

PATENT SPECIFICATION

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 (72) Inventor DIMITRIOS KARANATSIOS

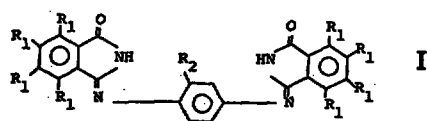


(54) ISO-INDOLINE COMPOUNDS

(71) We, SANDOZ LTD., of 35 Lichtstrasse, 4002 Basle, Switzerland, a Swiss Body Corporate, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The invention relates to iso-indolinone compounds.

The invention provides compounds of formula I,



in which the

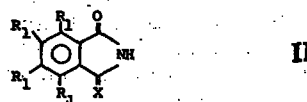
R_1 's, independently, are chlorine or bromine, and

R_2 is fluorine, chlorine, bromine, methyl, ethyl, ethoxy or tri-fluoromethyl.

Preferably the R_1 's are identical, most preferably all being chlorine.

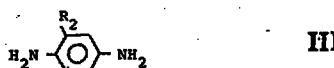
Preferably R_2 is fluorine, chlorine, bromine, methyl, ethyl or trifluoromethyl, more preferably fluorine, chlorine, bromine or methyl, still more preferably fluorine, chlorine or bromine and most preferably chlorine.

The invention also provides a process for the production of compounds of formula I, which process comprises condensing a compound of formula II,



wherein

X is NH, S, (Cl)₂ or (OR)₂ where R is alkyl, preferably C₁₋₄ alkyl and more preferably methyl or ethyl, with a compound of formula III,



The above process is carried out in conventional manner to obtain condensation of 2 mols of compound II with 1 mol of compound III. In the compound of formula II, X is preferably (Cl)₂.

The compounds of formulae II and III are either known or may be obtained from available starting materials in conventional manner.

The resulting compounds of formula I may be isolated and purified in conventional manner.

The compounds of formula I are useful as pigments, e.g. for the mass pigmenting of synthetic plastics and resins, free from or containing solvents, e.g. viscose, cellulose acetate, polyethylene, polystyrene, polyvinyl chloride and synthetic leathers and rubbers, of surface coatings, such as paints, whether of an oil or water base, lacquers and inks. They may also be used in pigment printing, textile coating and for pigmenting paper in the mass. They may be employed for such uses in conventional manner and in conventional amounts.

The pigmentations obtained employing the compounds of formula I possess notably good fastness to light, weathering, migration, bleeding, over-varnishing and solvents. They also have good chemical resistance. The compounds give notably good depths of colour and have notably good application properties, e.g. resistance to flocculation and crystallisation. Their dispersion and covering capacity are also notably good. Of particular interest, however, is the light-fastness of the pigmentations obtained.

The pigments can generally be employed without post-treatment, i.e. direct from synthesis and after washing.

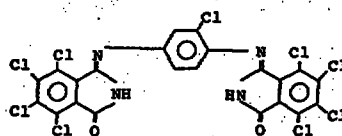
The following Examples, in which all parts and percentages are by weight and all temperatures in degrees centigrade, illustrate the invention.

Example 1.

34 Parts of 3,3,4,5,6,7 - hexachloro - iso-indolin - 1 - one are dissolved at 110° in 275 parts of o-dichlorobenzene. A solution of 7.2

parts of 2 - chloro - 1,4 - diaminobenzene in 180 parts of o-dichlorobenzene (dissolved under a nitrogen atmosphere at 110°) is added over the course of 12 minutes to this solution under a nitrogen atmosphere, and the yellow suspension which forms immediately is stirred under a nitrogen atmosphere at 110° for 17 hours.

The resulting solution is filtered whilst hot, the filter residue then being washed with hot o-dichlorobenzene, methanol and hot water. After drying, the pure, deep, greenish-yellow pigment dyestuff of formula



is obtained, which gives pigmentations of notable fastness to migration, light and solvents, and can be used without further after-treatment.

The pigments of the following table may be produced in a manner similar to the above procedure. They are of formula I, above.

TABLE

Example No.	R ₁ 's	R ₂
2	Cl	-CH ₃
3	do.	-CF ₃
4	do.	-F
5	do.	-OC ₂ H ₅
6	do.	-Br
7	Br	-Cl
8	do.	-F
9	Cl	-C ₂ H ₅

Application Example.

0.5 Parts of the pigment according to Example 1, and 5 parts of titanium dioxide pigment, are added to a basic mixture of

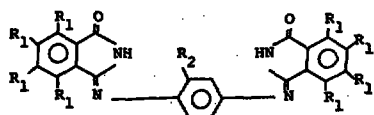
63 parts of polyvinyl chloride emulsion,
32. parts of dioctyl phthalate,
3 parts of commercial epoxy softener,
1.5 parts of stabilizer (barium-cadmium-stearate),

and they are mixed together intimately.

The mixture is milled for 8 minutes for improved pigment distribution in a mixing roller set which is heated to 160°C and which has friction rollers (one roller has a speed of 20, the other 25 revolutions per minute), and the sheet obtained which is of 0.3 mm thickness and is of a yellow shade, is removed.

WHAT WE CLAIM IS:—

1. A compound of formula I,



in which the

R₁'s, independently, are chlorine or bromine, and

R₂ is fluorine, chlorine, bromine, methyl, ethyl, ethoxy or trifluoromethyl.

2. A compound of Claim 1, wherein the R₁'s are identical.

3. A compound of Claim 2, wherein the R₁'s are all chlorine.

4. A compound of any preceding claim, wherein R₂ is fluorine, chlorine, bromine, methylethyl or trifluoromethyl.

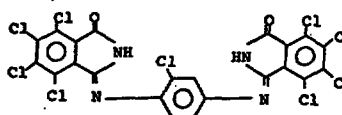
5. A compound of Claim 4, wherein R₂ is fluorine, chlorine, bromine or methyl.

6. A compound of Claim 5, wherein R₂ is fluorine, chlorine or bromine.

7. A compound of any one of the preceding claims, wherein R₂ is other than fluorine.

8. A compound of Claim 6, wherein R₂ is chlorine.

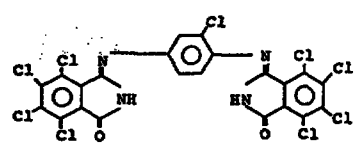
9. A compound of formula



(11)



- parts of 2 - chloro - 1,4 - diaminobenzene in 180 parts of o-dichlorobenzene (dissolved under a nitrogen atmosphere at 110°) is added over the course of 12 minutes to this
- 5 solution under a nitrogen atmosphere, and the yellow suspension which forms immediately is stirred under a nitrogen atmosphere at 110° for 17 hours.
- 10 The resulting solution is filtered whilst hot, the filter residue then being washed with hot o-dichlorobenzene, methanol and hot water. After drying, the pure, deep, greenish-yellow pigment dyestuff of formula



is obtained, which gives pigmentations of notable fastness to migration, light and solvents, and can be used without further after-treatment.

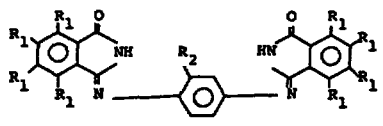
The pigments of the following table may be produced in a manner similar to the above procedure. They are of formula I, above.

TABLE

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2	Cl	-CH ₃
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7	Br	-Cl
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9	Cl	-C ₂ H ₅

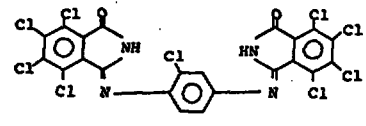
- Application Example.
- 25 0.5 Parts of the pigment according to Example 1, and 5 parts of titanium dioxide pigment, are added to a basic mixture of
- 63 parts of polyvinyl chloride emulsion,
32 parts of dioctyl phthalate,
3 parts of commercial epoxy softener,
- 30 1.5 parts of stabilizer (barium-cadmium-stearate),
- and they are mixed together intimately.
- The mixture is milled for 8 minutes for improved pigment distribution in a mixing roller set which is heated to 160°C and which has friction rollers (one roller has a speed of 20, the other 25 revolutions per minute), and the sheet obtained which is of 0.3 mm thickness and is of a yellow shade, is removed.

40 WHAT WE CLAIM IS:—
1. A compound of formula I,



I

- in which the R₁'s, independently, are chlorine or bromine, and
- R₂ is fluorine, chlorine, bromine, methyl, ethyl, ethoxy or trifluoromethyl.
2. A compound of Claim 1, wherein the R₁'s are identical.
3. A compound of Claim 2, wherein the R₁'s are all chlorine.
4. A compound of any preceding claim, wherein R₂ is fluorine, chlorine, bromine, methylethyl or trifluoromethyl.
5. A compound of Claim 4, wherein R₂ is fluorine, chlorine, bromine or methyl.
6. A compound of Claim 5, wherein R₂ is fluorine, chlorine or bromine.
7. A compound of any one of the preceding claims, wherein R₂ is other than fluorine.
8. A compound of Claim 6, wherein R₂ is chlorine.
9. A compound of formula



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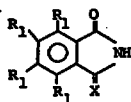
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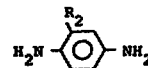
10. A compound of Claim 3, wherein R_2 is methyl.
11. A compound of Claim 3, wherein R_2 is trifluoromethyl.
5 12. A compound of Claim 3, wherein R_2 is fluorine.
13. A compound of Claim 3, wherein R_2 is ethoxy.
10 14. A compound of Claim 3, wherein R_2 is bromine.
15 15. A compound of Claim 3, wherein R_2 is ethyl.
16. A compound of Claim 2, wherein the R_1 's are all bromine and R_2 is chlorine.
15 17. A compound of Claim 2, wherein the R_1 's are all bromine and R_2 is fluorine.
18. A compound of Claim 1, in which R_2 is chlorine, bromine, methyl, ethyl, ethoxy or trifluoromethyl.
20 19. A process for the production of a compound of formula I, stated in Claim 1, comprising condensing a compound of formula II



II

- 25 wherein
X is NH , S, $(Cl)_2$ or $(OR)_2$, where R is

alkyl, with a compound of formula III,



III

20. A process according to Claim 19, substantially as hereinbefore described with reference to any one of the foregoing Examples 1 to 9. 30
21. A compound of formula I, stated in Claim 1, whenever obtained by a process according to Claim 19 or 20. 35
22. A process for pigmenting paper or synthetic plastics or resins in the mass comprising employing as pigmenting agent a compound of any one of Claims 1 to 18 or 21. 40
23. A pigment printing or textile coating process wherein a compound of any one of Claims 1 to 18 or 21 is employed as pig-
menting agent. 45
24. A pigmented surface coating medium comprising, as pigment, a compound of any one of Claims 1 to 18 or 21.

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