Methanolysis of the Cyclic Acetal Function of NanoKid Catalyzed by NanoGoblin, the Pyridinium Salt of Tetracyanocyclopentadienide

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Supporting Information

ABSTRACT: Small, “doll”-shaped tetracyanocyclopentadienide bearing a 1,3-dioxane acetal “head” and cyano “hands” and “feet” was synthesized. Its pyridinium salt, which was named NanoGoblin, exhibited catalytic activity in the methanolysis of acetals, as demonstrated by the reaction with NanoKid in methanol-\textit{d}_4, where the acetal head of NanoKid was converted to a deuterated dimethyl acetal moiety.

INTRODUCTION

Tetra- and pentacyanocyclopentadienides are conjugate bases of superacids and are stabilized by both the aromaticity of the cyclopentadienide ring and the inductive and mesomeric effects of cyano groups. 1,2 Indeed, these anions are primarily characterized by their \textit{C}_\text{5} symmetrical structure derived from the pentagonal cyclopentadienide ring and the linear cyano groups. In addition, these peripheral cyano groups can coordinate with metals through \textit{σ}-bonding because of the negative charge transfer from the cyclopentadienide ring to the cyano group. 3,4 For example, Wright reported that sodium pentacyanocyclopentadienide forms fullerene-like metal–organic frameworks in which the ligand coordinates to six Na\textsuperscript{+} cations through all five C\textequiv\text{N} groups. 5 More recently, this salt was found to be a potent open-channel blocker of the γ-aminobutyric acid receptor. 6 Following these discoveries, the interest toward the chemistry of functionalized cyclopentadienide derivatives has increased. 7,8 Indeed, we recently developed an efficient synthetic method for the preparation of tetracyanocyclopentadienides from tetracyanothiophene and a range of sulfones 9 and discovered that the sodium salts of tetracyanocyclopentadienides catalyze hydrolysis reactions in biphasic systems. 10 We also succeeded in the observation of rotamers around the C\textequiv\text{N}–O bond and the measurement of rotational barriers using 3,3′-disubstituted BINOL (1,1′-bi-2-naphthol) esters of (tetracyanocyclopentadienyl)carboxylic acid. 11

During the course of our studies on tetracyanocyclopentadienides, we employed the acetalization of aldehyde \textit{1} with 1,3-propanediol to protect the aldehyde as the cyclic acetal-substituted tetracyanocyclopentadienide \textit{2} (Scheme 1). Following the successful synthesis of \textit{2}, we attempted to exchange the sodium counter cation for a pyridinium cation using pyridine hydrochloride. However, hydrolysis of the 1,3-dioxane ring of \textit{2} occurred to afford the pyridinium salt of aldehyde \textit{3} instead of the desired product \textit{4}. This unexpected transformation likely occurred through an attack by pyridinium chloride at the acetal head of molecular doll \textit{2}, which bears cyano “hands” and “feet.” Indeed, this observation reminded us of a pioneering piece of work on anthropomorphic molecules by Tour and Chan
teur, 12,13 in which they reported the creation of a novel class of 2 nm tall molecules that resembled the human figure and were named NanoPutians. In this context, NanoKid (\textit{5}), which bears

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a simple dioxolane “head,” can serve as a precursor to other NanoPutians (Figure 1). Such compounds are of particular interest because these endearing molecular shapes\textsuperscript{14–24} attract the interest of young students who are entering into the world of organic chemistry. However, despite such interest, reactions between anthropomorphic molecules have received little attention to date. We therefore envisaged that the hydrolysis of NanoKid (5) using a pyridinium salt of acetal-substituted tetracyanocyclopentadienide could be envisaged as an “attack” on NanoKid (5) by a small doll-shaped molecule bearing a pyridinium weapon. We therefore designed the pyridinium salt of tetracyano(1,3-dioxan-5-yl)cyclopentadienide (6) (NanoGoblin), which contains a methylene acetal group rather than an arylidene acetal moiety (cf, NanoKid), thus making it more resistant to acid hydrolysis. We herein report the synthesis of this new compound, referred to as the arylidene acetal moiety (cf, NanoKid), thus making it more resistant to acid hydrolysis. We herein report the synthesis of NanoGoblin (6) and its reaction with NanoKid (5) to ultimately provide an intriguing topic for young students studying organic chemistry.

## RESULTS AND DISCUSSION

The synthesis of NanoGoblin (6) began with the construction of a 1,3-dioxane head unit through the acetalization of triol 7 with methylal (Scheme 2).\textsuperscript{25} The resulting alcohol 8 was then converted to iodide 9, and the subsequent treatment of 9 with CF$_3$SO$_2$Na in toluene at 100° C in the presence of 15-crown-5 afforded trifluoromethyl sulfone 10. Higher reaction temperatures and the replacement of toluene with dimethylformamide (DMF) decreased the yield of 10. Following the successful preparation of 10, construction of the tetracyanocyclopentadienide body was attempted, a process which required significant optimization of the reaction conditions, because the original conditions, where sulfone 10 was treated with NaH and tetracyanothiophene (11) in tetrahydrofuran (THF),\textsuperscript{6} provided the desired tetracyano(1,3-dioxan-5-yl)-cyclopentadienide (12, sodium form) in a poor 22% yield. Thus, after extensive screening of various additives [DMF, hexamethyldisilazane (HMPA), and 15-crown-5], solvents (THF and dimethyl sulfoxide), and bases (LiHMDS, n-BuLi, NaH, NaHMDS, KHMSD, and KOt-Bu), we found that the addition of HMPA under the original reaction conditions significantly promoted the formation of the desired product. However, the removal of HMPA from the product was challenging because of the strong coordination of HMPA to the sodium cation, as confirmed by NMR spectroscopy. As such, the desired product was isolated as its silver salt 12, following the cation exchange with AgNO$_3$ (52% overall yield).

Subsequently, methylene acetal 12 was armed with various aminium “weapons.” More specifically, the pyridinium, imidazolium, ammonium, and triethylammonium cations 6 and 13–15 were prepared in good yields (70–95%) by the treatment of silver salt 12 with the corresponding amine hydrochlorides in methanol at room temperature (rt), followed by the removal of the precipitated AgCl (Scheme 3).

The catalytic activities of the synthesized aminium salts 6 and 13–15 in the methanolysis of acetal 16 were then investigated in methanol-d$_4$ (CD$_3$OD), as outlined in Table 1. As indicated, in the presence of pyridinium salt 6, which has the strongest acidity of the four cations examined, methanolysis was completed (97% yield) within 6 h at rt. In addition, imidazolium 13 and ammonium 14 promoted methanolysis to give the desired product 17 in yields of 28 and 65%, respectively, after 24 h. By contrast, triethylammonium 15, which exhibited the weakest acidity of the four cations, did not catalyze the methanolysis reaction. These results therefore confirmed that the pyridinium salt of NanoGoblin (6) should serve as a successful “weapon” against NanoKid (5).

Thus, the methanolysis of NanoKid (5) with NanoGoblin (6) in methanol-d$_4$ was attempted (Scheme 4), and the transformation was monitored by $^1$H NMR spectroscopy (Figure 2). As indicated, NanoKid (5) remained intact for 5 min after commencing the reaction. However, after 8 h, the $^1$H NMR signals corresponding to 5 had decreased in intensity by approximately one-third, and the formation of new signals was observed at 5.55 and 5.70 ppm, corresponding to the acetal protons of dimethyl acetal 18 and cross-acetal 19, respectively. After 46 h, all signals corresponding to both NanoKid (5) and intermediary cross-acetal 19 had disappeared, with the formation of dimethyl acetal 18 being confirmed in 89% yield. Interestingly, the signals at 4.75 and 5.02 ppm corresponding to the methylenedioxy protons of 6 remained unchanged during the experiment, thereby indicating that NanoGoblin (6) could successfully “defeat” NanoKid (5) whilst remaining chemically unchanged itself (i.e., in a catalytic manner).
CONCLUSIONS

In conclusion, we successfully synthesized the pyridinium salt of 1,3-dioxan-5-yl-substituted tetracyanocyclopentadienide (NanoGoblin) and demonstrated its catalytic activity in the methanolysis of acetals. More specifically, the doll-shaped NanoGoblin catalyzed the methanolysis of the acetal “head” of NanoKid in methanol-d4 to yield the corresponding dimethyl acetal without exhibiting any structural changes (i.e., catalytic “destruction”). To the best of our knowledge, this is the first example of a “battle” (i.e., reaction) between NanoPutians. We expect that this study may have educational potential to attract the interest of young organic chemistry students and in particular ignite their interest in the areas of acetal chemistry and acid/base concepts.

EXPERIMENTAL SECTION

General Experimental Methods. All air- and moisture-sensitive reactions were carried out in dry solvents under an argon atmosphere. In all cases, rt ranged from 26 to 28 °C. Flash chromatography was carried out using silica gel 60 (spherical, neutral, 40–50 μm) or diol silica gel (spherical, 40–75 μm). Melting points are uncorrected. Chemical shifts are reported in parts per million (ppm) relative to the solvent signals (1.94 ppm for CD3CN and 3.31 ppm for CD3OD) or internal TMS (0.00 ppm for CDCl3) for the 1H NMR spectra and to the solvent signals (1.39 ppm for CD3CN and 77.0 ppm for CDCl3) for the 13C NMR spectra. Coupling constants (J) are reported in hertz (Hz). Data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, and br = broad), integration, and coupling constant. High-resolution mass spectra (HRMS) were recorded on magnetic sector electron impact (EI) or magnetic sector fast atom bombardment (FAB) mass spectrometers.

Cyclic Acetal 2. To a solution of aldehyde 1 (11.2 mg, 0.052 mmol) in 1,3-propanediol (394 mg, 5.18 mmol, 100 equiv) was added TsOH·H2O (9.5 mg, 0.052 mmol), and the mixture was stirred at rt for 2 h. After this time, the reaction was quenched using a saturated NaHCO3 solution, and the resulting mixture was extracted with ethyl acetate (EtOAc), dried over anhydrous Na2SO4, and concentrated under reduced pressure. Flash chromatography using diol silica (40% CH3CN in CH2Cl2) afforded acetal 2 (11.3 mg, 79%) as a yellow solid. mp 320–330 °C (decomp); IR (KBr) νmax: 3000, 2983, 2972, 2940, 2901, 2871, 2220, 1515, 1476, 1086, 982 cm−1; 1H NMR (400 MHz, CD3CN): δ 5.57 (s, 1H), 4.14 (m, 2H), 3.93 (m, 2H), 2.07 (ddd, 1H, J = 13.5, 12.4, 4.9 Hz), 1.42 (ddd, 1H, J = 13.5, 2.7, 1.4 Hz); 13C NMR (100 MHz, CD3CN): δ 135.4, 116.5, 116.2, 101.3, 97.9, 96.4, 82.6, 26.5; HRMS (FAB) m/z: [M − Na]− calc for C13H7N4O2, 251.0569; found, 251.0556.

Pyridinium Aldehyde 3. To a solution of cyclic acetal 2 (11 mg, 0.041 mmol) in a mixture of CH2Cl2 (3.5 mL) and water (3 mL) was added pyridinium hydrochloride (24 mg, 0.22 mmol), and the resulting mixture was stirred at rt for 5 min. After this time, the organic and aqueous layers were separated and the aqueous layer was extracted with CH2Cl2. The combined organic layers were dried over anhydrous Na2SO4, filtered, and concentrated under reduced pressure to afford pyridinium aldehyde 3 as a yellow solid (4.2 mg, 37%) which was of sufficient purity for spectroscopic analysis. mp 190–195 °C (decomp); IR (KBr) νmax: 3222, 3168, 2925, 2925, 2328, 2128, 1664, 1604, 1532, 1470 cm−1; 1H NMR (600 MHz, CD3OD).

Table 1. Investigation of Various Aminium “Weapons” in the Methanolysis of Acetal (16)

| entry | H−NR3 | time (h) | yield (%)a |
|-------|-------|----------|------------|
| 1     | 6     | 6        | 97         |
| 2     | 13    | 24       | 28         |
| 3     | 14    | 24       | 65         |
| 4     | 15    | 24       | 1          |

*Yields determined by 1H NMR spectroscopy using 1,2-dichloromethane as an internal standard.
Alcohol 8. Alcohol 8 was prepared according to Gensini’s method with a number of modifications.12 More specifically, to a solution of 2-hydroxymethyl-1,3-propanediol (7) (5.00 g, 47.1 mmol), LiBr (818 mg, 9.42 mmol), and p-TsOH·H2O (896 mg, 4.72 mmol, 0.1 equiv) in a mixture of CHCl3 (40 mL) and H2O (4.5 mL) were added CH3OH (15.1 mL, 471 mmol, 10.0 equiv) and methylal (17.0 mL, 189 mmol, 4.0 equiv). The resulting mixture was heated at 65 °C for 26 h; after this time, additional LiBr (818 mg, 9.42 mmol), p-TsOH·H2O (896 mg, 4.72 mmol), and methylal (9.5 mL, 106 mmol) were added. The reaction mixture was then heated for a further 46 h at 65 °C, cooled to rt, and concentrated to a volume of ~10 mL under reduced pressure. Flash chromatography using silica gel (60–100% Et2O in n-hexane) afforded acetal alcohol 8 (3.30 g, 59%). The O-MOM product (1.77 g) was also obtained as a by-product, and so removal of the O-MOM group was carried out as follows. To a solution of the O-MOM product (1.77 g, 10.8 mmol), LiBr (188 mg, 2.17 mmol), and p-TsOH·H2O (206 mg, 1.08 mmol, 0.1 equiv) in CHCl3 (9.2 mL) was added CH3OH (3.5 mL, 108 mmol, 10 equiv). The resulting mixture was stirred under reflux for 24 h, then cooled to rt, and concentrated under reduced pressure. Flash chromatography using silica gel (60–100% Et2O in n-hexane) afforded the desired acetal alcohol 8 (704 mg, 15%). In total, 4.00 g of 8 was obtained (74%) as a colorless oil. IR (film): 3420, 2953, 2923, 2853, 1463 cm⁻¹; 1H NMR (500 MHz, CDCl3): δ 4.87 (d, 1H, J = 6.1 Hz), 4.81 (d, 1H, J = 6.1 Hz), 4.02 (dd, 2H, J = 11.7, 3.7 Hz), 3.80 (dd, 2H, J = 11.6, 6.2 Hz), 3.76 (dd, 2H, J = 6.6, 5.0 Hz), 1.96 (tt, 1H, J = 6.7, 6.2, 3.7 Hz), 1.62 (t, 1H, J = 5.0 Hz); 13C NMR (150 MHz, CDCl3): δ 94.2, 68.3, 61.6, 37.4; HRMS (EI) m/z: [M − H]⁺ calcd for C₅H₉O₃, 117.0552; found, 117.0537.

Figure 2. Time-dependent changes in the 1H NMR spectrum of the reaction mixture during the methanolysis of NanoKid (5) using NanoGoblin (6).
Iodide 9. To a solution of alcohol 8 (4.00 g, 33.9 mmol), imidazole (4.62 g, 67.8 mmol, 2.0 equiv), and PPh3 (17.8 g, 67.8 mmol, 2.0 equiv) in benzene (80 mL) was added iodine (17.2 g, 67.8 mmol, 2.0 equiv). After stirring the reaction mixture at rt for 0.5 h, the reaction was quenched with a 10 wt% aqueous solution of Na2S2O3. The resulting mixture was extracted with Et2O, and the organic phase was washed with water, dried over anhydrous MgSO4, and concentrated under reduced pressure. Flash chromatography using silica gel (70% CH2Cl2 in n-hexane) afforded iodide 9 (5.48 g, 71%) as colorless needles. mp 33–35 °C; IR (film): 2965, 2916, 2847, 2773, 1156 cm⁻¹; ¹H NMR (600 MHz, CDCl3): δ 4.85 (d, 1H, J = 6.2 Hz), 4.76 (d, 1H, J = 6.2 Hz), 4.07 (dd, 2H, J = 11.5, 3.7 Hz), 3.69 (dd, 2H, J = 11.5, 6.6 Hz), 3.20 (d, 2H, J = 7.1 Hz), 2.04 (ttt, 1H, J = 7.1, 6.6, 3.7 Hz); ¹³C NMR (150 MHz, CDCl3): δ 134.7, 117.1, 116.0, 101.3, 97.0, 94.5, 70.5, 37.9; HRMS (FAB) m/z: [M + C3H7N4O2⁺]⁺ calcd for C19H17N8O2, 389.1474; found, 389.1490.

Trifluoromethylsulfone 10. A mixture of iodide 9 (1.82 g, 8.0 mmol), Na2SO4, and benzene (80 mL) was heated at 100 °C in a sealed tube in the absence of light. After 0.5 h, the mixture was cooled to rt, and concentrated under reduced pressure. Flash chromatography using silica gel (30% EtOAc in CH2Cl2) afforded trifluoromethylsulfone 10 (1.82 g, 92%) as a yellow solid. mp 251.0569; found, 251.0597.

Silver Salt 12. To a suspension of NaH (60% dispersion in mineral oil, 2.05 g, 51.2 mmol, 3.0 equiv) in THF (80 mL) and HMPA (36 mL) was added dropwise at 0 °C a solution of sulfone 10 (4.00 g, 17.1 mmol, 1.0 equiv) in THF (120 mL), and stirring was continued at 0 °C for 0.5 h. A solution of tetracyanodithiophosphine (11) (3.46 g, 18.8 mmol, 1.1 equiv) in THF (120 mL) was then added, and stirring was continued at 0 °C for 20 h. After this time, brine (40 mL) was added and the reaction mixture was extracted with EtOAc. The organic phase was then washed with brine, dried over anhydrous MgSO4, and concentrated under reduced pressure. Flash chromatography using silica gel (30% EtOAc in n-hexane) afforded imidazolium salt 12 (75 mg, 95%) as a yellow solid. mp 235–237 °C; IR (KBr pellet): 3078, 2987, 2877, 1449, 1137 cm⁻¹; ¹H NMR (500 MHz, CDCl3): δ 5.92 (s, 4H), 5.00 (d, 1H, J = 6.2 Hz), 4.71 (d, 1H, J = 6.2 Hz), 4.02 (dd, 2H, J = 11.5, 5.2 Hz), 3.99 (dd, 2H, J = 11.5, 10.5 Hz), 3.34 (tt, 1H, J = 10.5, 5.2 Hz); ¹³C NMR (125 MHz, CDCl3): δ 134.5, 117.0, 115.9, 101.2, 97.0, 94.4, 70.5, 37.8; HRMS (FAB) m/z: [M + C3H7N4⁺]⁺ calcd for C19H17N8O2, 389.1474; found, 389.1490.

Ammonium Salt 14. To a suspension of silver salt 12 (106 mg, 0.30 mmol) in CH3OH (1 mL) was added ammonium chloride (40 mg, 0.75 mmol), and the mixture stirred at rt for 3 h. After this time, the resulting suspension was filtered through a Celite pad and the filtrate was concentrated under reduced pressure. Flash chromatography using diol silica (30% CH3CN in CH2Cl2) provided ammonium salt 14 (75 mg, 95%) as a yellow solid. mp 179–182 °C; IR (KBr pellet): 3185, 3014, 2872, 2771, 1464, 1449, 1144, 1029, 937, 900 cm⁻¹; ¹H NMR (500 MHz, CDCl3): δ 5.83 (s, 1H), 14.78 (s, 2H), 4.99 (d, 1H, J = 5.2 Hz), 4.02 (dd, 2H, J = 11.5, 5.2 Hz), 3.99 (dd, 2H, J = 11.5, 10.5 Hz), 3.34 (tt, 1H, J = 10.5, 5.2 Hz); ¹³C NMR (125 MHz, CDCl3): δ 135.1, 134.5, 120.4, 117.0, 115.9, 101.2, 97.0, 94.4, 70.5, 37.8; HRMS (FAB) m/z: [M + C3H7N4⁺]⁺ calcd for C19H17N8O2, 389.1474; found, 389.1490.

Triethylammonium Salt 15. To a suspension of silver salt 12 (97 mg, 0.27 mmol) in CH3OH (1 mL) was added triethylamine hydrochloride (186 mg, 1.35 mmol, 5.0 equiv), and the mixture was stirred at rt for 3 h. After this time, the resulting suspension was filtered through a Celite pad and the filtrate was concentrated under reduced pressure. Flash chromatography using diol silica (30% CH3CN in CH2Cl2) provided triethylammonium salt 15 (70 mg, 73%) as a yellow solid. mp 157–160 °C; IR (KBr pellet): 3078, 2989, 2854, 2813, 2204, 1461, 1145, 1069, 1021, 934, 899 cm⁻¹; ¹H NMR (500 MHz, CDCl3): δ 7.75 (s, 1H), 5.06 (d, 1H, J = 6.4 Hz), 4.78 (d, 1H, J = 6.4 Hz), 4.07 (d, 4H, J = 7.8 Hz), 3.49 (quintet, 1H, J = 7.8 Hz), 3.28 (q, 6H, J = 7.3 Hz), 1.45 (t, 9H, J = 7.3 Hz); ¹³C NMR (125 MHz, CDCl3): δ 134.5, 116.5, 115.6.
2129, 1355, 1131 cm

7.59 (s, 1H), 7.51 (s, 1H), 7.45 (d, 2H, ppm) relative to that of the internal standard (benzene, 7.33 to be 89% by the integration of the dimethyl acetal signal (5.54 C9H6D3O2, 158.1214; found, 158.1205.

yield of the product was determined to be 97% based on the and 1,2-dichloromethane (3.77 ppm) after 6 h. For the reaction where 6 was employed (Table 1, entry 1), the reaction was quenched with Et₃N after the completion of methanolysis. The resulting mixture was concentrated and purified by flash chromatography using silica gel (20% EtOAc in n-hexane) to afford acetal 17 (23 mg, 84% isolated yield) as a

1H NMR (600 MHz, CDCl₃): δ 7.44 (m, 2H), 7.38 (m, 3H), 5.39 (s, 1H); 13C NMR (150 MHz, CDCl₃): 139.1, 135.3, 134.2, 133.6, 129.6, 125.7, 125.1, 124.5, 122.0, 104.7, 104.2, 102.2, 91.9, 91.3, 88.4, 79.4, 77.8, 75.9, 53.6 (septet, JCD = 21.5 Hz), 31.0, 30.9, 28.3 (x2), 22.1, 21.3, 13.5; HRMS (FAB) m/z: [M + Na]⁺ calc for C₁₇H₃₅D₅O₃Na, 549.3616; found, 549.3621.

ASSOCIATED CONTENT

Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acsomega.7b01748.

Comparisons of the aminium structures and weapon figures and copies of ¹H and ¹³C NMR spectra of all new compounds (PDF)

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Notes
The authors declare no competing financial interest.

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