Naphthalenes and Quinolines by Domino Reactions of Morita–Baylis–Hillman Acetates

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(E)-2-(Acetoxymethyl)-3-(2-fluoro-5-nitrophanyl)acrylonitrile (4, JAG-5-OAc, CCDC 2035023)

Comment
The selected crystal was a 3-component twin by non-merohedry. The scale factors for component 2 and 3 refined to 0.336 (3) and 0.095 (2). The displacement ellipsoids were drawn at the 50% probability level.

Experimental
A colorless block-shaped crystal of dimensions 0.084 × 0.102 × 0.240 mm was selected for structural analysis. Intensity data for this compound were collected using a D8 Quest-geometry diffractometer with a Bruker Photon II CMOS area detector [1,2] and an Incoatec Iис microfocus Mo Kα source (λ = 0.71073 Å). The sample was cooled to 100(2) K. Cell parameters were determined from a least-squares fit of 9784 peaks in the range 2.31 < < 27.08°. A total of 8358 data were measured in the range 2.311 < < 25.678° using and oscillation frames. The data were corrected for absorption by the empirical method [3] giving minimum and maximum set of 8358 independent data with R(int) = 0.0801 and a coverage of 99.1%.

The triclinic space group P1(1) was determined by statistical tests and verified by subsequent refinement. The structure was solved by dual-space methods and refined by full-matrix least-squares methods on $F^2$ [4,5]. The positions of hydrogens bonded to carbons were initially determined by geometry and were refined using a riding model. Non-hydrogen atoms were refined with anisotropic displacement parameters. Hydrogen atom displacement parameters were set to 1.2 (1.5 for methyl) times the isotropic equivalent displacement parameters of the bonded atoms. A total of 174 parameters were refined against 8358 data to give wR ($F^2$) = 0.2158 and S = 1.020 for weights of $w = 1/ [ \bar{2} (F^2) + (0.0500 P)^2 + 1.4000 P + 8.0000 + 6.0 \sin \phi ]$, where $P = [Fo^2 + 2Fc^2]/3$. The final R($F$) was 0.1022 for the 6452 observed, [$F > 4(\sigma(F))]$, data. The largest shift/s.u. was 0.000 in the final refinement cycle. The final difference map had maxima and minima of 0.405 and $-0.535 \ e/Å^3$, respectively.
Thermal Elipsoid Plot for Structure 4
Table 1. Crystal data and structure refinement for JAG-5-OAc.

| Property                        | Value                       |
|---------------------------------|-----------------------------|
| Empirical formula               | C_{12} H_{9} F N_{2} O_{4}  |
| Formula weight                  | 264.21                      |
| Crystal system                  | triclinic                   |
| Space group                     | P1 bar                      |
| Unit cell dimensions            |                             |
| \(a\) = 7.5185(10) Å           | \(= 67.309(4)°\)           |
| \(b\) = 9.1614(11) Å           | \(= 83.346(5)°\)           |
| \(c\) = 9.5536(12) Å           | \(= 73.576(5)°\)           |
| Volume                          | 582.33(13) Å³              |
| \(Z, Z'\)                      | 2, 1                        |
| Density (calculated)            | 1.507 Mg/m³                 |
| Wavelength                      | 0.71073 Å                   |
| Temperature                     | 100(2) K                    |
| \(F(000)\)                     | 272                         |
| Absorption coefficient          | 0.125 mm\(^{-1}\)          |
| Absorption correction          | semi-empirical from equivalents |
| Theta range for data collection | 2.311 to 25.678°            |
| Reflections collected           | 8358                         |
| Independent reflections         | 8358 \([R(int) = ?]\)       |
| Data / restraints / parameters  | 8358 / 0 / 174              |
| \(wR(F^2\text{ all data})\)    | \(wR2 = 0.2158\)           |
| \(R(F\text{ obsd data})\)      | \(R1 = 0.1022\)            |
| Goodness-of-fit on \(F^2\)      | 1.020                       |
| Observed data \([I > 2\sigma(I)]\) | 6452                       |
| Largest and mean shift / s.u.   | 0.000 and 0.000             |
| Largest diff. peak and hole     | 0.405 and -0.535 e/Å³       |

\[
wR2 = \frac{\sum[w(Fo^2 - Fc^2)^2]}{\sum[w(Fo^2)^2]}^{1/2}
\]

\[
R1 = \frac{\sum|Fo| - |Fc|}{\sum|Fo|}
\]
Table 2. Atomic coordinates and equivalent isotropic displacement parameters for JAG-5-OAc. \(U(\text{eq})\) is defined as one third of the trace of the orthogonalized \(U_{ij}\) tensor.

|     | \(x\)     | \(y\)     | \(z\)     | \(U(\text{eq})\) |
|-----|-----------|-----------|-----------|------------------|
| F(1)| 0.5585(5) | 0.6976(4) | 0.2162(4) | 0.0277(8)        |
| O(1)| 0.9855(7) | 0.1548(5) | 0.7798(5) | 0.0347(11)       |
| O(2)| 0.9652(6) | 0.3702(5) | 0.8307(5) | 0.0302(10)       |
| O(3)| 0.5743(6) | 0.1061(5) | 0.1328(5) | 0.0234(9)        |
| O(4)| 0.3522(6) | 0.3388(5) | 0.1039(5) | 0.0269(10)       |
| N(1)| 0.7553(8) | -0.0231(6)| 0.5043(6) | 0.0338(13)       |
| N(2)| 0.9346(7) | 0.3038(6) | 0.7494(6) | 0.0237(11)       |
| C(1)| 0.6514(8) | 0.6004(6) | 0.3461(6) | 0.0210(12)       |
| C(2)| 0.7338(8) | 0.4365(6) | 0.3674(6) | 0.0198(12)       |
| C(3)| 0.8266(8) | 0.3388(7) | 0.5035(6) | 0.0209(12)       |
| C(4)| 0.8318(8) | 0.4080(7) | 0.6081(6) | 0.0213(12)       |
| C(5)| 0.7517(8) | 0.5708(7) | 0.5852(7) | 0.0212(12)       |
| C(6)| 0.6583(8) | 0.6689(7) | 0.4502(7) | 0.0245(13)       |
| C(7)| 0.7203(8) | 0.3807(7) | 0.2449(7) | 0.0227(12)       |
| C(8)| 0.7274(8) | 0.2294(7) | 0.2534(6) | 0.0213(12)       |
| C(9)| 0.7445(9) | 0.0909(7) | 0.3945(7) | 0.0242(13)       |
| C(10)| 0.7115(8)| 0.1969(7) | 0.1131(7) | 0.0223(13)       |
| C(11)| 0.3948(8)| 0.1957(7) | 0.1210(6) | 0.0227(12)       |
| C(12)| 0.2642(9)| 0.0973(8) | 0.1271(7) | 0.0291(14)       |
Table 3. Bond lengths [Å] and angles [°] for JAG-5-OAc.

| Bond                        | Length [Å] 1 | Bond                          | Length [Å] 2 | Bond                          | Length [Å] 3 |
|-----------------------------|-------------|-------------------------------|-------------|-------------------------------|-------------|
| F(1)-C(1)                   | 1.348(6)    | C(4)-C(5)                     | 1.380(8)    |                               |             |
| O(1)-N(2)                   | 1.233(6)    | C(5)-C(6)                     | 1.386(8)    |                               |             |
| O(2)-N(2)                   | 1.229(6)    | C(5)-H(5)                     | 0.9500      |                               |             |
| O(3)-C(11)                  | 1.358(7)    | C(6)-H(6)                     | 0.9500      |                               |             |
| O(3)-C(10)                  | 1.453(6)    | C(7)-C(8)                     | 1.343(8)    |                               |             |
| O(4)-C(11)                  | 1.207(7)    | C(7)-H(7)                     | 0.9500      |                               |             |
| N(1)-C(9)                   | 1.150(8)    | C(8)-C(9)                     | 1.439(8)    |                               |             |
| N(2)-C(4)                   | 1.471(7)    | C(8)-C(10)                    | 1.505(8)    |                               |             |
| C(1)-C(6)                   | 1.377(8)    | C(10)-H(10A)                  | 0.9900      |                               |             |
| C(1)-C(2)                   | 1.396(7)    | C(10)-H(10B)                  | 0.9900      |                               |             |
| C(2)-C(3)                   | 1.392(8)    | C(11)-C(12)                   | 1.493(8)    |                               |             |
| C(2)-C(7)                   | 1.470(8)    | C(12)-H(12A)                  | 0.9800      |                               |             |
| C(3)-C(4)                   | 1.384(8)    | C(12)-H(12B)                  | 0.9800      |                               |             |
| C(3)-H(3)                   | 0.9500      | C(12)-H(12C)                  | 0.9800      |                               |             |
| C(11)-O(3)-C(10)            | 115.3(4)    | C(8)-C(7)-C(2)                | 129.0(5)    |                               |             |
| O(2)-N(2)-O(1)              | 123.6(5)    | C(8)-C(7)-H(7)                | 115.5       |                               |             |
| O(2)-N(2)-C(4)              | 118.2(5)    | C(2)-C(7)-H(7)                | 115.5       |                               |             |
| O(1)-N(2)-C(4)              | 118.2(4)    | C(7)-C(8)-C(9)                | 123.1(5)    |                               |             |
| F(1)-C(1)-C(6)              | 117.9(5)    | C(7)-C(8)-C(10)               | 120.9(5)    |                               |             |
| F(1)-C(1)-C(2)              | 118.1(5)    | C(9)-C(8)-C(10)               | 116.0(5)    |                               |             |
| C(6)-C(1)-C(2)              | 124.0(5)    | N(1)-C(9)-C(8)                | 177.1(6)    |                               |             |
| C(3)-C(2)-C(1)              | 116.8(5)    | O(3)-C(10)-C(8)               | 111.4(5)    |                               |             |
| C(3)-C(2)-C(7)              | 125.0(5)    | O(3)-C(10)-H(10A)             | 109.3       |                               |             |
| C(1)-C(2)-C(7)              | 118.3(5)    | C(8)-C(10)-H(10A)             | 109.3       |                               |             |
| C(4)-C(3)-C(2)              | 119.0(5)    | O(3)-C(10)-H(10B)             | 109.3       |                               |             |
| C(4)-C(3)-H(3)              | 120.5       | C(8)-C(10)-H(10B)             | 109.3       |                               |             |
| C(2)-C(3)-H(3)              | 120.5       | H(10A)-C(10)-H(10B)           | 108.0       |                               |             |
| C(5)-C(4)-C(3)              | 123.8(5)    | O(4)-C(11)-O(3)               | 122.2(5)    |                               |             |
| C(5)-C(4)-N(2)              | 118.0(5)    | O(4)-C(11)-C(12)              | 125.9(6)    |                               |             |
| C(3)-C(4)-N(2)              | 118.2(5)    | O(3)-C(11)-C(12)              | 111.9(5)    |                               |             |
| C(4)-C(5)-C(6)              | 117.6(5)    | C(11)-C(12)-H(12A)            | 109.8       |                               |             |
| C(4)-C(5)-H(5)              | 121.2       | C(11)-C(12)-H(12B)            | 109.2       |                               |             |
| C(6)-C(5)-H(5)              | 121.2       | H(12A)-C(12)-H(12B)           | 109.5       |                               |             |
| C(1)-C(6)-C(5)              | 118.9(5)    | C(11)-C(12)-H(12C)            | 109.4       |                               |             |
| C(1)-C(6)-H(6)              | 120.6       | H(12A)-C(12)-H(12C)           | 109.5       |                               |             |
| C(5)-C(6)-H(6)              | 120.6       | H(12B)-C(12)-H(12C)           | 109.5       |                               |             |
Table 4. Anisotropic displacement parameters (Å² x 10³) for JAG-5-OAc. The anisotropic displacement factor exponent takes the form:

\[-2 \pi^2 [ h^2 a^*^2 U_{11} + \ldots + 2 h k a^* b^* U_{12} ]\]

|       | U_{11} | U_{22} | U_{33} | U_{23} | U_{13} | U_{12} |
|-------|--------|--------|--------|--------|--------|--------|
| F(1)  | 34(2)  | 18(2)  | 29(2)  | -8(1)  | -4(2)  | -3(1)  |
| O(1)  | 48(3)  | 19(2)  | 36(3)  | -7(2)  | -8(2)  | -8(2)  |
| O(2)  | 35(3)  | 31(2)  | 31(2)  | -15(2) | 0(2)   | -13(2) |
| O(3)  | 25(2)  | 18(2)  | 31(2)  | -12(2) | -2(2)  | -8(2)  |
| O(4)  | 34(2)  | 21(2)  | 26(2)  | -11(2) | -3(2)  | -5(2)  |
| N(1)  | 54(4)  | 24(3)  | 32(3)  | -15(3) | -3(3)  | -16(3) |
| N(2)  | 25(3)  | 25(3)  | 25(3)  | -10(2) | 3(2)   | -12(2) |
| C(1)  | 23(3)  | 16(3)  | 25(3)  | -6(2)  | 0(2)   | -8(2)  |
| C(2)  | 21(3)  | 16(3)  | 24(3)  | -8(2)  | 4(2)   | -9(2)  |
| C(3)  | 24(3)  | 15(3)  | 26(3)  | -7(2)  | 3(2)   | -9(2)  |
| C(4)  | 22(3)  | 20(3)  | 24(3)  | -8(2)  | 2(2)   | -11(2) |
| C(5)  | 21(3)  | 22(3)  | 26(3)  | -14(2) | 5(2)   | -9(2)  |
| C(6)  | 26(3)  | 18(3)  | 34(3)  | -13(3) | 5(3)   | -10(3) |
| C(7)  | 24(3)  | 20(3)  | 22(3)  | -6(2)  | 0(2)   | -6(2)  |
| C(8)  | 23(3)  | 19(3)  | 23(3)  | -8(2)  | -1(2)  | -6(2)  |
| C(9)  | 31(3)  | 19(3)  | 29(3)  | -14(3) | 0(3)   | -10(2) |
| C(10)| 23(3)  | 22(3)  | 27(3)  | -13(3) | 3(2)   | -9(2)  |
| C(11)| 28(3)  | 22(3)  | 19(3)  | -9(2)  | -1(2)  | -7(2)  |
| C(12)| 30(4)  | 29(3)  | 32(4)  | -11(3) | -3(3)  | -12(3) |
Table 5. Hydrogen coordinates and isotropic displacement parameters for JAG-5-OAc.

|       |   x     |   y     |   z     |   U(eq) |
|-------|---------|---------|---------|---------|
| H(3)  | 0.885464| 0.226246| 0.524185| 0.025   |
| H(5)  | 0.760224| 0.614057| 0.659185| 0.025   |
| H(6)  | 0.600038| 0.781417| 0.429913| 0.029   |
| H(7)  | 0.704297| 0.463145| 0.145940| 0.027   |
| H(10A)| 0.676214| 0.302452| 0.026320| 0.027   |
| H(10B)| 0.833542| 0.133692| 0.089518| 0.027   |
| H(12A)| 0.258568| 0.091619| 0.027423| 0.044   |
| H(12B)| 0.140469| 0.149343| 0.156047| 0.044   |
| H(12C)| 0.307447| -0.013678| 0.202526| 0.044   |
| Bond Sequence | Torsion Angle (°) |
|--------------|-------------------|
| F(1)-C(1)-C(2)-C(3) | -179.2(5) |
| C(6)-C(1)-C(2)-C(3) | 0.4(8) |
| F(1)-C(1)-C(2)-C(7) | 2.1(8) |
| C(6)-C(1)-C(2)-C(7) | -178.2(6) |
| C(1)-C(2)-C(3)-C(4) | 0.1(8) |
| C(7)-C(2)-C(3)-C(4) | 178.6(5) |
| C(2)-C(3)-C(4)-C(5) | -0.9(8) |
| C(2)-C(3)-C(4)-N(2) | -178.4(5) |
| O(2)-N(2)-C(4)-C(5) | -9.4(7) |
| O(1)-N(2)-C(4)-C(5) | 170.7(5) |
| O(2)-N(2)-C(4)-C(3) | 168.3(5) |
| O(1)-N(2)-C(4)-C(3) | -11.6(7) |
| C(3)-C(4)-C(5)-C(6) | 1.1(8) |
| N(2)-C(4)-C(5)-C(6) | 178.7(5) |
| F(1)-C(1)-C(6)-C(5) | 179.4(5) |
| C(2)-C(1)-C(6)-C(5) | -0.2(9) |
| C(4)-C(5)-C(6)-C(1) | -0.6(8) |
| C(3)-C(2)-C(7)-C(8) | 26.9(10) |
| C(1)-C(2)-C(7)-C(8) | -154.6(6) |
| C(2)-C(7)-C(8)-C(9) | 2.2(10) |
| C(2)-C(7)-C(8)-C(10) | -179.7(5) |
| C(11)-O(3)-C(10)-C(8) | 77.7(6) |
| C(7)-C(8)-C(10)-O(3) | -130.1(6) |
| C(9)-C(8)-C(10)-O(3) | 48.1(7) |
| C(10)-O(3)-C(11)-O(4) | -4.0(8) |
| C(10)-O(3)-C(11)-C(12) | 174.3(5) |
Comment
The displacement ellipsoids were drawn at the 50% probability level.

Experimental
A colorless, block-shaped crystal of dimensions 0.048 × 0.068 × 0.150 mm was selected for structural analysis. Intensity data for this compound were collected using a D8 Quest -geometry diffractometer with a Bruker Photon II cmos area detector [1,2] and an Incoatec I s microfocus Mo K source ( = 0.71073 Å). The sample was cooled to 100(2) K. Cell parameters were determined from a least-squares fit of 9637 peaks in the range 2.66 < < 27.10°. A total of 42733 data were measured in the range 2.659 < < 27.192° using and oscillation frames. The data were corrected for absorption by the empirical method [3] giving minimum and maximum transmission factors of 0.708 and 0.746. The data were merged to form a set of 3947 independent data with R(int) = 0.0637 and a coverage of 99.9%.

The monoclinic space group P2_1_1/c was determined by systematic absences and statistical tests and verified by subsequent refinement. The structure was solved by direct methods and refined by full-matrix least-squares methods on F^2 [4,5]. The positions of hydrogens bonded to carbons were initially determined by geometry and were refined using a riding model. Non-hydrogen atoms were refined with anisotropic displacement parameters. Hydrogen atom displacement parameters were set to 1.2 (1.5 for methyl) times the isotropic equivalent displacement parameters of the bonded atoms. A total of 235 parameters were refined against 3947 data to give wR(F^2) = 0.1027 and S = 1.007 for weights of w = 1/[ F^2 + (0.0200 P)^2 + 2.8000 P], where P = [Fo^2 + 2Fc^2]/3. The final R(F) was 0.0459 for the 3251 observed, [F > 4 (F)], data. The largest shift/s.u. was 0.000 in the final refinement cycle. The final difference map had maxima and minima of 0.987 and -0.666 e/Å^3, respectively.
Thermal Elipsoid Plot for Structure 22
Table 1. Crystal data and structure refinement for JAG-1-CNTL.

| Property                              | Value                                      |
|---------------------------------------|--------------------------------------------|
| Empirical formula                     | C\textsubscript{19}H\textsubscript{16}FN\textsubscript{4}O\textsubscript{4}S         |
| Formula weight                        | 373.39                                     |
| Crystal system                        | monoclinic                                 |
| Space group                           | P21/c                                      |
| Unit cell dimensions                  | \(a = 11.2443(3)\) Å \(= 90^\circ\)        |
|                                       | \(b = 20.9387(5)\) Å \(= 91.1985(9)^\circ\) |
|                                       | \(c = 7.5398(2)\) Å \(= 90^\circ\)         |
| Volume                                | 1774.79(8) Å\(^3\)                         |
| Z, Z'                                 | 4, 1                                       |
| Density (calculated)                  | 1.397 Mg/m\(^3\)                           |
| Wavelength                            | 0.71073 Å                                  |
| Temperature                           | 100(2) K                                   |
| \(F(000)\)                            | 776                                         |
| Absorption coefficient                | 0.217 mm\(^{-1}\)                          |
| Absorption correction                 | semi-empirical from equivalents            |
| Max. and min. transmission            | 0.746 and 0.708                             |
| Theta range for data collection       | 2.659 to 27.192\(^\circ\)                  |
| Reflections collected                 | 42733                                       |
| Independent reflections               | 3947 [R(int) = 0.0637]                      |
| Data / restraints / parameters        | 3947 / 0 / 235                              |
| \(wR(F^2\text{ all data})\)           | \(wR2 = 0.1027\)                           |
| \(R(F\text{ obsd data})\)             | \(R1 = 0.0459\)                            |
| Goodness-of-fit on \(F^2\)            | 1.007                                       |
| Observed data [I > 2 \((I)\)]        | 3251                                        |
| Largest and mean shift / s.u.         | 0.000 and 0.000                             |
| Largest diff. peak and hole           | 0.987 and -0.666 e/Å\(^3\)                 |

\[ wR^2 = \left( \frac{\sum\left[w(F_o^2 - F_c^2)\right]}{\sum\left[w(F_o^2)\right]} \right)^{1/2} \]

\[ R1 = \left( \frac{\sum\left|F_o\right| - \left|F_c\right|}{\sum\left|F_o\right|} \right) / \left|F_o\right| \]
Table 2. Atomic coordinates and equivalent isotropic displacement parameters for JAG-5-OAc. $U(\text{eq})$ is defined as one third of the trace of the orthogonalized $U_{ij}$ tensor.

|     | $x$    | $y$    | $z$    | $U(\text{eq})$ |
|-----|--------|--------|--------|----------------|
| F(1)| 0.5585(5) | 0.6976(4) | 0.2162(4) | 0.0277(8)     |
| O(1)| 0.9855(7) | 0.1548(5) | 0.7798(5) | 0.0347(11)    |
| O(2)| 0.9652(6) | 0.3702(5) | 0.8307(5) | 0.0302(10)    |
| O(3)| 0.5743(6) | 0.1061(5) | 0.1328(5) | 0.0234(9)     |
| O(4)| 0.3522(6) | 0.3388(5) | 0.1039(5) | 0.0269(10)    |
| N(1)| 0.7553(8) | -0.0231(6) | 0.5043(6) | 0.0338(13)    |
| N(2)| 0.9346(7) | 0.3038(6) | 0.7494(6) | 0.0237(11)    |
| C(1)| 0.6514(8) | 0.6004(6) | 0.3461(6) | 0.0210(12)    |
| C(2)| 0.7338(8) | 0.4365(6) | 0.3674(6) | 0.0198(12)    |
| C(3)| 0.8266(8) | 0.3388(7) | 0.5035(6) | 0.0209(12)    |
| C(4)| 0.8318(8) | 0.4080(7) | 0.6081(6) | 0.0213(12)    |
| C(5)| 0.7517(8) | 0.5708(7) | 0.5852(7) | 0.0212(12)    |
| C(6)| 0.6583(8) | 0.6689(7) | 0.4502(7) | 0.0245(13)    |
| C(7)| 0.7203(8) | 0.3807(7) | 0.2449(7) | 0.0227(12)    |
| C(8)| 0.7274(8) | 0.2294(7) | 0.2534(6) | 0.0213(12)    |
| C(9)| 0.7445(9) | 0.0909(7) | 0.3945(7) | 0.0242(13)    |
| C(10)| 0.7115(8) | 0.1969(7) | 0.1131(7) | 0.0223(13)    |
| C(11)| 0.3948(8) | 0.1957(7) | 0.1210(6) | 0.0227(12)    |
| C(12)| 0.2642(9) | 0.0973(8) | 0.1271(7) | 0.0291(14)    |
Table 3. Bond lengths [Å] and angles [°] for JAG-1-CNTL.

| Bond                  | Length [Å] | Angle [°] |
|-----------------------|------------|-----------|
| S(1)-O(1)             | 1.4388(15) | C(8)-C(9) | 1.384(3) |
| S(1)-O(2)             | 1.4393(17) | C(8)-C(13) | 1.401(3) |
| S(1)-C(14)            | 1.759(2)   | C(9)-C(10) | 1.371(3) |
| S(1)-C(1)             | 1.822(2)   | C(10)-C(11) | 1.383(3) |
| F(1)-C(9)             | 1.355(3)   | C(10)-H(10) | 0.9500 |
| O(3)-C(2)             | 1.319(3)   | C(11)-C(12) | 1.388(3) |
| O(3)-C(3)             | 1.450(3)   | C(11)-H(11) | 0.9500 |
| O(4)-C(2)             | 1.202(3)   | C(12)-C(13) | 1.383(3) |
| N(1)-C(6)             | 1.150(3)   | C(12)-H(12) | 0.9500 |
| C(1)-C(2)             | 1.527(3)   | C(13)-H(13) | 0.9500 |
| C(1)-C(4)             | 1.531(3)   | C(14)-C(19) | 1.389(3) |
| C(1)-H(1)             | 1.0000     | C(14)-C(15) | 1.392(3) |
| C(3)-H(3A)            | 0.9800     | C(15)-C(16) | 1.383(3) |
| C(3)-H(3B)            | 0.9800     | C(15)-H(15) | 0.9500 |
| C(3)-H(3C)            | 0.9800     | C(16)-C(17) | 1.379(4) |
| C(4)-C(5)             | 1.515(3)   | C(16)-H(16) | 0.9500 |
| C(4)-H(4A)            | 0.9900     | C(17)-C(18) | 1.382(4) |
| C(4)-H(4AB)           | 0.9900     | C(17)-H(17) | 0.9500 |
| C(5)-C(7)             | 1.337(3)   | C(18)-C(19) | 1.391(3) |
| C(5)-C(6)             | 1.444(3)   | C(18)-H(18) | 0.9500 |
| C(7)-C(8)             | 1.469(3)   | C(19)-H(19) | 0.9500 |
| C(7)-H(7)             | 0.9500     |            |          |
| O(1)-S(1)-O(2)        | 118.91(10) | O(3)-C(3)-H(3C) | 109.5 |
| O(1)-S(1)-C(14)       | 108.76(10) | H(3A)-C(3)-H(3C) | 109.5 |
| O(2)-S(1)-C(14)       | 109.00(10) | H(3B)-C(3)-H(3C) | 109.5 |
| O(1)-S(1)-C(1)        | 106.07(9)  | C(5)-C(4)-C(1)  | 111.02(17) |
| O(2)-S(1)-C(1)        | 108.31(9)  | C(5)-C(4)-H(4A) | 109.4 |
| C(14)-S(1)-C(1)       | 104.90(9)  | C(1)-C(4)-H(4A) | 109.4 |
| C(2)-O(3)-C(3)        | 115.27(17) | C(5)-C(4)-H(4AB) | 109.4 |
| C(2)-C(1)-C(4)        | 111.55(17) | C(1)-C(4)-H(4AB) | 109.4 |
| C(2)-C(1)-S(1)        | 109.17(14) | H(4A)-C(4)-H(4AB) | 108.0 |
| C(4)-C(1)-S(1)        | 107.82(14) | C(7)-C(5)-C(6)  | 120.60(19) |
| C(2)-C(1)-H(1)        | 109.4      | C(7)-C(5)-C(4)  | 122.85(19) |
| C(4)-C(1)-H(1)        | 109.4      | C(6)-C(5)-C(4)  | 116.49(18) |
| S(1)-C(1)-H(1)        | 109.4      | N(1)-C(6)-C(5)  | 177.4(2) |
| O(4)-C(2)-O(3)        | 125.21(19) | C(5)-C(7)-C(8)  | 127.26(19) |
| O(4)-C(2)-C(1)        | 124.11(19) | C(5)-C(7)-H(7)  | 116.4 |
| O(3)-C(2)-C(1)        | 110.68(17) | C(8)-C(7)-H(7)  | 116.4 |
| O(3)-C(3)-H(3A)       | 109.5      | C(9)-C(8)-C(13) | 116.46(19) |
| O(3)-C(3)-H(3B)       | 109.5      | C(9)-C(8)-C(7)  | 118.82(19) |
| H(3A)-C(3)-H(3B)      | 109.5      | C(13)-C(8)-C(7) | 124.61(19) |
| Bond                  | Angle   | Bond                  | Angle   |
|-----------------------|---------|-----------------------|---------|
| F(1)-C(9)-C(10)       | 118.3(2)| C(15)-C(14)-S(1)     | 118.93(16) |
| F(1)-C(9)-C(8)        | 117.49(19)| C(16)-C(15)-C(14)   | 118.8(2) |
| C(10)-C(9)-C(8)       | 124.3(2)| C(16)-C(15)-H(15)    | 120.6    |
| C(9)-C(10)-C(11)      | 118.0(2)| C(14)-C(15)-H(15)    | 120.6    |
| C(9)-C(10)-H(10)      | 121.0   | C(17)-C(16)-C(15)    | 120.4(2) |
| C(11)-C(10)-H(10)     | 121.0   | C(17)-C(16)-H(16)    | 119.8    |
| C(10)-C(11)-C(12)     | 120.2(2)| C(15)-C(16)-H(16)    | 119.8    |
| C(10)-C(11)-H(11)     | 119.9   | C(16)-C(17)-C(18)    | 120.3(2) |
| C(12)-C(11)-H(11)     | 119.9   | C(16)-C(17)-H(17)    | 119.9    |
| C(13)-C(12)-C(11)     | 120.4(2)| C(18)-C(17)-H(17)    | 119.9    |
| C(13)-C(12)-H(12)     | 119.8   | C(17)-C(18)-C(19)    | 120.8(2) |
| C(11)-C(12)-H(12)     | 119.8   | C(17)-C(18)-H(18)    | 119.6    |
| C(12)-C(13)-C(8)      | 120.7(2)| C(19)-C(18)-H(18)    | 119.6    |
| C(12)-C(13)-H(13)     | 119.7   | C(14)-C(19)-C(18)    | 118.0(2) |
| C(8)-C(13)-H(13)      | 119.7   | C(14)-C(19)-H(19)    | 121.0    |
| C(19)-C(14)-C(15)     | 121.7(2)| C(18)-C(19)-H(19)    | 121.0    |
| C(19)-C(14)-S(1)      | 119.34(18)|
Table 4. Anisotropic displacement parameters ($\AA^2 \times 10^3$) for JAG-5-OAc. The anisotropic displacement factor exponent takes the form:

$$-2 \pi^2 [h^2 a^{*2} U_{11} + \ldots + 2hk a^* b^* U_{12}]$$

|     | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ |
|-----|----------|----------|----------|----------|----------|----------|
| F(1) | 34(2)    | 18(2)    | 29(2)    | -8(1)    | -4(2)    | -3(1)    |
| O(1) | 48(3)    | 19(2)    | 36(3)    | -7(2)    | -8(2)    | -8(2)    |
| O(2) | 35(3)    | 31(2)    | 31(2)    | -15(2)   | 0(2)     | -13(2)   |
| O(3) | 25(2)    | 18(2)    | 31(2)    | -12(2)   | -2(2)    | -8(2)    |
| O(4) | 34(2)    | 21(2)    | 26(2)    | -11(2)   | -3(2)    | -5(2)    |
| N(1) | 54(4)    | 24(3)    | 32(3)    | -15(3)   | -3(3)    | -16(3)   |
| N(2) | 25(3)    | 25(3)    | 25(3)    | -10(2)   | 3(2)     | -12(2)   |
| C(1) | 23(3)    | 16(3)    | 25(3)    | -6(2)    | 0(2)     | -8(2)    |
| C(2) | 21(3)    | 16(3)    | 24(3)    | -8(2)    | 4(2)     | -9(2)    |
| C(3) | 24(3)    | 15(3)    | 26(3)    | -7(2)    | 3(2)     | -9(2)    |
| C(4) | 22(3)    | 20(3)    | 24(3)    | -8(2)    | 2(2)     | -11(2)   |
| C(5) | 21(3)    | 22(3)    | 26(3)    | -14(2)   | 5(2)     | -9(2)    |
| C(6) | 26(3)    | 18(3)    | 34(3)    | -13(3)   | 5(3)     | -10(3)   |
| C(7) | 24(3)    | 20(3)    | 22(3)    | -6(2)    | 0(2)     | -6(2)    |
| C(8) | 23(3)    | 19(3)    | 23(3)    | -8(2)    | 1(2)     | -6(2)    |
| C(9) | 31(3)    | 19(3)    | 29(3)    | -14(3)   | 0(3)     | -10(2)   |
| C(10)| 23(3)    | 22(3)    | 27(3)    | -13(3)   | 3(2)     | -9(2)    |
| C(11)| 28(3)    | 22(3)    | 19(3)    | -9(2)    | 1(2)     | -7(2)    |
| C(12)| 30(4)    | 29(3)    | 32(4)    | -11(3)   | -3(3)    | -12(3)   |
Table 5. Hydrogen coordinates and isotropic displacement parameters for JAG-1-CNTL.

|     | x      | y      | z      | U(eq)  |
|-----|--------|--------|--------|--------|
| H(1 )| 0.329595 | 0.313612 | 0.406793 | 0.027  |
| H(3A) | 0.560398 | 0.431167 | 0.094771 | 0.056  |
| H(3B) | 0.557416 | 0.471752 | 0.274480 | 0.056  |
| H(3C) | 0.448167 | 0.475421 | 0.135375 | 0.056  |
| H(4A) | 0.175575 | 0.325745 | 0.628979 | 0.030  |
| H(4AB) | 0.190352 | 0.401552 | 0.610747 | 0.030  |
| H(7)  | 0.084888 | 0.452472 | 0.378571 | 0.031  |
| H(10) | -0.243854 | 0.507819 | 0.037014 | 0.041  |
| H(11) | -0.223145 | 0.447904 | -0.223428 | 0.041  |
| H(12) | -0.075160 | 0.370117 | -0.239303 | 0.038  |
| H(13) | 0.054097 | 0.352406 | 0.000453 | 0.033  |
| H(15) | 0.511275 | 0.213775 | 0.622754 | 0.033  |
| H(16) | 0.694108 | 0.176743 | 0.522216 | 0.042  |
| H(17) | 0.840514 | 0.248745 | 0.440674 | 0.047  |
| H(18) | 0.807296 | 0.358039 | 0.461481 | 0.047  |
| H(19) | 0.626788 | 0.397037 | 0.569703 | 0.037  |
Table 6.  Torsion angles [°] for JAG-1-CNTL.

| Bond Pair                        | Torsion Angle |
|----------------------------------|---------------|
| O(1)-S(1)-C(1)-C(2)             | 173.33(14)    |
| O(2)-S(1)-C(1)-C(2)             | 44.64(17)     |
| C(14)-S(1)-C(1)-C(2)            | -71.65(16)    |
| O(1)-S(1)-C(1)-C(4)             | 51.98(16)     |
| O(2)-S(1)-C(1)-C(4)             | -76.71(16)    |
| C(14)-S(1)-C(1)-C(4)            | 167.00(14)    |
| C(3)-O(3)-C(2)-O(4)             | 4.0(3)        |
| C(3)-O(3)-C(2)-C(1)             | -175.66(17)   |
| C(4)-C(1)-C(2)-O(4)             | 29.5(3)       |
| S(1)-C(1)-O(2)-O(4)             | -89.6(2)      |
| C(4)-C(1)-C(2)-O(3)             | -150.88(17)   |
| S(1)-C(1)-C(2)-O(3)             | 90.07(17)     |
| C(2)-C(1)-C(4)-C(5)             | 71.2(2)       |
| S(1)-C(1)-C(4)-C(5)             | -168.94(14)   |
| C(1)-C(4)-C(5)-C(7)             | -102.7(2)     |
| C(1)-C(4)-C(5)-C(6)             | 80.1(2)       |
| C(6)-C(5)-C(7)-C(8)             | -4.5(3)       |
| C(4)-C(5)-C(7)-C(8)             | 178.4(2)      |
| C(5)-C(7)-C(8)-C(9)             | 143.5(2)      |
| C(5)-C(7)-C(8)-C(13)            | -40.4(3)      |
| C(13)-C(8)-C(9)-F(1)            | 179.33(19)    |
| C(7)-C(8)-C(9)-F(1)             | -4.2(3)       |
| C(13)-C(8)-C(9)-C(10)           | -0.1(3)       |
| C(7)-C(8)-C(9)-C(10)            | 176.3(2)      |
| C(8)-C(9)-C(10)-C(11)           | 0.2(4)        |
| C(9)-C(10)-C(11)-C(12)          | 0.1(3)        |
| C(10)-C(11)-C(12)-C(13)         | -0.4(4)       |
| C(11)-C(12)-C(13)-C(8)          | 0.5(3)        |
| C(9)-C(8)-C(13)-C(12)           | -0.2(3)       |
| C(7)-C(8)-C(13)-C(12)           | -176.4(2)     |
| O(1)-S(1)-C(14)-C(19)           | -160.84(16)   |
| O(2)-S(1)-C(14)-C(19)           | -29.79(19)    |
| C(1)-S(1)-C(14)-C(19)           | 86.02(18)     |
| O(1)-S(1)-C(14)-C(15)           | 18.73(19)     |
| O(2)-S(1)-C(14)-C(15)           | 149.79(16)    |
| C(1)-S(1)-C(14)-C(15)           | -94.40(17)    |
| C(19)-C(14)-C(15)-C(16)         | 0.6(3)        |
| S(1)-C(14)-C(15)-C(16)          | -178.99(16)   |
| C(14)-C(15)-C(16)-C(17)         | -1.0(3)       |
| C(15)-C(16)-C(17)-C(18)         | 0.4(3)        |
| C(16)-C(17)-C(18)-C(19)         | 0.6(3)        |
| C(15)-C(14)-C(19)-C(18)         | 0.4(3)        |
| S(1)-C(14)-C(19)-C(18)          | 179.95(16)    |
| C(17)-C(18)-C(19)-C(14)         | -1.0(3)       |
Table 7. Hydrogen bonds for JAG-1-CNTL [Å and °].

| D-H…A                  | d(D-H) | d(H…A) | d(D…A)  | <(DHA) |
|------------------------|--------|--------|---------|--------|
| C(1)-H(1)...O(1)#1     | 1.00   | 2.31   | 3.290(2)| 166.1  |
| C(4)-H(4A)...N(1)#2    | 0.99   | 2.37   | 3.300(3)| 156.7  |
| C(4)-H(4AB)...F(1)#3   | 0.99   | 2.31   | 3.249(3)| 158.5  |
| C(15)-H(15)...O(3)#2   | 0.95   | 2.64   | 3.481(3)| 148.4  |

Symmetry transformations used to generate equivalent atoms:
#1 x, -y+1/2, z-1/2  #2 x, -y+1/2, z+1/2  #3 -x, -y+1, -z+1

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