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DISPOSAL OF SOLID "TEFLON" WASTES CONTAINING C-8 APFIC

### Introduction

Some of the soliids wastes from the Fine Powder and Dispersion Area of the "Teeflon" plant contain small amounts of toxic perfluorocarboxyllic dispersing agents. A cheap and acceptable method of disposing of these solids is in a land fill, provided that the toxic agents are reduced to an acceptable level. Since the dispersing agents are volatile at moderate temperatures, we set out to determine the conditions necessary to bake the resins to a non-toxic state.

### Summary and Conclusions:

- 1. The dispersing aggent level of the solids waste stream is reduced to a quite acceptable level (below 2 ppm) by baking at 175°C for 5 hours.
- 2. Without a pretreatment, a small amount of the perfluorocarboxyllic acid dispersing agent would be leached into the ground water.

#### Discussion:

A system for collecting the solid wastes from thee Dispersion and Fine Powder Area was proposed by Anderson amd Chren. This involved the use of sumps and settling basins to concentrate the solids. Solids in the form of coagulum from wax filters and wax separator settlings were also to be collected in this system. A flow sheet of the proposed system is atttached. The total amount of solids is estimated at ca. 768 pounds per day at current operating conditions.

Samples of solid wastes were obtained from the collection points in the proposed system. These samples were representative of materiall to be obtained from the fine powder sump, the wax trap, the bottom of the wax separator and the sump from the Dispersion Airea: The make-up of these samples was as follows:

August 331, 1966

- Fine Powder Sump This material looks like contaminated,, wet, fine powder. The material contained 36..2% water and 130 ppm perfluorocarbon dispersing agent calculated as C-8 APFC.
- Wax Trap appears like wax. Lost 8% by weight on heating to 175°C. Contains ca. 30 ppm C-8.
- Bottom of Wax: Separator appears like wet fime powder and cointains about 65% water and 240 ppm C-8.
- Dispersion Area Sump This sample was actuallly obtained from the bottom of the stabilization tanks and was a mixture of a 60% dispersion liquid and a solid containing about 30% water. The material (contained ca. 300 ppm water. In most of the work done with this mixture, both the liquid and solid phase were analysed.

# 1. Leaching

As a first step,, it was determined that the dispersing agent could be leached from these solids. This was done by adding 100 grams of the sample from the different sources to 100 ml of water in a Waring Blendor. After blending for 5 minutes, 1the water was filtered and anælysed for dispersing agent by thee titration method (see below). The data shown in Table I show that the C-8 dispersing agent could indeed be extracted by water. This is the conclusion remched by Crandell in 1964 (see atitachment).

# Table I

The Extraction of C-8 Dispersing Agent from "Tefflon" Samples in Water

	Gain 1 a	<b>a</b>			PPM		Extract		
	Sample	Source		<u>lst</u>	Extract	1on	2nd	Extraction	Ĩ,
Fin	e Powder	Sump			30			19 . *	:
Wax	Separat	or	***		103			15,,	· .
Wax	Trap	•		•	(39)		;	. 17	
2.	Baking		• •	• .					•

The samples were baked at 175°C for 3, 5 and 20 mours. Analysis of these samples showed almost complete removal off the dispersing agent at 5 or 200 hours; after 3 hours treatment only the sample from the fine prowder sump contained any residual C-8

TABLE II

# ANALYSIS OF PERIFLUOROCARBON DISPERSING AGENT IN "TIEFLON" SOLIDS WASTE

Hours Heated @ 175°C						
Sample Source	0 3 5	20				
Fine Powder Sump	130 100 2.7	0.24				
Bottom of Wax Trap	240 <10 2.4					
Wax Trap #8	(30), 0 0.73					
	460 130 <40 1.1 0.77	0.20 0.41				

August 311, 1966

APFC. The temperature of 175°C was chosen as one easily reached by most ovens and because it is below the flash point of the wax used (205°C).

## 3. Analysis

Two methods of analysis were used for the determination of perfluorocarbon dispersing agents in the waste streams. The procedures are included here for completeness. One made use of the property that perfluorocarbon dispersing agents can be distilled with water from a strongly acidified slurry. This distillate can then be titrated to determine the amount of acid (as perfluorocatanoic acid ( $C_7F_{15}C^2OH - MW$  431). In these studies the following procedure was followed:

- 1. A 100 gram samplee (+200 ml of distilled water) was cut in a Wariing Blendor. The slurry was added to a 500-mll flask and 25 ml of concentrated phosphoric acid added.
- 2. The flask was fitted with a distilling head and condenser. The solurry was heated to boiling and the condensate collected in an Erlynmeyer flask containing 50-ml of distilled water that had been carefully neutrallized to a pink end-point with phenolphthalein findicator. Distillate samples were collected each 5 minutes and titrated with 1/100 N sodium hydroxide.
- 3. When the titrations indicated that no additional (acid was being distilled (ca. 3 titrations or 15 minut(es) the titre was added and the amount of dispersing agent calculated according to the following.

 $ppm C-8 = m1 \times 43.1$ 

This method checked very well with known spiked amounts of C-8 im the slurry.

The other method is based on the formation of a complex between methylene blue and the dispersing agent. While methylene blue is not extracted from acid solution into chloroform, the complex is partitioned into the chloroform layer. The intensity of the coloration in the chloroform layer is related to the concentration of dispersing agent in the aqueous phase. Nonionic dispersing agents, such ass "Triton" X-100, do not interferce. The method is sensitive to 0.05 ppm dispersing agent in the aqueous phase.

In this method, the following procedure was used:

1. The sample was prepared (as in the method above) by cutting a 100 gram sample in a Waring Blendor,

with water, acidifying with H<sub>3</sub>PO<sub>4</sub> and distilling. For this test, 5(0-ml of distillate was collected.

- 2. A measured portion of the distillate was added too a 100 ml volumethric flask and diluted with water. The measured amount depended on the amount of C-83 in the distillate. For instance, in the baked samples, a 25 ml portion, diluted to 100 ml, was used; for the as-received sample from the stabilization tank, a 0.2 ml sample, diluted to 100-ml was used.
- 3. The 100-ml from the dilution of step 2 was added to a 250-ml sepairating funnel and 1.0 ml of 1-9 HCl solution, 1.(0 ml of methylene blue solution (0.500 gms/liter), and 10.0 ml of chloroform were added. The mixture was shaken vigorously for 2 minutes, allowed to settle and the chloroform layer withdrawn.
- 4. The chloroform layer was analysed in a spectrophotometer in a ll-cm glass cell by scanning from
  700 mμ to 600 mμ using chloroform in the reference
  cell. The peak absorbance for the sample at
  650 mμ was measured.
- 5. For these studiess, it had been shown that 0.0057 units on the spectrophotometer = 1x10<sup>-6</sup> grams of C-8. From the amount of absorbance and the factor from step 2, the amount of C-8 in the sample was calculated.

The two methods checked within 10% for the wax separator sample.

RGA:sc Attachment