In addition, in the case under consideration, *i.e.*, with three terminal CO groups at each osmium atom in cluster **3a** (IR spectroscopic data), only structure **A** has the 18e configuration at each metal atom. However, this uniform electron distribution is by no means a necessary condition for stability of triangular clusters.

The final structure of **3a** could be elucidated by X-ray diffraction analysis, however, we did not succeed in growing an appropriate crystal of this compound. To overcome this difficulty we synthesized cluster **3b** by reaction of 2 with alkyne $Me_3SiC{\equiv}CBu^n$ (**1b**). The spectroscopic similarity of **3a** and **3b** suggests that these complexes are identical in structure.

As shown by an X-ray diffraction study, molecule **3b** (space group $P2_12_12_1$, $Z = 4$, 2717 observed reflections, $R = 0.0448$) is composed of the Os_3 triangle with three terminal CO groups coordinatively bound to each metal atom. The organic ligand formed by alkyne-vinylidene coupling is linked to the Os_3 framework following the **A** pattern.

Metallacyclobutenes are known to be intermediates in reactions of carbene complexes with alkynes.[3] Therefore, the formation of type **A** clusters in the reaction under study indicates that the coupling of two hydrocarbon species may be preceded by alkyne-vinylidene rearrangement of the second alkyne molecule in the metal atom coordination sphere.

The synthesis, structure, and stereodynamic behavior of **3a** and **3b** will be detailed in the next publication.

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References

1. A. A. Koridze, N. M. Astakhova, P. V. Petrovskii, F. M. Dolgushin, A. I. Yanovsky, and Yu. T. Struchkov, *J. Organomet. Chem.*, 1994, **481**, 247.
2. D. Nucciarone, N. J. Taylor, A. J. Carty, A. Tiripicchio, M. Tiripicchio Camellini, and E. Sappa, *Organometallics*, 1988, 7, 118.
3. M. F. Semmelhack, R. Tamura, W. Schnatter, and J. Springer. *J. Am. Chem. Soc.*, 1984, **106**, 5363.

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Transformations of β-D-xylofuranosyl nucleosides. The effective synthesis of 2′,3′-dideoxy-2′,3′-didehydrothymidine

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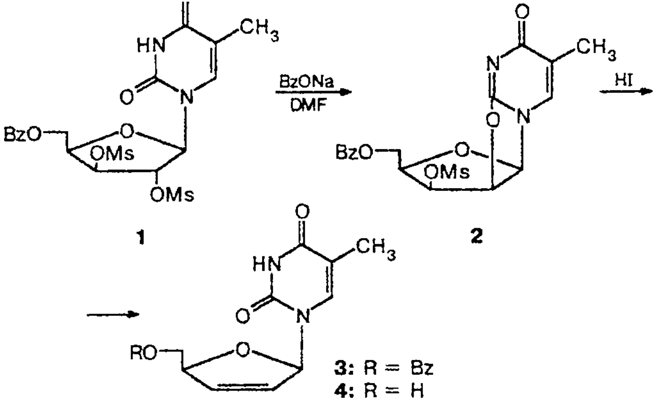
It is known that certain 2′,3′-didehydronucleosides possess high antiviral activity.[1,2] We showed earlier that heating 1-(2′-*O*-tosyl-3′,5′-di-*O*-benzoyl-β-D-xylofuranosyl)thymine with NaI leads to a mixture of 2,2′-anhydronucleoside and 5′-*O*-benzoyl-2′,3′-dideoxy-2′,3′-didehydrothymidine (in 30% yield).[3]

We continued the search for efficient methods of introducing a double bond at position 2′,3′ of the sugar moiety and found (Scheme 1) that the reaction of 2,2′-anhydro compound **2** with HI in anhydrous 1,2-dimethoxyethane at 20 °C for 8 h leads to 2′,3′-didehydronucleoside **3** in 93% yield.

The protecting group in compound **3** was removed with MeONa in methanol. The yield of 2,3′-dideoxy-2′,3′-didehydrothymidine (**4**) was 87%.

Scheme 1



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Compound **1** was prepared according to the procedure given in Ref 4. Heating compound **1** (2.4 g) with BzONa (1.55 g) in DMF (120 ml) gave 2,2′-anhydro-1-(5-*O*-benzoyl-3-*O*-methanesulfonyl-β-D-lyxofuranosyl)thymine (**2**, 1.61 g, 82%), m.p. 230—232 °C (acetone), $[\alpha]_D^{26}$ +47.3° (*c* 0.55, CH_3CN). UV spectrum (CH_3CN), λ_{max}/nm: 229.2 (ε 17460), 248 (ε 8320); λ_{min} 244 nm. ^1H NMR spectrum (acetone-d_6, δ, ppm, *J*/Hz): 3.26 (s, 3 H, CH_3); 1.70 (s, 3 H, CH_3); 6.25 (d, 1 H, H-1′, $J_{1',2'}$ = 5.84); 5.71 (dd, 1 H, H-2′, $J_{2',3'}$ = 5.68); 5.60 (dd, 1 H, H-3′, $J_{3',4'}$ = 5.18); 4.74 (m, 1 H, H-4′); 4.39 (dd, 1 H, H-5a′, $J_{5a',4'}$ = 4.91); 4.32 (dd, 1 H, H-5b′, $J_{5b',4'}$ = 7.73, J_{hem} = −11.85); 7.34—7.95 (m, 5 H, ArH); 7.88 (s, 1 H, H-6). Found (%): C, 50.94; H, 3.81; N, 6.33; S, 7.16. $C_{18}H_{18}N_2O_8S$. Calculated (%): C, 51.18; H, 4.27; N, 6.64; S, 7.58.

Work on the application of this reaction to β-D-xylofuranosyl nucleosides with the other nucleic acid bases is in progress.

References

1. M. Baba, R. Pauwels, P. Herdewijn, E. De Clerq, J. Deasmyter, and M. Wanderputte, *Biochem. Biophys. Res. Commun.*, 1987, **142**, 128.
2. H. Mitsuya and S. Broder, *Proc. Natl. Acad. Sci. U.S.A*, 1986, **83**, 1911.
3. G. A. Tolstikov, A. G. Mustafin, R. R. Gataullin, L. V. Spirikhin, V. S. Sultanova, and I. B. Abdrakhmanov, *Izv. AN, Ser. khim.*, 1993, 1137 [*Russ. Chem. Bull.*, 1993, **42**, 1095 (Engl. Transl.)].
4. R. R. Gataullin, A. G. Mustafin, I. B. Abdrakhmanov, and G. A. Tolstikov, Pat. RF No. 205 2465, *Byull. izobret.*, 1996, No. 2 (in Russian).

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