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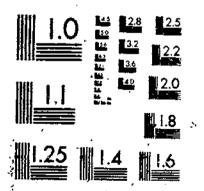
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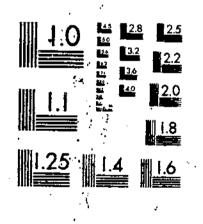
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BARRIERS IN SYNERGIZED PYRETHRINS-TREATED PAPER BAGS TO PREVENT MIGRATION OF PIPERONYL BUTOXIDE INTO CORNMEAL, FLOUR, AND CSM

Technical Bulletin No. 1475

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Washington, D.C.

Issued December 197 For sale by the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402 - Price: 30 cents Stock Number 0100-02873

BÁRRIERS IN SYNERGIZED. PYRETHRINS-TREATED PAPER BAGS TO PREVENT MIGRATION OF PIPERONYL BUTOXIDE INTO CORNMEAL, FLOUR, AND CSM

By HENRY A. HIGHLAND, research entomologist, and MARGARET SECREPST, chemist, Stored-Product Insects Research and Development Laboratory, Southern Region, Agricultural Research Service, U.S. Department of Agriculture, Savannah, Ga.

SUMMARY

Tests were conducted to determine the effects of insecticide barriers in synergized-pyrethrinstreated, insect-resistant, multiwall, paper bags. We determined the migration of piperonyl butoxide from the outer treated ply of bags having various barriers and containing flour, cornmeal, or CSM (a cereal consisting of cornmeal, soy flour, dry milk, vitamins, and minerals). Tests were conducted with eight sarancoated kraft barriers having styrene butadiene (SB) or polyethylene (PE) precoats, one PEcoated and two polyvinyl alcohol (PVA)-coated kraft barriers, and with glassine and greaseproof paper barriers. Experimental synergized pyrethrins/wax coatings were also tested. These tests indicated that (1) an effective barrier ply was required to prevent the occurrence of excessive residues of piperonyl butoxide in flour. cornmeal, and CSM stored in 25-pound, synergized-pyrethrins-treated kraft bags; (2) greaseproof paper was the best barrier tested; (3) SB/saran-coated kraft was almost as effective as greaseproof paper; (4) PE/saran-coated

kraft was considerably less effective than SB/ saran-coated kraft; (5) light coatings of PE and PVA was ineffective barriers or in fact promoted movement of piperonyl butoxide into the bagged commodities; (6) increasing the amount of synergized pyrethrins in insect-resistant-treated coatings did not extend the biological effectiveness of the coatings; (7) bags containing flour appeared to be much more susceptible to insect attack than were bags of CSM or cornmeal; (8) the rate and extent of the movement of piperonyl batoxide were greater into CSM than into cornmeal or flour; (9) two coatings of saran over SB or PE formed a better barrier against the migration of piperonyl butoxide than did a single coating; (10) piperonyl butoxide was absorbed and held by the saran-coated barrier plies; and (11) large numbers of pinholes in the saran coatings did not appear to greatly decrease the effectiveness of the coatings as barriers to the migration of piperonyl butoxide.

INTRODUCTION

Insect infestation of packaged foods can be a health hazard and cause economic losses. Available techniques make it possible to protect packages against infestation from the time they are closed at the processing plant until they are opened by the consumer. To be insect resistant, packages must not only have insectproof seals and seams, but must also be constructed to prevent the entrance of boring insects. To prevent attack by these insects, packages such as multiwall kraft bags must be treated with a repellent, in addition to being insect tight.

Environmental Protection Agency regulations allow three insect-resistant package treatments. One is pyrethrins synergized with piperonyl butoxide in the adhesive used to laminate polyethylene film to cellophane. The other two are pyrethrins synergized with piperonyl butoxide on the outer surface of multiwall paper or laminated cotton bags containing at least 50 pounds of dry food or feed. Extensive studies have shown that, when approved procedures are followed, residues resulting from the repellent migrating from the outer treated ply into the packaged product remain well within legal tolerances.¹

Even though the approved use of synergizedpyrethrins-treated, multiwall, kraft bags does not result in objectionable residues, there would be advantages in eliminating these residues altogether. A barrier ply that would prevent migration of the repellent into the inner treated plies of empty bags and into the commodity in filled bags may also extend the effectiveness of the repellent used on currently approved bags. An effective barrier would permit the use of the insect-resistant treatments on 25-pound or smaller packages for commodities such as dry food and rice.

Preliminary investigations at the Savannah

laboratory to determine the movement of the insect repellent through representative types of flexible packaging materials indicated that saran coatings, kraft coated with polyvinyl alcohol, greaseproof paper, and glassine paper were the most promising.² Subsequently, cooperative investigations were conducted with Battelle Memorial Institute to find the most effective, economical, and practical barriers to reduce or prevent migration of the repellent. Our tests of the barriers prepared by Battelle Memorial Institute indicated that saran coatings over styrene butadiene (SB) or polyethylene (PE) holdout coatings and kraft paper coated with polyvinyl alcohol (PVA) were effective barriers.

The long-term storage tests described here were conducted to determine the effectiveness of those barriers in preventing or minimizing residues in commodities stored in repellenttreated, 25-pound bags. Tests were conducted simultaneously to compare wax with standard clay as a carrier for the synergized pyrethrins applied to the outer ply.

EXPERIMENTAL MATERIALS AND METHODS

Triwall kraft bags, 17.5 by 25 inches, were made expressly for these tests by the St. Regis Paper Co. with the barriers given in table 1.^a Each bag had an outer insect-resistant-treated (IRT) kraft ply, an untreated kraft middle ply, and an inner barrier ply. The barrier ply consisted of coated kraft or a sheet of kraft, glassine, or greaseproof paper. The coated barrier plies were positioned with the coated surface facing outward, away from the commodity.

The IRT kraft and the barriers were manufactured according to standard commercial practices. Coating weights and basis weights were determined at the laboratories of the St. Regis Paper Co. The kraft paper had a basis weight of either 50 or 60 pounds per 3,000 square feet; the glassine weighed 29 pounds per 3,000 square feet; and the basis weight of the greaseproof paper was 40.5 pounds per 3,000 square feet. The SB copolymer emulsion (Dow 636, Dow Chemical Co.) precoat was applied by a coating machine equipped with a No. 3 wire-wound Mayer rod. The PE precoat (Capcote 100, St. Regis Paper Co.) was extruded. All saran coatings were from an emulsion (Serfene H32, Morton Co.) applied with an air knife. The PVA formulation consisted of 5 percent glycerine and 10 percent PVA (Elvanol 72–51, E. I. du Pont de Nemours & Co.) solids in water and was applied directly to kraft paper on a machine equipped with a No. 0 Mayer rod. The greaseproof paper was treated with 6 to 8 percent plasticizer.

The bags were filled within a 16-day period at the Savannah laboratory after the commodities and premises had been fumigated with methyl bromide to kill all insects. Twenty-five pounds of cornmeal (1.65 percent fat), flour

¹HIGHLAND, H. A., JAY, E. G., PHILLIPS, MARGARET, and DAVIS, D. F. THE MIGRATION OF PIPERONYL BUTOXIDE FROM TREATED MULTIWALL KRAFT BAGS INTO FOUR COM-MODITIES. JOUR. Econ. Ent. 59(3): 543-545. 1966.

^{&#}x27;The tables appear in the appendix.

² HIGHLAND, H. A., SECREAST, MARGARET, and MER-RITT, P. H. POLYVINYLIDENE-COATED KRAFT PAPER AS AN INSECTICIDE BARRIER IN INSECT-RESISTANT PACKAGES FOR FOOD. Jour. Econ. Ent. 61(5): 1459-1460. 1968.

PACKAGING MATERIALS AS BARRIERS TO PI-PERONYL BUTOXIDE MIGRATION. Jour. Econ. Ent. 63(1): 7-10. 1970.

(0.94 percent fat), or CSM (6 percent fat) were weighed into a bag. The bags were generally closed by one of two methods: sewn top and bottom, tape-over-stitching (TOS) closures, with IRT kraft tape heat-sealed over the stitching, or stepped-end, pasted-open-mouth (POM) closures. Some bags were closed with stitchingover-tape (SOT) closures as an experimental check.

After the bags were filled and closed, sixbag stacks were randomized on pallets in two rooms heavily infested with insects to simulate storage in a heavily infested warehouse. Large populations were maintained by periodically introducing additional insects into the rooms. The bags with the greaseproof liners were subjected only to chemical tests and were therefore placed in two-bag stacks. Each bag was carefully examined as it was moved into a room; if necessary, a hot-melt adhesive was applied to the tape ears to close possible entrances into the bag. The bags to be examined after 3 months' exposure were placed in one room and the remainder in an adjacent room having similar environmental conditions and insect populations.

The following insects, among the important pests affecting packaged food, were used : lesser grain borer, Rhyzopertha dominica (F.); cigarette beetle, Lasioderma serricorne (F.); sawtoothed grain beetle, Oryzaephilus surinamensis (L.); merchant grain beetle, Oryzaephilus mercator (Fauvel); red flour beetle, Tribolium castaneum (Herbst); and confused flour beetle, Tribolium confusum Jacquelin duVal. In addition, active populations of the following species were also present: flat grain beetle, Cryptolestes pusillus (Schönherr); cadelle, Tenebroides mauritanicus (L.); almond moth, Cadra cautella (Walker); Trogoderma inclusum LeConte; Anthrenus flavipes LeConte; black carpet beetle, Attagenus megatoma (F.); granary weevil, Sitophilus granarius (L.); rice weevil, Sitophilus oryzae (L.); maize weevil, Sitophilus zea-Motschulsky; Trogoderma glabrum maize (Herbst); and Trogoderma variabile Ballion.

Chemical Tests

At each examination, composite samples representing the entire contents of the two top bags in each stack were analyzed colorimetrically for piperonyl butoxide according to the procedure of Secreast and Cail.* Surface samples adjacent to the bag wall were also analyzed. The commodities were sampled by cutting the bag across the width of the upper surface at the middle of the bag. A metal separator was then inserted into the commodity to divide the contents into two equal portions. One end of the bag was then cut longitudinally down the middle and across the end. The plies were folded back, surface samples were taken, and the remaining contents of that half of the bag were discarded. To cbtain composite samples, the other half of the contents was poured onto a large sheet of kraft paper and mixed thoroughly; subsamples were then placed in 1-quart jars. At the 18-month examination, composite samples were collected from the exit port of the sifter as the commodities were examined for insects.

Samples of each ply were taken from the lower surfaces of the two top bags in each stack. Each sample was wrapped individually in aluminum foil and held for analysis.

All commodity and bag samples were held at 32° to 35° F., and extracts were held at 0° to 10° F. until analysis.

Biological Tests

After 3, 6, and 9 months' exposure, the bottom four bags in each stack were examined for insects in the commodities and for insect penetrations of the paper. The commodities were screened with a gyratory sifter, and the empty bags were then examined for penetrations. At about the time of the 12-month examination, the exposure rooms were fumigated with methyl bromide to eliminate highly active populations of two insects that are parasitic and predatory upon stored-product insects. The premises were reinfested with the species previously listed. After 15 and 17 months' exposure, two four-bag stacks of cornmeal and CSM, one four-bag stack of flour, and one six-bag stack of flour were examined for insect penetrations without opening the bags. After 19 months' exposure, all remaining cornmeal and flour bags were opened and examined for insects in the commodities and penetrations of the bags.

^{&#}x27;SECREAST, M. F., and CAIL, R. S. A CHROMATOGRAPH-IC-COLORIMETRIC METHOD FOR DETERMINING LOW RESIDUES OF PIPERONYL BUTOXIDE IN FLOUR. Agr. and Food Chem. 19(1): 192-193. 1971.

Within 2 weeks after the bags were placed in storage, grease stains were noted on the exterior surfaces of some CSM bags, indicating that the fat was migrating through the SB/ saran, PE/saran, PVA, and glassine barriers. These bags were examined visually after about 1 and 3 months' storage to estimate the fat stained areas on the exterior surfaces, and samples of the barriers were examined for pinholes formed during manufacture (table 1).

The most effective barrier to the migration of piperonyl butoxide was the greaseproof liner (tables 2-7). Cornneal protected by the greaseproof liner in bags with IRT/clay coatings usually contained only traces of piperonyl butoxide, and CSM generally had about 1 p.p.m. or less during 19 months of storage. Cornneal in bags with greaseproof liners and IRT/wax coatings contained less than 1 p.p.m., even though the coating contained a very high (96 milligrams per square foot) initial piperonyl butoxide deposit. CSM in similar bags contained 5.7 p.p.m. or less during 19 months of storage.

The kraft treated with an SB precoat and a double coat of saran was almost as effective as the greaseproof liner (tables 2-7). Residues of piperonyl butoxide in cornmeal and flour exceeded 1 p.p.m. only after 12 and 19 months' storage in bags with IRT/clay coatings. CSM had residues up to 6.7 p.p.m. Commeal protected by SB/saran-coated kraft in IRT/waxcoated bags contained residues that exceeded 10 p.p.m. only after 19 months' storage, even though the coatings on these bags contained almost twice the approved piperonyl butoxide content. However, the high initial piperonyl butoxide deposit in the wax coating produced residues of up to 23 p.p.m. in CSM in bags with SB/saran-coated kraft barriers.

The PE/saran (2C)-coated kraft also reduced migration of piperonyl butoxide into the three commodities, but it was considerably less effective than either the greaseproof paper or the SB/saran-coated kraft. PE-coated kraft was not a barrier; on the contrary, it promoted the movement of piperonyl butoxide. Residues in all commodities in bags with PE-coated kraft plies were usually higher than residues in the control bags. Residues in cornmeal and flour in bags with a PVA-coated kraft ply were very similar to residues in the control bags, whereas CSM in bags with a PVA-coated kraft ply had higher residues than CSM in the control bags.

Residues in flour and cornmeal stored in glassine-lined bags with IRT/clay coatings never exceeded 6.4 p.p.m., but CSM in similar bags contained up to 12.6 p.p.m. of piperonyl butoxide. Residues exceeded 10 p.p.m. in all commodities stored in IRT/wax-treated bags with glassine barriers.

Residues in composite samples (tables 2-4) were generally very similar to residues in surface samples (tables 5-7) from the same bag. This is not in agreement with Yeadon et al.,⁵ who found that most of the piperonyl butoxide remains in the layer of commodity immediately adjacent to the bag wall. These apparently conflicting conclusions probably resulted from the shallow depth of commodity in the 25-pound bags; thus, surface samples constituted a substantial portion of the depth from the surface to the center of the bag, thereby approximating composite samples.

Two coatings of saran over SB or PE were more effective than was a single coating over SB or PE in reducing the migration of piperonyl butoxide into the test commodities. This is especially evident in table 3, which shows that double coatings of saran provided CSM with much better protection from piperonyl butoxide contamination than did single coatings.

Within 3 months piperonyl butoxide migrated from the outer treated ply into the untreated kraft middle ply and also into the barriers containing a saran component (tables 8-11). After 19 months, less piperonyl butoxide was found on the saran barrier in CSM-filled bags than on saran barriers in cornneal or flour bags (table 11). This occurred because more migrated directly into the CSM packed in these bags. There was little buildup of piperonyl butoxide on the greaseproof and glassine plies.

Under conditions of this test, most of the IRT bags resisted infestation for 15 months regardless of the type of barrier in the bag (tables 12-14). Any variation in protection provided by the various barriers was obscured by

³ YEADON, DAVID A., DANNA, GARY F., and COOPER, ALBERT S., JR. AN ACCELERATED TEST FOR EVALUATING THE STABILITY OF PYRETHRINS-PIPERONYL BUTOXIDE TREATMENTS ON FOOD STORAGE BAG FABRIC. Jour. Stored Products Res. 6(1): 45-51, 1970.

the activity of predators and parasites at the critical 12-month exposure period and by the subsequent fumigation to eliminate these insects. With one exception, the wax/IRT coatings, all of which contained high initial deposits of synergized pyrethrins, were not superior to the clay/IRT formulation in preventing infestation. The exception was the wax/IRT bag with no barrier, which provided good protection for 17 months. However, the commodities in these bags contained very high deposits of piperonyl butoxide that may have provided protection from infestation.

Data in tables 12 to 14 show that treated

TOS bags provided good protection from infestation, while very little protection was provided by untreated TOS bags. This points out the necessity for an insect-resistant treatment on packages that are susceptible to penetration by insects.

These data also indicate that there was more insect activity in and around bags of flour than in bags of either CSM or commeal. There were generally more flour bags penetrated at both the 17- and 19-month examinations. The higher susceptibility of flour to insect attack was evident in the untreated control bags at all examinations.

APPENDIX.-TABLES

ABBREVIATIONS USED IN TABLES

- IRT Insect-resistant-treated (kraft).
- PE Polyethylene.
- POM Pasted-open-mouth (closure).
- PVA Polyvinyl alcohol.
- SB Styrene-butadiene copolymer.
- SOT Stitch-over-taping (closure).
- TOS Tape-over-stitching (closure).

TABLE 1.—Weights of components of barrier plies in synergized-pyrethrins-treated, triwall bags; pinholes in barrier plies; and grease-stained areas on outer ply of CSM-filled bags

Barrier coating or	Initial piperonyl		Costi	ng weight c			30	Pinholes	Grease stain on IRT ply after—	
sheet 1	butoxide deposit	SB	PE	Saran	PVA	Total	barrier ply	barrier	1 то.	3 mo.
			CLAY R	SPELLENT	CARRIER					
	Mg./ sq. ft.	Lb./ ream	Lb./ ream	Lb./ ream	Lb./ ream	Lb./ ream	Ľb./ ream	No./ sq. ft.	Pct.	Pct.
SB/saran (2C)	- 51	9.0		7.5		16.5	69.4	20	15	30
	49	4.2		16.5		20.7	72.7	3	8	15
	50	3.9	.	22.8		26.7	79.4	10	δ	10
SB/saran	52	6.7		4.1		10.8	61.9	>1,000	10	20
	54	4.0		7.7		11.7	65.4	40	4	10
	51	8.2		7.2		15.4	68.2	>1,000	8	15
PVA (2C)					1.8	1.8	54.4	>1,000	S	10
PVA					3.2	3.2	55.8	>1,000	3	5
PE/saran (2C)			4.8	9.6		14.4	66.0	0	7	7
• • •			5.8	4.5		10.3	64.5	0	7	8
PE/saran		+	4.0	1.0		4.0	58.3	50	5	7
PE			4.0				29.0	0	2	20
Glassine				• •			40.5	õ	<1	1
Greaseproof					••		40.0 50.8	v	100	100
Kraft	_ 47						00.0	• • • •	100	100

TABLE 1.—Weights of components of barrier plies in synergized-pyrethrins-treated, triwall bags; pinholes in barrier plies; and grease-stained areas on outer ply of CSM-filled bags---Continued

Barrier coating or sheet ¹	Initial piperonyl butoxide	yl						Pinholes in	Grease stain on IRT ply after—	
	deposit	SB	РE	Saran	PVA	Total	barrier ply	barrier	1 mo.	3 то.
			WAX RE	PELLENT	CARRIER					
	Mg./ sq. ft.	Lb./ ream	Lb./ ream	Lb./ ream	Lb./ ream	Lb./ ream	Lb./ ream	No./ sq. ft.	Pct.	Pct.
SB/saran		4.8		5.2		10.0	60.0	>1,000	1	20
PVA					1.5	1.5		>1,000	0	5
PE/saran			5.2	5.6		10.8	64.8	1	0	2
PE	113		5.0			5.0	58.8	40	0	5
Glassine	74						28.6	3	0	30
Greaseproof							39.9	0	<1	<1
Kraft	128						51.8		20	95

 $^{1}(2C) = 2$ coatings of saran or PVA; and coatings were applied to kraft paper.

'1 ream is 3,000 square feet.

'Estimated by subtracting average basis weight of the kraft paper of barrier piles in all other bags from total basis weight of this ply.

TABLE 2.—Piperonyl butoxide in composite samples of cornmeal stored in 25-pound, synergized-pyrethrins-treated, triwall bags with various barriers

Barrier coating or sheet 1	Initial piperonyl butoxide -		Piperonyl but	toxide in corn	meal after ² -	
or sneet -	deposit	3 mo.	6 mo.	9 mo.	12 mo.	19 mo.
	CLAY	REPELLENT	CARRIER			
· · · · · ·	Mg./ sq. ft.	P.p.m,	P.p.m.	P.p.m.	P.p.m.	P.p.m.
SB/saran (2C)	51	0.5	0.6	1.0	1.1	2.0
	49	.4	.6	.7	.6	1.5
	50	.3	.4	.6	.7	1,4
	52	.7	2.3	2.5	2.7	3.1
SB/saran	54	.5	1.3	1.5	1.8	2.6
VA (2C) VA	51	.5	1.0	1.3	1.3	1.5
PVA (2C)	46	6.6	7.8	9.1	8.4	6.2
PVA	49	8.0	9.9	10.5	8.7	7.5
PE/saran (2C)	50	1.4	5.2	5.3	4.8	4.6
PE/saran	53	4.5	9.1	8.2	9.9	7.9
PE	52	9.2	9.2	9.7	9.7	7.5
Glassine	50	3.3	5.7	6.4	6.4	6.1
Greaseproof	59	<.2	.3	.2	<.2	2.5
Kraft	47	6.2	8.7	7.6	7.5	6.2
	WA	X REPELLE?	NT CARRIER			
SB/zaran	92	2.8	6.0	6.7	7.0	10.6
PVA	80	12.6	15.0	15.6	14.6	10.9
PE/saran	77	7.1	14.6	12.3	13.7	11.0
PE	113	21.0	19.5	21.0	21.0	13.4
Glassine	74	7.3	11.5	15.3	14.6	10.0
Greaseproof	96	.3	.4	.4	.5	.7
Kraft	128	22.0	30.0	31.0	28.0	21.0

 $^{\circ}$ (2C) =2 coatings of saran or PVA; all coatings were applied to kraft paper.

² Each figure is the average of 2 samples from each of 2 bags.

Barrier coating or sheet 1	Initial piperonyl butoxide -		Piperonyl b	utoxide in Ca	5M sfter ² —	_
or sneet -	deposit	3 mo.	6 mo.	9 mo.	12 mo.	19 mo.
	CLA	y repeller	T CARRIER			
	Mg./ sq. ft.	P.p.m.	 P.p.m.	P.p.m.	P.p.m.	P.p.m.
SB/saran (2C)	51	0.9	2.6	3.0	4.1	5.3
	49	.9	3.1	3.1	4.8	6.7
	50	.9	1.4	1.7	3.3	5.3
SB/saran	52	5.0	9.7	8.5	9.5	12. 1
	54	3.7	8.0	7.9	9.3	9.7
	51	1.1	4.2	4.8	5.3	7.4
PVA (2C)	46	7.5	12.7	14.3	12.5	16.0
PVA	49	9.6	11.9	15.4	14.7	13.5
PE/saran (2C)	50	2.5	4.0	6.0	7.4	8.5
PE/saran	53	6.0	10.4	13.4	13.3	11.1
PE	52	13.0	13.5	24.0	17.2	16.6
Glassine	50	7.3	10.9	10.2	12.6	11.1
Greaseproof	59	.4	.9	.9	1.2	.7
Kraft	47	5.3	7.1	7,7	10.2	13.8
····	WA	X REPELLE	NT CARRIER			
SB/saran	92	7.6	21	16.1	19.4	23
PVA	80	17	23	30	25	22
PE/saran	77	11.8	15	20	24	20
PE	113	24	32	25	32	29
Glassine	74	17.4	27	22	23	16.2
Greaseproof	96	1.3	1.8	1,9	5.7	3.5
Kraft	128	17.5	24	30	29	35

TABLE 3.—Piperonyl butoxide in composite samples of CSM stored in 25-pound, synergized-pyrethrins-treated, triwall bags with various barriers

(2C) = 2 coatings of soran or PVA; all coatings were applied to kraft paper.

² Each figure is the average of 2 samples from each of 2 bags.

TABLE 4.—Piperonyl butoxide in composite samples of flour stored in25-pound, synergized-pyrethrins-treated, triwall bags with various barriers

Barrier coating	Initial piperonyl butoxide	Piperonyl butoxide in flour after 2-								
or sheet 1	deposit	3 mo.	6 mo.	9 mo.	12 mo.	19 mo.				
	CLA	Y REPELLE	NT CARRIER							
	Mg./ sq. ft.	P.p.m.	P.p.m.	P.p.m.	P.p.m.	P.p.m				
SB/saran (2C)	51	0.3	0.5	0.6	0.8	1.0				
,	49	.2	.4	,5	.8	.7				
	50	.3	.4	.5	.8	.5				
SB/saran	52	.5	1.4	1.1	2.5	2.4				
	54	.3	.8	1.2	2.1	1.9				
	51	.3	.6	.4	.7	1.3				
PVA (2C)	46	7.5	10.9	11.7	10	7.8				
PVA	49	10.3	9.9	15.6	10	9.5				
PE/saran (2C)	50	1.9	2.4	2,7	3	3.1				
PE/saran	53	3.5	6.8	9.4	8.2	9.6				
PE	52	8.6	12.5	13,2	17.3	7.8				
Glassine	50	2.1	4.1	4.3	5.0	3.7				
Greaseproof	59									
Kraft	47	6	10.4	11.2	9.7	8.3				

 TABLE 4.—Piperonyl butoxide in composite samples of flour stored in 25pound, synergized-pyrathrins-treated, triwall bags with various barriers

 —Continued

Barrier coating or sheet ¹	Initial piperonyl butoxide	ur after °—				
	deposit	3 mo.	6 mo.	9 mo.	12 mo.	19 mo.
	WA	X REPELLE	NT CARRIER			
	Mg./ sq. ft.	P.p.m.	P.p.m.	P.p.m.	P.p.m.	P.p.m.
SB_saran	92					
PVA	80	15	19.6	25	21	17.6
PE/saran	77	7.4	10.9	15	13	17.8
PE	113	20	18.5	21	24	9.9
Glassine	74	4.1	9.2	10.6	14	10.9
Greaseproof	96					
Kraft	128	28	26	30	27	20

'(2C)=2 coatings of saran or PVA; all coatings were applied to kraft paper.

*Each figure is the average of 2 samples from each of 2 bags.

TABLE 5.—Piperonyl butoxide in surface samples of cornmeal stored in 25-pound, synergized-pyrethrins-treated, triwall bags with various barriers

Barrier coating or shect ¹	Initia) piperonyl	Piperonyl butoxide in commeal after 2-					
or sneet -	butoxide - deposit	3 mo.	6 mo.	9 mo.	12 mo.		
CLAY	REPELLENT	CARRIER					
	Mg./				•		
	sq. ft.	P.p.m.	P.p.m.	P.p.m.	P.p.m.		
SB/saran (2C)	51	0.3	0.3	0.6	0.5		
	49	.3	.3	.5	.3		
	50	.2	.2	.3	.7		
SB/saran	52	-5	1,2	1.5	2.1		
	54	.4	.8	.8	1.1		
	51	.3	.5	.9	.9		
PVA (2C)	46	6.8	6.8	8.5	6,3		
PVA	49	6.7	9.8	8.5	5.9		
PE/saran (2C)	50	.7	1.9	4.1	2.8		
PE/saran	53	3.7	5.8	5.9	7.0		
PE	52	7.8	10.2	10.7	6.5		
Glassine	50	1.9	5.0	4.8	5.2		
Greaseproof	59	< .2	<.2	.2	<.2		
Kraft	47	6.9	8.9	9.7	7.5		
WAX	REPELLENT	CARRIER					
SB/saran	92	1.7	2,4	4.1	5.9		
PVA	80	11.5	13.4	14.0	11.0		
PE/saran	77	4.9	11.2	10.9	9.0		
PE	113	16.7	22.0	18.0	16.3		
Glassine	74	7.1	11.5	13.8	12.1		
Greaseproof	96	.2	.2	.3	.4		
Kraft	128	14.7	27	33	24		

(2C) = 2 coatings of saran or PVA; all coatings were applied to kraft paper.

²Each figure is the average of 2 samples from each of 2 bags.

Barrier coating or sheet ¹	Initial piperonyl butoxide	Piper	ronyl butoxide in CSM after ² —				
	deposit	3 mo.	6 mo.	9 mo.	12 mo.		
CL	AY REPELLE	NT CARRIES	3				
	Mg./ sq. ft.	P.p.m.	P.p.m.	P.p.m.	P.p.m.		
SB/saran (2C)	51	0.8	2.0	3.0	2.6		
	49	1.0	1.7	3.1	2.9		
	50	.7	.9	2.7	1.9		
SB/saran	52	3.6	4.9	5.3	7.6		
	54	2.8	6.4	5.9	9.0		
	51	.8	4.0	3.7	5.2		
PVA (2C)	46	8.8	12.4	14.1	12.0		
PVA	49	9.4	11.5	15.0	14.7		
PE/saran (2C)	50	.9	2.2	4.6	6.0		
PE/satan	53	6.7	8.3	12.1	9.9		
PE	52	10.5	13.2	18	17.3		
Glassine	50	5.6	10.2	13.8	12.4		
Greaseproof	59	.5	1.2	.7	.9		
Kraft	47	4.3	8.1	7.6	7.5		
WAX	REPELLENT	CARRIER					
SB/saran	92	3.1	10.6	12.2	18.9		
PVA	80	21	21	31	24		
PE/saran	77	11.0	13.9	10.5	17.1		
PE	113	22	26	25	30		
Glassine	74	23	21	23	24		
Greaseproof	96	.9	1.9	2.5	4.4		
Kraft	128	17.7	19.7	57	26		

TABLE 6.—Piperonyl butoxide in surface samples of CSM stored in 25-pound, synergized-pyrethrins-treated, triwall bags with various barriers

(2C) = 2 coatings of saran or PVA; all coatings were applied to kraft paper.

* Each figure is the average of 2 samples from each of 2 bags.

TABLE 7.—Piperonyl butoxide in surface samples of flour stored in 25-pound, synergized-pyrethrins-treated, triwall bags with various barriers

Barrier coating or sheet ¹	Initial piperonyl butoxide -	Pipere	onył butoxide	xide in flour after 2—		
or sneet -	deposit	3 mo.	6 mo.	9 mo.	12 mô.	
CLAY	REPELLENT	CARRIER				
	Mg./ sq.ft.	P.p.m.	P.p.m.	P.p.m.	P.p.m.	
SB/saran (2C)	51	0.2	0.3	0.5	0.5	
	49	<.2	.3	.4	.5	
	50	.2	.3	.3	.5	
SB/saran	52	.4	1.0	1.2	1.7	
	54	.2	.7	1.2	1.0	
	51	< .2	-4	,4	.5	
PVA (2C)	46	9.9	9.8	12.2	10.4	
PVA	49	7.6	10.7	13.7	9.4	
PE/saran (2C)	50	.9	2.8	2.3	3.7	
PE/saran	53	3.4	4.7	7.8	6.9	
PE	52	6.3	11.2	14.1	13.2	
Glassine	50	2.1	3.9	4.8	6.0	
Greaseproof	59					
Kraft	47	9	13.2	18.4	10.4	

Barrier coating or sheet ¹	Initial piperonyl butoxide	Piperonyl butoxide in flour after 2—					
	deposit	3 mo.	6 mo.	9 mo.	12 mo.		
WAX	REPELLENT	CARRIER					
SB/saran	Mg./ sq. ft. 92	P.p.m.	P.p.m.	P.p.m.	P.p.m.		
PVA	80	16.5	23	23	20		
PE/saran	77	4.0	8.4	15.1	10.9		
PE	113	24	17.8	25	17.4		
Glassine	74	5.1	11.7	15.9	13.8		
Greaseproof	96 128	25	28	44	28		

TABLE 7.—Piperonyl butoxide in surface samples of flour stored in25-pound, synergized-pyrethrins-treated, triwall bags with various barriers—Continued

 $^{1}(2C) = 2$ coatings of saran or PVA; all coatings were applied to kraft paper.

* Each figure is the average of 2 samples from each of 2 bags.

TABLE 8.—Piperonyl butoxide on plies of 25-pound, synergized-pyrethrins-treated, triwall bagsstored for 3 months

[Mg./sq. ft.]

	Initial		Piperonyl butoxide on indicated plies from bags of 7.3-								
Barrier coating or sheet 1	piperonyl butoxide		Cornmeal			Flour		CSM			
	deposit	1	2	3	1	2	3	1	2	3	
			CLAY I	REPELLENT	CARRIER			-			
SB/saran (2C)	51	30	3.9	5.7	32	4.6	3.7	61	5.8	4.6	
	49	27	4.9	8.6	27	4.8	6.7	29	9.6	7.1	
	50	27	4.9	8.2	28	4.0	5.6	27	6.8	5.8	
SB/saran	52	27	3.4	6.6	30	4.3	6.6	24	4.7	5.2	
	54	25	4.9	9.5	23	3.8	6.2	21	6.7	6.8	
	51	57	5.3	8.1	27	4.7	6.6	23	8.1	6.3	
PVA (2C)	46	17.7	3.3	4.4	19.0	2.9	1.5	15.2	2.9	3.3	
PVA	49	16.4	1.4	1.4	16.4	2.3	1.3	13.9	1.0	2.7	
PE/saran (2C)	50	24	3.8	8.3	20	3.5	8.6	23	3.7	8.5	
PE/saran	53	16.4	6.3	7.0	16.4	3.8	8.8	29	3.3	6.8	
PE	52	13.9	.9	1.4	12.6	1.6	1.5	12.6	.6	3.3	
Glassine	50	29	4.3	1.9	27	5.7	2.0	20	3.0	1.1	
Greaseproof	59	32	6.7	2.9				25	7.5	3.0	
Kraft	47	12.6	1.5	1.4	17.7	3.3	1.1	23	6.8	1.9	
			WAX R	EPELLENT	CARRIER						
SB/saran	92	47	10.2	29		· · · · · ·		13.9	9.9	19.0	
PVA	80	13.9	3.9	2.5	19.0	6.1	2.5	8.8	7.7	4.8	
PE/saran	77	20	5.2	6.8	27	8.0	15.2	6.3	3.7	14.0	
PE	113	23	3.8	4.2	19	4.8	4.0	10.1	2.3	6.1	
Glassine	74	33	9.2	5.1	46	12.4	9.0	16.4	7.7	3.0	
Greaseproof	96	52	17.7	4,4				46	16.4	6.1	
Kraft	128	17.7	4.3	3.2	24	4.8	2.5	24	12.6	4.9	

'(2C)=2 coatings of saran or PVA; all coatings were applied to kraft paper.

² Ply 1 is the outer ply.

'Each figure is the average from 2 bags.

TABLE 9.—Piperonyl butoxide on plies of 25-pound, synergized-pyrethrins-treated, triwall kraft bagsstored for 6 months

				Piperonyl	butoxide on	indicated p	lies from ba	gs of ³ ,3		
Barrier coating	Initial piperonyl	<u> </u>	Cornmeal			Flour			CSM	
or sheet 1	butoxide deposit	1	2	3	1	2	3	1	2	3
	· · · · ·		CLAY R	EPELLENT	CARRIER					
SB/saran (2C)	51	24	4.3	7.2	28	4.1	8.2	22	9.0	• 4.3
	49	21	4.5	11.1	22	4.5	9.6	17.4	8.5	8.7
	50	23	4.4	10.2	23	3.9	9.3	17.4	7.7	7.8
SB/saran	52	23	3.4	11.5	26	3.9	10.5	19.3	2.2	5.6
	54	20	4.0	9.0	23	4.1	12.1	12.8	3.0	6.7
	51	20	4.3	11.5	22	4.3	9.5	18.4	5.6	6.6
PVA (2C)	46	12.1	1.3	1.1	15.0	2.2	1.0	10.0	2.3	3.0
PVA	49	10.5	1.3	1.3	14.1	1.6	1.2	10.7	1.1	2.2
PE/saran (2C)	50	18.2	2.9	11.2	19.1	3.8	11.0	13.0	3.0	7.7
PE/saran	53	11.0	2.2	7.8	13.0	2.6	7,9	27	1.7	6.2
PE	52	10.0	.6	,9	12.3	.9	1.3	10.7	2.1	42.4
Glassine	50	24	3.0	1.6	26	4.5	1.8	16.8	2.7	.9
Greaseproof	59	26	9.0	2.1				22.6	10.0	2.7
Kraft	47	8,8	1.8	1.1	9.6	1.3	1.1	14.1	5.0	2.4
			WAX R	EPELLENT	CARRIER					
SB/saran	92	26	8.2	4.3				8.2	3.7	10.4
PVA	80	3.9	1.3	1.7	9.3	3.9	1.8	2.6	1.3	4.3
PE/saran	77	6.6	2.1	10.4	12.4	6.2	10.2	8.4	3.3	11.5
PE	113	6.7	1.7	2.4	8.2	3.0	2.3	7.3	2.2	4.1
Glassine	74	17.7	5.4	3.0	127	8.9	3.4	12.0	5.9	1.1
Greaseproof	96	48	2.0	3.5				27	38	• 5.0
Kraft	128	7.3	2.2	2.6	9.5	2.9	1.8	13.9	11.7	5.1

[Mg./sq. ft.]

(2C) = 2 coatings of saran or PVA; all coatings were applied to kraft paper.

² Ply 1 is outer ply.

'Each figure is the average from 2 bags.

'1 bag was examined.

TABLE 10.—Piperonyl butoxide on plies of 25-pound, synergized-pyrethrins-treated,	triwall	kraft
bags stored for 9 months		

[Mg./sq. ft.]

				Piperonyl I	outoxide on i	indicated p	lies from ba	gs of ^{2,3} —		
Barrier coating	Initial piperonyl		Cornmeal			Flour			CSM	
or sheet ¹	butoxide deposit	1	2	3	1	2	3	1	2	3
			CLAY R	EPELLENT	CARRIER					
SB/saran (2C)	51	20	4.0	9.0	22	3.9	10.0	23	6.9	4.7
(10) (10) (10)	49	19.8	5.3	9.2	17.4	4.5	14.6	16.8	6.9	7.9
	50	20	4.8	9.0	13.2	4.2	9.4	16.8	5.7	8.7
SB/saran	52	15.9	4.8	7.6	20	4.2	10.0	13.7	1.5	5.0
	54	18.7	8.0	9.8	10.9	4.4	11.3			
	51	16.3	6.3	12.3	14.3	3.5	7.5			
PVA (2C)	46	6.7	1.0	.9	9.1	1.5	.8	8.5	2.2	2.0
PVA	49	6.9	.8	.9	9.4	1.0	-8	8.3	1.7	2.9
PE/saran (2C)	50	9.0	2.1	5.5	10.0	2.6	6.6	10.6	1.8	4.8
PE/saran	53	12.6	1.0	4.6	9.6	1.4	4.3	24	1.1	5.1
P2	52	7.7	.6	.7	7.4	.6	.7	7.9	.4	1.5
Glassine	50	16.2	3.1	1.4	19.4	5.2	.9	10.8	1.9	.3
Greaseproof	59	28	11.6	2.2				12.7	8.5	2.0
Kraft	47	7.2	1.9	.9	6.9	1.5	1.0	11.2	2.9	2.2

TABLE 10.—Piperonyl butoxide on plies of 25-pound, synergized-pyrethrins-treated, triwall kraft bags stored for 9 months—Continued

Barrier coating or sheet 1	Initial			Piperonyl	butoxide on	indicated p	lies from ba	gs of 1.1-		
	piperonyl butoxide deposit	Cornmeal				Flour			CSM	
		1	2	3	1	2	3	1	2	8
			WAX RE	PELLENT	CARRIER					
SB/saran	92	14.3	4.7	2.8		.		4.1	3.1	8.7
PVA	80	4.7	1.4	1.7	6.4	2.2	1.8	2.0	1.0	2.3
PE/saran	77	3.5	1.3	6.7	9.8	3.5	11.9	7.7	1.7	5.2
PE	113	5.1	1.3	1.5	6.3	1.8	1.7	3.5	.8	2.6
Glassine	74	12.4	4.0	1.9	14.9	6.9	2.0	8.7	4.3	.7
Greaseproof	96	30	17.8	6.1				17.2	11.8	3.2
Kraft	128	5.2	1.3	1.7	6.2	1.4	1.4	6.9	5.9	2.9

[Mg./sq.ft.]

(2C) = 2 coatings of saran or PVA; all coatings were applied to kraft paper.

' Ply 1 is outer ply.

'Each figure is the average from 2 bags.

TABLE 11.—Piperonyl butoxide on plies 25-pound, synergized-pyrethrins-treated, triwall bags stored for 19 months

	Initia]			Piperonyl	butoxide on :	indicated pli	es from bag	s of ^{2,3} —		-
Barrier coating or sheet ¹	piperonyl butoxide		Cornmea	1		Flour			CSM	
UT BAEEL	deposit	1	2	3	1	2	3	1	2	3
			CLAY	REPELLENT	CARRIER					
SB/saran (2C)	51	13.6	2,0	6.6	21	2.5	5.1	13.3	4.1	1.8
	49	13.6	3.4	6.6	13.9	4.0	5.5	10.8	2.7	3.9
	50	17.0	2.6	6.7	15.8	3.2	6.4	12.1	3.1	3.2
SB/saran	52	9.5	.7	5.3	13.8	1.9	5.6	11.7	1.1	1.6
	54	9.9	1.7	7.3	11.8	2.1	6.0	7.8	1.1	1.9
	51		2.6	7.5	11.1	2.4	6.7	8.5	2.1	2.5
PVA (2C)	46	5.3	.2	.4	5,4	.9	4.4	5.3	.5	1.0
PVA	49	5.3	1.6	.4	5.3	.4	.4	4.9	.8	.6
PE/saran (2C)	50	6.1	.8	'1.4	9.1	1.4	4.2	6.3	.8	1.5
PE/saran	53	11.0	1.5	.8	6.9	1.1	3.0	10.9	.5	1.0
PE	52	5.4	.3	.5	7.9	.2	.6	5.2	.2	.7
Glassine	50	9.3	.9	.5	11.5	2.6	.5	8.9	1.6	.2
Greaseproof	59	10.1	5.3	.8				10.0	6.1	.9
Kraft	47	2.8	.6	.3	3.5	.5	.5	5.4	1.1	.5
			WAX	REPELLENT	CARRIER					
SB/saran	92	4.2	1.2	14.9				1.9	1.4	3.2
PVA	80	.4	.2	.7	1.0	0.4	0.7	1.9	.4	.7
PE/saran	77	.6	.3	2.5	3.4	1.4	6.4	1.8	.5	1.7
PE	113	1.8	.4	.6	.7	.3	1.1	1.6	.4	1.4
Glassine	74	3.3	1.0	.8	5.7	2.5	.7	2.0	1.2	.2
Greaseproof	96	22	14.5	2.7				11.5	10.0	2.2
Kraft	128	1.1	.2	.8	4.0	.9	.9	4.1	2.7	1.6

[Mg./sq. ft.]

'(2C)=2 coatings of saran or PVA; al' coatings were applied to kraft paper.

' Ply 1 is outer ply.

¹ Each figure is the average from 2 bags.

⁴ Single sample was examined.

Barrier coating	Repellent		Initial piperonyl			Penetrated	i bags after-	_	
or sheet '	CBITICE	Closure	butoxide deposit	8 то.	6 1210.	9 mo.	15 mo.	17 mo.	19 mo.
			Mg./sq. ft.	Pct.	Pct.	Pct.	Pct.	Pct.	Pct.
SB/saran (2C)	Clay	TOS	51	0	0	0	0	12	50
DD/ Curum (20) 1	Clay	TOS	49	0	0	0	0	12	14
	Clay	TOS	50	0	0	0	0	12	43
SB/saran	Clay	TOS	52	0	0	0	0	0	2 5
	Clay	TOS	54	0	0	0	0	12	37
	Clay	TOS	51	0	0	0	0	12	43
	Wax	TOS	92	0	0	0	0	12	50
PVA (2C)	Clay	TOS	46	0	0	0	0	17	20
PVA	Clay	TOS	49	0	0	0	0	0	37
	Wax	TOS	80	25	0	0	0	0	14
PE/saran (2C)	Clay	TOS	50	0	0	0	0	25	62
PE/saran	Clay	TOS	53	0	Û	0	17	37	75
	Wax	TOS	77	0	0	0	17	28	100
	Wax	POM	36						-
-	Clay	POM	70						
PE	Clay	TOS	52	0	0	0	0	14	86
	Wax	TOS	113	0	0	0	0	50	75
Glassine	Clay	TOS	50	0	0	0	0	12	62
	Wax	TOS	74	0	0	0	0	25	83
Greaseproof	Clay	TOS	59						
	Wax	TOS	96		-			 -	
Kraft	Clay	TOS	47	0	0	0	0	12	57
	Wax	TOS	128	0	0	0	0	0	33
	None	TOS	0	50	0	100	25	75	100
	None	POM	0			-			
	None	SOT	0	100	100	100	100	100	100

 TABLE 12.—Insect penetration of 25-pound, synergized-pyrethrins-treated, triwall bags containing commeal

'(2C)=2 coatings of saran or PVA; all coatings were applied to kraft paper.

 TABLE 13.—Insect penetration of 25-pound, synergized-pyrethrins-treated, triwall bags containing

 CSM

Barrier coating	Repellent		Initial piperonyl	Penctrated bags after-							
or sheet '	Carrier	Closure	butoxide deposit	3 mo.	6 mo.	9 mo.	15 mo.	17 mo.	19 mo.		
			Mg./sq. ft.	Pct.	Pct.	Pct.	Pct.	Pct.	Pet.		
SB/saran (2C)	Clay	TOS	51	0	0	0	14	43	100		
, ,	Clay	TOS	49	0	0	0	0	28	43		
	Clay	TOS	50	0	0	25	0	0	86		
SB/saran	Clay	TOS	52	0	0	0	0	28	87		
	Clay	TOS	54	0	0	0	0	43	100		
-	Clay	TOS	61	0	0	0	0	50	57		
	Wax	TOS	92	25	0	0	12	25	100		
PVA (2C)	Clay	TOS	46	0	0	0	0	37	75		
PVA	Clay	TOS	49	0	0	0	0	62	57		
• • • • • • • • • • • • • • • • • • •	Wax	TOS	80	0	0	0	0	12	71		
PE/saran (2C)	Clay	TOS	50	0	0	0	0	28	67		
PE/saran	Clay	TOS	53	0	0	0	0	20	40		
	Wax	TOS	77	0	0	0	0	43	83		
	Wax	POM	36						 ·		
	Clay	POM	70			· -					
PE	Clay	TOS	52	0	0	0	0	37	71		
	Wax	TOS	113	0	0	0	0	25	83		

Barrier coating	Repellent carrier	Closure	Initial piperonyl butoxide deposit	Penetrated bags after-							
	conner			3 mo.	6 mo.	9 mo.	15 mo.	17 то.	19 mo.		
			Mg./sq.ft.	Pct.	Pct.	Pct.	Pct.	Pct.	Pct.		
Glassine	Clay	TOS	50	0	0	0	25	75	100		
	Wax	TOS	74	0	0	0	0	86	100		
Greaseproof	Clay	TOS	59								
-	Wax	TOS	96					•			
Kraft	Clay	TOS	47	0	0	0	0	17	50		
	Wax	TOS	128	0	0	0	0	0	20		
	None	TOS	0	0	0	50	75	100	100		
	None	POM	0						200		
	None	SOT	0	50	100						

 TABLE 13.—Insect penetration of 25-pound, synergized-pyrethrins-treated, triwall bags containing CSM—Continued

'(2C)=2 coatings of saran or PVA; all coatings were applied to kraft paper.

 TABLE 14.—Insect penetration of 25-pound, synergized-pyrethrins-treated, trivall bags containing flour

Barrier coating or sheet ²	Repellent carrier	Ciosure	Initial piperonyl butoxide	Penetrated bags after—							
	carrier	Giosure	deposit	3 mo.	6 mo.	9 mo.	15 mo.	17 mo.	19 mo.		
		-	Mg./sq. ft.	Pct.	Pct.	Pct.	Pct.	Pct.	Pct.		
SB/saran (2C)	Clay	TOS	51	0	0	0	0	33	100		
	Clay	TOS	49	0	0	0	0	25	75		
	Clay	TOS	50	0	0	0	0	0	· 30		
SB/saran	Clay	TOS	52	0	0	0	0	50	86		
	Clay	TOS	54	0	0	0	0	10	60		
	Clay	TOS	51	0	0	0	0	0	33		
	Wax	TOS	92	. 		·					
PVA (2C)	Clay	TOS	46	0	0	0	0	60	100		
PVA	Clay	TOS	49	0	0	0	10	67	62		
	Wax	TOS	80	0	0	0	0	90	80		
PE/saran (2C)	Clay	TOS	50	0	0	0	0	67	100		
PE/saran	Clay	TOS	53	0	0	0	0	40	100		
	Wax	TOS	77	0	0	0	0	14	67		
	Wax	POM	36	0	50	0	0	87	88		
	Clay	POM	70	0	0	0	0	43	71		
PE	Clay	TOS	52	0	0	0	10	88	100		
	Wax	TOS	113	0	0	0	0	71	86		
Glassine	Clay	TOS	50	0	0	0	0	62	86		
	Wax	TOS	74	0	0	0	0	0	50		
Greaseproof	Clay	TOS	59					-			
	Wax	TOS	96		.						
Kraft	Clay	TOS	47 -	0	0	0	0	100	100		
	Wax	TOS	128	0	0	0	0	0	38		
	None	TOS	0	100	100	100	100	100	100		
	None	POM	0	75	75	100	100	100	100		
	None	SOT	0	100	100	100	100	100	100		

²(2C)=2 coatings of saran or PVA; all coatings were applied to kraft paper.

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