

$\frac{1}{4}$  in. O.D. copper column of silicone gum rubber on 60-80 mesh Diatoport-S was obtained from the F & M Scientific Corporation. All chromatograms were obtained on an F & M Model 720 gas chromatograph at 40° and a helium flow rate of 60 ml/min (variation of temperature and flow-rate from these values did not substantially improve the separations). The olefin mixture was approximately equimolar and the sample size was 0.002 ml.

The results of the separations are given in terms of retention times (Table I) and the chromatograms are shown in Fig. 1-3.

TABLE I  
RETENTION TIMES OF ALICYCLIC OLEFINS

Column used	Retention time (sec) of compounds			
	I	II	III	IV
Silicone gum rubber	120	76	45	120
Dinonyl phthalate	388	476	388	748
$\beta,\beta'$ -Oxydipropionitrile	132	212	320	465

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## Gas chromatography of derivatives of maleic hydrazide

### II. Trimethylsilyl maleic hydrazide

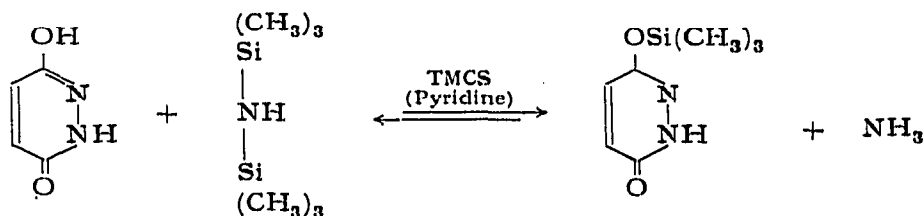
The gas chromatographic analysis of maleic hydrazide has been reported earlier to be extremely difficult owing to its extremely high melting point (296-298°)<sup>1</sup>. This same report described the preparation and gas chromatography of alkyl carbonate derivatives of maleic hydrazide.

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In view of the attention which has, in recent years, been afforded to the gas chromatographic analysis of various classes of trimethylsilyl (TMS) derivatives<sup>2-9</sup>, and since the enolic form of maleic hydrazide appeared to be conducive to silylation, it was of interest to investigate the gas chromatographic tendency of this derivative.

### Experimental

The maleic hydrazide sample was obtained from Naugatuck Chemical Division, U.S. Rubber Company (Naugatuck, Connecticut) and recrystallized from dimethyl formamide. The recrystallized product melted with decomposition at 300°. This was reacted in pyridine with hexamethyldisilazane<sup>10</sup> in the presence of trimethylchlor-silane (TMCS) as given below:



The melting point of the derivative was 63–65°.

Chromatography was carried out on an F & M model 1609 flame ionization chromatograph utilizing 6 ft. by 1/4 in. o.d. coiled pyrex glass columns containing 3% Carbowax 20M on 60–80 mesh acid-washed, DMCS-treated Chromosorb G and 4% SE-30 on 80–100 mesh HMDS-treated Chromosorb W. On-column injections were made using a conversion kit to provide an all-glass flow system (Applied Science Laboratories, State College, Pennsylvania). The specific operating conditions are given in Table I.

TABLE I  
ELUTION OF TMS-MALEIC HYDRAZIDE

	Minutes	Conditions				Range
		Col. T (°C)	N <sub>2</sub> (ml/min)	H <sub>2</sub> (ml/min)	Air (ml/min)	
Carbowax 20M	6.11	190	89	77	450	1000
SE-30	4.72	100	104	81	500	1000

Infrared spectra were obtained from a Perkin-Elmer model 337 grating infrared spectrophotometer.

### Results and discussion

The behavior of TMS-maleic hydrazide on polar (Carbowax 20M) and non-polar (SE-30) columns is shown in Table I. It is evident from the elution data that a fair degree of residual polarity still exists in the TMS derivative. Fig. 1 illustrates the elution of the derivative from SE-30. The temperature of analysis of TMS-maleic hydrazide

(100°) on this column is highly significant when one considers the melting point of the parent compound.

Infrared spectra were run on both the derivative and on maleic hydrazide. The TMS derivative gave a band at  $1250\text{ cm}^{-1}$  in the region of Si-C absorbance which was absent in the maleic hydrazide spectra.

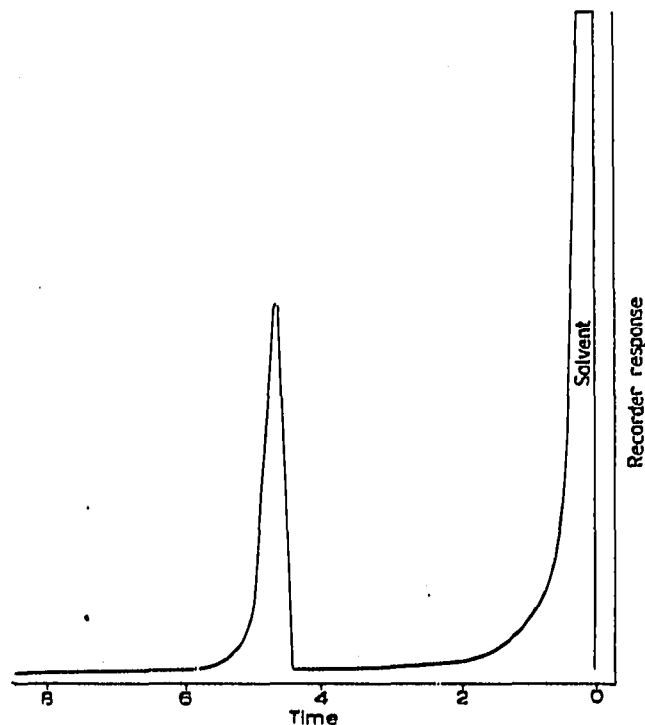


Fig. 1. TMS-maleic hydrazide on SE-30.

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