

2. No evidences of cumulative effects were apparent among three rabbits each of which was given 75 successive oral doses of 0.265 grams per kilogram of body weight, on each of five days per week, over a period of 106 days.
3. Rats fed diets containing 200 p.p.m., 2000 p.p.m., and 20,000 p.p.m. of Santicizer B-16 in their daily diets showed no toxic effects or retardation of growth.
4. Two dogs, each given a daily oral dose of 11.0 mgs. of Santicizer B-16 for two years, showed no toxic effects or retardation of growth.

IV. Extraction Studies³

A. Procedure

Two-mil polyvinyl chloride films plasticized with butyl phthalyl butyl glycolate (Santicizer B-16) were cast from 20% solids solution in cyclohexanone according to the method of Alben, Alfrey, Jansen, and Mark, "Journal of Polymer Science", 2, 178 (April 1947).

Research revealed that the Santicizer B-16 content of plasticized films could be determined by infra red spectrophotometry. Measurements made on such films containing Santicizer B-16 up to 20% showed an absorption band at 13.45μ , a region which is uniquely free from interfering absorption of polyvinyl chloride, hard, and peanut oil. It was therefore possible to establish the curve of optical density vs. percent Santicizer B-16 in the film as shown in Figure IV. From this curve, the percentage loss of Santicizer B-16 from the extracted film could be directly determined.

Approximately 1" diameter punchings from the cast film were fastened in clip-type brass rings and completely immersed in the following solvents for 24 hrs., 7 days, and 30 days:

Peanut Oil at 26° C., 80° C., and 110° C.
Pure Lard at 26° C., 80° C., and 110° C.
Water, pH 3, at 26° C. and 80° C.
Water, pH 7, at 26° C. and 80° C.
Water, pH 10, at 26° C. and 80° C.

The immersion was done in such a manner that both sides of the film were in complete contact with the solvents. After remaining in the solvent for 24 hours, the films were removed, the plasticizer content determined and the same films replaced in the respective solvents for the 7 day's test. Those films which showed considerable extraction after 7 days were not continued for 30 days.

³This portion of the study was performed by the Monsanto Chemical Co., St. Louis, Missouri

PLASTICIZER EXTRACTION FROM VINYL FILM

CALIBRATION CURVE FOR B-16 PLASTICIZER IN POLYVINYL CHLORIDE FILMS

PERCENT B-16 VS. OPTICAL DENSITY PER GRAM FOR 2" DISKS

Optical Density/gram/2" disk

B-16 in P.V.C. Film

20

15

10

5

02085702

BFG03540

The buffer solutions used for the various aqueous solutions were prepared as follows:

pH 3
11.26 gm. citric acid. H_2O
167 ml $N/2$ NaOH
236 ml $N/10$ NaI
Diluted to 1000 ml.

pH 7
6.8 gm. KH_2PO_4
59.26 ml $N/2$ NaOH
Diluted to 1000 ml.

pH 10
3.1 gm. H_2SO_4
3.73 gm. NaI
87.8 ml. $N/2$ NaOH
Diluted to 1000

In addition to the extraction tests described above, the effect on the physical properties of film when in contact with oleomargarine for 48 hours at 27° C. was determined. Test samples 5" long, 1" wide, and approximately 0.004" thick, containing 50 parts by weight of Santicizer B-16 (per 100 parts Geon resin 202), were exposed to Delrich colored oleomargarine. A quantity of the oleomargarine was spread into a glass baking dish and the film strips - previously conditioned for 48 hours - were placed on top. Another layer of oleomargarine was placed over the top of the film, thus exposing both sides of the film to the extracting effects of the oleomargarine. The dish was then placed in the constant temperature room (27° C.) for 48 hours. The films were removed, all excess oleomargarine wiped off, and conditioned for 48 hours at 27° C. The tensile strength, modulus, and elongation were determined on the exposed film and compared to similar data of unexposed film.

B. Results

The results of exposing the 1" diameter film punchings to various solvents under the conditions as outlined above, were as follows:

Solvent	Temp. °C.	Degree of Extraction - Percent of Original plasticizer content of film extracted		
		After 24 hours	After 7 days	30 days
Peanut Oil	26° C.	11.5%	19.5%	28%
Peanut Oil	80° C.	89%	98%	
Peanut Oil	116° C.	100%	100%	
Pure Lard	26° C.	12%	20.5%	28.5%
Pure Lard	80° C.	92%	100%	
Pure Lard	116° C.	100%	100%	
Water-pH 3	26° C.	9%	8.5%	15.5%
Water-pH 3	80° C.	24%	58.5%	
Water-pH 7	26° C.	9%	13.5%	18%
Water-pH 7	80° C.	37%	82%	
Water-pH 10	26° C.	5%	6%	14.5%
Water-pH 10	80° C.	57.5%	83%	

*Initial plasticizer content of films was 80% Santicizer B-16 in Geon 202.

20458021

These results show that at 80° C. and 100° C. peanut oil and lard effect essentially a total extraction of Santiciser B-16 from the film after 24 hours. At 26° C. the amount extracted by peanut oil and lard does not appear to be excessive even after 30 days. Water does not have as serious an effect on Santiciser B-16 as do the other solvents, and the pH does not appear to be a significant factor. Increased temperature significantly increases the extraction by water but such an increase is not initially as effective as in the case of peanut oil and lard.

The results of exposing the film test strips containing 50 parts Santiciser B-16 to oleomargarine were as follows:

Plasticiser	Parts Plasti- ciser	Before Exposure				After Exposure				% Change	
		T.S. E		Mod. T.S. E		T.S. E		Mod. T.S. E			
		psi	%	psi	%	psi	%	psi	%	psi	%
Santiciser B-16	50	980	175	600	1600	180	1150	65	0	90	

These films were rather poor due to the difficulty of casting high-solid polyvinyl chloride solutions. However, the data indicate strongly that polyvinyl chloride films plasticized with Santiciser B-16 does not lose its physical properties when exposed to oleomargarine.

C. Conclusions

As a result of the extraction studies the following conclusions can be made:

1. At 26° C., the extractability of Santiciser B-16 by peanut oil, lard, and water is relatively low.
2. At 80° C. and 100° C. virtually all of the Santiciser B-16 is extracted by the peanut oil and lard in 24 hours.
3. At 80° C. the extraction of Santiciser B-16 by water is much greater than at 26° C., but 100% extraction requires in excess of 7 days immersion.
4. The pH of the water does not appear to have a significant effect on the extractability of Santiciser B-16.
5. Films containing Santiciser B-16 still retain good physical properties after 48 hours exposure to oleomargarine at 27° C.
6. A film containing Santiciser B-16 showed an increase in tensile strength and modulus with practically no change in elongation after exposure to oleomargarine for 48 hours at 27° C.

20458022

AN-1175

AN-1175
02.04.00

BFG03543