Data Article

Data for the synthesis and characterization of fluorene-containing β-diketonato ligands and their rhodium(I) complexes

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A R T I C L E   I N F O

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A B S T R A C T

Three novel β-diketonato ligands were synthesized containing the fluorene-group as substituent. Rhodium(I) complexes thereof were synthesized as reported in Fluorene-containing β-diketonato ligands and their Rhodium(I) complexes. The \(^1\)H and \(^{13}\)C NMR’s of all 6 compounds are reported. Spectroscopic data of concentration variations vs. absorbance intensities are given for ligands and rhodium(I) complexes showing that they follow the Beer-Lambert law and extinction coefficients (\(\varepsilon\)) are determined. Fluorescence quantum yields are determined and graphs showing the integrated fluorescent intensities vs. absorbance are shown. The electrochemistry data of compounds 1 – 6 are reported for scan at 100 - 500 mV/s scan speeds. No electrochemical processes were observed for the fluorene-group, only the Rh(I) and β-diketonato processes were observed.

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**Specifications Table**

| Subject | Inorganic chemistry |
|---------|---------------------|
| Specific subject area | Organometallic chemistry and physical characterization |
| Type of data | Table |
| | Graph |
| | Figure |
| How data were acquired | NMR - Bruker 600 MHz AVANCE II NMR spectrometer with Topspin 3.6.2 software. |
| | UV/vis - Cary 60 spectrophotometer. |
| | Fluorescence - Cary Eclipse spectrophotometer. Spectra were integrated using OriginPro 2019b software version 9.6.5.169. |
| | Electrochemistry - BAS 100 B/W potentiostat |
| Data format | Analyzed |
| Parameters for data collection | NMR - 600 MHz AVANCE II NMR spectrometer operating at 25 °C, with 5 mm DUAL 13C-1H/D probe with z-gradients |
| | 1H frequency = 600.28 MHz |
| | 13C frequency = 150.95 MHz |
| | Fluorescence – Excitation at 330 nm, excitation and emission slits – 5 nm, excitation and emission filters open, scan rate – 600 nm/min, PMT voltage 530 V |
| Description of data collection | NMR – Compounds 1–6 were measured in CDCl₃. |
| | Absorbance and fluorescent spectra were measured in spectroscopic grade ethanol for 10, 8, 6, 4 and 2 ppm solutions of compounds 1–6. |
| | Electrochemistry of 0.5 mmol dm⁻³ solutions of 1–6 in was measured in acetonitrile with 0.10 mol dm⁻³ [N(Bu)₄][PF₆] under argon at 25 °C utilizing a BAS 100 B/W potentiostat. |
| Data source location | Institution: University of the Free State |
| | City: Bloemfontein |
| | Country: South Africa |
| Data accessibility | doi:10.17632/2vcvjycf63.1 |
| Related research article | Co-submission |
| | E. Fourie, Fluorene-containing β-diketonato ligands and their Rhodium(I) complexes, Inorg. Chim. Acta, (https://doi.org/10.1016/j.ica.2020.120209) |

**Value of the Data**

- This data is valuable in characterization of β-diketones and rhodium(I) complexes and comparative NMR characterization and spectroscopic purposes.
- Researchers in rhodium chemistry and ligand design can use NMR, UV/vis absorbance and fluorescence data.
- Data can be used to develop quicker and easier comparative characterizations of β-diketones and rhodium(I) complexes.
- Fluorescence quantum yields are reported.
- Electrochemical data is reported for β-diketones and rhodium(I) complexes.

**1. Data Description**

The data is in support of the article cited and reports characterization data used [1]. Full characterization of compounds 1–6 are reported. Compounds 1–3 are β-diketonato ligands, while compounds 4–6 are rhodium(I) complexes thereof. NMR is used for structural elucidation, additional 2D experiments, including HMBC and HSQC experiments were used to determine keto or enol tautomer. UV/vis and fluorescence was used to determine the fluorescent properties of all compounds. Electrochemical studies was carried out in order to determine the influence of the fluorene-moiety on the electrochemical properties of the β-diketone and Rh(I) complexes.
Determination of fluorescence quantum yield ($\Phi_\text{F}$): Calculations for compound 1 is shown, and the same method used to determine values for 2–6.

$$
\Phi_X = \Phi_A \left( \frac{\text{Grad}_A}{\text{Grad}_X} \right) \left( \frac{n_A^2}{n_X^2} \right)
$$

where $\Phi_A = 0.27$ [2] and $n_A^2 = 1.359$ [3]

Grad$_A$ was measured as 390 088

$$
\Phi_X = 0.27 \left( \frac{367.72}{390 \ 088} \right)(1) = 0.0003 \text{ for compound 1}
$$

2. Experimental Design, Materials and Methods

Synthesis of compounds 1–6 as described in E. Foure, Fluorene-containing $\beta$-diketonato ligands and their Rhodium(I) complexes, Inorg. Chim. Acta, (https://doi.org/10.1016/j.ica.2020.120209). Single measurements of NMRs and spectroscopic experiments were determined and are shown in Figures 1–6.

2.1. NMR spectroscopy

All NMR spectra were collected using a 600 MHz AVANCE II NMR spectrometer operating at 25 °C and utilizing a $^{13}$C and $^1$H = 5 mm DUAL 13C-1H/D probe with z-gradients. Chemical shifts (δ ppm) were determined with TMS as an internal reference. $J$ values are given in Hz. $^1$H frequency = 600.28 MHz and $^{13}$C frequency = 150.95 MHz.

2.2. Spectroscopy of 1–6

FTIR spectra were recorded on a Bruker Tensor 27 instrument with ATR attachment.

UV/vis absorbance measurements were carried out on a Cary 60 spectrophotometer for 10, 8, 6, 4 and 2 ppm solutions in spectroscopic grade ethanol. UV/vis absorbance spectra were used to determine extinction coefficients ($\varepsilon$).

Fluorescence measurements were performed on a Cary Eclipse spectrophotometer. Spectra of 1–6 were measured for 10, 8, 6, 4 and 2 ppm solutions in spectroscopic grade ethanol. Excitation at 330 nm were used an spectra were measured between 330 and 600 nm. Fluorescence intensity was measured by integration of fluorescent curves by OriginPro 2019b software version 9.6.5.169. Fluorescent intensities were plotted vs. absorbance at excitation wavelength [2].

2.3. Electrochemistry of 1–6

Cyclic voltammograms of 1–6 were measured on 0.5 mmol dm$^{-3}$ solutions in spectroscopic grade acetonitrile containing 0.10 mol dm$^{-3}$ [N$^{+}$(nBu)$_4$][PF$_6$] under a blanket of purified argon at 25 °C. Measurements were performed with a BAS 100 B/W potentiostat. A three electrode cell was used, consisting of a glassy carbon working electrode, Pt auxiliary electrode and a Ag-wire reference electrode. Successive experiments were performed with different limits and are shown as such. Results are reported against the ferrocene/ferrocinium (FcH/FcH$^+$) couple at 0 V. Experimentally FcH was used as internal standard for compounds 1–3. Decamethyl ferrocene (Fc$^+$) was employed as internal standard, for compounds 4–6. Care was taken to ensure that exact equimolar amounts of the internal standard were added. Experiments were first performed in the absence of ferrocene and decamethyl ferrocene, and then repeated in the presence of < 0.5 mmol dm$^{-3}$ ferrocene or decamethyl ferrocene (Fc$^+$). A separate experiment containing only ferrocene and decamethyl ferrocene were performed and showed that under our conditions, the Fc$^+$/Fc$^{++}$ couple was at −515 mV vs. FcH/FcH$^+$. Data were adjusted on a spreadsheet to set the formal reduction potential of FcH/FcH$^+$ to 0 V. Electrochemical data is reported in Table 1.
Table 1
All electrochemical values obtained for compounds 1 – 6, obtained at 100 – 500 mV/s and 25 °C.

| Scan rate / mV/s | E_{pa} / mV | E_{pc} / mV | i_{pa} / μA | i_{pc} / μA | ΔE_p / mV | E° / mV | i_{pa}/i_{pc} |
|------------------|-------------|-------------|-------------|-------------|-----------|--------|---------------|
| 1 – wave A       |             |             |             |             |           |        |               |
| 100              | −2005       | −2084       | 1.13        | 4.01        | 79        | −2045  | 0.28^        |
| 200              | −2012       | −2093       | 2.52        | 5.90        | 81        | −2053  | 0.43^        |
| 300              | −2018       | −2099       | 3.51        | 7.91        | 81        | −2059  | 0.44^        |
| 400              | −2021       | −2117       | 4.65        | 9.16        | 96        | −2069  | 0.51^        |
| 500              | −2028       | −2125       | 5.42        | 9.29        | 97        | −2077  | 0.58^        |
| 1 – wave B       |             |             |             |             |           |        |               |
| 100              | −1747       |             | 1.03        |             |           |        |               |
| 200              | −1763       |             | 2.01        |             |           |        |               |
| 300              | −1781       |             | 2.33        |             |           |        |               |
| 400              | −1796       |             | 2.37        |             |           |        |               |
| 500              | −1798       |             | 2.93        |             |           |        |               |
| 2 – wave A       |             |             |             |             |           |        |               |
| 100              | −2021       | −2097       | 1.24        | 3.22        | 76        | −2059  | 0.39^        |
| 200              | −2029       | −2106       | 2.48        | 5.46        | 77        | −2068  | 0.44^        |
| 300              | −2035       | −2114       | 3.03        | 6.45        | 79        | −2075  | 0.47^        |
| 400              | −2039       | −2126       | 4.08        | 7.53        | 87        | −2083  | 0.54^        |
| 500              | −2043       | −2134       | 4.98        | 9.05        | 91        | −2089  | 0.55^        |
| 2 – wave B       |             |             |             |             |           |        |               |
| 100              | −1765       |             | 0.38        |             |           |        |               |
| 200              | −1768       |             | 0.65        |             |           |        |               |
| 300              | −1772       |             | 0.94        |             |           |        |               |
| 400              | −1775       |             | 1.08        |             |           |        |               |
| 500              | −1787       |             | 1.42        |             |           |        |               |
| 3 – wave A       |             |             |             |             |           |        |               |
| 100              | −2048       | −2121       | 1.87        | 4.78        | 73        | −2085  | 0.39^        |
| 200              | −2050       | −2126       | 2.94        | 6.92        | 76        | −2088  | 0.42^        |
| 300              | −2053       | −2130       | 3.77        | 8.72        | 77        | −2092  | 0.43^        |
| 400              | −2062       | −2142       | 4.49        | 9.90        | 80        | −2102  | 0.45^        |
| 500              | −2066       | −2148       | 5.46        | 11.55       | 82        | −2107  | 0.47^        |
| 3 – wave B       |             |             |             |             |           |        |               |
| 100              | −1874       |             | 0.81        |             |           |        |               |
| 200              | −1877       |             | 1.21        |             |           |        |               |
| 300              | −1888       |             | 1.47        |             |           |        |               |
| 400              | −1894       |             | 1.84        |             |           |        |               |
| 500              | −1905       |             | 2.08        |             |           |        |               |
| 4 – wave Rh[III] |             |             |             |             |           |        |               |
| 100              | 287         | 201         | 1.40        | 0.54        | 86        | 244    | 0.39         |
| 200              | 290         | 192         | 2.41        | 0.98        | 98        | 241    | 0.41         |
| 300              | 292         | 189         | 2.72        | 1.12        | 103       | 241    | 0.41         |
| 400              | 295         | 185         | 3.22        | 1.35        | 110       | 240    | 0.42         |
| 500              | 297         | 181         | 3.50        | 1.71        | 116       | 239    | 0.49         |
| 4 – wave A       |             |             |             |             |           |        |               |
| 100              | −2219       | −2345       | 1.28        | 1.81        | 126       | −2282  | 0.71^        |
| 200              | −2227       | −2357       | 2.14        | 2.43        | 130       | −2292  | 0.88^        |
| 300              | −2229       | −2359       | 2.84        | 3.16        | 130       | −2294  | 0.90^        |
| 400              | −2230       | −2361       | 3.20        | 3.34        | 131       | −2296  | 0.96^        |
| 500              | −2230       | −2364       | 3.28        | 3.39        | 134       | −2297  | 0.97^        |

(continued on next page)
Table 1 (continued)

| Scan rate / mV/s | $E_{pa}$ / mV | $E_{pc}$ / mV | $i_{pa}$ / $\mu$A | $i_{pc}$ / $\mu$A | $\Delta E_{p}$ / mV | $E^0$ / mV | $i_{pa}/i_{pc}$ |
|------------------|---------------|---------------|-------------------|-------------------|---------------------|-------------|----------------|
| **4 – wave B**   |               |               |                   |                   |                     |             |                |
| 100              | 318           | 254           | 3.13              | 1.14              | 64                  | 286         | 0.36           |
| 200              | 319           | 242           | 4.25              | 1.74              | 77                  | 281         | 0.41           |
| 300              | 323           | 226           | 5.44              | 2.35              | 97                  | 275         | 0.43           |
| 400              | 327           | 218           | 6.49              | 2.87              | 109                 | 273         | 0.44           |
| 500              | 330           | 215           | 7.47              | 3.36              | 115                 | 273         | 0.45           |
| **5 – wave Rh^{III}** |           |               |                   |                   |                     |             |                |
| 100              | -2254         | -2332         | 2.06              | 2.71              | 78                  | -2293       | 0.76*          |
| 200              | -2258         | -2339         | 3.04              | 3.86              | 81                  | -2299       | 0.79*          |
| 300              | -2261         | -2343         | 3.87              | 4.54              | 82                  | -2302       | 0.85*          |
| 400              | -2267         | -2349         | 4.47              | 5.17              | 82                  | -2308       | 0.86*          |
| 500              | -2272         | -2355         | 4.75              | 5.48              | 83                  | -2314       | 0.87*          |
| **5 – wave A**   |               |               |                   |                   |                     |             |                |
| 100              | -2223         | -2296         | 1.81              | 2.29              | 73                  | -2260       | 0.79*          |
| 200              | -2227         | -2308         | 2.67              | 3.19              | 81                  | -2268       | 0.84*          |
| 300              | -2229         | -2313         | 3.08              | 3.63              | 84                  | -2271       | 0.85*          |
| 400              | -2232         | -2319         | 3.72              | 4.34              | 87                  | -2276       | 0.86*          |
| 500              | -2237         | -2237         | 4.25              | 4.94              | 100                 | -2287       | 0.86*          |
| **6 – wave B**   |               |               |                   |                   |                     |             |                |
| 100              | -1301         | -1301         | 0.38              |                   |                     |             |                |
| 200              | -1302         | -1304         | 0.83              |                   |                     |             |                |
| 300              | -1304         | -1304         | 1.20              |                   |                     |             |                |
| 400              | -1310         | -1310         | 1.54              |                   |                     |             |                |
| 500              | -1316         | -1316         | 1.68              |                   |                     |             |                |

* $i_{pc}/i_{pa}$ for reduction waves.
2.4. Data in repository

NMRs Compound 1 (Fig. 1)

Fig. 1. NMR spectra of compound 1 in CDCl$_3$ at 25 °C; top - $^1$H, middle - $^{13}$C, bottom left - 2D $^1$H, $^{13}$C HSQC, bottom right - 2D $^1$H, $^{13}$C HMBC. $^1$H and $^{13}$C NMR's account for all protons and carbons present in the structure, while $^1$H NMR shows that the compound is in the enol form, due to the position of the methine peak and it’s integration for 1 proton. $^1$H, $^{13}$C HSQC shows correlations between protons and carbons identifying the methine carbon as well as the CH$_2$ on the fluorene 2-position and the alkyl R group. $^1$H, $^{13}$C HMBC shows that the fluorene group is located next to the enol group, while the –CH$_3$ group is located next to the ketone.
NMRs Compound 2 (Fig. 2)

![NMR spectra of compound 2](image)

**Fig. 2.** NMR spectra of compound 2 in CDCl₃ at 25 °C; top - ¹H, middle - ¹³C, bottom left - 2D ¹H, ¹³C HSQC, bottom right - 2D ¹H, ¹³C HMBC. Similar information is obtained from the 1D and 2D NMR spectra as for compound 1.
NMRs Compound 3 (Fig. 3)

Fig. 3. NMR spectra of compound 3 in CDCl₃ at 25 °C; top - ¹H, middle - ¹³C, bottom left - 2D ¹H, ¹³C HSQC, bottom right - 2D ¹H, ¹³C HMBC. 1D and 2D NMR spectra give similar structural information as for compound 1 and 2.

NMRs Compound 4 (Fig. 4)
NMRs Compound 5 (Fig. 5)
NMRs Compound 6 (Fig. 6)
IRs Compounds 1 – 6 (Fig. 7)
Compound 1 UV & Fl spectra (Fig. 8)
Compound 2 UV & Fl spectra (Fig. 9)
Compound 3 UV & Fl spectra (Fig. 10)
Compound 4 UV & Fl spectra (Fig. 11)
Fig. 4. NMR spectra of compound 4 in CDCl₃ at 25 °C; top left - ¹H, top right - ¹³C, bottom - 2D ¹H, ¹⁰³Rh HMQC. ¹H and ¹³C NMR's account for all protons and carbons present. Spectra are similar to that of the free β-diketone, with the addition of 12 protons and 8 carbons due to the cyclooctadiene ligand also attached to the Rh(I) metal center. 2D ¹H, ¹⁰³Rh HMQC NMR was used to prove the presence and complexation to the Rh(I) metal center.
Fig. 5. NMR spectra of compound 5 in CDCl₃ at 25 °C; top left - ¹H, top right - ¹³C, bottom - 2D ¹H, ¹⁰³Rh HMQC. Similar information is obtained from the 1D and 2D NMR spectra as for compound 4.
Fig. 6. NMR spectra of compound 6 in CDCl₃ at 25 °C; top left - ^1H, top right - ^13C, bottom - 2D ^1H, ^103Rh HMQC. 1D and 2D NMR spectra give similar structural information as for compound 4 and 5.
Fig. 7. IR spectra of compounds 1 - 6. Spectra of ligands 1 – 3 show a single stretching frequency at ~1600 cm, representing the carbonyl group. This confirms that the ligand is formed in the enol form. In the spectra of the Rh(I) complexes 4 – 6, these stretching frequencies are absent indicating that the carbonyl group is not vibrating freely and is complexed to the metal center.

Fig. 8. UV/vis absorbance spectra (left) and fluorescence spectra of 2, 4, 6, 8 and 10 ppm solutions of compound 1 in ethanol at 25 °C. UV/vis spectra were measured on a Cary 60 dual beam spectrophotometer between 200 and 600 nm. No baseline correction was used and a scan rate of 600 nm/min was used. Fluorescence spectra were measured on a Cary Eclipse between 330 and 600 nm after excitation at 330 nm. Excitation and emission slits of 5 nm each were used. PMT voltage was set at 530 V.

Compound 5 UV & Fl spectra (Fig. 12)
Compound 6 UV & Fl spectra (Fig. 13)
Figs. 14–17
Fig. 18 echem 1–3
Fig. 19 echem 4–6
Fig. 9. UV/vis absorbance spectra (left) and fluorescence spectra of 2, 4, 6, 8 and 10 ppm solutions of compound 2 in ethanol at 25 °C. UV/vis and Fluorescence instrument settings were set with the exact parameters as described for compound 1. This is necessary for the calculation of fluorescence quantum yields.

Fig. 10. UV/vis absorbance spectra (left) and fluorescence spectra of 2, 4, 6, 8 and 10 ppm solutions of compound 3 in ethanol at 25 °C. UV/vis and Fluorescence instrument settings were set with the exact parameters as described for compound 1.
Fig. 11. UV/vis absorbance spectra (left) and fluorescence spectra of 2, 4, 6, 8 and 10 ppm solutions of compound 4 in ethanol at 25 °C. UV/vis and Fluorescence instrument settings were set with the exact parameters as described for compound 1.

Fig. 12. UV/vis absorbance spectra (left) and fluorescence spectra of 2, 4, 6, 8 and 10 ppm solutions of compound 5 in ethanol at 25 °C. UV/vis and Fluorescence instrument settings were set with the exact parameters as described for compound 1.
Fig. 13. UV/vis absorbance spectra (left) and fluorescence spectra of 2, 4, 6, 8 and 10 ppm solutions of compound 6 in ethanol at 25 °C. UV/vis and Fluorescence instrument settings were set with the exact parameters as described for compound 1.

Fig. 14. Data for the determination of extinction coefficients ($\varepsilon$) of $\beta$-diketones 1 – 3, measured at wavelengths 337, 339 and 340 nm respectively. Relative absorbance vs. concentration for $\beta$-diketones, 1 (■), 2 (▲) and 3 (●), measured in ethanol at 25 °C. Concentrations of 10, 8, 6, 4 and 2 ppm were used for all compounds and instrument settings as described in Fig. 8. $\varepsilon$(1) = 15 348 M$^{-1}$ cm$^{-1}$, $\varepsilon$(2) = 18 507 M$^{-1}$ cm$^{-1}$, $\varepsilon$(1) = 14 903 M$^{-1}$ cm$^{-1}$. 
Fig. 15. Data for the determination of extinction coefficients ($\varepsilon$) for Rh(I) complexes 4 – 6, measured at wavelengths 348, 349 and 348 nm respectively. Relative absorbance vs. concentration for rhodium(I) complexes, 4 (■), 5 (▲) and 6 (●), measured in ethanol at 25 °C. Concentrations of 10, 8, 6, 4 and 2 ppm were used for all complexes with instrumental settings as described in Fig. 8. $\varepsilon(4) = 19,164 \text{ M}^{-1} \text{ cm}^{-1}$, $\varepsilon(5) = 17,298 \text{ M}^{-1} \text{ cm}^{-1}$, $\varepsilon(6) = 27,511 \text{ M}^{-1} \text{ cm}^{-1}$.
Fig. 16. Integrated fluorescence intensity vs. absorbance, measured at 330 nm, for β-diketones, 1 (■), 2 (▲) and 3 (●), measured in ethanol at 25 °C. Integration of fluorescence curves were performed by OriginPro 2019b software version 9.6.5.169. Integration values of fluorescent spectra were determined as well as absorbance maxima measured for each concentration and plotted to give a straight line. The slopes of these straight lines were used to determine the fluorescent quantum yield of each compound according to the calculations shown above. \( \Phi_f(1) = 0.0003 \), \( \Phi_f(2) = 0.0001 \), \( \Phi_f(3) = 0.0008 \).
Fig. 17. Integrated fluorescence intensity vs. absorbance, measured at 330 nm, for rhodium(I) complexes, 4 (■), 5 (▲) and 6 (●), measured in ethanol at 25 °C. Integration of fluorescence curves were performed by OriginPro 2019b software version 9.6.5.169, and absorbance maxima measured for each concentration then plotted to give a straight line. The slopes of these straight lines were used to determine the fluorescent quantum yield of each compound as \( \Phi_F(4) = 0.0004, \Phi_F(5) = 0.0004, \Phi_F(6) = 0.0010. \)
Fig. 18. CV's of acetyl fluorene (top) and β-diketones 1 (middle top), 2 (middle bottom) and 3 (bottom) in CH$_3$CN and [nBu$_4$N][PF$_6$] are shown. Scans were measured at 100 – 500 mV/s and 25 °C. Ferrocene (FcH) is used as an internal standard. Scans were manipulated in a spreadsheet to adjust the E°'-value of the internal standard FcH/FcH$^+$-couple to 0 V as suggested by IUPAC. A three-electrode cell consisting of a Pt auxiliary electrode, a glassy carbon working electrode (surface area 0.0707 cm$^2$), and a Ag-wire reference electrode was used.
Fig. 19. CV’s of rhodium(I) complexes 4 (top), 5 (middle) and 6 (bottom) in CH$_3$CN and [nBu$_4$N][PF$_6$]. A scan of compound 5 was done with limits of −150 mV and −2 500 mV, in which the Rh(I) center is not oxidised. This helps to identify the reduction wave associated with the Rh(I) center. Scans were measured at 100 – 500 mV/s and 25 °C. Decamethylferrocene (Fc$^*$) is used as an internal standard. Scans were manipulated in a spreadsheet to adjust the E$^°$-value of the internal standard FcH/FcH$^+$-couple to 0 V as suggested by IUPAC. A three-electrode cell consisting of a Pt auxiliary electrode, a glassy carbon working electrode (surface area 0.0707 cm$^2$), and a Ag-wire reference electrode was used.
CRediT Author Statement

**Eleanor Fourie**: Conceptualization, data collection and curation, writing.

Declaration of Competing Interest

The author declare that they have no known competing financial interests or personal relationships which have, or could be perceived to have, influenced the work reported in this article.

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