Monsanto INDUSTRIAL CHEMICALS CO.

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SUBJECT DYE SOLVENTS: DO2-21-3985

REFERENCE WRE/JKS, 10/29/71

TO : Wayne R. Eberhardt
WEBER

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[redacted]. R. Darby, Res. 1
N. W. Touchette, Res. 3
W. R. Richard, WRICH

Wayne, I am enclosing five tables of new data to establish scope for our patent application on alcohols as synergists for color formation from CVL solutions.

Tables 1-4 show (usually) increased speed of color formation on the three resin CF papers from NCR. For this I used the rubber stamp test as for my first data to you in October. This time, however, I must have demanded a darker blue since I have indicated longer times for most of the solvents for which I have direct comparison. I hope this is not too confusing to an examiner. Actually, I could use a little math to reduce these numbers to an average value for the control if that would help.

Table 5 gives a different kind of data. I detect no change in speed of blue color formation on clay CF papers but I do see (usually) a slightly more intense blue color when small amounts of alcohols are used in the rubber stamp test. In Table 5, Method A, I made a disposable ink pad as usual from one layer of facial tissue on a microscope slide and coated it liberally with the CVL solution to be tested. I inked the rubber stamp well, then repeatedly stamped the clay CF receiving sheet. A + sign in the rating column indicates the solution containing the alcohol gave darker lines than the control solution without alcohol. The number (e.g., +6) indicates at least 6 lines stamped from one inking were darker than the corresponding control lines. A minus sign indicates the test solution gave a lighter color than the control. (The differences in all cases were slight but noticeable.) Method B was identical to Method A except only two drops of solution were used and the ink pads were allowed to stand overnight before using them.

SCOPE:

Aliphatic alcohols, linear and branched, work (with one or more of the phenolic resin CF papers) from at least ethanol (C_2) to HOE (C_{14}-C_{22}). The lower ones appear to be too volatile to last long. We might claim all the way down to one or two carbons as the broad claim. It looks like the C_{22} alcohol might be the top of the workable range, but to be safe how about C_{26} to get

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any "dimer" from tridecyl manufacture. The broad claim might run from methanol to C_{26} alcohols. The preferred range should cover from C_6 to C_{18} alcohols, I believe.

Cycloaliphatic alcohols should be claimed, e.g., cyclohexanol and its derivatives such as 2-methyl-cyclohexanol, 4-t-butyl-cyclohexanol, etc. I would suggest up to hydrogenated nonyl-phenol, $C_{15}OH$.

Benzyl Alcohols should be claimed from benzyl alcohol itself to at least α-methyl-α-propylbenzyl alcohol (2-phenyl-pentanol-2). 2-Methylbenzyl alcohol, 4-methylbenzyl alcohol, etc., could be included.

Aliphatic Diols with a ratio of C/OH of 5 or higher should be covered up to a total carbon content of, say, C_{26} if you think our one example will stretch that high.

Di- and Polyethylene and Propylene Glycols should be covered up to polyethylene glycol 400 (9-10 ethylene units) and its polypropylene glycol counterpart, again if one example will stretch that far.

Dicycloaliphatic Alcohols should be included, at least for enhancement of color on clay papers. Besides hydrogenated Bisphenol A or 2,2-di(4-hydroxycyclohexyl) propane we could include other hydrogenated bisphenols from the bisphenols listed in U.S. 3,186,961, but I would suggest abbreviating the list.

Concentration of Alcohol should be from about 0.25% to about 50% with the preferred range from 0.5% to 15%, but let's talk it over if you wish.

Primary solvents can be any of those known in the art, certainly including by name the ones used in this study.

If I have tried to claim the Universe or if I have left out the obvious, let me know.

Kern Sears

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Attachments

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Table 1.

Effect of Various Alcohols on Rate of Color Formation of 3% CVL Solutions On Three Resin CF Papers[a]

Primary Solvent: 2,4-Diphenyl-2-methyl pentene[b]

Alcohol Synergist 2%	Time (Seconds) To Good Blue Color[c] Paper A		Paper B		Paper B	
	Without Alcohol	With Alcohol	Without Alcohol	With Alcohol	without Alcohol	With Alcohol
Ethanol	15	15	15	15	10	7
Iso Amyl Alcohol	—	—	—	—	10	2
Ditol After Standing					7	7
Alfol 6[d]	14	10	10	8	10	3
Alfol 12[e]	15	8	15	6	10	3
711 Alcohol	15	8	15	8	8	2
HOE Alcohol	10	10	10+	10	10	5
Cyclohexanol	10	10	7	4	7	3
α,α-Dimethyl benzyl Alcohol	10+	10	8	7	7	4
1,10-Decanediol (Trace undissolved)	10	10	10	10	7	6
Diethylene glycol (Trace undissolved)	10+	10	10+	10	7	3
Hydrogenated Bisphenol A (Trace undissolved)	15	20	10	15	7	10

d. Alfol 6 (Continental Oil Co.) C_6 OH 98%, C_4 OH < 2%.
e. Alfol 12 (" " ") C_6 OH < 1%, C_8 OH < 1%,
C_{10} OH 2%, C_{12} OH 95.6%, C_{14} OH 1%

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a. Papers A, B and C are NCR's XP-770, XP-1875 and XP-1531.
b. Alpha methyl Styrene Dimer (liquid).
c. Time values should be taken in pairs for best understanding, since judgement of when a dark enough blue is reached is subjective

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Table 2

Effect of Various Alcohols on Rate of Color Formation of 3% CVL Solutions on 3 Resin CF Papers

Primary Solvent: HB-30

Alcohol Synergist 2%	Time (Seconds) To Good Blue Color Paper A		Paper B		Paper C	
	Without Alcohol	With Alcohol	Without Alcohol	With Alcohol	Without Alcohol	With Alcohol
711 Alcohols	ca 90	15-30	ca 60	20	35-40	7
α,α-Dimethylbenzyl Alcohol	70	70	60	35	40	20
Hydrogenated Bisphenol A	90	>90	60	90	45	50

Table 3

Effect of Various Alcohols on Rate of Color Formation of 3% CVL Solutions on 3 Resin CF Papers

Primary Solvent: Monobenzyl Toluene

Alcohol Synergist 2%	Time (Seconds) To Good Blue Color Paper A		Paper B		Paper C	
	Without Alcohol	With Alcohol	Without Alcohol	With Alcohol	Without Alcohol	With Alcohol
711 Alcohol	1+	1	1	<1	1	<1
HOE Alcohol	1+	1	1	<1	1	<1

Table 4

Effect of Various Alcohols etc

Primary Solvent: Therminol 77

Alcohol Synergist 2%	Time (Seconds) To Good Blue Color Paper A		Paper B		Paper C	
	WO	With	WO	With	WO	With
711 Alcohol	>180	120	>150	90	>120	20
Diethylene glycol	—	—	180	160	>120	13

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Table 5

Effect of Various Alcohols on Intensity of Lines, Stamped by Rubber Stamp from one Inking. Repeatability

Solutions: 3% CVL. Paper: Nashua's Clay CF Paper

Control Solution Solvent No		Test Solution Solvent No		Rating of Test Solution Method A	Method B
C11	HB-30	C12	HB-30 + 711 alcohol	+6	+2
"	"	"	"	+6	
C11	"	C13	HB-30 + α,α-dimethyl benzyl alcohol	+4	+1
				+3	
C11	"	C14	HB-30 + Hydrogenated Bisphenol A	+6	+5
				+5	
C18	Thermuloid 77	C19	Thermuloid 77 + 711 Alcohol	+10	+5
C18	"	C20	Thermuloid 77 + Diethylene glycol	+5	+5
C15	Monobenzyl Toluene	C17	Monobenzyl toluene + HB E Alcohol	-4	
				-3	

For the following solvent combinations one stamping only was made each time with the solution containing Alcohol giving either darker (+) or lighter (-) or the same (0) intensity of blue color as the control

599	2,4-Diphenyl-2-methyl pentene (AMSD-L)	C00	AMSD-L + Ethanol	+
599	"	C02	AMSD-L + Alfol 6	+
599	"	C04	AMSD-L + 711 Alcohol	0 to +
599	"	C05	AMSD-L + HB E Alcohol	—

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