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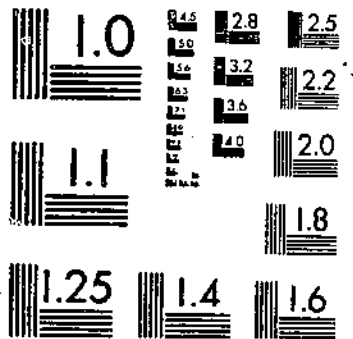
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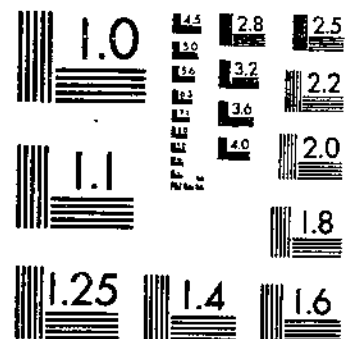
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DEVELOPMENT OF AN INSECT-RESISTANT COTTON BAG

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DEVELOPMENT OF AN INSECT-RESISTANT COTTON BAG

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Changes in food preferences, processing, and marketing have necessitated structural and functional changes in the design of food packages. To see how successfully the packaging industry has met this challenge, one need only walk through a modern supermarket and note the various types, shapes, sizes, colors, and uses of the containers displayed. One challenge the packaging industry has not yet fully met is the production of flexible or semi-rigid packages that resist insect infestation.

Numerous factors have increased the need and urgency for protective food packages. Among the more important are (1) the tremendous increase in the number of processed and prepared foods in our marketing and consumer channels; (2) the high susceptibility of many processed and prepared foods to spoilage or contamination; (3) the increased cost of food items; (4) the increasing demands for available food supplies; (5) the greater movement of processed foods in worldwide commercial systems and governmental programs; and (6) the ever-increasing demand by the public for cleaner, more wholesome foods, which in turn results in increased activities in promulgating and enforcing pure foods laws.

A significant amount of our raw and processed food becomes infested by insects, costing the American public millions of dollars annually (6).³ There are more than 50 species of stored-

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² Norman M. Dennis, chemist, Stored-Product Insects Research and Development Laboratory, assisted in conducting assays of analytical standards, scheduled and supervised the chemical analyses, and reviewed the residue data for this study. Richard H. Guy, biological technician, also of this Laboratory, conducted periodic biological examinations.

³ Italic numbers in parentheses refer to Literature Cited, p 17.

product insects, one or more of which can infest practically every dry plant and animal product used by man. Many of the more important stored-product insects feed on dry cereal products such as flour and cornmeal. Because we produce, consume, and export extremely large quantities of these foods, the insect problem is of serious economic importance.

Stored-product insects can be present wherever food is stored, processed, shipped, or handled. Thus, packaged foods can become infested in a processor's warehouse, in transit, during storage in wholesale and retail outlets, and while in the possession of the consumer. This was illustrated in a study which showed that 30 percent of the cotton bags in a large shipment of cornmeal were infested while awaiting shipment overseas and 56 percent were infested when unloaded at their destination (5). Such losses are needless, as extensive research has shown that dry cereal products can be effectively, practically, and economically protected from insect infestation by using insect-resistant packages. This method is reliable because intact packages will protect from infestation regardless of conditions that may exist after the packages are filled and closed and while they are in various distribution channels.

When research showed that certain insecticide treatments greatly improved the resistance of both paper and cotton bags to insect infestation (1), the paper bag industry, through the Paper Shipping Sack Manufacturers' Association, actively cooperated in developing more effective insect-resistant multiwall paper bags. Prior to overseas shipping tests in 1965, considerable research had been conducted on the development of insect-resistant multiwall paper shipping sacks, but little had been done to investigate possible means of insectproofing cotton bags. Studies were undertaken to determine the rate and extent of the migration of piperonyl butoxide used in insect-resistant treatments on the outer surface of multiwall kraft bags (3). Based on results of these studies, the U.S. Food and Drug Administration established a tolerance of 1 p.p.m. of pyrethrins and 10 p.p.m. of piperonyl butoxide on dry foods and feeds packaged in treated multiwall paper bags (7).

In 1965 an overseas shipping test showed that cornmeal in regular cotton bags was susceptible to insect infestation, whereas cornmeal in the newly developed insect-resistant multiwall kraft paper bags was provided a high degree of protection (5). The textile bag industry immediately realized that the heavy losses due to insect infestation could easily overcome the inherent ad-

vantages of cotton bags, such as strength, ease of closing, and reuse value. There was a sudden urgency for the development of an insect-resistant cotton bag. The potential decrease of cotton used for the manufacture of bags was of concern to the U.S. Department of Agriculture, particularly because of an existing surplus of cotton. Because of the reuse value of the cotton bags to developing countries in providing a source of fabric for clothing and of income through the sale of bags, the Agency for International Development, U.S. State Department, hoped to continue the use of cotton bags for overseas shipment of food.

An accelerated research program to develop an effective insect-resistant cotton bag was undertaken by the Stored-Product Insects Research and Development Laboratory and the Southern Regional Research Laboratory of the USDA's Agricultural Research Service. The plan was to conduct (1) preliminary laboratory tests to select the most promising insect-resistant treatments and bag constructions; (2) a large-scale laboratory test with the dual purpose of evaluating the efficacy of the most promising bag treatments and constructions and of obtaining the necessary biological and pesticide residue data required for approval of the treatment by the U.S. Department of Agriculture and for establishment of residue tolerances by the U.S. Food and Drug Administration; and (3) field tests of an overseas shipment to determine the efficacy and performance of the new insect-resistant cotton bag under actual shipping and storage conditions.

This report describes in detail the preliminary and large-scale tests.

PRELIMINARY CONSIDERATIONS

To minimize the time required to develop an insect-resistant cotton bag, maximum use was made of the knowledge and experience gained from our research on the development of other insect-resistant packages. We had learned that many of the principal species of insects that infest cereal products could easily and quickly penetrate through the weave of the cloth and through the stitching along the side seams and end closures of conventional cotton bags. That is, conventional cotton bags offered little or no physical resistance either to penetrating insects, which make holes in the bag, or to invading insects, which enter only through existing openings.

Two possible ways of making the cloth resistant to insect penetrations were considered: (1) Altering the physical properties by closing the weave and (2) treating the fabric with pyrethrins-

piperonyl butoxide, an insect-repellent treatment found effective for multiwall paper bags (5).

Numerous tests with insect-resistant multiwall paper bags showed that tape-over-stitch (TOS) closures using paper tape treated with pyrethrins-piperonyl butoxide were very effective in making bags insect tight (2). Therefore, this type of construction combined with the pyrethrins-piperonyl butoxide treatment was evaluated in storage tests of small cotton bags.

SMALL-BAG STORAGE TESTS

To determine whether synergized pyrethrins treatments and TOS closures would make cotton bags insect resistant, the small experimental bags listed in table 1 (appendix) were made and tested. The treated and untreated cloth for the test bags was supplied by the Southern Regional Research Laboratory. The bags were constructed, filled, closed, sealed, and tested by the Stored-Product Insects Research and Development Laboratory.

Materials and Procedures

Three types of fabrics were used in these tests to determine the effect of removing noncellulose components on the insect-resistant properties of the treated fabrics. (1) Greige fabric (unprocessed as it comes from the loom) was used without further processing except for the application of synergized pyrethrins. (2) Scoured fabric was prepared by desizing greige fabric by enzymatic treatment to remove the sizing that had been applied to the yarn prior to weaving, then scouring in boiling 2-percent sodium hydroxide to remove the natural waxes, oils, greases, pectins, and other noncellulose materials from the fibers. (3) The bleached fabric was prepared from the scoured fabric by bleaching with hydrogen peroxide to further purify it.

The three types of fabrics were treated by padding with emulsions of synergized pyrethrins prepared with Pyronyl 101, an emulsifiable concentrate containing 1.20 percent of pyrethrins, 12 percent of technical piperonyl butoxide, 15 percent of polyoxyethylene sorbitol ester of mixed fatty acids, and 71.8 percent of petroleum distillates. The treatment was done in a continuous range by immersing the fabric in the treating formulation and passing the treated cloth through squeeze rollers to remove the excess formulation. After two paddings the fabric was dried to about 10 percent of moisture in a forced-draft oven heated to 54°-60° C. Actual deposits of piperonyl butoxide as determined by chemical analysis are shown in table 1. Pyrethrins deposits

were calculated from the 1:8.8 ratio of pyrethrins to actual piperonyl butoxide in the treatment formulation.

One lot of synergized pyrethrins-treated greige fabric was calendered to decrease the size of the openings between threads by passing the fabric between squeeze rollers heated to 120°-180° C. at 0.4 to 0.6 ton per inch of fabric width. This process decreased air porosity from 100-150 cubic feet of air per minute per square foot through the uncalendered fabric to 5-15 cubic feet of air per minute per square foot through the calendered fabric. Chemical analyses indicated a slight loss of insecticide due to the calendering process (table 1).

The cloth was cut into 7- by 24-inch pieces to make bags measuring about 7 by 12 inches, and the sides were sewn with four-ply cotton thread, 3½ stitches per inch. The bags were turned inside out and the stitching and seams of each side of the TOS bags were completely covered with synergized pyrethrins-treated, extensible 70-pound kraft tape, using a heat-activated adhesive. Untreated tape was used on untreated bags. Each bag was filled with flour, the top was stitched, and the closure was covered with kraft tape. To compare the insect resistance of taped and untaped bags, one series of bags was left with all stitched seams (on sides and tops) exposed. Untreated single-ply kraft bags were also included for comparison. Thirty-six bags of each of the 14 variables were made.

The bags were placed in a room containing a heavy infestation of 16 species of stored-product insects (fig. 1). Stacks of three identical bags were randomized in six blocks, each block containing two 3-bag stacks for each periodic examination. After 1, 2, 3, 6, 9, and 12 months of exposure, two stacks of each variable were removed from the exposure room and opened, the flour was examined for insects, and the number of insects was recorded. The number of insect penetrations through each bag was also recorded.

The cotton was analyzed for piperonyl butoxide before the bags were made and after each periodic examination to determine the amount of piperonyl butoxide that remained on the treated fabric. Samples were taken from the bag-to-bag contacting surfaces of the top and bottom bags in each stack. After 12 months the flour was also analyzed to determine the amount of piperonyl butoxide present due to migration from the treated fabric. Throughout this bulletin all data for insecticide content are based on chemical analyses for piperonyl butoxide as there was no reliable method for analyzing the commodities, cotton, or paper for very



FIGURE 1. Small cotton bags filled with flour and exposed to heavy populations of stored-product insects.

small quantities of pyrethrins. The analytical procedure used was a modification of one described by Williams and Sweeney (9).

Results

None of the TOS bags with 6.8 mg. of pyrethrins plus 59.8 mg. of piperonyl butoxide per square foot of cloth or higher deposits on the fabric were infested during 12 months of exposure to intense insect activity (table 1). At the lower treatment level some TOS bags were infested, although the numbers of insects found in these bags were much lower than in treated sewn bags without the overtape (table 2). Half the treated cotton bags with no overtape covering the stitching were infested within 1 month at both treatment levels (table 1). All untreated bags were heavily

infested within 1 month, even though all stitching was completely covered with the overtape. These data show conclusively that the synergized pyrethrins treatment deterred infestation and that insect-tight seams were essential for maximum protection against outside infestation.

Nearly half of the insecticide disappeared from the treated cotton of the various bags within the first month of storage (table 3). However, even after 12 months' storage up to one-third of the original piperonyl butoxide deposit remained on the cotton fabric. Chemical analyses of the flour stored in treated bags for 12 months indicated average residues of up to 42 p.p.m. of piperonyl butoxide (table 4). These results indicated migration of some of the insecticide from the cloth into the flour, reducing the level of the treatment on the bag and producing extensive product contamination.

Comparison of the data from the biological and chemical tests indicated that there was little difference between results obtained with greige, scoured, and bleached fabrics.

LABORATORY INSECTICIDE BARRIER TESTS

Other more extensive investigations have shown that insecticides will migrate from the treated surface of full-sized shipping bags into the packaged commodity (1). To minimize food contamination, it was therefore necessary to develop means of reducing this migration of the insecticide.

Materials and Procedures

Laboratory tests were conducted to compare the effectiveness of waxed and natural kraft paper as barrier sheets. The test unit used is shown in figure 2.

To assemble the unit, synergized pyrethrins-treated cotton cloth was placed on a 6½-inch-square aluminum plate with a 4⅝-inch-diameter hole. The waxed or natural kraft barrier sheet was then placed on the cloth, followed by a cylinder 6 inches in diameter and seven-sixteenths inch high, which was then filled with 120 grams of flour. A barrier sheet was placed over the flour, followed by a sheet of treated cotton cloth and another aluminum plate. The assembled unit was then secured with four binder clips and stored on edge in a room maintained at 26.7° C. and 65-percent relative humidity. Units identical to these but without barrier sheets were included as controls.

After 1, 2, 4, and 6 months, duplicate units of each type were

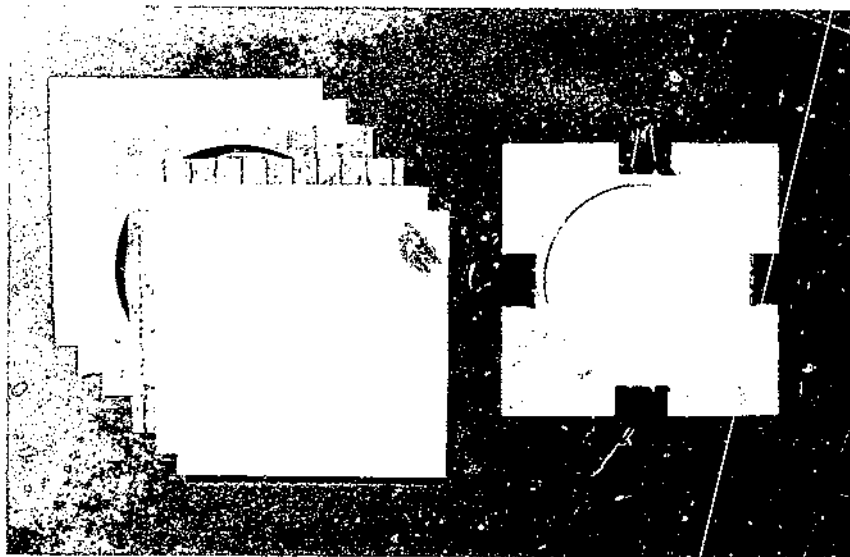


FIGURE 2.—Laboratory device to determine effectiveness of barriers to migration of insecticides from treated cotton into flour.

disassembled and the flour was analyzed for piperonyl butoxide. The effectiveness of the barrier sheets was determined by comparing the residues of piperonyl butoxide found in the flour.

Results

Results of these tests (table 5) indicated that both waxed kraft and natural kraft paper do protect flour against piperonyl butoxide contamination, but that waxed kraft is a more effective barrier than the natural kraft paper. The results obtained from these tests were sufficiently promising to warrant continuing the investigations utilizing commercial-size bags.

LARGE-BAG STORAGE TESTS

Comprehensive large-scale exposure tests of 100-pound bags were conducted to determine—

(1) The efficacy of pyrethrins-piperonyl butoxide treatments on variously constructed cotton bags against insect infestation.

(2) The piperonyl butoxide residue in selected cereal products resulting from 3, 6, 9, and 12 months' storage in treated bags.

(3) The piperonyl butoxide deposit remaining on the various

test bags containing three commodities after 3, 6, 9, and 12 months' storage.

The following types of bags (with indicated code number in table 6) were included in the large-bag storage tests:

(1) Insect-resistant-treated (IRT) cotton bag without liner, with sewn seams and end closures (3-100, 4-50, 4-100, 7-40, and 7-80).

(2) IRT cotton bag without liner, with seams and end closures overtaped with IRT kraft tape heat-sealed over the stitching (T-7-40 and T-7-80).

(3) IRT cotton bag with waxed, creped kraft liner ridge-laminated to the fabric, cemented longitudinal seam, and tape-over-stitch (TOS) end closures (5-50 and 5-100).

(4) Untreated cotton bag without liner, with sewn seams and end closures (1-0).

(5) Untreated cotton bag with creped kraft liner ridge-laminated to the fabric, cemented seam, and TOS end closures (6-0).

(6) IRT multiwall kraft bag with pasted-open-mouth (POM) closures (8-50).

(7) IRT multiwall kraft bag with TOS closures (9-50).

(8) Untreated multiwall kraft bag with stitch-over-tape (SOT) closures (9-0).

Description of Cotton Bags

Fabric for the cotton bags was prepared under the direction of the Southern Regional Research Laboratory. Greige cotton fabrics of 36- or 40-inch widths were used in these tests.

All fabric was treated with an emulsion containing various combinations of synergized pyrethrins emulsifiable concentrate, wax emulsion, polyalkylene glycol, an emulsifier, and water, as shown in table 7. Fabric for bags 3-100, 4-50, 4-100, 5-50, and 5-100 was treated about 10 months before the bags were filled. In treating the fabric the emulsion was applied as a bead or puddle behind a knife blade on a 45-inch textile tenter frame. The knife blade removed excess emulsion from the fabric, which was then dried to about 10-percent moisture content. Intended deposits were obtained by adjusting the concentrations of the various components in the treatment emulsion (table 7). The fabric for bags 3-100, 5-50, and 5-100 was calendered prior to being coated to determine the effect calendering would have on insect resistance of the treated cotton.

Fabric for bags 7-40, 7-80, T-7-40, and T-7-80 was treated on a 50-inch padder about 2 months before the bags were filled.

Excess insecticide emulsion was squeezed from the fabric with wringer rolls to produce the intended deposits. After a second immersion and squeezing, the treated fabric was passed through a tenter frame heated at 116°–149° C. to reduce the fabric moisture to about 10 percent. The treated fabric was calendered with a pressure of 0.6 ton per inch of fabric at ambient temperature, reducing air porosities from about 150 cubic feet per minute per square foot through the uncalendered fabric to 30–45 cubic feet per minute through the calendered fabric.

The hydroxyethyl cellulose in the treatment formulation was utilized to control the viscosity of the emulsions, whereas the polyalkylene glycol textile lubricant was intended to minimize the loss of the insecticide from the treated cotton over long storage periods. The wax was an absorbent carrier for the synergized pyrethrins.

Bags 5–50, 5–100, and 6–0 were fabricated with waxed, kraft paper liners, which were pleated, creped, and ridge-laminated to the cotton fabric. Longitudinal seams on these bags were fully glued with at least a 1-inch lap, utilizing a latex adhesive. All sewing thread on treated bags was soaked in solutions of synergized pyrethrins, and all kraft paper tape for TOS closures was treated with synergized pyrethrins.

Description of Paper Bags

Tape-over-stitch (TOS) bags were flat-tube, sewn-bottom, open-mouth type with the inner wall of class A heavy-duty, shipping-sack kraft paper, plain, meeting the requirements of Federal Specification UU–S–48C. The outer wall was treated with pyrethrins and piperonyl butoxide in accordance with Military Specification MIL–I–21330 and was of class B heavy-duty, shipping kraft paper, plain, wet strength, meeting the requirements of Federal Specification UU–S–48C. The bag had five plies, of which the four inner ones were of 50-pound basis weight and the fifth or outer ply was of 60-pound nominal basis weight. The longitudinal seam of the outer wall of each bag was glued so that there was no more than three-sixteenths inch of unglued edge on the outer surface of the bag.

Top and bottom closures were tightly sealed by covering the stitches along the top and bottom of the bag with 70-pound basis weight extensible kraft tape, the outer surface of which had been treated for insect repellency. The tape covering the top and bottom ends of the bag overlapped the stitches to provide at least one-half inch of bonded area between the stitch line and the edge

of the tape. The ends of the tape extended from the sides of the bag for one-half to 2 inches to enclose and cover the cutoff stitching. The tape was applied to the outer wall by means of a hot-melt resin adhesive so that there was no more than three-sixteenths inch of unbonded edge of the tape.

Stitch-over-tape (SOT) bags were similarly constructed but with no synergized pyrethrins treatment on the outer ply. The top and bottom closures on these bags consisted of stitching over creped kraft tape.

Pasted-open-mouth (POM) bags also had five plies with the outer ply treated for insect repellency, but the bags were gusseted and the pasted-open-mouth had stepped ends. The top and bottom of each bag were folded and sealed with a thermoplastic adhesive to the opposite face of the bag. The outer wall of each top and bottom foldover flap was stepped to extend beyond all inner walls to provide a positive seal over the ends of the inner walls. To prevent harboring of insects under the glued flaps there was no more than 3/16-inch unbonded edge beyond the adhesive line.

Procedures

Before filling the bags, the flour and cornmeal were fumigated under a tarpaulin with methyl bromide to assure that the commodities were insect free at the time of packing. Lots of each type of test bags except bags 9-50, as shown in table 8, were filled with the enriched all-purpose flour, extra-fine-ground degermed yellow cornmeal, or regular enriched yellow cornmeal. All the TOS multiwall kraft bags 9-50 were filled with, regular cornmeal.

The filled bags were placed in four-bag stacks in a room heavily infested with the following 16 species of stored-product insects: Black carpet beetle (*Attagenus megatoma* (F.)) cadelle (*Tenebroides mauritanicus* (L.)), cigarette beetle (*Lasioderma serricornis* (F.)), confused flour beetle (*Tribolium confusum* Jacquelin duVal), flat grain beetle (*Cryptolestes pusillus* (Schönherr)), furniture carpet beetle (*Anthrenus flavipes* Le Conte), granary weevil (*Sitophilus granarius* (L.)), Indian meal moth (*Plodia interpunctella* (Hübner)), lesser grain borer (*Rhyzopertha dominica* (F.)) maize weevil (*Sitophilus zeamais* Motschulsky), merchant grain beetle (*Oryzaephilus mercator* (Fauvel)), red flour beetle (*Tribolium castaneum* (Herbst)), rich weevil (*Sitophilus oryzae* (L.)), sawtoothed grain beetle (*Oryzaephilus surinamensis* (L.)), *Trogoderma glabrum* (Herbst), and *Trogoderma inclusum* LeConte.

The stacks of bags were randomized in blocks, each block con-

taining the bags for one periodic examination. Because of space limitations the bags containing the two types of cornmeal were stored in a room adjacent to the room containing the bagged flour. The bags from one stack of each test variable were examined after 3, 6, 9, and 12 months' exposure. One stack of bags of each test variable was used at each periodic examination.

To examine the contents, the surfaces of all four bags were thoroughly brushed and vacuum cleaned to remove all insects from the exterior. The two middle bags were opened and the contents screened; the insects thus collected were identified and counted. After samples were removed from the top and bottom bags of each stack for chemical analyses, as described in the following section, the remaining contents of each bag were processed in the same manner as the other two. All plies of each bag were examined for insect penetrations.

To obtain surface samples for chemical analysis of the commodities from the two middle bags in each stack, all plies were cut the length of the bag and across the width at each end. The plies were folded back without disturbing the contents and a flat scoop was used to obtain a 1-inch-deep surface sample of the commodity. The sample was placed in a paper bag and thoroughly mixed; subsamples were placed in two mason jars. Duplicate composite samples from the entire contents of each of the two middle bags of each stack were taken at the exit port of the sifter.

The various plies of the treated bags were also analyzed for piperonyl butoxide at each periodic examination. Samples of each bag were obtained from the upper surfaces of all bags in the stack and each ply was analyzed separately. The commodities and bags were analyzed for piperonyl butoxide by a modification of the method of Williams and Sweeney (9).

Results

The detailed data on the percentage of bags infested, average number of insects per bag, and average number of insect penetrations per bag are shown in tables 8, 9, and 10, respectively. The treated cotton bags with the waxed-paper liner (5-50 and 5-100) showed a very high level of resistance to infestation through 9 months of storage, about equivalent to the insect-resistant multiwall paper bags (8-50 and 9-50) used as standards for comparison (table 8). After 12 months' storage the treated, lined cotton bags with the lower level of treatment (5-50) had lost most of their effectiveness, as had the treated paper bags. (8-50 and 9-50).

Infestation and penetration

Most treated conventional cotton bags with sewn seams and closures (4-50, 7-40) were infested within 3 months. The only exceptions were some of those with the high levels of pyrethrins-piperonyl butoxide (3-100, 4-100, 7-80). Few of the bags in even the latter group were free of infestation after the 6-month exposures.

The addition of polyalkylene glycol apparently had some beneficial effects, for, as shown by the data (7-80 and T-7-80), the bags with this additive were about as effective as those treated with a higher level of the pyrethrins-piperonyl butoxide (4-100) but without the polyalkylene glycol. The data also show no significant difference between the number of infested cotton bags, with the TOS seams and closures (T-7-40 and T-7-80) and those with the conventional seams (7-40 and 7-80), indicating that the insects succeeded in infesting these bags through the weave of the fabric.

The data in table 9, which show the number of insects in the various bags during 12 months' storage, corroborate the data discussed previously. Based on the number of insects found in the bags, the cotton bags with the waxed-paper liner (5-50 and 5-100) were as effective in protecting the three commodities against infestation as were the standard insect-resistant multiwall paper bags (8-50 and 9-50) (fig. 3). After 12 months' storage the paper-lined cotton bags with comparable treatment had fewer insects than the treated multiwall paper bags with the POM closures. The cotton bags (no barrier sheets) with high levels of pyrethrins-piperonyl butoxide, including those with polyalkylene glycol additive and taped seams (T-7-80), offered a little more protection against infestation than did the untreated checks.

The protection against insect penetration rendered by the pyrethrins-piperonyl butoxide treatment is evident from the data in table 10. The higher the level of treatment, the fewer the number of penetrations. Protection against insect penetration appeared to be provided by the untreated paper-lined cotton bag (6-0), which contained fewer insect penetrations after 3 months than either the untreated unlined conventional bag (1-0) or the untreated multiwall kraft bag (9-0).

Piperonyl butoxide residues in commodities

As shown in table 11, piperonyl butoxide migrated from the package into the commodity in all treated bags. The only cotton

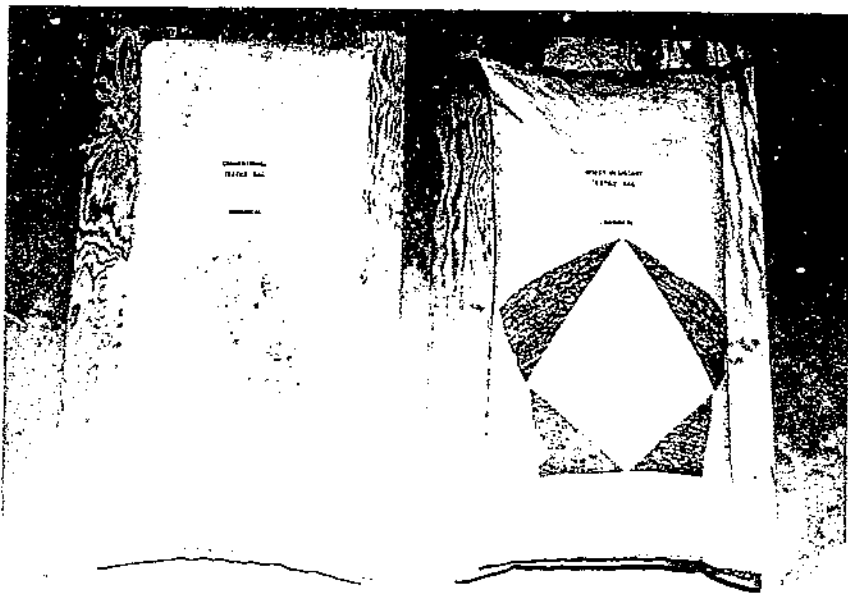


FIGURE 3.—Conventional untreated cotton bag (left) and treated cotton bag with waxed kraft liner (right) after 9 months' exposure to insects.

bags that were resistant to insects (table 8) and produced less than 10 p.p.m. of piperonyl butoxide in all the commodities (composite samples) during the entire 12-month storage period were the waxed-paper-lined cotton bags (5-50). Even the waxed-paper-lined cotton bags with 12.2 mg. of pyrethrins and 107 mg. of piperonyl butoxide per square foot (5-100) produced residues just slightly above the 10-p.p.m. level. However, residues produced by all but one (7-10) of the unlined cotton bags (3-100, 1-50, 1-100, 7-80) that had treated fabric in direct contact with the commodity far exceeded tolerances of 10 p.p.m. established for food packed in synergized pyrethrins-treated multiwall kraft bags. These data therefore showed that the waxed-paper liner was very effective in minimizing the migration of piperonyl butoxide from the treated cotton to the food inside the bags.

Table 12 shows the piperonyl butoxide residues in the 1-inch surface samples taken from the commodities lying next to the bag walls. The residues appearing in these samples were rather high, and a comparison of these with residues found in the composite samples of the entire contents of the bag (table 11) indicates that most of the piperonyl butoxide remains in the layer of commodity lying next to the bag wall.

Piperonyl butoxide deposits remaining on packaging materials

The data in table 13 show that there was a radical drop in the amount of piperonyl butoxide on the conventional cotton bags (3-100, 4-50, 4-100, 7-80) within 3 months. This is a consequence of the extensive loss due to migration, resulting in high residues in the commodities within these bags. The retention of the piperonyl butoxide by the waxed-paper-lined cotton bags was outstanding. After 12 months, 20 to 37 percent of the original amount was still present on these bags, whereas generally less than 9 percent remained on the conventional cotton bags and less than 16 percent on the multiwall paper bags. This is further evidence that the waxed-paper liner was an effective barrier for reducing migration of the piperonyl butoxide into the commodities.

DISCUSSION

The data and conclusions from these investigations were presented to the U.S. Food and Drug Administration (FDA) in a petition for a tolerance as provided in the Food, Drug, and Cosmetic Act. The petition was granted with certain specified limitations (8). According to the promulgated regulations, the cotton must be treated at a rate not to exceed 55 mg. of piperonyl butoxide and 5.5 mg. of pyrethrins per square foot of cloth. The treated bags must be of 50-pound or more capacity must be constructed with waxed-paper liners, and can be used to package dry foods containing no more than 4 percent of fat.

FDA's approval of a tolerance cleared the way for a large-scale test to evaluate the efficacy of paper-lined insect-resistant-treated cotton bags under practical shipping and storage conditions. Such an experiment involving 600,000 pounds of cornmeal shipped to and stored in the Philippines for 6 months was conducted by Highland and others (4). The results of the shipping and storage tests confirmed the findings of the static large-scale tests reported here.

SUMMARY

Preliminary tests were conducted to select the most promising insect-resistant treatments and bag constructions for protecting commodities in textile bags. Bags of the selected kinds were then subjected to large-scale laboratory tests to evaluate their efficacy and to obtain pesticide residue data.

The pyrethrins-piperonyl butoxide treatment on the cotton bag definitely provided some protection against insect infestation. However, the level of insecticide needed to adequately protect foods in unlined conventional cotton bags produced residues in the commodity far in excess of the 10 p.p.m. tolerance approved by the U.S. Food and Drug Administration for piperonyl butoxide residues in foods packaged in multiwall paper bags.

Cotton bags with the synergized pyrethrins treatment, waxed-paper liner, cemented longitudinal seams, and TOS (tape-over-stitch) closures (5-50) provided excellent protection against infestation for up to 9 months. The degree of protection was about equal to that provided by the treated multiwall paper bags with TOS and POM closures. The waxed-paper liner in treated cotton bags helped materially in minimizing the migration of piperonyl butoxide from the treated cotton cloth into the commodity inside the bag. Therefore these tests conclusively showed that a cotton bag can be made into an effective insect-resistant container for susceptible dry foods.

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| | | | | | | | | |
|-----------------------|---|---|-----|------------------|-----|-----|-----|-----|
| Sewn, scoured..... | 0 | 0 | 100 | ⁵ 100 | 100 | 100 | 100 | 100 |
| TOS, Kraft paper..... | 0 | 0 | 100 | 100 | 67 | 100 | 100 | 100 |

¹ Separate set of bags were opened at each periodic inspection.

⁴ 5 bags.

² Only this fabric was calendered after treatment with synergized pyrethrins.

⁵ 3 bags.

³ Many tapes were loose on these bags.

TABLE 2.—Average number of insects in small flour-filled treated cotton bags exposed to insects; results are based on inspection of 6 bags except as noted ¹

| Type of seams and type of cotton or paper | Initial deposit | | Insects in infested bags after indicated months | | | | | |
|---|-----------------|--------------------|---|--------|--------|--------|--------|--------|
| | Pyrethrins | Piperonyl butoxide | 1 | 2 | 3 | 6 | 9 | 12 |
| | Mg. per sq. fl. | Mg. per sq. fl. | Number | Number | Number | Number | Number | Number |
| TOS: | | | | | | | | |
| Greige..... | 3.7 | 32.5 | 0 | 0 | <1 | 0 | <1 | 60 |
| Greige ² | 3.5 | 30.8 | 0 | 0 | 0 | 0 | 23 | 0 |
| Scoured ³ | 3.5 | 30.8 | <1 | <1 | <1 | 5 | 2 | 4.4 |
| Bleached..... | 3.6 | 31.6 | 0 | 0 | <1 | <1 | <1 | 2.2 |
| Sewn, scoured..... | 3.5 | 30.8 | 3.6 | 0 | <1 | 108 | 81 | 43.6 |
| TOS: | | | | | | | | |
| Greige..... | 6.8 | 59.8 | 0 | 0 | 0 | 0 | 0 | 0 |
| Scoured..... | 6.9 | 60.7 | 0 | 0 | 0 | 0 | 0 | 0 |
| Bleached..... | 7.9 | 69.5 | 0 | 0 | 0 | 0 | 0 | 0 |
| Sewn, scoured..... | 6.9 | 60.7 | 4.0 | 7.0 | 0 | 38 | 4.8 | 2.2 |
| TOS: | | | | | | | | |
| Greige..... | 0 | 0 | 402 | 843 | 2,354 | 275 | 224 | 977 |
| Scoured..... | 0 | 0 | 320 | 264 | 1,244 | 501 | 407 | 682 |
| Bleached..... | 0 | 0 | 297 | 190 | 2,460 | 405 | 908 | 713 |

| | | | | | | | | |
|-----------------------|---|---|-----|------------------|-------|-----|-----|-------|
| Sewn, scoured..... | 0 | 0 | 746 | ⁶ 154 | 1,482 | 611 | 322 | 1,092 |
| TOS, Kraft paper..... | 0 | 0 | 697 | 326 | 1,069 | 540 | 851 | 830 |

¹ Separate sets of bags were opened at each periodic examination.

² Only this fabric was calendered after treatment with synergized pyrethrins.

³ Many tapes loose on these bags.

⁴ 5 bags.

⁵ 3 bags.

TABLE 3.—Persistence of piperonyl butoxide on small cotton bags filled with flour and aged for 12 months; data are from 4 bags at each examination

| Type of seams and type of cotton | Initial deposit | | Average piperonyl butoxide deposit on cotton after indicated months | | | | | |
|----------------------------------|-----------------|--------------------|---|-----------------|-----------------|-----------------|-----------------|-----------------|
| | Pyrethrins | Piperonyl butoxide | 1 | 2 | 3 | 6 | 9 | 12 |
| | Mg. per sq. ft. | Mg. per sq. ft. | Mg. per sq. ft. | Mg. per sq. ft. | Mg. per sq. ft. | Mg. per sq. ft. | Mg. per sq. ft. | Mg. per sq. ft. |
| TOS: | | | | | | | | |
| Greige..... | 3.7 | 32.5 | 13.6 | 9.6 | 11.1 | 6.7 | 6.4 | 5.9 |
| Greige ¹ | 3.5 | 30.8 | 11.9 | 10.2 | 10.2 | 6.9 | 6.5 | 6.1 |
| Scoured..... | 3.5 | 30.8 | 15.5 | 10.6 | 10.7 | 8.4 | 7.9 | 7.1 |
| Bleached..... | 3.6 | 31.6 | 15.3 | 11.9 | 10.0 | 9.3 | 8.8 | 7.7 |
| Sewn, scoured..... | 3.5 | 30.8 | 16.1 | 13.1 | 12.0 | 8.7 | 8.1 | 7.4 |
| TOS: | | | | | | | | |
| Greige..... | 6.8 | 59.8 | 23.8 | 16.7 | 14.4 | 16.0 | 11.9 | 10.6 |
| Scoured..... | 6.9 | 60.7 | 24.5 | 22.3 | 21.6 | 21.3 | 19.4 | 16.9 |
| Bleached..... | 7.9 | 69.5 | 27.8 | 20.9 | 24.9 | 21.9 | 21.7 | 19.5 |
| Sewn, scoured..... | 6.9 | 60.7 | 29.6 | 26.9 | 24.7 | 21.6 | 21.3 | 19.9 |

¹ Only this fabric was calendered after treatment with synergized pyrethrins.

TABLE 4.—*Piperonyl butoxide in flour from small scoured-cotton bags after 12 months' exposure; data are from inspection of 3 bags of each type*

| Type of seams | Initial deposit | | Piperonyl butoxide residue in flour after 12 months | |
|---------------|-----------------|--------------------|---|---------|
| | Pyrethrins | Piperonyl butoxide | Range | Average |
| | Mg. per sq. ft. | Mg. per sq. ft. | P.p.m. | P.p.m. |
| Sewn..... | 3.5 | 30.8 | 20.5-22.4 | 21 |
| Do..... | 6.9 | 60.7 | 35.2-39.6 | 37 |
| TOS..... | 6.9 | 60.7 | 40.4-44.8 | 42 |

TABLE 5.—*Piperonyl butoxide in flour exposed to synergized pyrethrins-treated cloth with and without barrier sheets; results are from analysis of flour from duplicate migration units*

| Initial piperonyl butoxide deposit on cotton cloth (mg. per sq. ft.) | Barrier sheet | Average residue of piperonyl butoxide after indicated months | | | |
|--|----------------------|--|--------|--------|--------|
| | | 1 | 2 | 4 | 6 |
| | | P.p.m. | P.p.m. | P.p.m. | P.p.m. |
| 31.6..... | { Waxed kraft..... | 2.7 | 6.4 | 13.0 | 10.4 |
| | { Natural kraft..... | 5.7 | 7.2 | 19.0 | 17.6 |
| | { None..... | 17.0 | 30 | 42 | 33 |
| 69.5..... | { Waxed kraft..... | 5.2 | 13.7 | 22 | 23 |
| | { Natural kraft..... | 11.8 | 20 | 39 | 48 |
| | { None..... | 39 | 39 | 82 | 67 |

TABLE 6.—Types of large bags for storage tests of insect-resistant-treated cotton bags

| Bag code No. | Initial deposit | | Construction | | | Calendered |
|-----------------------------------|------------------------|------------------------|-------------------|-----------------|-------|------------|
| | Pyrethrins | Piperonyl butoxide | Longitudinal seam | Ends | Liner | |
| | <i>Mg. per sq. ft.</i> | <i>Mg. per sq. ft.</i> | | | | |
| COTTON BAGS | | | | | | |
| 1-0 | 0 | 0 | Sewn | Sewn | No | No. |
| 3-100 | 11.6 | 102 | do | do | No | Yes. |
| 4-50 | 4.6 | 40 | do | do | No | No. |
| 4-100 | 12.9 | 113 | do | do | No | No. |
| 7-40 | ¹ 2.7 | 24 | do | do | No | Yes. |
| 7-80 | ¹ 7.2 | 63 | do | do | No | Yes. |
| T-7-40 | ¹ 2.7 | 24 | Sewn, taped. | Sewn, taped. | No | Yes. |
| T-7-80 | ¹ 7.2 | 63 | do | do | No | Yes. |
| 5-50 | 6.2 | 54 | Cemented | Sewn, taped. | Yes | Yes. |
| 5-100 | 12.2 | 107 | do | do | Yes | Yes. |
| 6-0 | 0 | 0 | do | do | Yes | Yes. |
| MULTIWALL KRAFT BAGS ² | | | | | | |
| 8-50 | 6.4 | 56 | | POM | | |
| 9-50 | 7.3 | 64 | | TOS | | |
| 9-0 | 0 | 0 | | SOT | | |

¹ Treatment of cotton for these bags includes a polyalkylene glycol additive.

² 5-ply, 260-pound basis weight.

TABLE 7.—*Synergized pyrethrins emulsion formulations used to treat cotton fabrics for long-term storage tests of insect-resistant-treated cotton bags*

| Component | Amount of component in treatment emulsion applied to bags of indicated code No. | | | | | | |
|---|---|-------------|-------------|-------------|-------------|-------------|-------------|
| | 5-50 | 4-50 | 3-100 | 5-100 | 4-100 | 7-40 | 7-80 |
| | <i>Pct.</i> | <i>Pct.</i> | <i>Pct.</i> | <i>Pct.</i> | <i>Pct.</i> | <i>Pct.</i> | <i>Pct.</i> |
| Synergized pyrethrins emulsifiable concentrate ¹ | 23.3 | 7.0 | 23.6 | 23.6 | 14.0 | 2.4 | 4.8 |
| Wax solids ² | 12.4 | 6.2 | 11.9 | 11.9 | 12.4 | 0 | 0 |
| Hydroxyethyl cellulose ³ | .8 | .8 | .8 | .8 | .8 | .5 | .5 |
| Water..... | 63.5 | 85.9 | 63.6 | 63.6 | 72.7 | 93.2 | 90.8 |
| Polyalkylene glycol ⁴ | 0 | 0 | 0 | 0 | 0 | 3.8 | 3.8 |
| Triton X-100 ⁵ | 0 | 0 | 0 | 0 | 0 | .1 | .1 |

¹ Pyronyl 101, Prentiss Drug & Chemical Co.

² Mobilicer HM wax emulsion, Socony Mobil Oil Co., Inc.

³ Cellosize QP 100-M, Union Carbide Corp.

⁴ UCON 50-HB-260, Union Carbide Corp.

⁵ Rohm and Haas Co.

TABLE 8.—Percentage of experimental bags infested after exposure to insects for 3, 6, 9, and 12 months; data are averages for 4 bags at each examination except as noted

| Type of bag and code No. | Initial deposit | | Exposure period | Infested bags of— | | | Average |
|--------------------------|------------------------|------------------------|-----------------|-------------------|------------------|------------------|-------------|
| | Pyrethrins | Piperonyl butoxide | | Flour | Cornmeal | | |
| | | | Pct. | | Pct. | Pct. | Pct. |
| | <i>Mg. per sq. ft.</i> | <i>Mg. per sq. ft.</i> | <i>Months</i> | <i>Pct.</i> | <i>Pct.</i> | <i>Pct.</i> | <i>Pct.</i> |
| Cotton, sewn closures: | | | | | | | |
| 3-100----- | 11.6 | 102 | 3 | 0 | 50 | ¹ 66 | 36 |
| | | | 6 | 100 | ¹ 100 | 100 | 100 |
| | | | 9 | 100 | 100 | 100 | 100 |
| | | | 12 | 75 | ² 100 | ¹ 100 | 89 |
| 4-100----- | 12.9 | 113 | 3 | 25 | 100 | ¹ 33 | 54 |
| | | | 6 | 0 | ¹ 66 | ¹ 66 | 40 |
| | | | 9 | 50 | ¹ 33 | ¹ 100 | 50 |
| | | | 12 | 100 | ² 50 | ² 100 | 87 |
| 4-50----- | 4.6 | 40 | 3 | 100 | 100 | 100 | 100 |
| | | | 6 | 100 | 100 | 100 | 100 |
| | | | 9 | 100 | ¹ 100 | 100 | 100 |
| | | | 12 | ¹ 100 | ¹ 100 | 100 | 100 |
| 7-40----- | 2.7 | 24 | 3 | 100 | 100 | 100 | 100 |
| | | | 6 | 100 | 100 | 100 | 100 |
| | | | 9 | 100 | ¹ 100 | 100 | 100 |
| | | | 12 | 100 | ² 100 | 100 | 100 |

| | | | | | | | |
|---|------|-----|----|-----|------------------|------------------|-----|
| 7-80----- | 7.2 | 63 | 3 | 0 | 100 | ¹ 66 | 54 |
| | | | 6 | 25 | 75 | 100 | 66 |
| | | | 9 | 25 | 100 | 100 | 75 |
| | | | 12 | 100 | 75 | 100 | 91 |
| 1-0----- | 0 | 0 | 3 | 100 | ² 100 | ¹ 100 | 100 |
| | | | 6 | 100 | ² 100 | ² 100 | 100 |
| | | | 9 | 100 | 100 | 100 | 100 |
| | | | 12 | 100 | 100 | ¹ 100 | 100 |
| Cotton, TOS closures: | | | | | | | |
| T-7-40----- | 2.7 | 24 | 3 | 100 | ² 100 | ³ 100 | 100 |
| | | | 6 | 100 | ² 100 | ² 100 | 100 |
| | | | 9 | 100 | ² 100 | ² 100 | 100 |
| | | | 12 | 100 | ² 100 | ² 100 | 100 |
| T-7-80----- | 7.2 | 63 | 3 | 0 | ² 100 | ¹ 66 | 44 |
| | | | 6 | 0 | ¹ 100 | 100 | 63 |
| | | | 9 | 75 | ² 50 | ¹ 100 | 77 |
| | | | 12 | 100 | ¹ 100 | ² 100 | 100 |
| Cotton, waxed-paper liner, TOS closures, cemented seam: | | | | | | | |
| 5-50----- | 6.2 | 54 | 3 | 0 | ¹ 0 | 50 | 18 |
| | | | 6 | 25 | ¹ 33 | ¹ 33 | 30 |
| | | | 9 | 25 | ¹ 33 | ¹ 33 | 33 |
| | | | 12 | 75 | ² 100 | ¹ 100 | 88 |
| 5-100----- | 12.2 | 107 | 3 | 0 | 25 | 0 | 8 |
| | | | 6 | 0 | 0 | 0 | 0 |
| | | | 9 | 0 | ¹ 0 | ¹ 0 | 0 |
| | | | 12 | 0 | ² 0 | 0 | 0 |

See footnotes at end of table.

TABLE 8.—Percentage of experimental bags infested after exposure to insects for 3, 6, 9, and 12 months; data are averages for 4 bags at each examination except as noted—Continued

| Type of bag and code No. | Initial deposit | | Exposure period | Infested bags of— | | | Average |
|--|------------------------|------------------------|-----------------|-------------------|------------------|------------------|-------------|
| | Pyrethrins | Piperonyl butoxide | | Flour | Cornmeal | | |
| | | | Pct. | | Pct. | Pct. | Pct. |
| Cotton, wax-paper liner, TOS closures, cemented seam— Continued | <i>Mg. per sq. ft.</i> | <i>Mg. per sq. ft.</i> | <i>Months</i> | <i>Pct.</i> | <i>Pct.</i> | <i>Pct.</i> | <i>Pct.</i> |
| 6-0..... | 0 | 0 | 3 | 100 | 100 | 100 | 100 |
| | | | 6 | 100 | 100 | 100 | 100 |
| | | | 9 | 100 | 100 | 100 | 100 |
| | | | 12 | 100 | 100 | ¹ 100 | 100 |
| Kraft, POM closures, 8-50..... | 6.4 | 56 | 3 | 25 | ² 33 | ¹ 0 | 22 |
| | | | 6 | ¹ 33 | 0 | ² 0 | 10 |
| | | | 9 | 25 | ² 100 | ¹ 33 | 44 |
| | | | 12 | ¹ 100 | ¹ 67 | ³ 100 | 86 |
| Kraft, TOS closures, 9-50..... | 7.3 | 64 | 3 | ----- | ----- | 0 | 0 |
| | | | 6 | ----- | ----- | 0 | 0 |
| | | | 9 | ----- | ----- | ¹ 0 | 0 |
| | | | 12 | ----- | ----- | ¹ 67 | 67 |

| | | | | | | | | |
|-------------------------------|---|---|---|----|-----|------------------|-----|-----|
| Kraft, SOT closures, 9-0----- | 0 | 0 | } | 3 | 100 | 100 | 100 | 100 |
| | | | | 6 | 100 | 100 | 100 | 100 |
| | | | | 9 | 100 | 100 | 100 | 100 |
| | | | | 12 | 100 | ¹ 100 | 100 | 100 |

¹ 3 bags examined.

² 2 bags examined.

³ 1 bag examined.

TABLE 9.—Average number of insects in experimental bags exposed to insects for 3, 6, 9, and 12 months; data are averages for 4 bags at each examination except as noted

| Type of bag and code No. | Initial deposit | | Exposure period | Insects per bag in— | | |
|--------------------------|-----------------|--------------------|-----------------|---------------------|--------------------|---------------------|
| | Pyrethrins | Piperonyl butoxide | | Flour | Cornmeal | |
| | Mg. per sq. ft. | Mg. per sq. ft. | Months | | Number | Degermed |
| Cotton, sewn closures: | | | | | | |
| 3-100----- | 11.6 | 102 | 3 | 0 | 1 | ¹ 161 |
| | | | 6 | 230 | ¹ 132 | 28 |
| | | | 9 | 734 | 11 | 4,086 |
| | | | 12 | 8,170 | ² 38 | ¹ 15,107 |
| 4-100----- | 12.9 | 113 | 3 | .5 | 57 | ¹ 704 |
| | | | 6 | 0 | ¹ 27 | ¹ 40 |
| | | | 9 | 3 | ¹ 64 | ¹ 2,049 |
| | | | 12 | 38,009 | ² 3,040 | ² 504 |
| 4-50----- | 4.6 | 40 | 3 | 426 | 963 | 617 |
| | | | 6 | 288 | 363 | 4,508 |
| | | | 9 | 32,280 | ¹ 3,884 | 16,535 |
| | | | 12 | ¹ 55,524 | ¹ 8,577 | 311,466 |
| 7-40----- | 2.7 | 24 | 3 | 60 | 127 | 3,051 |
| | | | 6 | 41 | 1,593 | 5,975 |
| | | | 9 | 17,231 | ¹ 2,832 | 14,489 |
| | | | 12 | 42,352 | ² 4,683 | 257,743 |

| | | | | | | | |
|---|------|-----|---|----|--------|---------------------|----------------------|
| 7-80----- | 7.2 | 63 | { | 3 | 0 | 25 | ¹ 802 |
| | | | | 6 | .5 | 24 | 1,852 |
| | | | | 9 | 1.2 | 119 | 7,198 |
| | | | | 12 | 11,693 | 725 | 178,718 |
| 1-0----- | 0 | 0 | { | 3 | 15,890 | ² 2,950 | ¹ 3,178 |
| | | | | 6 | 46,356 | ² 13,907 | ² 2,688 |
| | | | | 9 | 82,991 | 25,126 | 89,862 |
| | | | | 12 | 64,974 | 171,968 | ¹ 230,040 |
| Cotton, TOS closure : | | | | | | | |
| T-7-40----- | 2.7 | 24 | { | 3 | 79 | ² 309 | ³ 2,080 |
| | | | | 6 | 66 | ² 4,574 | ² 6,392 |
| | | | | 9 | 4,936 | ² 6,670 | ² 36,483 |
| | | | | 12 | 24,390 | ² 12,512 | ² 198,400 |
| T-7-80----- | 7.2 | 63 | { | 3 | 0 | ² 14 | ¹ 7 |
| | | | | 6 | 0 | ¹ 4.6 | 4.2 |
| | | | | 9 | 3 | ² 9 | ¹ 1,351 |
| | | | | 12 | 16 | ² 861 | ² 10,474 |
| Cotton, waxed-paper liner, TOS closures, cemented seam: | | | | | | | |
| 5-50----- | 6.2 | 54 | { | 3 | 0 | ¹ 0 | 1.2 |
| | | | | 6 | .2 | ¹ .6 | ¹ .2 |
| | | | | 9 | .2 | ¹ 747 | ¹ .3 |
| | | | | 12 | 14 | ² 50 | ¹ 3,125 |
| 5-100----- | 12.2 | 107 | { | 3 | 0 | .2 | 0 |
| | | | | 6 | 0 | 0 | 0 |
| | | | | 9 | 0 | ¹ 0 | ¹ 0 |
| | | | | 12 | 0 | ³ 0 | 0 |

INSECT-RESISTANT COTTON BAG

See footnotes at end of table.

TABLE 9.—Average number of insects in experimental bags exposed to insects for 3, 6, 9, and 12 months; data are averages for 4 bags at each examination except as noted—Continued

| Type of bag and code No. | Initial deposit | | Exposure period | Insects per bag in— | | |
|--|-----------------|--------------------|-----------------|---------------------|----------|----------|
| | Pyrethrins | Piperonyl butoxide | | Flour | Cornmeal | |
| | Mg. per sq. ft. | Mg. per sq. ft. | Months | | Number | Degermed |
| Cotton, wax-paper liner, TOS closures, cemented seam—Continued | | | | | | |
| 6-0----- | 0 | 0 | 3 | 262 | 98 | 281 |
| | | | 6 | 4,813 | 4,229 | 1,005 |
| | | | 9 | 13,760 | 3,480 | 20,849 |
| | | | 12 | 30,336 | 22,112 | 80,898 |
| Kraft, POM closures, 8-50----- | 6.4 | 56 | 3 | .2 | .6 | 10 |
| | | | 6 | 1.5 | 0 | 20 |
| | | | 9 | 1 | 121 | 1384 |
| | | | 12 | 21,837 | 15,487 | 52,912 |
| Kraft, TOS closures, 9-50----- | 7.3 | 64 | 3 | ----- | ----- | 0 |
| | | | 6 | ----- | ----- | 0 |
| | | | 9 | ----- | ----- | 10 |
| | | | 12 | ----- | ----- | 555 |

| | | | | | | | |
|-------------------------------|---|---|---|----|--------|--------|---------|
| Kraft, SOT closures, 9-0----- | 0 | 0 | { | 3 | 3,916 | 7,842 | 9,140 |
| | | | | 6 | 25,273 | 17,823 | 14,693 |
| | | | | 9 | 54,346 | 86,245 | 191,105 |
| | | | | 12 | 28,342 | 28,398 | 176,261 |

- ¹ 3 bags examined.
- ² 2 bags examined.
- ³ 1 bag examined.

TABLE 10.—Average number of penetrations in experimental textile bags exposed to insects for 3, 6, 9, and 12 months; data are averages for 4 bags at each examination except as noted

| Type of bag and code No. | Initial deposit | | Exposure period | Penetrations in bags of— | | |
|--------------------------|-----------------|--------------------|-----------------|--------------------------|-----------------|-----------------|
| | Pyrethrins | Piperonyl butoxide | | Flour | Cornmeal | |
| | Mg. per sq. ft. | Mg. per sq. ft. | Months | | Number | Degermed |
| Cotton, sewn closures:— | | | | | | |
| 3-100----- | 11.6 | 102 | 3 | 0 | 0.2 | ¹ 0 |
| | | | 6 | 0 | ¹ 0 | 0 |
| | | | 9 | 0 | 0 | .2 |
| | | | 12 | 0 | ² 1 | ¹ 11 |
| 4-100----- | 12.9 | 113 | 3 | 0 | 0 | ¹ 0 |
| | | | 6 | 0 | ¹ 0 | ¹ .3 |
| | | | 9 | .2 | ¹ 0 | ¹ 0 |
| | | | 12 | 0 | ² 1 | ² .5 |
| 4-50----- | 4.6 | 40 | 3 | .2 | 3.2 | 0 |
| | | | 6 | 1 | 7.5 | 17 |
| | | | 9 | 21 | ¹ 13 | 21 |
| | | | 12 | ¹ 119 | ¹ 29 | 145 |
| 7-40----- | 2.7 | 24 | 3 | 3.7 | 3 | 23 |
| | | | 6 | 4 | 66 | 129 |
| | | | 9 | 78 | ¹ 16 | 39 |
| | | | 12 | 290 | ² 51 | 228 |

| | | | | | | |
|---|------|-----|----|----------------|------------------|------------------|
| 7-80----- | 7.2 | 63 | 3 | 0 | 0 | ¹ 0 |
| | | | 6 | 0 | 0 | 1.7 |
| | | | 9 | .2 | .2 | 0 |
| | | | 12 | 1.0 | .7 | 7 |
| 1-0----- | 0 | 0 | 3 | 168 | ² 31 | ¹ 25 |
| | | | 6 | 188 | ² 111 | ² 84 |
| | | | 9 | 300 | 542 | 272 |
| | | | 12 | 330 | 183 | ¹ 568 |
| Cotton, TOS closures: | | | | | | |
| T-7-40----- | 2.7 | 24 | 3 | .7 | ² 2.5 | ³ 9 |
| | | | 6 | ² 5 | ² 65 | ² 136 |
| | | | 9 | 47 | ² 298 | ² 65 |
| | | | 12 | 160 | ² 250 | ² 260 |
| T-7-80----- | 7.2 | 63 | 3 | 0 | ² 1.5 | ¹ 0 |
| | | | 6 | 0 | ¹ 0 | .2 |
| | | | 9 | .2 | ² .5 | ¹ 13 |
| | | | 12 | 1.0 | ¹ 12 | ² 27 |
| Cotton, waxed-paper liner, TOS closures, cemented seam: | | | | | | |
| 5-50----- | 6.2 | 54 | 3 | 0 | ¹ 0 | 0 |
| | | | 6 | .2 | ¹ .2 | ¹ .3 |
| | | | 9 | 0 | ¹ 5 | ¹ 0 |
| | | | 12 | 1.0 | ² 2.5 | ¹ 16 |
| 5-100----- | 12.2 | 107 | 3 | 0 | 0 | 0 |
| | | | 6 | 0 | 0 | 0 |
| | | | 9 | 0 | ¹ 0 | ¹ 0 |
| | | | 12 | .5 | ³ 0 | 0 |

See footnotes at end of table.

TABLE 10.—Average number of penetrations in experimental textile bags exposed to insects for 3, 6, 9, and 12 months: data are averages for 4 bags at each examination except as noted—Continued

| Type of bag and code No. | Initial deposit | | Exposure period | Penetrations in bags of— | | |
|---|-----------------|--------------------|-----------------|--------------------------|-----------------|--------|
| | Pyrethrins | Piperonyl butoxide | | Flour | Cornmeal | |
| | | | Mg. per sq. ft. | | Mg. per sq. ft. | Months |
| Cotton, wax-paper liner, TOS closures, cemented seams—Continued | | | | | | |
| 6-0----- | 0 | 0 | 3 | 3.5 | 4.5 | 21 |
| | | | 6 | 30 | 39 | 17 |
| | | | 9 | 55 | 63 | 161 |
| | | | 12 | 265 | 615 | >1,000 |
| Kraft, POM closures, 8-50----- | 6.4 | 56 | 3 | .2 | 2.2 | 10 |
| | | | 6 | 10 | 0 | 20 |
| | | | 9 | 0 | 2.5 | 12.3 |
| | | | 12 | 202 | 146 | 525 |
| Kraft, TOS closures, 9-50----- | 7.3 | 64 | 3 | ----- | ----- | 0 |
| | | | 6 | ----- | ----- | 0 |
| | | | 9 | ----- | ----- | 20 |
| | | | 12 | ----- | ----- | 11 |
| Kraft, SOT closures, 9-0----- | 0 | 0 | 3 | 69 | 187 | 62 |
| | | | 6 | 196 | 275 | 121 |
| | | | 9 | 137 | 890 | >1,000 |
| | | | 12 | >1,000 | >1,000 | >1,000 |

¹ 3 bags examined. ² 2 bags examined. ³ 1 bag examined.

TABLE 11.—*Piperonyl butoxide residues in composite sample of commodities stores in treated bags; data are averages for duplicate samples from each of 2 bags except as noted*

| Type of bag and code No. | Initial deposit | | Exposure period | Average residue in— | | |
|--------------------------|-----------------|--------------------|------------------------|---------------------|------------------------|---------------|
| | Pyrethrins | Piperonyl butoxide | | Flour | Cornmeal | |
| | | | <i>Mg. per sq. ft.</i> | | <i>Mg. per sq. ft.</i> | <i>Months</i> |
| Cotton, sewn closures: | | | | | | |
| 3-100----- | 11.6 | 102 | { 3 | 27.0 | 25.4 | 25.1 |
| | | | { 6 | 22.0 | 25.9 | 25.5 |
| | | | { 9 | 31.2 | 28.1 | 18.5 |
| | | | { 12 | 31.2 | 25.5 | 15.6 |
| 4-100----- | 12.9 | 113 | { 3 | 32.2 | 25.1 | 22.7 |
| | | | { 6 | 41.3 | 22.9 | 20.6 |
| | | | { 9 | 31.4 | 25.1 | 20.5 |
| | | | { 12 | 34.9 | 29.0 | 24.3 |
| 4-50----- | 4.6 | 40 | { 3 | 11.2 | 8.5 | 11.7 |
| | | | { 6 | 13.4 | 9.9 | 9.5 |
| | | | { 9 | 13.2 | 10.5 | 11.4 |
| | | | { 12 | 13.2 | 10.2 | 10.0 |
| 7-40----- | 2.7 | 24 | { 3 | 5.1 | 6.5 | 7.9 |
| | | | { 6 | 9.1 | 5.2 | 6.6 |
| | | | { 9 | 9.2 | 7.9 | 9.9 |
| | | | { 12 | 12.1 | 10.4 | 5.4 |

INSECT-RESISTANT COTTON BAG

TABLE 11.—*Piperonyl butoxide residues in composite sample of commodities stored in treated bags; data are averages for duplicate samples from each of 2 bags except as noted—Continued*

| Type of bag and code No. | Initial deposit | | Exposure period | Average residue in— | | |
|---------------------------------|-----------------|--------------------|-----------------|---------------------|-------------------|-------------------|
| | Pyrethrins | Piperonyl butoxide | | Flour | Cornmeal | |
| | Mg. per sq. ft. | Mg. per sq. ft. | Months | | P.p.m. | P.p.m. |
| Cotton, sewn closures—Continued | | | | | | |
| 7-80----- | 7.2 | 63 | 3 | 10.9 | 13.2 | 13.9 |
| | | | 6 | 16.9 | 11.7 | 16.6 |
| | | | 9 | 11.1 | 15.3 | 14.5 |
| | | | 12 | 16.1 | 11.5 | 13.4 |
| Cotton, TOS closures: | | | | | | |
| T-7-40----- | 2.7 | 24 | 3 | 6.9 | ¹ 5.1 | ¹ 7.6 |
| | | | 6 | 5.5 | 4.7 | ¹ 6.6 |
| | | | 9 | 8.5 | 5.6 | 5.9 |
| | | | 12 | 7.1 | 6.4 | 3.8 |
| T-7-80----- | 7.2 | 63 | 3 | 10.3 | ¹ 11.1 | ¹ 10.0 |
| | | | 6 | 13.2 | 9.9 | ¹ 10.0 |
| | | | 9 | 16.4 | 10.7 | 11.0 |
| | | | 12 | 16.5 | 8.7 | 9.5 |

Cotton, waxed-paper liner, TOS closures, cemented seam:

| | | | | | | |
|--------------------------------|------|-----|----|-------|-------|------|
| 5-50----- | 6.2 | 54 | 3 | 5.0 | 5.1 | 6.0 |
| | | | 6 | 6.1 | 6.6 | 5.6 |
| | | | 9 | 8.9 | 7.7 | 7.8 |
| | | | 12 | 8.7 | 7.7 | 6.7 |
| 5-100----- | 12.2 | 107 | 3 | 6.8 | 9.0 | 9.0 |
| | | | 6 | 8.4 | 10.1 | 9.5 |
| | | | 9 | 10.4 | 12.5 | 10.2 |
| | | | 12 | 13.0 | 10.2 | 11.4 |
| Kraft, POM closures, 8-50----- | 6.4 | 56 | 3 | 2.7 | 2.4 | 3.3 |
| | | | 6 | 2.8 | 2.6 | 2.3 |
| | | | 9 | 1.9 | 2.2 | 1.1 |
| | | | 12 | 3.0 | 1.4 | 1.8 |
| Kraft, TOS closures, 9-50----- | 7.3 | 64 | 3 | ----- | ----- | 4.3 |
| | | | 6 | ----- | ----- | 4.0 |
| | | | 9 | ----- | ----- | 4.4 |
| | | | 12 | ----- | ----- | 4.4 |

¹ 2 samples from 1 bag.

TABLE 12.—*Piperonyl butoxide residues in surface samples of commodities stored in treated bags; data are averages for duplicate samples from each of 2 bags except as noted*

| Type of bag and code No. | Initial deposit | | Exposure period | Average residue in— | | |
|--------------------------------------|------------------------|------------------------|-----------------|---------------------|---------------|---------------|
| | Pyrethrins | Piperonyl butoxide | | Flour | Cornmeal | |
| | | | | | Degermed | Regular |
| | <i>Mg. per sq. ft.</i> | <i>Mg. per sq. ft.</i> | <i>Months</i> | <i>P.p.m.</i> | <i>P.p.m.</i> | <i>P.p.m.</i> |
| Cotton, sewn closures: 3-100----- | 11.6 | 102 | 3 | 47.0 | 54.1 | 48.4 |
| | | | 6 | 53.4 | 64.4 | 53.2 |
| | | | 9 | 57.6 | 51.0 | 45.2 |
| | | | 12 | 51.9 | 46.2 | 49.4 |
| 4-100----- | 12.9 | 113 | 3 | 43.2 | 46.6 | 42.2 |
| | | | 6 | 56.7 | 52.5 | 63.8 |
| | | | 9 | 60.8 | 40.4 | 48.3 |
| | | | 12 | 54.5 | 53.2 | 49.2 |
| 4-50----- | 4.6 | 40 | 3 | 18.2 | 16.9 | 21.7 |
| | | | 6 | 22.4 | 19.2 | 26.9 |
| | | | 9 | 23.5 | 17.1 | 20.7 |
| | | | 12 | 19.2 | 12.4 | 17.8 |
| 7-40----- | 2.7 | 24 | 3 | 6.8 | 8.2 | 8.7 |
| | | | 6 | 10.2 | 8.1 | 9.7 |
| | | | 9 | 9.1 | 9.4 | 12.5 |
| | | | 12 | 8.1 | 11.6 | 7.6 |

| | | | | | | |
|---|------|-----|----|-------|--------|--------|
| 7-80----- | 7.2 | 63 | 3 | 16.8 | 22.9 | 24.9 |
| | | | 6 | 27.1 | 29.2 | 26.5 |
| | | | 9 | 24.5 | 18.6 | 26.5 |
| | | | 12 | 24.5 | 24.2 | 22.8 |
| Cotton, TOS closures: | | | | | | |
| T-7-40----- | 2.7 | 24 | 3 | 5.3 | 1 8.3 | 1 7.6 |
| | | | 6 | 8.0 | 4.7 | 1 11.3 |
| | | | 9 | 10.5 | ----- | ----- |
| | | | 12 | 7.4 | ----- | ----- |
| T-7-80----- | 7.2 | 64 | 3 | 13.9 | 1 16.2 | 1 22.5 |
| | | | 6 | 24.1 | 1 21.9 | 29.4 |
| | | | 9 | 22.9 | 1 14.5 | 1 22.8 |
| | | | 12 | 22.9 | 1 12.4 | 1 14.9 |
| Cotton, waxed-paper liner, TOS closures, cemented seam: | | | | | | |
| 5-50----- | 6.2 | 54 | 3 | 11.1 | 1 11.0 | 15.5 |
| | | | 6 | 14.1 | 1 12.7 | 1 14.1 |
| | | | 9 | 17.3 | 20.5 | 15.0 |
| | | | 12 | 21.3 | 1 22.4 | 1 17.1 |
| 5-100----- | 12.2 | 107 | 3 | 20.8 | 29.6 | 20.1 |
| | | | 6 | 24.7 | 31.4 | 23.8 |
| | | | 9 | 23.6 | 29.9 | 1 14.9 |
| | | | 12 | 27.0 | 29.5 | 27.2 |
| Kraft, POM closures, 8-50----- | 6.4 | 56 | 3 | 8.0 | 3.9 | 1 8.7 |
| | | | 6 | 6.4 | 4.5 | ----- |
| | | | 9 | 6.8 | 4.1 | ----- |
| | | | 12 | 1 5.3 | 3.5 | 1 1.8 |

See footnote at end of table.

TABLE 12.—*Piperonyl butoxide residues in surface samples of commodities stored in treated bags; data are averages for duplicate samples from each of 2 bags except as noted—Continued*

| Type of bag and code No. | Initial deposit | | Exposure period | Average residue in— | | |
|--------------------------------|----------------------------|----------------------------|--------------------|---------------------|---------------|---------------|
| | Pyrethrins | Piperonyl butoxide | | Flour | Cornmeal | |
| | | | | | | Degermed |
| | <i>Mg. per sq. ft.</i> | <i>Mg. per sq. ft.</i> | <i>Months</i> | <i>P.p.m.</i> | <i>P.p.m.</i> | <i>P.p.m.</i> |
| Kraft, TOS closures, 9-50----- | 7.3 | 64 | 3 | ----- | ----- | 8.8 |
| | | | 6 | ----- | ----- | 7.5 |
| | | | 9 | ----- | ----- | 10.2 |
| | | | 12 | ----- | ----- | 11.0 |

¹ 2 samples from 1 bag.

TABLE 13.—*Piperonyl butoxide* deposits remaining on bags containing 3 commodities; data are averages for 4 bags at each examination except as noted

| Type of bag and code No. | Initial deposit | | Ply | Exposure period | Average deposit remaining on bags of— | | |
|--------------------------------------|-----------------|--------------------|--------|-----------------|---------------------------------------|-----------------|---------|
| | Pyrethrins | Piperonyl butoxide | | | Flour | Cornmeal | |
| | | | | | | Degermed | Regular |
| | Mg. per sq. ft. | Mg. per sq. ft. | Months | Mg. per sq. ft. | Mg. per sq. ft. | Mg. per sq. ft. | |
| Cotton, sewn closures: 3-100----- | 11.6 | 102 | ----- | 3 | 21.4 | 19.8 | 18.3 |
| | | | | 6 | 22.9 | 13.6 | 9.4 |
| | | | | 9 | 19.8 | 12.7 | 6.8 |
| | | | | 12 | 16.3 | 5.9 | 5.3 |
| 4-100----- | 12.9 | 113 | ----- | 3 | 27.0 | 23.7 | 17.7 |
| | | | | 6 | 17.2 | 13.5 | 11.5 |
| | | | | 9 | 19.0 | 10.0 | 10.4 |
| | | | | 12 | 9.1 | 8.0 | 6.2 |
| 4-50----- | 4.6 | 40 | ----- | 3 | 4.9 | 5.6 | 4.2 |
| | | | | 6 | 3.4 | 2.7 | 2.3 |
| | | | | 9 | 2.8 | 2.9 | 2.7 |
| | | | | 12 | 2.1 | 3.3 | 1.4 |
| 7-40----- | 2.7 | 24 | ----- | 3 | 7.5 | 5.1 | 4.6 |
| | | | | 6 | 5.8 | 3.1 | 2.0 |
| | | | | 9 | 4.1 | 2.9 | 1.6 |
| | | | | 12 | 3.6 | 2.4 | 1.5 |

TABLE 13.—*Piperonyl butoxide deposits remaining on bags containing 3 commodities; data are averages for 4 bags at each examination except as noted—Continued*

| Type of bag and code No. | Initial deposit | | Ply | Exposure period | Average deposit remaining on bags of— | | |
|---------------------------------|-----------------|--------------------|--------|-----------------|---------------------------------------|-------------------|-------------------|
| | Pyrethrins | Piperonyl butoxide | | | Flour | Cornmeal | |
| | | | | | | Degermed | Regular |
| | Mg. per sq. ft. | Mg. per sq. ft. | Months | Mg. per sq. ft. | Mg. per sq. ft. | Mg. per sq. ft. | |
| Cotton, sewn closures—Continued | | | | | | | |
| 7-80----- | 7.2 | 63 | } | 3 | 22.2 | 14.3 | 11.4 |
| | | | | 6 | 17.8 | 10.7 | 6.8 |
| | | | | 9 | 16.6 | 9.5 | 5.6 |
| | | | | 12 | 13.4 | 6.7 | 3.3 |
| Cotton, TOS closures: | | | | | | | |
| T-7-40----- | 2.7 | 23 | } | 3 | 8.9 | ² 5.7 | ² 5.1 |
| | | | | 6 | 6.4 | ² 2.9 | ² 2.2 |
| | | | | 9 | 4.9 | ² 2.4 | ² 2.2 |
| | | | | 12 | 3.0 | ² 5.0 | ² 1.4 |
| T-7-80----- | 7.2 | 63 | } | 3 | 24.9 | ² 18.2 | ² 16.5 |
| | | | | 6 | 19.8 | ¹ 13.2 | 8.0 |
| | | | | 9 | 14.8 | ¹ 12.4 | ¹ 5.1 |
| | | | | 12 | 12.7 | ¹ 7.6 | ¹ 4.9 |

Cotton, waxed-paper liner, TOS closures, cemented seam:

| | | | | | | | |
|-------|------|-----|--------|----|------|-------------------|-------------------|
| 5-50 | 6.2 | 54 | Cotton | 3 | 22.8 | ¹ 18.1 | 21.9 |
| | | | Kraft | | 15.5 | 11.4 | 12.5 |
| | | | Cotton | 6 | 20.9 | ¹ 17.0 | ¹ 14.8 |
| | | | Kraft | | 15.6 | 13.6 | 10.1 |
| 5-100 | 12.2 | 107 | Cotton | 9 | 14.3 | 13.3 | 9.0 |
| | | | Kraft | | 12.0 | 9.7 | 10.4 |
| | | | Cotton | 12 | 10.6 | ¹ 6.5 | ¹ 6.2 |
| | | | Kraft | | 9.6 | 7.1 | 6.3 |
| | | | Cotton | 3 | 33.9 | 38.1 | 44.1 |
| | | | Kraft | | 24.1 | 14.7 | 16.1 |
| | | | Cotton | 6 | 41.2 | 33.6 | 33.3 |
| | | | Kraft | | 16.1 | 11.7 | 13.7 |
| | | | Cotton | 9 | 42.6 | ¹ 31.6 | ¹ 27.2 |
| | | | Kraft | | 12.5 | 12.2 | 13.5 |
| | | | Cotton | 12 | 28.6 | ³ 10.6 | 17.4 |
| | | | Kraft | | 11.0 | 10.5 | 9.2 |

INSECT-RESISTANT COTTON BAG

See footnotes at end of table.

TABLE 13.—*Piperonyl butoxide deposits remaining on bags containing 3 commodities; data are averages for 4 bags at each examination except as noted—Continued*

| Type of bag and code No. | Initial deposit | | Ply | Exposure period | Average deposit remaining on bags of— | | |
|--|--------------------|--------------------|--------|------------------|---------------------------------------|-----------------|------------------|
| | Piperonyl butoxide | | | | Flour | Cornmeal | |
| | Pyrethrins | Piperonyl butoxide | | | | Degermed | Regular |
| | Mg. per sq. ft. | Mg. per sq. ft. | Months | Mg. per sq. ft. | Mg. per sq. ft. | Mg. per sq. ft. | |
| Cotton, wax-paper liner, TOS closures, cemented seam—Continued | | | | | | | |
| Kraft, POM closures, 8-50..... | 6.4 | 56 | 3 | Outer..... | 5.8 | 6.8 | ² 3.9 |
| | | | | 4..... | 2.2 | 3.0 | 2.4 |
| | | | | 3..... | 2.1 | 3.1 | 2.2 |
| | | | | 2..... | .8 | .7 | .7 |
| | | | | Inner..... | .1 | 1.1 | .5 |
| | | | 6 | Outer..... | 3.6 | 3.0 | ² 3.2 |
| | | | | 4..... | 1.6 | 1.4 | 1.8 |
| | | | | 3..... | 1.2 | .8 | 1.8 |
| | | | | 2..... | .5 | .2 | .8 |
| | | | | Inner..... | .2 | .3 | .4 |
| | | | 9 | Outer..... | 3.6 | 2.5 | ¹ 2.9 |
| | | | | 4..... | 1.9 | .8 | 1.7 |
| | | | | 3..... | 1.1 | .7 | 2.0 |
| | | | | 2..... | .6 | .3 | .9 |
| | | | | Inner..... | .3 | .2 | .3 |
| 12 | Outer..... | ¹ 4.2 | 2.6 | ² 1.7 | | | |
| | 4..... | 1.8 | 1.5 | 1.4 | | | |
| | 3..... | 1.7 | 1.7 | 1.6 | | | |
| | 2..... | .9 | .4 | .9 | | | |
| | Inner..... | .5 | .3 | .3 | | | |

Kraft, TOS closures, 9-50.....

7.3

64.0

| | | | | |
|------------|---|----|---|------|
| Outer..... | } | 3 | } | 10.4 |
| 4..... | | | | 2.6 |
| 3..... | | | | 3.0 |
| 2..... | | | | 1.4 |
| Inner..... | | | | .9 |
| Outer..... | } | 6 | } | 3.5 |
| 4..... | | | | 1.3 |
| 3..... | | | | 1.2 |
| 2..... | | | | .4 |
| Inner..... | | | | .3 |
| Outer..... | } | 9 | } | 3.9 |
| 4..... | | | | 1.1 |
| 3..... | | | | 1.0 |
| 2..... | | | | .4 |
| Inner..... | | | | .5 |
| Outer..... | } | 12 | } | 15.1 |
| 4..... | | | | 1.2 |
| 3..... | | | | 1.0 |
| 2..... | | | | .4 |
| Inner..... | | | | .6 |

¹ 3 bags analyzed.

² 2 bags analyzed.

³ 1 bag analyzed.

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