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UNITED STATES DISTRICT COURT
FOR THE DISTRICT OF NEW JERSEY

JOAN MAERTIN, Executrix of)
the Estate of Lothar Maerten,))
JOAN MAERTIN, individually)
and in her own right, et al.,)

v.) Civil Action L-95-cv
02849 (JBS)

ARMSTRONG WORLD INDUSTRIES,)
INC.,)

v.)

MONSANTO COMPANY and)
AMERICAN MINERAL SPIRITS)
COMPANY)

DEPOSITION OF WILLIAM PAPAGEORGE

ON BEHALF OF THE DEFENDANT/THIRD-PARTY
PLAINTIFF ARMSTRONG WORLD INDUSTRIES
APRIL 29, 1998

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1 INDEX 2 QUESTIONS BY: PAGE NO. 3 Mr. Taylor 4 4 5 INDEX OF EXHIBITS 6 PAPAGEORGE NO. DESCRIPTION PAGE MKD. 7 22 3/17/70 meeting notes- Bates 059941-45 11 8 23 4/3/70 memo- Bates 059946-47 14 9 24 Plasticizers info Bates 059971-73 39 10 25 8/28/69 "the Specialist" Bates 054389-90 43 11 26 Literature Survey Bates 059918-36 44 12 27 "Chemical Week" Bates 059894 44 13 28 Anniston Plant Newsletter Bates 059895-02 45 14 29 Products 1961 Bates 059907-08 50 15 16 (Exhibits attached to the original transcript only.) 17 18 19 20 21 22 23 24 25	1 APPEARANCES 2 3 For the Plaintiffs: 4 Mr. Gary Ginsburg, Esq. 5 Law Offices of Gary Ginsburg 6 Atrium II, Suite 101 7 3000 Atrium Way 8 Mt. Laurel, New Jersey 08054 9 10 For the Defendant/Third-Party Plaintiff Armstrong 11 World Industries: 12 Mr. Matthew A. Taylor, Esq. 13 Duane, Morris & Heckscher, LLP 14 One Liberty Place 15 Philadelphia, Pennsylvania 19103 16 17 For the Defendant Monsanto Company: 18 Mr. Christopher M. DiMuro, Esq. 19 Latham & Watkins 20 One Newark Center 21 Newark, New Jersey 07101 22 23 For the Defendant American Mineral Spirits Company: 24 Ms. Susan Karlovich, Esq. 25 Wilson, Elser, Moskowitz, Edelman & Dicker Two Gateway Center Newark, New Jersey 07102
1 UNITED STATES DISTRICT COURT 2 FOR THE DISTRICT OF NEW JERSEY 3 4 JOAN MAERTIN, Executrix of 5 the Estate of Lothar Maertin, 6 JOAN MAERTIN, individually 7 and in her own right, et al., 8 v. 9 ARMSTRONG WORLD INDUSTRIES, 10 INC., 11 v. 12 MONSANTO COMPANY and AMERICAN 13 MINERAL SPIRITS COMPANY 14 15 DEPOSITION OF WILLIAM PAPAGEORGE, produced, sworn and 16 examined on the 29th day of April, 1998, between the hours 17 of eight o'clock in the forenoon and six o'clock in the 18 afternoon of that day, at the offices of Taylor * 19 Schroeder Reporting & Video, 7494 Ethel Avenue, St. Louis, 20 Missouri before TAMMIE A. ST. ARBOR, Certified Shorthand 21 Reporter and Notary Public within and for the States of 22 Missouri and Illinois, in a certain cause now pending in 23 the United States District Court for the District of New 24 Jersey wherein JOAN MAERTIN, Executrix of the Estate of 25 Lothar Maertin, JOAN MAERTIN, individually and in her own right, et al. are Plaintiffs and Armstrong World Industries, Inc. is Defendant/Third-Party Plaintiff and Monsanto Company and American Mineral Spirits Company are Defendants.	1 IT IS HEREBY STIPULATED AND AGREED by and between 2 counsel for the Plaintiffs and counsel for the Defendants 3 that this deposition may be taken in shorthand by Tammie 4 A.St. Arbor, CSR and notary public, and afterwards 5 transcribed into printing, and signature by the witness 6 expressly reserved. 7 MR. DIMURO: Just before we start today, I want 8 to make a statement for the record. As everybody probably 9 knows by now, Mr. Papageorge is being produced again by 10 Monsanto in response to Magistrate Rosen's order that he 11 be produced to answer orders on DOP and Santicizers. 12 Mr. Taylor and I have reached an agreement that I would 13 also let him ask Mr. Papageorge questions about documents 14 which were subsequently produced by Monsanto after 15 Mr. Papageorge's first deposition and that the inquiry be 16 limited to those documents along with the DOP and 17 Santicizer questions. 18 * * * * * 19 WILLIAM PAPAGEORGE, 20 of lawful age, produced, sworn, and examined on behalf of 21 Defendant/Third-Party Plaintiff, Armstrong World 22 Industries, deposes and says: 23 EXAMINATION 24 QUESTIONS BY MR. TAYLOR: 25 Q. (By Mr. Taylor) Mr. Papageorge, my name is Matt

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1 Taylor, and I did not take your deposition the first time
2 in November of 1997, but I'm here today on behalf of
3 Armstrong World Industries. You may know it as Armstrong
4 Cork. During that deposition, you were asked some
5 questions regarding your time at the Queeny Plant. Do you
6 remember that?

7 A. I do.

8 Q. And as part of those questions, you were asked
9 about your involvement with DOP and the Santicizer
10 projects. Do you remember that?

11 A. I do.

12 Q. Santicizer products rather. In addition, you
13 were inquired generally regarding Plasticizers and your
14 name in Anniston about questions that came up from time to
15 time by employees regarding possible health effects that
16 may be caused them because of PCBs. Do you remember that?

17 A. I remember some of it. I don't remember all the
18 details.

19 Q. Well, were there questions raised by employees
20 at Queeny regarding possible ill effects that may be
21 caused them because of exposure to the DOP or Santicizer
22 products?

23 A. The questions that were raised were typical of
24 all of the industrial chemicals that the employees are
25 exposed to. They would be raised on an individual basis

1 A. I don't recall the specific wording, but the
2 gist of the instructions was one of continual respect for
3 all materials.

4 Q. Were those safe handling instructions in
5 connection with DOP and Santicizer similar to those
6 general instructions for PCB type products?

7 MR. DIMURO: Object to the form. You can
8 answer.

9 A. No. The PCBs were considered because of their
10 chlorine content to be, what's the proper word I'm looking
11 for, not as innocuous as the phthalic esters.

12 Q. (By Mr. Taylor) Would the word be toxicity?

13 A. That is, also lends itself to relative in terms
14 of how much it takes to cause any harm, detectable harm.
15 But the phthalic esters are less apt to cause problems even
16 if they are not respected. In other words, if you get it
17 on your hands, it's not apt to cause the chapped hands
18 effect that the PCBs showed.

19 Q. Do you remember any issues regarding toxicity in
20 connection with DOP and Santicizer?

21 MR. DIMURO: At that time period?

22 MR. TAYLOR: At any time.

23 A. No.

24 Q. (By Mr. Taylor) Were they considered to be
25 toxic?

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1 or at a safety meeting.

2 Q. And that was true of DOP and Santicizer as well
3 as PCBs?

4 A. And a thousand other chemicals, yes.

5 Q. I understand. Now, some time was spent at your,
6 the earlier deposition asking about the instructions on
7 safe handling of certain products. Do you remember that?

8 A. I do.

9 Q. And I'm going to focus in on instructions given
10 to employees of Monsanto or to anyone, customers that
11 would buy DOP or Santicizer regarding safe handling of
12 those products. Can you tell me what those instructions
13 were?

14 MR. DIMURO: Matt, just for clarification, are
15 you talking about the point in time he was at the Queeny
16 Plant when he worked with these products? I think in his
17 last deposition, that's where he said he had involvement,
18 in that window of time when he was at the Queeny Plant.

19 Q. (By Mr. Taylor) I'll ask you both, your
20 knowledge while you were at the Queeny Plant regarding
21 instructions on safe handling of DOP and Santicizer.
22 First, why don't we limit the question to your time
23 there.

24 A. You recognize this is four decades ago.

25 Q. I understand.

1 MR. DIMURO: I'll object to the form.

2 A. All chemicals are toxic, sir. It's a matter of
3 method of exposure and time of exposure and amount of
4 material the person is exposed to.

5 Q. (By Mr. Taylor) I understand. And I want to
6 know your general knowledge about toxicity of DOP and
7 Santicizer and in connection with instructing those
8 dealing with those products, how to handle them safely.

9 A. Well, handling the material at room temperature
10 is quite similar to the handling of mineral oil. But if
11 you take that same material and put it in a tank and heat
12 it to the boiling point, you're going to create a fume
13 problem. Now, that fume, not only because of its chemical
14 nature, but because of the temperatures involved, can
15 create an irritation to the breathing of a person.

16 Q. So the vaporization would be an issue for DOP
17 and Santicizer as well?

18 MR. DIMURO: I object to the form.

19 A. Not vapor at room temperature.

20 Q. (By Mr. Taylor) I understand.

21 A. But it's a function of temperature. The higher
22 the temperature, the more aggravating it can be. And it
23 varies from person to person for the same amount. People
24 react differently so this is why we always went back to
25 respect that chemical, if you smell it, stay away from

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1 it. If you have to go into the area, put on a
 2 respirator. Not that it's going to be like a cyanide,
 3 you're not going to fall over and die, but no sense in
 4 being exposed.
 5 Q. Was there a difference in the concerns generally
 6 between the heating of Santicizer products and Aroclor
 7 products containing PCBs?
 8 A. Yes.
 9 Q. And what were those differences and concerns?
 10 A. Well, the difference is that it was known that
 11 the PCBs, when exposure was excessive, would result in
 12 health symptoms. Some of these symptoms were looked upon
 13 as early warnings of unnecessary exposure. For example,
 14 breathing hot fumes excessively could lead to a situation
 15 where the worker complains of a symptom that reminds us
 16 all of a severe chest cold.
 17 Q. That's in connection with PCBs?
 18 A. PCBs is what I'm talking about. And these are
 19 what we refer to as early warnings. If that person didn't
 20 pay attention when his eyes were watering and his throat
 21 was sore, now he's got this cough. He is not protecting
 22 himself with the proper respirator.
 23 Another early warning was the reddening of the
 24 skin which indicated an exposure through the skin. And
 25 that's an early warning. Continued disregard for skin

1 A. True.
 2 Q. I'm talking about known situations that came to
 3 your knowledge whether it was through the medical
 4 department or through an individual on an individual basis
 5 where someone had ill effects because of exposure to
 6 Santicizer and DOPs and what that effect was.
 7 A. I have never experienced that, no.
 8 MR. TAYLOR: I would like to mark this as
 9 Papageorge No. 22 with today's date.
 10 (Papageorge No. 22 was marked for identification
 11 by the reporter.)
 12 Q. (By Mr. Taylor) Sir, some of these documents
 13 were produced after your initial deposition so I want to
 14 just refer to some additional documents which we're going
 15 to mark with today's date but continuing the numbering
 16 sequence utilized in the first part of your deposition.
 17 And I would just ask if you would review that for a
 18 minute. And just tell me when you're finished.
 19 A. I have reviewed the exhibit.
 20 Q. And what type of meeting is this referring to,
 21 the March 17, 1970 meeting? Was that a special committee
 22 or --
 23 A. This was a meeting called by the --
 24 MS. KARLOVICH: What document are you looking
 25 at? What are the Bates for it?

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1 exposure could lead to an ailment referred to as
 2 chloracne. And continued exposure beyond that point could
 3 lead to a liver damage and --
 4 Q. All right. I'm sorry, did you finish? I didn't
 5 mean to cut you off.
 6 A. I think I am. I just use those as examples,
 7 opportunities to correct your behavior before it gets too
 8 serious.
 9 Q. Let's take any of those symptoms that you just
 10 gave me. Let's start with the burning of the throat for
 11 example.
 12 A. Yes.
 13 Q. Were they, was that a concern for prolonged
 14 exposure to DOP or Santicizer?
 15 A. I don't recall any such reaction to the
 16 Santicizer or DOP.
 17 Q. What -- strike that.
 18 Were there any ill effects that you remember
 19 relating to DOP or Santicizer in situations where there
 20 was prolonged exposure?
 21 A. I don't recall any, sir, because we first of
 22 all, didn't tolerate prolonged exposure if there were hot
 23 vapors, for example, in the work area.
 24 Q. And that would be true whether it was Santicizer
 25 or Aroclors?

1 MR. TAYLOR: I'm sorry. It's titled Brief
 2 Summary of PCBs Meeting March 17th, 1970 bearing Bates
 3 MAE 059941-45.
 4 MS. KARLOVICH: Okay. Thank you very much.
 5 Q. (By Mr. Taylor) You can continue.
 6 A. These notes reflect the discussion held in
 7 Duluth, Minnesota at the Federal Water Laboratory on the
 8 day of March 17, 1970.
 9 Q. What was the purpose of the meeting?
 10 A. This was, it was called by the director of the
 11 laboratory, Dr. Mount, M-O-U-N-T, to offer an opportunity
 12 for those individuals who had shown an interest in PCBs in
 13 the environment to review their understandings and
 14 findings as of that date.
 15 Q. And I, and, sir, if you turn to Page 3 of the
 16 document, there's a heading with your name, J, under
 17 Section J. Do you see that?
 18 A. I do.
 19 Q. And was that your part of the program to talk
 20 about uses of Aroclors?
 21 A. Yes, sir.
 22 Q. And at that point, you had gone through an
 23 extensive historical review of the uses of Aroclors on a
 24 historical basis; is that correct?
 25 MR. DIMURO: I'll object to the form.

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1 A. I don't know what you mean by extensive
2 historical.
3 Q. (By Mr. Taylor) As I understood your earlier
4 testimony, you had gone through some tutorials to get an
5 understanding as to where the Aroclors may have gone after
6 leaving Monsanto.
7 A. Well, the tutorial was intended not only to
8 cover that narrow area of information, but it was to
9 introduce me to disciplines that related to the
10 environmental PCBs issue in areas that I had personally no
11 strong background. For example, the medical field, the
12 marketing field, and the like. And that was the purpose
13 of those early 1970 tutorials.
14 Q. And my question is: Say for example the
15 marketing field where these products had been marketed to,
16 you would know where they may be in the environment?
17 A. Yes. Now of course having been involved with
18 the manufacture and shipment of PCBs, I had a pretty good
19 understanding in a broad sense of the types of
20 applications. The tutorials were more specific in terms
21 of the types of PCBs and perhaps the general quantities
22 that went to certain customers, who the key customers
23 were. It's that kind of detail that I was brought up to
24 date on the tutorial.
25 Q. And your understanding of the biggest uses, and

1 see that name?
2 A. Yes, I do.
3 Q. Do you remember Mr. Wright?
4 A. Yes, I do.
5 Q. And it was, it's been testified by others that
6 Mr. Wright had some responsibility in relation to the
7 distributorship agreement between Monsanto and AMSCO.
8 Does that, are you aware of that?
9 A. Not that specific, no.
10 Q. Okay. Are you aware of that written agreement
11 between Monsanto and AMSCO?
12 MR. DIMURO: Objection to the form.
13 Q. (By Mr. Taylor) And when I say written
14 agreement, I mean memorializing the distribution or
15 distributorship relationship between the parties.
16 A. I was made aware of such an agreement. I have
17 never seen the document itself.
18 Q. Who made you aware of that agreement and when?
19 A. It was early 1970 and as best as I recall, it
20 was the director of marketing for the Plasticizers group,
21 Walter Schalk.
22 Q. And how did that topic come up?
23 A. He was the individual responsible for the
24 marketing of PCBs. And, of course, this involved the sale
25 of PCBs to distributors. And I don't know the specifics

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1 I'm just looking at this document, you refer to
2 Plasticizers in CI rubbers. Is that chlorinated rubbers?
3 A. Yes.
4 Q. And caulking materials. Do you see that?
5 A. Yes.
6 Q. I noticed for example that surface coatings are
7 not mentioned here. Is there any reason why?
8 A. Well, there was no exact number that served as a
9 guideline as to what is a big use or a little use. What I
10 mention here were the top five uses. I, at this point in
11 time, don't recall where coatings fell on the applications
12 list.
13 Q. Okay. Next document which we will mark as
14 Papageorge 23 is an April 3, 1970 memorandum from Cumming
15 Paton to various people. It bears Bates MAE 059946-47.
16 (Papageorge No. 23 was marked for identification
17 by the reporter.)
18 Q. (By Mr. Taylor) Mr. Papageorge, if you could
19 take a minute to review that memorandum and let me know
20 when you're finished.
21 A. I have finished reading the exhibit.
22 Q. Do you remember this memorandum?
23 A. Yes, I do.
24 Q. Sir, before we get into the body of the
25 memorandum, there's one gentleman, J.D. Wright. Do you

1 or the exact words, but he is the individual that I dealt
2 with when it came time to who were our customers and how
3 do we inform them.
4 Q. And what was your understanding of that
5 distribution agreement? How did it operate?
6 A. Well, I understood that there were several
7 entities involved in the distribution, that they would
8 warehouse certain types of PCBs in their facility, and
9 they would take care of an end user need in a geographic
10 area that was assigned each of these distributorships. I
11 don't know any of the other specifics.
12 Q. All right. Were you aware of, that Monsanto
13 trained anyone at AMSCO whether it was their, the AMSCO
14 salesmen or anyone else within AMSCO?
15 A. I don't know that.
16 Q. Were you aware that Monsanto or any of its
17 employees were asked to speak at conventions or meetings
18 of the AMSCO people?
19 MR. DIMURO: Object to the form. You can
20 answer.
21 A. I do not know that.
22 Q. (By Mr. Taylor) What role were you aware of
23 that J.D. Wright played vis-a-vis AMSCO?
24 A. I don't know any specific relationship with
25 AMSCO. Mr. Wright in my understanding represented, was a

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1 representative of the marketing department involved with
2 Plasticizers which included PCBs. And that's the extent
3 of my association with Mr. Wright of PCBs.

4 Q. Were you aware of anyone within Monsanto with
5 the specific role as a liaison between AMSCO and
6 Monsanto? And maybe liaison is the improper word, but the
7 point person responsible for that relationship.

8 A. I don't know the individual, no.

9 Q. Was there such an individual?

10 A. I always had the understanding that distributors
11 should, just like other customers, were assigned to
12 individuals in Monsanto's marketing team.

13 Q. Okay. Sir, if you look at Papageorge 24, which
14 is the April 3, it is 23, the memo in front of you, he has
15 this as personal and confidential. Do you see that?

16 A. I do.

17 Q. Do you know why it is so labeled?

18 A. That's the, that's Dr. Paton's. You'll have to
19 ask him. I don't know why he did that.

20 Q. He says many of you have asked about the
21 availability of Bulletin O/PL-306 which covers all
22 Aroclors. Do you see that?

23 A. Yes.

24 Q. Were you aware that people were asking for the
25 bulletin at this time, during this time frame?

1 Q. (By Mr. Taylor) Well, let me rephrase it. Were
2 you aware that customers would use the technical
3 bulletins, in this case, Technical Bulletin 306, for
4 guidance in and/or guidance for uses of the Monsanto
5 Aroclor PCB products?

6 MR. DIMURO: I'll object to the form again. You
7 can answer it.

8 A. This bulletin is not, at least in Monsanto's
9 viewpoint, a technical bulletin. It was a promotional
10 bulletin sharing with potential and current customers
11 Monsanto's awareness that some of the PCBs were put in
12 certain applications and they apparently were successful
13 and they wanted to let others know this. And they did it
14 through a bulletin like this one.

15 Q. (By Mr. Taylor) And, sir, my question is: Were
16 you aware that customers would utilize that bulletin in
17 their own use or their own formulation of uses for the PCB
18 products?

19 MR. DIMURO: I'll object to the form.

20 A. I, of course, don't know how the customers, each
21 and every one of them, used the bulletins specifically in
22 their application and their use of PCBs. There's nothing
23 in the bulletin that tells them how much PCBs to add to
24 products or components X, Y, and Z to reach some end
25 point. There's nothing like that. There are properties

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1 A. I don't know that I gave it any specific
2 thought. This, of course, brought me up to date as of
3 April 3rd, 1970.

4 Q. Were you aware that Bulletin 306 was out of
5 print, which Mr. Paton states in the second sentence of
6 that first paragraph?

7 A. No. Because as of April 3rd, 1970, that
8 particular bulletin was not a, did not have a high
9 priority in my list of things to do, so I didn't address
10 it.

11 Q. Were you aware of that bulletin?

12 A. Yes.

13 Q. And why was it low on your priority list?

14 A. Well, sir, there were dozens of bulletins. And
15 the PCBs issue was a fast evolving issue, so I had to keep
16 up with the new developments and I didn't get a chance to
17 go back to bulletins until much later on.

18 Q. Well, were you aware that that bulletin had been
19 distributed for some time to customers?

20 A. Yes.

21 Q. And were you aware that customers utilized that
22 bulletin to formulate applications for PCB products?

23 MR. DIMURO: Object to the form.

24 A. I have some problem, sir, understanding the
25 expression formulate their products.

1 in there like the specific gravity, and the viscosity, and
2 flame resistance, these kinds of things.

3 Q. (By Mr. Taylor) Well, you were certainly aware,
4 weren't you, that customers relied on those technical
5 bulletins to help them in utilizing products?

6 MR. DIMURO: I'm going to object to the form.

7 A. I don't know that I realized that. I suspected
8 it.

9 Q. (By Mr. Taylor) Okay. Well, what, let me ask
10 it to you this way: What did you think customers would do
11 with the Technical Bulletin 306?

12 MR. DIMURO: I'm going to object to the form
13 again.

14 A. There again, I'll have to share with you what I
15 would have done under the same circumstances. I don't
16 know what the customer did. But if I were to get a
17 bulletin like that and was serious about at least trying
18 out the PCBs that were described, I would get a hold of a
19 Monsanto representative and ask a lot of questions. I
20 would ask for samples. I would find, if I didn't have my
21 own laboratory, I would find somebody to do a little
22 studying or testing for me and so on.

23 Q. (By Mr. Taylor) And, in fact, that's often what
24 the Plasticizer Council would be used for, the Monsanto
25 Plasticizer Council would be used for?

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1 MR. DIMURO: I'll object to the form.
 2 A. I'm not familiar with the term of Plasticizer
 3 Council.
 4 Q. (By Mr. Taylor) Were you aware of a body within
 5 Monsanto called the Plasticizer Council?
 6 A. No.
 7 Q. And the first sentence or the next heading, he
 8 says, Mr. Paton writes, for your information only. Do you
 9 see that there's a heading that says, for your information
 10 only?
 11 A. I see it now, yes.
 12 Q. Do you have an understanding as to what
 13 Mr. Paton meant by that?
 14 A. Well, as a recipient of this document at that
 15 time, I have to share with you my understanding.
 16 Q. That's what I'm asking.
 17 A. I don't know what Dr. Paton had in mind.
 18 Q. What was your understanding?
 19 A. I understood that this was to bring me up to
 20 date, if you will, regarding the availability of this
 21 O/PL-306 Bulletin. And the intentions regarding the
 22 reissue and the intent to publish a new bulletin which
 23 would give physical properties without referencing
 24 applications. And that was for the, the readers'
 25 information.

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1 Q. Okay. Now he says in the first sentence that
 2 306 must drastically, must be drastically revised in light
 3 of the letters on PCBs that we have sent to our core
 4 customers in the U.S. and plan to send to U.K. and
 5 Canadian customers.
 6 MR. DIMURO: I just note it says letter, I think
 7 you said letters.
 8 MR. TAYLOR: I'm sorry. Letter.
 9 Q. (By Mr. Taylor) When he says must be
 10 drastically revised, do you know what he means by that?
 11 A. He's referring to a letter that was mailed in
 12 February of 1970 in which the latest information Monsanto
 13 had regarding the finding of some PCBs in the environment
 14 were reported to the customers. And in Dr. Paton's
 15 opinion, that information should be included in any
 16 revision of this O/PL-306 Bulletin.
 17 Q. Do you remember having a conversation with
 18 Mr. Paton regarding this topic?
 19 A. Yes.
 20 Q. Outside of this memorandum?
 21 A. Yes.
 22 Q. And what were the nature of those? Why don't
 23 you give me the details of those conversations as they
 24 just related to the revision of Technical Bulletin 306.
 25 A. Well, at that point in time, Monsanto was

1 seriously considering the discontinuance of the sale of
 2 PCBs to what was perceived to be open uses. So that
 3 thought was interwoven in this decision on how, if at all,
 4 we can make Bulletin 306 useful and current. It's that
 5 kind of discussion that I was involved with. And the
 6 decision was finally made since we're bowing out of the
 7 business, there's no, it makes no sense to say we
 8 recommend you use it in chlorinated rubbers and, and in
 9 caulks and --
 10 Q. I'm sorry. At this time?
 11 A. Yes.
 12 Q. Had the decision been made that you were going
 13 to bow out of the business by April 3 of 1970?
 14 A. The decision had been made by the business
 15 groups involved but had not been taken to the top
 16 authority within Monsanto for final approval.
 17 Q. And that didn't happen until sometime in August
 18 of 1970?
 19 A. No. That approval was made, or that, that was
 20 considered by the top management committee about two weeks
 21 after the date of this memo.
 22 Q. Okay.
 23 A. Two to three weeks. In April of '70.
 24 Q. In April of '70, the decision was made that for
 25 sure the Aroclor PCB products were going to be pulled from

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1 the market?
 2 A. Yes.
 3 Q. He goes on to say, as you know we have programs
 4 underway to solve the problems the PCB situation poses for
 5 our Aroclor business. Do you see that?
 6 A. I do, yes.
 7 Q. What programs were underway to solve the
 8 problems?
 9 A. There were many.
 10 Q. What types of programs?
 11 A. Improved analytical methodology so the
 12 analytical laboratories no matter where they were located
 13 could come up with results with high confidence. There
 14 was a concerted effort to demonstrate that, whether or not
 15 the only PCBs in the environment were the higher
 16 chlorinated or whether the others were also out there.
 17 There were studies made to find alternative chemicals to
 18 the PCBs to satisfy the needs of the customer.
 19 Q. Mr. Papageorge --
 20 MR. DIMURO: I don't think he was done.
 21 Q. (By Mr. Taylor) I'm sorry. I thought you were
 22 finished.
 23 MR. DIMURO: Are you done?
 24 A. Well, I forgot exactly what I covered, but when
 25 you say what program, there were many irons in the fire at

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1 the time to try to keep PCBs out of the environment and
 2 still provide Monsanto's customers with materials that
 3 would meet the customers' needs.
 4 Q. (By Mr. Taylor) I believe the first program you
 5 mentioned, and I didn't follow up with a question because
 6 you were going through a list of programs, you said
 7 something about to determine with confidence something.
 8 And if I asked you to elaborate more, on the confidence in
 9 what?

10 A. The analysis of PCBs even to this day is highly
 11 sophisticated. In my personal experience, every
 12 laboratory that called me to receive samples ran into
 13 problems invariably. The ability of virtually every
 14 laboratory that entered the PCBs analytical business, I'm
 15 going to call it, to be able to reproduce its results with
 16 a high degree of confidence was not easily achieved. Some
 17 laboratories, it would take them virtually almost a year
 18 to get to that point. So in early 1970, a lot of effort
 19 worldwide was going on by many, many laboratories to
 20 achieve that degree of confidence.

21 Q. Were any of these programs that were underway
 22 geared towards discrediting some of the tests that came
 23 out of say, Sweden, and to rebut them, in other words, to
 24 say that PCBs were in fact safe?

25 MR. DIMURO: I'll object to the form.

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1 A. No, sir. I would never have been involved in an
 2 activity like that personally.

3 Q. (By Mr. Taylor) Wasn't it true at some point
 4 Monsanto was sponsoring studies to show that what was
 5 being said about them in the news media and in various
 6 studies was incorrect?

7 MR. DIMURO: Object to the form again.

8 A. No. The studies were, that Monsanto did or
 9 sponsored, were to verify what was being reported by other
 10 entities. And if the results matched, we agreed. If they
 11 didn't, we'd have to sit down and discuss why the
 12 differences. An example was that previous meeting in
 13 Duluth where we compared notes. At no time did Monsanto
 14 conduct any study to prove somebody was lying. That was
 15 not the intent.

16 Q. (By Mr. Taylor) And that's not my question. My
 17 question was: Those studies were underway during this
 18 time period in April of 1970?

19 A. Yes.

20 Q. And that the decision to pull Aroclors from the
 21 market was delayed until some of those tests could confirm
 22 or discredit some of the tests that were in the media,
 23 correct?

24 MR. DIMURO: I'll object to form.

25 A. No. The decision, sir, was not delayed because

1 the decision was made and the studies were still
 2 underway. But there was enough evidence out there, not a
 3 hundred percent of it, but enough evidence to give
 4 Monsanto good reason to bow out of those uses that we knew
 5 resulted in immediate entry into the environment.

6 Q. (By Mr. Taylor) Okay. And when did Monsanto
 7 first become aware, at least according to your information
 8 that you were able to review through the tutorials or any,
 9 through that process of your learning process on the
 10 product, that PCBs were harmful or allegedly harmful to
 11 the environment?

12 MR. DIMURO: Object to the form.

13 A. I hesitate because I don't know that it was a
 14 single date. It was an evolving set of information that
 15 kept coming in. And some of it was later disproven, but
 16 at that point in time, it appeared reliable. As to when
 17 did Monsanto become aware, of course there were the
 18 reports of the pelicans off of the southern coast of
 19 California out of the University of Berkley.

20 Q. (By Mr. Taylor) And there was a report from
 21 Sweden?

22 A. No. This was Dr. Riesborough out of the
 23 University of California. The initial report came out of
 24 Sweden but that was confusing because the report, the
 25 reporters, the researchers who did the work were not

1 certain that it was PCBs the Biphenyl, they reported a
 2 Biphenol.

3 Q. Prior to that report, was there any awareness by
 4 Monsanto that there was a problem with PCBs?

5 A. Absolutely not.

6 Q. And that's, I'm referring to that there is, were
 7 allegedly problems with PCBs effecting the environment.

8 A. I'm confused, they're alleged. There was a
 9 presence in the environment.

10 Q. That's right and that's what the Swedish report
 11 stated?

12 A. Yes.

13 Q. Prior to that, was there any evidence in the
 14 hands of Monsanto that there was any, there should be any
 15 concern about PCBs in the environment?

16 A. PCBs were considered by Monsanto and its
 17 customers and the scientific community as being so
 18 inactive. And the expression that I've been using is like
 19 they were like pebbles on the beach. They were there, but
 20 no harm was noticeable.

21 Q. So my question is: Prior to that Swedish
 22 report, there was no study or indications that concerned
 23 Monsanto about PCBs?

24 MR. DIMURO: I'll object to the form. You can
 25 answer it.

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1 A. Monsanto had studies regarding an evaluation of
2 its toxicity as an industrial chemical. So there were
3 studies on PCBs.

4 Q. (By Mr. Taylor) I'm talking about any ill
5 effects to the environment.

6 A. To the environment, no.

7 Q. And how about prior? When was the first study
8 you're aware of that there were allegations that PCBs were
9 harmful to the human body?

10 MR. DIMURO: Object to the form.

11 A. Can you help me with your understanding of what
12 you mean by studies? You mean did we get human beings
13 lined up and feed them PCBs and that kind --

14 Q. (By Mr. Taylor) No. There were certain studies
15 whether or not, after some of the information came out
16 about the effect on the environment that focused on the
17 possible effects it may have on human beings, correct?

18 MR. DIMURO: Object to the form again. You're
19 not going to give any time period so it could be yesterday
20 studies came out.

21 Q. (By Mr. Taylor) I mean prior to it being pulled
22 from the market.

23 A. Prior to the market pull, I don't recall any
24 studies relating to human beings and PCBs and harmful
25 effects at that point in time. I just don't recall that.

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1 Q. Sir, if you go back to Cumming Paton's
2 memorandum, he states that we are cooperating with
3 scientists and public agencies who have interest in the
4 PCB situation. Is that what you were talking about, some
5 of the studies, some of the programs underway?

6 A. Yes.

7 Q. And he says that it would appear inconsistent
8 from an image standpoint that at the time we are promising
9 support to scientists slash agencies we publish a new
10 bulletin promoting PCBs. Do you see that?

11 MR. DIMURO: Well, he sees that, but you left
12 out a line.

13 A. You left out a parenthetical phrase there, in
14 all sincerity let me emphasize.

15 MR. DIMURO: So just for the record, the
16 sentence is: It would appear inconsistent from an image
17 standpoint that at the time we are promising open parens
18 in all sincerity let me emphasize close parens support to
19 scientists slash agencies we publish a new bulletin
20 promoting PCBs period.

21 Q. (By Mr. Taylor) What does he mean by it would
22 be inconsistent from an image standpoint?

23 A. You would have to ask Dr. Paton what he meant.

24 Q. Did you talk to him about that?

25 A. No.

1 Q. Do you have an understanding of what he meant by
2 that?

3 A. I have a personal understanding.

4 Q. What is that?

5 A. He didn't want a document to be issued promoting
6 PCBs when we had just sent out a letter cautioning people
7 about how careful they should be regarding the use of
8 those PCBs. And he just didn't want anything to be
9 perceived by any reader as an inconsistency.

10 Q. Well the next statement, sir, he states that the
11 lack of O/PL-306 should not inhibit your sales or
12 efforts. Do you see that?

13 A. Yes.

14 Q. Isn't that inconsistent, Mr. Papageorge?

15 MR. DIMURO: I'll object.

16 A. Well, you're assuming that the sales and efforts
17 are only related to PCBs. He's implying that substitute
18 products available to Monsanto would still be part of the
19 sales efforts.

20 Q. (By Mr. Taylor) How do you know he's implying
21 that?

22 A. I'm just repeating to you what I read in that
23 sentence. That's, as I said earlier, you need to ask
24 Dr. Paton.

25 Q. I'm reading the sentence as well and I -- well,

1 strike that.

2 He does not say in that sentence that it should
3 not inhibit your sales or your efforts to Aroclors which
4 do not contain PCBs, correct?

5 A. Not on the Aroclor but phthalic esters and maleic
6 esters and all kinds of Plasticizers.

7 Q. Yeah. But this bulletin is specifically
8 referring to Aroclor Bulletin 306?

9 A. Yes.

10 Q. All right. So those, the sales efforts --
11 strike that.

12 The Technical Bulletin 306 only deals with
13 Aroclors, correct?

14 A. Yes.

15 Q. And in fact, at this time, you were still
16 selling, Monsanto was still selling Aroclor 1254 for
17 example?

18 A. That's true.

19 Q. And you stated that within that month of April,
20 1970, the upper brass had made a decision to stop selling
21 PCB Aroclors, correct?

22 A. Yes.

23 Q. But, and that, in fact, the sales of such
24 Aroclors continued until August of 1970, correct?

25 A. Yes.

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1 Q. So the salesmen were using efforts to sell
2 Aroclor 1254 for five months after this bulletin, correct?

3 MR. DIMURO: I'll object to the form.

4 A. I hesitate because a salesman who has a customer
5 buying Aroclors for years is different from the salesman
6 who goes out seeking a new customer for Aroclors or PCBs.

7 Q. (By Mr. Taylor) Are you aware of any document
8 where salesmen were told not to seek new customers for PCB
9 Aroclors?

10 A. No. That wouldn't be done normally. But there
11 is a reference to salesmen who have been through training
12 school were given handouts on Aroclors. Those handouts,
13 as I recall, were cautionary types of remarks about
14 presence in the environment, unknown effects on the
15 environment, proper use of Aroclors. So these were not
16 handouts to promote the expanded use of Aroclors.

17 Q. So your testimony that his statement that the
18 lack of O/PL-306 should not inhibit your sales or your
19 efforts is referring to non-PCB Aroclors?

20 MR. DIMURO: I'll object. That's not what he
21 said. You can answer if you can.

22 A. The addressees of this memo are all marketing
23 men who sold at that time or were involved in the sales at
24 that time of the complete line of Monsanto's Plasticizers.

25 Q. (By Mr. Taylor) Including Aroclor 1254?

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1 A. Which was just a fraction of the total.

2 Q. And also Aroclor 1260?

3 A. Yes.

4 Q. And the other Aroclors that were PCBs?

5 A. The whole line, that's correct, yeah.

6 Q. But my question is, and I just want to make sure
7 I understand this sentence. The, well let me ask you
8 this: I just want to make sure I understood your earlier
9 testimony regarding this one sentence about when he says
10 the lack of O/PL-306 should not inhibit your sales or your
11 efforts. I want to focus on that sentence.

12 A. Yes.

13 Q. And as I understood your earlier testimony, and
14 correct me if I'm wrong, I thought you testified that that
15 sentence was referring to sales efforts for non-PCB
16 Aroclors?

17 MR. DIMURO: I'll object to the form.

18 Q. (By Mr. Taylor) Is that incorrect?

19 A. No. I understood that sentence to apply to the
20 total efforts of this group of marketing people a fraction
21 of which included the PCB materials of concern at the
22 time.

23 Q. Okay.

24 A. And it was not intended to promote further use
25 or misuse, or I don't know how else to put it except that.

1 Q. Okay.

2 A. Those are my understandings. Now if you want to
3 know what Cumming Paton actually meant, you'll have to ask
4 him.

5 Q. I will ask you though, in your opinion -- strike
6 that.

7 Mr. Paton makes a statement that it would appear
8 inconsistent from an image standpoint and we had talked
9 about that a little bit earlier. Does it appear
10 inconsistent to you sitting here today that the company is
11 pulling Aroclors from the market and at the same time
12 advising their sales force not to let that lack of
13 O/PL-306 inhibit the sales efforts?

14 MR. DIMURO: I'll object to the form.

15 Q. (By Mr. Taylor) Doesn't that seem inconsistent
16 to you?

17 A. No. Because I see other Plasticizers in the
18 picture.

19 Q. I understand. And I understand not every
20 Plasticizer has PCBs.

21 A. That is correct.

22 Q. But in that picture, are, PCB Aroclors are
23 Plasticizers, correct?

24 A. Some Aroclors are PCBs, and some of the
25 applications were described as Plasticizers. So both of

1 those terms do apply. But --

2 Q. You don't think it's inconsistent to continue
3 the sale of Aroclors that are PCBs with the company's
4 position that they were going to pull it from the market?

5 MR. DIMURO: I'm going to object to the form.

6 A. I don't read that in here anywhere, sir.

7 Q. (By Mr. Taylor) I'm ask asking you, I'm not
8 referring to the document. I'm asking you.

9 A. That's what I thought we were talking about,
10 this document.

11 Q. I'm asking generally. Don't you think it's
12 inconsistent. And you said you don't think it's
13 inconsistent. And I'm asking you what, why is it not
14 inconsistent?

15 A. Will you repeat your question again? I was
16 looking at it from a different perspective.

17 Q. In April of 1970, the company had decided to
18 pull Aroclors from the market. Correct?

19 A. The business directors had decided in April.
20 And later on they passed that recommendation on to the top
21 corporate management.

22 Q. I understand.

23 A. And that decision, that was approved in May of
24 1970.

25 Q. Okay. And we have also established that the

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1 Aroclors which contain PCBs were not pulled until August
2 of 1970?

3 A. Correct.

4 Q. And my question is: Is it not inconsistent for
5 the top brass to make the decision to pull it from the
6 market in May and for the sales force, the marketing force
7 to continue to sell the product until August? Isn't that
8 inconsistent?

9 MR. DIMURO: I'll object to the form. You can
10 answer.

11 A. I think we're nitpicking the definition of
12 inconsistency here --

13 Q. (By Mr. Taylor) Why don't you tell me your
14 version?

15 MR. DIMURO: Let him finish.

16 A. In the real world, sir, you do not arbitrarily
17 withdraw a product from the marketplace leaving the
18 customer in a position where he has no substitute
19 material. He can't make his products. So this is why the
20 August date was picked, to give them a little lead time to
21 find alternate materials to inform their customers. What
22 your question implies is that the minute the decision was
23 made in May by next midnight, we stop. That is an
24 irresponsible action.

25 Q. (By Mr. Taylor) Okay. I'm not arguing with you

1 MR. TAYLOR: I'm going to mark this Papageorge
2 24. This is a document that just has Plasticizer written
3 on top. It bears Bates MAE 059971-73.

4 (Papageorge No. 24 was marked for identification
5 by the reporter.)

6 Q. (By Mr. Taylor) Mr. Papageorge, if you could
7 just review that and tell me if you --

8 MR. GINSBURG: What were the numbers again,
9 please?

10 MR. DIMURO: 59971-73.

11 MR. GINSBURG: Thank you.

12 Q. (By Mr. Taylor) If you can tell me if you've
13 seen that document before.

14 A. I have read the exhibit and although this
15 subject matter is, I'm familiar with the subject matter, I
16 do not recall this document at all.

17 Q. Okay. Well, let's start with the second
18 paragraph on the first page. It refers to a 1969, I'm
19 quoting, exhaustive survey of our PCB customers was made
20 to determine exactly where these products were being
21 used. Do you see that?

22 A. Yes.

23 Q. In your position with Monsanto in the early
24 '70's, did you have an occasion to review the results of
25 that survey?

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1 whether it's irresponsible or responsible, but could that
2 have been done within the structure of Monsanto to stop it
3 immediately when your top brass made the decision to pull
4 it from the market?

5 A. Could it have been done? It could, but it would
6 produce more problems than solutions to a problem.

7 Q. And the problems would be in the nature of angry
8 customers?

9 MR. DIMURO: I'll object to the form.

10 A. No, sir. It would be irresponsible use of
11 PCBs. It would be the importation of PCBs from worldwide
12 producers. All kinds of problems.

13 Q. (By Mr. Taylor) I understand. In the last
14 sentence on the first page he writes, we plan to issue a
15 much condensed bulletin giving the physical properties of
16 the various Aroclors without reference to applications.
17 Do you see that?

18 A. Yes.

19 Q. Why was the, why were the references to
20 applications to be taken out of the technical bulletin?

21 A. Well, since we knew that we were going to
22 discontinue selling to what we perceived to be open uses,
23 we were in no position to list applications which we
24 thought would be closed uses in the Plasticizer field. We
25 just didn't have any.

1 A. No.

2 Q. Were you aware of an exhaustive survey?

3 A. I was aware that had been made, yes, sir.

4 Q. And can you give me, tell me about that survey
5 and I'll follow up with some questions. Who made it?
6 When it was made? Things like that.

7 A. I don't know that I can give you too much of the
8 details. I was informed that the marketing teams in
9 Monsanto had perused all the available records in which
10 they found names of customers, quantities of PCBs
11 purchased, and the types purchased. And an attempt was
12 made to establish the use of those PCBs, what the
13 application was.

14 Q. Well, who, do you know who directed that
15 survey? Let me start with this first: Who directed that
16 survey be conducted or who ordered that survey?

17 A. Well, the two directors of the business groups
18 involved were those who monitored and managed the study,
19 through their marketing directors.

20 Q. Okay. And why was that survey conducted?

21 A. I don't know.

22 Q. Was it part of the, part of the response to the
23 negative publicity that PCBs were getting --

24 MR. DIMURO: Objection.

25 Q. (By Mr. Taylor) -- in the late '60's?

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1 A. I don't know, sir.
 2 Q. Well, did you ever see the results of the
 3 survey?
 4 A. No.
 5 Q. Were you ever informed about the results of the
 6 survey?
 7 A. No.
 8 Q. Were, well you had talked in one of the earlier
 9 documents, I believe it was Papageorge 22 which was that
 10 brief summary of the March 17, 1970 meeting on PCBs in
 11 Minnesota --
 12 A. Yes, sir.
 13 Q. -- about the uses of Aroclors?
 14 A. Yes.
 15 Q. And I was wondering if you received some of your
 16 information on the uses of Aroclors from that survey?
 17 A. I received some numbers from Monsanto's
 18 accounting department. But I have no idea where those
 19 individuals got their numbers.
 20 Q. And then the bottom of that page, the first page
 21 of Papageorge 24, it says surface coatings 57 percent. Do
 22 you see that?
 23 A. Yes.
 24 Q. Give me some examples of surface coatings.
 25 A. Lacquers and varnishes. Specialty paints.

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1 Those are the only two that come to mind at the moment.
 2 Q. And miscellaneous. Do you see miscellaneous 6
 3 percent?
 4 A. Yes.
 5 Q. Do you know what types of applications would
 6 fall under that category?
 7 A. It's all the other sales. After subtracting the
 8 first three numbers, the 6 percent was referred to as
 9 miscellaneous.
 10 Q. So you weren't aware what applications were
 11 used --
 12 A. No.
 13 Q. -- from the PCBs?
 14 A. No.
 15 Q. Sir, I'm going to show you a document entitled
 16 "the Specialist" which has three pages attached to it
 17 entitled "technical service bulletin." And "the
 18 Specialist" that I'm going to show you is dated August
 19 28th, 1969 and the "technical service bulletin" behind it
 20 is dated June 24, 1968. And together, they're Bates
 21 stamped MAE 059911, and then 059911.01 through 03.
 22 MS. KARLOVICH: Can you just repeat that last
 23 series of Bates?
 24 MR. TAYLOR: Yeah. 059911.01, .02, .03. And I
 25 ask you if you can mark that.

1 (Papageorge No. 25 was marked for identification
 2 by the reporter.)
 3 A. I have scanned the document.
 4 Q. (By Mr. Taylor) Do you recognize that
 5 publication called "the Specialist?"
 6 A. This is the first time I've seen it.
 7 Q. And how about the "technical service bulletin"
 8 which is attached to it?
 9 A. I have never seen this before.
 10 MR. DIMURO: Matt, just a question, the first
 11 pages I have look exactly the same.
 12 MR. TAYLOR: It must have been a copying error.
 13 MR. DIMURO: They both have the same Bates
 14 number too.
 15 MR. TAYLOR: Yeah.
 16 MR. DIMURO: You're not going to refer to if
 17 there are differences in the document? Oh, the witness
 18 has one with only one page.
 19 MR. TAYLOR: Then that will be the official one.
 20 MR. DIMURO: Are you done with that one?
 21 MR. TAYLOR: I am.
 22 Q. (By Mr. Taylor) I'm going to show you, we'll
 23 mark this Papageorge 26. And I ask you if you recognize
 24 this document.
 25 (Papageorge No. 26 was marked for identification

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1 by the reporter.)
 2 MR. TAYLOR: For the record, it's called
 3 Literature Survey NCR Paper with Other Selected NCR
 4 Patents MAE 059918 through 26.
 5 A. I have scanned the exhibit.
 6 Q. (By Mr. Taylor) And do you recognize it?
 7 A. I do not. I've never seen it before.
 8 Q. Thanks. That's all. We're, I'm going to mark
 9 as Papageorge 27 an excerpt from "Chemical Week" January 4
 10 and February 15, 1964 bearing Bates MAE 059894.
 11 (Papageorge No. 27 was marked for identification
 12 by the reporter.)
 13 Q. (By Mr. Taylor) I'm not going to ask you to
 14 read the article right now, Mr. Papageorge, but is this,
 15 do you recognize this type of document?
 16 A. Recognize it in what way, sir?
 17 Q. As something that would either appear in a
 18 publication dealing with some of Monsanto's products.
 19 A. Well being specific "Chemical Week?"
 20 Q. Yeah.
 21 A. This is a publication dealing, of course, with
 22 the chemical industry and its products.
 23 Q. Is "Chemical Week" published by a third party or
 24 is it published by Monsanto?
 25 A. No, it's a third party.

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1 Q. And was it common -- strike that.
 2 Were you aware from time to time that they would
 3 run articles on a Monsanto product line?
 4 MR. DIMURO: I'll object to the form.
 5 A. This is the first I've seen of any such
 6 advertising or literature.
 7 Q. (By Mr. Taylor) Just from looking at that, can
 8 you tell if that is an advertisement or if this is an
 9 article?
 10 A. I cannot. It's the first I've ever seen it. I
 11 know nothing about this one.
 12 Q. And in your experience at Monsanto, were you
 13 ever aware of articles appearing or advertisements
 14 appearing on any of the Monsanto product lines in
 15 "Chemical Week?"
 16 A. I have never personally seen such articles.
 17 MR. TAYLOR: We'll mark this as Papageorge 28.
 18 (Papageorge No. 28 was marked for identification
 19 by the reporter.)
 20 Q. (By Mr. Taylor) Sir, this is a document bearing
 21 Bates MAE 059895 through 059902. Do you recognize this
 22 document?
 23 A. Yes, it appears to be a document that I was
 24 involved with.
 25 Q. Tell me what your involvement was with this

1 remember producing another newsletter on Aroclors?
 2 A. Correct.
 3 Q. If you turn to Bates 059898 of that document, it
 4 talks about other uses. Do you see that?
 5 A. I do.
 6 Q. And when they say only some of these uses are
 7 listed below -- strike that.
 8 Down at the bottom you'll see where it says, new
 9 uses for the various Aroclors are continually being
 10 developed. Who was developing those uses?
 11 A. Monsanto's research department was involved in
 12 not only conducting their own studies, but also monitoring
 13 what some of the customers, what activities might be
 14 taking place out in the market.
 15 Q. And is it fair to say that Monsanto was
 16 encouraging its customers in their own research efforts to
 17 find uses for Aroclors?
 18 A. Yes.
 19 Q. And it says, the last sentence there, that
 20 Aroclors has resulted in a widespread customer acceptance
 21 and limitless expansion of uses for them. Do you see
 22 that?
 23 A. Yes.
 24 Q. And I want to focus on the word limitless. Why,
 25 or what was your understanding of why the Aroclors had a

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1 document.
 2 A. I was the plant manager at the Monsanto plant in
 3 Anniston, Alabama where a program for communicating with
 4 our employees was instituted. And this document reflects
 5 the information, the handout prepared for the product line
 6 Monsanto referred to as Aroclors.
 7 Q. Okay. And it says on there, the first page,
 8 dear fellow employees, it says that this is the first in a
 9 series of quarterly Anniston Plant newsletters that you
 10 will be receiving during 1966. Do you see that?
 11 A. I do.
 12 Q. Do you remember additional newsletters going
 13 out?
 14 A. For other product lines, yes.
 15 Q. What about Aroclors?
 16 A. There was only one bulletin during that year of
 17 Aroclors. This covered it.
 18 Q. Were there other in subsequent years? Do you
 19 remember other newsletters on Aroclors?
 20 A. No.
 21 Q. You don't. Is that you don't remember or that
 22 there were none?
 23 A. There were none while I was there. I don't know
 24 what my successor did.
 25 Q. So in 1967, for example, '68 and '69, you don't

1 limitless potential for expansion?
 2 A. In 1967, the Aroclors were perceived to be
 3 chemicals with some unique and very attractive
 4 characteristics which included such properties as
 5 unreactive, so they would not, so they were compatible
 6 with many other materials.
 7 The characteristic of fire resistance was a
 8 very, very appealing characteristic for many applications
 9 for which the presence of a flame would not be a serious
 10 matter because the minute that the PCBs were present, that
 11 flame would not be propagated, would not extend any
 12 further.
 13 There were other characteristics that did appeal
 14 to the customers. And it depended really on each
 15 customer's perspective and needs. The fact that these
 16 Aroclors were available either in a solid or a liquid and
 17 the liquids could be very viscous or very thin, free
 18 flowing. The physical properties were appealing.
 19 So with those kinds of properties, it just
 20 appeared that there could well be many, many applications
 21 that had yet to be tried.
 22 Q. Well you had talked about fire resistance. Is
 23 your understanding that, is there any distinction in your
 24 mind between fire resistance and fire retardance as a
 25 benefit of the Aroclor PCB products?

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1 A. In my mind, they are almost synonymous.
2 Q. And I just want to go down a couple of the uses
3 that are in your bulletin, or the newsletter rather. That
4 it was recommended to be used in dental castings; is that
5 correct?
6 MR. DIMURO: I'm going to object to the form.
7 Where is that?
8 A. I see it, yes, sir.
9 Q. (By Mr. Taylor) Is that correct?
10 A. Yes.
11 Q. Okay. And that it was recommended as a vapor
12 suppressant to increase the kill life of insecticides?
13 MR. DIMURO: I'll object to the form again.
14 Q. (By Mr. Taylor) Do you see that? It's right
15 above that one.
16 A. Oh, yes. That was the common understanding in
17 '67. It had the approval of the U.S. Department of
18 Agriculture.
19 Q. And as castings for costume jewelry as well,
20 correct?
21 A. Correct.
22 Q. And what were the, what would the application,
23 well what is a casting, a dental casting?
24 A. Of course, I'm not an expert in the field. It
25 has to do with a process in which a mold is formed and

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1 that mold, whether it be a metal or clay or some other
2 substance, is filled with a material that's an awful lot
3 like wax. And the process, by the way, is called a lost
4 wax process.
5 The wax casting is then placed inside an outer
6 form, is melted, and the final material is injected into
7 that void space like the material used to make the
8 jewelry, for example, or the, the dental bridge work, or
9 what have you. I know I didn't describe that too well,
10 but the --
11 Q. I got the idea.
12 A. The idea is to start with a wax and the PCBs
13 were part of that wax formulation.
14 Q. I understand. I'll show you, and I think this
15 is the last one. There's one more. We'll mark this No.
16 29. Papageorge 29.
17 (Papageorge No. 29 was marked for identification
18 by the reporter.)
19 Q. (By Mr. Taylor) If you could just review that.
20 A. I have reviewed the exhibit.
21 Q. Do you remember seeing, well, do you recognize
22 this "Products 1961" document?
23 A. I do not.
24 MR. GINSBURG: I'm sorry. Do we have a Bates
25 number?

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1 MR. TAYLOR: MAE 059907-08.
2 MR. GINSBURG: Thank you.
3 Q. (By Mr. Taylor) And Page 50 of that document is
4 on the next page. We only have page 50. Do you remember
5 the issue of, that the Aroclor resins had low vaporization
6 losses? Do you remember that, some issue?
7 MR. DIMURO: I'll object to the form.
8 A. Yeah, that is a well known property of Aroclors.
9 Q. (By Mr. Taylor) But that is not a form or a
10 document that you remember seeing before?
11 A. I have not seen it before.
12 MR. TAYLOR: Let me just check my notes. I
13 think that's all I have. I just have one more document.
14 I want to look at. That's all I have.
15 MR. DIMURO: Gary?
16 MR. GINSBURG: I have nothing.
17 MR. DIMURO: Susan?
18 MS. KARLOVICH: No questions.

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1 NOTARIAL CERTIFICATE
2 STATE OF MISSOURI }
3 CITY OF ST. LOUIS }
4
5 I, Tammie A. St. Arbor, Certified Shorthand Reporter
6 and a duly commissioned Notary Public within and for the
7 States of Missouri and Illinois, do hereby certify that
8 there came before me at the offices of Taylor * Schroeder
9 Reporting & Video, 7494 Ethel Avenue, St. Louis, Missouri
10 63117,
11
12 WILLIAM PAPAGEORGE,
13 who was by me first duly sworn to tell the truth and
14 nothing but the truth of all the knowledge touching and
15 concerning the matters in controversy in this cause; that
16 the witness was thereupon carefully examined under oath
17 and said examination was reduced to writing by me; and
18 that this deposition is a true and correct record of the
19 testimony given by the witness.
20
21 I further certify that I am neither attorney nor
22 counsel for nor related nor employed by any of the parties
23 to the action in which this deposition is taken; further
24 that I am not a relative or employee of any attorney or
25 counsel employed by the parties hereto or financially
interested in this action.
17 IN WITNESS WHEREOF, I have hereunto set my hand and
18 seal this 11th day of May, 1998.

Tammie A. St. Arbor, CSR

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1 STATE OF MISSOURI)
2 CITY OF ST. LOUIS)
3
4
5 I, WILLIAM PAPAGEORGE, do hereby certify:
6 That I have read the foregoing deposition;
7 That I have made such changes in form and/or
8 substance to the within deposition as might be necessary
9 to render the same true and correct;
10 That having made such changes thereon, I hereby
11 subscribe my name to the deposition.
12 I declare under penalty of perjury that the
13 foregoing is true and correct.
14
15 Executed this day of ,
16 19 , at
17
18
19
20 WILLIAM PAPAGEORGE
21
22 My Commission Expires: _____
23 Notary Public: _____
24 Signature Page to Mr. DiMuro _____
25 Witness Letter Sent to _____

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1 Taylor * Schroeder Reporting & Video
2 7494 Ethel Avenue
3 St. Louis, Missouri 63117
4 Phone (314) 644-2191
5
6 May 11, 1998
7 Mr. William Papageorge
8 321 Pebble Valley
9 St. Louis, Missouri 63141
9 RE: Maertin, et al. Vs. Armstrong, Monsanto & American
10 Mineral Spirits
11 Dear Mr. Papageorge:
12 Please find enclosed a copy of your deposition taken on
13 April 29, 1998 in the above-referenced case. Also
14 enclosed is an original signature page and errata sheets.
15 Please read the copy of the transcript, indicate any
16 changes and/or corrections desired on the errata sheets,
17 and sign the signature page before a notary public.
18 Please return the errata sheets and notarized signature
19 page to my office at the above stated address within
20 thirty days of the receipt of this letter and deposition.
21 Please do not hesitate to call me at 314/644-2191 with any
22 questions.
23 Sincerely,
24 Tammie A. St. Arbor, CSR
25 Enclosures
cc: Mr. Taylor
Mr. DiMuro
Mr. Raditz
Ms. Karlovich
File

DEPOSITION OF WILLIAM PAPAGEORGE, 4/29/98

STATE OF MISSOURI)
CITY OF ST. LOUIS)

I, WILLIAM PAPAGEORGE, do hereby certify:

That I have read the foregoing deposition;

That I have made such changes in form and/or substance to the within deposition as might be necessary to render the same true and correct;

That having made such changes thereon, I hereby subscribe my name to the deposition.

I declare under penalty of perjury that the foregoing is true and correct.

Executed this 27th day of May,
1998, at National Bank, Creve Coeur, Mo.

William Papageorge

WILLIAM PAPAGEORGE

JEAN M. FUCHS
NOTARY PUBLIC—STATE OF MISSOURI
ST. LOUIS COUNTY
MY COMMISSION EXPIRES AUG. 18, 2000

My Commission Expires:

8-18-00

Notary Public:

Jean M. Fuchs

Signature Page to Mr. DiMuro

Witness Letter Sent to

Mr. Papageorge 5/11/98 X

7494 Ethel Avenue
St. Louis, MO 63117
(314) 644-2191
800-280-DEPO
(314) 644-1334 Fax

TAYLOR & ASSOCIATES REPORTING, INC. d/b/a

TAYLOR * SCHROEDER REPORTING & VIDEO

Witness Errata Sheet

Witness Name: William Papageorge

Case Name: Maertin, et al. vs Armstrong, Monsanto & American
mineral spirits

Date Taken: April 29, 1998

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Line # 11

Should Read: for, not as innocuous as the phthalic esters.

Reason for Change: Spelling

Page # 7

Line # 15

Should Read: But the phthalic esters are less apt to cause --

Reason for Change: Spelling

Page # _____

Line # _____

Should Read: _____

Reason for Change: _____

Page # _____

Line # _____

Should Read: _____

Reason for Change: _____

Page # _____

Line # _____

Should Read: _____

Reason for Change: _____

Page # _____

Line # _____

Should Read: _____

Reason for Change: _____

Witness: William Papageorge

Members: National Court Reporters Association
Missouri Court Reporters Association
DEPONENT

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WATER PCB-SD0000071955

BRIEF SUMMARY OF PCB MEETING
March 17, 1970

I. PRESENT RESEARCH.

A. G. Fred Lee, Water Chem. Lab., Univ. of Wisconsin.

Four questions are important to the study:

1. What are the compounds that interfere in the chlorinated hydrocarbon analyses in fish?
2. What is the relation with Monsanto's Aroclors?
3. What are the sources of PCB's in the environment?
4. What significance do these PCB's have?

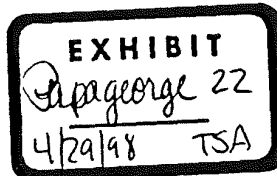
Fish have been sampled from the Milwaukee River area. Water contains PCB's in the low ppt range. Carp contain 1 - 5 ppm of PCB-like compounds. Peaks from gas chromatographs of these compounds were identical to those for Monsanto's Aroclors. Studies are now being conducted to determine the sources of these compounds within the river.

B. William T. Donaldson, Southeast Water Lab., FWPCA.

Discussed the analytical capabilities in quantitating the various peaks and trying to separate the isomers. They have used calibrated chlorine detectors, gas chromatographs, GC mass spectra and have attempted to determine the degree of chlorination of each compound. Thus far, they have gotten 95% pure samples. PCB's with less chlorine may be more toxic.

C. Matthew Zabik, Pesticide Research Center, Michigan State University.

While conducting DDT - DDE residue analyses on coho salmon, a series of chlorinated compounds whose GC patterns match those of PCB's were found along with trimethylbenzenes, phthalates, etc. Attempts have been made to separate the PCB's by nitration and sulfonation. No consistent pattern between



MAE 059941

the material first reported as DDE and actual DDE has been noted. It varied in coho salmon taken from the same area (1 to 30% other material).

D. J. J. Lichtenberg, FWPCA Analytical Quality Control Laboratory, Cincinnati.

PCB's have not been recognized as a real problem in the water monitoring program. They do interfere with DDT or DDE analyses.

E. E. H. Dustman, Patuxent Wildlife Research Center, Bureau of Sport Fisheries and Wildlife.

In tests conducted on ring-necked pheasant, mallard ducks, bobwhite quail, and Coturnix quail, the higher chlorinated compounds were found to be more toxic to birds (in cold-blooded animals the low Cl ones are more toxic). Aroclors tested: 1232, 1242, 1248, 1254, 1260, 1262. A great species difference in susceptibility was noted. PCB's were found in all eagles checked.

F. Richard A. Schoettger and David L. Stalling, Fish Pesticide Research Laboratory, Columbia, Missouri.

Tested Aroclors 1222 - 1268 and some of the 4,000 and 5,000 series. Found that the toxicity is greater at lower degrees of chlorination.

96-hour TL_m cutthroat trout: 1 ppm for 1221
Goes up for 1248
40 ppm for 1254

Gammarus sp. 50 ppm

The difference in toxicity to fish and birds in relation to the amount of chlorination of the PCB's might be due to the method of exposure (birds were fed PCB's while for fish the PCB's were in the water) or to water solubility limitations.

Discussed PCB analysis and interference with DDE, heptachlor, and aldrin analyses. Work is underway using mass spectroscopy to further identify compounds in flesh and other samples. Pond studies and lab bioassays are being started.

MAE 059942

G. Sidney Williams, Food and Drug Administration.

PCB's have been found only in a few foods: catfish 0.5-12 ppm. In one localized case it was found in milk (3-12 ppm). It was later discovered that oil that had leaked from a transformer was substituted for the fuel oil usually mixed with 2,4-D to spray for brush along power lines. FDA has no plans to establish food tolerances.

H. J. C. Street, Department of Animal Science, Utah State University.

In mammals the PCB's were found to cause hepatic microsomal enzyme induction (this was increasingly strong with higher Aroclors). There was extensive transformation during metabolism. 1248 and 1254 are commonly found in animal tissue. It appeared that ones with low Cl were more readily metabolized, so they would disappear from the residue. This may indicate that residue found in the animal does not indicate the source accurately. The interaction with other pesticides is an area where further research is needed.

I. John J. Birdsall, WARF Institute, Madison, WI.

Many samples from heavily industrialized areas contain substances like PCB's. Other chlorinated compounds are also found that have not been positively identified. BHC or Lindane may be one of these. Have completed a large number of samples from Lake Michigan, but positive confirmation of PCB's is needed.

J. William B. Papageorge, Monsanto Company.

Uses of Aroclors:

Biggest: Electrical transformers (1248, 1254)
Capacitors ^{and} in heat transfer systems
Plasticizer in Cl rubbers (1254, 1260)
Caulking materials
Hydraulic fluids (1242, 1248)

Production has increased 8% per year, but no dramatic increase recently. 1242, 1254, and 1260 account for most of the use.

MAE 059943

K. Jack T. Garrett, Monsanto Company.

Discussed toxicity to rats and beagles (paper distributed). Discussed the problem of establishing human tolerances.

II. ANALYTICAL.

1242, 1248, 1254, and 1260 Aroclors should be used as references. The separation of PCB's into isomers and homologs is a must for positive identification. Sample clean-up is critical and recovery measurements are necessary. The proposed method is:

1. Extraction with mixed ethers or methylene chloride.
2. For samples with fats, waxes or oils, use Jones-Riddich acetonitrile - petroleum ether partitioning.
3. Florisil column chromatography.
4. Silicic acid column chromatography.
5. Detection by GC using chlorine specific detector and support coated open tubular or capillary column.
6. Mass spectroscopy confirmation.

III. BIOLOGICAL.

A. Monitoring program.

1. Incorporate the monitoring of PCB's into the present pesticide monitoring program on Lake Michigan as a pilot study by 1971.
2. All fish residue monitoring should be based on total fish residues, although levels in fat should be determined concurrently.
3. All sampling should be done on a statistically sound basis.
4. Fish monitoring should receive priority but water measurements should be made where appropriate.

MAE 059944

5. Objectives of the monitoring program should be as follows:

- a. Establish existing PCB levels.
- b. Establish the major sources of PCB introduction into the environment.
- c. Establish a baseline of PCB concentrations which can be used later for determining long-term trends.

B. Biological Research Needs.

1. Initial laboratory bioassay should be made and limited to testing four Aroclors, 1242, 1254, and 1260 (high production) and 1248 (high persistence).
2. Representative species from all trophic levels should be tested to determine acute toxicities.
3. Representative species from all trophic levels should be tested through at least one generation with emphasis on the effects on survival, growth and reproduction.
4. Multiple generation tests should be made with some organisms having short life histories.
5. Physiological studies should be made to determine the effects of PCB's on enzyme systems, hormones, and tissues.
6. Studies should be made on the toxicity of PCB degradation products.
7. Food chain studies should be made to consider the PCB residues at each level. The compounds present at different levels must be characterized in relation to parent compounds.
8. PCB levels must be analytically measured in test tanks for all bioassays.

MAE 059945

TO: Cumming Paton - General Offices

DATE: April 3, 1970

SUBJECT: AROCLOR BULLETIN O/PL-306

REFERENCE:

TO : D. H. Bechtold - Chicago
R. N. Brell - Wilmington
J. H. Brydon - Montreal
G. Chew - Hong Kong
E. H. Fording - New York
J. D. Fowler - Melbourne
R. V. Johnson - RJOHN
J. J. McNamara - Everett
R. A. Onians - Brussels
G. W. Sperberg - GSPER
E. C. Wilde - Akron

cc M. W. Farrar - JFQ Research
R. G. Hutchison - RHUTC
E. V. John - EJOHN
Dr. R. E. Kelly - RKELL
W. B. Papageorge - WPAPA
Phocion S. Park - PPARK
W. E. Schalk - WSCHA
J. E. Smith - JSMT
J. E. Springgate - JSPRI
W. F. Waychoff
J. D. Wright - JWRIG

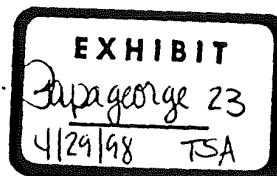
PERSONAL AND CONFIDENTIAL

Many of you have asked about the availability of Bulletin O/PL-306 which covers all Aroclors and all applications. It has been out of print for some time.

For your information only:

O/PL-306 must be drastically revised in the light of the letter on PCBs that we have sent to Aroclor customers in the U. S. and plan to send to U. K. and Canadian customers. As you know, we have programs underway to solve the problems the PCB situation poses for our Aroclor business. We are co-operating with the scientists and public agencies who have interests in the PCB situation. It would appear inconsistent from an image stand-point that at the time we are promising (in all sincerity let me emphasize) support to scientists/agencies we publish a NEW bulletin promoting PCBs. The lack of O/PL-306 should NOT inhibit your sales or your efforts. Salesmen who have been through the training school were given hand-outs on Aroclors. Many Specialists have been issued and O/PL-311A is available. We plan to issue a much-condensed bulletin giving the physical properties of the various Aroclors without reference to applications.

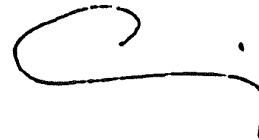
MAE 059946



For customers information:

Send them O/PL-311A (later you will have a physical properties pamphlet).

On O/PL-306, the line should be this. This bulletin is long out of date. Mere reprinting we feel would be misleading. We have various application research programs underway in hot melts, sealants and coatings involving Aroclors. We want to get the data and then publish it later. We feel this approach will be much more accurate and useful for our customers.



Cumming Paton

/dbw

MAE 059947

Plasticizers

Miscellaneous Uses

Monsanto's Aroclor[®] Plasticizer Bulletin O/PL-306 may appear to the layman to indicate that PCB found its way deliberately into almost every product produced in the nation. Applications such as dedusting agents, pesticide carriers, catalyst carriers, impregnating compounds etc. are listed as Aroclor[®] and have been so publicized by the press coverage given to PCBs. The fact is, however, that very few of these uses accounted for more than 4 or 5 drums of PCB per year and, in many cases, the Aroclor[®] used was not a PCB.

In 1969 an exhaustive survey of our PCB customers was made to determine exactly where these products were being used. An example from the survey may be of interest. We found only three customers in 1969 were using any PCB in pesticide formulations. Total consumption was between 5 to 10,000 pounds PCB per year. This application was developed by U.S.D.A. scientists at Beltsville, Maryland in the early 1950s.

The aim was to incorporate "chlorinated polyphenyls" into lindane and possibly chlordane to improve the residual effects. To Monsanto's knowledge there was never any recommendation by anyone for their use on food or other crops. The principal application was to spray metal or painted surfaces to increase the effectiveness of the active pesticide against flies, ants, roaches, etc. In April, 1970 we wrote to Dr. Hayes, Director, Pesticide Regulations Division, U.S.D.A. suggesting that no approvals should be given to pesticide formulations containing PCB.

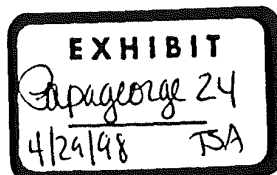
All applications of a "miscellaneous" category uncovered during our 1969 survey of customers buying PCBs through the Plasticizer Business Group of Monsanto were included in the sales ban put into effect in August, 1970.

PCB APPLICATIONS IN PLASTICIZERS

U.S.A. Only (Prior to Discontinuation)

Surface Coatings	57%
Sealants	20%
Adhesives	17%
Miscellaneous	6%

MAE 059971



WATER_PCB-SD0000071963

Encapsulants

The use of PCB as a solvent for the encapsulation of dyes for carbonless carbon paper began in the 1960s. Factors which influenced the selection of PCB were:

dye solubility
U.V. light stability
intensity of color development
speed of color development
moisture resistance
non-reactive with dyestuffs

In spite of research efforts for the past ten years it has only been in the past two years that the promise of satisfactory alternates have been brought to light. Since 1970 we have been supplying non-PCB alternates to this industry. The search for even better replacements continues but none of these is based on PCB.

ENCAPSULANTS U.S. SALES OF MONSANTO PCB

1965	3.5 M lb.
1966	4.1
1967	4.4
1968	6.0
1969	6.5
1970	6.7
1971 Estimate	1.2
1972 Projection	0

do not release

33

MAE 059972

Dye Carriers

do not disclose

Polyester fibers are notoriously difficult to dye and hence a usage of specialty dye-carriers has built up. Historically this has been based on biphenyl (i.e. a non-chlorinated product). In 1969 there was a trend toward the use of low chlorine content PCB. Biodegradation and medical feeding studies have shown this particular PCB to be non-hazardous to the environment. However, even this use is being discontinued within the next two months in favor of a monochlorinated product.

DYE CARRIERS

U.S. SALES OF MONSANTO PCBs

Prior to 1969	0
1970	1.0 M lb.
1971 Estimate	1.1
1972 Projection	0

MAE 059973

TO:

DATE: August 28, 1969

SUBJECT: HOT MELT ADHESIVE FORMULATIONS

We are indebted to Technical Service Department for supplying the attached formulations for hot melt adhesives. All contain an Aroclor 6.

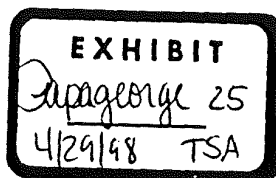
Furthermore, in formulation 6-26F, Ethyl 702 is recommended at 0.5% as antioxidant.

Santowhite Powder should be recommended as a replacement. Santowhite Powder is covered by the FDA to the same extent as Ethyl 702.

A separate Plastifacts will be issued.


Cumming Paton

/dbw



MAE 059911

technical service bulletin

June 24, 1968

FORMULA 6-3

PRODUCT: Hot Melt Adhesive -- Heat-Resistant Type

TYPICAL USE: Sealing seams of multiwall bags to be stored at temperatures to 140°F.

FORMULA:

<u>Wt. %</u>	<u>Material</u>	<u>Supplier</u>
12	Shellwax 700 (fully refined paraffin wax, 185°F melt point)	Shell Chemical Co.
32	Aroclor 5460 (chlorinated polyphenyl)	Monsanto Chemical Co.
20	Elvax 260 (E/VA copolymer)	E.I. duPont de Nemours
0.20	Butylated hydroxytoluene (antioxidant)	Eastman Chemical
36	UNIREZ 915 (modified phenolic resin)	Union Carbide Corp.

* or Filmed Resin Products Filrez 640.

PROCEDURE: Melt wax and Aroclor 5460 to about 350°F. Add Elvax slowly and mix until all is dissolved. Stir in BHT. Then stir in UNIREZ 915 at about 400°F.

PROPERTIES:

Softening Point, Ring & Ball, °C	-	192°F (89°C)
Color, 25% in toluene at 70°C,		
ASTM D-1544	-	9+
Brookfield Viscosity at 360°F	-	4000 cps
Oven Peel Temperature *	-	142°F

* Temperature at which kraft-kraft bond fails when loaded at 160 g/inch width.

MAE 059911.01

The formulas and statements herein are accurate to the best of our knowledge. However, since application in the user's plant is under conditions beyond our control, suggestions and recommendations are made without warranty or guarantee of any kind. Nothing contained herein shall be construed as a recommendation to use any product or process in conflict with existing patents covering any material or its use.

WATER_PCB-SD0000071967

technical service bulletin

July 24, 1968

Formula 6-14

Product: Hot melt adhesive for furniture

Typical Use: Bonding wood-to-wood, wood-to-coreboard

<u>Formula:</u>	<u>Wt. %</u>	<u>Material</u>	<u>Supplier Code</u>
	10	Paraflint SH (Fischer-Tropsch wax)	1
	13	Aroclor 1254 (chlorinated polyphenyl)	2
	12	Aroclor 5460 (" ")	2
	30	Elvax 150 (E/VA copolymer)	3
	0.2	Butylated hydroxytoluene (antioxidant)	4
	35	UNIREZ 9002 (modified phenolic resin)	5

Procedure: Melt wax and Aroclor plasticizers to about 375°F. Mix in BHT. Add Elvax in small portions and mix until each is completely dissolved. Stir in UNIREZ 9002 while reducing temperature to about 350°F. Hold at 350°F. until well blended.

Properties: Ring & Ball softening point - 228°F.
Brookfield viscosity (Model LVT
No. 3 spindle, 12 rpm) - 5700 cps.
Color, 25% in toluene at 70°C.,
ASTM D1544 - 6+
Oven peel temperature* - 160°F.

Supplier Code:

- 1 Moore & Munger, Inc.
- 2 Monsanto Chemical Co.
- 3 E. I. duPont de Nemours & Co.
- 4 Eastman Chemical Products, Inc.
- 5 Union Carbide Corp., Chemical Division

* Temperature at which kraft-to-kraft bond fails when loaded at 160 g/inch bond width

MAE 059911.02

The formulas and statements herein are accurate to the best of our knowledge. However, since application in the user's plant is under conditions beyond our control, suggestions and recommendations are made without warranty or guarantee of any kind. Nothing contained herein shall be construed as a recommendation to use any product or process in conflict with existing patents covering any material or its use.

WATER_PCB-SD0000071968

technical service bulletin

January 3, 1968

FORMULA 6-26F

PRODUCT: Hot Melt Adhesive for Polyolefin Film

TYPICAL USES: Polyolefin-kraft, polyolefin-polyolefin adhesion.

FORMULA:

<u>Wt. %</u>	<u>Material</u>	<u>Supplier</u>
45	UNIREZ XR-8 (experimental resin)	Union Camp Corp.
20	Aroclor 1254 (chlorinated poly-phenyl)	Monsanto Chemical Co.
35	Elvax 4320 (acid terpolymer)	E.I. duPont de Nemours
0.5	Ethyl 702 (antioxidant)	Ethyl Corp.

PROCEDURE: Blend XR-8 and Aroclor together at 300°F and mix in antioxidant. Increase temperature to 350°F and add Elvax in small portion with good agitation. Allow each portion of Elvax to melt and blend in before adding the next. After all components are added, allow to mix at 350°F until hot melt is completely homogenous.

PROPERTIES:

Softening Point, Ring & Ball, °C	- 69
Brookfield Viscosity (Model LVT, #3 spindle)	- 3150 cps at 350°F 8800 cps at 300°F

MAE 059911.03

formulas and statements herein are accurate to the best of our knowledge. However, since application in the user's plant is under conditions beyond our control, suggestions and recommendations are made without warranty or guarantee of any kind. Nothing contained herein shall be construed as a recommendation to use any product or process in conflict with existing patents covering any material or its use.

Literature Survey

NCR Paper

With other selected NCR Patents

(Chem Abst. 1947-1968)

1A37,
1819,
1943

Coating paper, such as that for impact printing. Barrett K. Green (to The National Cash Register Co.). U. S. 2,299,003, Oct. 20. A coating is formed from a mixt. of 2 emulsions, one being formed of a soln. of gum dammar 60, in toluene 60 parts together with di-(p-tert-butylphenyl) monophenyl phosphate 8.6 having dispersed therein glycerol 3.3, ferric ammonium sulfate 3.2 and water 9

a prototype NCR Paper

- 1 14 parts, and the other emulsion being otherwise similar but contg. gallic acid 0.4 parts instead of the ferric ammonium sulfate, both dispersed phases having a particle size of less than 0.1 mm. diam. U. S. 2,299,004 relates to a manifolding paper with a coating formed from an emulsion having a continuous phase which is a soln. of gum dammar 60 in toluene 60 parts, used with one-third its quantity of a discontinuous phase formed of glycerol 80, polyethylene glycol 14 and C black or other pigment 0.1 parts, emulsified so that the discontinuous phase is of a particle size invisible to the unaided eye.

EXHIBIT

Papagorge 26
4/29/98 TSA

MAE 059918

NCR Paper Literature Survey

A 39,
160
1945

Coating for marking paper. Barrett K. Green (to National Cash Register Co.). U.S. 2,374,862, May 17, 1945. A paper sheet is coated with a rupturable solid continuous film (I) having within it 2 types of oil inclusions (II) (cf. C.A. 37, 1839^a) which are chemically sepd. from each other but when the coating is ruptured by a stylus or printing type, a localized marking is produced, by chem. reaction, on an undersheet. Formulas for the prepn. of I and II are given. F. E. B.
Color coated noncarbon. Harry C. Fisher (to Con-

A 41,
7416a
1947

3,3-Bis(4-dimethylaminophenyl)-6-dimethylamino-phthalide. Clyde S. Adams (to National Cash Register Co.). U.S. 2,417,897, Mar. 25, 1947. 3,3-Bis(4-dimethylaminophenyl)-6-dimethylaminophthalide (I) is nearly colorless under normal conditions but intense blue when in adsorption contact with highly polar substances such as clay, SiO_2 , and $MgCO_3$, or when in contact with weak acids. Paper-coating compds. contg. I and including clay or other dry solid polar filler respond to marking pressures by producing a color. To 70 g. m-H₂NC₆H₄CO₂H, 225 g. MeI, and 700 ml. 60% MeOH was added 105 g. 85% KOH in 60% MeOH portionwise. The lower MeI layer disappeared after 3-5 days. The MeOH was distd. off and 75 ml. concd. HCl added. On standing and cooling, white crystals of m-OC₆H₄NMe₂O.H₂O (II) sepd. II was slowly heated

CVL dye -

Preparation of dye

up to 235° to give m-Me₂NC₆H₄CO₂Me (III), a waxlike solid. Sapon. of 64 g. III was effected in 5 min. by boiling with 200 ml. 18% HCl to give m-Me₂NC₆H₄CO₂H (IV). A mixt. of 18 g. IV and 27 g. (4-Me₂NC₆H₄)₂CHOH was added slowly with stirring to 212 g. 90% H₂SO₄ at 0°. After standing several hrs. at room temp. the mixt. was poured over ice, neutralized partially by NaOH, and then completely by Na₂CO₃ and NaOAc in turn. The solid products were sepd., redissolved in dil. HCl, the soln. neutralized by NaOH, boiled, and filtered, and acidified with HOAc. There sepd. 2-[4,4'-bis(dimethylamino)-benzhydryl]-5-(dimethylamino)benzoic acid (V), gray crystals from C₆H₆, m. 199-200°. To a soln. of 112 g. V in 121.009 N HCl was added 72 g. 90% PbO₂ slowly with stirring at 0°. The mixt. was treated with Na₂SO₃ and then with HCl, filtered, and made alk. with NaOH. I sepd., crysd. from 95% alc., white needles, m. 189°. Richard G. Kadesch

A 43,
3213c
1948

7902.
3,3-Bis(4-dimethylaminophenyl)-6-dimethylamino-phthalide. Clyde S. Adams (to The National Cash Register Co.). U.S. reissue 2,302,4, Aug. 17, 1948. A reissue of U.S. 2,417,897 (C.A. 41, 7416a). M. L. H.

Residue of above

MAE 059919

CA-43,
252c
1949

3,3-Bis(4-dimethylamino-3-methylphenyl)phthalide.
Clyde S. Adams (to National Cash Register Co.). U.S. 2,443,092, June 8, 1948. 3,3-Bis(4-dimethylamino-3-methylphenyl)phthalide is prepared by heating at 140° o-MeC₆H₄NMe₂, 68, phthalic anhydride 37, and fused anhyd. ZnCl₂ 70 g., pouring the crude product into dil. HCl, freeing it as an oil by adding NaOH soln., and steam-distg. to remove unreacted toluidine. The residual oil solidifies on cooling and crystallizes from EtOH in white crystals, m. 180°, bluish green when adsorbed on kaolin. The solid mixed with a binder and kaolin shows a color when marked with a stylus and may be used as a record-material coating. Richard H. Wiley.

Dye similar to CVL
Preparation

CA-44,
1140c
1950

Secondary amines of 3,3-bis(p-aminophenyl)phthalide.
Clyde S. Adams (to National Cash Register Co.). U.S. 2,474,084, June 21, 1949. 3,3-Dichlorophthalide 20, CO(NMe₂)₂ 23, and AlCl₃ 50 parts, are heated in PhNO₂ (1) 1 hr. at 100°, then poured into H₂O at 0°; steam, distn. of 1, then extrn. of the dried residue with Et₂O, gives the intermediate condensation product, which is hydrolyzed with HCl (d. 1.18) 6 hrs. at 140-60°. Pptn. with NH₄OH and recrystn. from EtOH gives 3,3-bis(p-methylamino-phenyl)phthalide. The Et analog was made similarly. These colorless compds., intimately mixed with "polar substances" (clay, silicates, carbonates, ZnS, CaF₂, etc.) turn a strong blue and hence are used in making pressure-sensitive record material. Arnold M. Collins

Dye similar to CVL
Preparation

CA-44
8109c
1950

Pressure-sensitive record material. Barrett K. Green (to National Cash Register Co.). U.S. 2,505,470, Apr. 25, 1950. The application of pressure upon paper coated with eosin (I), kaolin (II), 3,3-bis(p-dimethylamino-phenyl)-6-dimethylaminophthalide (III), and tetramethylbenzidine (IV), produces at the points of application a dark-blue mark that does not become discolored or stained owing to the effects of light, atm. conditions, or aging. To one-half of a soln. contg. 1.20 and water 139, with which extn. 28% NH₄OH (V) 8 g. has been mixed at 60°, a slurry is added of II 200 and water 200 g., and 0.3%

Several Patents
several dyes
Kaolin
No solvent for dye listed

Na metaphosphate by wt. as dispersing agent. The other half of the I soln. is mixed with V 5, and a soln. of III 5.1 and IV 0.9 g. in 50 cc. of 3.7% HCl. The paper is coated with the mixt. of both these solns. and dried. The coating weighs about 0.0023 lb. per sq. ft. of surface and is 0.0005-0.001 in. thick. U.S. 2,505,471 describes the process of prep. the paper with I, II, III, and IV. The following pairs of patents use alternatives for III and IV; the first being the material and the second the process patent: U.S. 2,505,472 and U.S. 2,505,481, 3,3-bis(p-dimethylaminophenyl)phthalide; U.S. 2,505,473 and U.S. 2,505,482, 2,4-bis(p-(p-dimethylaminophenylazo)-anilino)-6-hydroxy-sym-triazine; U.S. 2,505,474 and U.S. 2,505,483, o-(p-hydroxybenzylidene)acetophenone; U.S. 2,505,475 and U.S. 2,505,484, bis(p-dimethylaminophenyl)methanol or bis(p-dimethylaminophenyl)methoxymethane; U.S. 2,505,476, and U.S. 2,505,485, 3,3-bis(p-diethylaminophenyl)phthalide or 3,3-bis(p-dipropylaminophenyl)phthalide; U.S. 2,505,477 and U.S. 2,505,486, 3,3-bis(p-methylaminophenyl)phthalide or 3,3-bis(p-ethylaminophenyl)phthalide; U.S. 2,505,478 and U.S. 2,505,487, 2-(4,4'-bis(dimethylamino)benzoydryl)-6-dimethylaminobenzoic acid; U.S. 2,505,479 and U.S. 2,505,488, 3,3-bis(4-dimethylamino-3-methylphenyl)phthalide; and U.S. 2,505,480 and U.S. 2,505,489, 4'-(p-dimethylaminophenylazo)benzanilide.

R. J. Roth

Basic patent. No solvents
Solid-Solid Rx.

MAE 059920

CA 45,
5845-
1951

Pressure-sensitive record material. Barrett K. Green and Robert W. Sandberg (to National Cash Register Co.). U.S. 2,548,364-5, Apr. 10, 1951. A pressure-sensitive record material has been developed having minute particles of two kinds of substances which are color-reactive on contact, one of the substances being in a liquid form and the other being in solid form, the particles of the two substances are arranged in proximity in profuse numbers but insulated from contact by material rupturable when pressure is applied. This pressure acts to bring the two kinds of substances together at the points of rupture to produce a distinctly colored localized mark. One of the materials in liquid form is a soln. of 3,3-bis(p-dimethylaminophenyl)-6-dimethylaminophthalide (I), chlorinated in biphenyl. One of the solid color-forming materials is a zeolite. Compds. that may be used in place of I are 3,3-bis(p-dimethylaminophenyl)phthalide, 3,3-bis(p-dimethylaminophenyl)-4,5,6,7-tetrachlorophthalide, 3,3-bis(p-dimethylaminophenyl)-6-dimethylaminophthalide, and 2,4-bis[4-(4-dimethylamino-azo)anilino]-6-hydroxy-s-triazine. U.S. 2,550,470-1-2-3, Apr. 24, 1951. This new record material, although adapted for response to stylus pressures, is particularly adapted for response to pressure applied to impact printing operations such as by the striking or pressing of type against it. It is an improvement over U.S. 2,289,683 (C.A. 37, 1860) and also U.S. 2,505,470 (C.A. 44, 8109b). The pressure-sensitive record material includes the combination of color-forming substances of two kinds, one being solid and the other, kind being liquid which is insulated from contact with the

solid kind by being profusely dispersed in minute droplets in a solid rupturable film on the exterior of which is deposited a film bearing particles of the solid kind. The solid color-forming substance is selected from a group consisting of a finely divided Na Al silicate zeolite material and such material in which the Na base exchange cations have been replaced by cations of Ni, Cu, Fe, Zn, Hg, Ba, Pb, Cd, and K. The liquid color-forming substance is an org. electron donor aromatic compd., such as crystal violet lactone in chlorinated biphenyl, having a double bond system which is converted to a more highly polarized conjugated form upon taking part in an electron-donor-acceptor adsorption chem. reaction, giving it a distinct color. Malachite green lactone or tetrachloro malachite green may also be used in the liquid-forming material. Edgar R. Wright

2 Patents

capsules

CVL in Arranger

Clay

other dyes

capsules

zeolite

Theory
Arranger

other dyes

CA 45,
5846
1951

Manifold record material. Barrett K. Green and Robert W. Sandberg (to National Cash Register Co.). U.S. 2,548,366, Apr. 10, 1951; U.S. 2,550,466-7-8-9, Apr. 24, 1951. A process has been developed for making a record material which acts both as a receiving sheet and a transferring sheet. This material is suitable for use in a stack or pile wherein an original entry on the top sheet by impact or pressure is reproduced on the top surface of each undersheet without the necessity of using interleaved transfer sheets. Various liquid colors, such as crystal violet lactone, malachite green lactone, etc., (cf. U.S. 2,548,364-5, preceding abstr.) are dissolved in an inert oily liquid medium and are held as minute droplets profusely dispersed in a solid pressure-rupturable film, preferably of gelatin. The reactant material which changes color is an electron donor aromatic org. compd. having a double bond system which is convertible to a more highly polarized conjugated form upon taking part in an electron acceptor-donor surface chem. reaction. The adsorbant material is an inorg. substance and is in fine particle form to provide a large adsorbant surface area. Cf. C.A. 39, 8160. Edgar R. Wright

Arrangement of Paper

CVL

Gelatin Capsules

Dye Theory

Adsorbant inorganic

MAE 059921

CA-47,
128164
1953

Transfer paper. Chester Davis and Ned A. Thacker (to National Cash Register Co.). U.S. 2,646,367, July 21, 1953. A hydrophobic transfer coating applied to a manifold sheet (I) consists of a colorless recording oil (II) containing a colorless color-reactant (III) in a wax binder (IV). Thus, a mixt. of 65% by wt. of a petroleum lubricating oil of viscosity 200 sec. Saybolt and 45% by wt. of a mixt. of 50% crystal violet lactone and 50% benzoyl leuco methylene blue is added to a mixt. of sugar-cane wax 44.4, refined paraffin wax 44.4, and Montan wax 11.2%. I is coated with the thoroughly stirred aggregate by use of 6 lb. of coating per 30 x 48 in. ream and used with an under-sheet sensitized with acidlike inorg. materials (cf. C.A. 45, 6845d). Other II are chlorinated or alkylated biphenyls having equiv. viscosity, hydrocarbon terphenyls, dioctyl phthalate, or chlorinated paraffins. IV may be any microcryst. wax m. 170-200°F., a fatty acid amide, a synthetic wax such as hydrogenated castor oil, or a montanic acid ester.

Not an encapsulation pro
CVL dye in Lub oil
BLMB dye
Waxes of different kinds
Clay etc
Atroclor
Alkylated Diphenyl
HD-40 etc
DOP
Chlorinated Paraffins

CA 47,
13100
1953

Pressure-sensitive record material. Barrett K. Green (to National Cash Register Co.). U.S. 2,618,573, Nov. 18, 1952. A new material, which is an improvement over U.S. patent 2,518,304-5 (C.A. 45, 6845d), comprises a mixt. of tascin 8.85, kaolin 88.60, 2,3-bis(p-dimethylamino-phenyl)-6-dimethylaminophthalide 2.25, and tetramethylbenzidine 0.40%. The kaolin, which is in fine-particle form, provides a large absorbent surface area. The org. material is an electron-donor aromatic compd. having a double-bond system which is convertible to a more highly polarized conjugated form upon taking part in an electron acceptor-donor solid-surface chem. reaction. The inorg. reactant is a material which is an electron acceptor. Both materials are in fine-particle form in order to furnish a large reactant surface area per unit area of record material. A mark of distinctive color is produced by the chem. reaction of color-forming reactants in the absence of any liquid ionizing medium and only at the points of application of localized pressure. The distinctive color produced does not become discolored or stained because of the effect of light, atm. conditions, or aging. Edgar R. Wright

CVL dye + Tetramethyl benzid
clay
React dye; no ionizing ads

CA 48,
30430
1954

Colorless inks. David W. Steinhardt (to National Cash Register Co.). U.S. 2,654,673, Oct. 6, 1953. Two different color markings from a single ink may be produced on paper by varying the clay with which it has been coated or compounded. The ink consists of a toluene soln. of a chlorinated biphenyl in which have been dissolved 3,6-bis(diethylamino)-p-(p-nitroanilino)-p-xanthene-2-benzole acid lactam (I) and a compd. selected from the group consisting of 3,3-bis(p-dimethylaminophenyl)phthalide and 3,3-bis(p-diethylaminophenyl)phthalide (II). Ca silicate will produce color only with I. Attapulpa clay will develop color with both I and II to produce a color blend.

Atroclor in toluene
Dye
"Clays" with
Selection Activity

CA-49,
66242
1955

Paper sensitized with metallic silicates. David W. Steinhardt (to National Cash Register Co.). U.S. 2,702,765, Feb. 22, 1955. Paper is impregnated with Mg silicate particles and a latex binder by passing it through a bath of Na silicate and a butadiene-styrene copolymer and then through MgSO₄ soln. Color-reactants, e.g. colorless crystal violet lactone, applied in printing or writing operations, make distinctive marks thereon. Wills I. Guss

"Clays" = Mg silicate
Containing
CVL etc
Not encapsulation but
Direct printing

SPB
Lester
Hansen

MAE 059922

CA 50,
59512
1957

Pressure-responsive record materials. Barrett K. Green and Lowell Schleicher (to National Cash Register Co.). U.S. 2,730,457, Jan. 10, 1956. Pressure-sensitive record materials on which marks of distinctive color are made by application of localized pressure are prep'd. by application on a base web (paper, metal, wood, plastic, or glass) of a solid-particle reactant and an oily water-immiscible liquid,

the two substances normally being prevented from reacting by being contained in pressure-rupturable microscopic capsules of film-forming hydrophilic colloids. The solid adsorbent material is preferably attapulgite and the adsorbate may be crystal violet lactone or malachite green lactone (I) and a colorless comp'd. which oxidizes to form a color, preferably leuco-methylene blue (II). The preferred encapsulating material is gelatin. A coating of starch and attapulgite is first applied to the paper and allowed to dry. Into an emulsion of gum arabic and water, a soln. of I and II in trichlorobiphenyl is introduced and the gelatin soln. is then mixed in. By addn. of aq. HCHO and adjustment of temp. and pH, the emulsion is gelled and coated over the attapulgite by rollers, sprays, or brushes, without a binder. Cf. preceding abstr. Blanche B. White

Capsules - Gelatin

clay
CVL etc
BLMB
Anochrome

CA 50,
16814
1957

Crystal violet lactone. Nathan N. Crouse (to Sterling Drug Inc.). U.S. 2,742,483, Apr. 17, 1956. Crystal violet lactone (I) was prep'd. in improved yield and purity by oxidation of 2-[4,4'-bis(dimethylamino)benzhydrol]-8-dimethylaminobenzoic acid (II) (19.8 g.) stirred with 200 ml. lower alkylated benzenes. II (19.8 g.) stirred with 200 ml. H₂O and 100 ml. C₆H₆, 6 g. KMnO₄ in 200 ml. H₂O added in 1 hr. at 25-30°, 6 g. BaCl₂·2H₂O added, 6 g. activated C and 6 g. diatomaceous earth added after 5 min., the mixt.

Sterling Drug's - substance
Hilton - Davis in the support
for NCR
CVL Mfg Patent

stirred 20 min., filtered, the solid washed with 100 ml. C₆H₆, the C₆H₆ layer sup'd. from the combined filtrate and washings and conc'd. to 75 ml., 150 ml. aliphatic hydrocarbon (Varnolene, b. 305-405°F.) (III) added slowly with stirring, and the solid filtered off, washed with 50 ml. III, and dried *in vacuo* at 65° gave 13.0 g. I, m. 170-80°. PbO₂ was also used as oxidizing agent. Willis I. Guss

CA 51,
109552
1957

Desensitized clay-coated record sheets. Marjorie J. Cornack and Ned A. Thacker (to National Cash Register Co.). U.S. 2,777,780, Jan. 15, 1957. Parts of the receiving sheet of a manifold system are desensitized and rendered inconspicuous by application of highly polar, colorless, non-volatile org. compds. A typical desensitizing compn. is as follows: 60 parts oil tint base vehicle composed of a mixt. of linseed oil varnish and resinous binders, 25 parts 2,4,4-trimethyl-2-oxazoline, and 10 parts dodecyltrimethylammonium chloride. This can be applied to the record material alone or dissolved in a volatile solvent. Cf. U.S. 2,548,806 (CA. 43, 68465). G. J. C. Potter

Desensitization of Clay b.
Polar compounds.
(Related to print fold maybe?)

CA 51,
175412
1957

Naphthoylleucomethylene blue. Clyde R. Adams, Marjorie J. Cornack, and Mary L. Frazier (to National Cash Register Co.). U.S. 2,783,227, Feb. 20, 1957. Naphthoyl deriva. of leucomethylene blue provide more light-stable color reactants for use in manifold sheets than the benzoyl compds. The Zn double salt of leucomethylene blue 0.0705 and pyridine 1.25 is treated with stirring with 2-naphthoyl chloride (I) 0.131 moles, the mixt. is refluxed for 1 hr., poured into cold water 1 l., and stirred for 30 min., the brown semi-solid ppt. is washed with water, stirred with NaOH 17.6 g. in water 500 g. for 30 min., and the alk. soln. is decanted. The semisolid residue is washed with water, and ext'd. with hot benzene 2.2 l.; the dark benzene soln. is treated with Fuller's earth, filtered, and the amber filtrate is conc'd. to 400 ml. and ppt'd. with petr. ether to give light-yellow, cryst. 10-(2-naphthoyl)-3,7-bis(dimethylamino)phenothiazine. 1-Naphthoyl chloride in place of I gave the 1-naphthoyl deriv., also light-yellow crystals. The compds. will turn to a blue-green color when in prolonged contact with attapulgite or halloysite. B. A. W.

Search for better
leuco dyes

MAE 059924

CA-57,
17541-2
1957

Nitro derivatives of benzoylleucomethylene blue. Clyde S. Adams, Marjorie J. Cormack, and Mary L. Frazier (to National Cash Register Co.). U.S. 2,783,228, Feb. 26, 1957. The NO_2 derivs. of 10-benzoyl-3,7-bis(dimethylamino)phenothiazine provide more light-stable color (reagents for use in transfer coatings on the rear surfaces of manifold sheets) than the unnitrated compd. Thus, the ZnCl_2 double salt of leucomethylene blue (I) 100 was mixed with pyridine 225 and pulverized $p\text{-O}_2\text{NC}_6\text{H}_4\text{COCl}$ 75 parts.

The mixt. was refluxed for 2 hrs. with stirring, poured into 1000 parts water, and stirred for 30 min. The dark blue-green ppt. was filtered off, washed with water, and stirred for 30 min. with 40 parts NaOH in 1000 parts water. The filtered ppt. was extd. with 3000 parts hot $\text{C}_{12}\text{H}_{22}$. The resulting dark soln. was treated with Fuller's earth, filtered, and the orange-red filtrate concd. to 800 ml. and dild. with petr. ether to give the $p\text{-O}_2\text{NC}_6\text{H}_4\text{CO}$ deriv. (II) of I, light orange-yellow. The $m\text{-O}_2\text{NC}_6\text{H}_4\text{CO}$ deriv. of II was similarly prepd. The I derivs. become intensely blue-green upon contact with attapulgite or halloysite. E. A. W.

More search for
Tzith leuco dyes

Preparation

CA-51,
15842-2
1957

Microscopic capsules containing oil. Barrett K. Green and Lowell Schleicher (to National Cash Register Co.). U.S. 2,800,457, July 23, 1957. Oil-contg. microscopic capsules of complex hydrophilic colloid material can be made by coacervation. All oil is emulsified in an aq. sol of a colloid, and this emulsion is mixed with an aq. sol of another colloid, or, the 2 sols may be made and mixed and the oil emulsified in them. The resulting material can be used for coatings or films, protection of oils against deterioration, confinement of oil odors, prevention of the reaction of oils with other substances during handling or storage, and the confinement of medicinal oils to prevent their being tasted. B. I. Ferber

Encapsulation of oils

Misc. uses:
Coatings
Odor & taste
protection

CA-54
15842-2
1957

Microscopic capsules containing oil. Barrett K. Green (to National Cash Register Co.). U.S. 2,800,458, July 23, 1957. Oil-contg. microscopic capsules of gellable hydrophilic colloid material (I) can be made by emulsifying an oil with an aq. sol of I and then adding an aq. coacervate salt, such as $(\text{NH}_4)_2\text{SO}_4$ or Na_2SO_4 soln. to bring the emulsion into coacervate region as shown by clouding. The procedure is performed at a temp. above the gelation point of I, and I deposits around each oil droplet by coacervate forces, encapsulating each droplet. I is finally gelled by cooling. B. I. Ferber

Encapsulation

Preparation

CA 53
1777-2
1957

Printing fluid containing a red dye and crystal violet lactone. Barrett K. Green (to National Cash Register Co.). U.S. 2,850,395, Sept. 2, 1958. A printing fluid is prepd. from Sudan III 3, crystal violet lactone (2,2-bis(p-dimethylamino)phenyl)-O-dimethylarsinophthalide 8, and chlorinated biphenyl (48% Cl content) 94 parts by wt. This is printed in a suitable manner onto a paper sensitized with an adsorbent material (1), prepd. by 20% by wt. dispersing starch in water at 200°F. for 15 min. and then cooling to room temp. A 25% attapulgite dispersion in water (4 parts) is then added to 1 part by wt. 1, and a 0.0005-inch-thick coating is applied to the paper base and dried. On the sensitized material, the printed image is dark purple in color and is formed immediately. On unsensitized material, the image remains red. V. L. Marquart

Inks.

CVL
Red dye
Aroclor

Clay
Preparation

(How does this differ from
U.S. 2,714, 074 shown
earlier?)

MAE 059925

CA 55,
14511
(1960)

Pressure-sensitive transfer record sheet. Henry S. Pesa and Lowell Schleicher (to National Cash Register Co.). U.S. 2,932,582, Apr. 12, 1960. A supporting sheet of tissue paper is coated with microscopic pressure-rupturable capsules contg. an evaporable solvent, such as dichlorobenzene (D), CCl_4 , dichloroethane, toluene, or xylene. This coated sheet is then top-coated with a solvent soln. of chlorinated rubber (Parlon) contg. an oil-sol. dye. When used as a transfer sheet, an impression of a typewriter or of a writing instrument causes the capsules to rupture, releasing the solvent which dissolves the dye on the surface coating. This transfers dye to the receiving sheet. The capsules are prepd. at 50° by emulsifying 80 g. I in 20 g. gum arabic dissolved in 180 g. H_2O . A 2nd sol. is prepd. from 20 g. pork gelatin (isoelec. point pH 8) and 160 g. H_2O . These dispersions are mixed together, and H_2O is added dropwise until coacervation of the polymer is initiated and the desired amt. of material is deposited around the I droplets. When cool, the capsules are hardened by treating the dispersion with 20 g. of 37% HCHO at pH 9-11 for a min. of 10 min. Sufficient H_2O is then added to permit coating on the sup-

porting web of paper to a thickness of 0.0006 in. The 2nd color-contg. coating is then applied and dried.

V. L. Marquart

Capsules containing
Volatile Solvents
DEB, Toluene, Xylene
 CCl_4 , dichloroethylene
Dye in Parlon (C.Rub.)
Prep.

HCHO Hardening

CA
Vol 55
109194
(1961)

Oil-containing capsules. Carl Brynko (to National Cash Register Co.). U.S. 2,969,330, Jan. 24, 1961. Oil-contg. capsules are made by first dissolving at least one polymerizable monomer in an oil or H_2O -immiscible liquid in which the polymerized monomer is insol. The soln. is then dispersed as droplets in a polar liquid where the monomer is catalytically polymerized. The polymeric product, now insol., moves to the oil-polar liquid interfaces and forms a solid wall around each oil droplet. This results in discrete, pressure-rupturable, substantially spherical, oil-contg. capsules. Thus, a polar liquid is first prepd. by dissolving 5 g. gum arabic (emulsifying agent) in 500 cc. H_2O . Into this is emulsified a soln. previously heated to 55° for 15-20 min. to initiate polymerization contg. dichlorobiphenyl 78, a 10:1 soln. of styrene monomer and divinylbenzene 20, crystal violet lactone 2%, by wt., and 35 mg. Bz_2O_2 catalyst. The polymerization is carried out at 55° for 4 hrs. with agitation. The product can be directly coated on to paper leaving a coating of the capsules. After drying, the capsules can be ruptured by pressure to release the color which then acts as a printing ink. In place of the dichlorobiphenyl, a dispersed solid, such as C black, may be used. The oils have dissolved therein dyes, inks, perfumes, adhesives, medicines, or chem. reagents which need phys. protection against deterioration, evapn., and handling pressures. Cf. following abstr.

Sidney Braverman

Encapsulation by
Polymerization etc
styrene etc

Aroclor (or C black?)

C.V.L.

Uses: ink, dye, perfume
adhesives, medicines,
chem. Reagents.

CA 55
109194
(1961)

Dual-walled, oil-containing capsules. Carl Brynko and Joseph A. Scarpelli (to National Cash Register Co.). U.S. 2,969,331, Jan. 24, 1961. Oils are encapsulated by 2 layers of solid material. The inner layer is a polymerized monomer and the outer is a hydrophilic colloid deposited by coacervate forces from a polar solvent in which the colloid has been dispersed. Thus, an internal oil-monomer phase is prepd. by dissolving styrene monomer 8, divinylbenzene monomer 2, benzoyl leuco methylene blue 4, crystal violet lactone 6, and Bz_2O_2 catalyst 0.03 in dichlorobiphenyl 140 g. The phase is then heated to 85° for 8 min. to initiate polymerization. An external phase is prepd. at 55° by dissolving 20 g. gum arabic and 0.26 g. $\text{K}_2\text{S}_2\text{O}_8$ in 1220 g. H_2O and then adjusting the pH to 0.5. Into this phase is mixed 100 g. of the internal phase emulsified in 182 g. of an aq. sol. contg. 11% by wt. gelatin. The system is mixed at 55° while the pH is slowly adjusted to 4.6. This induces coacervation, the internal phase droplets acting as seeding points to form the liquid colloid outer capsule walls. The system is then chilled to 15° to cause gelation of the colloid material around the polymer walls of each droplet forming the dual-walled capsules. The colloid walls around the capsules can be hardened by the addn. of 3.8 cc. of a soln. of 37% HCHO in H_2O and 16 g. of a 5% aq. soln. of a maleic anhydride copolymer, preferably before chilling. In another example, xeroline is encapsulated with the monomer polymerized in the emulsion mixt. In like fashion pigments, dyes, fuels, and reagents may be encapsulated. Cf. preceding abstr.

Sidney Braverman

Encapsulation by
Polymerization
Styrene etc

C.V.L.

HCHO Hardening
MA-copolymer.
4 g.
Pigments, dye, fuels,
reagents.

MAE 059926

CA-58,
104182
(1963)

Microscopic gelatin capsules. William H. Anthony (to National Cash Register Co.). U.S. 3,041,288 (Cl. 252-316), June 26, 1962, Appl. Dec. 22, 1958; 1 p. The walls of the capsules consist of high-mol.-wt. gelatin. The capsules contain a single drop of 2 parts of trichlorobiphenyl and 1 part of light petroleum distillate (I). Gelatin provides a strong wall around the above drop, and thus eliminates the tendency of the capsules to aggregate when dispersed in a liquid. High-mol.-wt. gelatin is obtained by fractionation. Thus, pigskin gelatin with an isoelec. point at pH 8 has pH 4.6-5 in hot H₂O. The pH is adjusted to 7 with 20% NaOH. Upon adding EtOH, the higher-mol.-wt. gelatin seps. at the bottom. This is dried and powdered, and 20.1 g. is dissolved in 150 g. hot distd. H₂O, and emulsified with 72.6 g. of 1 to drops 1-3 μ in diam. The emulsion (63 g.) is dispersed in 1725 g. of distd. H₂O at 50° and the pH adjusted to 7; 5.8 g. of gum arabic in 200 ml. H₂O, also of pH 7, is added, the mixt. is stirred and cooled to 8°, and acidified to pH 4.3 with 10% AcOH within 30 min. The product is hardened and strengthened by treating with 195 ml. of 37% formalin. After 230 min., the pH is raised to 10.5. This method is useful for encapsulation of coloring material. The capsules can be incorporated in papermaking. Cl. CA 51, 15842d. S. K. Lim.

Encapsulation
Gelatin (high Mol wt.
Fractionation)
Arctox +
Petroleum distillate
(for thin Dispersion
MFO?)
HCHO Hardening
Dye

CA 60,
43114
(1964)

Pressure-sensitive and transfer sheet material. Theodore Materson (to National Cash Register Co.). U.S. 3,104,980 (Cl. 117-30.4), Sept. 24, 1963, Appl. Oct. 3, 1960; 7 pp. An improved pressure-sensitive material is prepd. by coating a porous web, e.g. paper, with a dispersion of a material, colored or potentially colored, and laying down over this a polymer film which has a controlled permeability to the disperse phase. By the application of pressure to the noncoated side of the web, with a stylus, etc., the dispersion breaks and the colored material is liberated and penetrates into the porous web and through the permeable film where it can be made to mark another sheet in contact. One suitable dispersion (I) conts. gelatin-encapsulated dyes was prepd. by the addn. of emulsion (II) 377.25 to a mixt. contg. 11% by wt. aq. gum arabic soln., 93.25, 2% by wt. polyethylene-maleic anhydride (sp. viscosity 0.6 as 1% soln. in HCONMe₂) soln., adjusted to pH 9, 21; and 2% by wt. similar resin (sp. viscosity 1.0) soln., adjusted to pH 9, 21; and H₂O 1240 parts. II was made by dispersing soln. (III) 129 parts in H₂O 155 parts and gelatin soln. (11%) 93.25 parts. III consisted of perchloroethylene 193, high flash naphtha 64.5, 1,4-bis-(butylamino)anthraquinone dye 7.7 and Oil Brown DN (azo dye) 6.1 parts. Dispersion I was treated in a colloid mill (10-15 μ), the pH was adjusted to 4.5 with AcOH (14%), and the gelatin hardened by the addn. of 5.2 parts glutaraldehyde. This dispersion was coated onto paper and over-coated with a polymer copolymer (Lytrol 820) in 300 parts water. The pH was adjusted to 8 with 20% LiOH and 16 parts of polyethylene glycol monolaurate 400 added. B. H. Wheeler

Encapsulation of dye
Gum arabic
PE/MA

Perchloroethylene +
High Flash naphtha + dye
Hardener
Glutaraldehyde

CA 60,
89182
(1964)

Encapsulation. Carl Brynko and Joseph A. Bakan (to National Cash Register Co.). U.S. 3,116,206 (Cl. 167-83), Dec. 31, 1963, Appl. Dec. 22, 1961; 8 pp. Minute capsules enclosing a water-immiscible core material can be made by dispersing a water-immiscible material in an aq. alk. soln. of zein or denatured zein and by lowering the pH of the system with continued agitation to below 5.6. The wall material is made water-insol. by treatment of the capsules with cross-linking agents for zein, e.g. with aldehydes. H. C. Silberman

Encapsulation
Zein
Hardening

MAE 059927

CA 63,
502 a
(1965)

- a Color-forming capsules for recording sheets. Bruce W. Brockett (to National Cash Register Co.), U.S. 3,179,600 (Cl. 252-183.3), April 20, 1966, Appl. March 10, 1964; 6 pp. The capsules consist of a rigid wall of film-forming polymer surrounding and locking up ≥ 1 droplets of H_2O -immiscible material comprising or containing ≥ 1 reactants, and having a coating associated with and adherent to the outside of the capsule wall, preferably consisting of ≥ 1 colloidal, solid, discrete particles of acid-like materials reactive with the liquid-carried reactant material but sepd. from phys. contact with it by the capsule wall until ruptured. Thus, the H_2O -immiscible liquid capsule-nucleus material is made from 152 g. trichlorodiphenyl and 1.6 g. low-viscosity Et cellulose heated to 220°F., cooled to 200°F., and then 6 g. crystal violet lactone and 3 g. benzoyl leuco methylene blue are dissolved in the mixt. This is the internal phase of the capsule. Then, a soln. of 40 g. pigskin gelatin at pH 8 is formed in 320 g. H_2O . To this is added 25 g. of a 2% by wt. aq. soln. of polyethylene-maleic anhydride and adjusted to 140°F. The internal phase is poured into this aq. mixt. and the system adjusted to pH 7. The internal phase is emulsified into the aq. medium and contents until the drop size of the internal phase liquid is of the order of 1-2 μ . The pH is then raised to 9 and 26 g. gum arabic dissolved in 212 g. of H_2O is added, keeping the system adjusted to pH 9, heated, and in const. agitation. Then, 1740 g. H_2O , holding the system at 140°, is added and the condition of the system is gradually changed to formation of the complex coacervate entities by lowering the pH over 10 min. to 4.6 by addn. of aq. AcOH. With continued agitation, the system is rapidly cooling in 0.5 hr. to 65°F. to cause setting to a firm condition and then agitated at 65°F. for 0.5 hr. Then there is added 230 g. of an aq. dispersion of colloidal SiO_2 (35% solids; particle size 10-22 μ). During 10 min. of agitation, the particles of SiO_2 deposit on the outside of each of the solidified walls of the capsules as discrete entities. The capsules are then hardened by addn. of 20 g. of a 25% by wt. aq. glutaraldehyde soln., hardening being completed by agitation for several hrs. John H. Dittmar
- b

Encapsulation
Gelatin

(Solid encapsulated Lin)

Arabin +
Et Cellulose
CVC

PE/MA

SiO₂ on outside of capsule

CA 63
6362 a
(1965)

Production of microscopic capsules. Carl Brynko and Joseph A. Scarpelli (to National Cash Register Co.), U.S. 3,190,837 (Cl. 252-316), June 22, 1966, Appl. Dec. 31, 1968; 6 pp. Small particles of a water-immiscible liquid or water-insol. solid are

encapsulated by the coacervate deposition of a hydrophilic colloid, the latter coagulated by acid, followed by a 2nd deposition of a similar film-former. Examples are gelatin (I) (isoelectric pH of about 8), gum arabic (II), and a polyethylene-maleic anhydride copolymer (III). The solidified film is hardened with cross-linking agents such as HCHO or glutaraldehyde (IV). I (160 g. of an 11 wt. % soln.) was added to 1600 ml. H_2O at 35° with stirring, which was continued during the addn. of the other materials. The pH was adjusted to 9; 160 g. of an 11 wt. %, aq. soln. of II was added, followed by 40 g. of a 2% by wt., aq. soln. of III (mol. wt. 1000-2000, "DX 843-11"). n-Decane was added dropwise as the core material under the surface of the moving liquid by a buret so that the drops were sheared off and carried away by the rotation of the dispersion. The size of the drops was regulated by the buret stopcock and the stirring speed. Then the pH was lowered to 4.8 with 10% HOAc. Remnants of the material not deposited on the liquid droplets were returned to the original state by raising the pH to 6.8 with 10% NaOH. Then 40 g. of a 2 wt. % aq. soln. of III (mol. wt. 60,000 to 70,000, "DX 843-31") was added and the pH was lowered to 5 with 10% HOAc. The temp. was lowered to 13° and 0.5 ml. of a 25 wt. % aq. soln. of IV per g. of I was added. HCHO may be used in the amt. of 0.10 ml. of a 37 wt. % aq. soln. per g. of I, in which case the pH is raised to 7-9. The capsules were recovered by filtration, centrifugation, or spray drying. The capsules may contain drugs, foods, fuels, dyes, chem. reactants, elec. and magnetic materials, perfumes, or flavoring substances. Robert F. Doerge

Encapsulation
Sil or Solid

Gelatin
Gum arabic
PE/MA
Hardened

n-Decane added & helps encapsulation.

Water -
drugs, fuels,
foods, dyes
perfume, reactants
flavor, Magnetics
electronic materials

MAE 059928

CA 67,
172181
(1966)

Encapsulating lipophilic material by coacervation. Erik H. Jensen (to National Cash Register Co.). U.S. 3,265,630 (Cl. 252-316), Aug. 9, 1966, Appl. Dec. 22, 1958; 5 pp. A soln. of an electrophoretically chargeable styrene-maleic acid copolymer (I) and a soln. of an oppositely chargeable gelable colloid (II), e.g., gelatin, albumin, or casein, are mixed in the presence of dispersed lipophilic material (III), e.g., cottonseed or soybean oil, lard, or cholesterol, at a temp. above the gelation temp. of II at a pH at which I is electrophoretically charged and II is oppositely charged and at which significant liquid-liquid phase separ. of I and II occurs. The encapsulation of III with a mantle of I and II is thereby induced. The mantle is set by cooling the mixt. below the gelation temp. of II, by mixing the encapsulated product with a highly-active carbonyl compd., or by maintaining the encapsulated product at a temp. above the gelation temp. of II until the mantle sets. For example, add with mixing 1 l. H_2SO_4 slowly to 20 kg. 25% aq. soln. of the half Na salt of I (Dulfont G-942), stir for 0.5 hr., dil. with 5 gal. deionized H_2O , stir for 0.5 hr., and centrifuge. Slurry the solids with 5 gal. deionized H_2O and centrifuge. Dry the solids in an air dryer for 120 hrs. at 35°, grind to a coarse powder, and dry in an air dryer at 35° for 40 hrs. to give an almost quant. yield of I. Disperse 20 g. I and 5 g. Sea Kem, Type 1, in 100 ml. propylene glycol. Add 600 ml. H_2O and heat to 80°. Heat 60 ml. Vitamin A oil to 80°, emulsify it into the I soln. with a homogenizer, and add 20 g. gelatin dispersed in 100 ml. H_2O at 80° dropwise. Stir at 80° for 15 min., cool to 4° during 30 min., hold at 4° for 1 hr., and add 20

ml. 37% aq. H_2CO_3 , then 10% aq. NaOH to give pH 8.0. Hold for 1 hr. at 4°, centrifuge, and resuspend the solids in 1% HCL and dry.

Ned E. Jaffa

Encapsulation

cottonseed oil,
soybean oil
cholesterol etc

Vitamin A

(Note how much more
"scientific" resuspension
sound. also they do
"show" the brownish?)

CA 67,
44953
(1967)

44953a Heat-rupturable capsules. Herman J. Eichel (to National Cash Register Co.). U.S. 3,317,433 (Cl. 252-316), May 2, 1967, Appl. Oct. 23, 1958, and Jan. 27, 1960; 4 pp. Capsules of minute size that can be ruptured by the application of heat to expose the capsule contents are described. The capsule consists essentially of a water-insol. core material surrounded by a seamless gas-pressure-rupturable solid wall of hydrophilic film-forming polymeric material, e.g. gelatin. A heat activatable material, when heated above an elevated predetd. temp., rapidly evolves an amt. of gas exerting a pressure sufficient to rupture the capsule wall abruptly, thereby releasing the confined core material. The heat activatable material may be solid or liquid, and is selected from the group consisting of p,p'-oxybis(benzene-sulfonyl hydrazide), azodicarbonamide, and dinitrosopentamethylenetetramine or from CBR_3CBR_3 , CCl_3FCCl_3 , and CCl_3FCCl_3 . The heat activatable material may be either in the capsule wall or core material or in both locations.

S. C. Cabbage

Capsules
Heat rupturable

Blowing agents etc

MAE 059929

Foreign Patents

CA 53,
18766
1958

Copying material. National Cash Register Co. Ger. 931,531, Aug. 11, 1955 (Cl. 154, 7m). Paper coated with an outer layer of wax or another pressure-sensitive material and an inner layer of dye, which does not stain adjacent paper on accidental pressure, is produced by admixt. of 1-10% cellulose fibers to the inner layer; the latter consists of a gelatin emulsion in which an oil contg. a dye is dispersed. The av. length of the fibers should exceed the av. diam. of the oil droplets. In an example, 1.6 parts crystal violet lactone and 1.6 parts benzoyl leucomethylene blue are dissolved in oily, chlorinated biphenyl (approx. 48% Cl). Then 1-10% (preferably 4%) of α -cellulose fibers (av. length 35 μ) is added to give the inner phase. For the outer phase one part animal gelatin (isoelec. point at pH 8; gelling power 276 g. in 100ml gelometer) is dispersed in water at 65° to give a sol. In 4 parts of the latter, 3 parts of the oil (plus the wt. of the fibers) are emulsified by stirring for 30 min. (diam. of droplets 2.6-3.5 μ). The mixt. is applied while hot onto the paper to give a layer of 0.025 mm. when dry. J. Klar

Capacitor
CVL
BLM 75
Cellulose film
Analog
Isolation

CA 54
39548
(1960)

Copying paper. National Cash Register Co. Ger. 1,001,292, Jan. 24, 1957 (Cl. 154). The manuf. of a coating substance for paper is described which contains a clay or a zeolite in an aq. dispersion of a binding agent. A writing or pressure fluid contg. a dye former is used to obtain a visible color. In the 1st step, the aq. dispersion of a binding agent is prepd., e.g. from a casein soln., latex, Na pyrophosphate, or Na silicate. In the 2nd step, the clay or zeolite is added in the form of a dry fine powder. R. Degenkolbe

mfg. 7^{NS.R} paper coated

CA 55
25095e
(9C1)

Oil-containing microcapsules suitable for magnetic recording. National Cash Register Co. Ger. 1,082,282, May 25, 1960 (Cl. 154). Black or red Fe oxide, Fe carbonyl, Ni, Co, etc., may be used as magnetic substances. The capsules capable of being broken by pressure consist of gelatin or gum arabic and have a diam. of 8-180 μ . They comprise chlorinated biphenyl or a light petroleum fraction. The magnetic powder:oil mole ratio is 1:2; oleic acid, Zn stearate, or di-Pb phthalate may be used as dispersing or wetting agents. Thus, 100 g. trichlorobiphenyl, 30 g. black magnetic Fe oxide of 1 μ particle size, 0.1 g. Zn stearate, and 1 g. dioctyl phthalate were mixed and adjusted to pH 6.5 to give soln. (a). Then 20 g. pigskin gelatin was dissolved in 100 g. H₂O at pH 8 and kept at 55° to obtain soln. (b). Approx. 20 g. gum arabic was dissolved in 100 g. H₂O at pH 6.5 and 55° to give soln. (c). Soln. (a) was emulsified in (b) up to a drop size of 10 μ . Then soln. (c) was added at 55°. About 400 g. of this mixt. was dild. to 1 l. with H₂O of 55° (pH 4.7), and 2.7 cc. of a 37% aq. HCHO soln. was added at 0°. Ten g. of a 6% aq. soln. of the poly(vinyl Me ether) of maleic anhydride was added at <10°. Finally, the pH was adjusted to 9-10.

Not NCR Paper but
Magnetic slab.
Analog?
Lt. petroleum fraction
Diphenyl phthalate
DOP
Encapsulation

CA 59
14202e
(1963)

Pressure-sensitive copying paper. National Cash Register Co. Ger. 1,152,429 (Cl. 154), Aug. 8, 1963; U.S. Appl. May 20, 1961; 2 pp. According to U.S. 2,605,481-4 (CA 44, 81106), paper coatings contg. attapulgite or another adsorbent capable of ion exchange, such as zeolite, react with org. color formers, e.g. Crystal Violet lactone, to yield a dye impression when subjected to pressure. By using 3.8-20% Na silicate, the tendency of such coatings, in which various binders may be employed, to brittleness during storage is reduced. For example, a coating is prepd. contg. attapulgite 10, 35 or 41% Be, Na silicate soln. 10, white dextrin 100, and water 232 parts by wt. G. A. Wieschahn

NCR paper +
Na Silicate to avoid
embrittlement

MAE 059930

CA 60,
1355
1944

Small heat-breakable capsules. National Cash Register Co. -
Ger. 1,151,784 (Cl. B 01j), July 26, 1963; U.S. Appl. Oct. 23,
1958; 5 pp. A substance that yields gas on heating (1) is in-
corporated in content or shell of capsules made by dispersing a
hydrophobic substance in a hydrophilic medium which forms the
walls by emulsification. Examples for 1 were p,p'-oxybis(ben-
zenesulfonyl hydrazide) (II), azodicarboxamide, and dinitroso-
pentamethylenetetramine, decomg. at 130°. Thus, 3 parts by
wt. II, dispersed in 17 chlorinated biphenyl of sp. gr. 1.378-1.388
contg. a dissolved dye, was emulsified in 29 of a 3.6% aq. soln. of
pigskin gelatin (w/v), point at pH 8, at pH 2.9 gum arabic soln.
of 11% and pH 9 was added, the emulsion dild. to 375 cc. and
AcOH added to pH 4.6. The capsules thus formed were solidified
at 10°, hardened by adding 0.5 parts of a 25% glutaraldehyde
soln., and water removed by filtration or spray-drying. Such
capsules can be used as carriers of fuels, vitamins, perfumes, ad-
hesives, pigments, or fire-extinguishing materials.

Eduard Farber d

Blowing agent
Encapsulated

Arachon

Gelatin + gum arabic

Use:
Fuels, vitamins, adhe-
sives, pigments, fire-extinguishers

CA 60

Small capsules containing oil. National Cash Register Co.
(by Everett N. Hiestand, Erik H. Jensen, and Peter H. Meister).
Ger. 1,184,323 (Cl. B 01g), Dec. 31, 1964; U.S. Appl. Dec. 22,
1958; 22 pp. Very small capsules contg. glycerol emulsified at
80° with 100 cc. peanut oil contg. 1 g. hydrocarbons ester oil are
prepd. by mixing the emulsion with a sol. prep. from 10 g.
styrene-maleic acid polymer with a mol. wt. of 120,000 dissolved
in 500 cc. H₂O. The emulsion and sol. mixt. are added slowly to
1200 cc. of a 20% Na₂SO₄ soln. at 80° while stirring vigorously.
To aid hardening the capsules formed, 200 cc. of 38% HOAc is

added, after which the hardened capsules are filtered off and dried.
Other emulsions can be similarly encapsulated.

Peter H. Ledermann

Encapsulation

glycerol
veg. oil

styrene MA

Hardening

CA 62
10632 L
(1965)

Microscopic beads, containing lyophobic material on the in-
side, produced by complex coacervation. National Cash Regis-
ter Co. (by Erik H. Jensen). Ger. 1,183,883 (Cl. B 01g), Jan.
31, 1965; U.S. Appl. Dec. 22, 1958; 4 pp. One l. chro. pure
H₂SO₄ was slowly added under agitation to 20 kg. of a 25% aq.
soln. of a Na salt of a styrene-maleic acid copolymer. The
mixt. was stirred for 30 min., dild. with 18.9 l. H₂O, stirred for
another 80 min., and centrifuged. The solid material was dis-
persed in 18.9 l. H₂O, centrifuged, dried for 120 hrs. at 35°,
ground, and dried again. The recovery of the copolymer was
complete. The copolymer (20 g.) was mixed with 5 g. carrageen
and dispersed in 100 cc. propylene glycol. After adding 600 cc.
H₂O, the mixt. was heated to 80° and 60 cc. vitamin A oil was
emulsified in the copolymer. A soln. of 20 g. gelatin in 100 cc.
H₂O, heated to 80°, was added dropwise to the emulsion. After
being agitated for 15 min., at 80°, the mixt. was cooled within 30
min. to 4°, kept at 4° for 1 hr., 20 cc. 37% aq. HCHO added,
and the pH adjusted to 8.0 with 10% aq. NaOH. After keeping
for 1 hr. at 4°, the solid material was collected by centrifugation,
resuspended in 1% HCl, and dried. α-Oxopropionaldehyde was
also used for hardening, instead of HCHO. J. J. Thassal

Encapsulation

St/MA copolymer

Gelatin

Hardening

MAE 059931

CA 62
14931
(1965)

Very small particles containing a hydrophilic liquid emulsified in oil, produced by simple coacervation. National Cash Register Co. (by Everett N. Hiestand, Erik H. Jensen, and Peter D. Meister). Ger. 1,185,153 (Cl. B 01g), Jan. 14, 1966; U.S. Appl. Dec. 22, 1968; 3 pp. Very small particles containing a hydrophilic liquid emulsified in oil are produced by simple coacervation by means of a gelling hydrophilic colloid. Thus, 40 cc. H₂O

contg. 20 g. men is emulsified in 25 cc. corn oil contg. 0.25 g. hydrated castor oil as stabilizer. Gelatin (10 g.) is dissolved in 100 cc. H₂O at 40°. Both solns. are added slowly with agitation to 100 cc. aq. Na₂SO₄ (20% wt./vol.) at 40°. After complete phase sep., the soln. is cooled to 5° and its pH adjusted to 9.5 by 10% NaOH. H₂CO is added for hardening. After 1 hr. at 5°, the coacervate is removed by centrifugation, washed, and spray-dried at 80°. The product is a fertilizer which slowly releases N. Similarly, particles containing ascorbic acid as fertilizer mixts. and particles contg. sorbitan monioleate are produced.

Jehudah Eliassaf

CA 62
14932
(1965)

Very small particles containing a liquid emulsified in oil produced by coacervation. National Cash Register Co. (by Everett N. Hiestand, Erik H. Jensen, and Peter D. Meister). Ger. 1,185,154 (Cl. B 01g), Jan. 14, 1966; U.S. Appl. Dec. 22, 1968; 3 pp. Very small particles containing an emulsion of a hydrophilic liquid in oil are produced by adding a stabilizer to the emulsion and coacervating in the presence of an aq. soln. of a hydrophilic colloid and either a styrene-maleic acid copolymer (I), a styrene-maleic acid amide copolymer or a carbohydrate acetate phthalate. Thus, 100 g. chloral hydrate dissolved in 50 cc. H₂O is emulsified at 40° in 125 cc. mineral oil contg. 25 g. beeswax. I (76 g.) is dissolved in H₂O by heating to 70° and adding 10% aq. NaOH to bring the pH to 7-8. The copolymer soln. is dispersed in the emulsion under const. agitation. A soln. of 76 g. gelatin in 500 cc. H₂O, at pH 7 (NaOH), is added dropwise at 70°, followed by dropwise addn. of HOAc to lower the pH to 4.5. After stirring for 30 min. at 70°, the mixt. is kept below 0° for 1 hr. and 76 cc. of 37% H₂CO is added. After 1 hr. the pH is raised to 8 by addn. of NaOH. The coacervate is filtered, washed with H₂O, and freeze dried at 40°. Slow acting fertilizers, fungicides, and bactericides are produced. The method is also applied to mixts. in animal foods, in cosmetics, and drugs.

Jehudah Eliassaf

Use
Fertilizer
Encapsulated

(water in oil emulsion encapsulated)

Stabilizer

Encapsulation

(water in oil emulsion)

slowly hydrolyt

Use

Fertilizer, fungicide, bactericide

Additives for food, cosmetics

CA 62
14934
(1965)

Capsules of hydrophilic substances. National Cash Register Co. (by Everett N. Hiestand, John G. Wagner, and Edwin L. Knoechel). Ger. 1,185,155 (Cl. B 01g), Jan. 14, 1966; U.S. Appl. Dec. 22, 1968; 5 pp. Hydrophilic substances can be encapsulated by dispersion in a nonaq. liquid coating with a hydrophobic coacervate, collecting and drying, and finally coating with a hydrophilic coacervate. Thus, 80 g. Na (2,4-dichlorophenoxy)acetate (I) is suspended in 300 cc. trichloroethylene contg. 10 g. benzyl cellulose. Phase sep. is brought about by addn. of PrOH (51% vol./vol.), thereby encapsulating I. After collecting by centrifugation, washing and drying, the capsules are suspended at 40-60° in 900 cc. H₂O contg. 100 g. dissolved gelatin, from which they are pptd. with a coating of gelatin by addn. of EtOH. After cooling to 0° and sep. by centrifugation, the capsules are washed and dried. The herbicidal action of I is shown down. Cosmetics and drugs, such as ascorbic acid, pancreatin, and methylcopolamine-HBr, are similarly encapsulated.

Jehudah Eliassaf

Encapsulation
(Complex)

Hydrophilic in
Hydrophobic in
Hydrophilic again

Weed killer (Herbicide)

Active Drug

Vitamin

A-63,
3660
(1965)

Minute capsules by coacervation. National Cash Register Co. Ger. 1,186,828 (Cl. B 01g), Feb. 11, 1966; U.S. Appl. Nov. 4, 1969; 2 pp. Minute capsules enclosing water-insol. solid or liquid materials are produced by coacervation of a hydrophilic film-forming macropolymer in an aq. soln. For example, a mixt. of 20 g. pig skin gelatin (low sec. point at pH = 8.9), 980 g. water, 800 cc. of 20 wt. % (NH₄)₂SO₄ aq. soln., and 200 cc. of mince oil is prepd. The mixt. is warmed to 35-60°, stirred until liquid walled capsules are formed, which become self-supporting upon cooling to room temp.

Hakke Chon

Encapsulation

Solid or liquid (oil) (corn oil)

Stabilizer

MAE 059932

CA 23,
1009
(1965)

Preparation of very small capsules. National Cash Register Co. Ger. 1,189,050 (Cl. B 01g), March 18, 1965; U.S. Appl. Oct. 28, 1960; 2 pp. The liquid or solid to be encapsulated is dispersed in an org. solvent contg. a hydrophobic polymer such as Et cellulose, poly(Me methacrylate) or cellulose acetate. After formation of a polymer coating around the particle by heating to 70°, addn. of a petroleum distillate b. >70° promotes phase sepn. and capsule recovery. T. F. Canning

Encapsulation
Solid or liquid
Polymer in solution
Et al, PMMA, CA
Pat. Detailed description

CA 64,
10040
(1966)

Recording papers with attapulgite. National Cash Register Co. Neth. Appl. 6,505,671 (Cl. B 41m), Nov. 8, 1965; U.S. Appl. May 8, 1964; 7 pp. Attapulgite is used as oil-absorbing coating on recording papers for color formation from colorless chromogenic compds. by electron donor-acceptor reactions. The high surface activity of attapulgite renders it prone to attract atm. impurities, which reduce its color-forming capacity. By the addn. of ≥ 0.1 mole of a water-sol. salt of the bivalent metals Zn, Cd, Hg, Ca, Sr, Mg, Mn, Co, or Ni this contamination is minimized. Also the coating wt. can be reduced to 3 g./sq. m. from 7.4-8.9 g. used previously, whereby the handling of the paper is facilitated. In an example, 20 parts ZnCl₂ is dissolved in 240 parts water, 100 parts attapulgite-clay is added in a high-speed mixer, followed by 40 parts each of the latex of butyl rubber and etherified starch. G. A. Wischehahn

NC R notes
Clay - absorbs dyes.
color fades
Zn, Cd salts - salts inhibit fading

CA 64,
15273
(1966)

pressure- and heat-sensitive copy materials. National Cash Register Co. Neth. Appl. 6,505,970 (Cl. B 41m), Nov. 12, 1965; U.S. Appl. May 11, 1964; 24 pp. Image formation is achieved by chem. reaction between a liquid chromophore A enclosed in microscopic pressure-rupturable polymer capsules and a normally solid, vaporizable component in the form of powder particles. Pressure causes the capsules to release the liquid for reaction with the solid component, whereas a heat pulse brings the vaporized solid into contact with the vapor-

Vaporizing all solid

permeable capsules. Five types of reactions are suggested: (1) Lewis acid, or Bronsted acid, plus a base (p-nitrobenzene-diazonium fluoroborate) liberating BF₃ or 4,4'-isopropylidene-diphenol, resp., with a soln. of Crystal Violet lactone or benzoyl-leuconmethylen blue; (2) transfer complex formation from Lewis acid plus base (tetracyanoethylene with benzene or xylene); (3) double decompn. ((NH₄)₂S with Co naphthenate); (4) chelate formation by double decompn. (dithiooxamide with Cu oleate); and (5) oxidn.-redn. reactions (N-bromosuccinimide with benzoylleuconmethylen blue). The 2 reagents may be coated on sep. sheets, forming an assembly in contact with each other; they may be coated on the surface of a common support (paper film), or one may be distributed in the support, the other coated on its surface. For example, Crystal Violet lactone in chlorinated biphenyl-petroleum ether soln. is dispersed in aq. 1% gelatin, concentrated with gum arabic and a vinyl Me ether-maleic anhydride copolymer, the resulting capsules are hardened with a glutaraldehyde-acetic acid mixt., and 4 parts of the capsules are mixed with 1 part of a dispersion of 4,4'-isopropylidene-diphenol in aq. hydroxyethyl cellulose for coating on film to a dry wt. of 6.8-7.4 g./m². G. A. Wischehahn

BPA. (vaporizing all?)
C/L
B/L M/D

Arrows

BPA in hydroxyethyl cellulose sol

CA 64,
20002
(1966)

Pressure-sensitive copy materials. National Cash Register Co. Neth. Appl. 6,511,224 (Cl. B 41m), Feb. 28, 1966; U.S. Appl. Aug. 27, 1964; 28 pp. In previous materials in which writing pressure releases a color component by rupturing microcapsules (6-10 μ) contg. a soln. of it to form a dye in contact with a sheet carrying an attapulgite coating, the latter tends to lose its reactivity with time, apparently due to absorption of substances from the atm. This deterioration is avoided by use of a resin sol. in the solvent for the encapsulated basic color former and with acidic groups to react with the latter. A suitable combination is that of crystal violet lactone with a p-phenylphenol-HCHO resin. The polymer may be used as fine particles or as solute droplets. Capsules and polymer may be on sep. carriers or on a single sheet and may be present as layers or distributed throughout the paper or film mass. G. A. Wischehahn

NC R notes

Resin
Phenolates to avoid print-fad in clay coated paper?

100 pages. not

MAE 059933

CA 67,
53935
(1967)

33035v Recording material. National Cash Register Co. (by Helmut Schwab). Belg. 663,660 Nov. 3, 1905; U.S. Appl. May 11, 1964; 20 pp. A pressure- or heat-sensitive recording medium contains liquid droplets sepd. from a solid surrounding medium reacting with the liquid to form an image by means of a barrier layer. The solid vaporizes on heating and the vapor penetrates the barrier layer to give an image. Single-layer or multi-layer transfer systems can be used. Acid-base, redox, or metathetic systems can be used. A typical system is 4,4'-isopropylidenediphenol (solid) and 3,3-bis[*p*-(dimethylamino)phenyl]-6-(dimethylamino)phthalide (liquid) surrounded by a mixt. of gelatin, gum arabic, and maleic anhydride-poly(vinyl methyl ether) copolymer. Geoffrey W. Bradbrook

Capsules
Solid-Vaporizer

BPA 2
CVL

CA 67,
65157
(1967)

65157j Manufacture of small capsules by coating with cellulose ethers or other polymers. National Cash Register Co. Neth. Appl. 6,611,061 (Cl. B 01f), Feb. 20, 1967; U.S. Appl. Aug. 18, 1965; 18 pp. Small capsules are prepd. by coating a core material with a hydrophobic polymer by liquid-liquid phase sepn. The solvent is chosen so as to give increased soly. of the polymer with increased temp. and phase sepn. is brought about by cooling. Thus, 2 g. Et cellulose contg. 47.8-49% EtO groups and with a viscosity of 22 cp. in a 5% soln. in 80% EtOH at 25° was dissolved in 98 g. boiling cyclohexane to form a clear soln. MgI₂ (8 g.) particles (20-40 μ) were added and the mixt. cooled to room temp. with stirring. Particles uniformly coated with Et cellulose and with little tendency to aggregation were obtained. With a more dil. soln. of the Et cellulose, even better wall formation was obtained. Similar coatings were obtained by pptn. of ethyl hydroxyethyl cellulose from petroleum distillate fractions, poly(chlorotrifluoroethylene) from iso-BuCOMe, or Et cellulose from a mixt. of cyclohexane and hexane. The process can also be used for encapsulation of liquids. CWPZ

Capsules

by
Phase separation
(liquid, liquid)

Et Cellulose
MgI₂

CA 67,
95338
(1967)

85338z Improvement of the physical characteristics of coacervate membranes. National Cash Register Co. (by David E. Guttman). Fr. 1,468,602 (Cl. B 01f), Feb. 10, 1965; U.S. Appl. Dec. 22, 1958; 8 pp. Membranes contg. gelifying hydrophilic colloid (I) were made. Coacervates, with pI < 7, were cooled below the gelification point of I and treated in acidic medium with alum having the formula M'M''(S)₂·12H₂O, where M' is a univalent alkali metal (K), M'' a trivalent metal (Al, Fe, Cr), and S a sulfate or selenate. The dry product contained: a coating of I, lyophilic type solid particles, and an oil emulsion of hydrophilic liquid in oil or vice versa. Jugal K. Sharma

Capsules?

Hardening

CA 67,
109438
(1967)

109438b Microcapsules containing an emulsion of oil in a hydrophilic liquid. National Cash Register Co. (by Everett N. Heistand, Erik H. Jensen, and Peter D. Meister). Ger. 1,249,219 (Cl. B 01g), Sept. 7, 1967; U.S. Appl. Dec. 22, 1958; 8 pp. Microcapsules contg. an emulsion of oil in a hydrophilic liquid are prepd. by the complex coacervation of ≥ 2 polymers, in which I is a natural hydrophilic colloid and the other a styrene

Capsules

Styrene/MA copolymer
caffeine

gelatin

Hardening

Water

MAE 059934

(I)-maleic acid (II) copolymer, a I-maleamide copolymer, or a carbohydrate acetate phthalate. Thus, a suspension of 8 g. Me cellulose and 60 g. caffeine in 100 ml. H₂O was heated to 80° and 100 ml. paraffin oil at 80° emulsified in the aq. suspension. A I-II copolymer (75 g.) was dispersed in 1500 ml. H₂O, heated, and treated with sufficient 10% NaOH until the copolymer dissolved. The emulsion was then dispersed in the copolymer. To 500 ml. water, 75 g. gelatin was added, the dispersion heated to 80°, and adjusted to a pI of 7.0 with 10% NaOH. The mixt. consisting of the emulsion and the copolymer was then added dropwise to the gelatin soln. Immediately afterwards, the pI was adjusted to 3.9 with addn. of 20% AcOH. The product was kept for 15 min. at 80°, then cooled to 4° within 30 min. The coacervate was hardened by addn. of 70 ml. of a 37% HClO₄ soln. and adjusted to a pI of 8.0 with 10% NaOH. The hardened product was centrifuged, washed with 2% HCl, and dried in air. Other components used in similarly prepd. compus. were rotenone, cellulose acetate phthalate, fibrinogen, CM-cellulose, corn oil, peanut oil, serum albumin, urea, tragacanth, and glycerol. The process is suitable for encapsulating such items as vitamins, drugs, or cosmetics. UNP

CA 68,
60312
(1968)

60312a Capsules of gelatinous polymeric material. National Cash Register Co. Ger. 1,256,195 (Cl. B 01g), Dec. 14, 1967; U.S. Appl. Sept. 14, 1961; 3 pp. Capsules of gelatinous, polymeric, hydrophilic properties, contg. water-immiscible materials, are prepd. by coacervation, lowering of the temp., and maintaining the temp. in the region of gelatinization until the desired wall thickness is obtained. Thus, a mixt. of 1000 ml. water, 180 ml. 11% aq. gelatin (isoelec. point of 8.9), 180 ml. 11% aq. arabic gum, 40 ml. 2% aq. ethylene-maleic anhydride copolymer (I) (mol. wt. 1600, pH 9), and 40 ml. 2% I soln. (mol. 100,000, pH 9) was agitated, the temp. raised to 38° and the pH adjusted to 0.8. To the system was then added 250 ml. PhMe (II) and agitation continued until I was dispersed to a particle size of 600 μ . Coacervation units deposited themselves in a 5 μ layer on the II droplet during which time the temp. was reduced over a period of 20 min. to 25 $\pm$ 5°. The deposition of the capsule-forming material continued until a thickness of 150-200 μ was formed around each II droplet. Cooling was continued to 10° whereby the polymeric deposit gelatinized. The capsules were now finished unless crosslinking was desired. BNP

Capsules

E/MR

DM Phellist encap

CA 68,
60704
(1968)

60704w Microcapsules containing oil emulsions in hydrophilic liquids. National Cash Register Co. (by Everett N. Hiestand, Erik H. Jensen, and Peter D. Meister). Ger. 1,256,196 (Cl. B 01g), Dec. 14, 1967; U.S. Appl. Dec. 22, 1968; 3 pp. Microcapsules that contain an emulsion of an oil in a hydrophilic liq. are prepd. by complex coacervation with an aq. dispersion of a

Capsules

gelating and isoelec. colloid, with the addn. of a thickener to the hydrophilic phase(s). Thus, 15 g. Me cellulose was added to a soln. of 60 g. urea in 25 ml. H₂O. The soln. was heated to 60°, and 60 ml. peanut oil at the same temp. was emulsified. A soln. was prepd. by dispersing 40 g. gelatin and 46 g. tragacanth gum in 400 ml. H₂O at 60° and the emulsion was then dispersed in the soln. with vigorous agitation. H₂O (1200 ml.) at 60° is then added dropwise to induce phase sepn. The mixt. is then cooled to ~5° in 30 min. and held at this temp. for an addnl. 10 min. The pH is raised to 9.5 by adding 10% NaOH and 40 ml. 37% HClO is added to harden the colloid envelope. The product is then collected, washed with H₂O, and air dried at 80°. Other thickeners, e.g. tragacanth, CM-cellulose, or polyglycols, can be used in equiv. amts. instead of Me cellulose. Similarly, other hydrophilic colloids, e.g. agar, albumin, alginates, casein, and similar gelating isoelec. colloids can be used instead of gelatin. The microcapsules have an impermeable envelope and are esp. suitable for slowly releasing agricultural compds., e.g. fertilizers, plant nutrients, growth-hormones, and pesticides, or for the administration of vitamins or nutrients, as well as for cosmetic and pharmaceutical purposes. M. W. Weiss

Water
Fertilizer
Hormone
Pesticide
Vitamin
Cosmetic & pharmaceutical

CA 68,
96486
(1968)

96486r Encapsulated curable resins. National Cash Register Co. Brit. 1,104,870 (Cl. B 65d), March 6, 1968; U.S. Appl. Feb. 28, 1966; 8 pp. Minute capsules contain a curable liq. resin and have a continuous polymeric wall, with solid particles of a resin curing agent adhering to the outside of the wall, formed by phase sepn. in a soln. of the polymeric coating material. Thus, 20 g. pigskin gelatin and 20 g. gum arabic were dissolved in 1120 g. distd. H₂O at 60°. The pH was adjusted to 4.5 with 20 wt.% aq. NaOH or 14 wt.% aq. HOAc, as required. The temp. was reduced to 40° and 100 g. polysulfide resin (LP-3) (I) was slowly poured into the warm system with stirring. The system was agitated until the desired resin globule size was obtained, and cooled to 25° and then to <10°, maintaining agitation. At <10°, 10 ml. 25 wt.% aq. pentanediol was added. The mixt. was stirred 16 hrs. as the temp. was gradually increased to 25°, and filtered. The filter cake was dispersed in 8 l. anhyd. 2-methyl-2,4-pentanediol, stirred 5 min., filtered, and the residue washed with n-C₄H₁₀. The capsules were sized on 105-250 μ screens and then shaken with orthorhombic TeO₂ of av. particle size 20 μ . The excess curing agent was removed from the capsules, giving a product that cured when crushed to release the polysulfide. I that had been ground with BaCrO₄ was also used. The use of MnO₂, PbO₂, and Sb₂O₃ as curing agents and SrCrO₄, Zn chromate, and Zn yellow as corrosion inhibitors is also claimed. When used as an adhesive or binder, the capsules can be applied to laying surfaces before the actual fabrication. Polyepoxides and polyisocyanates may also be used. CCI'N

Capsules
(containing)
Polysulfide
Curing Agent
(on outside)

MAE 059935

CA 68,
49040
(1968)

99040b Microencapsulation of aqueous emulsions. National Cash Register Co. (by Everett N. Hiestand, Erik H. Jensen, and Peter D. Meister). Ger. 1,248,015 (Cl. B 01g), Aug. 24, 1967; U.S. Appl. Dec. 22, 1958; 2 pp. Microcapsules contg. an emulsion of an oil in a hydrophilic liq. (H₂O) are prep'd. by simple concentration with a gelable hydrophilic colloid and the addn. of an aq. electrolyte soln. or a H₂O-sol. liq. in which the colloid is less sol. than in H₂O. A conventional thickening agent in the hydrophilic phase is used. Suitable electrolyte soln. thickeners include acacia gum, gum tragacanth, Me cellulose, CM-cellulose, Mg Al aluminate, polyglycols, and glycerol. Thus, an oil/H₂O emulsion was prep'd. at 60° from paraffin oil 33 ml., H₂O 25 ml., Mg Al silicate 2 g., and alizarin cyan green 0.5 g. A sol contg. 12.5 g. gelatin and 125 ml. H₂O at 60° was thoroughly mixed with the emulsion, and the resulting mixt. was then slowly added to 125 ml. 20% Na₂SO₄ soln. contg. 37 g. acacia gum. The temp. of the equil. mixt. was then reduced to 5°, and 10% NaOH was added to pH 9.5. To harden the envelope, 12.5 ml. 37% HCHO was added. The product was filtered after 5 hrs., washed, and spray-dried at 80° (exhaust steam temp.). The envelope was highly impermeable, as shown by tests with acids and alkalis. The microcapsules are esp. useful for releasing active substances at a slow rate under conditions that destroy the membrane envelope.

M. W. Weiss
99041c Microencapsulation of aqueous emulsions. National Cash Register Co. (by Everett N. Hiestand, Erik H. Jensen, and Peter D. Meister). Ger. 1,248,016 (Cl. B 01g), Aug. 24, 1967; U.S. Appl. Dec. 22, 1958; 2 pp. Microcapsules contg. an emulsion of an oil phase in a hydrophilic liq., e.g. H₂O, are prep'd. by adding a thickening agent to the latter phase and conserving in the presence of a colloidal aq. dispersion of a styrene-maleic acid or styrene-maleic amide copolymer. Suitable thickeners are insol. in the oil phase and have surfactant properties, e.g. gum acacia, tragacanth, Me cellulose, CM-cellulose, Mg Al silicate, polyglycols, and glycerol. The specific amts. of thickener may be varied according to the particular system, 5-10 wt. % of the hydrophilic phase being required as a

rule. The envelope of the microcapsule is impermeable to the contents but is readily destroyed in contact with a suitable pH or enzyme. Thus, rotenone 25 and tragacanth 100 g. were dispersed in glycerol 100 ml. and heated to 100°. Corn oil 100 ml. was emulsified into the glycerol dispersion. Styrene-maleic acid copolymer 100 and galactose polysaccharide 20 g. were dispersed in H₂O 1050 ml. and heated to 80°. The emulsion and the copolymer sol. were then added as a single stream to 450 ml. of 20% (NH₄)₂SO₄ soln., preheated to 80°, and the mixt. was stirred for 20 min. It was then poured into a soln. of 50 ml. AcOH in 3000 ml. of ice water with const. stirring. The resulting encapsulated material was then sep. by centrifugation, washed with H₂O, and spray-dried at 80°. The microcapsules are esp. suitable for releasing active substances at a slow rate, e.g. agricultural fertilizers, plant-growth hormones, fungicides and nematocides, nutritive substances and vitamins in foods, cosmetic or pharmaceutical substances, insecticides, and rodenticides.

M. W. Weiss

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49041
(1968)

106151x Water-insoluble microcapsules. National Cash Register Co. Ger. 1,265,130 (Cl. B 01g), April 4, 1968; U.S. Appl. Nov. 10, 1961; 2 pp. Small, water-insol. capsules are prep'd. from an aq. sol. contg. ≥ 1 hydrophilic, film forming polymer, dispersing the material to be encapsulated in the sol, starting the coagulation, and treating the capsules while the capsule walls are still in a liq. state with hardeners or metal chelates. The complete process can be carried out at 1 temp. Thus, the pH of a mixt. consisting of 5% aq. soln. of poly(vinyl-imidazole) 100, 5% aq. soln. of gum arabic 100, and H₂O 300 ml. was adjusted to > 8.5, 25 ml. trichlorotrifluoroethane added under vigorous stirring, and the pH reduced as quickly as possible to 0.5-7.0 while stirring was continued and 5 ml. 10% aq. soln. of CrCl₃ added. The capsule walls became hard and water insol. and the capsules could be sep'd. by the usual methods. Also used in a similarly prep'd. compn. were polyethylenimine and a soln. of poly(ethylene-maleic anhydride). BNPC

Capsules

Thickener (Misc)
Gelatin
dye - pigments

Capsules

Thickener (Misc.)
Styrene / MA copolymer

Water

Capsules

Mad
water-insoluble

Cyclic etc

MAE 059936

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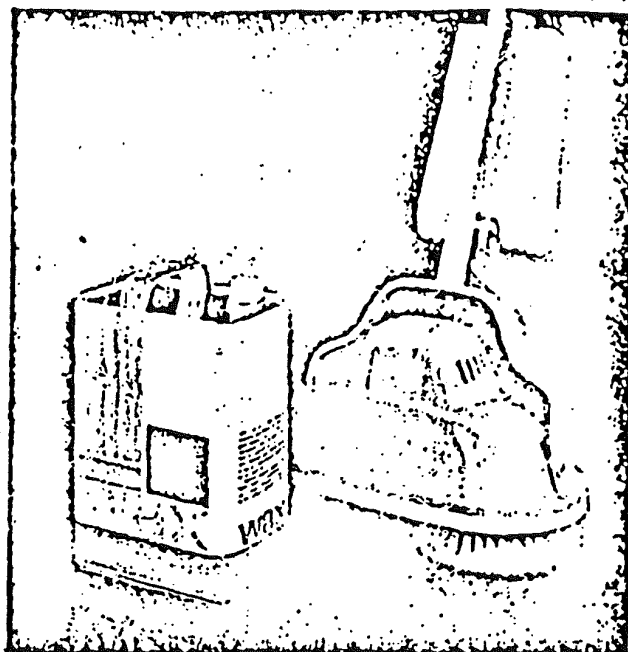
EXHIBITCupageorge 27
4/29/48 TSA

With the wax and polish
market making new demands,
formulators are looking
more closely at...

AROCLOR®

SIRUPS and RESINS

A shelf of stubborn "do-gooders"
for waxes, sealants, mastics



Compounders scratching their heads over plasticity, flow point, hardness, oxidation resistance, or volatility problems are discovering a bonanza of answers in low-cost Aroclor compounds. These various chlorinated biphenyls can fill a fair-sized laboratory shelf, and the range of physical modifications they impart is even wider. Besides their surprisingly close, predictable, and dependable modification of physical properties, compounders are getting bonuses in improvement of chemical properties. Most appealing, their cost per pound is in the cup of coffee and doughnut range.

Specialty wax formulators, polishing products makers, sealing compound producers, and mastic products compounders are finding new property improvements at their finger-tips... when they run their index finger down the list of available Aroclor compounds. Surprising, too, because the Aroclor "oils," "sirups," and "resins" have been around quite a while. And since their cost (in the 15-20¢/lb. range) is so low in relation to the functions they can perform, it could be assumed that by now virtually all floor polishes, wax coatings, caulking, and mastic sealing products would have some Aroclor liquid or resin in their recipes. But, to date, only manufacturers of top-quality investment casting waxes have adopted Aroclor compounds to give waxes high, sharp melting points and lower the cost of formulations.

Wherever polyethylene and Fischer-Tropsch waxes are bidding for the polish markets, formulators have overlooked what an Aroclor addition can do to modify the properties of these, as well as carnauba, candelilla, shellac, and micro-crystalline waxes.

The most intriguing possibility is this: manufacturers of the synthetic waxes could use the Aroclor compounds for "tailoring" their offerings; thus, with a given processed wax, they would have a wider range of properties and characteristics in the waxes they offer formulators. Recently, manufacturers of pressure sensitive adhesives have found combinations of ethylene/vinyl acetate with Aroclor give improved properties.

From a physical property standpoint alone, the Aroclor compounds appear to provide the wax product maker with a wide range of "do-good" compounds that have no peers. They all are fire-resistant and completely inert to chemicals, water, and weather. They impart these properties directly to formulations in which they are used. Most important, since they are virtually non-oxidizable and non-volatile—the properties stay. The Aroclor compounds are agreeable to handle, too. They are compatible with practically all commercial chemical solvents, waxes, resins, and asphalt, and they can be made into water emulsions. This combination of properties that makes them suitable for liquid, paste, or cake formulations, recommends their consideration in auto polishes and floor waxes, as well as furniture and shoe polishes. In addition to durability, they impart toughness, without brittleness, and either maintain or enhance gloss. Unlike other modifying additives, the Aroclor compounds offer modifying effects without imparting tackiness.

Outside the polish field, the Aroclor compounds can serve a variety of special functions in specialty waxes, wax binders, and decorating or marking-ink vehicles.

Such uses stem from their characteristic melting points and the fact that they will "burn out" without ashing. Thus, Aroclor liquids and resins provide a choice of materials: for property modification, for making dental-casting waxes, for the precision casting of aircraft parts, and for casting jewelry. They are highly useful in making powdered-metal binders, where the shaped product is fired and the binder must burn off without ashing or discoloration. A major use is for marking inks, used to decorate ceramic ware where the design and color is "fixed" in the glazing.

While Aroclor compounds have been widely adopted for improving the adhesion and chemical resistance of paints and surface coatings, their use in wax modification, asphalt sealants, and mastic has just begun. Manufacturers of wax-based products and polishes are just beginning to discover that they represent a whole shelf of "do-gooders" for wax and resin type formulations.

For further information on Aroclor compounds and their use in modifying and improving wax, asphalt, and resin-based products, request a copy of Specialty Data Report CS-18. Write to: MONSANTO, Organic Chemicals Division, Department 214, St. Louis, Missouri 63166.

MONSANTO COMPANY
ANNISTON, ALABAMA

Dear Fellow Employee,

Monsanto employees are continuously concerned about the Quality of the products and service we are providing our customers. Today, competitive products are readily available and the customer often buys on the supplier's ability to furnish dependable superior quality product and service.

This is the first in a series of quarterly Anniston Plant Newsletters that you will be receiving during 1966.

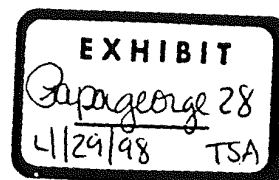
This issue highlights the Aroclor Department which has a 37 year history of continuous growth and high Quality Production. A few of the end uses of Aroclor and the employees who contribute to continued Quality Production are highlighted. The importance of Quality to our customers and therefore to our job security and growth of the Anniston Plant is illustrated.

Any comments you may have concerning Quality will be appreciated and should be forwarded to a member of the Quality Promotion Committee.

Congratulations on your record of producing superior Quality products which lead to the growth of the Anniston Plant.

W B Pope

MAE 059895



WATER_PCB-SD0000071990

AROCOLOR'S HISTORY

The first Aroclors (chlorinated biphenyls) were produced at the Anniston Plant on December 16, 1928. Biphenyl production began on February 28, 1928; Aroclor production then started as a natural result of the biphenyl raw material availability.

The number of Aroclor production employees has increased by eight times since December, 1928. Aroclor has become a "back-bone" department in the plant, providing new jobs, increased job security, and a higher standard of living to its employees throughout the department's thirty-seven year existence.

AROCOLOR USES ELECTRICAL EQUIPMENT

Monsanto Aroclors are used "as is" or in special formulations for the liquid coolant-insulation fluid in transformers and capacitors.

In transformers, they provide for power factor correction in electric utility transmission lines. In fixed power capacitors, Aroclors are the dielectric substance. A capacitor consists of two conducting metal plates separated a small distance apart by some non-conducting medium (Aroclor). The purpose of a capacitor is to store energy and release this energy a short time later. This is much the same as elevating a tank full of water. This water, being at a higher elevation than its surroundings, has a certain amount of potential energy to release, just as the capacitor has a potential to release stored voltage. These capacitors are used in home appliances such as air conditioners, furnaces, washers, and driers, in electric motors, and for ballast in fluorescent fixtures.

General Electric, Westinghouse, and other producers of electric capacitors use Monsanto Aroclors in special formulations for the dielectric substance. This helps to make their capacitors the best available in the industry.

Other important applications of both liquid and Solid Aroclors in the electrical field are in wire and cable coatings and as impregnants for cotton and asbestos based insulation. In addition, they are excellent dielectric solvents and are used to close the pores of carbon resistors and terminal electrical bushings and terminals.

Aroclors for electrical equipment use must be highly pure with only slight traces of contaminants. Two to three drops of sweat contain enough salt to reject a drum of Aroclor on chlorides. They must be chemically stable and non-corrosive to a wide variety of structural materials.

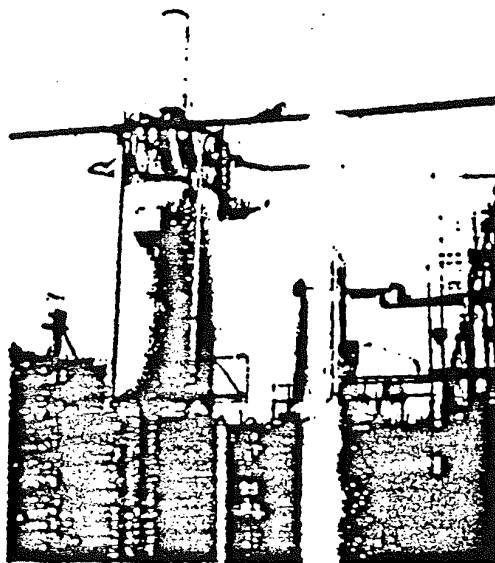
Obviously, continuous quality control must be maintained during manufacture, packaging, and shipping to assure that the customer is always satisfied with the performance of our Aroclors.

MAE 059896

Contamination must be controlled in the plant as analytical "bird dogging" may not detect traces of impurities which make the product unstable and useless. For instance, the dropping of one hard hat in a tank car could cause the failure of thousands of our customers' products. Normal power factor correcting capacitors used in air conditioners contain 10 cents worth of Aroclor. If the capacitor should explode and cause a fire, damage as high as \$20,000 could result. These capacitors are designed for 20 to 30 year life. Quality insulants, such as Anniston Aroclor, must be used to guarantee this life as there is no non-destructive procedure for pre-determining this life. This vividly illustrates the "mushrooming" damage and the major reason for such intense desire for quality products by our customers.

HEAT TRANSFER

Aroclors are outstanding for use as the heat transfer liquids in indirect heating systems. Aroclor systems can transfer closely controllable, uniform heat to chemical process vessels, food cookers, potato chip and frito fryers, drying ovens, to heat corrugating rolls, crimping presses, and calenders in materials processing, to heat treating metals such as Titanium and Vanadium, and to efficiently indirect heat distillation equipment. In fact, an Aroclor heating system is presently in use in the Anniston Plant Aroclor Department to distill Aroclors during routine production. This system is pictured below:



MAE 059897

It is interesting to note that as recently as 1941 only two methods of delivering process heat were known. Today non-pressurized, high temperature, fire resistant heat transfer systems based on Monsanto's Aroclor Liquids overcome practically all the deficiencies and drawbacks of *Direct-Firing* and *Pressurized Vapor Heating*.

Continuous development and Aroclor quality improvement has enabled Monsanto to continue to capture and add to a sizeable segment of the heat transfer market.

OTHER USES

Presently, there are numerous other uses for Liquid and Solid Aroclors and Aroclors in formulations. Only some of these uses are listed below:

As the actuating medium in bellows controls, thermostats, industrial temperature control regulators, and other kinds of automation equipment.

Liquid sealant for furnace roof.

Vacuum diffusion pump oil—pumps used to pull high vacuum for metalizing plastic, dehydrating foods, and medicinals.

Dust entrapment—used to coat fibrous glass air filter packs, metal mesh, and other material used for filtering air and gas streams.

As a vapor suppressant to increase the kill life of insecticides.

In waxes—waxes containing Aroclor are widely used in making dental castings, in the precision casting of aircraft parts, and for casting costume jewelry.

In specialized lubricants, adhesives, ink formulation, caulking compounds, plastics, rubbers, paints, varnishes, and lacquer formulations.

New uses for the various Aroclors are continually being developed. Continuous quality control, coupled with the unusual chemical and physical characteristics of Aroclors, has resulted in widespread customer acceptance and limitless expansion of uses for them.

MAE 059898

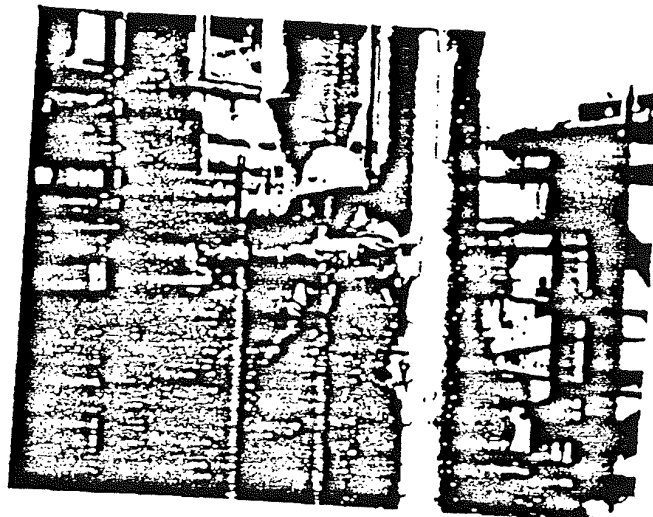
PRODUCTION QUALITY CONTROL

Over the years, the Anniston Plant Aroclor Department has done an outstanding job of continuous quality control. Customer satisfaction with the Aroclor product has led to continued increased sales. Illustrated below are some of the continuous routine quality control procedures used in the Aroclor Department.

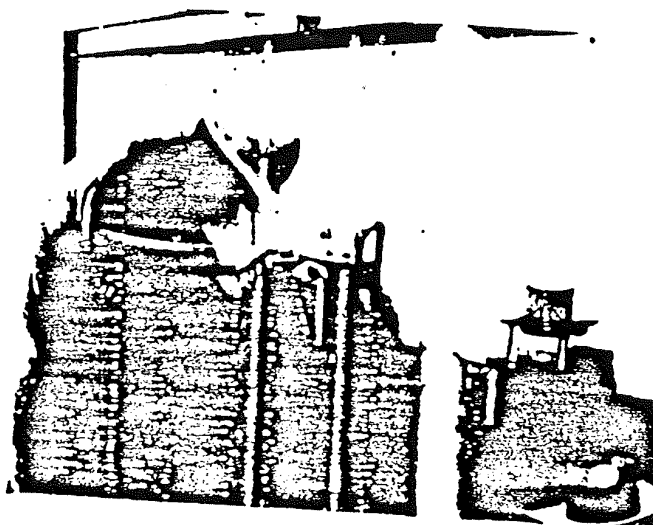


Norris Robinson is taking hourly temperature readings on Aroclor distillation equipment. Distillation temperature must be carefully controlled to enable production of uniform composition and color Aroclor product.

MAE 059899



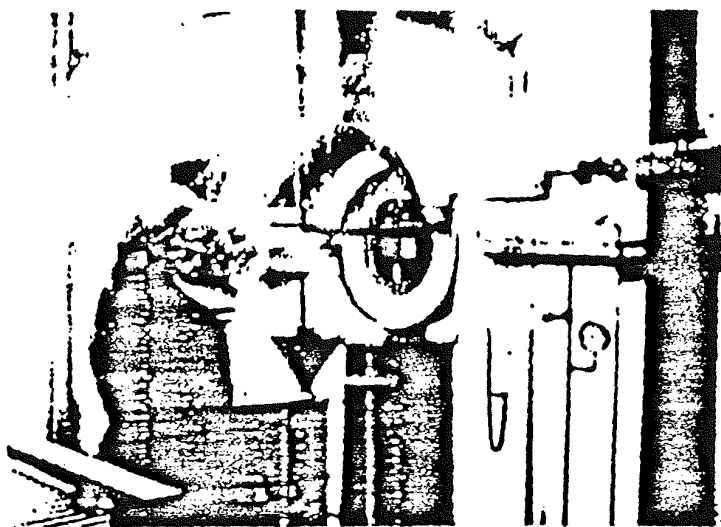
Edward Butler is sampling a chlorinator. It is important that this sample represent the actual composition of the Aroclor batch.



"Ham" Martin is determining the specific gravity of a representative sample of Aroclor. Careful adjustment of the sample

MAE 059900

temperature and accurate reading of the hydrometer is necessary to determine that the proper "end point" of the Aroclor batch has been reached.



Jesse Seay is drumming finished Aroclor product. The drum must be clean and dry before filling. The weight of Aroclor drummed must be accurate, the drum must be tight to prevent spillage, and clean before palletizing.



Ed Kirk and Robert Bell are loading finished Aroclor drums. This phase of the operation is as important as the actual manufacture.

MAE 059901

Each drum ready for shipment represents the Aroclor product to the customer. If appearance is poor or labelling is crooked, the customer can only assume that he is not buying a quality product.

Quality Promotion Committee

O. E. Dolan, Chairman

L. L. Froelkner

M. B. Mullally

F. O. Rogers

J. M. White

MAE 059902

DO NOT DISTRIBUTE
(Copies of this edn. have been
recalled for correction)
(Not for release) (This is Enacted legislation)

PRODUCTS 1961



EXHIBIT
Suppage 29
4/24/98 TSA

MAE 059907

GENERAL INFORMATION: Colloidal dispersion of silica in water. Wt. 100% at 32°. and the silica is precipitated irreversible.

USES: Anti-blocking agent in water finishes. Gloss control agent in water finishes. Increase bond strength of rubber latex adhesives. Increase modulus of latex film and thread. Sulfury harden water base laminating resins. Suspending agent for pigments in water systems. Mold lubricant. Binder for precision casting molds. Flame retarding agent for water-based paints.

SHIPPING INFORMATION: Colloidal Silica. Liquid QNB: Freezes 32°. Keep from freezing. 550-lb. drums.

"SYTON" DS AND DS-200 COLLOIDAL SILICA

ORGANIC DIVISION—LITERATURE AVAILABLE

GENERAL INFORMATION: Extremely fine colloidal aqueous dispersion of silica. In textile spinning: controls the slippage between fibers, thereby increasing tensile strength of spun yarns, permitting less twist in some cases, or fewer breaks during spinning and weaving. In textile finishing: controls slippage in worn regions, gives dry worsted-like hand, delusters in higher concentrations.

USES: Spinning, wet processing and finishing textiles.

SHIPPING INFORMATION: Colloidal Silica. Liquid QNB: Freezes 32°. Keep from freezing. 550-lb. drums.

"SYTON" P AND P-200 COLLOIDAL SILICA

ORGANIC DIVISION—LITERATURE AVAILABLE

GENERAL INFORMATION: The application of an extremely fine colloidal aqueous dispersion of silica applied either by spray or roller to multiwall paper bags or most any type of paper-lined boxes will materially reduce slippage between the coated surfaces. "Syton" P is proving to be the most effective anti-skid agent.

USES: For use on paper containers to make slip-resistant containers.

SHIPPING INFORMATION: Colloidal Silica. Liquid QNB: Freezes 32°. Keep from freezing. 550-lb. drums.

"SYTON" W-200 COLLOIDAL SILICA

ORGANIC DIVISION—LITERATURE AVAILABLE

GENERAL INFORMATION: "Syton" W-200 is an extremely fine colloidal aqueous silica dispersion and is used in wax emulsions primarily to give anti-slip properties to the wax applications.

USES: For use as an anti-slip agent in wax emulsion polishing compounds.

SHIPPING INFORMATION: Colloidal Silica. Liquid QNB: Freezes 32°. Keep from freezing. 550-lb. drums.

FUNCTIONAL FLUIDS

"AROCOR" POLYCHLORINATED POLYPHENYLS

ORGANIC DIVISION—LITERATURE AVAILABLE

STANDARD FORM: Liquids, resins or solids.

PROPERTIES: Chlorinated aromatic hydrocarbons varying from water-white mobile liquids and pale yellow viscous oils to light amber resins and opaque crystalline solids. Refractive indices above 1.6125. In general are stable on long heating at 150°C.; not affected by boiling with sodium hydroxide solution. The light oils can be distilled at atmospheric pressure without appreciable decomposition and the viscous oils and resins distill readily under vacuum. Viscous oils and resins almost odorless and tasteless at ordinary temperatures.

GENERAL INFORMATION: Most "Aroclor" materials flux readily with resins and pitch-like materials to give products having a decreased fire hazard. (Viscous oils and resins do not support combustion when heated alone even at their boiling points—temperatures above 360°C.) All have excellent electrical characteristics, high dielectric strength and resistivity, low power factor; certain of the liquids have a high dielectric constant as compared with mineral oils. The oils and resins easily soluble in most common organic solvents and drying oils; hard crystalline materials are, in general, less soluble, all are insoluble in water. The more viscous oils and resins have excellent adhesive properties. All forms are

thermoplastic, apparently undergo no further condensation or hardening upon repeated melting and cooling. No discoloring, when exposed to air, no noticeable oxidation or hardening takes place. The clear resins have rather low vaporization losses: at 65.8°C. (150°F.) less than 0.04 gni. per sq. in. of surface per month.

USES: Liquid "Aroclors" are used as dielectrics for condensers or capacitors and transformers. Solid "Aroclors" are used to impregnate carbon resistors, and when combined with other plastic materials, the "Aroclors" are excellent sealers or impregnating agents for a variety of electrical apparatus, wire coatings and tape. "Aroclors" are used as plasticizers and resins in lacquers including nitrocellulose, ethyl cellulose and cellulose acetate butyrate; to extend varnish resins and oils and as plasticizer and resin extenders for rubber-base paints, including chlorinated rubber styrene, butadiene and vinyls. In these protective coatings for metal, masonry and wood, "Aroclors" impart adhesion, resistance against moisture, acids, alkalis or other corrosive influence and they improve electrical insulating properties, durability and reduce flammability. They function as sealers in waterproofing compounds, wood preservative formulations, caulking compounds and putty. Employed in asphaltic siding materials and cellulose products impregnated or laminated with asphaltic materials, the "Aroclors" plasticize the asphalt, reduce flammability and improve durability. For printing inks; in precision-casting waxes, floor waxes and other wax products. Used widely in synthetic adhesives including ethyl cellulose, styrene, vinyls and rubber-base materials. "Aroclors" improve adhesion and resistance against corrosive influences. Selected liquid "Aroclors" are used as nonflammable hydraulic fluids for die-casting machines. They are used as actuating media for thermostats. "Aroclors" are useful in cutting oils as extreme pressure lubricants, as high-temperature lubricants and as grinding fluids. "Aroclor" 1248 is employed as a nonflammable, liquid-phase, heat-transfer medium for continuous operation in closed systems at temperatures up to 800°C. and at normal atmospheric pressure.

SHIPPING INFORMATION: Resin Synthetic NOIBN without filler, 600-lb. steel drums; 500-lb. net fiber drums.

"COOLANOL 45" DIELECTRIC COOLANT FOR ELECTRONIC EQUIPMENT

ORGANIC DIVISION—LITERATURE AVAILABLE

STANDARD FORM: Liquid.

PROPERTIES: A clear, amber liquid with dielectric, heat transfer and hydraulic properties—over a -65°F. to 400°F. temperature range.

USES: Dielectric coolant for both airborne and ground environmental electronic equipment. Also utilized in hydraulic applications in electronic equipment.

SHIPPING INFORMATION: Chemicals NOIBN, 1-qt. cans; 1-gal. cans; 5-gal. cans; 55-gal. drums.

"PYDRAUL F-9" FUNCTIONAL FLUID

ORGANIC DIVISION—LITERATURE AVAILABLE

STANDARD FORM: Liquid.

TYPICAL ANALYSIS: Viscosity at 100°F.—245 SUS, 210°F.—45 SUS; moisture—0.20% max.; neutralization number acid—0.15 max.; sp. gr. at 25°/25°C.—1.275-1.285.

PROPERTIES: A light, amber, cloudy liquid; straight synthetic fire resistant fluid with excellent lubricity and pumping characteristics; non-corrosive, extremely stable to both heat and air; recoverable by filtration; designed for long service life.

USES: Fire resistant hydraulic fluid in die-casting machines, hydraulic presses, automatic welding machines and similar applications requiring a medium viscosity fluid.

SHIPPING INFORMATION: Hydraulic system fluids other than petroleum, 55-gal. steel drums and 5-gal. cans.

"PYDRAUL 60" FUNCTIONAL FLUID

ORGANIC DIVISION—LITERATURE AVAILABLE

STANDARD FORM: Liquid.

TYPICAL ANALYSIS: Viscosity at 100°F.—60 STS, 210°F.—34 SUS; moisture—0.25% max.; neutralization number acid—0.15 max.; sp. gr. at 25°/25°C.—1.069-1.073.

PROPERTIES: A clear blue, oily, straight synthetic fire resistant hydraulic fluid with excellent lubricity and pumping characteristics; highly resistant to mechanical shear and breakdown; inert to common metals of construction; an excellent fluid for low temperature operations.

USES: A fire resistant hydraulic fluid in systems requiring extremely low viscosity. Low viscosity is excellent for high speed precision equipment such as machine tools. Low temperature properties suited for many outdoor operations.