**Supporting Information for:**

Towards a Better Understanding of the Electrochemical Synthesis of 2,5-dicarboxy-2,5-dihydrofurans: Structure, Mechanism and Influence over Stereochemistry

Michael A. Shipman, Stephen Sproules, Claire Wilson and Mark D. Symes\*

WestCHEM, School of Chemistry, University of Glasgow, University Avenue, Glasgow, G12 8QQ, UK.

\*E-mail: mark.symes@glasgow.ac.uk

EXPERIMENTAL SECTION

**General Experimental Remarks:** Unless otherwise stated, all syntheses were conducted under nitrogen in air- and moisture-free solvents obtained from a commercial solvent purification system. Water used was of ‘ultra-pure’ grade (18.2 MΩ resistivity) and dispensed from a SG Ultraclear TWF UV device. Sodium acetate (≥99%), sodium butyrate (98%), butyric acid (≥99%), furan (≥99%), diethyl ether (≥99.8%), and lead(IV) acetate (96% + 5-10% glacial acetic acid) were supplied by Sigma Aldrich. Acetonitrile (≥99%), sodium carbonate (≥99.9%) and nitric acid (70%) were supplied by Fisher Scientific. Acetic acid (99.9%) was purchased from VWR. Liquid nitrogen was supplied by BOC. HCl was obtained from Honeywell Fluka.

All 1H and 13C NMR spectra were recorded on a Bruker AV 400 instrument (unless otherwise stated), at a constant temperature of 300 K. Chemical shifts are reported in parts per million from low to high field. Standard abbreviations indicating multiplicity were used as follows: m = multiplet, s = singlet. Melting points were gauged using a Stuart Scientific SMP10 melting point apparatus. Experiments performed at “room temperature” were carried out at 20 °C. Electrochemical experiments were performed as below. Chemical synthesis of 2,5-diacetoxy-2,5-dihydrofuran was carried out by adapting the method of Holzapfel and Williams S[[1]](#endnote-2) (in particular the workup step), which allowed crystals of suitable quality for single crystal X-ray diffraction to be obtained (see below).

**Electrochemical Methods:** Electrochemical studies were performed in a single chamber cell in a three-electrode configuration using a CH Instruments CHI600 series potentiostat. Bulk electrolyses were performed using a large surface area reticulated vitreous carbon electrode (Alvatek Ltd., UK) as the working electrode, a Pt wire pseudo reference electrode, and a graphite rod (99.9995%, Alfa Aesar) as the counter electrode. Working electrodes were washed with water, followed by aqua regia (3:1 mixture of conc. HCl : HNO3), then rinsed with water and acetone prior to use. Counter electrodes were sanded using 800 grit sandpaper to remove surface contamination. They were then sonicated for 1 minute in water to remove any fine graphite particles, followed by a rinsing with water, then acetone. The electrochemical cells were rinsed with aqua regia, water, and acetone prior to use. Electrodes and glassware were dried under a stream of N2 to facilitate removal of any remaining acetone.

A typical cell set-up is shown below. The use of a sealable cell allowed the entire experiment to be conducted under an inert atmosphere of nitrogen. The electrolyte was first transferred to the cell under a constant stream of N2, and the cell was then lowered into an ice bath and the temperature allowed to equilibrate before furan was injected through a septum into the electrolyte. Bulk electrolyses were performed under potentiostatic control with stirring at a potential of 3 V *vs.* Pt (~2 V *vs*. SHE, gauged by obtaining the position of the standard couple potassium ferricyanide in the electrolyte).



***A typical set-up used for the bulk electrolysis experiments reported in this paper.***

**Supplementary Computational Data**

Geometry Optimized Coordinates for Furan

 C -1.80824864713518 1.18328544324190 0.00011953239508

 C -0.37469075882240 1.07750643071161 -0.00010138117473

 C -0.08714155159193 -0.25699783013190 0.00004363692989

 O -1.24815447430304 -0.99513387907364 -0.00001745284793

 C -2.28849117222436 -0.09480797473561 -0.00002977173500

 H -2.40054186472663 2.09347377423108 0.00019764194279

 H 0.34447802827272 1.89125230034324 -0.00021466082149

 H 0.83460133246966 -0.82864952790133 0.00015737735687

 H -3.28399648193884 -0.52511507568535 -0.00015492204549

Total energy = -229.98146206 Eh

Geometry Optimized Coordinates for Furanium Radical

 C -1.37546756220224 1.86058597676796 0.13889118583408

 C -0.03714131176468 1.54050828105590 0.28585559904490

 C 0.06096371172575 0.14666608950156 0.06889551754110

 O -1.16012327287906 -0.38725705425565 -0.20176323860802

 C -2.03947702317764 0.64832551949901 -0.16175055487928

 H -1.85472164882365 2.83044065241948 0.22911335114645

 H 0.79540753431498 2.19692740123955 0.51943193739830

 H 0.89930225904897 -0.54552750576724 0.07886844115849

 H -3.08116149724242 0.40723972953944 -0.35864919163603

Total energy = - 229.75460866 Eh

Geometry Optimized Coordinates for Furanium Dication

 C -1.33914566742290 1.88614291247649 0.14826737183340

 C -0.01885027288570 1.56192073901730 0.29192474887618

 C 0.07493437616790 0.18102023188911 0.07625234416883

 O -1.22207737505797 -0.40048416111189 -0.21513544209537

 C -2.04793883885400 0.57680695469328 -0.17517019715449

 H -1.85107280907828 2.84510155900215 0.23137912572293

 H 0.82601967567981 2.20742060682124 0.52673357764649

 H 0.89668595221618 -0.54211329405187 0.07994508893994

 H -3.11097385176502 0.38209354126420 -0.36530357093793

Total energy = - 229.38755187 Eh

Cartesian Coordinates for **TS1**

 C -1.375467562 1.860585977 0.138891186

 C -0.037141312 1.540508281 0.285855599

 C 0.060963712 0.146666090 0.068895518

 O -1.160123273 -0.387257054 -0.201763239

 C -2.039477023 0.648325520 -0.161750555

 H -1.854721649 2.830440653 0.229113351

 H 0.795407534 2.196927402 0.519431937

 H 0.899302259 -0.545527506 0.078868441

 H -3.081161498 0.407239730 -0.358649192

 O -0.282547581 0.180596687 4.489137538

 C 0.412287991 0.034603866 3.436342144

 O 0.050838055 0.351148158 2.259348593

 C 1.818831422 -0.578341728 3.582481956

 H 2.052505676 -0.826611423 4.625858846

 H 2.574039494 0.126186924 3.202235282

 H 1.894589779 -1.489659884 2.969402387

Total energy = -458.36740651 Eh

Geometry Optimized Coordinates for 2-Acetylfuranyl Radical

 C -1.48638333037037 1.58841764178893 -0.12077957774466

 C -0.12853358175708 1.42749359117565 0.12182256629350

 C 0.10342767444013 -0.00405345718842 0.45770867953988

 O -1.18073897538139 -0.64378169402084 0.28685427106476

 C -2.09306630405854 0.34303081938490 -0.01200107808121

 O 0.44950475087082 -0.16979540934410 1.88277530934526

 C 1.61817801213295 -0.75230583571833 2.29314411663903

 O 1.78689299955278 -0.84711000791430 3.49850958295502

 C 2.61626295304655 -1.23080598736280 1.27149664914237

 H -2.00538789870576 2.51311535810562 -0.35890346927573

 H 0.65445659515682 2.17987695440035 0.11365381044604

 H 0.83607191507893 -0.54741585616931 -0.15011106820177

 H -3.12060059000076 0.01584836218334 -0.13452228712748

 H 2.20229688112145 -2.06299324584646 0.68311677461839

 H 3.50675944514269 -1.58133599664703 1.80204804552251

 H 2.89897389273078 -0.43114814782721 0.57365513786410

Total energy = - 458.40312718 Eh

Geometry Optimized Coordinates for 2-Acetylfuranium

 C -1.59864633197084 1.43612351333527 -0.16920240533906

 C -0.33590203832187 1.67805117649723 0.26236759013765

 C 0.32936426408478 0.39202706687443 0.61446330063384

 O -0.79009702368525 -0.62442315024354 0.40815947019756

 C -1.79898037050747 0.02719911964158 -0.06374233694946

 O 0.78248072616115 0.33411739055909 1.91375882469501

 C 1.80313039457685 -0.58832547073091 2.29290243403328

 O 2.11063885911612 -0.54125003856071 3.45463431840178

 C 2.36932855818910 -1.48689871232631 1.24108874552746

 H -2.33975287217210 2.14261303474712 -0.52999145281193

 H 0.18211070729256 2.63284376706853 0.32981456327194

 H 1.06642437029187 0.09799638280963 -0.15102317838163

 H -2.67971251283210 -0.55797289049481 -0.33700105845649

 H 1.58433050230597 -2.09457154224282 0.76984962559406

 H 3.10133512919021 -2.14597890086042 1.71713570166993

 H 2.87206207728101 -0.90451365707337 0.45525332077605

Total energy = -458.23565691 Eh

Cartesian Coordinates for **TS2-t**

 C -1.592972418 1.435963281 -0.185853997

 C -0.330228111 1.677890943 0.245716036

 C 0.335038203 0.391866833 0.597811766

 O -0.784423090 -0.624583384 0.391507902

 C -1.793306452 0.027038887 -0.080393935

 O 0.788154704 0.333957156 1.897107304

 C 1.808804384 -0.588485706 2.276250943

 O 2.116312883 -0.541410273 3.437982837

 C 2.375002517 -1.487058948 1.224437271

 H -2.334078970 2.142452803 -0.546643066

 H 0.187784635 2.632683533 0.313163025

 H 1.072098286 0.097836147 -0.167674691

 H -2.674038602 -0.558133123 -0.353652683

 H 1.590004448 -2.094731778 0.753198127

 H 3.107009103 -2.146139137 1.700484249

 H 2.877736013 -0.904673894 0.438601861

 O -1.068454912 -0.421987390 -2.108438770

 C -1.235884475 -0.735793852 -3.327445498

 O -0.310424258 -0.975885944 -4.165323953

 C -2.681709351 -0.838698502 -3.851293864

 H -3.418921816 -0.613720773 -3.069769500

 H -2.866484096 -1.851789192 -4.239607878

 H -2.824486382 -0.143028087 -4.692578657

Total energy = -686.85446843 Eh

Cartesian Coordinates for **TS2-c**

 C -1.592972418 1.435963281 -0.185853997

 C -0.330228111 1.677890943 0.245716036

 C 0.335038203 0.391866833 0.597811766

 O -0.784423090 -0.624583384 0.391507902

 C -1.793306452 0.027038887 -0.080393935

 O 0.788154704 0.333957156 1.897107304

 C 1.808804384 -0.588485706 2.276250943

 O 2.116312883 -0.541410273 3.437982837

 C 2.375002517 -1.487058948 1.224437271

 H -2.334078970 2.142452803 -0.546643066

 H 0.187784635 2.632683533 0.313163025

 H 1.072098286 0.097836147 -0.167674691

 H -2.674038602 -0.558133123 -0.353652683

 H 1.590004448 -2.094731778 0.753198127

 H 3.107009103 -2.146139137 1.700484249

 H 2.877736013 -0.904673894 0.438601861

 O -2.502856248 0.047076030 2.001945553

 C -3.371225715 0.136519633 2.924129082

 O -3.125134195 0.273808893 4.163786430

 C -4.857430412 0.075122171 2.520588834

 H -4.984332836 -0.047676226 1.437217704

 H -5.369219289 0.995278427 2.841011168

 H -5.350742721 -0.761841022 3.038506451

Total energy = -686.84963368 Eh

Geometry Optimized Coordinates for *trans*-2,5-diacetoxy-2,5-dihydrofuran

 C -1.16188681483011 2.65806055702918 0.19892864367968

 C 0.05182559377903 2.31088361174873 0.62487245204403

 C 0.22004810117031 0.82573265673963 0.50748459281452

 O -1.00190255822555 0.34751159159462 -0.05584491881233

 C -1.89235195709133 1.44053536908628 -0.28377561942716

 O -2.12153097839456 1.59173821661760 -1.71125148254906

 H -1.59963616924252 3.65332550017193 0.16526031364183

 H 0.83230191709904 2.95769394115257 1.01952259951963

 H 1.06879945816188 0.50646525977400 -0.11267122488642

 H -2.85273483840374 1.25132453923867 0.21428057646828

 O 0.38359459222412 0.28351624602545 1.84576538321577

 C 0.85098649986012 -0.99764467629276 2.02830794761875

 O 0.94032612998005 -1.38642332075066 3.17950314068857

 C 1.21637357941986 -1.82057183906698 0.82353567545207

 C -3.01955522145056 0.77933085279274 -2.36435273258420

 C -3.79146634082239 -0.24081512336656 -1.57279276957992

 O -3.13513636484826 0.94850493928437 -3.56528771377764

 H 0.37430841387465 -1.89115687226279 0.12143763837446

 H 1.50164729279713 -2.82128458417193 1.16020325857485

 H 2.06313323881584 -1.36685907085769 0.28750801847888

 H -4.47235688952017 0.25678174074110 -0.86613273369490

 H -3.12138790205308 -0.88753223223116 -0.99095506833081

 H -4.38041440429974 -0.84595413899631 -2.26859095692887

Total energy = -686.90202676 Eh

Geometry Optimized Coordinates for *cis*-2,5-diacetoxy-2,5-dihydrofuran

 C -1.29787539536271 2.16900305239171 -0.58160221923907

 C -0.02424253862330 1.96484979642927 -0.24575811227983

 C 0.15351279872392 0.54558664654466 0.20407072056486

 O -1.10869404050805 -0.08466387900383 -0.02223678851610

 C -2.07578631396932 0.90205925658421 -0.38448640558030

 O -3.02802894834537 1.12475419869815 0.69163180327801

 H -1.75940549603715 3.09050698697197 -0.92897371462735

 H 0.79369627511426 2.68216374184082 -0.25419667597166

 H 0.92148248901055 -0.01755309955241 -0.34068226920453

 H -2.61241662467693 0.54720830107437 -1.27331818807751

 O 0.48977762539329 0.56548462911073 1.61857463924666

 C 0.99919429639763 -0.55480127401026 2.23338159085233

 O 1.24051441507686 -0.46257567706722 3.42398784908668

 C 1.22322585504136 -1.79808324677748 1.41680715631590

 C -4.08802466723540 0.26868299722569 0.87837965056345

 O -4.82214685439851 0.50643476235507 1.82138196737499

 C -4.27902829280533 -0.87863426066729 -0.07562988799449

 H 0.30609247228562 -2.10134160072251 0.89446238211558

 H 1.54881495911764 -2.59869153340447 2.08759936719740

 H 2.00183711525004 -1.62778862439241 0.65833825697609

 H -5.11087294302964 -1.49251544287013 0.28328701602399

 H -3.37150731428548 -1.49234843182660 -0.15387382097079

 H -4.52034773713398 -0.50582323493207 -1.08191811213430

Total energy = -686.90273226 Eh

**Additional single crystal crystallographic data for *cis-*2,5-diacetoxy-2,5-dihydrofuran**



View showing the structure of *cis-*2,5-diacetoxy-2,5-dihydrofuran. Displacement ellipsoids drawn at 50% probability level.

**Refinement**

Crystal data, data collection and structure refinement details are summarized in Table 1.

**Computing details**

Data collection: *APEX3* Ver. 2016.9-0 (Bruker-AXS, 2016); cell refinement: *SAINT* V8.37A (Bruker-AXS, 2016); data reduction: *APEX3* Ver. 2016.9-0 (Bruker-AXS, 2016); program(s) used to solve structure: XT (Sheldrick, 2015); program(s) used to refine structure: XL (Sheldrick, 2015); molecular graphics: Olex2 (Dolomanov *et al.*, 2009); software used to prepare material for publication: Olex2 (Dolomanov *et al.*, 2009).

**References**

Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.S[[2]](#endnote-3)

Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3-8.S[[3]](#endnote-4)

Sheldrick, G. M. (2015). *Acta Cryst.* A**71**, 3–8.S[[4]](#endnote-5)

**(2018gu0020\_150k\_r1)**

*Crystal data*

|  |  |
| --- | --- |
|  C8H10O5 | *F*(000) = 392 |
| *Mr* = 186.16 | *D*x = 1.381 Mg m-3 |
| Monoclinic, *P*21/*c* | Mo *K* radiation,  = 0.71073 Å |
| *a* = 11.7447 (17) Å | Cell parameters from 5942 reflections |
| *b* = 9.7503 (12) Å |  = 2.7–25.3° |
| *c* = 7.8734 (10) Å |  = 0.12 mm-1 |
|  = 96.676 (5)° | *T* = 150 K |
| *V* = 895.5 (2) Å3 | Block, colourless |
| *Z* = 4 | 0.32 × 0.2 × 0.11 mm |

*Data collection*

|  |  |
| --- | --- |
|  Bruker D8 VENTURE diffractometer | 1631 independent reflections |
| Radiation source: microfocus sealed tube, INCOATEC Is 3.0 | 1517 reflections with *I* > 2(*I*) |
| Multilayer mirror optics monochromator | *R*int = 0.025 |
| Detector resolution: 7.4074 pixels mm-1 | max = 25.3°, min = 2.7° |
|  and  scans | *h* = -1214 |
| Absorption correction: multi-scan *SADABS2016*/2 (Bruker,2016/2) was used for absorption correction. wR2(int) was 0.1582 before and 0.0643 after correction. The Ratio of minimum to maximum transmission is 0.8875. The /2 correction factor is Not present. | *k* = -1111 |
| *T*min = 0.661, *T*max = 0.745 | *l* = -99 |
| 6125 measured reflections |  |

*Refinement*

|  |  |
| --- | --- |
|  Refinement on *F*2 | Primary atom site location: dual |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| *R*[*F*2 > 2(*F*2)] = 0.040 | H-atom parameters constrained |
| *wR*(*F*2) = 0.107 |  *w* = 1/[2(*F*o2) + (0.0445*P*)2 + 0.554*P*] where *P* = (*F*o2 + 2*F*c2)/3 |
| *S* = 1.10 | (/)max = 0.001 |
| 1631 reflections | max = 0.36 e Å-3 |
| 120 parameters | min = -0.25 e Å-3 |
| 0 restraints |  |

*Special details*

|  |
| --- |
|  *Geometry*. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes. |

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å2)*

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
|  | *x* | *y* | *z* | *U*iso\*/*U*eq |
| O1 | 0.27502 (10) | 0.45847 (11) | 0.20054 (14) | 0.0260 (3) |
| O2 | 0.39227 (9) | 0.65113 (11) | 0.25709 (14) | 0.0268 (3) |
| O3 | 0.54271 (11) | 0.51178 (13) | 0.23740 (17) | 0.0367 (3) |
| O4 | 0.14957 (10) | 0.52238 (11) | 0.39486 (14) | 0.0270 (3) |
| O5 | 0.11600 (11) | 0.30235 (12) | 0.45999 (17) | 0.0364 (3) |
| C2 | 0.15928 (14) | 0.48707 (17) | 0.2187 (2) | 0.0249 (4) |
| H2 | 0.108113 | 0.408574 | 0.179267 | 0.030\* |
| C3 | 0.13112 (14) | 0.61265 (17) | 0.1134 (2) | 0.0278 (4) |
| H3 | 0.056790 | 0.651233 | 0.088365 | 0.033\* |
| C4 | 0.22511 (15) | 0.66125 (17) | 0.0609 (2) | 0.0282 (4) |
| H4 | 0.229359 | 0.740379 | -0.008498 | 0.034\* |
| C5 | 0.32422 (14) | 0.57320 (17) | 0.1268 (2) | 0.0255 (4) |
| H5 | 0.370093 | 0.545145 | 0.033274 | 0.031\* |
| C6 | 0.50173 (14) | 0.60878 (17) | 0.3021 (2) | 0.0256 (4) |
| C7 | 0.56068 (16) | 0.69836 (19) | 0.4384 (2) | 0.0352 (4) |
| H7A | 0.529621 | 0.791561 | 0.425588 | 0.053\* |
| H7B | 0.643053 | 0.700262 | 0.428329 | 0.053\* |
| H7C | 0.548128 | 0.662185 | 0.550831 | 0.053\* |
| C8 | 0.13694 (13) | 0.41800 (17) | 0.5055 (2) | 0.0252 (4) |
| C9 | 0.15346 (15) | 0.46831 (19) | 0.6857 (2) | 0.0317 (4) |
| H9A | 0.233709 | 0.494960 | 0.715955 | 0.048\* |
| H9B | 0.133661 | 0.395190 | 0.762455 | 0.048\* |
| H9C | 0.103850 | 0.547820 | 0.696982 | 0.048\* |

*Atomic displacement parameters (Å2)*

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
|  | *U*11 | *U*22 | *U*33 | *U*12 | *U*13 | *U*23 |
| O1 | 0.0276 (6) | 0.0195 (6) | 0.0308 (6) | 0.0027 (4) | 0.0032 (5) | 0.0007 (5) |
| O2 | 0.0262 (6) | 0.0251 (6) | 0.0292 (6) | 0.0009 (5) | 0.0033 (5) | -0.0041 (5) |
| O3 | 0.0332 (7) | 0.0334 (7) | 0.0433 (7) | 0.0074 (5) | 0.0034 (6) | -0.0061 (6) |
| O4 | 0.0352 (7) | 0.0214 (6) | 0.0247 (6) | -0.0035 (5) | 0.0048 (5) | -0.0029 (5) |
| O5 | 0.0470 (8) | 0.0230 (6) | 0.0390 (7) | -0.0075 (5) | 0.0043 (6) | -0.0005 (5) |
| C2 | 0.0268 (8) | 0.0240 (8) | 0.0235 (8) | -0.0005 (6) | 0.0011 (6) | -0.0037 (6) |
| C3 | 0.0286 (9) | 0.0293 (9) | 0.0244 (8) | 0.0058 (7) | -0.0025 (6) | -0.0033 (7) |
| C4 | 0.0385 (10) | 0.0247 (8) | 0.0210 (8) | 0.0045 (7) | 0.0013 (7) | 0.0022 (7) |
| C5 | 0.0301 (9) | 0.0242 (8) | 0.0225 (8) | 0.0001 (7) | 0.0044 (6) | -0.0013 (6) |
| C6 | 0.0258 (8) | 0.0245 (8) | 0.0273 (8) | -0.0004 (7) | 0.0068 (7) | 0.0044 (7) |
| C7 | 0.0363 (10) | 0.0316 (9) | 0.0363 (10) | -0.0021 (8) | -0.0019 (7) | -0.0005 (8) |
| C8 | 0.0194 (8) | 0.0264 (9) | 0.0299 (8) | -0.0008 (6) | 0.0033 (6) | 0.0014 (7) |
| C9 | 0.0330 (9) | 0.0347 (10) | 0.0277 (9) | -0.0037 (7) | 0.0050 (7) | 0.0008 (7) |

*Geometric parameters (Å, º) for (2018gu0020\_150k\_r1)*

|  |  |  |  |
| --- | --- | --- | --- |
| O1—C2 | 1.4110 (19) | C4—H4 | 0.9500 |
| O1—C5 | 1.414 (2) | C4—C5 | 1.490 (2) |
| O2—C5 | 1.4413 (19) | C5—H5 | 1.0000 |
| O2—C6 | 1.358 (2) | C6—C7 | 1.490 (2) |
| O3—C6 | 1.201 (2) | C7—H7A | 0.9800 |
| O4—C2 | 1.4463 (19) | C7—H7B | 0.9800 |
| O4—C8 | 1.359 (2) | C7—H7C | 0.9800 |
| O5—C8 | 1.200 (2) | C8—C9 | 1.493 (2) |
| C2—H2 | 1.0000 | C9—H9A | 0.9800 |
| C2—C3 | 1.494 (2) | C9—H9B | 0.9800 |
| C3—H3 | 0.9500 | C9—H9C | 0.9800 |
| C3—C4 | 1.312 (2) |  |  |
|  |  |  |  |
| C2—O1—C5 | 109.25 (12) | C4—C5—H5 | 111.5 |
| C6—O2—C5 | 117.09 (12) | O2—C6—C7 | 110.87 (14) |
| C8—O4—C2 | 117.57 (12) | O3—C6—O2 | 122.87 (15) |
| O1—C2—O4 | 109.18 (12) | O3—C6—C7 | 126.26 (16) |
| O1—C2—H2 | 111.6 | C6—C7—H7A | 109.5 |
| O1—C2—C3 | 105.03 (13) | C6—C7—H7B | 109.5 |
| O4—C2—H2 | 111.6 | C6—C7—H7C | 109.5 |
| O4—C2—C3 | 107.46 (13) | H7A—C7—H7B | 109.5 |
| C3—C2—H2 | 111.6 | H7A—C7—H7C | 109.5 |
| C2—C3—H3 | 125.3 | H7B—C7—H7C | 109.5 |
| C4—C3—C2 | 109.32 (15) | O4—C8—C9 | 110.57 (14) |
| C4—C3—H3 | 125.3 | O5—C8—O4 | 123.13 (15) |
| C3—C4—H4 | 125.2 | O5—C8—C9 | 126.30 (16) |
| C3—C4—C5 | 109.51 (15) | C8—C9—H9A | 109.5 |
| C5—C4—H4 | 125.2 | C8—C9—H9B | 109.5 |
| O1—C5—O2 | 110.14 (12) | C8—C9—H9C | 109.5 |
| O1—C5—C4 | 105.00 (13) | H9A—C9—H9B | 109.5 |
| O1—C5—H5 | 111.5 | H9A—C9—H9C | 109.5 |
| O2—C5—C4 | 106.87 (13) | H9B—C9—H9C | 109.5 |
| O2—C5—H5 | 111.5 |  |  |
|  |  |  |  |
| O1—C2—C3—C4 | -8.05 (18) | C5—O1—C2—O4 | -101.54 (14) |
| O4—C2—C3—C4 | 108.12 (15) | C5—O1—C2—C3 | 13.45 (16) |
| C2—O1—C5—O2 | 101.13 (14) | C5—O2—C6—O3 | 0.7 (2) |
| C2—O1—C5—C4 | -13.59 (16) | C5—O2—C6—C7 | -179.57 (13) |
| C2—O4—C8—O5 | -11.8 (2) | C6—O2—C5—O1 | 84.18 (16) |
| C2—O4—C8—C9 | 167.82 (13) | C6—O2—C5—C4 | -162.30 (13) |
| C2—C3—C4—C5 | -0.26 (18) | C8—O4—C2—O1 | -86.10 (16) |
| C3—C4—C5—O1 | 8.45 (17) | C8—O4—C2—C3 | 160.49 (13) |
| C3—C4—C5—O2 | -108.53 (15) |  |  |

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