

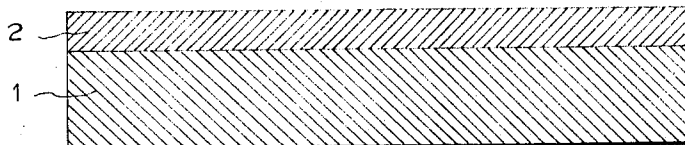
July 20, 1971

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3,594,209

INK TRANSFER MATERIAL AND METHOD FOR MANUFACTURE THEREOF

Filed May 27, 1968



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INK TRANSFER MATERIAL AND METHOD FOR MANUFACTURE THEREOF

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Filed May 27, 1968, Ser. No. 732,246

Claims priority, application Austria, May 26, 1967, A 4,906/67

Int. Cl. B41m 5/10

U.S. Cl. 117—36.1

13 Claims

ABSTRACT OF THE DISCLOSURE

Ink transfer material is produced by heating a mixture of at least a hard and a soft wax in a non-polar or weakly polar organic liquid along with a high molecular weight binding agent. The solution is agitated and cooled below 25° C. to separate out 40% to 70% of the wax as gel particles and after pigment material is added the dispersion/solution mixture is applied to a carrier having at least surface absorbency. The liquid is expelled to produce a carbon paper for single use with greater blackening.

The invention relates to a method for the manufacture of ink transfer material, in particular carbon paper, for use on one occasion only, by coating a carrier which is absorbent at least on the surface with a mixture which contains polymeric binding agent and pigment together with waxes in an organic liquid.

Carbon papers, including carbon papers for repeated use and carbon papers for single use, copying paper for handwritten and typewritten copies and similar ink transfer materials generally have a coating which is transferred under writing pressure; the dope which forms the coating consists of wax, oil and pigment. These materials with a dope layer containing oil, e.g. made of hot carbon; have certain major drawbacks which are well known. The dope layer smears in the fresh state and becomes dry and hard with the passage of time; the oil and other low molecular substances migrate into the backing paper and the intensity of writing obtained with the dope layer diminishes. The copies also are not resistant to smudging.

In the case of carbon paper for use on a single occasion only, i.e. the cheapest copy paper for single use, there are other disadvantages. Such paper is manufactured exclusively by the application of hot carbon, i.e. molten wax/oil compounds which contain carbon black or similar pigments. Papers of this type which are available commercially always have excessive ink application, which far exceeds the quantity required for typing. Last but not least, the dope layer which is applied must be abnormally thick because of an inevitable reduction in blackness due to alteration of the light absorption capacity of the secondary carbon black particles in the case of hot carbon dopes. Because of all these defects it is scarcely possible to obtain a significant improvement in the quality in particular of carbon papers for single use, beyond the quality available at present.

These disadvantages are characteristic of the hot carbon method since the latter presupposes the use of fusible dopes which still enable a cohesive layer to be produced on the coating or printing machines at the normal, high coating speeds.

The rheological requirements placed on the hot carbon dopes limit the quantities of pigment which can be processed. It is normally impossible to add more than 20% to the dope even in the case of carbon black with low colour intensity or in bead form. In the case of carbon blacks with a high colour intensity the quantity is even

lower if indeed it is possible to process carbon blacks of this type at all. Working from carbon blacks with high colour intensity and high oil binding it is practically impossible to produce hot carbon dopes which will flow, since because of the high degree of thickening processing is not possible. However, these types would be particularly advantageous because of the considerable colour depth.

There have therefore been many experiments designed to develop oil-free ink transfer layers for copying materials. In particular materials are known which have a layer of ink-binding agent on the carrier and a wax coating on this layer of ink. An intermediate layer may be arranged between the carrier and the ink layer. One of the disadvantages of these known oil-free materials results from their multi-layer structure, i.e. the complicated and expensive method of manufacture in several different working stages. Not only do the individual layers have to be applied and dried in a corresponding number of operations under special conditions, but the individual preparations also have to be matched to each other in such a way that they do not react unfavourably with each other or impair the layers produced from them.

The combination of contradictory characteristics, namely good adhesion of the dope layer to the copying material in order to give on the one hand a high degree of cleanliness when the material is touched, and on the other good adhesion of the particles of pigment which are transferred to the copy in order to achieve high resistance of the copies to smudging, always leads to difficulties relating to the materials and processes used.

Single layer copying materials have also previously been described. The layer generally consists of a continuous and practically homogeneous solid coating of a wax type material. Since large quantities of this material which serves as binding agent are also transferred with the particles of pigment, and the material itself is soft and adhesive, the copies are not resistant to smudging and the copying material itself tends to smear for the same reason. In this context, it is more or less immaterial whether the carbon compound is applied to the carrier with a liquid ink, from the melt with a suitable solid binding agent, as solution in a liquid organic solvent, or in the form of an emulsion or dispersion in suitable liquids.

An ink transfer material of the single layer type which has become known fairly recently has a coating which contains carbon black as the pigment in the normal manner in a non-adhesive wax, the wax being, however, mixed with talcum and held on the carrier with the latter by low molecular ethyl cellulose as the binding agent. This known material is produced by dispersing the pigment and talcum in a solution of wax and ethyl cellulose in toluene; the dispersion is coated on to the carrier and the solvent evaporates; at the same time the wax crystallizes out. The function of the talcum is to lead to the formation of a layer of fine, discrete particles and also to reduce the coefficient of friction of the surface of the layer so that the latter is more resistant to scratching or abrasion while on the other hand the transferability of the wax/pigment particles under writing pressure is not impaired.

Practical tests have shown that the requirements are not fully met by such product. The dope layer is not clean to the touch and the copies which are obtained are not intensive and resistant to smudging. The principal disadvantage of this known material lies in a difficulty relating to the actual process technology. This mixture of wax, talcum, carbon black, ethyl cellulose and toluene has an excessively small proportion of pigment, giving copies with intense colouring, and settles quickly after agitation so that a layer of sludge is formed with a clear to white cloudy layer of liquid on top. It is not possible to prepare a fine dispersion with stability at least for a limited period

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of time. It is therefore also impossible to produce the material described on normal coating machines and in exactly reproducible quality, since poor homogeneity always occurs even when the coating compound is carefully circulated.

The object of the present invention is to remedy these drawbacks and to solve the problem of developing a process which gives a copying material in a single coating operation, intended in particular for use on one single occasion, with a thin application of pigment which is resistant to ageing, diffusion resistant, dry and non-smearing and which provides intensive, sharp, smudge-proof and erasable copies and which also enables in the place of fillers, remarkably high quantities of pigments to be incorporated with intense colouring.

This object is achieved by way of the present invention by dissolving (1) a gel forming wax component, namely at least one wax with a penetration of 1 to 10, preferably 2 to 8, and at least one wax with a penetration of 10 to 60, preferably 20 to 25, or a mixture of the two with a mean penetration of about 10 to 20, and (2) up to 20% by weight on dry basis, preferably up to approximately 10% by weight, of a binding agent which, because of its high molecular weight, does not penetrate or penetrates only slightly into the paper backing and/or the pigment, the dissolution being in a non-polar or weakly polar organic liquid which gives solvation of the waxes to form a clear solution which is as concentrated as possible. In such solution about 10 to about 13 parts by weight of the gel wax component are employed per each 100 parts by volume of organic liquid. The solution formed is one in which approximately 40 to 70% by weight, and preferably 50 to 60% by weight, of the wax separates out at room temperature (about 20 to 25° C.) in the form of gel particles upon lowering the dissolving capacity of the liquid. The dispersion/solution thus formed is diluted with further liquid or wax solution while setting or maintaining this quantity ratio between separated and dissolved wax proportions and with maintenance or additional formation of the gel particles. The pigment is then dispersed in thoroughly and the composition obtained in this way is applied as a coating to the carrier in the known manner and the known manner and the liquid is expelled at a temperature below the softening temperature of the wax particles which separate out in solid form.

Preferably the procedure is such that a liquid with a positive and preferably large solubility coefficient is used as the liquid giving solvation and dissolving or separation is obtained respectively by heating or cooling, preferably rapid, of the liquid. It is preferable to use a liquid which can be easily removed from the layer, in particular aliphatic and aromatic hydrocarbons or chlorinated hydrocarbons, preferably light benzines, 1,1,1-trichlorethane, benzene, toluene or mixtures of these latter are acceptable. The amount of the liquid employed will vary with the particular materials employed and the amount used will be chosen based upon the characteristics necessary for the use of dye dispersion/solution in the coating of the carrier material. In general the amount will be about 100 parts by volume per each about 10 to about 13 parts by weight of the gel wax component.

Regarding classification of wax systems and in particular of gel forming waxes as used herein, reference is made to Fischer-Presting "Laboratoriumsbuch für die Untersuchung technischer Wachse Harz- und Ölmenge," 3rd edition, VEB Wilhelm Knapp Verlag, Halle (Saale), 1958, page 463.

The two types of gel forming waxes defined above are referred to below as harder and softer wax for greater simplicity.

The following are particularly suitable as harder waxes: natural or synthetic waxes, oxidized mineral waxes as well as esters and amides of the latter, solid hydrocarbons, mineral waxes or their mixtures and preferably Pennsylvanian mineral waxes of the branch chain type.

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Specific examples of suitable waxes are the commercial products listed below which have proven particularly suitable for practical realization of the invention:

(1) BRANCH CHAIN PENNSYLVANIAN MINERAL OIL WAXES

In particular the unblended products which are obtained exclusively from crude oil residue by selective separation with a narrow melting point range; these products have a very fine structure and a high degree of plasticity, adhesion and cohesion, in particular the grades having a melting point in accordance with ASTM B 127-49 between 84 and 86° C. or 88 and 90° C. and a penetration index in accordance with ASTM D 1321-57T at 77° F., 100 g., of maximum 8 to 7; these waxes are either white or light in colour.

(2) S WAX

Acid wax on a mineral wax base, dripping point 78 to 83° C., solidification point 73 to 77° C., acid number 135-155, saponification index 155-175, density at 20° C. 1.00-1.02 (all values determined by the DGS standard methods); colour light yellow.

(3) L WAX

Acid wax on mineral wax base, dripping point 80 to 85° C., solidification point 75 to 80° C., acid number 120-140, saponification index 140-160, density at 20° C. 1.00-1.02 (all values determined by the DGF standard methods); colour yellowish.

Candelilla wax can also be used.

Microwaxes in the narrower sense of the definition are suitable as the softer waxes. These include the ozokerites and soft cerines and similar petrolatum waxes which, unlike the paraffins, have a micro crystalline structure and a melting point of approximately 60-70° C., in particular about 65° C. Esteric waxes with an acid number of less than 60, preferably between 10 and 30, a saponification index between 80 and 150 and a melting point below 85° C. are also suitable. Japan wax and fine crystalline bees wax have also proved suitable.

The amount of the gel wax component will vary as will the proportions of the various waxes which make up such component. The dispersion/solution formed with the binding agent and liquid is one in which about 40% to 70% by weight of the wax separates out at room temperature and the amount of wax component must maintain this relationship. In general about 10 to 13 parts by weight of the gel wax is used per each 100 parts by volume of liquid solvent. It is preferred, although not essential, that the hard and soft waxes be employed in a ratio of about 7 to 6 parts by weight.

The waxes may also be used in mixtures of two or more representatives of the same group or of both groups. The products mentioned above are in any case in general themselves mixtures of various compounds even if these compounds are related. For example, mineral waxes are preferably extracted from mineral oil separations by more or less intensive refinery. These products are in general therefore refining cuts, so that it is also possible to use, instead of one harder and one softer wax, a medium type wax with a corresponding fraction width, provided that the latter corresponds to a mixture of the two waxes in the above mentioned quantity proportions and has the same properties.

The binding agents are used to bind the wax and ink particles together and to the layer carrier. The nature and quantity of these layer forming substances may be easily determined by the expert within the framework of the explanations given below.

Preferably a binding agent will be used which is at least predominantly soluble in the liquid in the temperature range between the dissolving and drying temperatures.

Polyvinyl ethers of straight or branched chain aliphatic alcohols with 1 to 20 carbon atoms are particularly suit-

able. In particular are those ethers of the fatty alcohol which are preferably used in conjunction with alkyd resins modified with styrene and its derivatives (e.g. methyl styrene, vinyl toluene, etc.), epoxy resins and/or with phenolic resins which are modified with natural resin acids.

This group of binding agents is preferred because, even in concentrations of a few percent by weight, the substances within this group clearly reduce the viscosity of the wax dispersion produced in accordance with the invention and in particular also reduce the coating dope, especially if the dispersion is obtained by cooling the wax solution. These binding agents are therefore referred to as viscosity regulators. Their effect is particularly surprising in that carbon paper dopes, as mentioned earlier, present special difficulties with regard to their viscosity and flow characteristics due to the carbon black or pigment particles which are incorporated therein.

In order to explain this phenomenon it is possible to make the following assumption, without limiting the invention to any particular theory.

It is well known that microcrystalline waxes have the ability of retaining solvents with swelling phenomena (retention effect). This phenomenon is all the more marked the lower the proportion of straight chain paraffins. Since the waxes mentioned above are generally characterised by a particularly high degree of branching, and often by a large proportion of paraffins in ring-form, this retention effect is particularly marked. The proportions which are only poorly soluble are precipitated out first from the hot solution when the latter cools and form relatively large swelling elements. During the further cooling of the solution, the proportions which are rather less poorly soluble are also gradually precipitated out, again in the form of swelling elements. In this way a viscous composition is obtained in grain or gel form which is non-homogeneous in the order of magnitude of these swelling elements. In addition to the precipitated and solvated waxes, this composition also contains waxes which are dissolved in the solvent. The composition is thus termed a dispersion/solution.

The retention effect of the waxes used in accordance with the invention does, however, have the disadvantage that a relatively large amount of solvent must be used in order to produce a composition which is suitable for coating. In particular, in the case of the addition of finely divided active carbon blacks as the pigment, the dispersion assumes, unless sufficient solvent is added, a viscosity which is so high that it cannot be mixed adequately or coated satisfactorily. The use of large quantities of solvents is unsatisfactory from the economic standpoint since the losses are high and/or expensive solvent recovery installations are required.

In the instant case the viscosity regulators mentioned above provide a remedy since they reduce the viscosity of the dispersion. It is possible that their influence on the viscosity—in practice preferably a reduction in viscosity—can be explained in terms of colloidal peptization, but electrical charge transfer processes with the pigments at the phase limits are also involved. In this context it is probably not necessary for these binding or layer forming substances to genuinely soluble in the solvent provided that they do have greater solubility than the waxes. In any event substances which only go into a cloudy solution in the solvent have a weaker effect as far as viscosity reduction is concerned, so that larger quantities either of such substance or of the solvent, must be used to obtain the same reduction in viscosity as will be achieved with a substance which is well soluble. In particular about 1–2% by weight of the total dispersion/solution of these latter well soluble materials will generally be sufficient but determination of the quantity which must be added bearing in mind the requisite reduction in viscosity and the binding effect which the substance exercises on the one hand in the dispersion/solution and on the other

hand in the final layer of ink, can be determined by the specialist through simple experimentation.

The following commercial products have proved particularly suitable as viscosity regulators:

(1) Medium viscosity polyvinyl methyl ethers with a K value of 40.

(2) Vinyl toluene modified alkyd resin, 60% in test benzene.

Characteristic data:	Percent
Phthalic acid anhydride -----	19
Linolic acid content -----	33
Viscosity at 20° C. (in benzene in accordance with DIN 4) -----	40

(3) Natural resin modified phenolic resin melting range 95–105°, soluble in aromatics, etc.

(4) Polymers on a vinyl acetate base in organic solution.

(5) Thermoplastic polyvinyl isobutyl ethers.

It is also possible to use mixtures of these viscosity regulators with themselves and/or with other layer forming substances which do not have this effect in the solvent which is used, e.g. ethyl cellulose in 1,1,1-trichloroethane.

If it is also necessary to match the binding agent and the liquid for dispersion or dissolving of the waxes and the binding agent in order to obtain optimal results in the sense described earlier, it is possible to obtain good to very good results for the above mentioned substance groups with the non-polar to weakly polar solvents previously referred to.

The viscosity reducing effect of the substances mentioned enables carbon blacks to be incorporated as pigment in the coating compositions having marked thixotropy due to the large surface area of such pigments.

By carefully choosing the type and quantity of the components of the layer, in particular the waxes and layer forming substances, the binding and adhesion of the ink layer on the carrier can be matched and the plastic separation of the ink layer under writing pressure regulated. It is assumed that the pigment particles are surrounded by the fine wax particles which have passed into the dry state through gel-forming swelling elements, but are not, or not completely, filled by the latter, and are also not filled by the latter, and are also not filled by the layer forming binding agents which on account of their high molecular weight and linear structure cannot penetrate the pigment particles but only bind the gel and pigment particles together and influence the hardness of the ink layer.

Paper is used primarily as the carrier, in particular a machine glazed material or a material which is glazed on one side such as 20 gram (g.) raw carbon paper. It is however also possible to use substances obtained from synthetic fibers and other materials which have a certain absorption characteristic at least on the surface, without however being too porous. It is also possible for the carrier material to be provided with a backing layer and/or intermediate layer or to be otherwise prepared.

In order to achieve a high ink depth it is particularly advantageous to precipitate out the waxes which have poor solubility as gel particles, which are at least partially larger than the pigment particles or their secondary particles or agglomerates.

From the point of view of production technology one process characteristic has proved particularly successful. This comprises blowing the layer with air at a temperature of between 5–25° C. prior to drying and preferably directly after coating the dispersion onto the carrier.

When the term pigments is used here, the expression must be understood in its general sense to refer to ink transfer material pigments; for instance it is possible to use instead of carbin black, a pigment which is applied to a carrier. The amount of the pigment employed is preferably about 7 to 15 parts by weight per 100 parts by volume of organic liquid. The amount of pigment is such that the

weight is not exceeded by the total weight of the soluble wax and binding agent.

The term wax also does not designate waxes in the narrow (chemical) sense but, as the substances named above show, substances of appropriate characteristics similar to those of waxes.

The invention leads to a result that e.g. in the case of inking carbon blacks, the layer consists of a high proportion of carbon blacks which could not be achieved in the case of hot carbon, these carbon blacks also having a significantly higher blackening effect while the ink layers have a hitherto unknown resistance of wiping off in spite of the high carbon black proportion. In addition, the ink layers have a dry, non-adhesive characteristic; they are particularly suitable for use with sets of forms and for processing on compiling machines. The layers produced in accordance with the invention do not suffer from the known alteration of the hot carbon inks after extended storage, and in particular they do not subsequently become hard. It is particularly important that the ink layers produced in accordance with the invention have improved efficiency but only between $\frac{1}{4}$ – $\frac{1}{3}$ the weight of conventional ink layers used in carbon paper for single utilization.

Without limiting the invention to a specific theory, the following remarks are intended to clarify outstanding characteristics of the materials produced by means of the method in accordance with the invention, and in particular the greater ink depth of the ink layers.

In spite of extensive experiments with carbon blacks with the highest possible colour intensity, it has previously been impossible in the case of hot carbon inks to improve the degree of blackening of copies beyond the known extent. Carbon blacks always undergo a reduction in the extent of blackening in hot carbon inks. The cause of this lies in the liquid oil or molten wax which fills the carbon black particle in the liquid state and therefore renders the hot carbon ink suitable for flowing.

The wax particles have approximately the same size as the pigment particles and in the case of carbon blacks the same size as that of the agglomerates of the secondary particles. The size of the first named wax particles is on the average of 10 to 20 microns (μ) and that of the pigment particles on the average of 20 to 30 μ .

During the layer formation drying is controlled in such a way that the gel particles remain in solid form and this they are unable to penetrate into the particles of carbon black or other pigment. The weight of the soluble wax and binding agent proportions in the composition should not exceed the weight of the carbon blacks. They will then essentially surround the carbon black particles without however filling the latter after the layer formation.

Whether or not the interior of the pigment particles is filled with oil or wax leads to a basic difference in the optical characteristics of the product. This results from the following consideration based in this example on a single secondary particle of carbon black.

A hot carbon particle, i.e. a carbon black particle which is filled with wax and oil, can be compared approximately with a drop in which the carbon black chain is incorporated in a knot form. A light beam which falls on a drop of this kind from outside, will emerge from the latter after single or repeated total reflection within the drop and there is a high degree of probability that it will not be directed further into the layer but will be deflected outwardly. During this process only a slight weakening of the beam generally occurs since the volume of the oil drop is much larger than that of the carbon black chain incorporated within it and the light absorption is therefore relatively low. The oil/wax medium which is denser than air increases refraction and reduces absorption of light. The particle of carbon black produced in accordance with this invention and which is to all intents and purposes unfilled, contains air so that when it is exposed to light there is no refraction and no total reflection. This means that, unless the light is absorbed on the carbon chain, it will pass

through the particle in practically linear form and disappear in the depth of the layer. It is finally absorbed in a particle of carbon black after refraction on the surrounding wax. The blackening of the layer therefore appears to be greater. The same applies to the copies which are produced.

This high colour intensity enables the weight of the ink layer to be reduced to a previously unknown extent while at the same time the opacity and blackening are increased. Carbon paper for single use produced in accordance with the method of the invention proves perfectly satisfactory with a colour layer of 2–3 g./m.², while a hot carbon type carbon paper for single use requires applications of 8–10 g./m.² to give copies which are at all satisfactory and even then such paper suffers from other disadvantages. This finding makes the method in accordance with the invention economically viable in spite of conventional views and even provides products which are cheaper than known one way carbon papers. The fact that the wax particles of the material produced in accordance with this invention do not penetrate into the pigments can be seen since the transfer capacity is immediately cancelled out when the layer is heated to melting point.

The invention is described in greater detail below on the basis of examples of practical embodiments. In these examples the term parts refers to parts by weight unless otherwise specified. Percentage is also by weight unless otherwise indicated. Parts by weight herein bears the same relationship to parts by volume as kilograms to liters.

In general the procedure employed is as follows: The waxes are dissolved into clear solution in the smallest possible quantity of hot liquid. This solution is cooled under strong agitation with an emulsifying machine or a dispersing unit. An initially oily dispersion is formed the particles of which become solid under further cooling and the entire solution becomes opaque and assumes a pasty consistency.

At the latest at this stage the viscosity regulator which is preferably used will be dissolved in the dispersion and the latter will assume the desired thin viscosity. If necessary the dispersion will be mixed at 20–25° C. under continued agitation with further proportions of solvents which do not impair gel formation at 20–25° C., so that an opaque liquid which flows easily and may have thixotropic characteristics is obtained. This dispersion contains gel particles in solid form which are solvated by solvents as well as genuinely dissolved wax proportions, and in addition dissolved waxes and binding agents which remain soluble at room temperature. The ink pigment is distributed into and mixed with this dispersion/solution while further dispersion takes place; the preparation which has good slip characteristics is now ready for coating.

After coating the paper carrier it is assumed that a type of filtration occurs which leads to a certain zone structure in the finished material. Solvent or wax solution will preferably penetrate into the surface of the paper. On the fibre, especially if cold air is immediately blown on to the layer, the dissolved wax will separate out as fine particles. The wax particles reduce on the one hand the porosity of the surface of the carrier and on the other hand prevent direct contact between the pigment and fibre. The pigment particles which are surrounded by gel particles are deposited on the zone formed in this way and consisting preferably of soft wax proportions they are deposited in the surface of the paper. These particles are bound together and to the carrier through the long chained binding agent which is slowly separated out as the solvent evaporates but is prevented from penetrating the paper because of its high molecular weight. As microscopic examinations have shown, during the writing process affecting the ink layer, the pigment and wax particles are apparently removed from the zone above the transitional zone mentioned earlier since the fibres at the

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surface points on the paper carrier at which writing takes place are still extensively coated with wax which does not contain any pigment.

The figure of drawing is a diagrammatic representation of a carbon paper produced as described. In the figure, 1 represents the carrier material and 2 represents the coating remaining after the liquid is expelled.

EXAMPLE 1

7 parts of the branch chained Pennsylvania petroleum wax described above, 6 parts microwax 60/65 with a softening range between 60–65° and 2 parts stearyl alcohol polyvinyl ether are dissolved to a clear solution by heating to 80–100° C. in 50 parts benzine with a boiling range between 100 and 125°. The clear solution is rapidly cooled to a temperature of 25° or less with intensive agitation. A white-opaque mass with almost pasty consistency is obtained which not only contains dissolved wax but also wax particles precipitated out of the solution in dispersed form. 35 parts of the same benzine are added to this dispersion. The mixture is treated with an effective dispersion unit. The fine opaque dispersion now has a flowing consistency; 7 parts of a subsequently compressed fission or flame black, i.e. an ink black with a mean particle size of 500–720 A. and a BET surface of 32 m.²/g. as well as 1 part of a finely dispersed silicic acid in the form of secondary particles, are now stirred into this dispersion and then dispersed at a temperature below 25° C. This preparation is applied by known coating methods to the coating surface of a 20 g. cellulose paper in a quantity such that a layer with a dry weight of approximately 2–3 g./m.² is obtained. Immediately after coating on to the carrier, the preparation which is still liquid is treated by blowing with a cold stream of air and is then dried in the air stream at room temperature. In order to remove the final residue of solvents the material is then exposed for a short time to a temperature of approximately 40–50° C. The product can be defined as carbon paper for single use. It is completely flat, clean to the touch and, in spite of the low ink application gives a deep black copy which detaches itself completely from the carrier, resists wiping and is dry and gives copies with sharp outlines.

EXAMPLE 2

7 parts of S wax and 6 parts of Japan wax are dissolved in the hot state in 45 parts benzine 100/125 and cooled rapidly to 20° under the action of a dispersing unit. An opaque paste is formed which is diluted with further 30 parts cold benzine 100/125 while further dispersion takes place. To this mixture which contains gel particles dispersed in a wax solution, is added 4 parts of the vinyl toluol modified alkyde resin mentioned above and a further 10 parts of benzine 100/125 after which the entire mixture is agitated well.

7 parts of ink black are further dispersed in the dispersion obtained in this way.

A	B	C
Harder wax..... 5 parts (26.7%)	7 parts (46.7%)	9 parts (60.0%).
Softer wax..... 8 parts (60.0%)	6 parts (40.0%)	4 parts (26.7%).
Viscosity regulator..... 2 parts (13.3%)	2 parts (13.3%)	2 parts (13.3%).
Very poor writing, only small amount of ink transferred, copies of low colour intensity.	Very good writing elastic layer, copies with precise outline.	Usable writing but not sharp, brittle layer, copies not perfectly clear.

The finished dispersion is coated in the known way on a cellulose paper in such a way that in the case of ink black the application is 2–3 g./m.². Immediately after coating the preparation, drying is carried out as described in Example 1. The results are comparable with those obtained in Example 1.

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EXAMPLE 3

Proceeding in the same way as in Example 2 but instead of S wax using an identical quantity of L wax, instead of Japan wax using bees wax and instead of 7 parts of ink black 35 parts of inactive black, i.e. a thermal fission black with mean particle size of 3,000–5,000 A. and a BET surface of 6–8 m.²/g., a product comparable to that obtained in Examples 1 and 2 is obtained.

EXAMPLE 4

A comparable product to that of the previous examples is obtained proceeding as in Example 1 but using the soft cerine instead of microwax 60/65 and, as a viscosity regulator, 4.0 parts polyvinyl methyl ether K 40 (20% in toluol).

EXAMPLE 5

Proceeding as in Example 1 but using Candelilla wax as a harder wax and 8 parts copper phthalocyanine blue as pigment a product comparable to those of the preceding examples is obtained.

EXAMPLE 6

The procedure of Example 1 is followed by using 1,1,1-trichloroethane as the solvent or diluting agent. A product comparable to that of Example 1 is obtained.

EXAMPLE 7

Proceeding as in Example 1 but using instead of carbon black 7 parts ultramarine blue a product comparable to that of Example 1 is produced.

EXAMPLE 8

Proceeding as in Example 1 but using 3 parts of a highly compressed ink black a product comparable to Example 1 is obtained. In this Example the dispersion is diluted with 70 instead of 35 parts benzine.

EXAMPLE 9

The procedure of Example 1 is followed but using instead of the 50 parts benzine originally used for dissolving, 50 parts of toluene, and instead of the 35 parts of benzine used to dilute the dispersion, 50 parts of toluene. A comparable product to that of Example 1 is obtained.

EXAMPLE 10

This example illustrates that by means of simple experiments it is possible to select suitable plasticities for the production of a material with an ink layer which can be completely transferred to the points of the paper which are exposed to writing pressure in single use.

Preparation 10 A contains significantly more than 50% dissolved proportions. The layer is adhesive and is not satisfactorily detached from the carrier. On the other hand preparation 10 C contains too little soft wax. The copy does not have adequate contrast and the transfer is not sharp and not precise. The layer also has the disadvantage of already showing a certain degree of brittleness. Preparation 10 B gives optimal results.

From this comparison of the preparations it can be seen that the hardness or elasticity of the ink layer can be varied as required to five satisfactory copies.

EXAMPLE 11

Proceeding as in Example 1 but using as the softer wax 6 parts bees wax and as the viscosity regulator 2 parts

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polyvinyl butyl ether a product comparable to that of Example 1 is obtained.

In all the above examples materials were obtained which meet the requirements described in the description of the invention in spite of the lower quantities of solvent which are used, preparations are obtained which can be coated easily upon the carrier.

EXAMPLE 12

The procedure of Example 1 is employed but using in place of the stearyl alcohol polyvinyl ether, 1.5 parts ethyl cellulose, and instead of 50 parts benzine there is used for dissolving, 100 parts 1,1,1-trichloroethane and instead of 35 parts benzine for diluting, 200 parts 1,1,1-trichloroethane. In this case no reduction in viscosity is observed so that the quantity of solvent must be significantly increased in order to permit the incorporation of the carbon black and to make the composition which is otherwise practically solid, dispersible and thus suitable for coating.

In the table below the soluble proportions and penetration values are shown for a series of waxes or wax mixtures. These values are applicable at 20° C. for a dispersion in benzene 100/125, which was produced by cooling a hot solution with 15% by weight wax. The penetration values are calculated for the soluble or insoluble proportions of this dispersion in the solid, solvent-free state after in each case 4 processing operations, i.e. the gel was separated by cooling, dissolved again, brought to separation by cooling and separated. This cycle was repeated 4 times.

Designation	Soluble proportion, percent	Penetration of—	
		Soluble proportion	Soluble proportion
Pennsylvanian wax.....	23.7	21	8
S wax.....	5.4	10	2
L wax.....	18.1	10	1
Fine crystalline micro wax...	17.6	12	3
Cerine.....	63.5	30	7
Micro wax 60/65.....	77.3	51	8
Ozokerite.....	79.4	38	4
Wax mixture of Example 1....	53.8	24	7

EXAMPLE 13

Similar to Examples 1 to 11, a yellow transfer dope is produced by dissolving, through heating in 70 parts by weight of 1,1,1-trichloroethane, 7 parts by weight of the above-mentioned branched-chain Pennsylvania petroleum wax, 3 parts by weight of microwax 60/65, 3 parts by weight of an externally strongly plastic, vaseline-like micro wax, and 2 parts by weight of polyvinyl ether of stearyl alcohol. This solution is cooled quickly to room temperature under vigorous agitation. A paste is formed. This is diluted with 100 parts by weight of 1,1,1-trichloroethane and 30 parts by weight of toluene. Into the diluted mixture, 13 parts by weight of a yellow pigment are dispersed with the aid of a highly effective dispersing aggregate.

Then, a black transfer dope is produced, as in Example 6, by dissolving by heating in 70 parts by weight of 1,1,1-trichloroethane, 7 parts by weight of the previously-mentioned vaseline-like microwax and 2 parts by weight of polyvinyl ether of stearyl alcohol, then quickly cooling the so-formed solution under vigorous agitation to room temperature, and diluting it with 100 parts by weight of 1,1,1-trichloroethane and 30 parts by weight of cold toluene, and finally mixing it with 10 parts by weight of color-intensive flame carbon black, under vigorous dispensing.

The yellow transfer dope is applied to cellulose paper suitable for copying purposes with a surface weight of 20 to 35 g./m.² in such a way that the ready, dried coating has a layer thickness of about 2 g./m.²; the material is dried in a warm air jet below 55° C.

The black transfer dope is applied to the reverse side

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of the thus coated paper in the same manner as described above.

Copy material coated with, for instance, a yellow dope layer on the front side and a black dope layer on the reverse side, is particularly well suited for the production of sets of forms. These are arranged in such a way that in order to achieve a white print, a copy, or for an increase in strength of the original's copyability, the reverse side of the sheet of paper records the characters in yellow color. The black coat applied to the reverse side of the transfer sheet produces the next copy.

The transfer material furnishes excellent copies of handwriting as well as type. The dope layers are wipe resistant and do not cause soiled hands when used. Particularly, the yellow color possesses high absorptivity of actinic light, especially for such light sources used in blueprinting equipment. The produced characters also show great wipe resistance on writing paper.

Instead of the yellow pigment used, the coloring layer may also be made with other pigment substances with high absorptivity of actinic light, for equally good results.

The following is a description of the experiments by means of which the dissolved wax proportions can be determined in a cold wax solvent dispersion.

The following mixture ratio is used as an example for carrying out the experiments to determine the dissolved wax proportions in a cold wax solvent dispersion:

Wax		Parts by weight
Harder wax.....	Pennsylvanian petroleum wax....	14
Softer wax.....	Micro wax.....	12
Viscosity regulator.....	Stearyl alcohol polyvinyl ether.....	4
Solvent.....	Benzine 100/125.....	170
Total.....		200

DESCRIPTION OF THE EXPERIMENTS

14 g. harder wax, 12 g. softer wax and 4 g. viscosity regulator are dissolved in the hot state in 100 g. solvent in a 500 ml. three-necked flask with agitation at the reflux. This hot solution is then cooled as quickly as possible with agitation in cold water to a temperature of less than 25° C. A dispersion is formed with rather pasty consistency. This is diluted with 70 g. benzine 100/125 at room temperature so that a 15% was dispersion is obtained in benzine 100/125. The dispersion is centrifuged. After centrifuging for 30 minutes the liquid phase is determined as 168 ml. of the specific weight of 0.73 g./ml. After evaporating the liquid phase there is left a wax residue of 11.8 g. The 11.8 g. corresponds to 39.3% of the wax mixture which was used.

The quantity of the solid centrifuged residue amounts to 16.9 g. after drying at 110–120° C. in the drying cabinet. The dry, solid centrifuge residue is used again to produce a 15% wax benzine dispersion under the test conditions described above, although fresh benzine 100/125 is used.

After centrifuging and evaporating the solvent a wax residue of 2.9 g. in the liquid phase remains. This 2.9 g. corresponds to 9.6% of the initial wax mixture.

12.7 g. remains as the solid, dry centrifuge residue and this quality is used again to produce a 15% dispersion.

The experiment described above is repeated a third and fourth time and a soluble wax residue of 0.8 g.=2.7% and 0.6 g.=2.0% of the initial quantity of the wax mixture is obtained.

By adding the percentage value of the dissolved wax proportions in a cold wax benzine dispersion, the percentage proportion of the wax which is present in the genuinely dissolved state in a cold wax benzine dispersion is obtained.

In the above-mentioned example the value is found as 53.6%.

	Percent
(I) -----	39.3
(II) -----	9.6
(III) -----	2.7
(IV) -----	2.0
	53.6

What is claimed is:

1. A method for the manufacture of ink transfer material which comprises:

(A) dissolving by heating in a non-polar or weakly polar organic liquid

(1) about 10 to 13 parts by weight per 100 parts of said organic liquid of a gel forming wax consisting of at least one hard wax having a penetration of from 1 to 10 and at least one soft wax having a penetration of from 10 to 60 or a mixture of said waxes having a mean penetration of about 10 to 20, and

(2) up to about 20% by weight on dry basis of a high molecular weight binding agent which will not substantially penetrate the surface of an ink transfer material carried on the surface of an ink transfer material pigment to achieve solvation of the waxes and yield a substantially clear solution,

(B) bringing the temperature of the solution to below about 25° C. while agitating the same to reduce the dissolving capacity of the liquid and to cause the separation of from about 40% to about 70% by weight of the wax present in the form of gel particles,

(C) dispersing an ink transfer material pigment into the solution containing gel particles,

(D) coating the thus obtained solution onto a transfer material carrier having at least surface absorbancy, and

(E) expelling the liquid from the coating at a temperature below the softening temperature of the wax particles contained in the coating.

2. A method as in claim 1 wherein the organic liquid has a positive, high solubility coefficient for the gel wax component.

3. A method as in claim 2 wherein the organic liquid is a member of the group consisting of aliphatic hydrocarbon, aromatic hydrocarbons, chlorinated aliphatic hydrocarbons, and mixtures thereof.

4. A method as in claim 3 wherein the organic liquid is a member of the group consisting of benzene, 1,1,1-trichloroethane, benzene, toluol and mixtures thereof.

5. A method as in claim 1 wherein the amount of pig-

ment present by weight is not substantially exceeded by the total amount by weight of the soluble wax and binding agent in the dispersion/solution.

6. A method as in claim 1 wherein the hard wax is selected from the group consisting of true natural and synthetic waxes, oxidized mineral waxes and esters and amides thereof, solid hydrocarbons, mineral waxes and mixtures thereof and the soft wax is a member selected from the group of Japan wax, micro waxes, bees wax, and soft cerines.

7. A method as in claim 6 wherein the hard wax is branch chain Pennsylvanian mineral wax and the soft wax is micro wax 60/65.

8. A method as in claim 1 wherein the binding agent is at least predominantly soluble in the organic liquid in the range of temperature between the dissolving temperature of the solution and the drying temperature of the coating.

9. A method as in claim 1 wherein the binding agent is a polyvinyl ether of straight or branched chain aliphatic alcohol of 1 to 20 carbon atoms, alkyd resin modified with styrene or a derivative thereof, an epoxy resin, phenolic resins modified with natural resin acids, or mixtures thereof.

10. A method as in claim 1 wherein the coating on the carrier is treated, prior to complete expelling of liquid, with air at a temperature of from about 5° to about 25° C.

11. The process of claim 1 wherein the ratio of hard to soft wax is about 7 parts by weight hard wax to 6 parts by weight soft wax.

12. The process of claim 1 wherein the pigment is present at about 7 to 15 parts by weight per 100 parts by volume organic liquid.

13. The product of the process of claim 1.

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U.S. Cl. X.R.

106—31, 272; 117—155, 158; 208—21