for carbons attached to, respectively, one and two nitrogen atoms.[11] The ^1H signal (δ_H 7.72) for the hydrogen attached to the former carbon (δ_C 128.0) showed long-range coupling (J = 1.0 Hz) to an anomeric proton (δ_H 4.89, H-1′). These data suggested that the base unit is either pyrrolo[2,3-*d*]pyrimidine (i.e., 7-deazaadenine) or pyrrolo[3,2-*d*]pyrimidine (i.e., 9-deazaadenine). The ^{13}C signals due to the aromatic carbons of **1** resemble those reported for 9-deazaadenine derivatives[12] rather than 7-deazaadenine derivatives,[13] although no pyrrolo[3,2-*d*]pyrimidine derivative has been reported from natural sources.[3] UV spectra of **1**, especially the shifts of absorption maxima in acidic solution, were also more like those of 9-deazaadenine[14] than those of tubercidin (7-deazaadenosine).[15] Accordingly, the base unit in **1** is most likely 9-deazaadenine.

The ^{13}C signals assigned to the hexose unit of **1** closely resembled those of methyl α-D-glucopyranoside,[16] suggesting that **1** is the α-D-glucopyranoside of 9-deazaadenosine. 5′-α-D-Glucopyranosides of tubercidin and toyocamycin (**3** and **4**, respectively, Scheme II) have been isolated from cyanobacteria.[17] ^1H and ^{13}C NMR data for the sugar units of **1** were very similar to those for **3** and **4**, except for the signals due to the C-1′ position. Moreover, enzymatic deglycosidation of **1** with α-D-glucosidase gave D-glucose and **2**, which was isolated as the minor component (11% of **1**) from the same cyanobacterium.

Compound **2**, $[\alpha]^{28}_D$ −28.4° (*c* 0.016, H_2O), showed a molecular ion peak at *m/z* 267.1090 ($C_{11}H_{15}N_4O_4$, M + H, Δ +0.3 mDa) by HRFABMS. FABMS/CID/MS of **2** gave the same fragment ion peaks at *m/z* 177, 163, and 147 observed for **1** (Scheme I). The ^1H NMR spectrum of **2** showed the signals ascribable to a ribose unit and two aromatic proton signals.[18]

From the results above, the structure of **2** can be assigned as 9-deazaadenosine, which has been synthesized by Lim and Klein as a cytotoxic C-nucleoside isostere of adenosine.[19] The direct comparison of **2** with a synthetic sample of 9-deazaadenosine[20] by HPLC, TLC, and UV spectra confirmed that **2** was identical to synthetic 9-deazaadenosine.[6] ^1H NMR data for natural **2** hydrochloride were also identical with those for synthetic **2** hydrochloride.[21]

Consequently, the structure of **1** was assigned as the 9-deazaadenosine 5′-α-D-glucopyranoside, as shown in Scheme I. Compounds **1** and **2** are pyrrolo[3,2-*d*]pyrimidine derivatives which have not been reported previously as biosynthetic products,[3] i.e., from natural sources. Their biosynthesis will be of considerable interest.

The IC_{50}s of **1** and **2** vs L1210 murine leukemia cells were 0.01 and 0.002 μg/mL, respectively. These compounds also showed lethal toxicity to the aquatic invertebrate *Ceriodaphnia dubia*; the LC_{50}s for acute (48 h) and chronic (7 day) toxicities were, respectively, 0.5 and 0.3 μg/mL for **1** and 0.3 and 0.1 μg/mL for **2**.

Acknowledgment. This study was supported by a grant from the National Institute of Allergy and Infectious Diseases (AI 04769, to KLR) and a subcontract from the same grant to W. W.C. We thank Dr. Robert S. Klein of Montefiore Medical Center for the synthetic sample of 9-deazaadenosine hydrochloride and for useful information and Dr. John Gilbert of Dartmouth College for the culture of *A. affinis* strain VS-1.

Supplementary Material Available: ^1H NMR, FABMS/CID/MS, and UV spectra of **1** and **2**; ^{13}C NMR, ^1H-^1H COSY, ^1H-^{13}C COSY spectra of **1**; and ^1H NMR spectra of natural and synthetic **2** hydrochloride (12 pages). Ordering information is given on any current masthead page.

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(18) ^1H NMR data (500 MHz, 18 °C) for **2** in DMSO-d_6 (2.49 ppm): δ 11.95 (1 H, br s, NH), 8.17 (1 H, s, H-2), 7.72 (2 H, br s, NH_2), 7.58 (1 H, s, H-6), 4.85 (1 H, d, J = 3.1 Hz, OH), 4.77 (1 H, d, J = 7.4 Hz, H-1′), 4.22 (1 H, dd, J = 7.4, 5.1 Hz, H-2′), 4.14 (1 H, d, J = 3.9 Hz, OH), 4.00 (1 H, dd, J = 5.1, 2.8 Hz, H-3′), 3.86 (1 H, ddd, J = 3.1, 3.1, 2.8 Hz, H-4′), 3.60 (1 H, dd, J = 12.0, 3.1 Hz, H-5′), 3.51 (1 H, dd, J = 12.0, 3.1 Hz, H-5′); assigned by single-frequency decoupling experiments.
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(20) The synthetic sample of 9-deazaadenosine hydrochloride was provided by Dr. Robert S. Klein, Montefiore Medical Center.
(21) ^1H NMR data (500 MHz, 18 °C) for **2** hydrochloride in DMSO-d_6 (2.49 ppm): δ 12.80 (1 H, s, NH), 9.03 and 8.99 (each 1 H, s, NH_2), 8.50 (1 H, s, H-2), 7.86 (1 H, d, J = 1.0 Hz, H-6), 4.86 (1 H, d, J = 7.0 Hz, H-1′), 3.97 (1 H, dd, J = 7.0, 5.1 Hz, H-3′), 3.94 (1 H, dd, J = 5.1, 3.1 Hz, H-2′), 3.87 (1 H, dt, J = 3.2, 3.1 Hz, H-4′), 3.62 (2 H, d, J = 3.2 Hz, H_2-5′).

[C_6H_6 iso-$C_3H_7^+$] and [$C_6H_7^+$ C_3H_6] Ion–Molecule Complexes: Theoretical Calculations

D. Berthomieu,*,† V. Brenner,‡ G. Ohanessian,§ J. P. Denhez,§ P. Millié,‡ and H. E. Audier§

Laboratoire de Chimie Organique Structurale CNRS URA 455, Université P. et M. Curie, 4 place Jussieu 75005 Paris, France
CEA-CE Saclay, DSM/DRECAM/SPAM 91191 GIF SUR Yvette, France
DCMR, Ecole Polytechnique, CNRS URA 1307 91128 Palaiseau Cedex, France

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While arenium ions are known to be intermediates in electrophilic aromatic substitution reactions,[1] the existence of π-complexes between an aromatic group and a cation remains elusive. In the gas phase the intermediacy of π-complexes has often been postulated in the unimolecular fragmentation of aromatic cations,[2–8] but there is as yet no irrefutable evidence for their existence. The existence of ion–molecule complexes [$C_6H_7^+$ alkene] has been proposed from experimental results.[8] In this work we have, for the first time, calculated the energy and structure of π-complexes and ion–molecule complexes involving the benzenium ion and an alkene. This kind of system is a good example of the use of molecular orbital calculations in order to calculate the energy and to study the structure of ion–neutral complexes. This has been recently reviewed.[9]

We have chosen to focus on one model: the complex intermediates presumed to be involved in the unimolecular reaction of metastable[10] protonated isopropylbenzene. It has been pre-

† Université P. et M. Curie.
‡ CEA-CE Saclay.
§ Ecole Polytechnique.
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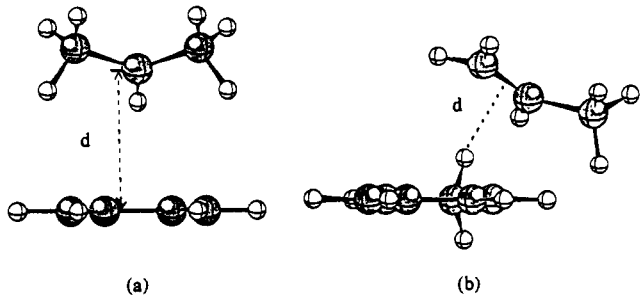
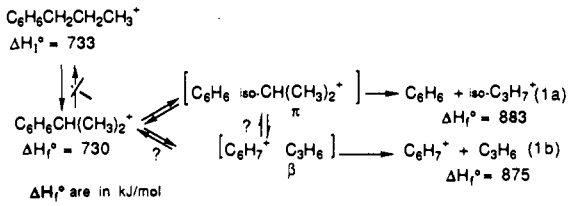


Figure 1. Structures for (a) the $[C_6H_6$ iso-$C_3H_7^+]$ complex and (b) the $[C_6H_7^+ C_3H_6]$ complex. In (a), the *d* value is 3.11 Å, with the ab initio calculation and 2.95 Å with the direct calculation. In (b), the *d* value is 2.51 Å with the ab initio calculation and 2.02 Å with the direct calculation.

viously shown[8] that protonated *n*-propylbenzene isomerizes irreversibly to protonated isopropylbenzene prior to dissociation. Both cations lead to $C_6H_7^+$ (87%) and iso-$C_3H_7^+$ (13%) product ions (eq 1). Dissociations are preceded by an incomplete but specific



H-exchange between the primary and aromatic hydrogen atoms. These data can be explained only if a $[C_6H_7^+ C_3H_6]$ complex, hereafter called β, is postulated (eq 1b). The $[C_6H_6$ iso-$C_3H_7^+]$ complex, hereafter called π, may be the precursor of iso-$C_3H_7^+$ (eq 1a). In this model the H-exchange can occur by reversible isomerization between protonated isopropylbenzene and the β-complex, between π- and β-complexes, or both.

First, ab initio calculations[11] were carried out at the 6-31G**//3-21G level and led to the existence of local minima corresponding to both β- and π-complexes. All optimized structures were characterized as true minima through vibrational energy analysis at the 3-21G level. After correction for zero-point vibrational energy, the well depths are 39 kJ/mol for the π-complex and 13 kJ/mol for the β-complex, relative to final products (values before correction are 46 and 20 kJ/mol, respectively).

However, ab initio calculations are intractable for a complete exploration of the potential energy surface (PES) and remain approximate since electron correlation calculations are not feasible at present. In such complexes, the single most important contribution to electron correlation is the dispersion energy. We thus performed direct calculations of the interaction energy calculated as the sum of terms: electrostatic, polarization, dispersion, short-range repulsion, and exchange dispersion energies, according to the method developed by Claverie.[12-14] We then determinated the most significant minima on the PES on the basis of a simulated annealing method.[15-17]

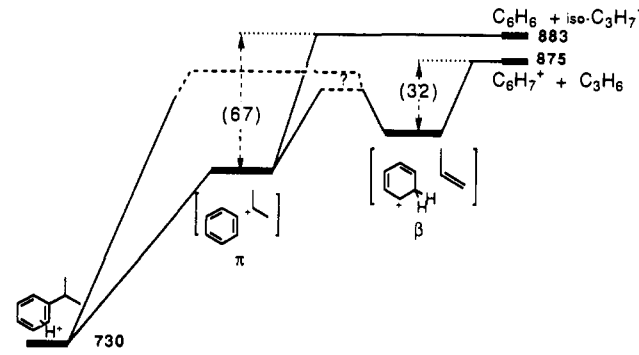


Figure 2. Potential energy surface for the unimolecular decomposition of metastable protonated isopropylbenzene. Bold characters correspond to $\Delta H_f°$ in kJ/mol.[18] The numbers in parentheses correspond to stabilization energies calculated by the semiempirical method.

From the simulated annealing calculations, only one stable form has been found for the $[C_6H_6$ iso-$C_3H_7^+]$ complex. This structure is almost the same as the ab initio optimized structure. However, due to dispersion forces, the structure is a little more compact. The interaction energy is 67 kJ/mol, and the structure of this complex corresponds to a π-complex. In fact, the isopropyl cation is centered on the benzene, with its three carbon atoms inclined with respect to the plane of the six carbon atoms of the benzene (Figure 1). The dihedral angle between these two planes is 40°. The distances between the center of gravity of the benzene and the secondary carbon are 2.95 and 2.39 Å, respectively, for the hydrogen atom linked to this carbon. The calculations for the β-complex $[C_6H_7^+ C_3H_6]$ show many minima on the PES. For the most stable β-complex the interaction energy is 32 kJ/mol (Figure 1). As in the case of the π-complexes, the geometry is very similar to ab initio values with shorter distances between the two components. One hydrogen bonded to an sp^3 carbon is in a favorable position for further transfer to the propene molecule (d = 2.02 Å, Figure 1). This distance is a little smaller than with the ab initio calculation. The important number of local minima for the β-complexes obtained from the simulated annealing study corresponds to the interaction of the propene with almost every hydrogen atom of the protonated benzene. There is no minimum with the propene molecule above the protonated benzene. For all these complexes, intermolecular distances smaller than 2.3 Å are observed between the two components.

From these calculations we can now consider the potential energy profile of the fragmentation reactions (Figure 2). The stabilization energy of the π-complex is 67 kJ/mol, and it is 32 kJ/mol for the β-complex, both relative to final products.[18] The π-complex lies below the most stable final state, corresponding to formation of $C_6H_7^+$. This result leads us to conclude that the intermediacy of π- and β-complexes is certainly a pathway leading to H-exchange and therefore to the formation of iso-$C_3H_7^+$ and $C_6H_7^+$ from low-energy protonated isopropyl benzene ions.

Metastable protonated *tert*-butylbenzene yields *tert*-$C_4H_9^+$ without H-exchange. Simulated annealing calculations show that π- and β-complexes correspond to stable structures. However, the β-complex lies higher in energy than the final state (*tert*-$C_4H_9^+$

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(17) The electronic distribution obtained by the SCF calculation of each molecule or ion is superseded by a monopole, dipole, and quadrupole centered on each atom and at the middle of each bond so that the true SCF electrostatic potential is correctly fitted. The polarization energy is calculated by using experimental atomic and bond polarizabilities. The repulsion–dispersion energy is calculated as the sum of atom–atom repulsion–dispersion energy terms. Thus, the calculation of the interaction energy and its derivatives is achieved by using analytical formulae. In this way, simulated annealing and quasi-Newtonian local minimization calculations are easily tractable, even for such complex systems.

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plus benzene), which explains why H-exchange does not occur. The interaction energy for the more stable π-complex structure is 56 kJ/mol.

The stability of π-complexes is a general phenomenon. In a further publication we will give other theoretical and experimental evidences which confirm their existence in related cases.

Crystal Structure and Reactivity of a Pentacoordinate 1,2-Oxastannetanide: An Intermediate of the Tin-Peterson Reaction

Takayuki Kawashima,* Naoshi Iwama, and Renji Okazaki*

Department of Chemistry, Faculty of Science
The University of Tokyo
7-3-1 Hongo, Bunkyo-ku, Tokyo 113, Japan
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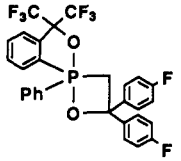
The Peterson reaction has been widely utilized for olefin synthesis as a silicon analog of the Wittig or Horner-Emmons reaction, providing the method for selective synthesis of (*E*)- or (*Z*)-isomer from a single diastereomer of (β-hydroxyalkyl)silanes.[1] The reactions using homologs such as β-hydroxy germanes, stannanes, and plumbanes are well-known to give the corresponding olefins under acidic and neutral (or basic) conditions.[1e,2] Very recently, we succeeded in the synthesis of pentacoordinate 1,2-oxaphosphetane **1**[3] and 1,2-oxasiletanide **2**[4] bearing the Martin ligand, intermediates of the Wittig and the Peterson reactions, respectively.



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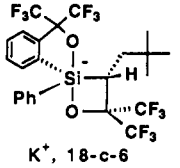

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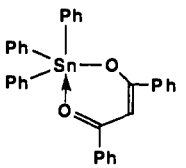

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We now report the first synthesis, crystal structure, and reactivities of a 1,2-oxastannetanide, an intermediate of the tin-Peterson reaction.

Sequential treatment of [(phenylthio)methyl]triphenylstannane (**3**)[5] with 3 equiv of lithium diisopropylamide (LDA) (THF, -20 °C, 2 h), 5 equiv of HMPA, excess hexafluoroacetone (THF, -78 °C, 15 min), and aqueous NH_4Cl gave the corresponding β-hydroxy stannane **4a** (23%) with recovery of **3** (50%) (Scheme I).[6]

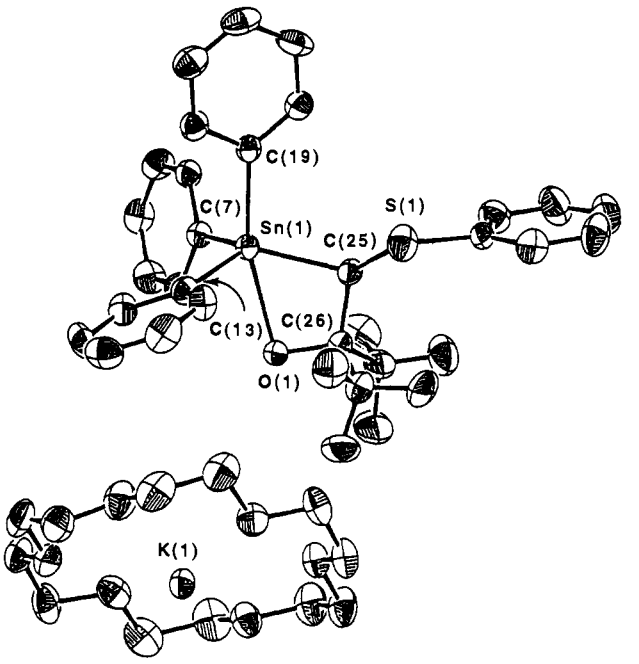
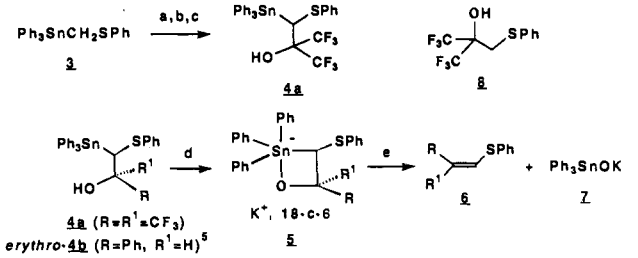


Figure 1. ORTEP drawing of **5a** (omitting CH_2Cl_2 and H_2O). Selected bond lengths (Å) and bond angles (deg): Sn(1)-O(1), 2.401(5); Sn(1)-C(7), 2.140(7); Sn(1)-C(13), 2.136(8); Sn(1)-C(19), 2.188(8); Sn(1)-C(25), 2.200(7); O(1)-C(26), 1.370(8); C(25)-C(26), 1.55(1); O(1)-Sn(1)-C(19), 165.1(2); C(7)-Sn(1)-C(13), 111.1(3); C(7)-Sn(1)-C(25), 113.5(3); C(13)-Sn(1)-C(25), 123.1(3); O(1)-Sn(1)-C(25), 61.3(2); O(1)-C(26)-C(25), 107.2(6); Sn(1)-O(1)-C(26), 93.2(4); Sn(1)-C(25)-C(26), 96.5(5).

Scheme I[a]



[a] (a) 3 equiv of LDA, THF, -20 °C, 2 h; (b) $(CF_3)_2C{=}O$, THF, -78 °C, 15 min; (c) aqueous NH_4Cl, -78 °C; (d) KH, 18-crown-6, THF, room temperature (**4a**) or -30 °C (**4b**); (e) 70 °C, CH_3CN, 36 h (**5a**) or 50 °C, THF, 5 h (**5b**).

Deprotonation of **4a** with KH in the presence of 18-crown-6 in THF was monitored by ^{19}F and ^{119}Sn NMR spectroscopy to show that 1,2-oxastannetanide **5a** was formed quantitatively as evidenced by the appearance of a double quartet with centers of δ_F -75.09 ($^4J_{FF}$ = 8.8 Hz) and -71.77 ($^4J_{FF}$ = 8.8 Hz) and a singlet (δ_{Sn} -229.65), respectively. The large upfield shift (105 ppm) in δ_{Sn} from **4a** (-125.09) to **5a** (-229.65) strongly supports the structure of a pentacoordinate tin ate complex.[7] The ^1H and ^{13}C NMR spectra showed only one set of signals for Sn-Ph, indicating the presence of very fast pseudorotation. It is surprising that a stable pentacoordinate 1,2-oxastannetanide can be obtained even without resort to the Martin ligand.

It was found by ^{19}F NMR spectroscopy that **5a** provided olefin **6a**[8] (95%) and probably potassium triphenylstannoxide (**7**) upon heating (70 °C, CH_3CN, 36 h), indicating that **5a** has a reactivity similar to that of silicon analog **2**.[4] In contrast to **2**, **5a** was

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(6) **4a**: colorless viscous oil; HRMS (70 eV) *m/z* calcd for $C_{28}H_{22}F_6OS^{120}Sn$ 640.0317, found 640.0312; ^1H NMR ($CDCl_3$) δ 3.75 (s, 1 H, $^2J_{HSn}$ = 67 Hz, C*H*SPh), 4.36 (s, 1 H, O*H*), 7.16-7.20 (m, 4 H), 7.33-7.41 (m, 10 H), 7.55-7.67 (m, 6 H); ^{13}C NMR ($CDCl_3$) δ 37.83 (s, $^1J_{CSn}$ = 290 Hz, Sn*C*H), 77.21 [sept, $^2J_{CF}$ = 30 Hz, *C*(CF_3)$_2$], 122.83 [q, $^1J_{CF}$ = 287 Hz, C(*C*F_3)(C'F_3)], 123.18 [q, $^1J_{CF}$ = 288 Hz, C(CF_3)(*C*'F_3)], 127.50, 128.60 ($^3J_{CSn}$ = 54 Hz, *m*-C of SnPh), 129.18, 129.39 ($^4J_{CSn}$ = 12 Hz, *p*-C of SnPh), 129.86, 136.71 ($^3J_{CSn}$ = 22 Hz, *ipso*-C of SPh), 137.17 ($^2J_{CSn}$ = 38 Hz, *o*-C of SnPh), 137.67 ($^1J_{CSn}$ = 556 Hz, *ipso*-C of SnPh); the coupling constants (J_{HSn} and J_{CSn}) for **4a** and **5b**[11] were obtained from the satellite peaks; ^{19}F NMR ($CDCl_3$) δ -74.84 (q, $^4J_{FF}$ = 9.0 Hz, 3 F), -73.78 (q, $^4J_{FF}$ = 9.0 Hz, 3 F); ^{119}Sn NMR (THF) δ -125.09 (m).

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