Monsanto

FROM (NAME & LOCATION) W. G. Krummrich

DATE : July 7, 1969

cc: Dan Roman
Harry Yapp
H. J. Horner

SUBJECT : S-1 Odor - Chemical Service

REFERENCE :

TO : H. Sl ec

Samples of Santophen-1 and Chlorophen-1 received from Chemical Service, Division of IMOCO-GATEWAY Corporation, Baltimore, Md. were checked for possible sources of mercaptan type odor.

The samples were fused with metallic sodium and qualtitative tests for sulfur performed. The Santophen-1 showed the presence of sulfur, which was expected because of the thiourea inhibitor. Chlorophen-1 did not show any sulfur present. An additional method for sulfur involving the reduction with Raney-nickel, evolution of H_2S, and formation of the methylene lue complex did not show the presence of sulfur in Chlorophen-1. This method is sensitive to tenth's of a ppm.

A G.L.C. analysis of both samples showed a difference of about 5:1 in one impurity. This material is higher boiling than o-benzyl-p-chlorophenol and was more concentrated in the chlorophen-1 material.

Isolation and identification of this impurity would require considerable effort. (> 10 man-days).

If additional work on this problem is requested, please inform and assign priority.

D J Roth

D. J. Roth

dv

DSW 298986

IN-10 REV. 11-65

STLCOPCB4068899

REAGENTS:

Benzophenone (Marker)	Fisher Scientific B-270
Hexamethyldisilazane (HMDS)	Pierce Chemical Company Stock No. 847704
Trimethylchlorosilane (TMCS)	Pierce Chemical Company Stock No. 85530
Tetrahydrofuran (THF)	Pierce Chemical Company Stock No. 278604
SE 30 Silcone	Wilkens Instrument and Research Company
Chromosorb G AW DMCS Treated	Varian Aerograph

EQUIPMENT:

Varian Aerograph Model 1820 GLC Varian Aerograph
Varian Aerograph Model 30 Recorder with disc integrater
$\frac{1}{4}$" SS column 6' long packed with 5% SE 30 on 70-80 chromosorb G AW DMCS treated
Syringe 50 ul #705 Hamilton Company
Syringe Luer 1 and 5 cc Fisher Scientific 14-820

INSTRUMENT CONDITIONS:

Detector Block Temperature	325°C
Injection Port Temperature	310°C
Detector Voltage	150 Ma.
Helium Flow	60 cc/min.
Helium Back Pressure	10 lbs.
Column Temperature	Start 75°C Finish 320°C

Program immediately @ 10°C/min. Hold until all comp. elute
Column Temperature limit control 350°C

DETERMINATION:

Using an analytical balance, weigh 0.35± 0.05 grams of sample to the nearest 0.0001 gram into a 20 ml. serum bottle. Add 0.2 grams of Benzophenone marker, weighed to the nearest 0.0001 gram, to the serum bottle.

Add, by pipet, 2.0 ml of tetrahydrofuran and swirl to dissolve the sample.

Using a 1 ml. syringe add 0.5 ml. trimethylchlorosilane. Swirl to mix.

Using a 5 ml. syringe add 5.0 ml. hexamethyldisilozane to the serum bottle swirling the bottle while adding the HMDS.

Stopper the serum bottle and insert a #20 syringe needle through the stopper for a vent. Place the bottle in a 75°C bath for 30 minutes. Swirl occasionally to help complete the reaction. Remove from bath at the end of the 30 min. period and swirl vigorously. Allow to settle.

Inject a 10 ul sample of the supernatant liquid into the GLC.

Program immediately at 10°C/min.

Attenuations will have to be determined according to the materials present in the sample.

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Correct peak areas for base line drift.

CALCULATION:

$$\% \text{ Component} = \frac{\text{Area Comp.}}{\text{Area Marker}} \times \frac{\text{Wt. Marker}}{\text{Wt. Sample}} \times \text{Comp. ACF} \times 100$$

Area correction factors for each component were determined by weighing given amounts of pure reagents and running several chromatograms per standard sample. The average value of each component was used as the AFC.

$$\text{AFC} = \frac{\text{Weight Comp.}}{\text{Weight Marker}} \times \frac{\text{Area Market}}{\text{Area Comp.}}$$

Note: This analytical procedure was developed for a special project by the WGK Process Research and Development Group. Its precision and accuracy have not been determined. Accordingly this analytical procedure is not recommended for routine analytical use, particularly Assay of Santophen 1.

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