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The authors congratulate Academician I.L. Eremenko with a 70th birthday

New Gallium Hydrides with Dianionic Acenaphthene-1,2-Diimine Ligands

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Abstract—Heating of a solution of [(Dpp-bian)GaH_2] (**I**) (Dpp-bian = 1,2-bis[(2,6-diisopropylphenyl)imino]acenaphthene) in pyridine leads to elimination of a hydrogen atom and reduction of the diimine ligand to dianion, resulting in the complex [(Dpp-bian)GaH(Py)] (**II**). The reaction of **I** with 1,3-diisopropyl-4,5-dimethylimidazol-2-ylidene (i-Pr_2Im^{Me}) is accompanied by a similar process, with the reaction product being identified as [(Dpp-bian)GaH(i-Pr_2Im^{Me})] (**III**). Diamagnetic derivatives **II** and **III** were characterized by ^1H NMR spectroscopy. The molecular structure of compound **II** was established by X-ray diffraction (CIF file CCDC no. 1973549).

Keywords: gallium, hydrides, redox active ligands, diimine ligands, synthesis, molecular structure

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Compounds with the Ga–H bonds frequently occur in the chemistry of Group 13 metal hydrides [1–4]. These compounds cause considerable interest for both practical applications and fundamental research. For example, volatile compounds based on gallium trihydride are potential precursors for the fabrication of GaN and GaAs films and particles in MOCVD processes [2, 5–8]. In addition, dichlorogallane Cl_2GaH [9–11] and dialkylgallanes R_2GaH [12–14] react with unsaturated substrates (ketones, nitriles, olefins, and alkynes) to give hydrohalogenation products [4, 10, 12, 15–29]. The hydrometallation of substituted alkynes with dichloro- or dialkylgallanes was studied in sufficient detail and the diversity of reaction pathways and gallium-containing products formed was demonstrated [4, 17–29]. It is noteworthy that hydrogallation reactions are, in some cases, more selective than hydroalumination reactions [3, 4]. An important class of gallium hydrides are neutral complexes in which the Ga–H group is chelated by *N*,*N*-bidentate ligands [30–38]. For instance, the diketiminate derivative [(Dpp-Nacnac)GaH(t-Bu)] (Dpp = 2,6-i-$Pr_2C_6H_3$) was shown to be applicable as a catalyst for CO_2 reduction to HC(O)OBpin (Pin = $OCMe_2CMe_2O$) or to MeOBpin, with excess HBPin (4,4,5,5-tetramethyl-1,3,2-dioxaborolane) [37]. Thus, *N*,*N*-bidentate ligands can expand the scope of applicability of gallium hydrides, providing the development of molecular systems alternative to *d*-element-based catalysts.

We found that main group metal complexes with redox-active nitrogen-containing *N*,*N*-chelating ligands show unusual reactivity towards various classes of compounds [39–46]. For example, digallane [(Dpp-bian)Ga–Ga(Dpp-bian)] (Dpp-bian = 1,2-bis[(2,6-diisopropylphenyl)imino]acenaphthene) exhibits a dual electron transfer in reactions with some oxidants: depending on the substrate, electrons are transferred to the substrate either from the ligands or from the metal–metal bond [42, 43]. Like transition metals, digallane is capable of two-electron oxidative addition of organic halides [47]. Also, gallium and aluminum complexes with the Dpp-bian dianion reversibly add alkynes and enones [39, 40, 44, 48]. In these reactions, the substrate forms new bonds both with the metal atom and the redox-active ligand. It is evident that the use of redox-active ligands in the chemistry of metal hydrides may produce metal complexes with a reactivity unusual for hydrides. This opens prospects for the development of efficient, accessible, and relatively nontoxic gallium hydride reagents or catalysts for reduction and isomerization of, for example, unsaturated substrates.

Recently, we prepared and characterized new monomeric gallium hydride derivatives, [(Dpp-bian)GaH(R)] (R = Cl, H, $OSi(Ph)H_2$), containing the redox-active Dpp-bian ligand. Despite the fact that these compounds proved to be inert towards some alkynes and alkenes, the implemented benzaldehyde hydrosilylation with phenylsilane in the presence of catalytic amounts of the dihydride [(Dpp-bian)GaH$_2$] implies the possibility of insertion of substrates with activated multiple bonds into the Ga–H moiety [49].

This communication describes the synthesis, molecular structures, and spectral characteristics of new gallium hydrides with acenaphthene-1,2-diimine ligands, [(Dpp-bian)GaH(Py)] (**II**) and [(Dpp-bian)GaH(*i*-Pr$_2$Im^{Me})] (**III**).

EXPERIMENTAL

The compound [(Dpp-bian)GaH$_2$] (**I**) [49] was synthesized by a known procedure. Compounds **I**–**III** are sensitive to oxygen and moisture; therefore, all manipulations on their synthesis, isolation, and identification were performed in vacuo either using Schlenk technique or nitrogen atmosphere (Glovebox M. Braun). Toluene and benzene (Aldrich) were dried and stored over sodium benzophenone, pyridine (Aldrich) was dried with sodium and collected by condensation in vacuo immediately before use. Deuteropyridine (Aldrich) was dried with sodium, and deuterobenzene (Aldrich) was dried with sodium benzophenone. The solvents were collected by condensation in vacuo into NMR tubes containing samples of compounds.

IR spectra were measured on an FSM-1201 spectrometer in the 3998–449 cm^{-1} range (mineral oil mulls). The ^{1}H NMR spectra were recorded on Bruker DPX-200 (200 MHz) and Bruker Avance III (400 MHz) spectrometers. Elemental analysis was carried out by burning samples in oxygen using the Pregl method.

Synthesis of [(Dpp-bian)GaH(Py)] (II). A solution of compound **I** (0.73 g, 1.27 mmol) in pyridine (20 mL) was heated for 4 h at 160°C. After replacement of pyridine by toluene, the resulting blue solution was concentrated to 10 mL volume. Blue crystals of compound **II** were formed in 24 h at 10°C. The yield was 0.73 g (77%).

^{1}H NMR (200 MHz; C_5D_5N; 298 K; δ, ppm; *J*, Hz): 7.42–7.22 (m, 9H), 7.22–7.10 (m, 4H), 7.00–6.88 (m, 2H), 6.23 (d, 2H, *J* = 6.8), 3.72 (sept., 4H, *J* = 6.8), 1.13 (d, 12H, *J* = 6.8), 0.88 (d, 12H, *J* = 6.8).

IR (ν, cm^{-1}): 1933 s, 1610 m, 1593 w, 1574 w, 1514 s, 1364 w, 1332 w, 1316 w, 1251 m, 1211 w, 1178 w, 1158 w, 1136 w, 1105 w, 1081 w, 1065 m, 1054 w, 1045 m, 1015 w, 997 w, 997 w, 968 w, 925 s, 903 m, 813 m, 806 m, 767 s, 734 s, 695 s, 663 m, 636 m, 624 m, 544 w, 534 w, 522 w, 465 m.

For $C_{48}H_{54}N_3Ga$

Anal. calcd., %	C, 77.63	H, 7.33
Found, %	C, 77.21	H, 7.09

Synthesis of [(Dpp-bian)GaH(*i*-Pr$_2$Im^{Me})] (III). *Method A.* 1,3-Diisopropyl-4,5-dimethylimidazol-2-ylidene (*i*-Pr$_2$Im^{Me}) (0.12 g, 0.69 mmol) was added to a solution of compound **I** (0.40 g, 0.69 mmol) in benzene (20 mL). The mixture was stirred for 15 min at 50°C and cooled to room temperature. The solvent was removed, and the remaining finely crystalline precipitate of compound **III** was washed with hexane (5 mL) and dried in vacuo. The yield was 0.39 g (75%).

Method B. *i*-Pr$_2$Im^{Me} (0.004 g, 0.02 mmol) was added to a solution of compound **II** (0.02 g, 0.02 mmol) in an NMR tube. The mixture was stirred for 5 min. The ^{1}H NMR spectrum corresponds to the product obtained by method A.

^{1}H NMR (400 MHz; C_6D_6; 298 K; δ, ppm; *J*, Hz): 7.37–7.25 (m., 6H), 7.07 (d, 2H, *J* = 6.7), 6.89 (t, 2H), 6.40 (d, 2H, *J* = 6.7), 5.42 (sept., 2H, *J* = 7.0), 4.23 (sept., 2H, *J* = 6.7), 3.67 (sept., 2H, *J* = 6.7), 1.48 (d, 6H, *J* = 6.7), 1.46 (s, 6H), 1.34 (d, 6H, *J* = 6.7), 1.21 (d, 6H, *J* = 6.7), 0.97 (d, 12H, *J* = 7.0), 0.88 (d, 6H, *J* = 6.7).

IR (ν, cm^{-1}): 1860 s, 1614 w, 1589 w, 1511 s, 1335 m, 1312 w, 1256 m, 1208 w, 1179 m, 1133 w, 1109 m, 1076 w, 1056 w, 999 w, 936 w, 924 m, 893 w, 810 s, 762 s, 722 m, 683 w, 634 m, 622 w, 608 w, 548 m, 517 w, 497 s.

For $C_{47}H_{61}N_4Ga$

Anal. calcd., %	C, 75.09	H, 8.18
Found, %	C, 75.03	H, 8.15

X-ray diffraction study of II. Crystals of compound **II** suitable for X-ray diffraction were prepared by crystallization from toluene. The crystal chosen for analysis was coated by mineral oil (Aldrich), mounted on a glass capillary, and placed into a cold nitrogen flow of a Bruker D8 Quest diffractometer. The experimental sets of intensities (MoK_α radiation, λ = 0.71073 Å, ω- and φ-scan modes) were integrated using the SAINT program package [50]. The absorption corrections were applied using the SADABS program [51]. The structure was solved by the dual-space method using the SHELXT program [52]. The nonhydrogen atoms were refined by the full-matrix least squares method on F^2_{hkl} in the anisotropic approximation with SHELXTL program packages [53]. The hydrogen atoms were placed into geometrically calculated positions and refined in the riding model, except for the hydrogen atoms at gallium, which were found from the electron density difference maps and refined isotropically. The asymmetric part of the crystal cell of **II** was found to contain a toluene solvent molecule in a general position. The key crystallographic characteristics and X-ray diffraction experiment details for compound **II** are summarized in Table 1; selected bond lengths and bond angles are presented in Table 2.

Table 1. Crystallographic data and X-ray experiment and structure refinement details for structure **II**

Parameter	Value
M	742.66
System	Monoclinic
Space group	$P2_1/n$
Temperature, K	100(2)
a, Å	10.5769(3)
b, Å	25.2796(6)
c, Å	15.4536(4)
β, deg	104.613(1)
V, Å^3	3998.32(18)
Z	4
ρ(calcd.), g/cm^3	1.234
μ, mm^{-1}	0.725
F(000)	1576
Crystal size, mm	0.27 × 0.17 × 0.04
θ Range, deg	2.11–28.00
Ranges of indices	$-13 \le h \le 13$, $-33 \le k \le 33$, $-20 \le l \le 18$
Number of measured reflections	32870
Number of unique reflections (R_{int})	9633 (0.0346)
Number of reflections with $I > 2\sigma(I)$	8126
Absorption corrections (max/min)	0.9144/0.7898
Data/constraints/parameters	9633/0/482
GOOF	1.023
R_1, wR_2 ($I > 2\sigma(I)$)	0.0322, 0.0742
R_1, wR_2 (for all reflections)	0.0427, 0.0773
$\Delta\rho_{max}/\Delta\rho_{min}$, e Å^{-3}	0.402/–0.427

The structure of **II** was deposited with the Cambridge Crystallographic Data Centre (CCDC no. 1973549; ccdc.cam.ac.uk/getstructures).

RESULTS AND DISCUSSION

Our studies in the field of Group 13 metal hydrides with redox-active ligands demonstrated that aluminum hydrides can be stabilized by both the Dpp-bian dianion and the corresponding radical anion [54–56], whereas all of the obtained gallium derivatives are stabilized only by the radical anion ligand [49].

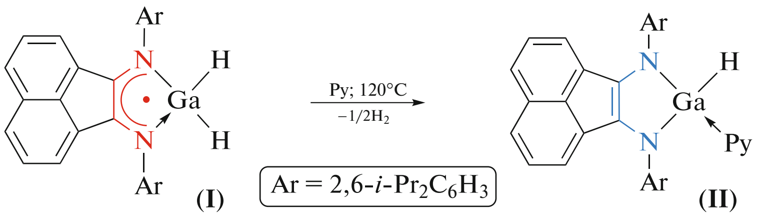
The synthesis of aluminum hydride with the Dpp-bian dianion by exchange reaction of [(Dpp-bian)Na_2] with $HAlCl_2$ gives the complex [(Dpp-bian)AlH(THF)] [55], whereas a similar reaction of [(Dpp-bian)Na_2] with $HGaCl_2$ affords dianionic [(Dpp-bian)Ga–Ga(Dpp-bian)] [57, 58]. Digallane is also formed in the reaction of the mixed halogen hydride complex [(Dpp-bian)Ga(H)Cl] with sodium metal, which attests to instability of dianionic gallium monohydrate. Nevertheless, previously we found that

Table 2. Selected bond lengths (Å) and bond angles (deg) in compound **II**

II	
Bond	d, Å
Ga–N(1)	1.8930(11)
Ga–N(2)	1.9022(12)
Ga–N(3)	2.0535(12)
Ga–H(1)	1.460(19)
N(1)–C(1)	1.3850(18)
N(2)–C(2)	1.3991(17)
C(1)–C(2)	1.3750(19)
Angle	ω, deg
N(1)GaN(2)	90.25(5)
N(1)GaN(3)	106.88(5)
N(1)GaH(1)	124.8(7)
N(2)GaH(1)	127.6(8)
N(3)GaH(1)	102.1(8)
N(2)GaN(3)	102.55(5)

gallium halogen derivatives with dianionic diimine ligands [(Dpp-bian)Ga(I)Py] [42] and [(Dpp-bian)Ga(Cl)Py] [47] can be stabilized by a strong Lewis base such as pyridine. The gallium monohydride with the diazadiene ligand, [{((Dpp)NCH)$_2$}-GaH(Mes$_2$Im^H)] (Mes$_2$Im^H = 1,3-mesitylimidazol-2-ylidene) [32], also contains an electron-donor carbene molecule, which prompts that the dianionic acenaphthenediimine gallium hydride complex can be prepared in the same way.

Indeed, heating of a solution of compound **I** in pyridine results in elimination of one hydrogen atom and formation of [(Dpp-bian)GaH(Py)] **(II)** (Scheme 1). Complex **II** was isolated as blue crystals in 77% yield.

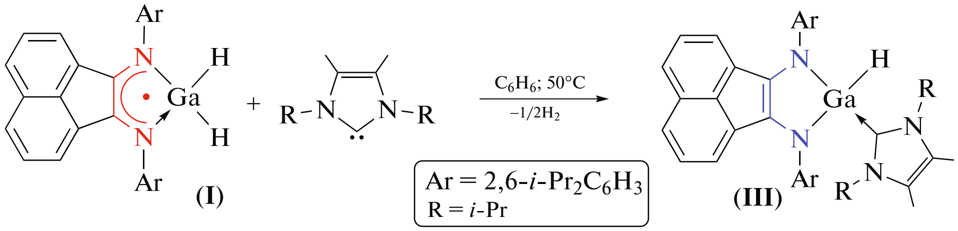


Scheme 1.

In the IR spectrum of complex **II**, the Ga–H vibrations are manifested as a strong band at 1933 cm^{-1}. This value is rather close to those for compounds **I** (1897 and 1872 cm^{-1}) and [(Dpp-bian)GaH(Cl)] (1939 cm^{-1}) [49], but differs from the value for diazadiene hydride [{((Dpp)NCH)$_2$}GaH(Mes$_2$Im^H)] (1854 cm^{-1}) [32].

The ^{1}H NMR spectrum of compound **II** (Fig. 1a) in deuteropyridine exhibits signals for the methyl protons of the 2,6-*i*-Pr$_2$-C$_6$H$_3$ substituents (doublets at 0.88 ppm (12H) and 1.13 ppm (12H)) and a septet for methine protons (3.72 ppm (4H)) of the same substituents. The doublet at 6.23 ppm (2H) and multiplets in the ranges of 6.88–7.00 ppm (2H), 7.10–7.22 ppm (4H), and 7.22–7.42 ppm (9H) refer to the aromatic protons of the naphthalene moiety, phenyl substituents at the nitrogen atoms, and coordinated pyridine.

A similar situation occurs for the reaction of compound **I** with the carbene *i*-Pr$_2$Im^{Me} (Scheme 2). The reaction proceeds in benzene at 50°C for 15 min; it is accompanied by a color change from brown to green and affords [(Dpp-bian)GaH(*i*-Pr$_2$Im^{Me})] **(III)**. The IR spectrum of complex **III** shows the Ga–H vibrations as a strong band at 1860 cm^{-1}. This value is close to the corresponding value for [{((Dpp)NCH)$_2$}-GaH(Mes$_2$Im^H)] (1854 cm^{-1}) [32], but markedly differs from that for **II** (1933 cm^{-1}) and for the aluminum hydride [(Dpp-bian)AlH(*i*-Pr$_2$Im^{Me})] (1732 cm^{-1}) [59].



Scheme 2.

The ^{1}H NMR spectrum of compound **III** (Fig. 1b) shows signals for the unsymmetrical Dpp-bian ligand: four pairwise-equivalent methine protons of the isopropyl groups in 2,6-*i*-Pr$_2$C$_6$H$_3$ substituents are manifested as septets at 4.23 ppm (2H) and 3.67 ppm (2H). The methyl protons of the isopropyl substituents produce four doublets (ppm): 1.48 (6H), 1.34 (6H), 1.21 (6H), and 0.88 (6H). The septet and doublet for *i*-Pr protons and the signals for methyl protons of the carbene ligand occur at 5.42 ppm (2H), 0.97 ppm (12H), and 1.46 ppm (6H), respectively. The aromatic protons give rise to two doublets at 7.07 ppm (2H) and 6.40 ppm (2H), a pseudo-triplet at 6.89 ppm (2H), and a multiplet in the 7.37–7.25 ppm range (6H).

The complex [(Dpp-bian)AlH(*i*-Pr$_2$Im^{Me})], similar to compound **III**, has been prepared by the reaction of [(Dpp-bian)AlH(THF)] with imidazol-2-ylidene in which the coordinated THF molecule is replaced by carbene [59]. The reaction of compound **II** with *i*-Pr$_2$Im^{Me} carried out in an NMR tube is accompanied by a change in the solution color from blue to green, the resulting spectrum is similar to that described above, indicating replacement of the pyridine molecule by the NHC ligand to give complex **III**

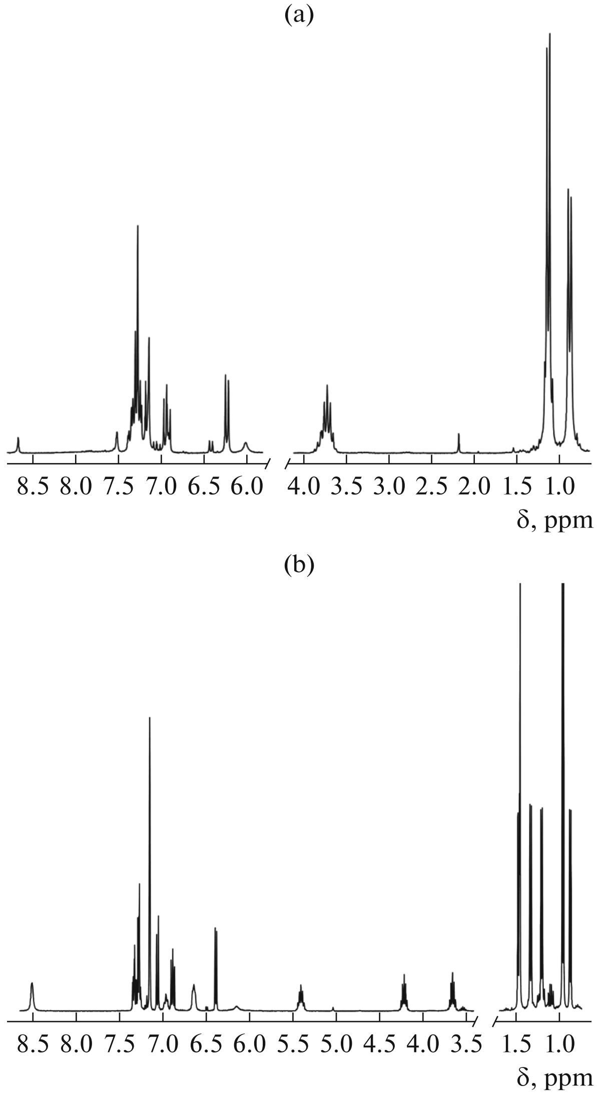


Fig. 1. ^{1}H NMR spectrum of compounds (a) **II** (200 MHz, C_5D_5N, 298 K) and (b) **III** (400 MHz, C_6D_6, 298 K).

and free pyridine (8.53 ppm (2H), 6.98 ppm (1H), 6.66 ppm (2H)) (Fig. 1b).

According to X-ray diffraction data, compound **II** is a monomeric four-coordinate gallium complex (Fig. 2). Analysis of the bond lengths in the metallacycle of **II** attests to the dianionic state of the Dpp-bian ligand. The N(1)–C(1) (1.3850(18) Å) and N(2)–C(2) (1.3991(17) Å) bond lengths are elongated compared with the corresponding bonds in complexes **I** (N(1)–C(1), 1.3238(18); N(2)–C(2), 1.3291(18) Å) and [(Dpp-bian)Ga(H)Cl] (N(1)–C(1), 1.327(3); N(2)–C(2), 1.326(3) Å) with the Dpp-bian radical anion [49]. On the other hand, the Ga–N distances in complex **II** (1.8930(11) and 1.9022(12) Å) are much shorter than the Ga–N distances in gallium hydrides (Ga–N(1), 1.9825(12); Ga–N(2), 1.9968(12) Å for **I**; Ga–N(1), 1.9581(17); Ga–N(2), 1.9519(17) Å for [(Dpp-bian)Ga(H)Cl]), which is indicative of stronger binding of gallium to the Dpp-bian dianion compared with the radical anion. The Ga–H bond length in **II** (1.460(19) Å) is close to the corresponding distances in hydride **I** (1.53(2) and 1.47(2) Å) and compound [{((Dpp)NCH)$_2$}GaH(Mes$_2$Im^H)] (1.498(16) Å) [32].

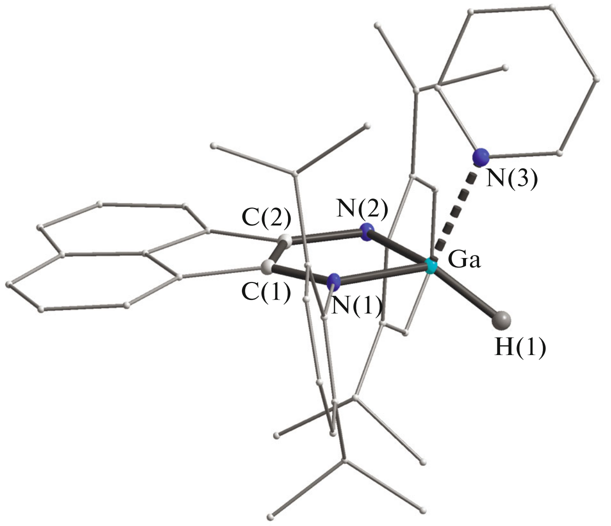


Fig. 2. Molecular structure of **II**. Thermal ellipsoids are given at the 30% probability level. The hydrogen atoms, except H(1), are omitted.

Thus, we prepared and characterized new monomeric gallium hydrides [(Dpp-bian)GaH(Py)] (**II**) and [(Dpp-bian)GaH(*i*-Pr$_2$Im^{Me})] (**III**) containing the redox-active Dpp-bian ligand in the dianionic form. Gallium hydrides that we obtained previously proved to be inert towards some unsaturated compounds; however, the reductive elimination of hydrogen atom on treatment with donor molecules such as pyridine and imidazol-2-ylidene can apparently be used for hydrogenation of such substrates. It was demonstrated that the dianionic gallium monohydride can be stabilized by a strong Lewis base. In the near future, we plan to study the reactivity of new complexes **II** and **III** towards some organic compounds and small molecules such as CO and CO_2. Thus we will be able to determine the applicability of these derivatives as catalysts for organic synthesis and to expand the fundamental views on the chemistry of metal complexes with redox-active ligands.

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CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

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