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AN IMPROVED METHOD
OF
LOADING AEROSOL CONTAINERS

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AN IMPROVED METHOD OF LOADING AEROSOL CONTAINERS

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The loading of aerosol products is now being done by one of two methods—cold filling or pressure filling.

In the cold-loading process, the concentrate and propellent are refrigerated and added to the open aerosol container in separate steps. This filling procedure is limited to products which have reasonable viscosities at low temperatures and will not be adversely affected by refrigeration. A second disadvantage is the rather expensive necessity of removing heat from the formulation prior to loading, and of replacing this heat immediately after loading. The purchase and maintenance of the refrigeration equipment is another disadvantage of the cold-filling technique.

Pressure loading of aerosol products avoids the problems associated with refrigeration in cold filling, but presents two new problems in their place. The pressure-filling procedure is complicated by the introduction of a third step—purging the air from the aerosol container prior to loading the propellent. The second disadvantage is the relatively long time required to load the propellent through the aerosol valve.

Air must be purged from an aerosol container prior to capping, because any air remaining in the container is compressed when the propellent is pressure loaded. This compressed air exerts its pressure independent of, and in addition to, the vapor pressure of the complete formulation. Since this compression increases when larger quantities of propellent are used, the problem is most acute when formulations containing a high proportion of propellent are loaded. Excessive pressures resulting from this trapped air can cause the total pressure to exceed the safe and legal limits for the aerosol package. Some specific examples will be cited in the description of the experimental work.

The object of this study was to explore the possibility of loading a mixture of concentrate and a portion of the propellent at atmospheric pressure and ambient temperature. The balance of the propellent is then pressure loaded in the usual manner. Since all operations are conducted at ambient temperature, the problems connected with refrigeration are eliminated. Purging is effected automatically by the vaporizing propellent, thus eliminating the troublesome purging step of pressure filling. Any propellent which can be added to the open container will reduce the quantity of propellent to be loaded through the valve, thus relieving a part of the time disadvantage of pressure filling.

Preliminary experiments were designed to examine the feasibility of automatically purging the aerosol container by loading a mixture of concentrate and propellent having sufficient vapor pressure to evolve small amounts of propellent vapor during loading. For these experiments, "Freon-11" propellent, "Deobase" deodorized kerosene, and absolute ethanol were selected as model concentrates. Equal volumes of each model

TABLE I

Purging of Aerosol Containers
(100 ml "Concentrate" + 30 ml "F-12")

	Pressure (Psig at 70F)			
Concentrate	No Purging	5% "F-12"	10% "F-12"	Cold Load
"Freon-11"	21	19	17	18
Alcohol (SD 39C)	48	42	36	29
"Deobase"	44	37	31	26

concentrate and propellent were used in every case to eliminate variables associated with differing volume fill. Reference points were established by pressure loading the formulation without purging, and by cold filling the same formulation. Thirty seconds were allowed to elapse between filling and capping the container in every case.

The data in Table I show clearly that the presence of a volatile component in the concentrate has the desired effect of removing much of the air from the container. Column 2 lists the final pressures of the formulations when the concentrate contains no "Freon-12" propellent. Columns 3 and 4 list the final pressures of the formulations when the concentrate contains 5 and 10 weight per cent "Freon-12." In the latter two cases, sufficient "Freon-12" was pressure loaded to make a total of 30 ml of "Freon-12" per can. These results were obtained by filling 6-oz Continental cans 75 per cent volume full at 75F. Somewhat different final pressures may be expected if a different fraction of the can's volume is utilized. The fact that the formulation containing "Freon-11" is completely purged while the "Deobase" and alcohol-based formulations are not, indicates that the "Freon-11" itself makes a significant contribution to the purging process when the formulation is loaded at 75F.

A second series of experiments was then made to examine the effect of loading a model insecticide formulation containing 15 per cent "Deobase," 42.5 per cent "Freon-12" and 42.5 per cent "Freon-11," and a model hair-spray formulation containing 30 per cent absolute ethanol, 35 per cent "Freon-12" and 35 per cent "Freon-11." In one group of experiments, a mixture of concentrate and "Freon-11" was loaded into the open cans, and in a second group of experiments a mixture of concentrate, "Freon-11," and "Freon-12" was loaded into the open containers, which were capped 30 seconds after filling. Table II

TABLE II

Purging by Solvent/Propellent Mixtures

Formulation		Pressure (Psig at 72F)	
"Concentrate"	"Freon-12"	Pressure Load	Cold Load
"Deobase" (26g) "Freon-11" (72g)	72g	37	33
"Deobase" (26g) "Freon-11" (72g) "Freon-12" (10g)	62g	34	33
Ethanol (52g) "Freon-11" (59g)	59g	41	31
Ethanol (52g) "Freon-11" (59g) "Freon-12" (11g)	48g	34	31

shows the formulation details and typical results of these experiments. Column 1 shows the composition of the concentrate-propellent mixture which was loaded into the open cans, and Column 2 shows the quantity of "Freon-12" which was pressure loaded. In the case where the formulation was cold loaded, the propellent mixture was added to the refrigerated concentrate. The pressures of the same formulations loaded by the cold-filling procedure are included for comparison.

These data demonstrate clearly that premixing the concentrate and "Freon-11" contributes to the effectiveness of purging and provides an excellent way of introducing significant quantities of propellent at atmospheric pressure. The presence of some "Freon-12" is desirable, since this provides the necessary pressure for rapid, efficient purging.

These experiments were duplicated using a pyrethrum-based insecticide formulation, a PVP-based hair-spray formulation, and a commercial paint formulation. With all of these materials, the results were essentially the same as those obtained with the model concentrates described before.

Since one of the objectives of this study was to investigate the possibility of reducing the time required for pressure loading an aerosol package, it was necessary to investigate the effect of loading time on purging efficiency. This was done by loading aerosol containers with a self-purging concentrate and allowing varying periods of time to elapse before capping the container. The results of these experiments are summarized in Table III. Times listed in this table are the total time elapsed between the start of loading and capping the container.

TABLE III

Effect of Filling Time on Pressure

	Pressure (Psig at 72F)	
Time Sec.	"Deobase" Formulation	Ethanol Formulation
25	36	35
10	36	37
5	36	38

As the data in Table III show, there is no significant difference in pressures as the loading time varies. It appears that purging is essentially complete immediately after the liquid is loaded. While the increasing pressure trend of the ethanol-based formulation is not considered significant, it may be an indication that the lower time limit for this type formulation is in the vicinity of five seconds.

The effect of varying ambient temperatures of the concentrate on purging was briefly examined. When the sulf-purging hair-spray concentrate was loaded at 85F and at 65F, the final pressures were 37 and 36 psig at 70F, respectively. This demonstrates that for this formulation, ordinary variations in ambient temperature will not have a significant effect on the effectiveness of the purging step.

A cooperative production-scale test loading has been made by G. Barr & Company, using the method described. In this test, a hair-spray formulation containing 30 per cent concentrate, 24.5 per cent "Freon-12," and 45.5 per cent "Freon-11" was loaded. A concentrate-propellent mixture containing 36 per cent concentrate, 55 per cent "Freon-11" and 9 per cent "Freon-12" was loaded into the open aerosol container. "Freon-12" was pressure loaded to reach the final formulation. Existing equipment was utilized without modification. Analysis of the contents of the cans after loading showed that the purging process was successful. The pressurized concentrate containing 9 per cent "Freon-12," which performed well in laboratory experiments, proved to be unsuitable for a full-scale loading operation. The proportion of "Freon-12" is so high that agitation of the liquid inherent in a high-speed operation caused serious losses by frothing of the formulation. This violent boiling occurred during gravity loading from an open bowl, and from the cans themselves during loading from a pressurized system.

Following these tests, a small laboratory apparatus was constructed which permitted loading of aerosol containers at approximately the same rate as commercial loading equipment. By use of this apparatus, it was found that the concentrate-propellent mixture containing 10 per cent "Freon-12," which gave good results in initial laboratory experiments, was unsuitable for high-speed loading because of the frothing problem described before. It has been possible to load a mixture of 35.5 per cent hair-spray concentrate, 61.5 per cent "Freon-11," and 3 per cent "Freon-12" at the rate of 130-140 g/sec into open 6-oz cans, without incurring losses from frothing. Where the end use of the product will permit, frothing can be further controlled by adding approximately 0.05 per cent of an antifoaming agent to the formulations. If a smaller volume fill is desired, the filling rate can be increased without causing frothing.

Propellent losses were estimated by preparing a quantity of PVP-based self-purging hair-spray concentrate. This concentrate was loaded into a group of tared aerosol cans. The total weight of material discharged from the loading apparatus was compared to the total quantity of material being retained in the cans. Average losses in this operation amount to approximately 1.5 per cent per can during a five-second loading cycle. Most of these losses occur during the actual loading, while the propellent is actively boiling off. These losses are related to the violence of loading, which affects the rate at which propellent vapors are released from the liquid, and to the proportion of "Freon-12" in the propellent-concentrate mixture. Losses which occur after the loading step are small, since active boiling ceases immediately after the liquid flow is stopped. From the quiescent liquid, the losses amount to about 0.5 per cent per can per minute.

The quantity of air remaining in the vapor phase in the containers after loading by this method was estimated to be 5-8 volume per cent from the excess pressure of the containers. Chromatographic analysis of a number of commercial insecticides showed that the vapor phase contained 5-20 volume per cent air, with most samples having approximately 10 volume per cent air in the vapor phase.

These laboratory experiments demonstrate that loading a self-purging concentrate offers several possibilities of improvement over presently used filling methods. The refrigeration problems associated with cold filling can be avoided, as can the purging problems normally connected with pressure filling. Loading time and purging efficiency are comparable to those now being attained.

There are still mechanical problems to be solved. For instance, it may be necessary to provide filling bowls and associated fixtures which can withstand 5-10 psi pressure, to avoid excessive losses from the concentrate-propellent mixture. Large, smooth filling nozzles would help to avoid the turbulence which contributes to the frothing problem. Answers to problems of this type are outside the scope of this particular study. These problems occur only in the aerosol loading plant, and can be solved most effectively there.