Supporting Information

Photoinitiated Thiol–Ene Reactions of Various 2,3- Unsaturated O-, C- S- and N-Glycosides – Scope and Limitations Study

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X-Ray Crystallographic Data

Computing details

Data collection: Bruker APEX3; cell refinement: Bruker APEX3; data reduction: Bruker Saint; program(s) used to solve structure: SHELXL2016/4 (Sheldrick, 2016); program(s) used to refine structure: SHELXL2016/4 (Sheldrick, 2016); molecular graphics: Mercury; software used to prepare material for publication: Bruker APEX3, WinGX.

(Compound 31)

Crystal data

| C_{26}H_{38}O_{15}S | F(000) = 660 |
|-------------------|--------------|
| M_r = 622.62      | D_x = 1.286 Mg m^{-3} |
| Monoclinic, P2_1  | Cu Kα radiation, λ = 1.54178 Å |
| a = 15.8457 (9) Å | Cell parameters from 318 reflections |
| b = 5.5284 (4) Å  | θ = 28.1–78.6° |
| c = 18.9922 (12) Å | µ = 1.48 mm^{-1} |
| β = 104.967 (5)^{\circ} | T = 280 K |
| V = 1607.30 (18) Å\(^3\) | Needle, colourless |
| Z = 2             | 0.11 × 0.04 × 0.02 mm |

Data collection

| Bruker D8 Venture diffractometer | 3979 reflections with I > 2σ(I) |
|----------------------------------|---------------------------------|
| ω scan                          | R_{int} = 0.054 |
| Absorption correction: multi-scan SADABS 2016/2: Krause, L., Herbst-Irmer, R., Sheldrick G.M. & Stalke D., J. Appl. Cryst. 48 (2015) 3-10 | θ_{max} = 66.6°, θ_{min} = 2.4° |
| T_{min} = 0.662, T_{max} = 0.753 | h = -15→18 |
| 16876 measured reflections      | k = -6→6 |
| 5664 independent reflections    | l = -22→22 |

Refinement

| Refinement on F^2 | Secondary atom site location: difference Fourier map |
|-------------------|-----------------------------------------------------|
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| R[F^2 > 2σ(F^2)] = 0.049 | H-atom parameters constrained |
| wR(F^2) = 0.134 | w = 1/[σ^2(F_o^2) + (0.0582P)^2] where P = (F_o^2 + 2F_c^2)/3 |
| S = 1.06 | (Δσ)_{max} < 0.001 |
5664 reflections
386 parameters
1 restraint
Primary atom site location: structure-invariant direct methods

Absolute structure: Flack x determined using 1308 quotients \[(I^+)-(I^-)/[(I^+)+(I^-)]\] (Parsons, Flack and Wagner, Acta Cryst. B69 (2013) 249-259).

Absolute structure parameter: 0.040 (17)

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) for (31)

|   | x    | y    | z    | \(U_{eq}\) |
|---|------|------|------|------------|
| C1 | 0.2891 (3) | 0.4459 (12) | 0.2850 (3) | 0.0807 (15) |
| H1 | 0.307276 | 0.338160 | 0.327207 | 0.097* |
| C1' | 0.5473 (3) | 0.5753 (10) | 0.3013 (2) | 0.0657 (12) |
| H1' | 0.531963 | 0.734118 | 0.317256 | 0.079* |
| C2' | 0.6304 (3) | 0.4814 (10) | 0.3549 (2) | 0.0636 (11) |
| H2' | 0.644919 | 0.320494 | 0.339836 | 0.076* |
| C2 | 0.3684 (3) | 0.5871 (11) | 0.2762 (3) | 0.0716 (13) |
| H2 | 0.382340 | 0.708318 | 0.315123 | 0.086* |
| C3 | 0.3484 (3) | 0.7224 (10) | 0.2037 (3) | 0.0730 (13) |
| H3A | 0.314363 | 0.865292 | 0.207566 | 0.088* |
| H3B | 0.403031 | 0.776019 | 0.194807 | 0.088* |
| C3' | 0.7060 (3) | 0.6532 (10) | 0.3588 (3) | 0.0664 (12) |
| C4 | 0.7159 (3) | 0.7140 (9) | 0.2834 (3) | 0.0678 (12) |
| H4 | 0.277606 | 0.681902 | 0.097726 | 0.081* |
| C5 | 0.2994 (3) | 0.5746 (10) | 0.1396 (3) | 0.0678 (12) |
| H5 | 0.607828 | 0.936060 | 0.247296 | 0.085* |
| C6 | 0.2224 (3) | 0.4427 (10) | 0.1566 (3) | 0.0715 (13) |
| H6C | 0.576571 | 0.833606 | 0.120903 | 0.093* |
| Atom  | X       | Y       | Z       | Uiso  |
|-------|---------|---------|---------|-------|
| H6D   | 0.668600| 0.956179| 0.149244| 0.093*|
| C6    | 0.1795 (3)| 0.2673 (12)| 0.0985 (3)| 0.0814 (15)|
| H6A   | 0.167605| 0.344169| 0.051037| 0.098*|
| H6B   | 0.217415| 0.129588| 0.098533| 0.098*|
| C11   | 0.1542 (5)| 0.511 (2)| 0.3172 (6)| 0.169 (5)|
| H11A  | 0.118427| 0.428542| 0.274808| 0.203*|
| H11B  | 0.173839| 0.391332| 0.355410| 0.203*|
| C12   | 0.1070 (8)| 0.676 (4)| 0.3394 (9)| 0.268 (10)|
| H12A  | 0.139335| 0.739932| 0.385439| 0.402*|
| H12B  | 0.053848| 0.603786| 0.344771| 0.402*|
| H12C  | 0.093289| 0.803887| 0.304178| 0.402*|
| C21'  | 0.6052 (4)| 0.2534 (12)| 0.4544 (3)| 0.0802 (14)|
| C22'  | 0.5982 (6)| 0.2745 (18)| 0.5310 (4)| 0.125 (3)|
| H22D  | 0.539302| 0.315482| 0.530817| 0.187*|
| H22E  | 0.637107| 0.398508| 0.555715| 0.187*|
| H22F  | 0.613703| 0.122967| 0.555609| 0.187*|
| C31'  | 0.8393 (4)| 0.6425 (18)| 0.4540 (4)| 0.110 (2)|
| C32'  | 0.9140 (5)| 0.4819 (19)| 0.4876 (5)| 0.143 (4)|
| H32D  | 0.894030| 0.317459| 0.485826| 0.215*|
| H32E  | 0.938187| 0.528704| 0.537358| 0.215*|
| H32F  | 0.957896| 0.495581| 0.461180| 0.215*|
| C41'  | 0.8523 (3)| 0.8971 (12)| 0.2800 (4)| 0.0886 (16)|
| C41   | 0.4099 (3)| 0.4669 (10)| 0.0812 (3)| 0.0696 (12)|
| C42   | 0.4686 (4)| 0.2681 (11)| 0.0716 (3)| 0.0858 (16)|
| H42A  | 0.495656| 0.309454| 0.033494| 0.129*|
| H42B  | 0.435259| 0.122185| 0.058846| 0.129*|
| H42C  | 0.512770| 0.243693| 0.116281| 0.129*|
| C42'  | 0.9022 (4)| 1.1217 (14)| 0.2899 (5)| 0.115 (2)|
| H42D  | 0.962771| 1.086151| 0.295047| 0.172*|
| H42E  | 0.895653| 1.202604| 0.332907| 0.172*|
| H42F  | 0.880934| 1.224175| 0.248291| 0.172*|
| C61   | 0.0629 (4)| -0.0100 (14)| 0.0813 (3)| 0.0908 (18)|
| C61'  | 0.7319 (4)| 0.6334 (11)| 0.0919 (3)| 0.0797 (15)|
| C62   | 0.7702 (5)| 0.4077 (13)| 0.0759 (4)| 0.104 (2)|
| H62D  | 0.724969| 0.303200| 0.048948| 0.156*|
| H62E  | 0.799975| 0.330435| 0.120747| 0.156*|
| H62F  | 0.811102| 0.440500| 0.047604| 0.156*|
| C62   | -0.0189 (4)| -0.076 (2)| 0.1005 (4)| 0.130 (3)|
| H62A  | -0.066616| -0.073624| 0.057427| 0.195*|
| H62B  | -0.029887| 0.037106| 0.135327| 0.195*|
|    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |
|----|----|----|----|----|----|----|----|----|----|----|----|----|----|----|
| H62C | -0.013238 | -0.235967 | 0.121170 | 0.195* |
| O1  | 0.2294 (3)  | 0.6170 (10) | 0.2989 (2) | 0.1044 (14) |
| O2' | 0.6189 (2)  | 0.4707 (7)  | 0.42777 (18) | 0.0738 (9) |
| O3' | 0.7845 (2)  | 0.5303 (7)  | 0.3991 (2)  | 0.0809 (10) |
| O4' | 0.7719 (2)  | 0.9226 (6)  | 0.2891 (2)  | 0.0786 (10) |
| O4  | 0.35493 (19) | 0.3922 (6)  | 0.12043 (16) | 0.0659 (8) |
| O5' | 0.5662 (2)  | 0.5972 (7)  | 0.23182 (17) | 0.0710 (9) |
| O5  | 0.2509 (2)  | 0.3054 (7)  | 0.22239 (18) | 0.0747 (10) |
| O6' | 0.6748 (2)  | 0.6058 (7)  | 0.1321 (2)  | 0.0786 (9) |
| O6  | 0.0992 (2)  | 0.1896 (10) | 0.1134 (2)  | 0.0999 (14) |
| O21' | 0.5981 (3)  | 0.0681 (9)  | 0.4199 (3)  | 0.1032 (13) |
| O31' | 0.8285 (5)  | 0.8446 (16) | 0.4710 (5)  | 0.193 (4) |
| O41  | 0.4115 (3)  | 0.6684 (8)  | 0.0600 (2)  | 0.0905 (11) |
| O41' | 0.8782 (3)  | 0.7065 (11) | 0.2649 (5)  | 0.168 (3) |
| O61  | 0.0935 (3)  | -0.1235 (11) | 0.0398 (3) | 0.1197 (16) |
| O61' | 0.7467 (5)  | 0.8279 (11) | 0.0712 (4)  | 0.163 (3) |
| S1   | 0.45800 (8) | 0.3721 (3)  | 0.29321 (7) | 0.0735 (3) |

**Atomic displacement parameters (Å²) for (31)**

|    | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|----|----------|----------|----------|----------|----------|----------|
| C1  | 0.067 (3) | 0.107 (5) | 0.069 (3) | 0.004 (3) | 0.019 (2) | -0.006 (3) |
| C1' | 0.064 (3) | 0.077 (3) | 0.057 (2) | 0.003 (2) | 0.017 (2) | -0.001 (2) |
| C2' | 0.071 (3) | 0.066 (3) | 0.054 (2) | 0.000 (2) | 0.016 (2) | -0.003 (2) |
| C2  | 0.068 (3) | 0.085 (3) | 0.064 (3) | -0.002 (3) | 0.020 (2) | -0.013 (3) |
| C3  | 0.076 (3) | 0.066 (3) | 0.078 (3) | 0.008 (2) | 0.022 (2) | -0.009 (3) |
| C3' | 0.064 (3) | 0.068 (3) | 0.064 (3) | 0.003 (2) | 0.009 (2) | -0.003 (2) |
| C4' | 0.068 (3) | 0.058 (3) | 0.078 (3) | 0.000 (2) | 0.020 (2) | 0.001 (2) |
| C4  | 0.076 (3) | 0.064 (3) | 0.064 (3) | 0.017 (2) | 0.020 (2) | 0.003 (2) |
| C5  | 0.071 (3) | 0.070 (3) | 0.072 (3) | 0.003 (2) | 0.021 (2) | 0.001 (2) |
| C5' | 0.062 (3) | 0.085 (4) | 0.066 (3) | 0.009 (2) | 0.015 (2) | 0.001 (2) |
| C6  | 0.068 (3) | 0.103 (4) | 0.073 (3) | -0.004 (3) | 0.019 (2) | -0.009 (3) |
| C11 | 0.096 (5) | 0.266 (15) | 0.171 (8) | -0.005 (7) | 0.081 (6) | -0.045 (9) |
| C12 | 0.152 (9) | 0.37 (2)  | 0.33 (2)  | 0.007 (13) | 0.150 (12) | -0.099 (19) |
| C21' | 0.087 (4) | 0.080 (4) | 0.076 (3) | 0.009 (3) | 0.024 (3) | 0.013 (3) |
| C22' | 0.156 (6) | 0.149 (7) | 0.074 (4) | -0.008 (6) | 0.036 (4) | 0.025 (4) |
| C31' | 0.088 (4) | 0.126 (7) | 0.096 (5) | -0.011 (4) | -0.010 (3) | -0.016 (5) |
| C32' | 0.091 (4) | 0.177 (9) | 0.129 (6) | -0.013 (5) | -0.031 (4) | 0.030 (6) |
| C41' | 0.070 (3) | 0.072 (4) | 0.127 (5) | 0.001 (3) | 0.031 (3) | 0.002 (4) |
| C41 | 0.076 (3) | 0.072 (3) | 0.063 (3) | -0.002 (3) | 0.022 (2) | 0.003 (2) |
|      |     |     |     |     |     |
|------|-----|-----|-----|-----|-----|
| C42  | 0.090 (4) | 0.081 (4) | 0.096 (4) | -0.001 (3) | 0.040 (3) |
| C42' | 0.094 (4) | 0.090 (5) | 0.159 (7) | -0.017 (4) | 0.032 (4) |
| C61  | 0.077 (3) | 0.115 (5) | 0.077 (4) | -0.008 (3) | 0.012 (3) |
| C61' | 0.088 (3) | 0.074 (4) | 0.086 (4) | -0.005 (3) | 0.037 (3) |
| C62  | 0.135 (5) | 0.087 (4) | 0.106 (5) | 0.000 (4) | 0.057 (4) |
| C62' | 0.094 (4) | 0.090 (5) | 0.159 (7) | -0.017 (4) | 0.032 (4) |
| C61  | 0.077 (3) | 0.115 (5) | 0.077 (4) | -0.008 (3) | 0.012 (3) |
| C61' | 0.088 (3) | 0.074 (4) | 0.086 (4) | -0.005 (3) | 0.037 (3) |
| C62  | 0.135 (5) | 0.087 (4) | 0.106 (5) | 0.000 (4) | 0.057 (4) |
| C62' | 0.094 (4) | 0.090 (5) | 0.159 (7) | -0.017 (4) | 0.032 (4) |

**Geometric parameters (Å, º) for (31)**

|      |     |     |     |     |     |
|------|-----|-----|-----|-----|-----|
| C1—O1 | 1.410 (7) | C11—H11B | 0.9700 |
| C1—O5 | 1.418 (7) | C12—H12A | 0.9600 |
| C1—C2 | 1.525 (7) | C12—H12B | 0.9600 |
| C1—H1 | 0.9800 | C12—H12C | 0.9600 |
| C1'—O5' | 1.431 (5) | C21'—O21' | 1.205 (8) |
| C1'—C2' | 1.532 (7) | C21'—O2'' | 1.343 (7) |
| C1'—S1 | 1.782 (5) | C21'—C22' | 1.492 (9) |
| C1'—H1' | 0.9800 | C22'—H22D | 0.9600 |
| C2'—O2' | 1.443 (6) | C22'—H22E | 0.9600 |
| C2'—C3' | 1.516 (7) | C22'—H22F | 0.9600 |
| C2'—H2' | 0.9800 | C31'—O31' | 1.187 (11) |
| C2—C3 | 1.526 (8) | C31'—O3' | 1.326 (8) |
| C2—S1 | 1.816 (5) | C31'—C32' | 1.485 (12) |
| C2—H2 | 0.9800 | C32'—H32D | 0.9600 |
| C3—C4 | 1.506 (7) | C32'—H32E | 0.9600 |
| C3—H3A | 0.9700 | C32'—H32F | 0.9600 |
| Bond                  | Distance (Å) | Bond                  | Distance (Å) |
|----------------------|--------------|----------------------|--------------|
| C3—H3B               | 0.9700       | C41'—O41'            | 1.192 (8)    |
| C3'—O3'              | 1.450 (6)    | C41'—O4'             | 1.337 (6)    |
| C3'—C4'              | 1.519 (7)    | C41'—C42'            | 1.457 (10)   |
| C3'—H3'              | 0.9800       | C41—O41              | 1.187 (6)    |
| C4'—O4'              | 1.442 (6)    | C41—O4               | 1.349 (6)    |
| C4'—C5'              | 1.534 (7)    | C41—C42              | 1.481 (6)    |
| C4'—H4'              | 0.9800       | C42—H42A             | 0.9600       |
| C4—C5                | 1.526 (7)    | C42—H42B             | 0.9600       |
| C4—H4                | 0.9800       | C42—H42C             | 0.9600       |
| C5'—O5'              | 1.431 (6)    | C42'—H42E            | 0.9600       |
| C5'—C6'              | 1.515 (7)    | C42'—H42F            | 0.9600       |
| C5'—H5'              | 0.9800       | C61—O61              | 1.204 (8)    |
| C5—O5                | 1.431 (6)    | C61—O6               | 1.318 (8)    |
| C5—C6                | 1.494 (8)    | C61—C62              | 1.480 (9)    |
| C5—H5                | 0.9800       | C61'—O61'            | 1.188 (8)    |
| C6'—O6'              | 1.419 (7)    | C61'—O6'             | 1.334 (6)    |
| C6'—H6C              | 0.9700       | C61'—C62'            | 1.453 (9)    |
| C6'—H6D              | 0.9700       | C62'—H62D            | 0.9600       |
| C6—H6A               | 1.438 (6)    | C62'—H62E            | 0.9600       |
| C6—H6B               | 0.9700       | C62—H62A             | 0.9600       |
| C11—C12              | 1.317 (16)   | C62—H62B             | 0.9600       |
| C11—O1               | 1.450 (9)    | C62—H62C             | 0.9600       |
| C11—H11A             | 0.9700       |                      |              |
| O1—C1—O5             | 111.3 (4)    | O1—C11—H11A         | 109.3        |
| O1—C1—C2             | 106.7 (5)    | C12—C11—H11B        | 109.3        |
| O5—C1—C2             | 111.9 (4)    | O1—C11—H11B         | 109.3        |
| O1—C1—H1             | 108.9        | H11A—C11—H11B       | 108.0        |
| O5—C1—H1             | 108.9        | C11—C12—H12A        | 109.5        |
| C2—C1—H1             | 108.9        | C11—C12—H12B        | 109.5        |
| O5'—C1'—C2'          | 107.0 (3)    | H12A—C12—H12B       | 109.5        |
| O5'—C1'—S1           | 109.0 (3)    | C11—C12—H12C        | 109.5        |
| C2'—C1'—S1           | 111.8 (4)    | H12A—C12—H12C       | 109.5        |
| O5'—C1'—H1'          | 109.7        | H12B—C12—H12C       | 109.5        |
| C2'—C1'—H1'          | 109.7        | O21'—C21'—O2'       | 123.9 (5)    |
| S1—C1'—H1'           | 109.7        | O21'—C21'—C22'      | 125.1 (6)    |
| O2'—C2'—C3'          | 105.8 (4)    | O2'—C21'—C22'       | 110.9 (6)    |
| O2'—C2'—C1'          | 110.6 (4)    | C21'—C22'—H22D      | 109.5        |
| Bond       | Angle (°) | Bond       | Angle (°) |
|------------|-----------|------------|-----------|
| C3'—C2'—C1' | 110.7 (4) | C21'—C22'—H22E | 109.5 |
| O2'—C2'—H2' | 109.9 | H22D—C22'—H22E | 109.5 |
| C3'—C2'—H2' | 109.9 | C21'—C22'—H22F | 109.5 |
| C1'—C2'—H2' | 109.9 | H22D—C22'—H22F | 109.5 |
| C1—C2—C3   | 111.5 (4) | H22E—C22'—H22F | 109.5 |
| C1—C2—S1   | 105.9 (4) | O31'—C31'—O3' | 122.9 (8) |
| C3—C2—S1   | 116.9 (3) | O31'—C31'—C32' | 126.9 (8) |
| C1—C2—H2   | 107.4 | O3'—C31'—C32' | 110.2 (8) |
| C3—C2—H2   | 107.4 | C31'—C32'—H32D | 109.5 |
| S1—C2—H2   | 107.4 | C31'—C32'—H32E | 109.5 |
| C4—C3—C2   | 114.0 (5) | H32D—C32'—H32E | 109.5 |
| C4—C3—H3A  | 108.7 | C31'—C32'—H32F | 109.5 |
| C2—C3—H3A  | 108.7 | H32D—C32'—H32F | 109.5 |
| C4—C3—H3B  | 108.7 | H32E—C32'—H32F | 109.5 |
| C2—C3—H3B  | 108.7 | O41'—C41'—O4' | 121.5 (5) |
| H3A—C3—H3B | 107.6 | O41'—C41'—C42' | 125.1 (5) |
| O3'—C3'—C2' | 110.0 (4) | O4'—C41'—C42' | 113.3 (6) |
| O3'—C3'—C4' | 108.5 (4) | O41'—C41—O4 | 123.2 (5) |
| C2'—C3'—C4' | 111.4 (4) | O41—C41—C42 | 125.8 (5) |
| O3'—C3'—H3' | 110.0 | O4—C41—C42 | 110.9 (5) |
| C2'—C3'—H3' | 110.0 | C41—C42—H42A | 109.5 |
| C4'—C3'—H3' | 110.0 | C41—C42—H42B | 109.5 |
| O4'—C4'—C3' | 108.5 (4) | H42A—C42—H42B | 109.5 |
| O4'—C4'—C5' | 106.4 (4) | C41—C42—H42C | 109.5 |
| C3'—C4'—C5' | 111.8 (4) | H42A—C42—H42C | 109.5 |
| O4'—C4'—H4' | 110.0 | H42B—C42—H42C | 109.5 |
| C3'—C4'—H4' | 110.0 | C41'—C42'—H42D | 109.5 |
| C5'—C4'—H4' | 110.0 | C41'—C42'—H42E | 109.5 |
| O4—C4—C3   | 111.2 (4) | H42D—C42—H42E | 109.5 |
| O4—C4—C5   | 107.0 (4) | C41'—C42'—H42F | 109.5 |
| C3—C4—C5   | 111.2 (4) | H42D—C42—H42F | 109.5 |
| O4—C4—H4   | 109.2 | H42E—C42'—H42F | 109.5 |
| C3—C4—H4   | 109.2 | O61—C61—O6 | 122.5 (6) |
| C5—C4—H4   | 109.2 | O61—C61—C62 | 123.9 (7) |
| O5'—C5'—C6' | 107.9 (4) | O6—C61—C62 | 113.6 (7) |
| O5'—C5'—C4' | 108.6 (4) | O61'—C61'—O6' | 120.8 (6) |
| C6'—C5'—C4' | 112.6 (4) | O61'—C61'—C62' | 125.4 (5) |
| O5'—C5'—H5' | 109.2 | O6—C61'—C62' | 113.7 (5) |
| C6'—C5'—H5' | 109.2 | C61'—C62'—H62D | 109.5 |
| C4'—C5'—H5' | 109.2 | C61'—C62'—H62E | 109.5 |
| Bond  | Distance (Å) | Angle (°)  |
|-------|--------------|------------|
| O5—C5—C6  | 106.4 (5)  | H62D—C62'—H62E  | 109.5 |
| O5—C5—C4  | 110.4 (4)  | C61'—C62'—H62F  | 109.5 |
| C6—C5—C4  | 112.9 (4)  | H62D—C62'—H62F  | 109.5 |
| O5—C5—H5  | 109.0  | H62E—C62'—H62F  | 109.5 |
| C6—C5—H5  | 109.0  | C61—C62—H62A  | 109.5 |
| C4—C5—H5  | 109.0  | C61—C62—H62B  | 109.5 |
| O6'—C6'—C5'  | 109.6 (4)  | H62A—C62—H62B  | 109.5 |
| O6'—C6'—H6C  | 109.7  | C61—C62—H62C  | 109.5 |
| C5'—C6'—H6C  | 109.7  | H62A—C62—H62C  | 109.5 |
| O6'—C6'—H6D  | 109.7  | H62B—C62—H62C  | 109.5 |
| C5'—C6'—H6D  | 109.7  | C1—O1—C11  | 113.9 (7) |
| H6C—C6'—H6D  | 108.2  | C21'—O2'—C2'  | 118.0 (4) |
| O6—C6—C5  | 107.8 (4)  | C31'—O3'—C3'  | 119.6 (5) |
| O6—C6—H6A  | 110.1  | C41'—O4'—C4'  | 119.6 (4) |
| C5—C6—H6A  | 110.1  | C41—O4—C4  | 116.6 (4) |
| O6—C6—H6B  | 110.1  | C5'—O5'—C1'  | 112.4 (4) |
| C5—C6—H6B  | 110.1  | C1—O5—C5  | 114.3 (4) |
| H6A—C6—H6B  | 108.5  | C61—O6—C6'  | 119.3 (4) |
| C12—C11—O1  | 111.5 (11)  | C61—O6—C6  | 117.6 (5) |
| C12—C11—H11A  | 109.3  | C1'—S1—C2  | 99.8 (2) |

**Hydrogen-bond geometry (Å, °) for (31)**

| D—H···A  | D—H  | H···A  | D···A  | D—H···A  |
|----------|-------|--------|--------|----------|
| C3'—H3'···O21'_$1^i$  | 0.98  | 2.38  | 3.249 (7)  | 148  |
| C42—H42B···O41_S2^ii  | 0.96  | 2.54  | 3.428 (7)  | 154  |
| C62F···O61_S3^iii  | 0.96  | 2.54  | 3.460 (8)  | 160  |

Symmetry codes: (i) x, y+1, z; (ii) x, y-1, z; (iii) -x+1, y+1/2, -z.

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S10
$^{1}$H and $^{13}$C NMR spectra of compounds

$^{1}$H and $^{13}$C NMR spectra of compound 19
$^1$H and $^{13}$C NMR spectra of compound 20
$^1$H and $^{13}$C NMR spectra of compound 21
$^1$H and $^{13}$C NMR spectra of compound 22
$^1$H and $^{13}$C NMR spectra of compound 23
$^1$H and $^{13}$C NMR spectra of compound 24
1H and 13C NMR spectra of compound 25
$^1$H and $^{13}$C NMR spectra of compound 26

$^1$H NMR [400 MHz, CDCl$_3$]

$^{13}$C NMR [100 MHz, CDCl$_3$]
$^1$H and $^{13}$C NMR spectra of compound 27
$^1$H and $^{13}$C NMR spectra of compound 29
$^1$H and $^{13}$C NMR spectra of compound 31
$^1$H and $^{13}$C NMR spectra of compound 32
$^1$H and $^{13}$C NMR spectra of compound 33
$^1$H and $^{13}$C NMR spectra of compound 34
$^1$H and $^{13}$C NMR spectra of compound 36
$^1$H and $^{13}$C NMR spectra of compound 37
$^1$H and $^{13}$C NMR spectra of compound 38
$^1$H and $^{13}$C NMR spectra of compound 39
$^{1}$H and $^{13}$C NMR spectra of compound 41

$^{1}$H NMR (400 MHz, CDCl$_3$)

$^{13}$C NMR (100 MHz, CDCl$_3$)
$^1$H and $^{13}$C NMR spectra of compound 43

$^1$H NMR (400 MHz, CDCl$_3$)

$^{13}$C NMR (100 MHz, CDCl$_3$)
$^1$H and $^{13}$C NMR spectra of compound 44

$^1$H NMR (400 MHz, CDCl$_3$)

$^{13}$C NMR (100 MHz, CDCl$_3$)
The "S59" page contains a section titled "\(^1\)H and \(^{13}\)C NMR spectra of compound 46". The page features two NMR spectra: one is labeled as "\(^1\)H NMR (CDCl\(_3\), 400 MHz)" and the other as "\(^1\)H-\(^{13}\)C NMR (CDCl\(_3\), 100 MHz)". The spectra include detailed chemical structures with various peak annotations.
$^1$H and $^{13}$C NMR spectra of compound 48
$^1\text{H}$ and $^{13}\text{C}$ NMR spectra of compound 49
