

Figure 2. Oscillating light absorption at 450 nm (a), oscillating photoluminescence emission at 610 nm (b), and oscillating chemiluminescence emission at 610 nm (c) in the $\text{Ru}(\text{bpy})_3^{2+}$ -catalyzed BZ reaction. Experimental conditions are as in Figure 1.

3) of $\text{Ru}(\text{bpy})_3^{2+}$ can be obtained. Actually, when both reactions 2 and 3 are thermodynamically allowed, reaction 3 predominates for kinetic reasons,²⁴ and since the quantum yield of $^*\text{Ru}(\text{bpy})_3^{2+}$ emission is high,²⁴ luminescence can be easily observed. Rubinstein and Bard²⁵ have recently shown that chemiluminescence can be obtained when organic acids are oxidized by $\text{Ru}(\text{bpy})_3^{3+}$ or in the presence of $\text{Ru}(\text{bpy})_3^{2+}$. The reaction mechanism involves reduction of $\text{Ru}(\text{bpy})_3^{3+}$ by strongly reducing radicals generated in the one-electron oxidation of the organic acid. We have observed that chemiluminescence is also obtained on mixing aqueous solutions of $\text{Ru}(\text{bpy})_3^{3+}$ and malonic acid in 1 M H_2SO_4 . Since the $\text{Ru}(\text{bpy})_3^{3+}$ concentration oscillates^{8,15} in the BZ reaction and since a large concentration of malonic acid is also present, we thought that the oscillating reaction has to be accompanied by an oscillating chemiluminescence emission. When an aqueous solution containing 0.25 M malonic acid, 0.06 M KBrO_3 , 1 M H_2SO_4 , and 1.0×10^{-4} M $\text{Ru}(\text{bpy})_3^{2+}$ was examined for chemiluminescence in a fluorimeter, an oscillating signal was indeed recorded, although the light emission was too weak to be observed by eye.

Subsequent systematic investigations showed that (i) the chemiluminescence spectrum is identical (except for oscillations) with the photoluminescence spectrum of $\text{Ru}(\text{bpy})_3^{2+}$ (Figure 1), (ii) the intensity and period of the oscillating chemiluminescence depend on the reactant concentrations and decrease with time, and (iii) the oscillating chemiluminescence has the same period but is out of phase compared with the oscillating light absorption at 450 nm (where the extinction coefficient of $\text{Ru}(\text{bpy})_3^{2+}$ is much higher than that of $\text{Ru}(\text{bpy})_3^{3+}$) and with the oscillating photoluminescence emission (which is due to $\text{Ru}(\text{bpy})_3^{2+}$ absorption) (Figure 2).

The shapes of the oscillating curves shown in Figure 2 merit some comments. The shape of the light absorption oscillation (Figure 2a) shows that the interconversion of $\text{Ru}(\text{bpy})_3^{2+}$ and $\text{Ru}(\text{bpy})_3^{3+}$ is due to a smooth reaction. By contrast, the pho-

toluminescence oscillation (Figure 2b) shows a shoulder and then a sharp peak before decreasing. These features cannot be due to concomitant variations in the $\text{Ru}(\text{bpy})_3^{2+}$ concentration because in such a case they should also appear in the light absorption oscillation curve of Figure 2a. In the same way, we can note that the chemiluminescence curve (Figure 2c) exhibits an oscillatory behavior that does not exactly reflect the changes in the $\text{Ru}(\text{bpy})_3^{3+}$ concentrations shown by Figure 2a. We believe that the peculiar features of the photoluminescence and chemiluminescence curves are due to the oscillating formation of radical species which may act as quenchers for $^*\text{Ru}(\text{bpy})_3^{2+}$ and as reactants for the $\text{Ru}(\text{bpy})_3^{3+}$ chemiluminescent reaction. A more thorough investigation of this system might reveal new details of the mechanism of the BZ reaction.

Other experiments on this artificial "firefly" system are in progress in our laboratory.

Registry No. $\text{Ru}(\text{bpy})_3^{2+}$, 15158-62-0; BrO_3^- , 15541-45-4; malonic acid, 141-82-2.

Chiral Total Synthesis of Compactin

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Compactin^{1a} (from *Penicillium brevicompactum*) [or ML-236B^{1b} (from *P. citrinum*), **1a**], monacolin K^{2a} (or mevinolin,^{2b} **1b**), and dihydro compounds **2a** and **2b**,³ which are potent inhibitors of 3-hydroxy-3-methylglutaryl coenzyme A (HMG-CoA) reductase, have attracted considerable interest because of their high hypocholesterolemic activity.⁴ This important family of polyketide-derived compounds possesses a highly functionalized hexahydronaphthalene (or *trans*-octalin) skeleton substituted with a β -hydroxy δ -lactone moiety.

In this communication, we report an enantioselective and convergent synthesis of **1a**,⁵ which is also adaptable to the synthesis of the other related compounds **1b**, **2a**, and **2b**. Our synthetic strategy outlined in Scheme I encompasses several interesting synthetic facets. (1) The intramolecular Diels-Alder reaction of **4** via *exo* orientation was considered to be a viable approach for the construction of the *trans*-octalone system **3**, since decal-1,7,9-trien-3-one cyclized exclusively to *trans*-octalone.⁶ (2) The concomitant asymmetric induction of four asymmetric centers, C8, C9, C14, and C17 in **3**, might be realized in this [4 + 2]

(1) (a) Brown, A. G.; Smale, T. C.; King, T. J.; Hasenkamp, R.; Thompson, R. H. *J. Chem. Soc., Perkin Trans. 1* **1976**, 1165. (b) Compactin and ML-236B are identical: Endo, A.; Kuroda, M.; Tsujita, Y. *J. Antibiot.* **1976**, *29*, 1346. Endo, A.; Kuroda, M.; Tanzawa, K. *FEBS Lett.* **1976**, *72*, 323.

(2) (a) Endo, A. *J. Antibiot.* **1979**, *32*, 852. (b) Alberts, A. W.; Chen, J.; Kuron, G.; Hunt, V.; Huff, J.; Hoffman, C.; Rothrock, J.; Lopez, M.; Joshua, H.; Harris, E.; Patchett, A.; Monaghan, R.; Currie, S.; Stapley, S.; A.-Schönberg, G.; Hensens, O.; Hirshfield, J.; Hoogsteen, K.; Liesch, J.; Springer, J. *Proc. Natl. Acad. Sci. U.S.A.* **1980**, *77*, 3957.

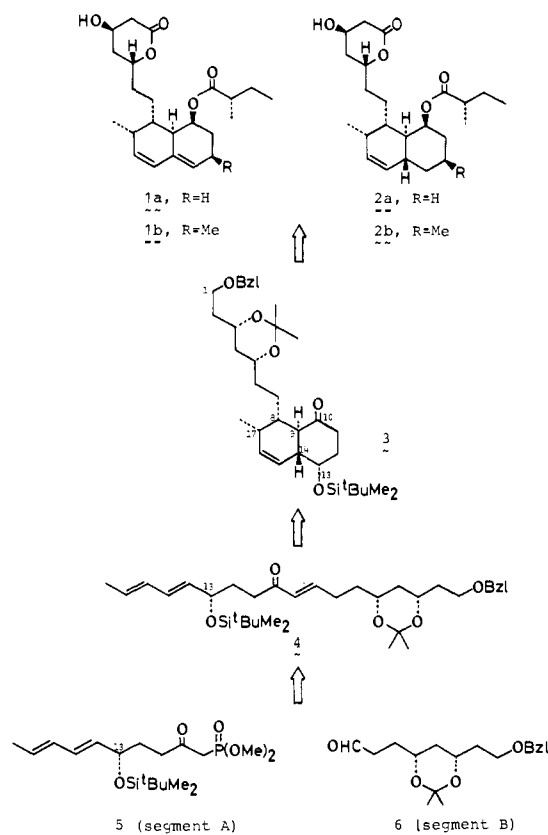
(3) Lam, Y. K. T.; Gullo, V. P.; Goegelman, R. T.; Jorn, D.; Huang, L.; DeRiso, C.; Monaghan, R. L.; Putter, I. *J. Antibiot.* **1981**, *34*, 614. A.-Schönberg, G.; Joshua, H.; Lopez, M. B.; Hensens, O. D.; Springer, J. P.; Chen, J.; Ostrove, S.; Hoffman, C. H.; Alberts, A. W.; Patchett, A. A. *Ibid.* **1981**, *34*, 507.

(4) Tsujita, Y.; Kuroda, M.; Tanzawa, K.; Kitano, N.; Endo, A. *Atherosclerosis* **1979**, *32*, 307. Kuroda, M.; Tsujita, Y.; Tanzawa, K.; Endo, A. *Lipids* **1979**, *14*, 585. Yamamoto, A.; Sudo, H.; Endo, A. *Atherosclerosis* **1980**, *35*, 259.

(5) (a) For a first synthesis of (+)-compactin, see: Wang, Nai-Y.; Hsu, Chi-T.; Sih, C. J. *J. Am. Chem. Soc.* **1981**, *103*, 6538. (b) For very recent synthetic studies, see: Danishefsky, S.; Kerwin, J. F., Jr.; Kobayashi, S. *J. Am. Chem. Soc.* **1982**, *104*, 358. Prugh, J. D.; Deana, A. A. *Tetrahedron Lett.* **1982**, 281. Funk, R. L.; Zeller, W. E. *J. Org. Chem.* **1982**, *47*, 180.

(6) (a) Oppolzer, W.; Snowden, R. L. *Tetrahedron Lett.* **1976**, 4187. (b) The *endo* rule of Diels-Alder reactions does not play a dominant role in intramolecular cycloadditions; see: Boeckman, R. K., Jr.; Ko, S. S.; *J. Am. Chem. Soc.* **1980**, *102*, 7146. Roush, W. R. *J. Org. Chem.* **1979**, *44*, 4008. Roush, W. R.; Hall, S. E. *J. Am. Chem. Soc.* **1981**, *103*, 5200 and references cited therein.

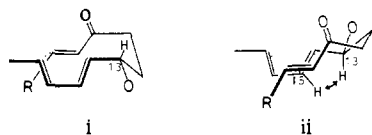
Scheme I



reaction, because the asymmetric center at C13 was expected to control the approach of the dienophile from a single diastereomeric face by steric and stereoelectronic interactions.⁷ (3) Wadsworth–Emmons coupling of the optically active segments A (5) and B (6) was envisioned for the expeditious preparation of the requisite (*E,E,E*)-trienone 4. (4) Development of an asymmetric synthesis of 6, a masked β -hydroxy δ -lactone, should be valuable for the preparation of polyketide-derived natural products.

Preliminary studies on the intramolecular Diels–Alder reaction of racemic **7**⁹ led to *trans*-octalone **8** with the desired relative stereochemistry.¹⁰ Thus, *S* configuration at C13 in **7** (4) is

(7) Although the preference of one diastereomeric transition state over the other in *endo* (or *exo*) intramolecular Diels–Alder reactions has been accounted for only in terms of steric interaction,^{8a} stereoelectronic control due to favorable orbital overlap^{8b} between an allylic substituent bond and an incipient bond should play a significant role: in the present case, the transition states in which the allylic C13–O bond is nearly perpendicular to the diene plane may be greatly stabilized. Thus, we postulated that the two *exo* transition states i, leading to desired **3**, and ii, leading to the undesired isomer, are preferred by stereoelectronic interaction and that i is more favored because of the absence of an unfavorable steric interaction between 13-H and 15-H in ii.

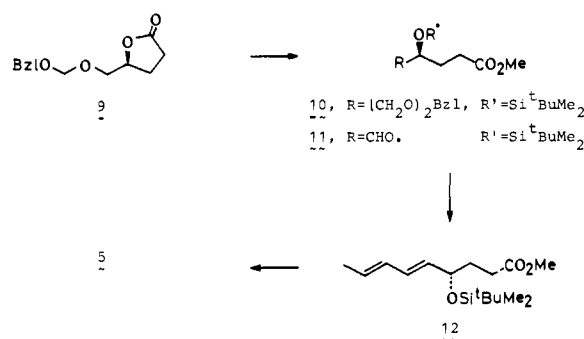


(8) (a) Nicolaou, K. C.; Magolda, R. L. *J. Org. Chem.* **1981**, *46*, 1506. Roush, W. R.; Myers, A. G. *Ibid.* **1981**, *46*, 1509. (b) For the importance of antiperiplanarity between an incipient bond and an adjacent σ bond in 1,2-asymmetric induction, see: Anh, N. T.; Eisenstein, O. *Nouv. J. Chim.* **1977**, *1*, 61.

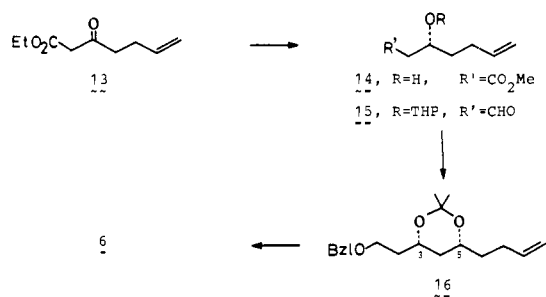
(9) (a) All new compounds exhibited satisfactory IR and ¹H NMR spectral properties as well as analytical ($\pm 0.3\%$) or exact mass data. (b) The specific rotations, $[\alpha]_D$, (concentration) in CHCl₃ of representative intermediates are as follows: **3**, +37.4° (0.4); **4**, -1.4° (2.3); **5**, -1.2° (1.5); **6**, -2.5° (1.1); **10**, -13.4° (1.0); **11**, -37.5° (1.2); **12**, +3.1° (1.9); **14**, -22.1° (1.1); **15**, +7.8° (1.0); **16**, -16.0° (1.1); **17**, +27.4° (2.0); **18**, +50.5° (1.3); **19**, +57.6° (0.6); **20**, +120.4° (0.8); **21**, +120.8° (0.6).

(10) These studies including the C13-substituent and Lewis acid effects on stereoselectivity will be discussed in a separate publication.

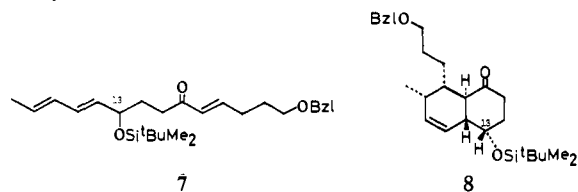
Scheme II



Scheme III



required for obtaining the desired absolute stereochemistry in the optically active series.



The synthesis of segment A (5) in its optically active form started from readily available 4(*S*)- γ -lactone **9**¹¹ (Scheme II). Lactone opening (LiOMe, MeOH) and silylation (*t*-BuMe₂SiCl) gave a chromatographically easily separable 1:1 mixture of recovered **9** and silyl ether **10** in 94% overall yield. Recovered **9** was recycled to produce **10**. Hydrogenolysis (H₂/Pd, EtOH) of **10** followed by Collins oxidation yielded the aldehyde **11** (79%). Stereoselective preparation of (*E,E*)-diene **12** was accomplished by addition of *trans*-crotyl phenyl sulfone anion¹² (-78 °C, 3 min) followed by quenching with Ac₂O and subsequent reductive elimination of sulfone acetate¹³ [3% Na(Hg), -24 °C] in 75% overall yield.¹⁴ Condensation of **12** with the lithium anion of dimethyl methylphosphonate produced segment A (5), 85% yield.¹⁴

The synthesis of segment B (6) was designed to utilize the inexpensive and convenient asymmetric reduction of β -keto acid derivatives with baker's yeast.¹⁵ After considerable preliminary experiments **13**¹⁶ was chosen as a starting material because it carried the functionality capable of further elaboration to **6**

(11) The benzyloxymethylated **9** ($[\alpha]_D^{25} +18.0^\circ$ (*c* 1.6, CHCl₃)) was prepared from 4(*S*)-(hydroxymethyl)butyrolactone [Taniguchi, M.; Koga, K.; Yamada, S. *Tetrahedron* **1974**, *30*, 3574] by the standard method (PhCH₂OCH₂Cl, *i*-Pr₂NEt, CH₂Cl₂).

(12) It is essential to use *trans*-crotyl phenyl sulfone of high isomeric purity for stereoselective formation of **12**. The sulfone of 96% *E* was prepared from commercial *trans*-2-buten-1-ol (*E* $\geq 96\%$) in two steps (36% yield): (i) 1.15 equiv of Ph₃P, 1.0 equiv of NBS, CH₂Cl₂, 0 °C, 30 min; (ii) 0.5 equiv of NaSO₂Ph·2H₂O, MeOH, 25 °C, 42 h.

(13) Kocienski, P. J.; Lythgoe, B.; Ruston, S. *J. Chem. Soc., Perkin Trans. I* **1978**, 829.

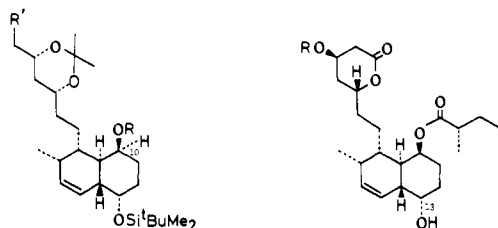
(14) 360-MHz ¹H NMR spectra of **12** and **5** revealed the presence of 4% of 14-*E*,16-*Z* and 6% of 14-*Z*,16-*E* isomers.

(15) (a) Lemieux, R. U.; Giguere, J. *Can. J. Chem.* **1951**, *29*, 678. (b) Deol, B. S.; Ridley, D. D.; Simpson, G. W. *Aust. J. Chem.* **1976**, *29*, 2459. Fräter, G. *Helv. Chim. Acta* **1979**, *62*, 2829.

(16) Prepared from 5-hexen-2-one [CO(OEt)₂, NaH; 76%; bp 115–116 °C (20 mmHg)] cf.: Soloway, S. B.; LaForge, F. B. *J. Am. Chem. Soc.* **1947**, *69*, 2677.

(Scheme III). Saponification (KOH, EtOH, H₂O), reduction (baker's yeast, D-glucose, H₂O, 25 °C, 2 days), and methylation (CH₂N₂) produced chiral β-hydroxy ester **14**, 35% yield, in >99% ee as determined by ¹H NMR chiral shift studies [360 MHz, Eu(tfc)₃]. The absolute configuration was tentatively assigned by analogy to the reduction of 3-oxohexanoate^{15a} and confirmed by eventual conversion to natural product **1a** (vide infra). This ester **14**, after conversion to its THP ether, was reduced with DIBAL-H to afford the aldehyde **15** in 85% yield. Transformation of **15** to a chromatographically readily separable 1:1 mixture of **16**¹⁷ and its 3-*R* isomer was performed in a straightforward manner in five steps (48% overall): (i) aldol condensation (EtOAc, LDA, -78 °C), (ii) deprotection of THP ether (PPTS, EtOH), (iii) acetonide formation (2,2-dimethoxypropane, *p*-TsOH), (iv) reduction (LiAlH₄), and (v) protection (PhCH₂Br, NaH, DMF). Finally, careful ozonolysis of **16** (MeOH, -78 °C → Me₂S) completed the preparation of segment B (**6**) in 88% yield.

The coupling of **5** and **6** in THF (1.3 equiv of NaH, 1.0 equiv of **6**, 0 °C, 5 min → 25 °C, 10 min) smoothly produced the (*E,E,E*)-trienone **4**, 86% yield. Cyclization of **4** in refluxing chlorobenzene (N₂, 82 h) proceeded more slowly than **7** to give the desired *trans*-octalone **3** (28%) and two *cis* isomers (45% and 9%).¹⁸ The stereochemistry on the octalone ring of **3**, which embodies five correct asymmetric centers out of six in the carbon framework of **1a**, was evident from its ¹H NMR spectrum.¹⁸ K-Selectride reduction (2 equiv in THF, 25 °C) of **3** introduced selectively the requisite axial alcohol **17** (87%).¹⁹ Esterification



17, R = H; R' = CH₂OBz
18, R = COCH(Me)Et; R' = CH₂OBz
19, R = COCH(Me)Et; R' = CO₂Me

20, R = H
21, R = Si-*t*-BuMe₂

with 2(*S*)-methylbutyric anhydride (DMAP, pyridine, 25 °C, 20 h) yielded **18** (70%),^{19,20} which was debenzylated (Li/NH₃, -78 °C, 10 min; 73%) and oxidized [Collins, PDC(DMF), and CH₂N₂] to the methyl ester **19** (65%). Exposure of **19** to 47% aqueous HF-CH₃CN (1:10) at 25 °C for 1 h resulted in desilylation, deprotection of the acetonide, and subsequent lactonization to afford **20**, mp 178–180 °C, 70% yield. The final operation remaining for the completion of the synthesis, regioselective dehydration of the C13 axial OH in **20**, was initiated by selective protection of the lactonic OH as the *tert*-butyldimethylsilyl ether **21** (65%). Dehydration under the mild condition (SOCl₂, pyridine; 0 °C, 15 min → 25 °C, 15 min) followed by removal of the silyl protecting group [47% aqueous HF-CH₃CN (1:10), 25 °C, 30 min] completed the synthesis of **1a** (51% from **21**), which was identical (mp, 360-MHz NMR, IR, UV, MS, [α]_D, TLC) with natural ML-236B (compactin).

Application of the described methodology to the synthesis of analogues and refinement of stereoselectivity are currently under investigation.

Acknowledgment. We thank Professor Akira Endo, Tokyo Noko University, for a generous sample of ML-236B and Drs.

(17) The desired stereochemistry of **16** was evident from ¹H NMR data: H_{4a} (δ 1.15) appeared as doublet of triplet (*J*_{4a,4b} = 12.7 Hz, *J*_{4a,3} = *J*_{4a,5} = 11.5 Hz).

(18) Characteristic ¹H NMR data of **3**: *J*_{9,14} = *J*_{8,9} = 11.5 Hz, *J*_{8,17} = 5.3 Hz, *J*_{14,15} = 1.8 Hz, *J*_{14,16} = 2.7 Hz, *J*_{15,16} = 10.0 Hz, *J*_{16,17} = 5.2 Hz, *J*_{15,17} = 1.2 Hz, *J*_{13,14} ≈ 2 Hz; major *cis*, *J*_{9,14} = 6.2 Hz; minor *cis*, *J*_{9,14} = 6.0 Hz.

(19) The narrow *W*_{1/2} (7 Hz) of the ester carbinol proton (10-H, δ 5.14) of **18** supported the depicted C10 stereochemistry in **17**.

(20) 2(*S*)-Methylbutyric anhydride [bp 65.5 °C (~1 mmHg)], [α]_D²⁴ +29.2° (neat)] was prepared from 2(*S*)-methylbutanol (Nakarai) by the standard procedures; see ref 5a.

Takeo Sakan, Koji Nakanishi, and Kyosuke Nomoto for valuable discussions.

Registry No. **1a**, 73573-88-3; **3**, 82080-53-3; **4**, 82080-54-4; **5**, 82065-57-4; **6**, 82065-58-5; **7**, 82080-55-5; **8**, 82065-59-6; **9**, 82065-60-9; **10**, 82065-61-0; **11**, 82065-62-1; **12**, 82065-63-2; **13**, 17605-06-0; **14**, 82065-64-3; **15**, 82065-65-4; **16**, 82065-66-5; **17**, 82065-67-6; **18**, 82065-68-7; **18** debenzylated derivative, 82065-69-8; **19**, 82065-70-1; **20**, 82065-71-2; **21**, 82080-56-6; **16**, 3(*R*) isomer, 82110-38-1; 2(*S*)-methylbutyric anhydride, 65527-79-9; *trans*-crotlyl phenyl sulfone, 72863-24-2; *trans*-2-buten-1-ol, 504-61-0; 5-hexen-2-one, 109-49-9.

Supplementary Material Available: Spectroscopic data (NMR and IR) for new compounds described in this paper (27 pages). Ordering information is given on any current masthead page.

Novel Binuclear Platinum(III) Diphosphite Complexes

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Relatively few platinum(III) complexes have been reported to date. The best characterized are Pt-Pt-bonded binuclear species, with Pt-Pt distances in the range 2.47–2.56 Å.² In recent experiments we have found that binuclear Pt(III) complexes can be generated readily through oxidative addition to a binuclear platinum(II) tetrakis(diphosphite), Pt₂(pop)₄⁴⁻ (pop = P₂O₅H₂²⁻).³ The platinum(III) products of halogen and methyl iodide oxidative-addition reactions are described in this communication.

The binuclear Pt(II) species Pt₂(pop)₄⁴⁻ reacts rapidly with halogens (or CH₃I) to give Pt₂(pop)₄X₂⁴⁻ (or Pt₂(pop)₄(CH₃)I⁴⁻).⁴ The Pt-Pt distance in Pt₂(pop)₄Cl₂⁴⁻ (Figure 1)⁵ is 2.695 (1) Å,

(1) (a) California Institute of Technology; (b) Washington State University.

(2) (a) K₂[Pt₂(SO₄)₄(H₂O)₂], Pt-Pt = 2.47 Å: Muravieskaya, G. S.; Kukina, G. A.; Orlova, V. S.; Evstefera, O. N.; Porai-Koshits, M. A. *Dokl. Akad. Nauk. SSSR* **1976**, *226*, 596–599. (b) Pt₂(O₂C₂F₃)₂(CH₃)₄(NC₆H₇)₂, Pt-Pt = 2.557 Å: Schagen, J. D.; Overbeck, A. R.; Schenk, H. *Inorg. Chem.* **1978**, *17*, 1938–1940. (c) Pt₂(C₃H₄NO)₂(NH₃)₄(NO₂)₂, Pt-Pt = 2.539 (1) Å: Hollis, L. S.; Lippard, S. J. *J. Am. Chem. Soc.* **1981**, *103*, 6761–6763. (d) Na₂[Pt₂(HPO₄)₄(H₂O)₂], Pt-Pt = 2.486 (2) Å: Cotton, F. A.; Falvello, L. R.; Han, S. *Inorg. Chem.* **1982**, *21*, 1709–1710.

(3) Striking luminescence led to the discovery of the platinum anion (Sperline, R. P.; Dickson, M. K.; Roundhill, D. M. *J. Chem. Soc., Chem. Commun.* **1977**, 62–63), and the emission intensity linearity has been used for the spectrophotometric detection of trace platinum (Dickson, M. K.; Peltee, S. K.; Roundhill, D. M. *Anal. Chem.* **1981**, *53*, 2159–2160). The compound K₄[Pt₂(pop)₄]·2H₂O has been structurally characterized (Filomena Dos Remedios Pinto, M. A. Sadler, P. J.; Neidle, S.; Sanderson, M. R.; Subbiah, A. J. *Chem. Soc., Chem. Commun.* **1980**, 13–15).

(4) K₄[Pt₂(pop)₄X₂] (X = Cl, Br) was prepared by adding excess X₂ and then KX to an aqueous solution of K₄[Pt₂(pop)₄]·2H₂O at room temperature. [Ph₄As]₄[Pt₂(pop)₄I₂] was prepared by adding excess I₂ to [Ph₄As]₄[Pt₂(pop)₄] in acetonitrile solution. Anal. Calcd for K₄[Pt₂(pop)₄Cl₂]·2H₂O: P, 20.2; Cl, 5.77. Found: P, 19.0; Cl, 6.17. Anal. Calcd for K₄[Pt₂(pop)₄Br₂]: P, 19.3; Br, 12.5. Found: P, 19.2; Br, 12.4. Anal. Calcd for [Ph₄As]₄[Pt₂(pop)₄I₂]: P, 9.00; I, 9.22. Found: P, 9.80; I, 9.22. Anal. Calcd for K₄[Pt₂(pop)₄(CH₃)I]: C, 0.95; H, 0.88; I, 10.0; P, 19.6. Found: C, 1.25; H, 0.78; I, 10.5; P, 19.4. The complexes are diamagnetic 1:4 electrolytes and are very stable both in the solid state and in solution.