Supplementary Material

Variation of the bromination site on the reaction of (E)-1-[5-methyl-1-(aryl)-1H-1,2,3-triazol-4-yl]-3-arylpiper-2-en-1-ones with N-bromosuccinimide

Bakr F. Abdel-Wahab, a Hanan A. Mohamed, a Ehab M. Zayed, b Benson M. Kariuki, c and Gamal A. El-Hiti d, *

a Applied Organic Chemistry Department, Chemical Industries Research Institute, National Research Centre, Dokki, Giza 12622, Egypt; b Green Chemistry Department, Chemical Industries Research Institute, National Research Centre, Dokki, Giza 12622, Egypt. c School of Chemistry, Cardiff University, Main Building, Park Place, Cardiff CF10 3AT, UK; d Department of Optometry, College of Applied Medical Sciences, King Saud University, Riyadh 11433, Saudi Arabia

Email: gelhiti@ksu.edu.sa

Table of Contents

<table>
<thead>
<tr>
<th>Figure</th>
<th>Description</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>S1</td>
<td>FTIR spectrum of 2</td>
<td>S2</td>
</tr>
<tr>
<td>S2</td>
<td>1H NMR spectrum of 2</td>
<td>S3</td>
</tr>
<tr>
<td>S3</td>
<td>13C NMR spectrum of 2</td>
<td>S4</td>
</tr>
<tr>
<td>S4</td>
<td>FTIR spectrum of 3</td>
<td>S5</td>
</tr>
<tr>
<td>S5</td>
<td>1H NMR spectrum of 3</td>
<td>S6</td>
</tr>
<tr>
<td>S6</td>
<td>13C NMR spectrum of 3</td>
<td>S7</td>
</tr>
<tr>
<td>S7</td>
<td>FTIR spectrum of 4</td>
<td>S8</td>
</tr>
<tr>
<td>S8</td>
<td>1H NMR spectrum of 4</td>
<td>S9</td>
</tr>
<tr>
<td>S9</td>
<td>13C NMR spectrum of 4</td>
<td>S10</td>
</tr>
<tr>
<td>S10</td>
<td>CheckCIF of 2</td>
<td>S12</td>
</tr>
<tr>
<td>S11</td>
<td>CheckCIF of 3</td>
<td>S16</td>
</tr>
<tr>
<td>S16</td>
<td>CheckCIF of 4</td>
<td>S20</td>
</tr>
</tbody>
</table>
Figure S1. FTIR spectrum of 2.
Figure S2. $^1$H NMR spectrum of 2.
Figure S3. $^{13}$C NMR spectrum of 2.
Figure S4. FTIR spectrum of 3.
Figure S5. $^1$H NMR spectrum of 3.
Figure S6. $^{13}$C NMR spectrum of 3.
Figure S7. FTIR spectrum of 4.
Figure S8. $^1$H NMR spectrum of 4.
Figure S9. $^{13}$C NMR spectrum of 4.
CheckCIF of 2–4
CheckCIF of 2

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) bmk2375

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: bmk2375

<table>
<thead>
<tr>
<th>Bond precision:</th>
<th>C–C = 0.0044 A</th>
<th>Wavelength=0.71073</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cell:</td>
<td>a=15.1917(10)</td>
<td>b=7.3491(4)</td>
</tr>
<tr>
<td></td>
<td>alpha=90</td>
<td>beta=100.068(6)</td>
</tr>
<tr>
<td>Temperature:</td>
<td>296 K</td>
<td></td>
</tr>
</tbody>
</table>

Calculated Reported
Volume 1909.49(19) 1909.49(19)
Space group P 21/n P 21/n
Hall group -P 2yn -P 2yn
Moiety formula C20 H20 Br N3 O2 ?
Sum formula C20 H20 Br N3 O2 C20 H20 Br N3 O2
M r 414.29 414.30
D x, g cm−3 1.441 1.441
Z 4 4
M u (mm−1) 2.172 2.172
F000 848.0 848.0
F000’ 847.16
h,k,lmax 21,10,24 20,10,22
Nref 5498 4730
Tmin,Tmax 0.666,0.805 0.353,1.000
Tmin’ 0.357

Correction method= # Reported T Limits: Tmin=0.353 Tmax=1.000
AbsCorr = GAUSSIAN

Data completeness= 0.860 Theta(max)= 29.836

R(reflections)= 0.0496( 2831) wR2(reflections)= 0.1207( 4730)
S = 1.057 Npar= 238
The following ALERTs were generated. Each ALERT has the format test-name_ALERT_alert-type_alert-level. Click on the hyperlinks for more details of the test.

**Alert level C**

<table>
<thead>
<tr>
<th>ALERT</th>
<th>Description</th>
<th>Severity</th>
</tr>
</thead>
<tbody>
<tr>
<td>PLAT910_ALERT_3_C</td>
<td>Missing # of FCF Reflection(s) Below Theta(Min).</td>
<td>8 Note</td>
</tr>
<tr>
<td></td>
<td>1 1 0, 2 0 0, -1 0 1, -1 1 1, 0 1 1, 1 0 1, -2 0 2, 0 0 2,</td>
<td></td>
</tr>
</tbody>
</table>

**Alert level G**

<table>
<thead>
<tr>
<th>ALERT</th>
<th>Description</th>
<th>Severity</th>
</tr>
</thead>
<tbody>
<tr>
<td>PLAT480_ALERT_4_G</td>
<td>Long H...A H-Bond Reported H6..BR1.</td>
<td>3.09 Ang.</td>
</tr>
<tr>
<td>PLAT683_ALERT_1_G</td>
<td>No Info/Value for _atom_sites_solution_primary.</td>
<td>Please Do!</td>
</tr>
<tr>
<td>PLAT912_ALERT_4_G</td>
<td>Missing # of FCF Reflections Above Sth/L= 0.600</td>
<td>705 Note</td>
</tr>
<tr>
<td>PLAT941_ALERT_3_G</td>
<td>Average HKL Measurement Multiplicity.</td>
<td>3.7 Low</td>
</tr>
<tr>
<td>PLAT952_ALERT_5_G</td>
<td>Calculated (ThMax) and CIF-Reported Lmax Differ.</td>
<td>2 Units</td>
</tr>
<tr>
<td>PLAT958_ALERT_1_G</td>
<td>Calculated (ThMax) and Actual (FCF) Lmax Differ.</td>
<td>2 Units</td>
</tr>
<tr>
<td>PLAT969_ALERT_5_G</td>
<td>The ‘Henn et al.’ R-Factor-gap value.</td>
<td>3.33 Note</td>
</tr>
<tr>
<td>Predicted WR2: Based on SigI**2</td>
<td>3.63 or SHELX Weight 11.73</td>
<td></td>
</tr>
<tr>
<td>PLAT978_ALERT_2_G</td>
<td>Number C-C Bonds with Positive Residual Density.</td>
<td>3 Info</td>
</tr>
</tbody>
</table>

0 ALERT level A = Most likely a serious problem - resolve or explain
0 ALERT level B = A potentially serious problem, consider carefully
1 ALERT level C = Check. Ensure it is not caused by an omission or oversight
8 ALERT level G = General information/check it is not something unexpected

2 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
1 ALERT type 2 Indicator that the structure model may be wrong or deficient
2 ALERT type 3 Indicator that the structure quality may be low
2 ALERT type 4 Improvement, methodology, query or suggestion
2 ALERT type 5 Informative message, check
It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

Publication of your CIF in IUCr journals

A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (Acta Crystallographica, Journal of Applied Crystallography, Journal of Synchrotron Radiation); however, if you intend to submit to Acta Crystallographica Section C or E or IUCrData, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

Publication of your CIF in other journals

Please refer to the Notes for Authors of the relevant journal for any special instructions relating to CIF submission.

PLATON version of 06/01/2024; check.def file version of 05/01/2024
checkCIF/PLATON report

Structure factors have been supplied for datablock(s) bmk2378

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: bmk2378

Bond precision: C-C = 0.0054 Å Wavelength=0.71073 Å

Cell: a=8.6657(6) b=11.3808(7) c=11.7740(8)
alpha=66.237(6) beta=77.532(6) gamma=74.221(6)
Temperature: 293 K

Calculated Reported
Volume 1015.21(13) 1015.21(13)
Space group P -1 P -1
Hall group -P 1 -P 1
Moiety formula C20 H19.58 Br1.42 N3 O3 ?
Sum formula C20 H19.58 Br1.42 N3 O3 C20 H19.58 Br1.42 N3 O3
Mr 463.09 463.44
Dx, g cm-3 1.515 1.516
Z 2 2
Mu (mm-1) 2.868 2.876
F000 468.3 469.0
F000’ 467.65
h,k,lmax 12,15,16 11,15,16
Nref 5797 4833
Tmin,Tmax 0.412,0.563 0.546,1.000
Tmin’ 0.264

Correction method= # Reported T Limits: Tmin=0.546 Tmax=1.000
AbsCorr = GAUSSIAN

Data completeness= 0.834 Theta(max)= 29.759

R(reflections) = 0.0475 (2992) wR2(reflections) = 0.1202 (4833)
S = 1.017 Npar= 257
The following ALERTS were generated. Each ALERT has the format `test-name_ALERT_alert-type_alert-level`. Click on the hyperlinks for more details of the test.

**Alert level C**
PLAT077_ALERT_4-C Unitcell Contains Non-integer Number of Atoms Please Check
PLAT910_ALERT_3-C Missing # of FCF Reflection(s) Below Theta (Min) 9 Note
  1 0 0, 0 1 0, 1 1 0, 0 -1 1, -1 0 1,
  1 0 1, 0 1 1, 1 1 1,
PLAT911_ALERT_3-C Missing FCF Refl Between Thmin & STh/L= 0.600 2 Report
-7 7 3, 1 13 7,

**Alert level G**
PLAT068_ALERT_1-G Reported F000 Differs from Calcd (or Missing)... Please Check
PLAT154_ALERT_1-G The s.u.’s on the Cell Angles are Equal .. (Note) 0.006 Degree
PLAT168_ALERT_4-G The CIF-Embedded .res File Contains EXYZ Records 1 Report
PLAT171_ALERT_4-G The CIF-Embedded .res File Contains EADP Records 1 Report
PLAT199_ALERT_1-G Reported _cell_measurement_temperature ...... (X) 293 Check
PLAT200_ALERT_1-G Reported _diffrn Ambient_temperature ...... (X) 293 Check
PLAT301_ALERT_3-G Main Residue Disorder .................. (Read 1) 5% Note
PLAT883_ALERT_1-G No Info/Value for _atom_sites_solution_primary . Please Do!
PLAT912_ALERT_4-G Missing # of FCF Reflections Above STh/L= 0.600 939 Note
PLAT941_ALERT_3-G Average HKL Measurement Multiplicity ............ 2.0 Low
PLAT969_ALERT_5-G The ‘Henn et al.’ R-Factor-gap Value ............ 2.21 Note
  Predicted WR2: Based on Sigm2 5.44 or SHELLX Weight 12.14
PLAT978.Alert_2-G Number C-C Bonds with Positive Residual Density. 1 Info

0 ALERT level A - Most likely a serious problem - resolve or explain
0 ALERT level B - A potentially serious problem, consider carefully
3 ALERT level C - Check. Ensure it is not caused by an omission or oversight
12 ALERT level G - General information/check it is not something unexpected

5 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
1 ALERT type 2 Indicator that the structure model may be wrong or deficient
4 ALERT type 3 Indicator that the structure quality may be low
4 ALERT type 4 Improvement, methodology, query or suggestion
1 ALERT type 5 Informative message, check
It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

**Publication of your CIF in IUCr journals**

A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica, Journal of Applied Crystallography, Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section C* or *E* or *IUCrData*, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

**Publication of your CIF in other journals**

Please refer to the *Notes for Authors* of the relevant journal for any special instructions relating to CIF submission.

---

PLATON version of 06/01/2024; check.def file version of 05/01/2024
checkCIF/PLATON report

Structure factors have been supplied for datablock(s) bmk2379

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: bmk2379

<table>
<thead>
<tr>
<th>Bond precision: C-C = 0.0059 A</th>
<th>Wavelength=0.71073</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cell:</td>
<td></td>
</tr>
<tr>
<td>a=7.9084(7)</td>
<td>b=9.6713(6)</td>
</tr>
<tr>
<td>alpha=74.369(6)</td>
<td>beta=77.424(7)</td>
</tr>
<tr>
<td>c=14.9540(12)</td>
<td>gamma=86.856(6)</td>
</tr>
<tr>
<td>Temperature:</td>
<td></td>
</tr>
<tr>
<td>293 K</td>
<td></td>
</tr>
<tr>
<td>Calculated</td>
<td>Reported</td>
</tr>
<tr>
<td>Volume 1075.02(15)</td>
<td>1075.02(15)</td>
</tr>
<tr>
<td>Space group P -1</td>
<td>P -1</td>
</tr>
<tr>
<td>Hall group -P 1</td>
<td>-P 1</td>
</tr>
<tr>
<td>Moleety formula C21 H21 Br2 N3 O4</td>
<td>?</td>
</tr>
<tr>
<td>Sum formula C21 H21 Br2 N3 O4</td>
<td>C21 H21 Br2 N3 O4</td>
</tr>
<tr>
<td>Mr 539.21</td>
<td>539.23</td>
</tr>
<tr>
<td>Dx,g cm-3 1.666</td>
<td>1.666</td>
</tr>
<tr>
<td>Z 2</td>
<td>2</td>
</tr>
<tr>
<td>Mu (mm-1) 3.803</td>
<td>3.803</td>
</tr>
<tr>
<td>F000 540.0</td>
<td>540.0</td>
</tr>
<tr>
<td>F000’ 539.09</td>
<td></td>
</tr>
<tr>
<td>h,k,lmax 11,13,20</td>
<td>10,13,20</td>
</tr>
<tr>
<td>Nref 6075</td>
<td>5107</td>
</tr>
<tr>
<td>Tmin,Tmax 0.176,0.285</td>
<td>0.274,1.000</td>
</tr>
<tr>
<td>Tmin’ 0.087</td>
<td></td>
</tr>
<tr>
<td>Correction method= # Reported T Limits: Tmin=0.274 Tmax=1.000 AbsCorr = GAUSSIAN</td>
<td></td>
</tr>
<tr>
<td>Data completeness= 0.841</td>
<td>Theta(max)= 29.654</td>
</tr>
<tr>
<td>R(reflections)= 0.0481(3498)</td>
<td>wR2(reflections)=</td>
</tr>
<tr>
<td>S = 1.047</td>
<td>0.1367(5107)</td>
</tr>
<tr>
<td>Npar= 275</td>
<td></td>
</tr>
</tbody>
</table>
The following ALERTS were generated. Each ALERT has the format:

test-name_ALERT_alert-type_alert-level.

Click on the hyperlinks for more details of the test.

**Alert level B**

PLAT910_ALERT_3_B Missing # of PCP Reflection(s) Below Theta(Min). 11 Note

1 0 0, -1 1 0, 0 1 0, 0 -1 1, -1 0 1, 0 0 1, 1 0 1, 0 1 1, 1 1 1, 0 0 2, 0 1 2

**Alert level G**

PLAT063_ALERT_4_G Crystal Size Possibly too Large for Beam Size .. 0.62 mm
PLAT199_ALERT_1_G Reported _cell_measurement_temperature ..... (K) 293 Check
PLAT200_ALERT_1_G Reported _diffrn_ambient_temperature ..... (K) 293 Check
PLAT480_ALERT_4_G Long H...A H-Bond Reported H6 ..BR2 .. 3.09 Ång.
PLAT480_ALERT_4_G Long H...A H-Bond Reported H7C ..BR2 .. 3.07 Ång.
PLAT883_ALERT_1_G No Info/Value for _atom_sites_solution_primary . Please Do !
PLAT912_ALERT_4_G Missing # of PCP Reflections Above STh/L= 0.600 954 Note
PLAT941_ALERT_3_G Average HKL Measurement Multiplicity ......... 2.0 Low
PLAT969_ALERT_5_G The ’Henn et al.’ R-Factor-gap value ............ 2.87 Note
Predicted wr2: Based on SigI**2 4.76 or SHELX Weight 13.41
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density. 6 Info

0 ALERT level A - Most likely a serious problem - resolve or explain
1 ALERT level B - A potentially serious problem, consider carefully
0 ALERT level C - Check. Ensure it is not caused by an omission or oversight
10 ALERT level G - General information/check it is not something unexpected

3 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
1 ALERT type 2 Indicator that the structure model may be wrong or deficient
2 ALERT type 3 Indicator that the structure quality may be low
4 ALERT type 4 Improvement, methodology, query or suggestion
1 ALERT type 5 Informative message, check
It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

**Publication of your CIF in IUCr journals**

A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica, Journal of Applied Crystallography, Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section C* or *E* or IUCrData, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

**Publication of your CIF in other journals**

Please refer to the *Notes for Authors* of the relevant journal for any special instructions relating to CIF submission.

---

**PLATON version of 06/01/2024; check.def file version of 05/01/2024**
Data block bmk2379 - ellipsoid plot

PLATON - Apr 25 14:58:55 2024 - (60124)
Z 26 bmk2379 P -1 R = 0.05 RES = 0 52 X