

TABLE VII

Uvescence intensity as a function of cure temperature at constant cure time for thermal curing of ethylenically unsaturated monomers monitored using dibenzofulvene.			
Heizbank Temperature, °C.	Uvescence Intensity at 318 nm, arbitrary units		
	Sample A	Sample B	Sample C
80	7	(a)	1
100	10	5	5
110	26	9	22
120	49	47	38
130	80	62	67
140	94	79	92
150	(a)	89	(a)

(a) not measured

Data of Table VII can be used to construct a calibration curve which shows the temperature at which cure is effectively complete as a function of uvescence intensity.

## EXAMPLE 16

This example demonstrates the inhibitor effect of dibenzofulvene for free radical polymerization.

A stock solution of 3.0 g 1,6-hexanediol diacrylate, 0.06 g AIBN, and 0.03 g dibenzofulvene (I, X=CH<sub>2</sub>) was prepared. A second stock solution was also prepared but without the dibenzofulvene. 5 to 10 mg samples were subjected to Differential Scanning Calorimetry in sealed pans using a DuPont Model 9900 Thermal Analyzer (E. I. duPont de Nemours, Wilmington, Del.)

The sample without dibenzofulvene had a polymerization exotherm at 80° C., while the sample containing dibenzofulvene had a polymerization exotherm at 118° C., showing the inhibition effect of dibenzofulvene on free radical polymerization.

## EXAMPLE 17

This example demonstrates cure monitoring of hydride curable silicones using dibenzofulvene.

To 1.51 g of the ethylenically unsaturated polysiloxane as used in Example 7 was added 0.0157 g dibenzofulvene (I, X=CH<sub>2</sub>) in minimal chloroform (approximately 0.2 g), 1.53 g of the ethylenically unsaturated polysiloxane containing 100 ppm Pt(O) catalyst as used in Example 7, and 0.084 g of polyhydrosiloxane as used in Example 7. The mixture was coated on 25 micrometer thick polypropylene film using a #14 wire wound bar and overlaid with a second film of polypropylene. Strips of the layered construction were cured by exposure to a thermal gradient (60°-150° C., approximately 7° C./cm) on a Heizbank Thermal Gradient (Reichert Type 7841, Austria) for 10 minutes. The resulting films were analyzed by fluorescence spectroscopy (Perkin-Elmer MPF44B fluorescence spectrometer, excitation 254 nm, emission monitored at 318 nm, through the polypropylene), and uvescence intensity was measured according to the procedure of Example 15. Data is shown in Table VIII below for three samples. After the fluorescence spectra were obtained, the polypropylene overlayer was removed and the film was tested for cure by rubbing with a finger. The coating was solid, non-greasy, and did not crumble where the strip had been exposed to temperatures greater than or equal to 120° C.

TABLE VIII

Uvescence intensity as a function of cure temperature at constant cure time for thermal curing of hydride curable silicones monitored using dibenzofulvene.			
Heizbank Temperature, °C.	Uvescence Intensity at 318 nm, arbitrary units		
	Sample A	Sample B	Sample C
70	36	31	46
80	42	55	59
90	66	77	106
100	88	77	152
110	176	169	239
120	471	316	423
130	507	454	547
140	(a)	626	672

(a) not measured

Data of Table VIII can be used to construct a calibration curve which shows the temperature at which cure is complete as a function of uvescence intensity.

## EXAMPLE 18

This example describes cure monitoring at variable temperatures of hydride curable silicones containing 9-fluorenone [I, X=oxygen].

A sample was prepared of 5.00 g ethylenically unsaturated polysiloxane, 0.053 g 9-fluorenone (dissolved with slight warming), 0.013 g maleate inhibitor, 0.052 g Pt(O) catalyst, and 0.13 g polyhydrosiloxane, as described in Example 7. 9-fluorenone is commercially available, e.g., from Aldrich Chemical Co. The sample was coated and heated on a Heizbank Thermal Gradient for 10 minutes, as described in Example 17. Using the IR absorption method described in Example 7, the extent of disappearance of Si—H bonds was measured. Data are shown in Table IX, below:

TABLE IX

Heizbank Temperature (°C.)	%	Uvescence Intensity (arbitrary units)	
		Si—H reacted <sup>(a)</sup>	328 nm 355 nm
70	51	62	90
80	57	138	191
90	63	205	296
100	(b)	217	327
110	57	401	621
120	(b)	580	962
130	65	465	764

<sup>(a)</sup> assuming a value at t = 0 for A<sub>v</sub>/B<sub>v</sub> of 4.0, for the calculation described in Table I, Example 7.

<sup>(b)</sup> poor quality IR spectra precluded measurement.

Data of Table IX show that when 9-fluorenone is used as latent uvaphore, uvescence increases by a factor of at least 3 at the temperature where the curable silicone is fully cured, and by a factor of 10 or more at somewhat higher temperatures.

Various modifications and alterations of this invention will become apparent to those skilled in the art without departing from the scope and spirit of this invention, and it should be understood that this invention is not to be unduly limited to the illustrative embodiments set forth herein.

We claim:

1. A curable composition consisting essentially of at least one of a) a hydride curable silicone comprising an ethylenically unsaturated siloxane and a polyhy-