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FACILE REDUCTION OF SULFOXIDES WITH
SODIUM BIS(2-METHOXYETHOXY) ALUMINUM HYDRIDE

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In the course of investigating the reducing power of sodium bis-(2-methoxyethoxy) aluminum hydride¹ (Red-al^R), we have subjected oxygenated sulfur compounds to this reducing agent. It was found that sulfones can be converted into sulfides under prolonged reflux in benzene, but the reaction was always incomplete thus rendering it impractical. However, deoxygenation of sulfoxides to sulfides by Red-al^R is extremely facile and exothermic.

Two procedures have been developed for the reduction of sulfoxides. Low boiling sulfides (e.g. dimethyl sulfide) may be fractionated from the reaction flask directly since the reduction does not require hydrolysis. The high boiling or solid sulfides were isolated by routine extraction. The yields were in general nearly quantitative; the lower figure from dimethyl sulfoxide reduction might be due to mechanical loss as dimethyl sulfide is extremely volatile. The use of Red-al^R to reduce sulfoxides is convenient and homogeneity is maintained.² In our opinion, the procedure is superior or comparable to any existing method.³

EXPERIMENTAL

Reduction of Dimethyl Sulfoxide - Red-al (15 ml, 3.46 M in benzene, from Aldrich Chemical Co.), was added dropwise to stirred, warm (65°) dimethyl sulfoxide (10 ml). The distillate was collected in a Dry-Ice-acetone

T-L. HO AND C. M. WONG

cooled flask and was fractionated through a Vigreux column to give dimethyl sulfide (2.26 g, 70%; bp. 36-38°).

Reduction of Higher Molecular Weight sulfoxides.- A Red-al solution (4.3 ml, 3.46 M) was added slowly to the sulfoxide (10 mmole) in benzene (10 ml). The resulting mixture was stirred under reflux for 0.5 hr., cooled and poured into 1N hydrochloric acid. The layers were separated and the aqueous phase was extracted with chloroform (25 ml). The combined organic phase was dried over MgSO₄, evaporated to dryness and the residue (tlc pure) was distilled or recrystallized to give the sulfide.

TABLE 1 - RED AL^R REDUCTION OF SULFOXIDES

Sulfoxide	Yield (%)	Sulfide Bp. (mp.)	Lit. ⁴
Dimethyl sulfoxide	70	36-38°	38°
Di- <u>n</u> -butyl sulfoxide	98	183-186°	182°
Thioanisole S-oxide	98	191-194°	194-196°
Diphenyl sulfoxide	97	192-5°/60torr	189°/50torr
Dibenzothiophene oxide	95	(96-99°)	(99°)

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