

5 to afford malonate derivative 8 (46% from 5) was accomplished by reaction with dimethyl oxomalonate¹⁷ to obtain 6, conversion to chloride 7 (SOCl₂, pyridine, THF), and reduction (Zn, HOAc).¹⁸ Cleavage of the acetal functionality of 8 (NBS, C₆H₆) afforded crystalline 9 (64%).¹⁹

Construction of the enolpyruvate ester moiety from malonate derivative 9 was achieved by formation of Mannich base 10 [CH₂=N⁺(CH₃)₂I⁻, (C₂H₅)₃N, CH₂Cl₂], quaternization of the amino group (CH₃I, CH₂Cl₂), and thermolysis at 80–85 °C in dimethyl sulfoxide to obtain 11²⁰ (47% from 9). Reaction of 11 with CH₃O⁻ in CH₃OH gave 12 and a small amount of 13 (~15%) from which pure, crystalline epoxide 12²¹ (46%) was obtained by crystallization from CH₃OH. Epoxide 12 underwent regio-specific ring opening with PhSe⁻ (Ph₂Se₂, NaBH₄, CH₃OH) to give 14.²² Treatment of 14 with 30% H₂O₂ in CH₂Cl₂:THF at 0 °C afforded the selenoxide derivative. After addition of 3 equiv of NaHCO₃, the mixture was kept at room temperature for 1.5 h, during which time selenoxide elimination occurred to give dimethyl chorismate (15)^{23,24} in 28% yield from 12.

Hydrolysis of 15 (2.2 equiv of NaOH in THF–H₂O, 3.5 h, 0 °C) followed by treatment with Amberlite IR-120 resin and removal of water afforded a 3:2 mixture of 1 and 16 from which pure, racemic 1 was obtained by successive recrystallization from ethyl acetate/hexane. The IR, UV, NMR, and mass spectral data of synthetic 1 (mp 139.5–141 °C dec) were identical with the spectral data of 1 obtained from culture growth of *A. aerogenes* (62-1).^{5,26}

Acknowledgment. We are grateful to the National Institutes of Health, Grant GM 19103, for financial support.

(16) Satisfactory spectral data have been obtained for all new substances described. Satisfactory analytical data (combustion or high-resolution mass spectrum) have been obtained for 1, 4, 5, 7, 9, 11, 12, and 15. High-resolution mass spectra were provided by the facility supported by National Institutes of Health Grant RR00317 (principal investigator Professor K. Biemann) from the Biotechnology Resources Branch, Division of Research Resources.

(17) The most convenient preparation of dimethyl oxomalonate is by modification of Salomon's procedure for the diethyl ester: Pardo, S. N.; Salomon, R. G. *J. Org. Chem.* **1981**, *46*, 2598–2599.

(18) The sequence 5 → 8 is based on literature procedures developed to effect similar substitution on the nitrogen atom of β-lactams: (a) Schmitt, S. M.; Johnston, D. B. R.; Christensen, B. G. *J. Org. Chem.* **1980**, *45*, 1135–1142, 1142–1148. (b) Scartazzini, R.; Peter, H.; Bickel, H.; Heusler, K.; Woodward, R. B. *Helv. Chim. Acta* **1972**, *55*, 408–417.

(19) Cleavage of the acetal group of 8 is not regio-specific. The 4-bromo-5-benzoyloxy derivative is obtained also (24%), and the two isomers are separated by chromatography on silica gel. Although we used only isomer 9 for further transformations, in principle it should be possible to convert the second isomer to 12 by procedures similar to those used to convert 9 to 12.

(20) 11: IR (CH₂Cl₂) 1721, 1619 cm⁻¹; ¹H NMR (CDCl₃, 250 MHz) δ 8.03 (dd, 2 H, J = 8.5, 1.5 Hz, *o*-ArH), 7.59 (t, 1 H, J = 7 Hz, *p*-ArH), 7.46 (dt, 2 H, J = 7.7, 2 Hz, *m*-ArH), 6.94 (dd, 1 H, J = 2.7, 1.7 Hz, H₂), 5.73 (dd, 1 H, J = 10.7, 7.3 Hz, H₄), 5.50 (d, 1 H, J = 2.6 Hz, *trans*-OC=CH), 4.93 (m, 1 H, H₃), 4.88 (d, 1 H, J = 2.6 Hz, *cis*-OC=CH), 4.34 (dt, 1 H, J = 10.3, 5.9 Hz, H₅), 3.80 (s, 3 H, OCH₃), 3.69 (s, 3 H, OCH₃), 3.27 (dd, 1 H, J = 18.2, 5.7 Hz, H₆), 2.99 (m, 1 H, H₆).

(21) 12: mp 119–120 °C; IR (CH₂Cl₂) 1720, 1621 cm⁻¹; ¹H NMR (CDCl₃, 250 MHz) δ 6.83 (m, 1 H, H₂), 5.59 (d, 1 H, J = 3.3 Hz, *trans*-OC=CH), 4.94 (m, 1 H, H₃), 4.93 (d, 1 H, J = 3.3 Hz, *cis*-OC=CH), 3.82 (s, 3 H, OCH₃), 3.76 (s, 3 H, OCH₃), 3.48 (m, 1 H, H₄ or H₅), 3.40 (m, 1 H, H₄ or H₅), 2.94 (br d, 1 H, J = 19.8 Hz, H₆), 2.76 (br d, 1 H, J = 19.8 Hz, H₆).

(22) 14: ¹H NMR (CDCl₃, 250 MHz) δ 7.6–7.2 (m, 5 H, Ar–H), 6.71 (br s, 1 H, H₂), 5.58 (d, 1 H, J = 2.9 Hz, *trans*-OC=CH), 4.89 (d, 1 H, J = 2.9 Hz, *cis*-OC=CH), 4.67 (m, 1 H, H₃), 3.81 (s, 3 H, OCH₃ and m, 1 H, H₄), 3.71 (s, 3 H, OCH₃), 3.42 (s, 1 H, OH), 3.30 (dt, 1 H, J = 11.4, 5.4 Hz, H₅), 2.95 (dd, 1 H, J = 18.1, 5.3 Hz, H₆), 2.46 (m, 1 H, H₆).

(23) 15: mp 94–96 °C; IR (CH₂Cl₂) 1730, 1627 cm⁻¹; ¹H NMR (CDCl₃, 250 MHz) δ 6.91 (br s, 1 H, H₂), 6.35 (br d, 1 H, J = 10.3 Hz, H₅ or H₆), 6.05 (dd, 1 H, J = 10.3, 2.0 Hz, H₅ or H₆), 5.56 (d, 1 H, J = 2.9 Hz, *trans*-OC=CH), 4.90 (d, 1 H, J = 13.2 Hz, H₃ or H₄), 4.86 (d, 1 H, J = 13.2 Hz, H₃ or H₄), 4.80 (d, 1 H, J = 2.9 Hz, *cis*-OC=CH), 3.83 (s, 3 H, OCH₃), 3.79 (s, 3 H, OCH₃), 3.15 (br s, 1 H, OH); ¹³C NMR (CDCl₃) 164.7 (s), 163.6 (s), 149.4 (s), 133.7 (d), 132.0 (d), 129.4 (s), 121.7 (d), 98.7 (t), 81.6 (d), 70.9 (d), 52.6 (q), 52.1 (q).

(24) The IR and ¹H NMR spectral data were in agreement with those reported for 15 prepared from reaction of CH₂N₂ with 1 isolated from culture growth of *A. aerogenes*.²⁵

(25) Ife, R. J.; Ball, L. F.; Lowe, P.; Haslam, E. *J. Chem. Soc., Perkin Trans. 1* **1976**, 1776–1783.

(26) We thank Professor F. Gibson for providing a sample of *A. aerogenes* (62-1).

Hexamethylenetetratellurafulvalene

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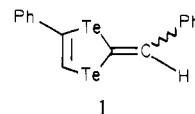
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One of the most formidable challenges to the designer of organic metals over the past eight years has been the preparation of tellurium analogues of tetrathiafulvalene (TTF) and its alkylated derivatives.¹ Engler² and Cowan³ have shown that dramatic changes in physical properties can be gained in going from the thiafulvalene to selenafulvalene TCNQ salts. Of these, hexamethylenetetraselenafulvalene was the most interesting because its TCNQ salt behaved like a semimetal,⁴ and contrary to all other members of its class it did not become an insulator at low temperature. More recently, Bechgaard⁵ showed that tetramethyltetraselenafulvalene (TMTSF) could be converted to a family of salts [(TMTSF)₂X; X = univalent anion], one of which [(TMTSF)₂ClO₄] was found to be a superconductor at 1.3 K. Would the tellurium analogue be a superconductor at higher temperature?

In this communication we report on the first directed synthesis of a tetratellurafulvalene. All previous reports on organotellurium chemistry dealt with methodology for carbon–tellurium bond-forming reactions.

Work on the synthesis of a tellurafulvalene was pursued very seriously only after the Russian claim⁷ that a 1,3-ditellurole (1)



could be isolated without difficulty and that it was stable.⁶ Prior to this finding, it was assumed that such a molecule (and consequently the tellurafulvalenes) would be very unstable because tellurium was thought to be too large (covalent radius 1.37 Å) to be incorporated into a five-membered heterocycle with the two tellurium heteroatoms in a 1,3 relationship.

The heterocycle 1 (cis isomer) was prepared in reasonable yield along with a number of small-ring telluroheterocycles⁷ via phenyltelluroacetylide (PhC≡CTe⁻Na⁺) in a modified version of the original claim. One of the most versatile synthetic reactions for the preparation of tellurium heterocycles is the addition of telluride to an acetylene linkage,⁸ and synthetic schemes for the preparation of 1,3 ditelluroles based on this reaction were reported⁹ and are being explored.

Scheme I (with the following conditions: (i) *t*-BuLi, THF, –80 °C; (ii) Te, –15 °C; (iii) Cl₂C=CCl₂, –80 to 25 °C) depicts our approach to the title compound, 7. From the outset we were pessimistic about the coupling of two ditellurole moieties, the most effective method employed for the preparation of thia- and sel-

(1) "The Physics and Chemistry of Low Dimensional Solids"; L. Alcacer, Ed.; D. Reidel Publishing Co.: Dordrecht, Holland, 1980.

(2) Engler, E. M.; Patel, V. V.; Andersen, J. R.; Tomkiewicz, Y.; Craven, R. A.; Scott, B.; Etemad, W. *Ann. N.Y. Acad. Sci.* **1978**, *313*, 343 and references therein.

(3) Bloch, A. N.; Carruthers, T. F.; Poehler, T. O.; Cowan, D. O. In "Chemistry and Physics of One Dimensional Metals"; H. J. Keller, Ed.; Plenum: New York, 1977; p 47. Cowan, D. O.; Shu, P.; Hu, C.; Krug, W.; Carruthers, T. F.; Poehler, T. O.; Bloch, A. N. *Ibid.*, p 25.

(4) Reference 3. Jerome, D.; Weger, M. In ref 3, p 341.

(5) Bechgaard, K.; Carneiro, K.; Rassmussen, F. B.; Rinsdorf, G.; Jacobsen, C. S.; Pedersen, H. J.; Scott, J. C. *J. Am. Chem. Soc.* **1981**, *103*, 2440 and references therein.

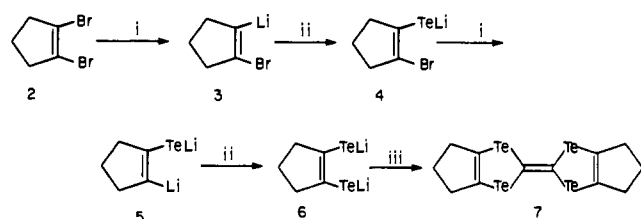
(6) Petrov, M. L.; Iaihev, V. Z.; Petrov, A. A. *Russ. J. Org. Chem. (Engl. Transl.)* **1979**, *15*, 2346.

(7) The Russian claim was premature; see: Lakshminantham, M. V.; Cava, M. P.; Albeck, M.; Engman, L.; Wudl, F.; Aharon-Shalom, E. *J. Chem. Soc., Chem. Commun.* **1981**, 828. Bender, S. L.; Haley, N. F.; Luss, H. R. *Tetrahedron Lett.* **1981**, 22, 1495.

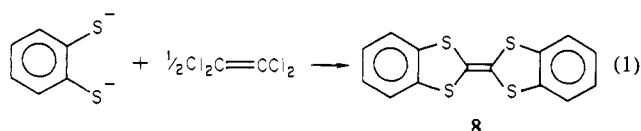
(8) Lehner, W.; Praefcke, K. *Chem. Ber.* **1978**, *111*, 3745.

(9) Sukhai, R. S.; Verboov, W.; Meijer, J.; Schoufs, M. J. M.; Brandsma, I. *Recl. Trav. Chim. Pays-Bas* **1981**, *100*, 10.

Scheme I



enfulvalenes. There existed a precedent for the preparation of benzologues of tetrathiafulvalene,¹⁰ shown in reaction 1. However,



this reaction was carried out at elevated temperature in a protic solvent, hardly the conditions conducive to the survival of an organotelluride anion.¹¹ We were counting on the very high nucleophilicity of telluride for the success (under much milder conditions) of the last step of our synthesis.

We could have attempted the presumably simpler synthesis of the benzologue of a tetratellurafulvalene since *o*-dilithiobenzene is known,¹² but we decided against it because we expected this tellurafulvalene to be extremely insoluble; also, the sulfur analogue **8**, to date, has not given rise to any compounds with spectacular physical properties.¹³

While it was possible to trap the dianion derived from reduction of the two bromines in **2** via Rieke magnesium,¹⁴ the yield was irreproducible and mostly low. The 1,2-dilithiocyclopentene was, not unexpectedly, extremely reactive. Its presence could be detected as trapping products (I₂, D₂O), but once again the yield was poor. The observations that **3** could be generated¹⁵ quantitatively¹⁶ at -78 °C and that it could be quenched with Te were not surprising. What was somewhat surprising was that the newly formed carbon-tellurium bond (**4**) survived further lithium halogen exchange. The dianion **5** presented no difficulties in its further reaction with Te to produce the dilithioditelluride¹⁷ **6**.

The crucial step, reaction with tetrachloroethylene, occurred rapidly at -78 °C as evidenced by sudden color changes and formation of a precipitate. Further test reactions showed that it was not a simple nucleophilic substitution but a more complicated process.¹⁸ Ironically, in our hands, this synthetic step does not lead to the production of detectable amounts of the selenium analogue (HMTSF).

Thus, all the steps in the scheme could be carried out in a single reaction vessel, and the crude (unoptimized) yield of THF insoluble, CS₂ extractable solids was 32% based on C₁₂H₁₂Te₄. The product consisted of two fractions distinguishable by relative

(10) Bajwa, J. S.; Berlin, K. D.; Pohl, H. A. *J. Org. Chem.* **1976**, *41*, 145 and references within.

(11) "The Organic Chemistry of Tellurium"; K. Irgolic, Ed.; Gordon and Breach Science Publishers: New York, 1974.

(12) Wittig, G.; Bickelhaupt, F. *Chem. Ber.* **1958**, *91*, 883.

(13) Schjibaeva, R. P.; Yarochkina, O. V. *Sov. Phys.-Dokl. (Engl. Transl.)* **1975**, *20*, 304. Lyubovskaya, R. N.; Aldoshina, M. Z.; Rodionov, V. Ya.; Chibisova, T. A.; Khidekel, M. L. *Izv. Akad. Nauk SSSR, Ser. Khim.* **1975**, 177. Jones, M. T.; Kellerman, R.; Troup, A.; Sandman, D. *J. Chem. Scr.* **1981**, *17*, 43.

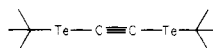
(14) Rieke, R. D.; Bales, S. E. *J. Am. Chem. Soc.* **1974**, *96*, 1775.

(15) Wittig, G.; Weinlich, I.; Wilson, E. R. *Chem. Ber.* **1965**, *98*, 458.

(16) Neumann, H.; Seebach, D. *Chem. Ber.* **1978**, *111*, 2785.

(17) We use ClCH₂OC₂H₅ as quantitative trapping reagent: Wudl, F.; Nalewajek, D. *J. Organomet. Chem.* **1981**, *217*, 329.

(18) Treatment of lithium *tert*-butylltelluroate (from *tert*-butyllithium and tellurium) with tetrachloroethylene afforded



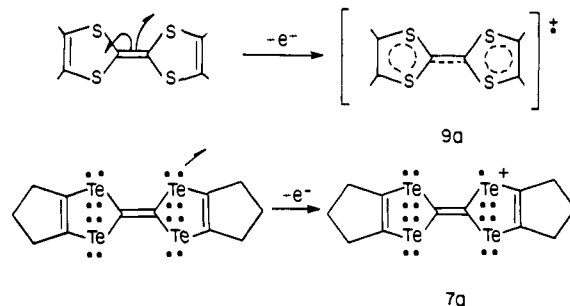
and di-*tert*-butyl ditelluride. This indicates that the reaction leading from **6** to **7** (see Scheme I) is probably not a straightforward addition-elimination process but may involve alkyne intermediates.

Table I

compd ^a	UV-vis, nm	E ₁ ^{1/2c} , V	E ₂ ^{1/2c} , V	ΔE, V
TMTTF	315 (14 000), ^b 327 (13 800), ^b 473 (248) ^b	0.24	0.73	0.49
TMTSF	299 (12 600), ^d 508 (200), ^d 534 (176) ^e	0.42	0.81	0.39
HMTTeF ^g	320 (15 560), 335 (19 350), 410 (9115), 610 (189), ^f 653 (121) ^e	0.40	0.69	0.29

^a HMTTF and TMTTF as well as HMTSF and TMTSF have practically identical electronic spectra and REDOX properties.^{23,24}
^b ClCH₂CH₂Cl, ref 23. ^c V vs. SCE; C₆H₅CN; Bu₄N AsF₆, electrolyte, reversible waves, scan rate 100 mV/s. ^d CH₂Cl₂, ref 25. ^e CS₂. ^f CH₂ClCHCl₂. ^g IR (sample sublimed on a NaCl disc, cm⁻¹): 2965, s; 2940, s; 2900, vs; 2840, s; 1560, w; 1475, s; 1302, m; 1265, w; 1200, w; 1080, s; 1020, w; 812, m; 795, w; 636, m. Note that C=C stretch shifted²⁷ from 1618 to 1560 cm⁻¹.

Scheme II



solubility in carbon disulfide. The less soluble component is assigned the title compound's structure on the basis of elemental analysis,¹⁹ mass spectroscopy (fragmentation pattern identical with HMTSF,²⁰ except for the relative *m/e* of Te and Se and perfect match to a computer simulation of the parent peak), and other physical properties shown in Table I. The most salient feature of this collection of data is the cyclic voltammetry. The tellurium fulvalene exhibits reversible waves in both redox steps in benzonitrile; in 1,1,2-trichloroethane, *only the first sweep* shows the two reversible waves because the electrode is immediately coated with a substance (radical cation salt?) that is insoluble in this solvent. The trend to smaller values of ΔE in the series TMTTF > TMTSF > HMTTeF, coupled with the fact that electron transfer is slowest from TMTTF and fastest from HMTTeF, allows us to propose that ionization in the sulfur fulvalene occurs primarily from the π-bonded network, whereas ionization in the tellurium fulvalene originates from the tellurium lone pairs, viz. Scheme II.

Stabilization of **7a** is probably derived, to a greater extent, from the lower electronegativity of Te vs. Se and S and from transannular lone-pair interaction²¹ but to a lower degree from π delocalization through the carbon framework because of the disparate size of tellurium and carbon.²²

(19) Correct elemental analysis for C, H, and Te was obtained.

(20) Andersen, J. R.; Egsgaard, H.; Larsen, E.; Bechgaard, K.; Engler, E. *M. Org. Mass Spectrom.* **1978**, *13*, 121.

(21) Musker, W. K.; Wolford, T. L.; Roush, P. B. *J. Am. Chem. Soc.* **1978**, *100*, 6416. Asmus, K.-D.; Bahnmann, D.; Fischer, Ch.-H.; Weltwisch, D. *Ibid.* **1979**, *101*, 5322. If these authors could find such stabilization for the much smaller and more electronegative sulfur atom, then it stands to reason that the less electronegative and more polarizable Te atom could participate in this process.

(22) Tellurium only reluctantly forms "double bonds" to carbon: Lerstrup, K. A.; Henriksen, L. *Chem. Commun.* **1979**, 1102. Barrett, A. G. M.; Barton, D. H. R.; Read, R. W. *Ibid.* **1979**, 645. Barrett et al. found they could prepare a C=Te functional group but that it could be best represented by C⁺-Te⁻ and that it was unstable.

(23) Bechgaard, K.; Andersen, J. R. In ref 3, p 247.

(24) Engler, E. M., private communication.

(25) Bechgaard, K.; Cowan, D. O.; Bloch, A. N. *Chem. Commun.* **1974**, 937.

The low solubility of **7** in the usual polar electrochemistry solvents (acetonitrile, methylene chloride, etc.) has, so far, thwarted our attempts to grow salts electrochemically. We have, however, been able to prepare a TCNQ salt but so far only in the form of microcrystalline powder. Preliminary ESR measurements on this solid give a g value of 2.0039 and a line width of 4 G, indicating that the spins seem to be observable only on the TCNQ molecules at 4 K. To date we measured only compressed powders of the TCNQ salt and found a resistivity of 0.6–1 Ω cm, comparable to TTF TCNQ.

We are currently attempting to improve crystal growth of the TCNQ salt as well as prepare other salts of HMTTeF and synthesize TMTTeF.²⁶

Acknowledgment. We thank S. H. Bertz for helpful discussions and some initial work related to cyclopentene dianion, E. A. Chandross for helpful suggestions and critical reading of the

manuscript, and R. S. Hutton and W. M. Walsh for solid-state ESR measurements.

Registry No. 2, 75415-78-0; 3, 23586-46-1; 4, 80631-57-8; 5, 80631-58-9; 6, 80631-59-0; 7, 80631-60-3; 7 TCNQ salt, 80631-61-4.

(26) A nonfulvenoid organic compound containing four tellurium atoms has recently been reported: D. J. Sandman, J. C. Stark, G. P. Hamill, W. A. Burke, B. M. Foxman, International Conference on Low Dimensional Conductors, Boulder, CO, August 9–14, 1981. See also: Shibaeva, R. P.; Kaminskii, V. F. *Cryst. Struct. Commun.* **1981**, *10*, 663.

(27) This weakening of the endocyclic C=C bond is probably due to transannular nonbonded Te–Te interactions in the five-membered ring and across rings. A six-membered ring isomer²⁸ would be expected to be less strained and consequently not show as dramatic a shift.

(28) A referee has correctly pointed out a fact, which has also not escaped our notice, that the synthetic method could also yield the six-membered ring isomer. The only way to resolve this structural problem is via X-ray crystallography. Dr. E. M. Engler has kindly supplied us with a sample of HMTSF-TCNQ for this purpose.

Additions and Corrections

Total Syntheses of (\pm)-Daunomycinone: Regiospecific Preparations of (\pm)-7,9-Dideoxydaunomycinone and 6,11-Dihydroxy-4-methoxy-7,8,9,10-tetrahydronaphthacene-5,9,12-trione [*J. Am. Chem. Soc.* **1981**, *103*, 6378]. FRANK M. HAUSER* and SUBBARAO PRASANNA, Department of Chemistry and Biological Sciences, The Oregon Graduate Center, Beaverton, Oregon 97006.

Page 6380: The three lines under Scheme II beginning with endo-18, exo-19,20, and endo-4 do not belong with this paper.

Carbon-14 Kinetic Isotope Effects in the Menshutkin-Type Reaction of Benzyl Benzenesulfonates with *N,N*-Dimethylanilines. Variation of the Effects with Substituents [*J. Am. Chem. Soc.* **1979**, *101*, 266]. HIROSHI YAMATAKA and TAKASHI ANDO,* The Institute of Scientific and Industrial Research, Osaka University, Suita, Osaka 565, Japan.

The reaction temperature shown in eq 1 and Table I should be 35 °C.

Catalysis of Ester Aminolysis by Divalent Metal Ions [*J. Am. Chem. Soc.* **1981**, *103*, 3233–3235]. R. M. PROPST III and L. S. TRZUPEK,* Department of Chemistry, Furman University, Greenville, South Carolina 29613.

Page 3234: The correct value for the second-order rate coefficient, k , in eq 1 is $1.5 \times 10^{-4} \text{ M}^{-1} \text{ s}^{-1}$, not 1.5×10^{-1} as shown.

Facile Reduction of Carbon Dioxide by Anionic Group 6B Metal Hydrides. Chemistry Relevant to Catalysis of the Water-Gas Shift Reaction [*J. Am. Chem. Soc.* **1981**, *103*, 3223]. DONALD J. DARENSBOURG,* ANDRZEJ ROKICKI, and MARCETTA Y. DARENSBOURG, Department of Chemistry, Tulane University, New Orleans, Louisiana 70118.

Page 3223, Table I: Two sets of numbers were interchanged. The ¹³C NMR data for HC(O)OCr(CO)₅⁻ is $\delta\text{C}(\text{eq}) = 216.0$ and $\delta\text{C}(\text{ax}) = 222.7$; for HC(S)SCr(CO)₅⁻ the $\delta\text{C}(\text{eq}) = 218.0$ and the $\delta\text{C}(\text{ax}) = 225.4$ ppm.

Optically Detected Magnetic Resonance Evidence for Carcinogen–Nucleic Acid Interaction in the Tetrahydro-9,10-epoxybenzo[*e*]pyrene–DNA Adduct [*J. Am. Chem. Soc.* **1981**, *103*, 5257–5259]. STEVEN M. LEFKOWITZ and HENRY C. BRENNER,* Department of Chemistry, New York University, New York, New York 10003.

Page 5258, left column, first paragraph: The limits of error in the ODMR frequencies should be ± 2 MHz (megahertz), rather

than ± 2 GHz, as printed. The ODMR frequencies themselves are in GHz.

Transition-Metal Complexes of Vitamin B₆ Related Compounds. 3. X-ray, Mössbauer, and Magnetic Properties of a Binuclear Iron(III) Complex Containing an Unusual Pyridoxal Derivative [*J. Am. Chem. Soc.* **1980**, *102*, 6040]. GARY J. LONG,* JAMES T. WROBLESKI, RAJU V. THUNDATHIL, DON M. SPARLIN, and E. O. SCHLEMPER, Departments of Chemistry, University of Missouri—Rolla, Rolla, Missouri 65401, and University of Missouri—Columbia, Columbia, Missouri 65201.

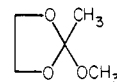
The space group and the extinctions reported in the abstract and on page 6041 are in error. The correct space group is *Pbca* and the correct extinctions are odd k absent for $0kl$, and l absent for $h0l$, and odd h absent for $hk0$. The structure was refined in the correct space group and all the structural results presented in the paper are correct.

Structure of Palytoxin [*J. Am. Chem. Soc.* **1981**, *103*, 2491–2494]. RICHARD E. MOORE* and GIOVANNI BARTOLINI, Department of Chemistry, University of Hawaii, Honolulu, Hawaii 96822.

The authors deeply apologize for misspelling the name of Professor Josef Dadok (not Datok) in the acknowledgment of this communication and a preceding communication on the periodate oxidation of *N*-(*p*-bromobenzoyl)palytoxin [Moore, R. E.; Woolard, F. X.; Bartolini, G. *J. Am. Chem. Soc.* **1980**, *102*, 7370].

Ortho Ester Hydrolysis: Direct Evidence for a Three-Stage Reaction Mechanism [*J. Am. Chem. Soc.* **1979**, *101*, 2669]. M. AHMAD, R. G. BERGSTROM, M. J. CASHEN, Y. CHIANG, A. J. KRESGE,* R. A. MCCLELLAND,* and M. F. POWELL, Department of Chemistry, University of Toronto, Scarborough College, West Hill, Ontario M1C 1A4, Canada.

We have detected an error in our calculation of the hydronium-ion catalytic coefficient for the hydrolysis of 2-methyl-2-methoxy-1,3-dioxolane (**11**) in biphosphate buffers:



11

The correct value is $k_{\text{H}^+} = (1.50 \pm 0.03) \times 10^4 \text{ M}^{-1} \text{ s}^{-1}$ rather than the $k_{\text{H}^+} = (1.50 \pm 0.03) \times 10^3 \text{ M}^{-1} \text{ s}^{-1}$ originally reported. This correction requires reversal of our original conclusion that hydrolysis of this ortho ester does not undergo a change in