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### LEWIS ACID CATALYZED PREPARATION OF TETRAMETHYLHEXATHIOADAMANTANE

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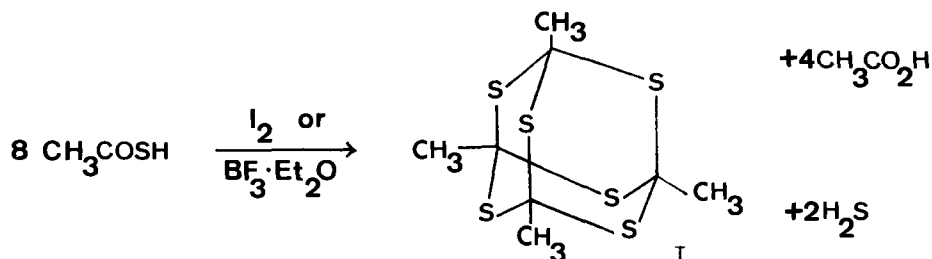
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LEWIS ACID CATALYZED PREPARATION OF  
 TETRAMETHYLHEXATHIOADAMANTANE

Submitted by C. S. Giam\*, R. L. Tabor and T. E. Goodwin<sup>†</sup>  
 (5/2/80)

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We have utilized two new and convenient processes for the  
 synthesis of tetramethylhexathioadamantane (I)<sup>1,2</sup> from thiol-  
 acetic acid. The compound is useful both as a lubricant addi-



tive,<sup>3</sup> and as a thermal stabilizer in poly(methylmethacrylate)<sup>4</sup>  
 where it promotes antiseize properties and confers oxidative

stability. The two synthetic procedures reported herein involve the use of boron trifluoride etherate or iodine as the catalyst.

These procedures are less hazardous and require less time for the isolation of products than the hydrogen sulfide/Lewis acid treatment of acetic acid used previously.<sup>5</sup>

#### EXPERIMENTAL

Procedure 1 (Boron Trifluoride).— Thiolacetic acid (9 ml, 127.1 mmoles) was stirred with boron trifluoride etherate (7.0 ml, 55.5 mmoles) for approximately one week under a  $\text{CaCl}_2$  drying tube. The white precipitate which was formed was collected and washed with 150 ml of a 5% sodium bicarbonate solution. The residual white powder was dried thoroughly under vacuum to yield 2.61 g (55%) of crude I, mp.  $214\text{--}216^\circ$ , which was then recrystallized from chloroform (1.5 M solution) giving 2.10 g (44%) of white crystals, mp.  $224\text{--}225^\circ$  (corr.), lit.<sup>1</sup> mp.  $224\text{--}225^\circ$ .

Procedure 2 (Iodine).— Thiolacetic acid (1.0 ml, 11.0 mmoles) was allowed to react with 608 mg (2.39 mmoles) of iodine in a round bottom flask under a  $\text{CaCl}_2$  drying tube at room temperature for one day. The contents of the flask were dissolved in 30 ml of chloroform and washed with two 30 ml portions of a 5% sodium bicarbonate solution. The chloroform layer was dried over magnesium sulfate, filtered and concentrated to about 20 ml. The resulting solution was then chromatographed over a basic alumina column (20 x 3.3 cm) and eluted with chloroform, yielding 0.31 g of crude product. Upon recrystallization from chloroform (1.5 M solution), 0.22 g (52%) of I was obtained, mp.

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224-225°.

IR (KBr): 2960  $\text{cm}^{-1}$  (m), 2908  $\text{cm}^{-1}$  (w), 1433  $\text{cm}^{-1}$  (m), 1362  $\text{cm}^{-1}$  (s), 1352  $\text{cm}^{-1}$  (s), 1086  $\text{cm}^{-1}$  (s), 1022  $\text{cm}^{-1}$  (s), 715  $\text{cm}^{-1}$  (vs).

$^1\text{H}$  NMR ( $\text{CDCl}_3$ ): 2.09 ppm (s,  $\text{CH}_3$ ).

$^{13}\text{C}$  NMR ( $\text{CDCl}_3$ ); 58.4 ppm (s, quaternary carbon); 29.14 ppm (quartet,  $\text{CH}_3$ ).

Anal. Calcd for  $\text{C}_8\text{H}_{12}\text{S}_6$ : C, 31.96; H, 4.02.

Found: C, 32.04; H, 3.88

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