tion was applied by the program XABS based on F_o and F_c differences. (H. Hope, B. Moezzi, Chemistry Department, University of California, Davis), $\mu = 1.477\ mm^{-1}$, max./min. transmission = 0.76/0.61, structure was solved by direct method using SHELXL-93, and refined by a full-matrix least-squares method based on F^2 with 429 parameters, hydrogen atoms were located or added in calculated positions, and refined by using the riding model, $R = 0.0339$; $wR = 0.0944$, largest difference peak = 0.480 e $Å^{-3}$. Crystallographic data (excluding structure factors) for the structure reported in this paper have been deposited with Cambridge Crystallographic Data Centre as supplementary publication no. CCDC-179-157. Copies of the data can be obtained free of charge on application to The Director, CCDC, 12 Union Road, GB-Cambridge CB21EZ UK (fax: int. code +(1223)336-033; e-mail: deposit@chemcrys.cam.ac.uk).

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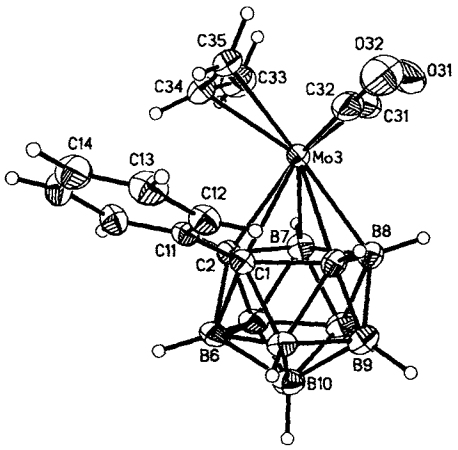

Figure 1. Perspective view of $\mathbf{1}^-$. Selected bond lengths [Å] and angles [°]: C1–C2 1.616(4), C1–C11 1.509(4), Mo3–C1 2.438(3), Mo3–C2 2.418(3), Mo3–C31 1.914(4), Mo3–C32 1.918(4), Mo3–C33 2.335(4), Mo3–C34 2.234(4), Mo3–C35 2.379(3), C31–O31 1.161(4), C32–O32 1.163(4); C31-Mo3-C32 79.4(2).

Isolation of a Nonicosahedral Intermediate in the Isomerization of an Icosahedral Metallacarborane**

Shirley Dunn, Georgina M. Rosair, Rhodri Ll. Thomas, Andrew S. Weller, and Alan J. Welch*

The mechanism by which the icosahedral carboranes, their C-substituted derivatives, and their heteroatom analogues rearrange has been the subject of speculation since the 1,2- to 1,7-isomerization of *closo*-$C_2B_{10}H_{12}$ was reported more than 30 years ago.[1] A number of possible mechanisms have been proposed[2] on the basis of both experimental observations (product distributions) and theoretical calculations. As far as we are aware, however, the high temperatures generally required for these isomerizations have so far precluded isolation of any intermediate species; the identification of such intermediates is clearly important for judging the relative merits of the various mechanistic proposals.

We have recently shown that targeting molecules that are severely overcrowded can dramatically lower isomerization temperatures in icosahedral metallacarboranes.[3] We now report the unexpected first isolation and characterization of an intermediate in an icosahedral-to-icosahedral rearrangement. This intermediate, which is closed but not icosahedral in shape, has previously only been identified theoretically.[2f]

Reaction of Na_2[7-Ph-7,8-*nido*-$C_2B_9H_{10}$] and [$(CH_3CN)_2$-$MoBr(\eta^3$-$C_3H_5)(CO)_2$] in THF at 0 °C affords the metallacarborane $\mathbf{1}^-$ as a benzyltrimethylammonium ($BTMA^+$) salt in good yield. The anion $\mathbf{1}^-$ exhibits two bands in the carbonyl region of the IR spectrum at 1924 and 1833 cm^{-1} (CH_2Cl_2). Crystallographic analysis[4] confirms that $\mathbf{1}^-$ has the expected icosahedral structure with a 3,1,2-MoC_2 heteroatom arrangement (Figure 1).

[1-Ph-3,3-$(CO)_2$-3-(η^3-C_3H_5)-3,1,2-*closo*-$MoC_2B_9H_{10}$]$^-$ $\mathbf{1}^-$

[$Ph_2(CO)_2(\eta^3$-$C_3H_5)MoC_2B_9H_9$]$^-$ $\mathbf{2}^-$

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An analogous product is not obtained from the diphenyl carborane anion. Rather, treatment of Na_2[7,8-Ph_2-7,8-*nido*-$C_2B_9H_9$] with the same metal substrate under the same conditions yields $\mathbf{2}^-$, which shows IR stretching bands at significantly higher frequencies (1954 and 1893 cm^{-1}). A crystallographic study[5] of the $BTMA^+$ salt of $\mathbf{2}^-$ reveals a nonicosahedral *closo* geometry with two 4-connected cluster vertices (occupied by carbon atoms) and two 6-connected vertices (one occupied by Mo, Figure 2). There is only one Mo–C (cluster) connectivity, and it is significant that the two cage carbon atoms are substantially separated.

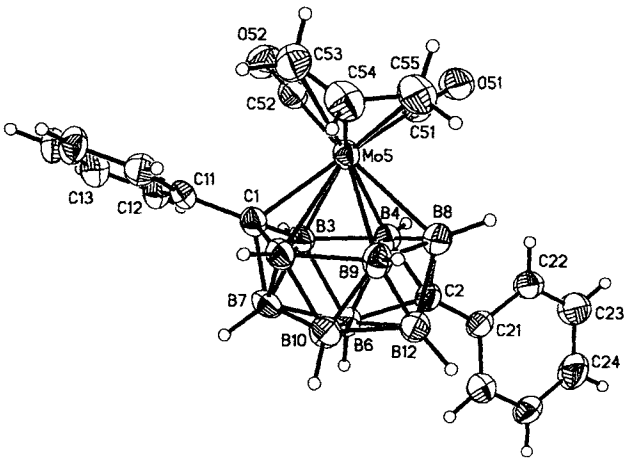


Figure 2. Perspective view of $\mathbf{2}^-$. Selected bond lengths [Å] and angles [°]: C1–C11 1.495(10), C2–C21 1.478(10), Mo5–C1 2.260(7), Mo5–C51 1.950(8), Mo5–C52 1.986(9), Mo5–C53 2.395(9), Mo5–C54 2.248(9), Mo5–C55 2.338(9), C51–O51 1.155(9), C52–O52 1.150(9); C51-Mo5-C52 79.4(3).

The ^1H NMR spectrum of $\mathbf{2}^-$ in CD_3CN slowly changes at room temperature (over days). This suggests that $\mathbf{2}^-$ is a kinetic rather than a thermodynamic product, and identifies it as a potential intermediate in a carbon atom isomerization process. This is confirmed by the quantitative conversion of $\mathbf{2}^-$ to $\mathbf{3}^-$ upon heating in THF at reflux (30 min); the product $Et_4N^+\mathbf{3}^-$ shows carbonyl IR bands (1920 and 1831 cm^{-1}) that are very similar to those of $BTMA^+\mathbf{1}^-$. A single crystal X-ray

[1,9-Ph_2-3,3-$(CO)_2$-3-(η^3-C_3H_5)-3,1,9-*closo*-$MoC_2B_9H_9$]$^-$ $\mathbf{3}^-$

diffraction study[6] of $Et_4N^+\mathbf{3}^-$ (Figure 3) shows that this anion also has an icosahedral geometry, but this time with a 3,1,9-MoC_2 arrangement.

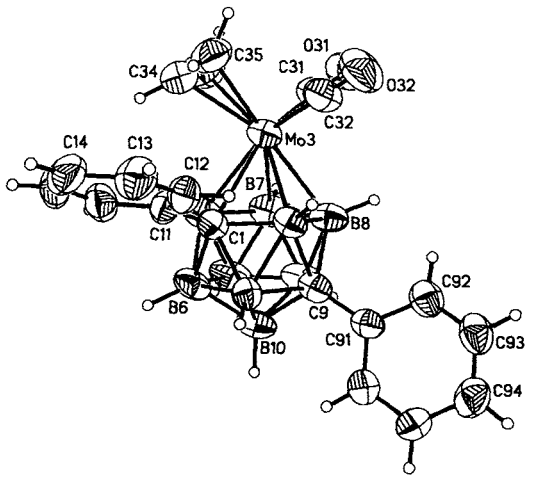
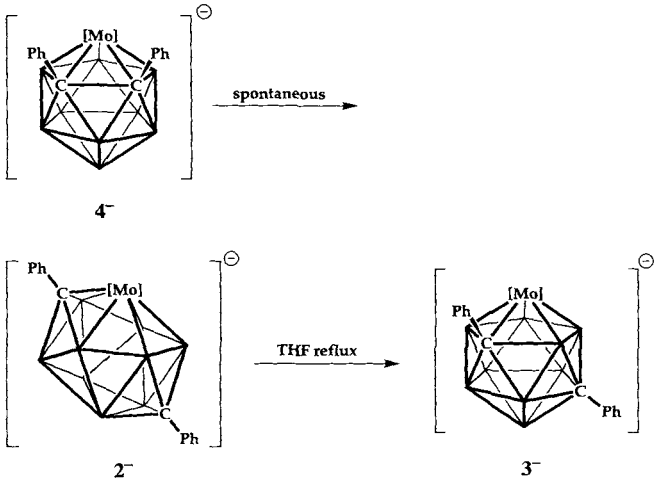


Figure 3. Perspective view of $\mathbf{3}^-$. Selected bond lengths [Å] and angles [°]: C1–C11 1.508(14), C9–C91 1.483(14), Mo3–C1 2.459(8), Mo3–C31 1.948(13), Mo3–C32 1.957(14), Mo3–C33 2.36(2), Mo3–C34 2.252(14), Mo3–C35 2.36(2), C31–O31 1.148(14), C32–O32 1.14(2); C31-Mo3-C32 79.6(5).

Collectively, these results are entirely consistent with the spontaneous conversion of the nonisolable metallacarborane $\mathbf{4}^-$ into nonicosahedral $\mathbf{2}^-$ to relieve steric crowding (Scheme 1).

$[1,2\text{-}Ph_2\text{-}3\text{-}(\eta^3\text{-}C_3H_5)\text{-}3,3\text{-}(CO)_2\text{-}3,1,2\text{-}closo\text{-}MoC_2B_9H_9]^-$ $\mathbf{4}^-$

Gentle thermolysis of $\mathbf{2}^-$ then affords the icosahedral $\mathbf{3}^-$, which is related to $\mathbf{4}^-$ in exactly the same way that 1,7-*closo*-$C_2B_{10}H_{12}$ is related to 1,2-*closo*-$C_2B_{10}H_{12}$.



Scheme 1. Proposed isomerization of the sterically overcrowded icosahedral anion $\mathbf{4}^-$ into $\mathbf{3}^-$ via the nonicosahedral intermediate $\mathbf{2}^-$.

Thus $\mathbf{2}^-$ is an intermediate in an icosahedral-to-icosahedral rearrangement. The nonicosahedral structure of $\mathbf{2}^-$ is experimentally unprecedented, but the analogous carborane (with $\{Mo(\eta^3\text{-}C_3H_5)(CO)_2\}^-$ replaced by $\{BH\}$) of C_2 symmetry has been identified by Wales[2f] as a stable intermediate in the 1,2- to 1,7-$C_2B_{10}H_{12}$ isomerization and a viable synthetic target. It is possible that $\mathbf{2}^-$ is isolable, hence realizing this prediction, by virtue of stabilization of the metal center in a highly-connected cluster vertex.

Experimental Section

$BTMA^+\mathbf{1}^-$: $Na_2[7\text{-}Ph\text{-}7,8\text{-}nido\text{-}C_2B_9H_{10}]$ (0.6 mmol) in THF (30 mL) was added to a stirred solution of $[(CH_3CN)_2MoBr(\eta^3\text{-}C_3H_5)(CO)_2]$ (0.6 mmol) in THF (20 mL) at 0 °C. After removal of solvent in vacuo, the resulting brown oil was dissolved in dichloromethane (30 mL). BTMACl (0.6 mmol) was added, and NaBr that was formed in situ removed by filtration. Purification by column chromatography (silica, 200–400 mesh, water-cooled) with dichloromethane eluent followed by recrystallization from dichloromethane/(diethyl ether) afforded $BTMA^+\mathbf{1}^-$ as a yellow solid (42% yield). IR (CH_2Cl_2): $\tilde{\nu}$ = 1924, 1833 cm^{-1} (CO); ^1H NMR (200 MHz, CD_3CN): δ = 7.7–7.5 (m, 5H, $BTMA^+$), 7.2–6.9 (m, 5H, phenyl), 4.4 (s, 2H, $BTMA^+$), 3.35 (m, 1H, $allyl_{center}$), 3.0 (s, 9H, $BTMA^+$), 2.7 (dd, 1H, $allyl_{syn}$), 1.45 (dd, 1H, $allyl_{syn}$), 1.1 (d, 1H, $allyl_{anti}$), 0.7 (d, 1H, $allyl_{anti}$); $^{11}B\{^1H\}$ NMR (128.4 MHz, CD_3CN): δ = −6.4 (1B), −7.9 (1B), −10.4 (1B), −14.3 (1B), −15.8 (2B), −19.1 (1B), −29.9 (1B), −33.1 (1B).

$BTMA^+\mathbf{2}^-$: Prepared analogously to $BTMA^+$ $\mathbf{1}^-$ from $Na_2[7,8\text{-}Ph_2\text{-}7,8\text{-}nido\text{-}C_2B_9H_9]$ (1.0 mmol), $[(CH_3CN)_2MoBr(\eta^3\text{-}C_3H_5)(CO)_2]$ (1.1 mmol), and BTMACl (0.9 mmol) to afford $BTMA^+$ $\mathbf{2}^-$ as a yellow solid (54% yield). IR (CH_2Cl_2): $\tilde{\nu}$ = 1954, 1893 cm^{-1} (CO); ^1H NMR (200 MHz, CD_3CN) δ = 7.9 (m, 2H, phenyl), 7.45–7.75 (m, 5H, $BTMA^+$), 7.1–7.35 (m, 8H, phenyl), 4.1 (s, 2H, $BTMA^+$), 3.75 (m, 1H, $allyl_{center}$), 2.8 (s, 9H, $BTMA^+$), 2.15 (dd, 1H, $allyl_{syn}$), 2.05 (dd, 1H, $allyl_{syn}$), 1.4 (d, 1H, $allyl_{anti}$), 0.95 (d, 1H, $allyl_{anti}$); $^{11}B\{^1H\}$ NMR (128.4 MHz, CD_3CN): δ = 16.2 (1B), 5.9 (1B), 3.8 (1B), −0.5 (1B), −4.5 (1B), −6.5 (1B), −11.7 (1B), −22.6 (1B), −23.8 (1B).

$A^+\mathbf{3}^-$ (A^+ = $BTMA^+$, Et_4N^+, Ph_4P^+, $(Ph_3P{=}N)_2N^+$) Method 1: $BTMA^+\mathbf{2}^-$ (0.5 mmol) was heated at reflux in THF (30 mL). Purification by column chromatography (silica, 200–400 mesh, water-cooled) with dichloromethane eluent afforded $BTMA^+\mathbf{3}^-$ as a yellow solid after trituration with pentane. Method 2: anion $\mathbf{2}^-$ was prepared by allowing equimolar solutions of $Na_2[7,8\text{-}Ph_2\text{-}7,8\text{-}nido\text{-}C_2B_9H_9]$ and $[MoBr(CH_3CN)_2(\eta^3\text{-}C_3H_5)(CO)_2]$ in THF to react. Subsequent heating at reflux in THF formed $\mathbf{3}^-$. The solvent was removed in vacuo, and the residual brown oil dissolved in dichloromethane (30 mL). Addition of a salt of the counterion (1 molequiv), filtration, and purification by column chromatography (silica, 200–400 mesh, water-cooled) with dichloromethane as eluent afforded $A^+\mathbf{3}^-$ as yellow oils. Trituration with pentane provided the products as yellow solids in quantitative yields. Data for $BTMA^+$ $\mathbf{3}^-$: IR (CH_2Cl_2): $\tilde{\nu}$ = 1920, 1831 cm^{-1} (CO); ^1H NMR (200 MHz, CD_3CN): δ = 7.45–7.65 (m, 7H, $BTMA^+$ (5H), phenyl (2H)), 7.25 (m, 5H, phenyl), 6.9 (m, 3H, phenyl), 4.4 (s, 2H, $BTMA^+$), 3.0 (m, 10H, $BTMA^+$ (9H), $allyl_{center}$ (1H)), 2.6 (dd, 1H, $allyl_{syn}$), 1.8 (dd, 1H, $allyl_{syn}$), 1.2 (d, 1H, $allyl_{anti}$), 1.05 (d, 1H, $allyl_{anti}$); $^{11}B\{^1H\}$ NMR (128.4 MHz, CD_3CN): δ = −1.9 (2B), −4.5 (2B), −6.7 (1B), −9.5 (1B), −10.4 (1B), −11.6 (2B).

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[4] For all structure determinations: Siemens P4 diffractometer, 293(2) K, $Mo_{K\alpha}$ radiation, λ = 0.71073 Å, $2\theta_{max}$ = 50°, corrections for absorption (φ scans), Lorentz, and polarization effects, structure solved by direct and refined (against F^2) by full-matrix least-squares methods. Crystal data for $BTMA^+\mathbf{1}^-$: $C_{23}H_{36}B_9MoNO_2$, M_r = 551.8, crystal size 0.2 × 0.4 × 0.6 mm, monoclinic, $P2_1/c$, a = 12.8544(9), b = 23.5801(12), c = 9.2640(6) Å, β = 105.625(7)°, V = 2704.2(3) $Å^3$, Z = 4, ρ_{calcd} = 1.355 $g\,cm^{-3}$, $F(000)$ = 1136, μ = 0.51 mm^{-1}, of 4738 unique reflections 3650 were observed ($F_o > 4\sigma(F_o)$), 364 parameters, R_1 = 0.0360, wR_2 = 0.0736 (for observed data), S = 1.032, max. and min. residual electron density: 0.40 and −0.33 e $Å^{-3}$. Crystallographic data (excluding structure factors) for the structures reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC-100001. Copies of the data can be obtained free of charge on application to The Director, CCDC, 12 Union Road, Cambridge CB21EZ, UK (fax: Int. code +(1223)336-033; e-mail: deposit@chemcrys.cam.ac.uk).

[5] Crystal data for BTMA$^+$**2**$^-\cdot 2CH_2Cl_2$: $C_{31}H_{44}B_9Cl_4MoNO_2$, $M_r = 797.7$, crystal size $0.2\times0.3\times0.8$ mm, monoclinic, $P2_1/n$, $a = 11.003(2)$, $b = 26.100(2)$, $c = 14.2707(14)$ Å, $\beta = 108.646(10)°$, $V = 3883.3(8)$ Å^3, $Z = 4$, $\rho_{calcd} = 1.364$ g cm^{-3}, $F(000) = 1632$, $\mu = 0.64$ mm^{-1}, of 6771 unique reflections 3490 were observed ($F_o > 4\sigma(F_o)$), 464 parameters, $R_1 = 0.0660$, $wR_2 = 0.1371$ (for observed data), $S = 1.014$, max. and min. residual electron density: 0.55 and -0.56 e Å^{-3}. See ref. [4].

[6] Crystal data for Et_4N^+**3**$^-$: $C_{27}H_{44}B_9MoNO_2$, $M_r = 607.9$, crystal size $0.2\times0.3\times0.7$ mm, monoclinic, Cc, $a = 24.403(10)$, $b = 10.1090(10)$, $c = 17.612(3)$ Å, $\beta = 133.060(10)°$, $V = 3174.4(14)$ Å^3, $Z = 4$, $\rho_{calcd} = 1.272$ g cm^{-3}, $F(000) = 1264$, $\mu = 0.44$ mm^{-1}, of 2963 unique reflections 2240 were observed ($F_o > 4\sigma(F_o)$). 362 parameters, $R_1 = 0.0536$, $wR_2 = 0.1343$ (for observed data), $S = 1.039$, Flack parameter $= -0.3(2)$, max and min. residual electron density: 0.58 and -0.50 e Å^{-3}. See ref. [4]

The Phenylacetyl Group—The First Amino Protecting Group That Can Be Removed Enzymatically from Oligonucleotides in Solution and on a Solid Support**

Herbert Waldmann* and Armin Reidel

A central problem in the chemical synthesis of oligodeoxynucleotides on solid supports is the protection and deprotection of the amino groups of the different nucleobases.[1] Even under the strongly basic conditions required for the removal of the established blocking groups deprotection may remain incomplete,[2] and unwanted side reactions like the formation of 2,6-diaminopurines from 6-O-alkylated guanine moieties may occur.[3] In addition, new protecting groups are needed, for example, for the construction of complex and sensitive nucleopeptides,[4] nonradioactive DNA probes,[5] and aminoacyl-modified tRNAs[6] and for the preparation of matrix-bound deprotected DNA fragments.[7a] Therefore, the development of new methods for the selective protection and deprotection of the amino functions of nucleobases under alternative and mild conditions in solution and on solid supports is of particular *interest in natural product synthesis*.[1,7]

Enzymatic protecting group techniques[8] have proven their efficiency (completely selective removal under mild conditions, for example pH 7, room temperature) in particular, in the construction of sensitive multifunctional acid- and base-labile peptide conjugates like lipo-,[9] glyco-,[10,11] and phosphopeptides,[11,12] and might also open up viable alternatives to established classical chemical methods for solution- and solid-phase oligonucleotide chemistry. Furthermore, in the light of the intense recent interest in combinatorial chemistry,[13] the successful implementation of enzymatic transformations on solid supports[14] is of general relevance. The introduction of biocatalysts for this challenging task would greatly expand the toolbox of methods available for combinatorial synthesis. We now report that the enzyme-labile phenylacetyl (PhAc) group[15] can be removed from oligonucleotides with the enzyme penicillin G acylase under very mild conditions (pH 7, room temperature) both in solution and on a solid support.

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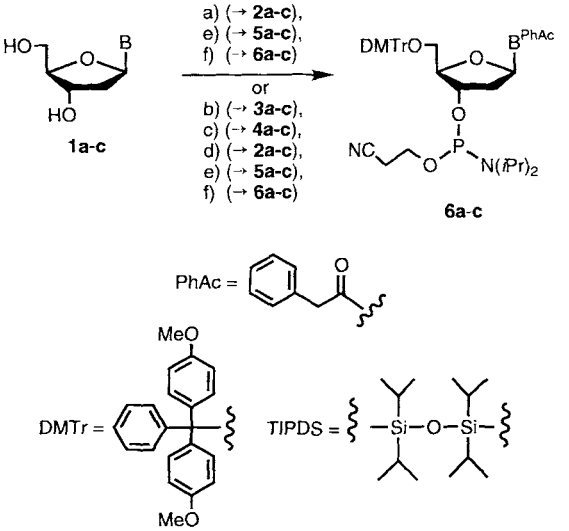
The synthesis of the selectively protected phosphoramidite building blocks **6**, which are needed for the solid-phase synthesis of the PhAc-protected oligonucleotides, is detailed in Scheme 1. The unprotected 2′-deoxynucleosides **1** were N-acylated directly by means of the "transient protection" method:[16] simultaneous O- and N-silylation was followed by treatment of the



Scheme 1. Synthesis of the PhAc-protected nucleoside phosphoramidites **6**. a) Trimethylsilyl chloride (TMSCl, 5 equiv), pyridine, room temperature, 30 min; $PhCH_2C(O)Cl$ (1.5 equiv), *N*-hydroxybenzotriazole (HOBt, 1.5 equiv), CH_3CN/pyridine 2:1, 0 °C → room temperature; yields: **2a**: 71%, **2b**: 51%; **2c**: 63%; b) 1,1,3,3-tetraisopropyldisiloxane-1,3-diyl chloride (TIPDSCl, 1.1 equiv), pyridine, room temperature; yields: **3a**; 96%, **3b**: 95%, **3c**: 97%; c) $(PhAc)_2O$ (4 equiv), pyridine, 120 °C, 30 min; yields: **4a**: 87%, **4b**: 77%, **4c**: 85%; d) $(nBu)_4NF$, (5 equiv), THF, room temperature, 1 h; yields: **2a**: 60%, **2b**: 88%; **2c**: 86%; e) 4,4′-dimethoxytriphenylmethyl chloride (DMTrCl, 1.4 equiv), 4-dimethylaminopyridine (DMAP, cat.), pyridine, room temperature, 3–5 h; yields: **5a**: 83%, **5b**: 82%, **5c**: 81%; f) $NC(CH_2)_2OP(Cl)N(iPr)_2$ (2 equiv), $(iPr)_2NEt$ (3 equiv), THF, room temperature, 20 min; yields: **6a**: 90%, **6b**: 80%, **6c**: 55%. B = adenine (**a**), guanine (**b**), cytosine (**c**).

silylated nucleosides with phenylacetyl chloride and *N*-hydroxybenzotriazole (HOBt) to deliver the desired N-PhAc nucleosides **2** in yields of 51–71%. Alternatively, the OH groups of **1a–c** were simultaneously masked with the 1,1,3,3-tetraisopropyldisiloxane-1,3-diyl (TIPDS) group.[17] The resulting TIPDS ethers **3** (obtained in 95–97% yield) were then N-acylated with phenylacetic anhydride to give the amides **4**, which were subsequently desilylated to deliver the desired N-protected nucleosides **2** in overall yields of 52–73%. After the primary OH groups of **2a–c** had been masked as DMTr ethers by alkylation with DMTrCl in the presence of DMAP, the secondary OH groups were treated with chloro(β-cyanoethoxy)-*N*,*N*-diisopropylaminophosphane to provide the desired phosphoramidites **6**. The crude products were precipitated with cold hexane and used without further purification for solid-phase synthesis of the PhAc-masked oligonucleotides **7–11** (Table 1).[18]

Table 1. Yields of oligonucleotides **7–11** produced by solid-phase synthesis [a].

pentanucleotide	d(5′-AAA AT-3′)	**7** (77)
	d(5′-GGG GT-3′)	**8** (95)
	d(5′-CCC CT-3′)	**9** (91)
dodecanucleotide	d(5′-AAT TCC GGA ATT-3′)	**10** (86)
hexadecanucleotide	d(5′-GTC ATA GCT GTT TCCT-3′)	**11** (95)

[a] All A, G, and C bases are PhAc-protected; the average yields of the couplings are given in brackets (%).

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