Silicon-Based Synthetic Reagents:

Protected Organic Molecules

Gelest

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Maxfield Parish

Morning

Maxfield Parish (1870-1966) is among the most important American illustrators of the 20th century. He carried out his undergraduate studies at Haverford College in Pennsylvania, where as part of a liberal arts program, he completed a course in chemistry. Our front cover is a reproduction of his laboratory notebook pages dated Friday May 23, 1890.

His more well-known pieces maintain the fantasy and freshness demonstrated in his chemistry notebook. His celebrated works are known for their photorealism and a luminescent quality that he later perfected. From a chemist's perspective this was, without doubt, due to the chemistry training that encouraged him to experiment with new materials and techniques. (Cover Art Courtesy of Haverford College Library, Haverford, PA - Quaker Collection)

Silicon-Based Synthetic Reagents: Organosilyl-Protected Organic Molecules

Di- and trisubstituted silyl groups have long been used as a temporary replacement for active hydrogens and, in fact, trialkylsilyl groups have been referred to "fat hydrogens". The advantages of this chemistry can be attributed to several factors among which are: 1) the facile ability to replace an active hydrogen, such as that of an OH or NH group with a silyl group; 2) the capability for the ready removal of the silyl group; 3) the ability to sterically and electronically vary the silyl group, thereby modifying the stability of the protected system; and 4) the ability to modify the chemistry of the original group by making it more sterically encumbered or eliminating the ability to form hydrogen bonds. Herein is presented a sampling of organosilanes wherein an active hydrogen has been replaced by a silyl group. Among these are silyl carboxylates, silyl ethers, silyl amines, and silyl enol ethers.

RESISTANCE OF SILYLATED ALCOHOLS TO CHEMICAL TRANSFORMATIONS

$t^{1/2}$ for Si-OR bond scission at room temperature

		reagents					
Blocking group	**Substrate**	**HCl - THF**	**KF - methanol**	**CH_3MgBr in ether**	**n-Butyl lithium**	**LAH - THF**	**Pyridinium Chlorochromate**
	n-butanol	<15 min	2 min	48 hr	2 hr	30 min	<30 min
$(CH_3)_3Si$-	cyclohexanol	<15 min	2 min	>48 hr	3 hr	1 hr	<30 min
	t-butanol	<15 min	24 hr	>48 hr	50 hr	24 hr	<30 min
	n-butanol	<15 min	2 hr	no reaction	24 hr	1 hr	<30 min
$(C_2H_5)_3Si$-	cyclohexanol	<15 min	20 hr	no reaction	>48 hr	2 hr	<30 min
	t-butanol	<15 min	no reaction	no reaction	no reaction	no reaction	1 hr
cyclohexylMe_2Si-	cyclohexanol	< 15 min	10 hr	no reaction	36 hr	2 hr	<30 min
$^{i}Pr(CH_3)_2Si$-	cyclohexanol	10-30 min	24-30 hr	no reaction	>60 hr	3 hr	<30 min
	n-butanol	>3 hr	no reaction	no reaction	no reaction	25 hr	10 hr
$^{t}BuMe_2Si$-	cyclohexanol	>3 hr	no reaction	no reaction	no reaction	>50 hr	>20 hr
	t-butanol	no reaction	no reaction	no reaction	no reaction	no reaction	>20 hr
	n-butanol	16 hr	no reaction	no reaction	no reaction	>30 hr	22 hr
^{t}HexylMe_2Si-	cyclohexanol	30 hr	no reaction	no reaction	no reaction	no reaction	50 hr
	t-butanol	no reaction	no reaction	no reaction	no reaction	no reaction	no reaction
$^{i}Pr_3Si$-	cyclohexanol	no reaction	no reaction	no reaction	no reaction	no reaction	>72 hr
	n-butanol	no reaction	100 hr	no reaction	no reaction	no reaction	no reaction
$^{t}BuPh_2Si$-	cyclohexanol	no reaction	no reaction	no reaction	no reaction	no reaction	no reaction
	t-butanol	no reaction	no reaction	no reaction	no reaction	no reaction	no reaction

SILYLATED AMINES – Section 1

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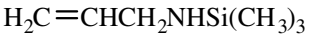

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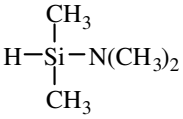

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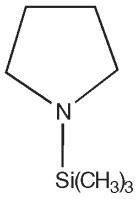

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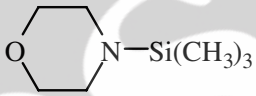

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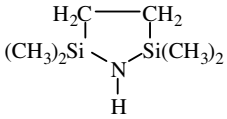

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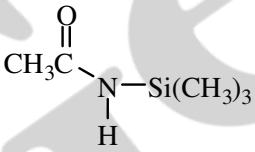

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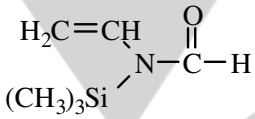

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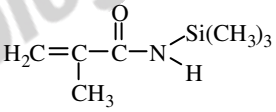

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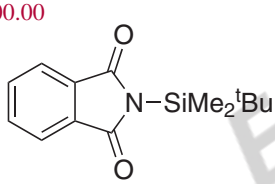

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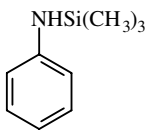

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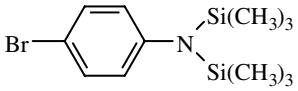

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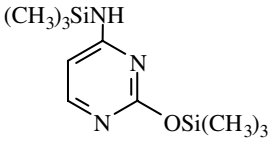

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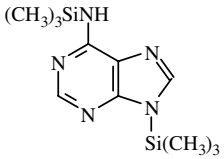

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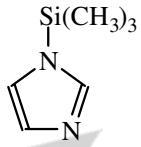

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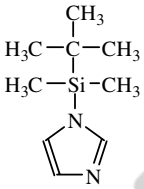

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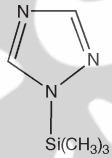

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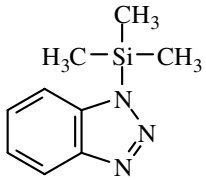

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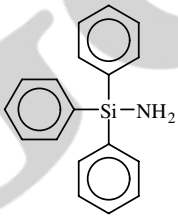

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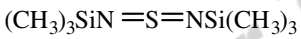

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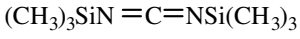

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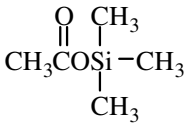

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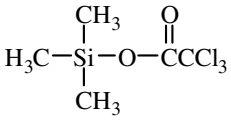

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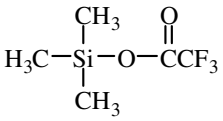

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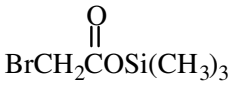

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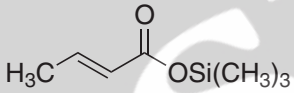

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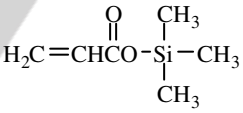

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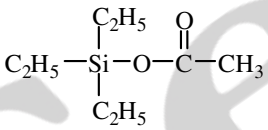

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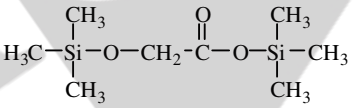

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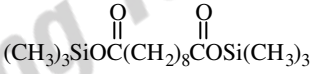

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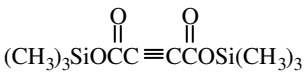

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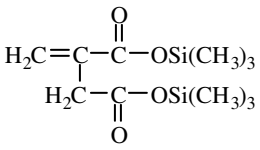

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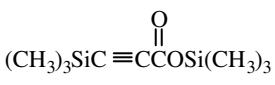

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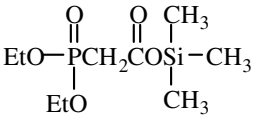

SILYL ENOL ETHERS – Section 3

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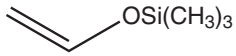

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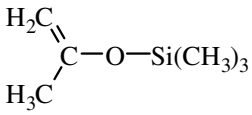

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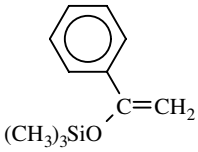

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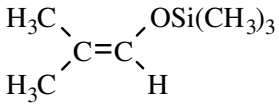

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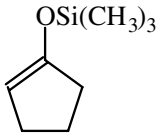

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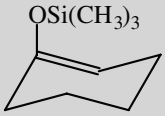

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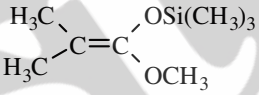

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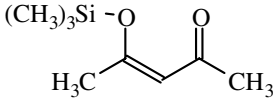

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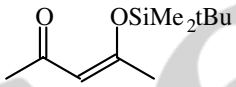

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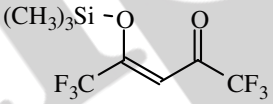

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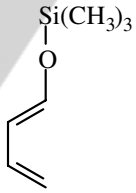

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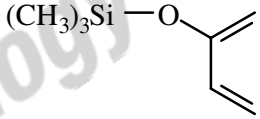

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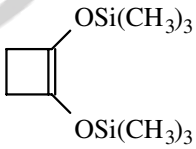

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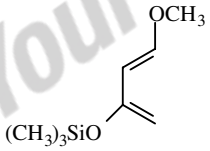

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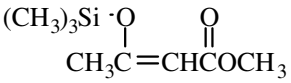

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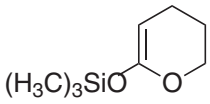

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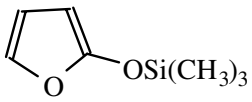

SILYLATED ALCOHOLS – Section 4

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SIA0476.0
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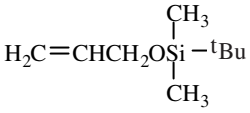

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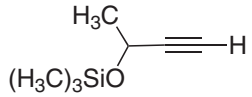

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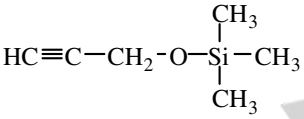

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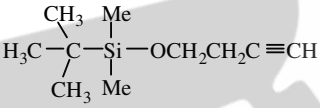

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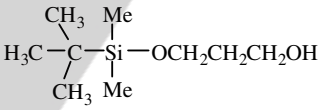

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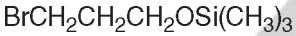

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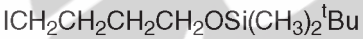

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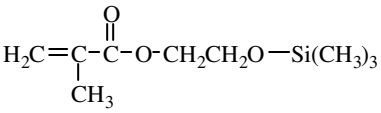

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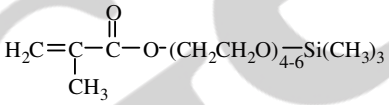

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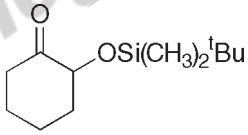

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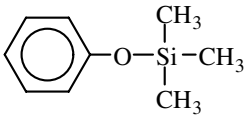

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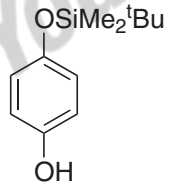

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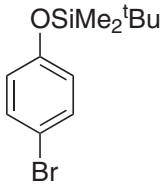

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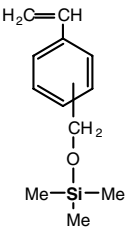

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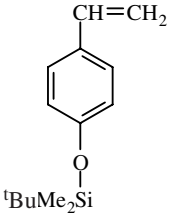

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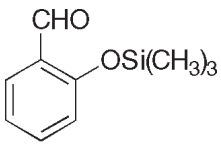

SILYLATED ALCOHOLS – Section 4 (cont.) –

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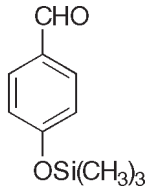

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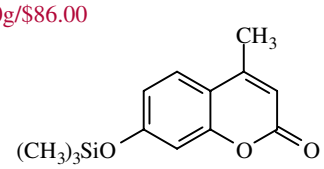

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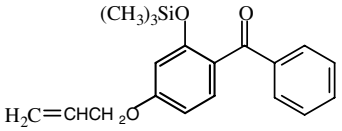

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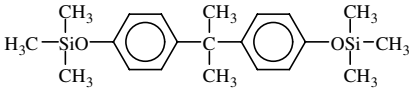

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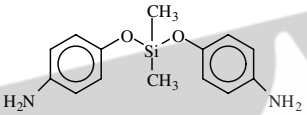

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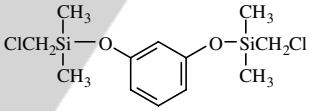

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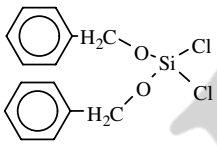

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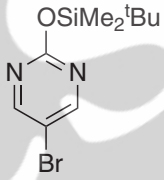

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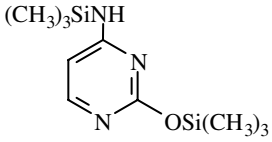

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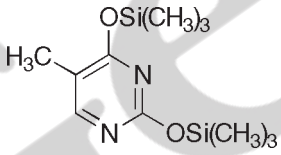

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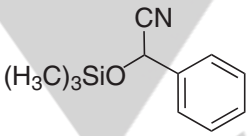

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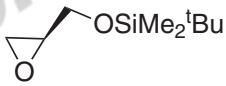

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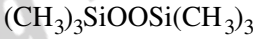

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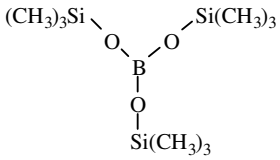

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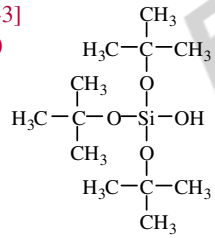

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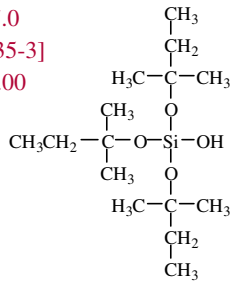

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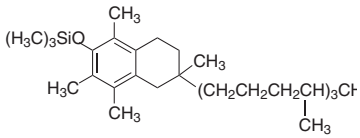

SILYLATED THIOLS – Section 5

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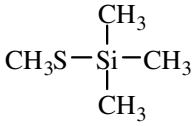

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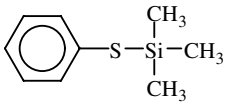

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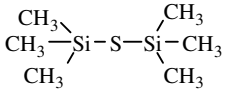

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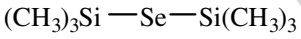

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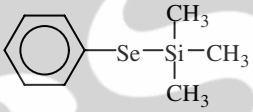
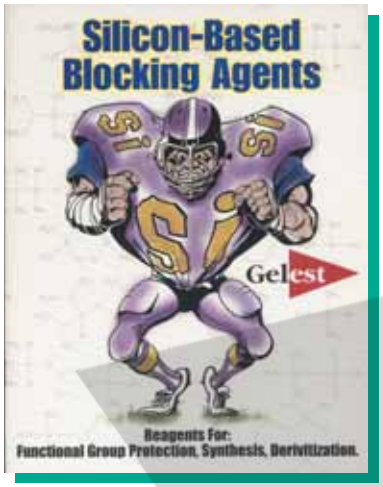


Gelest Synthetic Chemistry Product Literature

Silicon-Based Blocking Agents

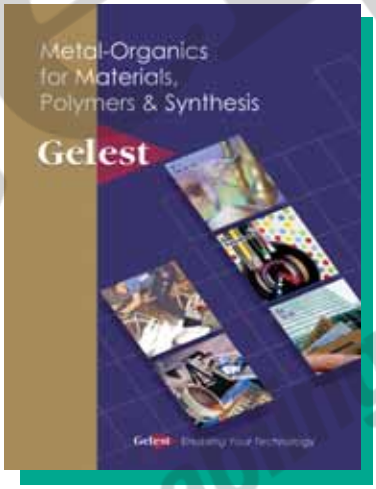


These silicon reagents are used for functional group protection, synthesis and derivatization. The 28 page brochure presents detailed application information on silylation reagents for pharmaceutical synthesis and analysis. Detailed descriptions are presented on selectivity for reactions, resistance to chemical transformations and selective deblocking conditions. Over 300 references are provided.

Silicon Based Reducing Agents

These silicon-based reagents are employed in the reduction of various organic and inorganic systems. The 24 page brochure presents information complete with literature references for a variety of reductions using organosilanes.

Metal-Organics for Materials, Polymers & Synthesis

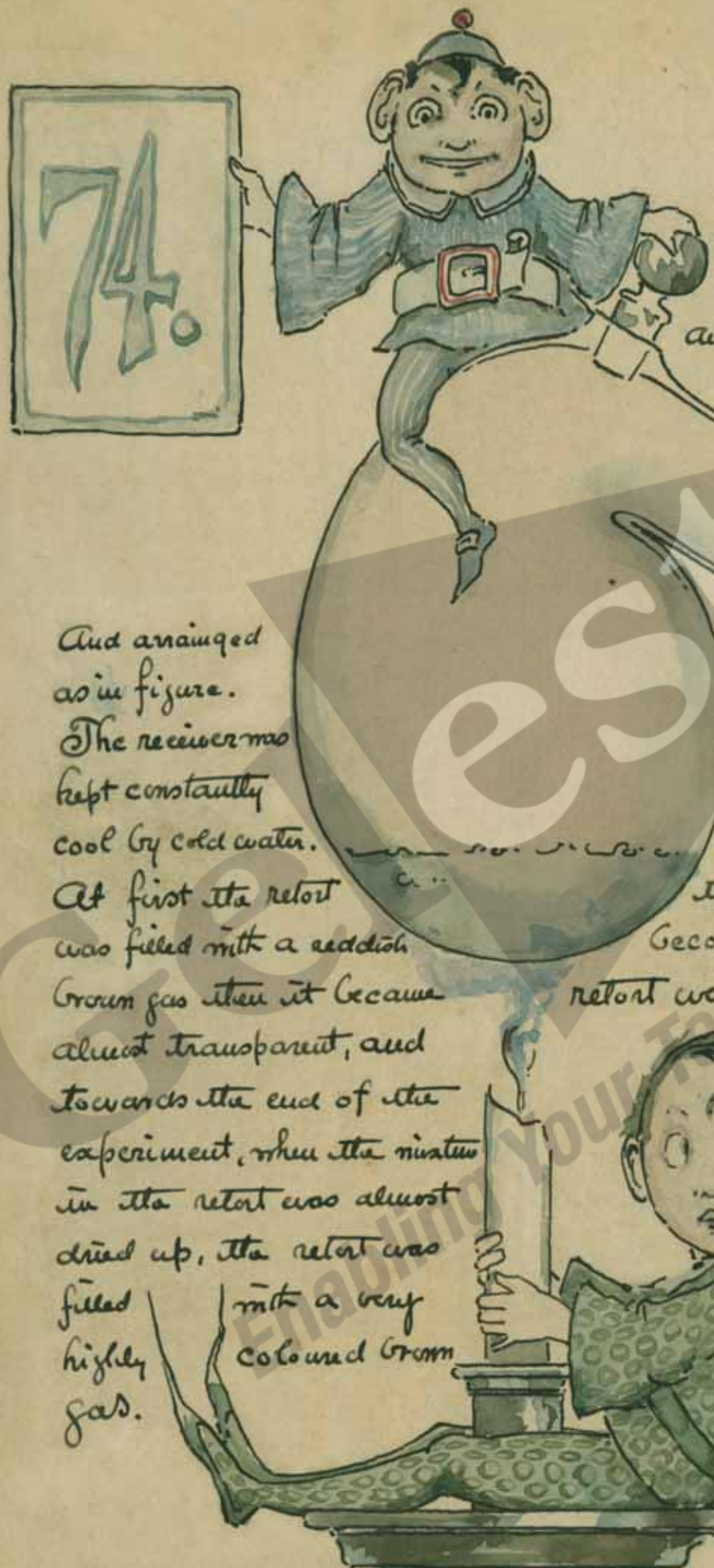


The latest Gelest catalog provides many new compounds with applications on optical, microelectronic, diagnostic and materials applications. Highly referenced listings and device applications are presented.

Silicon Compounds: Silanes & Silicones

Detailed chemical properties and reference articles for over 1500 compounds. The 560 page catalog of silane and silicone chemistry includes scholarly reviews as well as detailed application information.

74.



This experiment was to distill over nitric acid from a mixture of sodium nitrate and sulphuric acid. 25 grams of Sodium nitrate or (Chili saltpetre.) and 15 grams of concentrated sulphuric acid, were put into a retort.

And arranged as in figure. The receiver was kept constantly cool by cold water. At first the retort was filled with a reddish brown gas then it became almost transparent, and towards the end of the experiment, when the mixture in the retort was almost dried up, the retort was filled highly gas.

with a very coloured brown

After enough acid had been distilled, or rather when the sodium nitrate had become dry the contents of retort were crystallized from water.

The nitric acid obtained had a yellowish brown colour and appeared to be very strong as it gave off white fumes in contact with air.