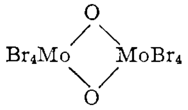
is quite strong. Because of the unambiguous stoichiometry of the isolated compound, a dimeric structure with two bridging groups seems to be the most plausible; either



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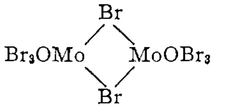
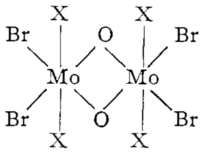


If bridging is by bromines the steric strain will probably be relieved principally by lengthening of the central Mo–Br bonds, without much additional distortion. If the bridging is by oxygen atoms it would appear difficult to maintain bond angles of 90° about the Mo atoms and the Mo–O bonds would probably be unusually long. In either case the Mo–Mo distance would undoubtedly be too great to lead to a decrease in the magnetic moment. Hence the magnetic moments of 1.77–1.78 reported by Klemm and Steinberg[19] for two salts of the type $M(MoOBr_4)$ do not rule out the dimeric structure.

A dimer with the formula



where X is either OH or O might not be subjected to such steric effects and thus might be diamagnetic. The existence of both paramagnetic and diamagnetic dimers might then be expected. Such has actually been observed in the chloro complexes of Mo(V).[7–9]

It is the consensus of workers on the similar system in HCl that monomeric $MoOCl_5^{2-}$ is the predominant form in high HCl concentrations and that a series of dimers appears at lower concentrations. The absence of significant amounts of $MoOBr_5^{2-}$ in the HBr solutions is the most marked difference between the bromo and chloro systems.

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[19] W. Klemm and H. Steinberg, *Z. anorg. allgem. Chem.*, **227**, 193 1936).

CONTRIBUTION FROM THE SAVANNAH RIVER LABORATORY,
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The Absorption Spectra of Uranium(V) Compounds[1]

BY D. G. KARRAKER

Received May 4, 1964

The absorption spectra of the uranium(V) ion in CCl_4 solutions of $UCl_5 \cdot SOCl_2$ and $[U(OC_2H_5)_5]_2$ were measured, and energy levels were assigned on the basis of a $5f^1$ configuration for the U(V) ion. Comparison of these energy levels with the theoretical levels for a $5f^1$ configuration was satisfactory, and the spin–orbit coupling constant for U(V) was calculated to be 1900 cm.$^{-1}$. The shifts in the energy levels between the two compounds were interpreted to result from a 30% stronger field on the U(V) ion in $[U(OC_2H_5)_5]_2$.

Introduction

The uranium(V) ion has one unpaired electron, probably in a $5f^1$ configuration, and is thus isoelectronic with Pa(IV) and Np(VI). The optical spectra and paramagnetic resonance of both Pa(IV) and Np(VI) have been previously measured and interpreted.[2–7] Similar measurements for U(V) would also be of interest, but are complicated by the difficulty of preparing U(V) compounds that have a known symmetry and are chemically stable. Absorption spectra of alkali metal–U(V) fluorides has been recently reported by Penneman, *et al.*[8,9] A partial interpretation of the spectra of these compounds has also been proposed.[10] This paper reports the absorption spectra of two U(V) compounds, uranium pentaethoxide and the thionyl chloride complex of uranium pentachloride, and proposes an interpretation of the spectra consistent with the energy levels in a $5f^1$ configuration.

Experimental

Materials. $UCl_5 \cdot SOCl_2$.[11]—$UCl_5 \cdot SOCl_2$ was prepared by refluxing uranium trioxide (20 g.) with an excess of thionyl chloride (300 ml.) for 3 weeks. The solution was filtered to remove unreacted UO_3, and the excess thionyl chloride was removed by distillation at atmospheric pressure, followed by vacuum distillation. The yield was essentially quantitative. *Anal.* Calcd. for $UCl_5 \cdot SOCl_2$: U, 44.6; Cl, 46.5. Found: U, 45.1; Cl, 46.2.

$[U(OC_2H_5)_5]_2$.[12]—Uranium(IV) chloride (19 g., 0.05 mole), dissolved in dry absolute ethanol, was added slowly to an ethanolic solution of sodium ethoxide (13.6 g., 0.20 mole) in a nitrogen

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[1] The information contained in this article was developed during the course of work under contract AT(07-2)-1 with the U. S. Atomic Energy Commission. Presented at the 147th National Meeting of the American Chemical Society, Philadelphia, Pa., April, 1964.

[2] J. D. Axe, R. Kyi, and H. J. Stapleton, *J. Chem. Phys.*, **32**, 1261 (1960).

[3] J. D. Axe, H. J. Stapleton, and C. D. Jeffries, *Phys. Rev.*, **121**, 1630 (1961).

[4] C. H. Hutchinson, Jr., and B. Weinstock, *J. Chem. Phys.*, **32**, 56 (1960).

[5] G. L. Goodman, Ph.D. Thesis, Harvard University, 1959.

[6] G. L. Goodman and M. Fred, *J. Chem. Phys.*, **30**, 849 (1959).

[7] J. C. Eisenstein and M. H. L. Pryce, *Proc. Roy. Soc.* (London), **A255**, 181 (1960).

[8] R. A. Penneman, G. D. Sturgeon, and L. B. Asprey, *Inorg. Chem.*, **3**, 126 (1964).

[9] L. B. Asprey and R. A. Penneman, *ibid.*, **3**, 727 (1964).

[10] M. J. Reisfield and G. A. Crosby, *J. Mol. Spectry.*, **10**, 232 (1963).

[11] D. C. Bradley, B. N. Chakravarti, and A. K. Chatterjee, *J. Inorg. Nucl. Chem.*, **3**, 367 (1957).

[12] R. G. Jones, *et al.*, *J. Am. Chem. Soc.*, **78**, 4287 (1956).

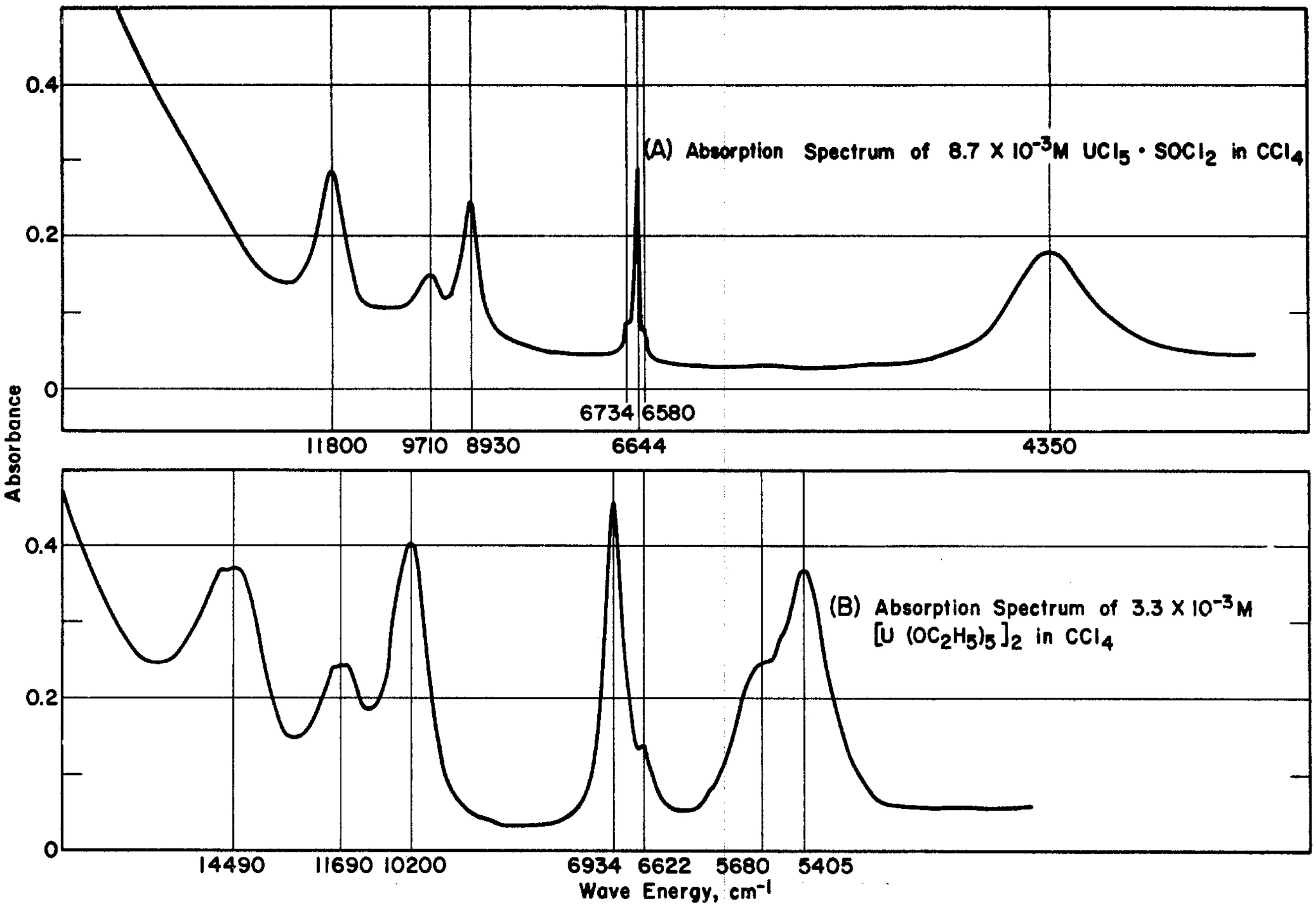

Fig. 1.—Absorption spectra of U(V) ion.

atmosphere to form uranium(IV) ethoxide. The uranium(IV) ethoxide was oxidized to a uranium(V) intermediate *in situ* by the slow addition of a benzene solution of bromine (4 g. of Br_2, 0.05 mole). The reaction mixture was stirred for about 45 min., then an ethanolic solution of sodium ethoxide (3.4 g., 0.05 mole) was added to produce uranium(V) ethoxide. The solvent was removed by vacuum evaporation, and the uranium(V) ethoxide was recovered from the residue by distillation at 160° under a diffusion-pump vacuum. The yield was 18 g., 80% of theoretical.

Anal. Calcd. for $[U(OC_2H_5)_5]_2$: U, 51.4. Found: U, 51.5.

CCl_4.—Carbon tetrachloride was Eastman Spectro Grade material that was further purified before use by refluxing overnight with mercury metal, distilling through a column, and vacuum distilling from phosphorus pentoxide.

Analytical Methods.—Standard analytical methods were used; uranium concentrations were determined from the absorbance of the uranyl thiocyanate complex, and chloride by titration with mercuric nitrate using diphenylcarbazone as an indicator.[13]

Spectral Measurements.—Absorption spectra for the range 2800–300 mμ (3600 to 33,000 cm.$^{-1}$) were measured at room temperature with a Beckman DK-2 spectrophotometer; the lead sulfide detector was used from 600 to 2800 mμ, and the photomultiplier detection unit for wave lengths between 300 and 600 mμ. Carbon tetrachloride solutions of $UCl_5 \cdot SOCl_2$ and $[U(OC_2H_5)_5]_2$ were prepared in an argon atmosphere, since both are rapidly oxidized by exposure to air. The samples were contained in 1-cm. quartz cells fitted with ground-glass stoppers to prevent exposure of the solutions to air. A matching quartz cell filled with CCl_4 was used as reference.

Solutions of ethanol and thionyl chloride in CCl_4 were measured to allow separation of the absorption spectra due to the ethoxy radical and thionyl chloride from absorption spectra due to the U(V) ion.

Absorption in the infrared region (2.5 to 15 μ) was measured on a Perkin-Elmer Model 221 spectrophotometer with NaCl optics. Solid samples were measured as mulls with paraffin oil or "Fluorolubes"[14]; $[U(OC_2H_5)_5]_2$ was measured as a liquid and as a 20% solution in CCl_4.

Results

The absorption spectra in the visible and near-infrared region for $UCl_5 \cdot SOCl_2$ and $[U(OC_2H_5)_5]_2$ are shown in Fig. 1, and the molar absorptivities at different wave lengths are tabulated in Table I. Bands at 9710 and 11,800 cm.$^{-1}$ found in CCl_4 solutions of $UCl_5 \cdot SOCl_2$ have also been observed in $POCl_3$ solutions of $UCl_5 \cdot PCl_5$.[15] Absorption in the ultraviolet region, resulting from charge-transfer transitions, was not investigated.

No infrared absorption was detected for any solid samples of $UCl_5 \cdot SOCl_2$ over the range 2.6–15 μ. Samples mulled with both paraffin oil and "Fluorolubes" were used to ensure that weak bands were not concealed by the absorption of one of the oils.

The infrared spectrum of uranium(V) ethoxide was reasonably complex; absorption occurred at 2990, 2880, 2720, 1455, 1375, 1350, 1130, 1100, 1052, 1025, 908, and 875 cm.$^{-1}$. All the absorption bands except those at 908 and 875 cm.$^{-1}$ could be assigned to vibrations occurring in the ethoxy radical.

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(13) F. E. Clarke, *Anal. Chem.*, **22**, 553 (1950).

(14) Hooker Electrochemical Co., Niagara Falls, N. Y.

(15) R. E. Panzer and J. F. Suttle, *J. Inorg. Nucl. Chem.*, **20**, 229 (1961).

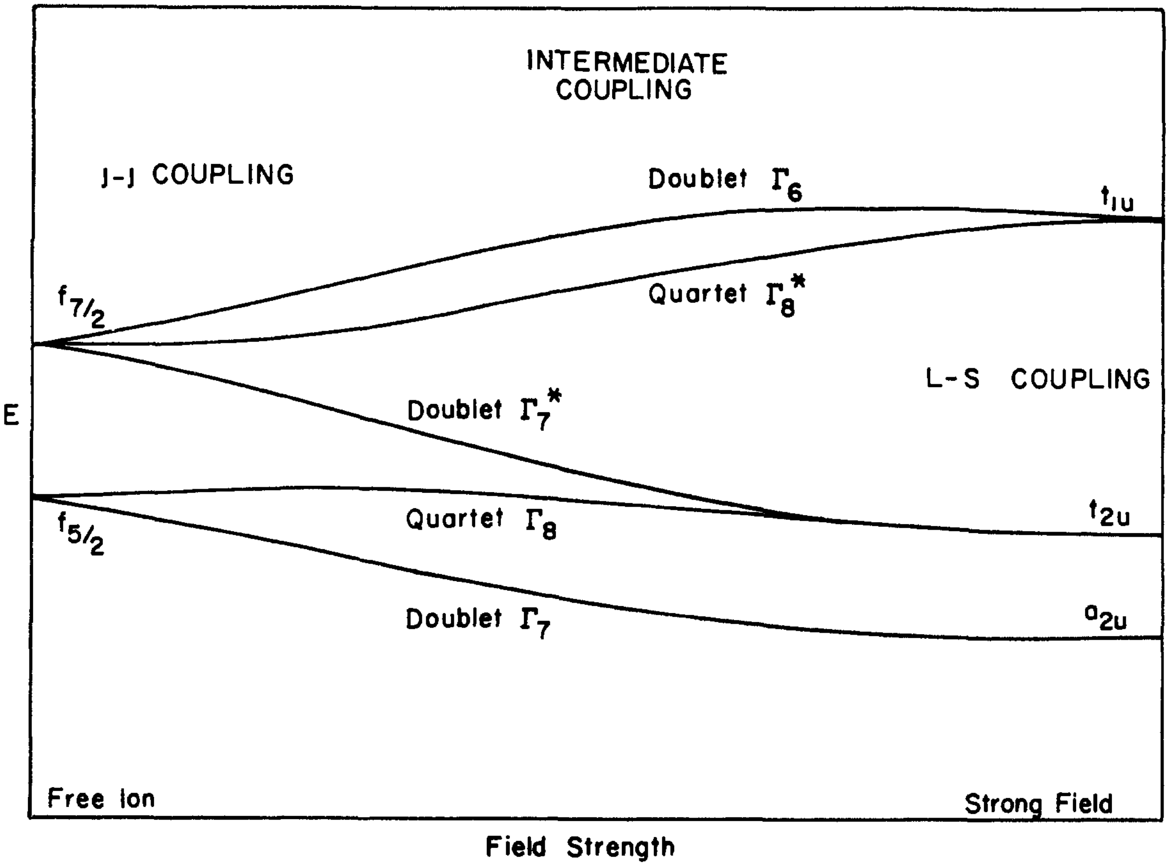

Fig. 2.—Field effects on energy levels of a 5f electron.

TABLE I

MOLAR ABSORPTIVITIES

$UCl_5 \cdot SOCl_2$		$[U(OC_2H_5)_5]_2$	
Wave energy, cm.$^{-1}$	ϵ_{max}, M^{-1} cm.$^{-1}$	Wave energy, cm.$^{-1}$	ϵ_{max}, M^{-1} cm.$^{-1}$
4,350	21	5,405	9.7
6,580	~2	5,680	5.5
6,644	33	6,622	~1
6,734	~2	6,934	13
8,930	22	10,200	12
9,710	5.8	11,690	4.2
11,800	21	14,490	6.7

Discussion

The energy levels for ions with a 5f^1 configuration have been calculated by several workers[4,5,7,16] for an octahedral field surrounding the central ion. Figure 2 reproduces a schematic by Goodman[5] (the graphs of ref. 4 and 16 are essentially the same) that illustrates the shifts in energy levels going from the free ion in space (j–j coupling) to the strong crystal-field limit (L–S coupling). The energy levels for actinides normally lie in the intermediate coupling region, where both spin–orbit coupling and crystal-field effects are observed. Goodman[5] has also derived explicit expressions for the energy levels of the 5f^1 ions in an octahedral field; these expressions, in a rearranged form, were used to fit the observed levels to the theoretical.[17]

The symmetry of the field surrounding the U(V) ion in CCl_4 solution is unknown, but a reasonable assumption is that $UCl_5 \cdot SOCl_2$ provides a field of C_{4v} symmetry, like an octahedral MA_5B molecule. The most likely alternate possibility, dissociation into UCl_5 and $SOCl_2$ in CCl_4 solution, can be ruled out by noting that attempts during this study to obtain the spectra of UCl_5 in CCl_4 were unsuccessful because of the decomposition of UCl_5 to form an unidentified precipitate. Solutions of UCl_5 in $SOCl_2$ are reasonably stable because of the formation of the $UCl_5 \cdot SOCl_2$ complex.

Uranium(V) ethoxide is a dimer in benzene[12] and would be expected to be a dimer in CCl_4. Evidence for dimer structure is afforded by two infrared absorption

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[16] J. D. Axe, Ph.D. Thesis, University of California (Berkeley), 1960 (UCRL-9293).

[17] Energy Levels for 5f^1 Electrons.—Goodman's equations for the energy levels were rearranged to set the Γ_7 level equal to zero, resulting in the equations

$$E_{\Gamma_8} = \frac{7}{2}\left\{R + Q + \frac{1}{2}(\zeta^2 - 4\zeta R/7 + 4R^2)^{1/2} - \frac{1}{2}(\zeta^2 - 8\zeta Q/7 + 4Q^2)^{1/2}\right\}$$

$$E_{\Gamma_8^*} = \frac{7}{2}\left\{R + Q + \frac{1}{2}(\zeta^2 - 4\zeta R/7 + 4R^2)^{1/2} + \frac{1}{2}(\zeta^2 - 8\zeta Q/7 + 4Q^2)^{1/2}\right\}$$

$$E_{\Gamma_7^*} = \frac{7}{2}(\zeta^2 - 4\zeta R/7 + 4R^2)^{1/2}$$

$$E_{\Gamma_6} = \frac{7}{2}\left\{R + 2Q + \zeta/2 + \frac{1}{2}(\zeta^2 - 4\zeta R/7 + 4R^2)^{1/2}\right\}$$

ζ is the spin–orbit coupling constant; Q and R are energy terms for the electric field and are related to the customary energy terms by the equations

$$A_4^0\langle r^4\rangle = 21(3Q + 2R)/4$$

$$A_6^0\langle r^6\rangle = 39(5Q - 4R)/40$$

bands, at 908 and 875 cm.$^{-1}$. These bands are near the UO_2^{2+} absorption at 965 cm.$^{-1}$,[18] and thus are assigned to U–O stretching frequencies. The area under the 908 cm.$^{-1}$ absorption is four times as great as the area under the 875 cm.$^{-1}$ band. This is consistent with a dimer, where the 908 cm.$^{-1}$ band results from a U–OR stretching frequency and the 875 cm.$^{-1}$ band results from a $\text{U–}\overset{\text{R}}{\text{O}}\text{U}$ stretching frequency. The dimer molecule may be assumed to be two octahedra joined along one edge by sharing two ethoxy oxygen atoms between them.

The assignment of energy levels for a $5f^1$ configuration for the spectral data was made by assuming the uranium ion in both molecules could be considered to be in a distorted octahedral field. The effect of the distortion was treated as equivalent to the Jahn–Teller distortion of an octahedral molecule. In Jahn–Teller distortions, doublet levels occurring from Kramers degeneracy (Γ_7, Γ_7^*, Γ_6) will be stabilized by spin–orbit coupling[19] and would be essentially unaffected by the distortion; quartet levels (Γ_8, Γ_8^*) have been predicted[7] to split into two doublets or interact with the vibration modes to produce weak, broad absorption bands.

The energy levels for the U(V) ion in the $UCl_5 \cdot SOCl_2$ molecule were assigned as $\Gamma_8 = 4350$, $\Gamma_7^* = 6580$, $\Gamma_8^* = 9320$, and $\Gamma_6 = 11{,}800$ cm.$^{-1}$, referred to $\Gamma_7 = 0$. The Γ_8 level was assigned at the midpoint of the broad absorption at 4350 cm.$^{-1}$, and the Γ_8^* level was chosen as the average of 8930 and 9710 cm.$^{-1}$, following the arguments presented above. The experimental values for the Γ_7^*, Γ_8^*, and Γ_6 levels were substituted into Goodman's equations[17] and the equation solved by iteration for values of the spin–orbit coupling constant, ζ, and the energy terms Q and R that gave the best fit to the experimental levels. (Q and R are related to the energy of the electric field on the electron and are defined[17] in terms of the conventional parameters $A_4^0 \langle r^4 \rangle$ and $A_6^0 \langle r \rangle^6$.) From the values for ζ, Q, and R, the energy of Γ_8 was calculated. A comparison between the assigned and the calculated levels is shown in Table II.

TABLE II

ENERGY LEVELS FOR U(V) IN $UCl_5 \cdot SOCl_2$[a]

Level	Assigned	Calculated
Γ_7	0	0
Γ_8	4,350	3,020
Γ_7^*	6,580	6,604
Γ_8^*	9,320	9,780
Γ_6	11,800	11,831

[a] All values in cm.$^{-1}$; $Q = 654$, $R = 274$, $\zeta = 1900$ cm.$^{-1}$.

A comparison of the assigned and calculated levels for the U(V) ion in $[U(OC_2H_5)_5]_2$ is shown in Table III.

The parameters calculated for U(V) ion are compared with those for the isoelectronic species Pa(IV) and Np(VI) in Table IV.

TABLE III

ENERGY LEVELS FOR U(V) IN $[U(OC_2H_5)_5]_2$[a]

Level	Assigned	Calculated
Γ_7	0	0
Γ_8	5,542	3,890
Γ_7^*	6,622	6,686
Γ_8^*	10,845	11,810
Γ_6	14,490	14,524

[a] All values in cm.$^{-1}$; $Q = 975$, $R = 293$, $\zeta = 1905$ cm.$^{-1}$.

TABLE IV

PARAMETERS FOR $5f^1$ IONS

	Cs_2PaCl_6[16]	$UCl_5 \cdot SOCl_2$	$[U(OC_2H_5)_5]_2$	NpF_6[7]
$A_4^0\langle r^4 \rangle$	888	12,500	18,400	5738
$A_6^0\langle r^6 \rangle$	41.9	970	3,630	540.5
ζ	1490	1,900	1,905	2405

[a] All values in cm.$^{-1}$.

The agreement between calculated levels and assigned levels is considered to be fairly good for both $UCl_5 \cdot SOCl_2$ and $[U(OC_2H_5)_5]_2$. The value for the spin–orbit coupling constant is in good agreement with an average of the Pa(IV) and Np(VI) values, as predicted by Conway.[20] The values of $A_4^0\langle r^4 \rangle$ and $A_6^0\langle r^6 \rangle$ are less satisfactory and indicate that higher terms arising from deviations from octahedral symmetry are also important. The parameters for $[U(OC_2H_5)_5]_2$ deviate much farther from a reasonable value than do those for $UCl_5 \cdot SOCl_2$—a predictable result, since the assumption of distorted octahedral symmetry is obviously less justified for $[U(OC_2H_5)_5]_2$ than for $UCl_5 \cdot SOCl_2$.

The values of R and ζ were used to calculate g, the spectroscopic splitting factor, from the expression[5]

$$g = \frac{(6 - 8a)}{(a^2 + 3)}$$

where

$$a = \frac{1}{4}\{1 - 14(R/\zeta) + 7[1 - 4R/7\zeta + 4(R/\zeta)^2]^{1/2}\}$$

The calculations yielded $g = -1.18$ for $UCl_5 \cdot SOCl_2$ and $g = -1.16$ for $[U(OC_2H_5)_5]_2$. A determination of g for U(V) ions in a matrix of ThO_2 by paramagnetic resonance showed $|g| = \sim 1.25$,[21] with the sign undetermined. The value of g is expected to be negative, since $g = -1.14$ for Pa(IV)[3] and $g = -0.60$ for Np(VI).[4] Accepting the sign of g as negative, the agreement between the experimental value and the calculated values is reasonable and sustains the interpretation proposed for the data.

Alternative interpretations of the spectra were attempted and discarded. These interpretations assumed that an additional forbidden level not due to f–f transitions was present at 11,800 cm.$^{-1}$ or 9710 cm.$^{-1}$ (for $UCl_5 \cdot SOCl_2$). Fitting the data on either assumption led to values of ζ of about 1400 cm.$^{-1}$ and $g = -0.6$, neither of which is credible. The possibility that the energy levels of U(V) might be explained by a $6d^1$ configuration was also considered. The d–d transition would be predicted to show two major absorption bands,

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(18) L. H. Jones and R. A. Penneman, *J. Chem. Phys.*, **21**, 542 (1953).

(19) C. J. Ballhausen, "Introduction to Ligand Field Theory," McGraw-Hill Book Co., Inc., New York, N. Y., 1962, p. 200.

(20) J. G. Conway, *J. Chem. Phys.*, **31**, 1002 (1959).

(21) P. M. Llewellyn, unpublished, quoted in W. Low, "Paramagnetic Resonance in Solid State Physics," Suppl. 2, Academic Press, New York, N. Y., 1960.

rather than the five observed. The sharp absorption bands are also characteristic of f–f transitions; d–d bands are ordinarily much broader than those observed.

The qualitative differences between U(V) spectra in $[U(OC_2H_5)_5]_2$ and in $UCl_5 \cdot SOCl_2$ indicate a stronger crystal field in $[U(OC_2H_5)_5]_2$. In $[U(OC_2H_5)_5]_2$ all absorption bands are shifted to shorter wave lengths and spaced over a 1600 cm.$^{-1}$ wider energy range. The directions of the energy shifts are as predicted in Fig. 2 for an increased crystal field in $[U(OC_2H_5)_5]_2$. The energy change between the Γ_7^* levels is small, but the Γ_8, Γ_8^*, and Γ_6 levels are shifted upward in $[U(OC_2H_5)_5]_2$ by 1190, 1525, and 2690 cm.$^{-1}$, respectively. Taking $Q/(Q + \zeta)$ as a measure of field strength, the field on the U(V) ion in $[U(OC_2H_5)_5]_2$ is about 30% stronger than the field in $UCl_5 \cdot SOCl_2$. The magnitude of this change is surprising, but it should be noted that no other data on a $5f^1$ configuration are available for comparison. The increase in field strength conforms to a greater strength for a U–O bond than for a U–Cl bond, as would be expected from a comparison of the electronegativities of oxygen and chlorine.

The effect of the field on the U(V) ion is in marked contrast to the effect of crystal field on U(IV)[22,23] where only minor shifts in the energy levels have been observed. The field effect on the U(V) ion indicates that the f orbitals extend near the region of bonding; f orbitals in the U(IV) ion are still well shielded from bond effects, and thus a sharp decrease in the spatial extension of the 5f orbitals must occur when a second f electron is added. A similar, but smaller, decrease in the spatial extension of the 4f orbitals has been noted by comparison of the field parameters for Ce^{3+} and Pr^{3+}.[24] Further investigations of the spectra of $5f^1$ ions in different fields would be of interest in defining both the field effect and the changes between $5f^1$ and $5f^2$ ions.

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(24) R. J. Elliott and K. W. H. Stevens, *Proc. Roy. Soc.* (London), **A219**, 387 (1953).

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The Synthesis and Properties of N-Substituted Cyclotrisilazanes

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N-Substituted cyclotrisilazanes are obtained when bis(alkylamino)diorganosilanes are heated in the presence of ammonium sulfate. Good yields are reported for the methylamino and the ethylamino derivatives, but lower yields are obtained when bulkier groups are substituted on nitrogen. In the series Si-substituted groups include methyl, ethyl, vinyl, phenyl, and *p*-anisyl. The ease of hydrolysis of various silicon–nitrogen compounds is compared to show the steric effects on hydrolytic stability. *cis* and *trans* isomers of hexamethyl-2,4,6-triphenylcyclotrisilazane and hexamethyl-2,4,6-tri-*p*-anisylcyclotrisilazane were separated, and the configuration of both isomers was assigned on the basis of their n.m.r. spectra. Infrared spectra are discussed.

Introduction

Although $\equiv SiNH_2$ compounds condense readily to $\equiv SiNHSi \equiv$ derivatives, $\equiv SiNHR$ compounds, where R is an alkyl or aryl group, exhibit little tendency to condense because of the marked steric effect exhibited by groups that are substituted on nitrogen. As a consequence, only a few N-substituted cyclotrisilazanes are described in the literature and little is known of their properties.

When dichlorodiorganosilanes are treated with primary amines, the products are generally disubstituted alkylamino or arylamino diorganosilanes rather than the N-alkyl or N-aryl cyclotrisilazanes that might be expected. Reported exceptions are the formation of a small quantity (3%) of hexamethyl-1,3,5-triphenylcyclotrisilazane from the reaction of dichlorodimethylsilane with aniline, and the partial condensation to bis(dimethylmethylaminosilyl)methylamine of a part of the dimethylbis(methylamino)silane obtained on treating dichlorodimethylsilane with methylamine.[1] The disubstituted compounds are usually sufficiently stable against self-condensation that they can be recovered in high yields by distillation at moderate temperatures.

Indirect methods have been described for preparing several N-substituted cyclotrisilazanes, but no general method has been reported that is useful in preparing compounds with varied structures. Andrianov prepared hexamethyl-1,3,5-triphenylcyclotrisilazane and hexamethyl-1,3,5-tripentylcyclotrisilazane from hexamethylcyclotrisilazane and the appropriate amines, but the yields were not reported.[2] An approach recently described for preparing nonamethylcyclotrisilazane is to treat the dilithium derivative of bis(dimethyl-

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(1) E. Larsson and B. Smith, *Acta Chem. Scand.*, **3**, 487 (1949).

(2) K. A. Andrianov and G. Rumba, *Zh. Obshch. Khim.*, **32**, 1993 (1962).