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### Hazards in the Chemical Laboratory



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Edited by L. Bretherick, Safety Consultant

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Hazards in the Chemical Laboratory
4th Edition is edited by Les Bretherick,
presently a consultant and formerly a
Senior Project Leader with BP
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Thames. Les Bretherick is well known
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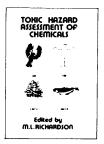
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Edited by:

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## Disilane Dialog

A literature search on **hexachlorodisilane** quickly reveals the interdisciplinary interest in this versatile chemical. The patent literature reflects considerable activity in areas such as catalysis and olefin metathesis, <sup>1,2</sup> deposition and epitaxial doping, <sup>3-5</sup> photoconductive elements, <sup>6,7</sup> and polymer research. <sup>6,9</sup> Organosilane chemistry is another active area <sup>10-13</sup> which includes hetero-π-systems, such as silabenzenes. <sup>14,15</sup>

Use of **hexachlorodisilane** as a reagent in organic synthesis grew from the realization that the Si-Si bond could be cleaved by various agents, most notably oxides. <sup>16,17</sup> Higher silicon chlorides were suggested as reducing agents for use in nonaqueous systems where hydride or metallic reducing agents were incompatible. <sup>18</sup> Its use in the synthesis of optically active phosphines promptly followed (eq. 1). <sup>19</sup> Mild conditions give good yields and high stereospecificities with nearly complete inversion of configuration.

$$Si_2Cl_6$$
 +  $O=P \xrightarrow{R_1} R_2 \xrightarrow{R_2 \longrightarrow P} P$ : +  $Si_2OCl_6''$ 
 $R_3$  (eq. 1)

**Hexachlorodisilane** also reduces amine oxides to amines and sulfoxides to sulfides. <sup>20</sup> However, phosphine sulfides are reduced to phosphines with retention of configuration in high optical yields. <sup>21</sup> Similar results are observed in the reduction of cyclic phosphine oxides, <sup>22</sup> where structural constraints apparently promote the retention of configuration. Applications of this chemistry include conversion of *cis*-azoxyalkanes to unstable *cis*-azo compounds (eq. 2), <sup>23</sup> reduction of 1-halophospholine oxides (eq. 3), <sup>24</sup> and deoxygenation of a diphenylthiirene 1-oxide (eq. 4). <sup>25</sup>

In addition to the advantages provided by the mild reaction conditions (ice bath/ambient temperature), the selectivity of **hexachlorodisilane** deoxygenation of N-oxides minimizes product rearrangement and over-reduction of C = N bonds<sup>26</sup> (eq. 5).

This technique has been extended to certain novel dehydroxylations (eq. 6).

Hexamethyldisilane is less sensitive to water and oxygen than hexachlorodisilane and, along with several other disilanes, has been used in the deoxygenation of nitrobenzenes and nitrotoluenes? (eq. 7); severe reaction conditions may limit the exploitation of this method.

The chemistry of **trichlorosilane**, a widely used reagent, has been extended to the reduction of bridged cyclic phosphine oxides.<sup>28</sup>

For a computer printout of all our organosilanes write or call our Technical Services Department (toll-free 800-231-8327).

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