.

Some of the compounds that may be used containing three or more than three phenolic hydroxyl groups are for example:

phloroglucinol,

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

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

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

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

tri-(4-hydroxyphenyl)-phenylmethane,

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

2,4-bis-(4-hydroxyphenylisopropyl)-phenol,

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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.

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

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The catalysts used in the phase interface synthesis are tertiary amines, in particular triethylamine, tributylamine, trioctylamine, N-ethylpiperidine, N-methylpiperidine, N-i/n-propylpiperidine; quaternary ammonium salts such as tetrabutylammonium/tributylbenzylammonium/tetraethylammonium hydroxide/chloride/bromide/hydrogen sulfate/tetrafluoroborate; as well as the phosphonium compounds

corresponding to the ammonium compounds. These compounds are described as typical phase interface catalysts in the literature, are commercially obtainable, and are well known to the person skilled in the art. The catalysts may be added individually, as a mixture, or also together and successively to the synthesis, optionally also before the phosgenation, although addition of catalyst after the addition of phosgene is preferred unless an onium compound or mixtures of onium compounds are used as catalysts, in which case an addition before the addition of phosgene is preferred. The metering in of the catalyst or catalysts may take place in bulk, in an inert solvent, preferably the solvent of the polycarbonate synthesis, or also as aqueous solution, and in the case of tertiary amines as their ammonium salts with acids, preferably mineral acids, in particular hydrochloric acid. When using several catalysts or when metering in partial amounts of the total catalyst amount, different metering methods may of course also be employed at different sites or at different times. The total amount of catalysts used is between 0.001 and 10 mole % referred to moles of bisphenols used, preferably 0.01 to 8 mole %, particularly preferably 0.05 to 5 mole %.

Additives conventionally used for polycarbonates may also be added in the usual amounts to the polycarbonates according to the invention. The addition of additives serves to extend the service life or the colour (stabilisers), to simplify the processing (e.g. mould release agents, anti-blocking agents, antistatics) or to match the polymer properties to specific stresses (impact modifiers such as rubbers; flame retardants, colourants, glass fibres).

These additives may be added individually or in arbitrary mixtures or as several different mixtures to the polymer melt, and more particularly directly during the isolation of the polymer but also after the melting of granular material in a so-called compounding step. In this connection the additives or their mixtures may be added as a solid, i.e. as powder or as a melt, to the polymer melt. Another method of metering is to use master batches or mixtures of master batches 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.

5 Preferred heat stabilisers are for example organic phosphites, phosphonates and phosphanes, generally those compounds in which the organic radicals consist wholly or partially of optionally substituted aromatic radicals. Substituted benzotriazoles for example are used as UV stabilisers. These and other stabilisers may be used individually or in combinations and are added in the aforementioned forms to the
10 polymer.

Processing auxiliaries such as mould release agents, generally derivatives of long-chain fatty acids, may also be added. Pentaerythritol tetrastearate and glycerol monostearate for example are preferred. They are generally used alone or as a
15 mixture, preferably in an amount of 0.02 to 1 wt.%, referred to the mass of the composition.

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

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

30 Furthermore colourants such as organic dyes or pigments or inorganic pigments, IR absorbers, may be added individually, as a mixture, or also in combination with stabilisers, glass fibres, (hollow) glass spheres or inorganic fillers.

The present invention furthermore provides the extrudates and moulded parts obtainable from the polycarbonates according to the invention, in particular those extrudates and moulded parts for use in transparent applications, most particularly in the optical applications sector, such as for example panels, spider-type panels, glazings, diffuser panels, lamp coverings, or optical data loggers, such as audio CDs, CD-R(W)s, DVDs, DVD-R(W)s, minidiscs in their various read-only but also write-once and possibly also repeatedly writeable embodiments.

10 The present invention moreover provides for the use of the polycarbonates according to the invention for the production of extrudates and moulded parts.

Further applications are given by way of example, without however restricting the subject matter of the present invention:

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1. Safety panels, which as is known are necessary in many areas of buildings, vehicles and aircraft, and as visors for helmets,

2. Films,

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3. Blow mouldings (see for example US patent 2 964 794), for example 1 to 5 gallon water bottles,

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4. Translucent sheets, such as solid sheets or in particular twin-wall sheets, for example for covering buildings such as stations, greenhouses and lighting installations,

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5. Optical data storage media, such as audio-CDs, CD-R(W)s, DCDs, DVD-R(W)s, minidisks, and next generation developments.

6. Traffic light housings or road signs,

7. Foams with an open or closed, optionally printable, surface.

8. Threads and wires (see for example DE-A 1 137 167),
- 5 9. Light technology applications, optionally using glass fibres for applications in the translucent range,
- 10 10. Translucent formulations containing barium sulphate and/or titanium dioxide and/or zirconium oxide or organic polymeric acrylate rubbers (EP-A 0 634 445, EP-A 0 269 324) for producing translucent and light-scattering moulded parts,
- 15 11. Precision injection mouldings, such as holders, e.g. lens holders; here polycarbonates having a content of glass fibres and optionally additionally containing 1-10 wt.% MoS₂, referred to the total weight, are optionally used.
- 20 12. Optical instrument parts, in particular lenses for photographic and film cameras (see for example DE-A 27 01 173),
13. Light carriers, in particular optical cables (see for example EP-A 0 089 801) and lighting strips,
- 25 14. Electrical insulating materials for electrical cables and for connector housings and plug-in connectors, as well as capacitors.
15. Mobile telephone casings,
16. Network interface devices,
17. Support materials for organic photoconductors,
- 30 18. Lamps, headlights, diffusers or internal lenses,
19. Medical applications such as oxygenators, dialysis machines,

20. Food applications, such as bottles, crockery and chocolate moulds,
21. Applications in the automotive sector, such as glazings or in the form of
5 blends with ABS as bumpers.
22. Sports articles, such as slalom poles or ski boot clips,
23. Domestic items such as kitchen sinks, washbasins and letterboxes,
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24. Housings, such as electrical distribution cabinets,
25. Casings for electrical devices such as toothbrushes, hairdryers, coffee
machines, machine tools such as drills, milling machines, planers and saws.
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26. Transparent washing machine portholes.
27. Protective goggles, sunglasses, optical correction spectacles and lenses
therefor.
20
28. Lamp covers.
29. Packaging films.
- 25 30. Chip boxes, chip carriers, containers for silicon chips.
31. Miscellaneous applications such as stable doors or animal cages.

Examples

The procedure for measuring the content of compounds of the formula (1) in the polycarbonate is described hereinafter:

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500 mg of polycarbonate are dissolved in 20 g of tetrahydrofuran (THF), 1.91 g of 32% sodium hydroxide and 5 g of water are added, and the mixture is saponified overnight (at least 15 hours) while shaking. After saponification the solution is acidified with hydrochloric acid and made up to 50 ml with THF. 15 μ l are injected
10 into an HPLC instrument. Detection is carried out either with a diode array detector (DAD), fluorescence detector (FLD) or by mass spectrometry (MS).

The calibration is carried out by an external standard method (multipoint calibration).

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The method for measuring the electrostatic field on the corresponding injection moulded body, which in the present case is an optical disc, is as follows:

In order to measure the electrical field strength a field meter (EMF 581230) from the Eltec company is used. Immediately after the end of the injection moulding process
20 the moulded part (disc) is removed by a rotor arm and set aside. In this connection the disc must not come into contact with metal since this interferes in the measurement. In addition possible existing ionisers must be screened.

25 The field meter is positioned above the disc at a distance of 100 mm from the horizontal disc surface. The distance of the field meter to the inner edge of the disc is 29 mm and is thus aligned centrally above the writeable surface. The disc is not moved. The measurement of the field thus takes place in a period of 3 to 10 seconds after completion of the injection moulding process.

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The measurement instrument is connected to an x/y printer, on which the respective values are printed out. A specific integral value of the electrical field is thus associated with each measured disc. In order to limit the amount of data, 100

measurements are carried out after the start of the process, i.e. the corresponding electrical field is recorded from the first 100 discs. After in each case 60 minutes a further 100 measurements are made. After the fourth series of measurements, i.e. after ca. 3 hours, the measurement procedure is stopped.

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When carrying out the measurements care should be taken to ensure that the atmospheric humidity during the measurement is 30 to 60%, preferably 35 to 50%, and that the room temperature is 25° to 28°C.

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

Example 1

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Preparation of 1-(4-tert-butylphenyloxycarbonyloxy)-1'(piperidine carboxylic acid)-4,4'-isopropoxydiphenyl ester

9.30 g (0.025 mole) of isopropylidene diphenylbischlorocarbonic acid ester are added under argon to 150 ml of dichloromethane and cooled to 0°C. 48.49 g (0.428 mole) of N-ethylpiperidine are dissolved in 20 ml of dichloromethane and added dropwise at 0°C to the bischlorocarbonic acid ester solution. 3.76 g (0.025 mole) of tert-butylphenol dissolved in 10 ml of dichloromethane are then added dropwise at 0°C to this solution. The mixture is heated to room temperature and stirred for 3 hours. The solvent is then removed *in vacuo*. The residue is boiled in 500 ml of toluene and filtered hot. On cooling, crystals precipitate out in the mother liquor. The mother liquor is filtered and concentrated by evaporation (95°C, 25 mbar). 13.2 g of a highly viscous red oil are obtained. This is dissolved in 100 ml of ethyl acetate and, after addition of 10 g of silica gel (silica gel 60; 0.04-0.063 µm; Merck 109385/Lt.: 948 785 203), is concentrated by evaporation and added to a silica gel column (column Ø 5 cm, filling height ca. 25 cm). After chromatography with a solvent mixture of n-hexane/ethyl acetate (9:1), 2.3 g of a glassy solid are obtained.

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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).

5 Example 2

Preparation of 1-(4-tert-butylphenoxy)carbonyloxy)-1'-(4,4'-isopropylidene-diphenyl)-N,N-diethyl carbamate

10 5.0 g (0.013 mole) of isopropylidene diphenylbischlorocarbonic acid ester are added at 0°C under argon to 100 ml of dichloromethane. 4.29 g (0.042 mole) of triethylamine dissolved in 30 ml of dichloromethane are then added dropwise at 0°C to this solution. 2.02 g (0.013 mole) of tert.-butylphenol dissolved in 30 ml of dichloromethane are next added dropwise. The mixture is heated to room
15 temperature and stirred for 3 hours. The solvent is then removed *in vacuo*. The residue is boiled in 500 ml of toluene and filtered hot.

The solvent is removed *in vacuo*. The crude product is chromatographed on silica gel (height: 16 cm, Ø 5 cm, solvent n-hexane/ethyl acetate 9:1).

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2.1 g of a yellow highly viscous resin are obtained.

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).

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Example 3

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

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0.5 g of 1-(4-tert-butylphenoxy)carbonyloxy)-1'-(piperidinecarboxylic acid)-4,4'-isopropylidene diphenyl ester is dissolved in 20 g of THF, 0.5 g of 32% sodium

hydroxide and 5 g of water are added, and the mixture is saponified overnight (at least 15 hours) while shaking.

Working up:

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The aqueous phase of the THF solution is separated and the organic phase is concentrated by evaporation. The residue is taken up in diethyl ether and washed several times with water. The organic phase is dried over magnesium sulfate, filtered off from the drying agent, and the solvent is removed *in vacuo*. 1.46 g of
10 crude product are obtained, which 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 $\varnothing$ 5cm, filling height ca. 25 cm). During the further course of the working up hexane/ethyl acetate (5:1) is used as solvent mixture. 1.0 g of a white solid is obtained.

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^1H -NMR (400 MHz, CDCl_3) δ = 7.20-7.15 (m, 2 H), 7.10-7.05 (m, 2 H), 7.02-6.95 (m, 2 H), 6.75-6.68 (m, 2 H), 3.65-3.45 (m, 4 H), 1.63 (s, 6 H).

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

0.5 g of 1-(4-tert-butylphenyloxycarbonyloxy)-1'-(4,4'-isopropylidenediphenyl)-N,N'-diethyl carbamate are dissolved in 20 g of THF, 0.5 g of 32% sodium hydroxide and 5 g of water are added, and the mixture is saponified overnight (at least 15 hours) while shaking.

Purification:

The aqueous phase of the THF solution is separated and the organic phase is concentrated by evaporation. The residue is taken up in diethyl ether and washed several times with water. The organic phase is dried over magnesium sulfate, filtered off from the drying agent, and the solvent is removed *in vacuo*. The crude 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 $\varnothing$ 3 cm, filling height ca. 25 cm). During the further course of the working up hexane/ethyl acetate (1:1) is used as solvent mixture. 0.29 g of a white solid is obtained.

$^1\text{H-NMR}$ (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

The production of the polycarbonate is carried out according to the known phase interface process. A continuous production process is employed.

The bisphenolate solution (bisphenol A; alkali content 2.12 mole NaOH/mole BPA) is fed in at a rate of 750 kg/hour (14.93 wt.%), the solvent (dichloromethane/chlorobenzene 1:1) is fed in at a rate of 646 kg/hour and the

phosgene is fed in at a rate of 56.4 kg/hour, and the mixture is reacted. The temperature in the reactor is 35°C. In addition sodium hydroxide (32 wt.%) is also metered in at a rate of 9.97 kg/hour. In the course of the condensation a second amount of sodium hydroxide (32 wt.%) is fed in at a rate of 29.27 kg/hour, as well as a solution of chain terminators (11.7 wt.% of tert.-butylphenol in methylene chloride/chlorobenzene 1:1) at a rate of 34.18 kg/hour. Next N-ethylpiperidine dissolved in methylene chloride/chlorobenzene (1:1; 2.95 wt.% of N-ethylpiperidine) is fed in as catalyst at a rate of 33.0 kg/hour. 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 by evaporation, concentrated in an evaporation vessel and the polymer melt is spun through an evaporation extruder and granulated.

0.5 g of the polycarbonate thereby produced (see Table 1) is dissolved in 20 g of THF, 1.9 g of 32% sodium hydroxide and 5 g of water are added, and the mixture is saponified overnight (at least 15 hours) while shaking. After saponification the solution is acidified with hydrochloric acid and made up to 50 ml with THF. 15 µl of the solution are injected into an HPLC instrument. Detection is carried out with FLD.

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The calibration is performed according to an external standard method (multipoint calibration) using the reference substance from Example 3.

The content of carbamate compounds of Example 2 in the polycarbonate according to Example 5 is 37 mg/kg (37 ppm).

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Example 6

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Comparison example

The preparation of the polycarbonate is carried out as described in Example 5. However, the bisphenolate solution (bisphenol A) is fed at a rate of 750 kg/hour (14.93 wt.%), the solvent (dichloromethane/chlorobenzene 1:1) is fed at a rate of 646 kg/hour and the phosgene is fed at a rate of 58.25 kg/hour into the reactor. In addition sodium hydroxide (32 wt.%) is also metered in at a rate of 12.34 kg/hour. The second amount of sodium hydroxide is 36.20 kg/hour; the amount of chain terminator is 34.18 kg/hour at the concentrations specified in Example 5. The amount of catalyst is 33 kg/hour. The working up is carried out as described in Example 5.

0.5 g of the polycarbonate thereby produced (see Table 1) is dissolved in 20 g of THF, 1.9 g of 32% sodium hydroxide and 5.0 g of water are added, and the mixture is saponified overnight (at least 15 hours) while shaking. After saponification the solution is acidified with hydrochloric acid and made up to 5 ml with THF. 15 µl of the solution are injected into an HPLC instrument. The detection is carried out with FLD.

The calibration is carried out according to an external standard method (multipoint calibration) using the reference substance from Example 3.

The content of carbamate compounds of Example 2 in this polycarbonate sample is 285 mg/kg (285 ppm)

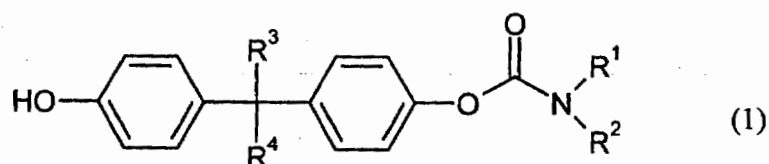
Table 1

Example No.	Molecular Weight M _w [g/mole]	T _g [°C]	Carbamate Derivative of Example 2 after Hydrolysis [mg/kg]	Electrical Field on Discs after 3 hours [kV/m]
5	17,500	145	37	<18
6	17,700	145	285	>18

5 As is evident from the table, the polycarbonate according to the invention has carbamate concentrations in the desired range and exhibits the good electrostatic behaviour associated therewith.

Patent Claims

1. A polycarbonate resin produced by the continuous interfacial process characterized in that after alkaline hydrolysis with sodium hydroxide contains an amount of 0.01 to 150 ppm of carbamate compounds according to formula (1)



said amount measured by high pressure liquid chromatography

wherein

R^1 and R^2 independently of one another denote hydrogen or C_1 - C_{12} -alkyl, or together denote C_4 - C_{12} -alkylidene,

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

said process comprising phosgene reacting with at least one bisphenol at 8 to 17% molar excess of phosgene relative to said bisphenol.

2. Polycarbonate according to claim 1, containing compounds of the formula (1) in an amount of 0.01 to 100 ppm.
3. Polycarbonate according to claim 1, in which the proportion of compounds of the formula (1) is 0.01 to 50 ppm.
4. Polycarbonate according to claim 1, wherein in the formula (1)

R^1 and R^2 independently of one another denote hydrogen, methyl, ethyl, propyl or butyl, or R^1 and R^2 together denote C_5 - C_6 -alkylidene,

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

5. Polycarbonate according to claim 1 as substrate material.
6. Polycarbonate according to claim 1, in which the electrostatic field measured at a distance of 100 mm from injection-moulded parts produced from the polycarbonate is not more than 18 kV/m.
7. Use of polycarbonate according to claim 1 for the production of injection-moulded parts.
8. Injection-moulded parts obtainable from polycarbonate according to claim 1, in which the electrostatic field measured at a distance of 100 mm is not more than 18 kV/m.
9. Carrier according to claim 8 for an optical disc.
10. Optical data storage media containing a carrier that can be obtained from polycarbonate according to claim 1.