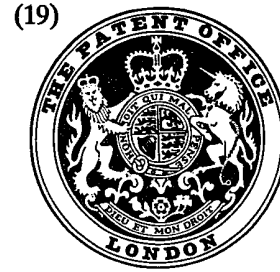


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C6C K

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(54) DISPERSIONS

5 (71) We, IMPERIAL CHEMICAL INDUSTRIES LIMITED, Imperial Chemical House, Millbank, London SW1P 3JF a British Company 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:

10 This invention relates to dispersions of solids in polar organic liquids in the presence of a dispersing agent.

According to the present invention we provide a dispersion of a particulate solid in a polar organic medium in the presence of a dispersing agent which is a tertiary amine or a salt thereof with an aromatic carboxylic or sulphonic acid, the amine containing at least one polymeric group which is a poly(lower alkylene oxy) chain comprising at least 50% propylene oxy groups or a poly(carbonyl lower alkylene oxy) chain.

15 By the term lower alkylene in the above definition of the dispersing agent and elsewhere in the specification and claims we mean an alkylene group containing from one to eight and preferably from two to six carbon atoms.

Where the dispersing agent contains a poly(lower alkylene oxy) chain or chains it is preferred that from 75% to 100% of the lower alkylene oxy groups are propylene oxy groups, the remainder preferably being ethylene oxy groups. It is however further preferred that the polymeric group comprises a poly(propylene oxy) chain linked to the nitrogen atom of the tertiary amine by an ethylene oxy group.

20 Each poly(lower alkylene oxy) chain preferably carries from 3 to 50 lower alkylene oxy groups and more preferably from 7 to 20 of such groups. It is also preferred that there are from 1 to 3 and more preferably 1 such poly(lower alkylene oxy) chains contained in each molecule of the dispersing agent.

Where the dispersing agent contains a poly(carbonyl lower alkylene oxy) chain or chains it is preferred that the lower alkylene group contains 5 carbon atoms preferably as a chain of 5 methylene groups which lie between the oxygen atom and the carbonyl group, so that the dispersing agent contains one or more poly(E-caprolactone) chains. Each poly(carbonyl lower alkylene oxy) chain preferably contains from 3 to 50 carbonyl lower alkylene oxy groups and more preferably from 7 to 20 such groups. It is also preferred that there are one or two, especially one, poly(carbonyl lower alkylene oxy) chain contained in the dispersing agent.

35 The term polar organic medium includes organic liquids and resins capable of forming moderate or strong hydrogen bonds as described in the article entitled "A three dimensional approach to solubility" by Crowley et al in Journal of Paint Technology, Vol. 38, 1966, at page 269. Such organic liquids generally have a hydrogen bonding number of 5 or more as defined in the above-mentioned article.

40

As examples of such polarorganic liquids there may be mentioned, amines, ethers, especially lower alkyl ethers, organic acids, esters, ketones, glycols, alcohols and amides. Numerous specific examples of such moderately and strongly hydrogen bonding liquids are given in the book entitled "Compatibility and Solubility" by Ibert Mellan (published in 1968 by Noyes Development Corporation) in Table 2.14 on pages 39 and 40 and these liquids all fall within the scope of the term polar organic liquid as used in this specification.

Preferred polar organic liquids are dialkyl ketones, alkyl esters of alkanecarboxylic acids, alkanols and especially those containing up to and including a total of 6 carbon atoms. As examples of the preferred and especially preferred liquids there may be mentioned dialkyl ketones such as acetone, methylethylketone (MEK), diethylketone, diisopropylketone, methylisobutylketone and diisobutylketone; alkyl esters such as methyl acetate, ethyl acetate, isopropyl acetate, ethylformate, methyl propionate and ethyl butyrate and alkanols such as methanol, ethanol, *n*-propanol, isopropanol, *n*-butanol and isobutanol.

Preferred resins are film forming resins such as are suitable for use in the preparation of inks, paints and pigment chips for use in inks and paints. As examples of such polar resins there may be mentioned polyesters such as nitrocellulose, cellulose acetate/propionate (cap) and acrylics, polyamides such as Versamid (Trade Mark) and Wolfamide (Trade Mark) and cellulose ethers such as ethylcellulose and ethyl hydroxy ethylcellulose.

It is preferred that the particulate solid is a pigment or a dyestuff. Where the solid is an inorganic pigment it is preferred that the dispersing agent is a free tertiary amine as hereinbefore defined and that the polar organic liquid is a lower alkanol. Where the solid is an organic pigment or dyestuff it is preferred that the dispersing agent is the salt of a tertiary amine as hereinbefore defined with an aromatic acid containing at least two benzene rings and more especially a coloured acid. By a coloured acid we mean an organic pigment or a dyestuff containing one or more sulphonic acid or carboxylic acid groups. It is preferred also that the coloured acid is an acid of the same organic pigment or dyestuff that forms the particulate solid. An especially preferred coloured acid is a copper phthalocyanine sulphonic acid especially one containing an average of from 1 to 2 sulphonic acid groups when used in the preparation of a dispersing agent for dispersing a copper phthalocyanine pigment.

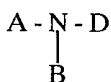
As examples of inorganic pigments there may be mentioned titanium dioxide, zinc oxide, cadmium sulphide, iron oxides, vermilion, ultramarine and chrome pigments including chromates of lead, zinc, barium and calcium, the various mixtures and modifications thereof such as are commercially available as greenish-yellow to red pigments under the names primrose, lemon, middle, orange, scarlet and red chromes and especially Prussian Blue.

As examples of organic pigments there may be mentioned pigments of the azo, thioindigo, anthraquinone, anthanthrone, isobenzanthrone or triphendioxazine series, vat dye pigments, phthalocyanine pigments such as copper phthalocyanine and its nuclear halogenated derivatives and copper tetraphenyl and octaphenyl phthalocyanines, quinacridone pigments and lakes of acid, basic and mordant dyestuffs, and especially α and β form copper phthalocyanines and carbon black which for dispersion purposes is more conveniently classified as an organic pigment.

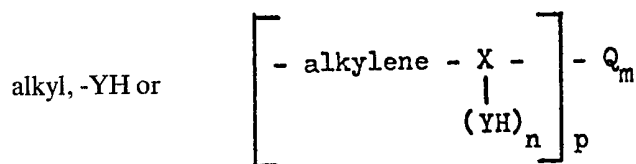
Such pigments are described in, for example, Volume 2 of the 2nd Edition of the Colour Index which was published in 1956 under the heading "Pigments" and in subsequent authorised amendments thereto.

As examples of dyestuffs there may be mentioned dyestuffs of the azo series, including monazo and diazo dyestuffs and metallised derivatives thereof, anthraquinone, nitro, phthalocyanine, methine, styryl, naphthoperinone, quinphthalone, diarylmethane, triaryl-methane, xanthine, azine, oxazine and thiazine series. Such dyestuffs are preferably disperse dyestuffs but may be water-soluble dyestuffs such as basic, acid, or direct dyestuffs, and if desired the dyestuffs can contain reactive groups, that is to say groups capable of forming covalent bonds with textile materials so that the dyestuffs become chemically attached to the textile materials. Examples of such classes of dyestuffs are given in the appropriate sections of Volumes 1 and 2 of the second edition of Colour Index and the subsequent authorised amendments thereto.

A preferred form of the tertiary amine which may be used as the dispersing agent either directly, or in the form of a substituted ammonium with a mineral acid or an aromatic sulphonic or carboxylic acid, has the general formula:



wherein each of A, B and D independently represents



wherein each X independently represents an oxygen or a nitrogen atom

provided that, where X is oxygen $m + n = 1$ and $p = 1$

and where X is nitrogen $m + n = 2$ and p is from 1 to 30;

each Q independently represents alkyl or -YH; and

each Y independently represents a poly(lower alkylene oxy) chain containing from 3 to 50 lower alkylene oxy groups comprising at least 50% propylene oxy groups provided that,

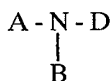
(i) at least one of A, B and D contains a poly(lower alkylene oxy) chain, and

(ii) not more than one of the alkylene or alkyl groups contains more than 8 carbon atoms.

In the above formula the alkyl and alkylene groups, none of which preferably contains more than 8 carbon atoms, may be substituted provided the substituents do not contain active or ionisable hydrogen atoms but it is preferred that they be unsubstituted. As examples of suitable substituents there may be mentioned chlorine, bromine and cyano.

It is preferred that each poly(lower alkylene oxy) chain contains not more than one ethylene oxy group and further that this is attached directly to the N atom of the amine, the remaining groups being propylene oxy groups. The preferred value for p is 1 and it is further preferred that X is oxygen.

Preferred amines of the general formula:



are:

alkyl-N-(-YH)₂,

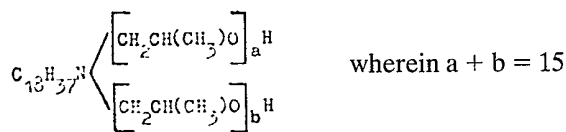
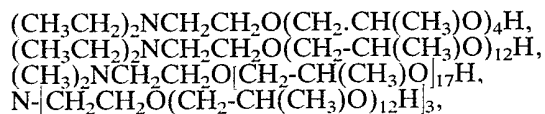
alkyl N-(alkylene-O-YH)₂,

N-(alkylene-O-YH)₃,

and especially (alkyl)₂N-alkylene-O-YH.

wherein Y has the meaning hereinbefore defined, and especially wherein the alkylene is ethylene and YH is a poly(propylene oxy) chain.

As specific examples of suitable amines there may be mentioned,



The dispersions of the invention preferably contain from 10% to 80% and more especially from 20% to 70% by weight of the solid based on the total weight of the dispersion depending on the density of the solid. The quantity of dispersing agent contained in these dispersions is preferably from 2% to 50% and more especially from 5% to 30% by weight based on the weight of the solid.

The dispersions of the invention are particularly useful in the preparation of inks based on polar solvents especially printing inks for use in package printing. They are however also of use in stationery inks and paints which are based on polar solvents. Where the solvent is compatible with a plastics material, e.g. a plasticiser, the dispersion may be used to introduce the solid, especially where the solid is a pigment, into the plastics material.

According to a further feature of the invention we provide an ink especially a printing ink containing a dispersion as hereinbefore defined.

The inks which are made from the above mentioned dispersions have enhanced gloss, transparency, brightness and strength compared with such compositions not containing these components or prepared from conventional pigment chips which are expensive and difficult to prepare.

5 The dispersions of the invention can be obtained by any of the conventional and well known methods of preparing dispersions. Thus the solid, the dispersing agent and the polar organic medium preferably in a liquid form may be mixed in any order and the mixture then subjected to a mechanical treatment to reduce the particle size of the solid, for example by ball milling, bead milling or gravel milling until a dispersion is formed in which the mean diameter of the particles of the solid is desirably less than 10 microns and preferably less than 1 micron. 10

Where the dispersing agent is soluble in the polar organic medium, the solid can be treated to reduce its particle size independently of the dispersing agent and this added subsequently following which addition the dispersion can be obtained by stirring or otherwise agitating the mixture. 15

The dispersing agents can be added to a mixture of a pigment or dyestuff and a resin, such as nitrocellulose to prepare pigment compositions such as chips and the similar compositions by for example a hot milling process. Such compositions may subsequently be dissolved in a polar organic solvent to form a liquid pigment dispersion or ink. 20

Alternatively the dispersing agents can be added to a conventional mill bases, that is dispersions of pigments in solvent/resin solutions either before, during or after milling in order to effect improvements in the inks produced therefrom. 25

According to further features of the invention we provide pigment/resin chips and mill bases as hereinbefore defined, which contain the dispersing agents as hereinbefore defined and also inks prepared from such chips and millbases. 30

Where the dispersing agent is the salt of a tertiary amine with an acid it is preferred to prepare the dispersing agent in situ by mixing together the amine, the acid and the polar organic medium following which the amine and acid react together to form the amine salt which is the dispersing agent. The solid is then added the dispersion prepared as hereinbefore described. 35

Whilst the dispersing agent may be soluble to a substantial extent in the polar organic medium it is not essential that it is more than sparingly soluble and in fact there is some advantage with sparingly soluble dispersing agents since these have a lesser tendency to bleed during applications of the dispersions and inks made from them. 40

The dispersing agents containing a poly(lower alkylene oxy) chain which are used to form the dispersions of the invention may be prepared by reacting the appropriate lower alkylene oxide or mixture of such oxides with an amine containing active hydrogen atoms, e.g. those present in amino and hydroxyl groups, so that each active hydrogen atom is replaced by a poly(lower alkylene oxy) chain, in a known manner. 45

The dispersing agents containing a poly(carbonyl lower alkylene oxy) chain which are used to form the dispersions of the invention may be prepared by reacting the appropriate lower alkylene lactone with a tertiary amine containing at least one primary or secondary amino group or a hydroxyl group in a known manner so that each of such groups becomes substituted by a poly(carbonyl lower alkylene oxy) chain. 50

The agents labelled Agent A to I described below are used either as such or after neutralisation with an acid as dispersing agents to form the dispersions described in the Examples which follow and which serve to further illustrate the present invention. 55

Agent A

8 Parts of potassium are dissolved in 450 parts of 2-diethyl-aminoethanol and propylene oxide is added at 105°C under increased pressure until the equivalent by titration with acid reaches 420. 60

The following are prepared in a similar manner from hydroxyamines and propylene oxide:-

Agent	Hydroxyamine	Equivalent
B	2-diethylaminoethanol	806
C	triethanolamine	875
D	"	2130
E	2-dimethylaminoethanol	1095

Agent F

A mixture of 71.3 parts of E-caprolactone, 8.1 parts of 3-dimethylaminopropylamine and 0.1 part of tetrabutyl titanate is stirred for 1½ hours at 160-165°C under nitrogen. On cooling it forms a hard wax of equivalent 1016. 65

Agent G

This is prepared similarly but using 150.6 parts of caprolactone. The wax has an equivalent of 2050.

Agent H

- 5 This is an adduct prepared from one molecular proportion of 2-diethylaminoethanol and a mixture of 9 molecular proportions of ethylene oxide and 9 molecular proportions of propylene oxide. 5

Agent I

- 10 30 Parts of polyethyleneimine of molecular weight 1200 (sold as PEI-12 by Dow Corp.) is stirred at 40-50°C whilst 81.8 parts of propylene oxide is gradually added during 11 hours. After removing unchanged propylene oxide at 100°/18 mm the product weighs 74.3 parts. A solution of 1.55 parts of potassium hydroxide in 2.65 parts of water is added and the water removed at 100°C/18 mm. The mixture is stirred at 100-110°C while 204 parts of propylene oxide is added during 30 hours. After removing unchanged propylene oxide at 100/18 mm the yield is 275 parts of pale amber coloured viscous liquid. 15

Example 1

- 20 A mixture of 0.53 part of Agent B, 0.37 part of a copper phthalocyanine sulphonic acid of average degree of sulphonation 1.3 and 6.1 parts of ethanol is ball milled for 2 hours to form the salt, then 3 parts of β-form copper phthalocyanine is added and milling continued for a further 16 hours, to give a fluid dispersion of the pigment which is suitable for use in a printing ink, e.g. for gravure or flexographic printing. 20

- 25 The following table gives further examples of fluid dispersions of the invention which are obtained by milling together in the manner described above (except that when the copper phthalocyanine sulphonic acid is omitted the initial 2 hours' milling without pigment is unnecessary) the pigment and the number of parts thereof listed in column 2 of the table, the agents and the number of parts thereof listed in columns 3 and 4 of the table and sufficient of the organic liquids listed in column 5 of the table to bring the total weight to 10 parts. In the table CPC stands for copper phthalocyanine. Propomeen HT/25 is an adduct from crude octadecylamine and 15 molecular proportions of propylene oxide (Propomeen is a Trade Mark belonging to Armour-Hess). 30

<i>Ex.</i>	<i>Pigment and amount thereof</i>	<i>Agent and amount thereof</i>	<i>COPPER PHTHALOCYANINE sulphonic acid and amount thereof</i>	<i>Organic liquid</i>	
35	2 3 parts of β-form CPC	0.38 part of Agent A	0.52 part of CPC (SO ₃ H) _{1.3}	ethanol	35
40	3 "	0.51 part of Agent B	0.31 part of CPC monosulphonic acid	"	40
	4 "	0.54 part of Agent C	0.36 part of CPC (SO ₃ H) _{1.3}	"	
45	5 "	0.71 part of Agent D	0.19 part of CPC (SO ₃ H) _{1.3}	"	45
	6 "	0.59 part of Agent E	0.31 part of CPC (SO ₃ H) _{1.3}	"	
50	7 "	0.42 part of Agent B	0.18 part of CPC (SO ₃ H) _{1.3}	"	50
55	8 3 part of polychloro CPC	0.53 part of Agent B	0.37 part of CPC (SO ₃ H) _{1.3}	"	55
	9 3 parts of carbon black	"	"	"	
60	10 3 parts of indanthrone	"	"	"	60

	<i>Ex.</i>	<i>Pigment and amount thereof</i>	<i>Agent and amount thereof</i>	<i>copper phthalocyanine sulphonic acid and amount thereof</i>	<i>Organic liquid</i>	
5	11	3 parts of α -form partially chlorinated CPC	0.53 part of Agent B	0.37 parts of CPC (SO ₃ H) _{1,3}	ethanol	5
10	12	3 parts β -form CPC	"	"	isopropanol	10
	13	"	0.6 part of Propomeen HT/25	0.3 part of CPC (SO ₃ H) _{1,3}	"	
15	14	"	0.58 part of Agent F	0.32 part of CPC (SO ₃ H) _{1,3}	methylethylketone	15
	15	"	0.7 part of Agent G	0.2 part of CPC (SO ₃ H) _{1,3}	"	
20	16	"	"	"	ethyl acetate	20
	17	"	0.53 part of Agent B	0.37 part of CPC (SO ₃ H) _{1,3}	butanol	
25	18	3 parts of Prussian Blue	0.9 part of Agent A	Nil	ethanol	25
	19	"	0.1 part of Agent B	"	"	30
	20	"	0.5 part of Propomeen HT/25	"	"	
35	21	7 parts of lead sulphochromate	0.35 part of Agent B	"	"	35
	22	5 parts of chromium oxide	0.5 part of Agent B	"	"	40
	23	5 parts of titanium dioxide	0.5 part of Agent B	"	"	45
	24	7 parts of iron oxide	0.7 part of Agent B	"	"	
50	25	5 parts of Prussian Blue	0.5 part of Propomeen HT/25	"	isopropanol	50
	26	"	0.5 part of the benzoate of Agent B	"	"	55

Ex.	Pigment and amount thereof	Agent and amount thereof	copper phthalocyanine sulphonic acid and amount thereof	Organic liquid	
5	27	"	0.5 part of the p-toluene-sulphonate of Agent B	"	5
10	28	3 parts of β -form CPC	0.61 part of Agent H	9.29 part of CPC (SO ₃ H) _{1,3}	10
	29	"	0.53 part of Agent I	0.37 part of CPC (SO ₃ H) _{1,3}	
15	<i>Example 30</i>				15
20	20 parts of β -copper phthalocyanine is mixed with 80 parts of a varnish medium consisting of 15 parts of nitrocellulose, 17 parts of isopropyl acetate, 58 parts of ethanol 74 O.P., 4.8 parts of Agent B and 3.2 parts of CPC (SO ₃ H) _{1,3} . The mixture is milled for 18 hours in a Ball Mill before dilution with 100 parts of a varnish medium comprising 15 parts nitrocellulose, 17 parts isopropyl acetate and 68 parts of ethanol 74OP. This mill base is suitable for mixing with further solvent, varnish or other conventional additives for use in printing inks or other surface coating compositions.				20
25	WHAT WE CLAIM IS:				25
25	1. A dispersion of a particulate solid in a polar organic medium in the presence of a dispersing agent which is a tertiary amine or a salt thereof with an aromatic carboxylic or sulphonic acid, the amine containing at least one polymeric group which is a poly(lower alkylene oxy) chain comprising at least 50% propylene oxy groups or a poly(carbonyl lower alkylene oxy) chain.				25
30	2. A dispersion according to Claim 1 wherein the polymeric group is a poly(lower alkylene oxy) chain in which from 75% to 100% of the lower alkylene groups are propylene.				30
35	3. A dispersion according to Claim 1 or Claim 2 wherein the polymeric group is a poly(propylene oxy) group linked to the nitrogen atom of the tertiary amine by an ethylene oxy group.				35
35	4. A dispersion according to any one of Claims 1 to 3 wherein there are from 1 to 3 poly(lower alkylene oxy) chains contained in each molecule of the dispersing agent.				35
40	5. A dispersion according to Claim 1 wherein the polymeric group is a poly(carbonyl lower alkylene oxy) chain and the lower alkylene group comprises a chain of 5 methylene groups between the oxygen atom and the carbonyl group.				40
40	6. A dispersion according to any one of Claims 1 to 5 wherein there is a single polymeric group in each chain.				40
45	7. A dispersion according to any one of Claims 1 to 6 wherein there are from 3 to 50 monomeric units in each polymeric group.				45
45	8. A dispersion according to any one of Claims 1 to 7 wherein there are from 7 to 20 monomeric units in each polymeric group.				45
50	9. A dispersion according to any one of Claims 1 to 8 wherein the polar organic medium is capable of forming moderate to strong hydrogen bonds as hereinbefore defined.				50
50	10. A dispersion according to any one of Claims 1 to 9 wherein the polar organic medium has a hydrogen bonding number of 5 or more.				50
55	11. A dispersion according to any one of Claims 1 to 10 wherein the polar organic medium is a liquid at ambient temperatures.				55
55	12. A dispersion according to any one of Claims 1 to 11 wherein the polar organic medium is selected from dialkyl ketones, alkyl esters of alkane carboxylic acids and alkanols.				55
55	13. A dispersion according to Claim 12 wherein each molecule of the organic medium contains not more than 6 carbon atoms.				55
60	14. A dispersion according to any one of Claims 1 to 10 wherein the organic medium is a film-forming resin.				60
60	15. A dispersion according to Claim 14 wherein the resin is selected from the group consisting of polyesters, polyamides and cellulose ethers.				60
65	16. A dispersion according to any one of Claims 1 to 15 wherein the solid is a pigment or a dyestuff.				65
65	17. A dispersion according to Claim 16 wherein the pigment is inorganic and the dispersing agent is a free tertiary amine as defined in Claim 1.				65

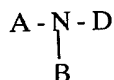
18. A dispersion according to Claim 16 wherein the pigment or dyestuff is organic and the dispersing agent is the salt of a free tertiary amine as defined in Claim 1 with a coloured acid.

5 19. A dispersion according to Claim 18 wherein the coloured acid is an acid of the pigment or dyestuff contained in the dispersion. 5

20. A dispersion according to Claim 18 or Claim 19 wherein the coloured acid is a copper phthalocyanine sulphonic acid.

21. A dispersion according to Claim 20 wherein the copper phthalocyanine sulphonic acid contains an average of from 1 to 2 sulphonic acid groups per molecule.

10 22. A dispersion according to any one of Claims 1 to 21 wherein the tertiary amine conforms to the formula: 10

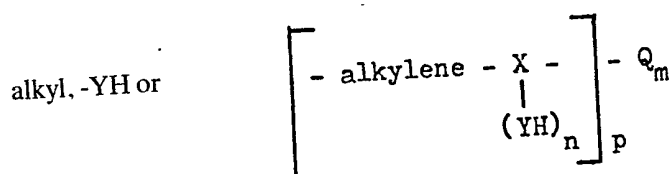


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wherein each of A, B and D independently represents

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wherein each X independently represents an oxygen or a nitrogen atom

30 provided that, where X is oxygen $m + n = 1$ and $p = 1$ and where X is nitrogen $m + n = 2$ and p is from 1 to 30; 30

each Q independently represents alkyl or -YH, and each Y independently represents a poly(lower alkylene oxy) chain containing from 3 to 50 lower alkylene oxy groups comprising at least 50% propylene oxy groups provided that.

35 (i) at least one of A, B and D contains a poly(lower alkylene oxy) chain, and 35

(ii) not more than one of the alkylene or alkyl groups contains more than 8 carbon atoms.

23. A dispersion according to Claim 22 wherein none of the alkyl or alkylene groups contains more than 8 carbon atoms.

40 24. A dispersion according to Claim 22 or Claim 23 wherein the poly(lower alkylene oxy) chain comprises a chain of propylene oxy units terminated at the end adjacent to the nitrogen atom by an ethylene oxy unit. 40

25. A dispersion according to any one of Claims 11 to 24 wherein the tertiary amine is selected from the group consisting of alkyl $\text{N}-(\text{CH}_2\text{CH}_2\text{O}-\text{YH})_2$, $(\text{alkyl})_2\text{-N-CH}_2\text{-CH}_2\text{OYH}$ and $\text{N}-(\text{CH}_2\text{CH}_2\text{OYH})_3$ wherein YH is a poly(propylene oxy) chain.

45 26. A dispersion according to any one of Claims 1 to 25 containing from 10% to 80% by weight of the solid based on the total weight of the dispersion and from 2% to 50% by weight of the dispersing agent based on the weight of the solid. 45

27. An ink or a mill base therefor containing a dispersion according to any one of Claims 1 to 26.

50 28. A pigment/resin chip comprising a dispersion of a pigment in a resin containing a dispersing agent as defined in any one of Claims 1 to 8, and Claims 22 to 25. 50

29. A process for the preparation of a dispersion according to any one of Claims 1 to 26 which comprises mixing the ingredients together and subjecting the mixture to a mechanical treatment to reduce the particle size of the solid.

55 30. A dispersion according to Claim 1 as described in any one of Examples 1 to 29. 55

31. A mill base according to Claim 27 as described in Example 30.

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Agent for the Applicants.

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