

BFG TECHNICAL DOCUMENT

BFG CHEMICAL CO.

ANALYTICAL

REPORT

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GC/MS ANALYSES ASSOCIATED WITH TEMPREL[®] HOSE FUMES

A problem at BFG Marion involving Temprel hose stock fumes, which causes throat and eye irritation has existed since at least 1976 (see attachment). In 1976, the fumes generated during the manufacture of the Temprel hose were investigated by the Environmental Health Department. While the fumes were not specifically characterized, certain observations were reported by the investigators. Diallyl phthalate (DAP), triallyl isocyanurate (TAIC) which has since been replaced by triallyl trimellitate (TATM) and peroxide related materials could be detected in air samples. However, belief was expressed (see attachment) that no single Temprel ingredient, when heated, could produce the irritating fumes associated with Temprel stock. GC studies related the fumes to peroxide decomposition products and to allyl phthalate but these could not be confirmed in plant samples.

More recently, customers using Temprel hoses have complained about the irritating fumes resulting from the cutting of these materials with hot knives which can approach the pyrolytic decomposition temperature of these materials. These considerations and the fume problem at BFG Marion resulted in a planned study of the Temprel stock fumes and the fumes generated from the ingredients most likely to be responsible with TGA-DTA and GC/MS. S. W. Newell noted a definite lessening of the fumes when Uniflex 300 (a polyglycol ester derived from a mixture of 1,2-propane diol (major), 1,3-butane-diol, 1,4-butane diol and adipic acid⁽¹⁾) was not used in the Temprel compound⁽²⁾.

A series of compound formulations with dicumyl peroxide, lead stearate, Barcol, triallyl mellitate, Uniflex 300, diallyl phthalate, dicumyl adipate and chlorinated polyethylene were prepared to cover the possible combinations of the ingredients used in Temprel hoses without revealing the actual recipe. Of these, three compounded samples, and three stock samples, and the plasticizers with and without dicumyl peroxide were examined by GC/MS-headspace techniques. This report summarizes the materials identified in these GC/MS-headspace studies.

(1) The analysis of the Uniflex 300 material will be reported separately by J. L. Dorsch based on ¹³C NMR measurements (see attachment).

(2) J. C. McCool communication to J. A. Nihara, 1/21/80.

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Experimental

The following compounds were heated in standard, evacuated headspace vials at 180°C for 20 minutes:

	3	7	10
CPE	100	100	100
BFM-1 ^a	30	30	30
BFM-3 ^b	12	12	12
TATMC	3.6	3.6	3.6
Uniflex 300 ^d	10	--	--
DAP ^e	--	10	--
DOA ^f	--	--	10

^a 60% lead silicate, 18% Desical and 22% CPE.

^b 40% DiCup (40% dicumylperoxide on calcium carbonate) and 60% CPE.

^c 74% triallyl mellitate on Microcel.

^d Polyester based on a mixture of diols (primarily 1,2-propane diol) and adipic acid(1).

^e Diallyl phthalate.

^f Di-2-ethylhexyl adipate.

In addition to these compounds, the two masterbatches, BFM-1 and BFM-3, and the three plasticizers with and without dicumyl peroxide were examined under the same conditions. Pure dicumyl peroxide was obtained by extracting DiCup with diethyl ether and removing the ether by rotary evaporation. (Interestingly, too much BFM-3 masterbatch in a headspace vial ruptured the septum at 180°C and the escaping fumes generated from the dicumyl peroxide were irritating to the nose, throat, and eyes.)

At the end of the 20 minute-180°C heating period, the gas samples were removed with a 5cc gas tight syringe and injected onto a 3.2mm x 4.3m column packed with 80/100 mesh Porapak Q5. The materials evolved by gas chromatography were identified from their individual mass spectra by comparison with reference spectra.

Results and Discussion

A series of GC/MS total ion current profiles (TICP's) were obtained from the headspace gases generated at 180°C. Figures 1 and 2 show the materials evolved from BFM-1 and BFM-3. The only volatiles expected from BFM-3, which contains dicumyl peroxide and chlorinated polyethylene, were expected to result from the peroxide decomposition. The following were identified:

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