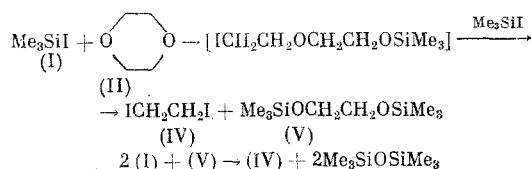
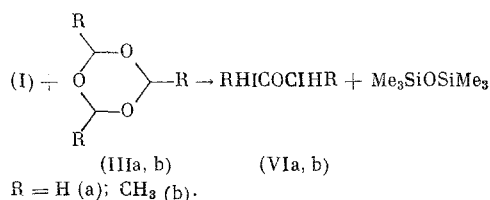


UDC 542.92:547.593.23:547.1'128

Contrary to the data in [6], we were able to cleave (II) using (I).



From (I) and the trioxanes (III) under analogous conditions are easily formed the bis(iodomethyl) (VIa) and bis( $\alpha$ -iodoethyl) (VIb) ethers:



## EXPERIMENTAL

The reactions were run in a dry argon atmosphere. Trimethyliodosilane (I) was obtained as described in [7].

Reaction of 1,4-Dioxane (II). To 7.2 g (0.082 mole) of (II) was added 32.9 g (0.164 mole) of (I) in drops. Then the mixture was heated for 3 h at 40°. Distillation gave 3.9 g (30%) of HMDS, bp 96°, 22 g (95%) of ICH<sub>2</sub>-CH<sub>2</sub>I (IV), mp 79°. Found: C 8.31; H 1.23; I 90.93%. C<sub>2</sub>H<sub>4</sub>I<sub>2</sub>. Calculated: C 8.52; H 1.43; I 90.05%. PMR spectrum ( $\delta$ , ppm): 3.5 s (CH<sub>2</sub>I); 5.9 g (34%) of 1,2-bis(trimethylsiloxy)ethane (V), bp 60° (4 mm); d<sub>4</sub><sup>20</sup> 0.8422, n<sub>D</sub><sup>20</sup>

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TABLE 1. Reaction of Trimethyl-iodosilane (I) with Trioxanes (III)

Trioxane	(I)/(IIIa, b) mole ratio	Yield, %	
		(VI)	HMDS
(III a)	1	96	91
	2	96	95
	3	99,5	98,5
	4	67	60
	6	46	50
(III b)	2	93	94
	3	97	96
	4	70	70

1.4031. Found: C 46.02; H 10.82; Si 27.01%.  $C_8H_{22}Si_2O_2$ . Calculated: C 46.61; H 10.75; Si 27.25%. PMR spectrum ( $\delta$ , ppm): 0.08 s  $[(CH_3)_3SiO]$ ; 3.55 s  $(OCH_2)$ .

Reaction of 1,3,5-Trioxane (IIIa) and 2,4,6-Trimethyl-1,3,5-trioxane (IIIb). To 5.2 g (0.058 mole) of (IIIa) was added dropwise in 30 min 35 g (0.174 mole) of (I). The mixture was heated for 10 h at 40°. Distillation gave 13.8 g (98%) of HMDS and 25.8 g (99.5%) of bis(iodomethyl) ether (VIa), bp 62° (5 mm);  $d_4^{20}$  2.8259;  $n_D^{20}$  1.6720. Found: C 8.17; H 1.07; I 87.53%.  $C_2H_4OI_2$ . Calculated: C 8.06; H 0.75; I 88.04%. PMR spectrum ( $\delta$ , ppm): 5.62 s  $(ICH_2O)$ .

$\alpha, \alpha'$ -Diiododiethyl ether was obtained in a similar manner from (IIIb), bp 71° (5 mm),  $d_4^{20}$  2.2213;  $n_D^{20}$  1.5750. Found: C 14.73; H 2.46; I 78.03%.  $C_4H_8OI_2$ . Calculated: C 14.74; H 2.47; I 77.88%. PMR spectrum ( $\delta$ , ppm): 6.09 m  $(OCH_2I)$ ; 2.17 d  $(CH_3C)$ .

Reaction of 1,2-Bis(trimethylsiloxy)ethane (V). To 16.9 g (0.08 mole) of (V) was added 32.9 g (0.164 mole) of (I) in drops and the mixture was heated for 6 h. We isolated 12.6 g (95%) of HMDS and 22.7 g (98%) of 1,2-diiodoethane (IV).

## CONCLUSIONS

The reaction of  $Me_3SiI$  with 1,4-dioxane gives 1,2-diiodoethane, 1,2-bis(trimethylsiloxy)ethane, and hexamethyldisiloxane, while reaction with 1,3,5-trioxane and 2,4,6-trimethyl-1,3,5-trioxane respectively gives the  $\alpha, \alpha'$ -diiodomethyl and ethyl ethers and HMDS.

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