On a TLC plate and in the dry state, 2 is destroyed when not protected from air and light. In chloroform solution at −20°, 2 seems to be stable.

The conditions necessary to mono-brominate 1 were somewhat more vigorous than those needed to produce the 9- or 7-bromo derivatives of the 1,3,6-triazacycl-[3.3.3]azine 3.[3]

Efforts to prepare 2, by treating 1 with bromine in glacial acetic acid were unsuccessful since 1 is unstable in this medium.

Attempts to decarboxylate 1 by the method used to prepare 4 [5,6] from its 4-carbethoxy derivative (diphenyl ether, traces of p-toluenesulphonic acid,[6] 100−258°) in order to obtain the symmetrical system 5 failed, since 1 was unstable under these conditions.
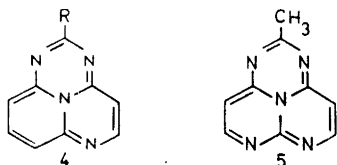
R = CH₃ and H

The observations reported above thus indicate that the 1,3,6,7-tetraazacycl[3.3.3]-azine system 1 is less susceptible to electrophilic substitution and, at least in some respects, chemically more unstable than the corresponding 1,3,6-analog 3.

Experimental. General. Nuclear magnetic resonance (NMR) spectra were determined in $CDCl_3$ with a Varian Model A-60 spectrometer, using tetramethylsilane as internal reference. Mass spectra were recorded with a GEC-AEI MS 902 instrument at the Department of Medical Biochemistry, University of Göteborg. Thin-layer chromatography (TLC) was performed on Silica Gel GF₂₅₄ (Merck) according to Stahl and the spots were visualized by means of short-wave ultraviolet light. For column chromatography, silica gel, 0.05−0.2 mm (Merck), was used.

Bromination of 1 with NBS. A mixture of 45 mg of 1 and 135 mg of N-bromosuccinimide in 9 ml of chloroform was stirred at ca. 25°. The formation of 2 was followed by TLC (EtOAc). After 6 h the starting material had vanished and red-violet 2 was present (R_F= 0.68). The volume of the reaction solution was reduced to ca. 5 ml and succinimide and un-

reacted NBS, which then precipitated, were removed by filtration. The filtrate was poured on to a column of silica gel (25 × 2.5 cm) and the red-violet band was eluted with chloroform−ethylacetate (1:1). After careful evap-, oration under reduced pressure at ca. 30° 36 mg (60 %) of 2 was obtained. It was immediately dissolved in chloroform and kept in the dark in a Dewar vessel together with dry ice.

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The Structure of 3,4-Dimethyl-6a-selenathiophthene

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So far X-ray structure determinations of two 6a-selenathiophthenes have been reported.[1,2] The Se−S bonds in 6a-selenathiophthene (I) [1] were found to be 2.446(5) Å, and the Se−S bonds in 2,5-diphenyl-6a-selenathiophthene (II) [2] were found to be 2.433(3) Å and 2.419(3) Å, respectively.

A structure investigation of 3,4-dimethyl-6a-selenathiophthene (III) has been carried out in order to obtain further information about the bonding in the 6a-

selenathiophthene system. The preliminary results are given here.

The 3,4-dimethyl-6a-selenathiophtheno molecule lies with the Se − C bond on a crystallographic two-fold axis, and the two halves of the molecule are therefore
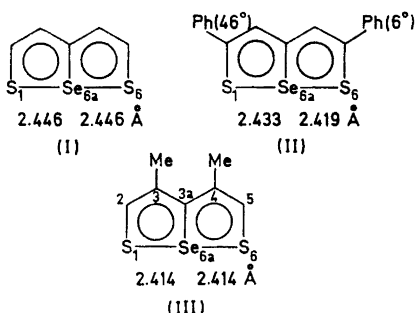
exactly equal. The Se − S distances are 2.414(1) Å with the S − Se − S angle equal to 176.2(1)°. Other bond lengths in the molecule are, $S(1) - C(2) = 1.691(3)$ Å, $C(2) - C(3) = 1.375(4)$ Å, $C(3) - C(3a) = 1.420(3)$ Å, and $Se(6a) - C(3a) = 1.917(3)$ Å.

A sample of III was generously supplied by Reid.[3] The crystals are deep red and belong to the monoclinic space group $C2/c$ with the cell dimensions $a = 15.913(2)$ Å, $b = 7.503(1)$ Å, $c = 7.280(2)$ Å, and $\beta = 99.79(2)°$. There are four molecules per unit cell; $D_c = 1.824$ g/cm³, $D_m = 1.83$ g/cm³.

The structure analysis is based on X-ray data collected on a paper-tape controlled Siemens AED diffractometer using MoKα radiation. 1874 reflections were observed within $\theta = 35°$.

The structure was solved by the heavy atom method and refined by full matrix least squares. The final R factor is 0.041.

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The Enthalpies of Formation of the Solid Compounds K₂MgCl₄, Rb₂MgCl₄, Cs₂MgCl₄, and KMgCl₃, RbMgCl₃, CsMgCl₃

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It is well-known that compounds of the type A_2MgCl_4 and $AMgCl_3$ (A = K, Rb, and Cs) are found in the systems ACl-MgCl₂. For instance, the phase diagram of the system KCl-MgCl₂ has recently been examined by Grjotheim, Holm and Røtnes.[1] They found two congruently melting compounds, K_2MgCl_4 and $KMgCl_3$, in this system. In the case of the two systems RbCl-MgCl₂ and CsCl-MgCl₂, phase diagram examinations by Markov and Panchenko[2] show the existence of the following four congruently-melting compounds: Rb_2MgCl_4 and $RbMgCl_3$ in the former system, and Cs_2MgCl_4 and $CsMgCl_3$ in the latter. In Table 1 are given the structures and melt-

Table 1. Structures and melting temperatures for alkali chloride-magnesium chloride compounds.

Compound	Structure [3]	T_f/K
K_2MgCl_4	tetragonal	705[1]
Rb_2MgCl_4	tetragonal	740[2]
Cs_2MgCl_4	orthorhombic	813[2]
$KMgCl_3$	hexagonal	755[1]
$RbMgCl_3$	hexagonal	825[2]
$CsMgCl_3$	hexagonal	883[2]

ing points of the six compounds. The structures have been determined by Svedahl.[3]

While the enthalpies of mixing in the liquid state have been determined,[4] the enthalpies of reaction between the solid compounds are not known. These enthalpies can, however, be calculated from enthalpy data as shown by the following two cycles.