geometry and a metal-metal bond, d(Ir-Ir) = 2.7861 (6) Å. Complex 3 has one unpaired electron, which is introduced by the addition of a neutral 'AlEt2 radical to the diamagnetic complex 1. At -150 °C the EPR powder spectrum of 3 exhibits a completely isotropic signal with g = 2.005. The isotropic nature of the low-temperature powder spectrum suggests that the unpaired electron 3 resides in a molecular orbital with essentially no contribution from the iridium atoms. This result is in accord with FTIR  $\nu$ (CN) data which suggest no change in the formal oxidation state of 3 compared to 1 and indicates the unpaired electron likely is delocalized exclusively within the  $C_2N_2Al$  ring of 3.

The formation of 3 by annulation of two  $\mu$ -isocyanides with an AlEt2 radical is unprecedented, eq 1. The 'AlEt2 radical is



presumably formed by AlEt<sub>3</sub> abstraction of C<sub>2</sub>H<sub>5</sub><sup>-</sup> from an Ncomplexed  $AlEt_3$  followed by electron transfer. The complexation of 1 equiv of AlEt<sub>1</sub> to 1 is observed in the early stages of reaction (vide supra). Upon abstraction of  $C_2H_5^-$  from the initial adduct, one would expect formation of the species  $[Ir_2[C_2(NR)_2AlEt_2]$ -(CNR)<sub>2</sub>(dmpm)<sub>2</sub>]<sup>+</sup>[AlEt<sub>4</sub>]<sup>-</sup>, [3<sup>+</sup>][AlEt<sub>4</sub><sup>-</sup>]. We note Schmidbaur has reported an apparently similar disproportionation of Al<sub>2</sub>Me<sub>6</sub> to  $[AlMe_2^+]/[AlMe_4^-]$  and annulation of  $[AlMe_2^+]$  in the case of bis(trialkylphosphoranylimino)silanes.<sup>31</sup> We find the molecular cation  $3^+$  can be prepared by one-electron oxidation of 3. Cyclic voltammetric studies of 3 in THF reveal one reversible oxidation,  $E_{1/2}(3^+/3) = -0.22$  V versus SCE. Chemical oxidation of 3 with  $[FeCp_2][PF_6]$  affords  $[3^+][PF_6^-]$ . The cationic species  $3^+$  does not show any  $\nu(CN)$  band in the 1700–1450-cm<sup>-1</sup> region, suggesting that the carbon-carbon bond in the  $C_2N_2Al$  ring of 3 is not cleaved by one-electron oxidation. The diamagnetic cation  $3^+$  is however reducible by AlEt<sub>4</sub><sup>-</sup> to yield the isolated radical product 3. Our results thus imply that it is the condensation of an AlEt<sub>2</sub><sup>+</sup> fragment with two bridging isocyanide ligands which induces carbon-carbon bond formation, not the injection of an electron from AlEt<sub>4</sub><sup>-</sup>, formed during the coupling reaction. Our studies of the relative importance of the Lewis acid employed in ligand coupling versus electron transfer are continuing.

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Supplementary Material Available: Tables consisting of crystal data and data collection parameters for 1 and 3 (Table I), positional parameters for 1 and 3 (Tables II and VII, respectively), temperature factor expressions for 1 and 3 (Tables III and VIII, respectively), bond distances and angles for 1 and 3 (Tables IV and IX, respectively), least-squares planes and dihedral angles for 1 and 3 (Tables V and X, respectively) (30 pages); tables consisting of observed and calculated structure factors for 1 and 3 (Tables VI and XI, respectively) (51 pages). Ordering information is given on any current masthead page.

## Total Synthesis of (+)-CC-1065 and ent-(-)-CC-1065

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CC-1065 (1, NSC-298223), an antitumor-antibiotic isolated from Streptomyces zelensis<sup>2</sup> initially identified by spectroscopic techniques<sup>3a</sup> and confirmed in a single-crystal X-ray structure determination,<sup>3b</sup> has been shown to possess exceptional, potent in vitro cytotoxic activity, antimicrobial activity, and confirmed, potent in vivo antitumor activity.<sup>4</sup> In sharp contrast to the early observations made with simplified analogues of CC-1065 bearing modified central and right-hand subunits, e.g., U-71,184,5 in which the antitumor activity and DNA binding properties have been found to be restricted primarily to the agent enantiomer bearing the natural 3bR,4aS-CPI left-hand segment,6 recent efforts have

Scheme I<sup>4</sup>



<sup>a</sup> (a) 1.10 equiv of 1-piperidino-1-propene, CH<sub>2</sub>Cl<sub>2</sub>, 0-23 °C, 12 h; (b) 10% aqueous HCl-THF (1:5), 23 °C, 12 h; (c) 1.0 equiv of *N*-bromosuccinimide, THF, H<sub>2</sub>SO<sub>4</sub> (catalyst), -23 °C, 1 h, 97%; (d) 1.1 equiv of NaH, DMF, 23 °C, 15 min; 3 equiv of 3-bromopropyne, DMF 23 °C, 12 °C, 12 °C, 14 °C, 14 °C, 15 °C, 14 °C, 16 °C, DMF, 23 °C, 3 h, 67% from 7; (e) 2.1 equiv of n-Bu<sub>3</sub>SnH, AIBN (catalyst), benzene, 80 °C, 4-5 h; (f) 2-3 equiv of BH<sub>3</sub>·SMe<sub>2</sub>, THF, 0-23 °C, 1-3 h; 1 equiv of 2 N aqueous NaOH, 3 equiv of 30% H<sub>2</sub>O<sub>2</sub>, 45 °C, 30 min, 40% from 9; (g) 5% anhydrous HCl-CH<sub>3</sub>OH, 50 °C, 2 h, 83%; (h) 1 atm of H<sub>2</sub>, 10% Pd/C, EtOAc, 23 °C, 20 h, 85%; (i) 1.5 equiv of Ph<sub>3</sub>P, 1.95 equiv of diethyl azodicarboxylate, THF, 23 °C, 3 h, 50%.

(1) (a) National Institutes of Health research career development award recipient, 1983-1988 (CA 01134). Alfred P. Sloan research fellow, 1985-1989. (b) National Institutes of Health predoctoral trainee, 1984-1985 (GM 07775). David Ross Fellow, Purdue University, 1986-1987.

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<sup>(27)</sup> The Al-N bond distances of 1.939 (9) Å and 1.896 (9) Å lie at the extrema for known Al-N distances, 1.937 (5)-1.902 (4) Å.<sup>28</sup>
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revealed that the enantiomeric pairs (+)-CC-1065/ent-(-)-CC-1065<sup>7</sup> and (+)-CPI-CDPI<sub>2</sub>/(-)-CPI-CDPI<sub>2</sub><sup>8</sup> possess indistinguishable in vitro cytotoxic activity. Herein we detail the preparation of 3, the spirobicyclo[5.2.0]octa-2,5-dien-4-one bearing left-hand segment of CC-1065 (CPI),9 the coupling of its resolved, immediate precursors (-)-1S-15 and (+)-1R-15 to synthetic PDE-I dimer (5),<sup>10</sup> and incorporation into the total syntheses of natural (+)-CC-1065 (1) and unnatural ent-(-)-CC-1065 (2), respectively.

Treatment of the selectively activated 2-benzyloxy-pquinonediimide  $6^{11}$  with 1-piperidino-1-propene (CH<sub>2</sub>Cl<sub>2</sub>, 0-23 C, 2-12 h) followed by acid-catalyzed elimination of piperidine (10% aqueous HCl/THF 1:5, 23 °C, 12 h)<sup>12</sup> afforded 3methylindole 7. Selective C-4 bromination of 7 (1.0 equiv NBS, THF, catalytic  $H_2SO_4$ , -23 °C, 1 h) afforded 8 (97%), and subsequent benzenesulfonamide alkylation (NaH, DMF; 3bromopropyne, 23 °C, 3 h) provided 9 (67% overall from 7). 5-Exo-Dig aryl radical-alkyne cyclization<sup>13</sup> (2.1 equiv n-Bu<sub>3</sub>SnH, catalytic AIBN, benzene, 80 °C, 4-5 h) afforded 3methylideneindoline 10, which proved unstable to chromatographic

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purification and was subjected directly to the conditions of hydroboration (2-3 equiv of BH<sub>3</sub>·SMe<sub>2</sub>, THF, 0-23 °C, 1-3 h) and subsequent oxidation (2 N NaOH, 30% aqueous H<sub>2</sub>O<sub>21</sub> 45 °C, 30 min) to provide 3-hydroxymethylindoline 11 (40% overall from 9). Indole deprotection (5% anhydrous HCl-CH<sub>3</sub>OH, 50 °C, 2 h) afforded 12, and hydrogenolysis of the benzyl ether (1 atm of H<sub>2</sub>, 10% Pd/C, EtOAc, 23 °C, 20 h) provided 13. Final closure of 13 directly to the spirocyclopropylquinone was achieved by employing the Mitsunobu activation and intramolecular alkylation conditions detailed by Magnus and co-workers (Ph<sub>3</sub>P, diethyl azodicarboxylate, THF, 23 °C, 3 h),<sup>9b</sup> affording N<sup>2</sup>-benzenesulfonyl CPI (3),% and completing the preparation of the protected left-hand segment of CC-1065.

The resolution of 4<sup>8</sup> and its subsequent incorporation into the total synthesis of (+)- and (-)-CC-1065 is detailed in Scheme II. Reductive removal of the indoline N-benzenesulfonamide group of 12 (6 equiv of sodium bis(2-methoxyethoxy)aluminum hydride, toluene, 100 °C, 3 h)<sup>7,14</sup> followed by acylation of the unstable free indoline (2 equiv of di-tert-butyldicarbonate, THF, 23 °C, 3 h) afforded 4 (60% from 12). Resolution of alcohol 4 was achieved by chromatographic separation of the corresponding diastereomeric (R)-(-)-O-acetylmandelate esters (14),<sup>8</sup> providing 1R,2'R-14 and 1S,2'R-14, which were independently subjected to base-promoted hydrolysis (3 equiv of LiOH, THF/ CH<sub>3</sub>OH/H<sub>2</sub>O 3:2:1, 20 °C, 3 h) to provide the enantiomeric alcohols (+)-1*R*-4 ( $[\alpha]_{578}^{23}$  = +8.1° (*c* 0.63, CH<sub>2</sub>Cl<sub>2</sub>)) and (-)-1*S*-4 ( $\alpha]_{578}^{23}$  = -8.3° (*c* 0.74, CH<sub>2</sub>Cl<sub>2</sub>)).<sup>15</sup> Independent conversion of (-)-1S-4 and (+)-1R-4 to the corresponding primary chlorides (1.5 equiv of Ph<sub>3</sub>P, 1.5 equiv of CCl<sub>4</sub>, CH<sub>2</sub>Cl<sub>2</sub>, 23 °C, 12 h)<sup>16</sup> afforded (-)-1*S*-15 ( $[\alpha]_{578}^{23} = -12.8^{\circ}$  (*c* 0.51, CH<sub>2</sub>Cl<sub>2</sub>)) and (+)-1*R*-15 ([ $\alpha$ ]23<sub>578</sub> = +13.1° (*c* 0.66, CH<sub>2</sub>Cl<sub>2</sub>)),<sup>15</sup> respectively, constituting the resolved substrates for incorporation into the total synthesis of (+)- and (-)-CC-1065.<sup>7</sup> Following the conditions described by Kelly and co-workers,7 sequential removal of the

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<sup>(15)</sup> HPLC analysis of the separated diastereomers showed the faster eluting diastereomer (1*R*,2'*R*·14) was ≥99% pure, and the slower eluting diastereomer (1*S*,2'*R*·14) was 97–98% diastereomerically pure. (16) Appel, R. Angew. Chem., Int. Ed. Engl. 1975, 14, 801.

benzyl ether (HCO<sub>2</sub>NH<sub>4</sub>, THF/H<sub>2</sub>O 4:1, 10% Pd/C, 23 °C, 30 min, 100%) and the *t*-BOC protecting groups (3 N anhydrous HCl-EtOAc, 23 °C, 30 min) of (-)-1*S*-15 and (+)-1*R*-15 afforded the unstable indoline hydrochlorides 16, which were coupled directly with synthetic PDE-I dimer (5)<sup>10</sup> in the presence of EDCI (3 equiv, DMF, NaHCO<sub>3</sub>, 23 °C, 24 h) to provide 1*S*-17 and 1*R*-17, respectively. Final spirocyclization (Wierenga-Kelly Winstein Ar-3' alkylation)<sup>7</sup> was effected by treatment of 1*S*-17 and 1*R*-17 with 1:1:1 Et<sub>3</sub>N/H<sub>2</sub>O/CH<sub>3</sub>CN (23 °C, 30 min) and afforded (+)-CC-1065 (1,  $[\alpha]_{578}^{23} = +90^{\circ}$  (*c* 0.062, DMF)) and *ent*-(-)-CC-1065 (2,  $[\alpha]_{578}^{23} = -92^{\circ}$  (*c* 0.075, DMF)),<sup>15</sup> respectively.<sup>17</sup>

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Upjohn Company for providing a comparison sample of naturally occurring (+)-CC-1065, for preprints of their work (ref 5 and 7), and for many helpful discussions.

Supplementary Material Available: Full physical and spectral characterizations of 3–17, 1, and 2 are provided (8 pages). Ordering information is given on any current masthead page.

(17) Synthetic CC-1065 prepared in this manner was shown to be identical by SiO<sub>2</sub> TLC (20% DMF-toluene),  $[\alpha]_{278}^{27}$ , <sup>1</sup>H NMR (DMSO- $d_6$ , 300 MHz), IR (KBr), and FABMS with an authentic sample of natural (+)-CC-1065. The  $[\alpha]_{25}^{27}$  for natural (+)-, semisynthetic (+)-, and semisynthetic ent-(-)-CC-1065 have been reported to be +97°, +98°, and -96° (c 0.2, DMF), respectively. In the present investigation,  $[\alpha]_{378}^{23}$  for natural (+)-CC-1065 was determined to be +93° (c 0.067, DMF). In the present investigation, the in vitro cytotoxic activity of natural (+)-, synthetic (+)-, and synthetic ent-(-)-CC-1065, respectively, were determined to be the following: 1.1, 1.2, and 1.3 × 10<sup>-5</sup> µg/mL (L1210); 1.4, 1.8, 1.3 × 10<sup>-5</sup> µg/mL (B16).

## Additions and Corrections

Remarkable Effects of Lone Pair-Lone Pair Interactions on the Extremely Stereoselective [2 + 2] Cycloaddition of Azidoketene to Chiral 3-Imino- $\beta$ -lactams [J. Am. Chem. Soc. 1987, 109, 1798]. IWAO OJIMA,\* KAZUAKI NAKAHASHI, STEPHAN M. BRAND-STADTER, and NAOTO HATANAKA

Page 1801: In Scheme V, the compound designations for IIA and IIB should be reversed.

## Book Reviews\*

Advances in Heterocyclic Chemistry. Volume 41. Edited by A. R. Katritzky. Academic Press: Orlando. 1987. vii + 376 pp. \$85.00. ISBN 0-12-020641-2

The six chapters in this volume treat subjects new to the series. Synthesis of Heterocycles from Hydrogen Cyanide Derivatives, by D. S. Donald and O. W. Webster, deals with the elaborate chemistry built upon the drimers and oligomers of HCN, important industrially and in the search for insight into the paleological origin of organic compounds. T. S. Gilchrist reviews the anions of 5-membered heteroaromatic rings with attention to ring-opening reactions. F. Taddei and colleagues present a critical discussion of the barriers to free rotation in the C-acyl and N-acyl heterocycles. The Basicity and Acidity of Azoles is the subject of a chapter by J. Catalan, J. L. M. Abbond, and J. Elguero. Oxidative Transformations of Heteroaromatic Iminium Salts, exemplified by pyridinium salts, have been the subject of much recent attention and are reviewed by H. Weber. A ring system not reviewed before, the pyrazolopyrimidines, is the subject of the final chapter, by M. H. El Nagadi, M. R. H. El Moghayar, and G. E. H. El Gemeie. The high quality of content and production that characterizes this series is maintained.

High-Energy Processes in Organometallic Chemistry. ACS Symposium Series. No. 333. Edited by Kenneth S. Suslick (University of Illinois at Urbana—Champaign). American Chemical Society: Washington, DC. 1987. vii + 336 pp. \$69.95. ISBN 0-8412-1018-7

This book, which was developed from a symposium sponsored by the division of inorganic chemistry at the 192nd meeting of the American chemical society, Anaheim, Ca, is composed of 19 chapters and author and subject indexes. It is written by academic and industrial researchers who are currently very active in high-energy processes in organometallic chemistry. The first chapter gives a historical background to the development of high-energy processes, which started as early as 1929, by F. A. Paneth. Some of the chapters discuss different aspects of high-energy processes, from current studies in the gas phase of the organometallic chemistry to the study of photochemical reactions and the investigation of some highly reactive intermediates and metal powders. The remaining chapters discuss some techniques and applications in high-energy processes involving infrared spectroscopy for examining the structure and behavior of intermediates involved in organometallic chemistry, electron spin resonance spectroscopy in the study of radiolysis of transition-metal compounds, ultrasonic waves for heterogeneous reactions, electrochemiluminescence of organometallics and finally plasma, and ion and electron beams for synthesis and modification of inorganic systems.

Sultan T. Abu-Orabi, Yarmouk University, Jordan

Formulas, Facts and Constants, For Students and Professionals in Engineering, Chemistry and Physics. Second Editlon. By Harold J. Fischbeck (University of Oklahoma) and Kurt H. Fischbeck (University of Heidelberg). Springer-Verlag: Berlin, Heidelberg, and New York. 1987. xv + 260 pp. \$23.00. ISBN 3-540-17610-1

Although the title identifies chemists as one of the groups for whom this book is intended, only those whose work is very close to engineering will find it useful. The first section (108 pages) is essentially mathematical and includes trigonometric functions, vectors, etc. The second part is headed "Units, conversion factors, and constants" and is arranged according to kind of property: mass, pressure, energy, etc. This is a useful arrangement. Section 3, "Spectroscopy and atomic structure", considers only the spectroscopy of atoms and totally ignores molecular spectroscopy. The fourth section is on wave mechanics and includes a range of equations. The last section is called "Facts, figures, and data useful in the laboratory". It is concerned with vacuum phenomena, flow, electronic properties, and radiation (especially  $\gamma$ ), for the most part. Some physical-chemistry laboratories would find it useful; those who

<sup>\*</sup>Unsigned book reviews are by the Book Review Editor.