Pentasilicon dodecachloride reacts with anhydrous HCl at room temperature producing a mixture of $SiHCl_3$ and $SiCl_4$ along with traces of hydrogen and Si_2Cl_6. The reaction is essentially complete in 24 hr.

We are continuing investigations of the properties of this new substance with a particular interest in the structure. The fact that it melts at a higher temperature than Si_6Cl_{14} and is more soluble in $SiCl_4$ would be consistent with a more symmetrical structure. The possibility that this compound is the neopentasilane is suggested.

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The synthesis of N,N-difluorourea

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WHEN urea is subjected to fluorine, diluted with nitrogen (ca. 1:5 to 1:10) in a flow reactor at 0°, a complex yellow, corrosive liquid containing up to 20 per cent active fluorine (to HI) and about 45–55 per cent total fluorine is obtained. Among the gaseous products obtained from the distillation of this corrosive liquid was difluoramine as previously reported by LAWTON and WEBER.(1) However distillation of the liquid also yields a solid sublimate and a solid or solid-liquid pot residue. These solid products were shown to have oxidizing power by reaction with hydriodic acid, and the oxidizing solid species are soluble in water, dichloromethane, and tetrahydrofuran, but slightly soluble in diethylether. Extraction with either dichloromethane or tetrahydrofuran, and evaporation of the resulting solutions yields crude difluorourea. The difluorourea may be purified by sublimation to form long thin needles melting at 41·0°–41·5°.

TABLE 1.—INFRA-RED SPECTRUM OF UNSYMMETRICAL DIFLUOROUREA

Dilute soln (cm^{-1})	Solid state (cm^{-1}) (Nujol mull)	Intensity	Assignment
3520		*m*	N—H stretch (free)
	3425	*m*	N—H stretch (bonded)
3420		*m*	N—H stretch (free)
	3320	*m*	N—H stretch (bonded)
3200	3200	*w*	—
2940	—	*w*	—
1803	1790	*s*	Amide I
1592	1613	*m*	Amide II
1345	1343	*m*	—
1115	1118	*w*	—
1018	1020	*m*	—
925	925	*m*	N—F (tentative)
892	—	*w*	—
836	836	*w*	—

The solid was identified as unsymmetrical difluorourea by infra-red analysis and the structure was confirmed by wet chemical analysis. Infra-red spectra were obtained in dilute dichloromethane solution and in the solid phase as a mineral oil mull. All spectra were obtained on a Beckman IR-7 infra-red spectrophotometer. In dilute solution in non-polar solvents, primary amides give two absorption bands in the region 3500–3300 cm^{-1}. The first of these bands corresponds to the asymmetric stretching mode and is generally found around 3500 cm^{-1}. The second band, arising from the corresponding symmetrical mode, occurs around 3300 cm^{-1}. In dilute solution secondary amides

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(1) E. A. LAWTON and J. Q. WEBER, *J. Amer. Chem. Soc.* **81**, 4755 (1959).

give rise to a single absorption in the range of 3460–3420 cm^{-1}. Unsymmetrical difluorourea has two strong bands at 3520 and 3420 cm^{-1} respectively, indicating the presence of an NH_2 grouping.

The bands at 1803 cm^{-1} and 1592 cm^{-1} in dilute solution in a non-polar solvent have been assigned as the Amide I and Amide II bands of an amide, respectively. The assignment of these bands was confirmed by the shift of these bands in the solid state spectrum. The Amide I band shifted from 1803 cm^{-1} in solution to 1790 cm^{-1} in the solid state while the Amide II band shifted from 1592 cm^{-1} in dilute solution to 1613 cm^{-1} in the solid state. The direction of the shifts is in keeping with the proposed structure.(2) The large frequency shift of the Amide I band from a nominal 1700 cm^{-1} to 1803 cm^{-1} in dilute solution in a non-polar solvent indicates the presence of a strong electronegative group such as NF_2 attached directly to the CO—NH_2 structure. Accordingly, from the infra-red spectra, the chemical structure was confirmed as that of a primary amide.

TABLE 2.—SUMMARY OF WET CHEMICAL ANALYSES

Sought	Found	Calc
Active fluorine	38	39·6
Total fluorine	38·6	39·6
Total nitrogen	29·3	29·2
Amino nitrogen (NH_2)	14·6	14·6

The infra-red structural interpretation was confirmed by wet chemical analysis. Total fluorine was determined by the lead chlorofluoride titration,(3) while active fluorine was determined by reaction with potassium iodide and titration of the iodine liberated. Standard Kjeldahl techniques for nitrogen yielded one half of the theoretical nitrogen content because of the reaction of the —HF_2 group in acid to yield volatile difluoramine. Prior reduction of the —NF_2 group with HI yielded the theoretical nitrogen values. Accordingly, it was possible to differentiate between the NH_2 and NF_2 groups and confirm the unsymmetrical structure. The results of these analyses are shown in Table 2.

These data unambiguously identify the compound as unsymmetrical difluorourea, F_2NCONH_2. The hygroscopic compound may be stored in polyethylene but decomposes exothermically on being suddenly heated. The compound is a lachrymator and strong vesicant.

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Preparation of long-lived hafnium-182*

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IN a continuation of our programme(1,2) to investigate long-lived isotopes in the rare-earth region, a successful search for hafnium-182, produced by high flux neutron irradiation of hafnium-180, has been undertaken. For tantalum-182, the product of the β-decay of even–even hafnium-182, ground-state spins of 3 or 4 and odd parity are suggested by the coupling rule for odd–odd deformed core nuclei;(3)

* This work was performed under the auspices of the U.S. Atomic Energy Commission.

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