Diastereoselective Synthesis of Functionalised Carbazoles via a Sequential Diels-Alder/Ene Reaction Strategy

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General experimental information

\(^1\)H and \(^{13}\)C NMR Spectra:  
2a - (3a\(^S\)*,10a\(^S\)*,10b\(^S\)*)-2-methyl-10-tosyl-4,10,10a,10b-tetrahydropyrrolo[3,4-a]carbazole-1,3(2H,3aH)-dione  
2b - 2-phenyl-11-tosyl-1,11a-di-hydro-1\(^H\),5\(^H\)-[1,2,4]triazolo[1',2':1,2]pyridazino[3,4-b]indole-1,3(2H)-dione  
2c - ethyl 3-((3a\(^S\)*,4R*,10a\(^S\)*,10b\(^S\)*)-2-methyl-1,3-dioxo-10-tosyl-1,2,3,3a,4,10,10a,10b-octahydropyrrolo[3,4-\(a\)]carbazol-4-yl)propanoate  
2d - ethyl 3-((3a\(^S\)*,4R*,10a\(^S\)*,10b\(^S\)*)-1,3-dioxo-10-tosyl-1,2,3,3a,4,10,10a,10b-octahydropyrrolo[3,4-\(a\)]carbazol-4-yl)propanoate  
2e - ethyl 3-((3a\(^S\)*,4R*,10a\(^S\)*,10b\(^S\)*)-1,3-dioxo-2-phenyl-10-tosyl-1,2,3,3a,4,10,10a,10b-octahydropyrrolo[3,4-\(a\)]carbazol-4-yl)propanoate  
2f - (3a\(^S\)*,5\(^S\)*,10b\(^S\)*)-5-(hydroxy(phenyl)amino)-2-methyl-10-tosyl-4,5,10,10b-tetrahydropyrrolo[3,4-\(a\)]carbazole-1,3(2H,3aH)-dione  
2g - (3a\(^S\)*,5\(^S\)*,10b\(^S\)*)-5-(hydroxy(o-tolyl)amino)-2-methyl-10-tosyl-4,5,10,10b-tetrahydropyrrolo[3,4-\(a\)]carbazole-1,3(2H,3aH)-dione  
2h - (3a\(^S\)*,5\(^S\)*,10b\(^S\)*)-5-(3,5-dioxo-4-phenyl-1,2,4-triazolidin-1-yl)-2-methyl-1,3-dioxo-10-tosyl-1,2,3,3a,4,5,10,10b-octahydropyrrolo[3,4-\(a\)]carbazol-4-yl)propanoate  
2i - (3a\(^S\)*,5\(^S\)*,10b\(^S\)*)-5-(3,5-dioxo-4-phenyl-1,2,4-triazolidin-1-yl)-1,3-dioxo-10-tosyl-1,2,3,3a,4,5,10,10b-octahydropyrrolo[3,4-\(a\)]carbazol-4-yl)propanoate  
2j - (3a\(^S\)*,5\(^S\)*,10b\(^S\)*)-5-(3,5-dioxo-4-phenyl-1,2,4-triazolidin-1-yl)-2-methyl-1,3-dioxo-10-tosyl-1,2,3,3a,4,5,10,10b-octahydropyrrolo[3,4-\(a\)]carbazol-4-yl)propanoate

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3k - (3aS*,5S*,10bS*)-5-(hydroxy(phenyl)amino)-10-tosyl-4,5,10,10b-tetrahydropyrrolo[3,4-a]carbazole-1,3(2H,3aH)-dione
3l - (3aS*,5S*,10bS*)-5-(hydroxy(o-tolyl)amino)-10-tosyl-4,5,10,10b-tetrahydropyrrolo[3,4-a]carbazole-1,3(2H,3aH)-dione
3m - (3aS*,5S*,10bS*)-5-((S*)-hydroxy(perfluorophenyl)methyl)-10-tosyl-4,5,10,10b-tetrahydropyrrolo[3,4-a]carbazole-1,3(2H,3aH)-dione
3n - (3aS*,5S*,10bS*)-5-(3,5-dioxo-4-phenyl-1,2,4-triazolidin-1-yl)-10-tosyl-4,5,10,10b-tetrahydropyrrolo[3,4-a]carbazole-1,3(2H,3aH)-dione
3o - (3aS*,5S*,10bS*)-5-(hydroxy(o-tolyl)amino)-7-methoxy-10-tosyl-4,5,10,10b-tetrahydropyrrolo[3,4-a]carbazole-1,3(2H,3aH)-dione
3p - (3aS*,5S*,10bS*)-5-((S*)-hydroxy(perfluorophenyl)methyl)-7-methoxy-2-methyl-10-tosyl-4,5,10,10b-tetrahydropyrrolo[3,4-a]carbazole-1,3(2H,3aH)-dione and (3aS*,5S*,10bS*)-5-((R*)-hydroxy(perfluorophenyl)methyl)-7-methoxy-2-methyl-10-tosyl-4,5,10,10b-tetrahydropyrrolo[3,4-a]carbazole-1,3(2H,3aH)-dione
3q - (3aS*,5S*,10bS*)-5-(3,5-dioxo-4-phenyl-1,2,4-triazolidin-1-yl)-7-methoxy-10-tosyl-4,5,10,10b-tetrahydropyrrolo[3,4-a]carbazole-1,3(2H,3aH)-dione
3r - 6-(3,5-dioxo-4-phenyl-1,2,4-triazolidin-1-yl)-2-phenyl-11-tosyl-5,6-dihydro-[1,2,4]triazolo[1′,2′:1,2]pyridazino[3,4-b]indole-1,3(1H,11H)-dione
3s - 6-(hydroxy(o-tolyl)amino)-8-methoxy-2-phenyl-11-tosyl-5,6-dihydro-[1,2,4]triazolo[1′,2′:1,2]pyridazino[3,4-b]indole-1,3(1H,11H)-dione
3t - (3aS*,5S*,10bS*)-5-(hydroxy(o-tolyl)amino)-N,N,2-trimethyl-1,3-dioxo-1,3,3a,4,5,10b-hexahydropyrrolo[3,4-a]carbazole-10(2H)-sulfonamide
3u - (3aS*,5S*,10bS*)-5-(2,6-dibromophenyl)(hydroxy)amino)-N,N-dimethyl-1,3-dioxo-2-phenyl-1,3a,4,5,10b-hexahydropyrrolo[3,4-a]carbazole-10(2H)-sulfonamide
3v - (3aS*,5S*,10bS*)-5-((S*)-hydroxy(perfluorophenyl)methyl)-N,N-dimethyl-1,3-dioxo-1,3a,4,5,10b-hexahydropyrrolo[3,4-a]carbazole-10(2H)-sulfonamide
3w - 6-(hydroxy(o-tolyl)amino)-N,N-dimethyl-1,3-dioxo-2-phenyl-2,3,5,6-tetrahydro-[1,2,4]triazolo[1′,2′:1,2]pyridazino[3,4-b]indole-11(1H)-sulfonamide
3x - (3aS*,5S*,10bS*)-5-(hydroxy(o-tolyl)amino)-N,N-dimethyl-1,3-dioxo-1,3a,4,5,10b-hexahydropyrrolo[3,4-a]carbazole-10(2H)-sulfonamide
3y - (3aS*,5S*,10bS*)-benzyl 5-(hydroxy(phenyl)amino)-2-methyl-1,3-dioxo-1,3a,4,5,10b-hexahydropyrrolo[3,4-a]carbazole-10(2H)-carboxylate
3z - (3aS*,5S*,10bS*)-benzyl 5-(hydroxy(o-tolyl)amino)-2-methyl-1,3-dioxo-1,3a,4,5,10b-hexahydropyrrolo[3,4-a]carbazole-10(2H)-carboxylate
3aa - (3aS*,5S*,10bS*)-5-(3,5-dioxo-4-phenyl-1,2,4-triazolidin-1-yl)-2-methyl-1,3-dioxo-2,3a,4,5,10b-hexahydropyrrolo[3,4-a]carbazole-10(1H)-carboxylate
3bb - benzyl (3aS*,5S*,10bS*)-5-(hydroxy(phenyl)amino)-1,3-dioxo-2,3a,4,5,10b-hexahydropyrrolo[3,4-a]carbazole-10(1H)-carboxylate
3cc - benzyl (3aS*,5S*,10bS*)-5-(hydroxy(o-tolyl)amino)-1,3-dioxo-2,3a,4,5,10b-hexahydropyrrolo[3,4-a]carbazole-10(1H)-carboxylate
3dd - benzyl (3aS*,5S*,10bS*)-5-(3,5-dioxo-4-phenyl-1,2,4-triazolidin-1-yl)-1,3-dioxo-2,3a,4,5,10b-hexahydropyrrolo[3,4-a]carbazole-10(1H)-carboxylate
3ee - benzyl (R*)-6-(hydroxy(phenyl)amino)-1,3-dioxo-2-phenyl-2,3,5,6-tetrahydro-1H,11H-[1,2,4]triazolo[1′,2′:1,2]pyridazino[3,4-b]indole-11(1H)-carboxylate
3ff - benzyl (R*)-6-(hydroxy(o-tolyl)amino)-1,3-dioxo-2-phenyl-2,3,5,6-tetrahydro-1H,11H-[1,2,4]triazolo[1′,2′:1,2]pyridazino[3,4-b]indole-11(1H)-carboxylate
3gg - benzyl (3aS*,5S*,10bS*)-5-(hydroxy(phenyl)amino)-7-methoxy-2-methyl-1,3-dioxo-2,3a,4,5,10b-hexahydropyrrolo[3,4-a]carbazole-10(1H)-carboxylate
3hh - benzyl (3aS*,5S*,10bS*)-5-(hydroxy(o-tolyl)amino)-7-methoxy-2-methyl-1,3-dioxo-2,3a,4,5,10b-hexahydropyrrolo[3,4-a]carbazole-10(1H)-carboxylate
1,3-dioxo-2,3,3a,4,5,10b-hexahydropyrrolo[3,4-a]carbazole-10(1H)-carboxylate

3ii - benzyl (3aS*, 5S*, 10bS*)-5-(hydroxy(phenyl)amino)-7-methoxy-1,3-dioxo-2,3,3a,4,5,10b-hexahydropyrrolo[3,4-a]carbazole-10(1H)-carboxylate

3jj - benzyl (3aS*, 5S*, 10bS*)-5-(hydroxy(o-toly)amino)-7-methoxy-1,3-dioxo-2,3,3a,4,5,10b-hexahydropyrrolo[3,4-a]carbazole-10(1H)-carboxylate

3kk - benzyl (R*)-6-(hydroxy(phenyl)amino)-8-methoxy-1,3-dioxo-2-phenyl-2,3,5,6-tetrahydro-1H,11H-[1,2,4]triazolo[1',2':1,2]pyridazino[3,4-b]indole-11-carboxylate

3ll - benzyl (R*)-6-(hydroxy(o-toly)amino)-8-methoxy-1,3-dioxo-2-phenyl-2,3,5,6-tetrahydro-1H,11H-[1,2,4]triazolo[1',2':1,2]pyridazino[3,4-b]indole-11-carboxylate

4a - (3aS*, 5S*, 10bS*)-5-(3,5-dioxo-4-phenyl-1,2,4-triazolidin-1-yl)-2-methyl-4,5,10,10b-tetrahydropyrrolo[3,4-a]carbazole-1,3(2H,3aH)-dione

4b - (3aS*, 5S*, 10bS*)-5-ethoxy-2-methyl-4,5,10,10b-tetrahydropyrrolo[3,4-a]carbazole-1,3(2H,3aH)-dione

4c - (3aS*, 5S*, 10bS*)-5-(hydroxy(phenyl)amino)-2-methyl-4,5,10,10b-tetrahydropyrrolo[3,4-a]carbazole-1,3(2H,3aH)-dione

4d - (3aS*, 5S*, 10bS*)-5-(hydroxy(o-toly)amino)-2-methyl-4,5,10,10b-tetrahydropyrrolo[3,4-a]carbazole-1,3(2H,3aH)-dione

4e - (3aS*, 5S*, 10bS*)-5-(3,5-dioxo-4-phenyl-1,2,4-triazolidin-1-yl)-2-methyl-4,5,10,10b-tetrahydropyrrolo[3,4-a]carbazole-1,3(2H,3aH)-dione

4f - (3aS*, 5S*, 10bS*)-5-(hydroxy(phenyl)amino)-4,5,10,10b-tetrahydropyrrolo[3,4-a]carbazole-1,3(2H,3aH)-dione

4g - (3aS*, 5S*, 10bS*)-5-(hydroxy(o-toly)amino)-4,5,10,10b-tetrahydropyrrolo[3,4-a]carbazole-1,3(2H,3aH)-dione

4h - (3aS*, 5S*, 10bS*)-5-(3,5-dioxo-4-phenyl-1,2,4-triazolidin-1-yl)-4,5,10,10b-tetrahydropyrrolo[3,4-a]carbazole-1,3(2H,3aH)-dione

4i - (R*)-6-(hydroxy(phenyl)amino)-2-phenyl-6,11-dihydro-1H,5H-[1,2,4]triazolo[1',2':1,2]pyridazino[3,4-b]indole-1,3(2H)-dione

4j - (R*)-6-(hydroxy(o-toly)amino)-2-phenyl-6,11-dihydro-1H,5H-[1,2,4]triazolo[1',2':1,2]pyridazino[3,4-b]indole-1,3(2H)-dione

4k - (3aS*, 5S*, 10bS*)-5-(hydroxy(phenyl)amino)-7-methoxy-2-methyl-4,5,10,10b-tetrahydropyrrolo[3,4-a]carbazole-1,3(2H,3aH)-dione

4l - (3aS*, 5S*, 10bS*)-5-(hydroxy(o-toly)amino)-7-methoxy-2-methyl-4,5,10,10b-tetrahydropyrrolo[3,4-a]carbazole-1,3(2H,3aH)-dione

4m - (3aS*, 5S*, 10bS*)-5-(hydroxy(phenyl)amino)-7-methoxy-4,5,10,10b-tetrahydropyrrolo[3,4-a]carbazole-1,3(2H,3aH)-dione

4n - (3aS*, 5S*, 10bS*)-5-(hydroxy(o-toly)amino)-7-methoxy-4,5,10,10b-tetrahydropyrrolo[3,4-a]carbazole-1,3(2H,3aH)-dione

4o - (R*)-6-(hydroxy(phenyl)amino)-8-methoxy-2-phenyl-6,11-dihydro-1H,5H-[1,2,4]triazolo[1',2':1,2]pyridazino[3,4-b]indole-1,3(2H)-dione

4p - (R*)-6-(hydroxy(o-toly)amino)-8-methoxy-2-phenyl-6,11-dihydro-1H,5H-[1,2,4]triazolo[1',2':1,2]pyridazino[3,4-b]indole-1,3(2H)-dione
Crystal Data and Structure Refinement Tables

2a - (3aS*,10aS*,10bS*)-2-methyl-10-tosyl-4,10,10a,10b-tetrahydropyrrolo[3,4-a]carbazole-1,3(2H,3aH)-dione  
2b - 2-phenyl-11-tosyl-11,11a-dihydro-1H,5H-[1,2,4]triazolo[1',2':1,2]pyridazino[3,4-b]indole-1,3(2H)-dione  
2d - ethyl 3-((3aS*,4R*,10aS*,10bS*)-1,3-dioxo-10-tosyl-1,2,3,3a,4,10,10a,10b-octahydropyrrolo[3,4-a]carbazol-4-yl)propanoate

3l - (3aS*,5S*,10bS*)-5-(hydroxy(o-tolyl)amino)-10-tosyl-4,5,10,10b-tetrahydropyrrolo[3,4-a]carbazole-1,3(2H,3aH)-dione  
3r - 6-(3,5-dioxo-4-phenyl-1,2,4-triazolidin-1-yl)-2-phenyl-11-tosyl-5,6-dihydro-[1,2,4]triazolo[1',2':1,2]pyridazino[3,4-b]indole-1,3(2H,11H)-dione  
3u - (3aS*,5S*,10bS*)-5-((2,6-dibromophenyl)(hydroxy)amino)-N,N-dimethyl-1,3-dioxo-2-phenyl-1,3,3a,4,5,10b-hexahydropyrrolo[3,4-a]carbazole-10(2H)-sulfonamide  
3v - (3aS*,5S*,10bS*)-5-((S*)-hydroxy(perfluorophenyl)methyl)-N,N-dimethyl-1,3-dioxo-1,3,3a,4,5,10b-hexahydropyrrolo[3,4-a]carbazole-10(2H)-sulfonamide

General experimental information

$^1$H and $^{13}$C NMR spectra were recorded directly with a Jeol Lambda 500 MHz, Jeol ECS-400 MHz or Bruker Avance 300 MHz. HRMS data were provided by the EPSRC National Mass Spectrometry Service (University of Swansea). X-ray diffraction data was obtained on an Oxford Diffraction Gemini. IR spectra were obtained as neat samples using a Varian 800 FT-IR Scimitar Series spectrometer scanning from 4000-600 cm$^{-1}$. THF and Et$_2$O were distilled from sodium/benzophenone and used directly. DCM was distilled from calcium hydride and used directly.
The image contains a chemical structure labeled as 3h, along with a 1D NMR spectrum. The spectrum shows a series of peaks with various intensities across different ppm ranges.
2a - (3aS*,10aS*,10bS*)-2-methyl-10-tosyl-4,10,10a,10b-tetrahydropyrrolo[3,4-\(\alpha\)]carbazole-1,3(2H,3aH)-dione

Table 1 Crystal data and structure refinement for MJH80.

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<td>(\beta/)°</td>
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<tr>
<td>(\gamma/)°</td>
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<td>Description</td>
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**2b** - 2-phenyl-11-tosyl-11a-dihydro-1H,5H-[1,2,4]triazolo[1',2':1,2]pyridazino[3,4-\(b\)]indole-1,3(2H)-dione

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**Table 1 Crystal data and structure refinement for MJH105.**

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<td>Crystal size/mm(^3)</td>
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<td>Radiation</td>
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<td>Independent reflections</td>
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<td>R$_1$ = 0.0702, wR$_2$ = 0.1528</td>
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<td>Largest diff. peak/hole / e Å$^{-3}$</td>
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</tbody>
</table>
**2d** - ethyl 3-((3aS*,4R*,10aS*,10bS*)-1,3-dioxo-10-tosyl-1,2,3,3a,4,10,10a,10b-octahydropyrrolo[3,4-α]carbazol-4-yl)propanoate

Table 1 Crystal data and structure refinement for mjh140029_fa.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Identification code</td>
<td>mjh140029_fa</td>
</tr>
<tr>
<td>Empirical formula</td>
<td>C_{26}H_{26}N_{2}O_{6}S</td>
</tr>
<tr>
<td>Formula weight</td>
<td>494.55</td>
</tr>
<tr>
<td>Temperature/K</td>
<td>150.00(10)</td>
</tr>
<tr>
<td>Crystal system</td>
<td>monoclinic</td>
</tr>
<tr>
<td>Space group</td>
<td>P2_1/n</td>
</tr>
<tr>
<td>a/Å</td>
<td>16.0840(2)</td>
</tr>
<tr>
<td>b/Å</td>
<td>8.84163(10)</td>
</tr>
<tr>
<td>c/Å</td>
<td>18.0999(2)</td>
</tr>
<tr>
<td>α/°</td>
<td>90</td>
</tr>
<tr>
<td>β/°</td>
<td>106.2054(14)</td>
</tr>
<tr>
<td>γ/°</td>
<td>90</td>
</tr>
<tr>
<td>Volume/Å³</td>
<td>2471.70(6)</td>
</tr>
<tr>
<td>Z</td>
<td>4</td>
</tr>
<tr>
<td>ρ calc/g/cm³</td>
<td>1.329</td>
</tr>
<tr>
<td>µ/mm⁻¹</td>
<td>1.537</td>
</tr>
<tr>
<td>F(000)</td>
<td>1040.0</td>
</tr>
<tr>
<td>Crystal size/mm³</td>
<td>0.2871 × 0.2849 × 0.1359</td>
</tr>
<tr>
<td>Radiation</td>
<td>CuKα (λ = 1.54184)</td>
</tr>
<tr>
<td>2Θ range for data collection/°</td>
<td>6.51 to 133.56</td>
</tr>
<tr>
<td>Index ranges</td>
<td>-19 ≤ h ≤ 19, -10 ≤ k ≤ 10, -21 ≤ l ≤ 21</td>
</tr>
<tr>
<td>Reflections collected</td>
<td>65129</td>
</tr>
<tr>
<td>Independent reflections</td>
<td>4373 [R_{int} = 0.0518, R_{sigma} = 0.0153]</td>
</tr>
<tr>
<td>Data/restraints/parameters</td>
<td>4373/345/341</td>
</tr>
<tr>
<td>Goodness-of-fit on F²</td>
<td>1.031</td>
</tr>
<tr>
<td>Final R indexes [I&gt;2σ (I)]</td>
<td>R₁ = 0.0351, wR₂ = 0.0896</td>
</tr>
<tr>
<td>Final R indexes [all data]</td>
<td>R₁ = 0.0392, wR₂ = 0.0929</td>
</tr>
<tr>
<td>Largest diff. peak/hole / e Å⁻³</td>
<td>0.32/-0.48</td>
</tr>
</tbody>
</table>
31 - (3aS*,5S*,10bS*)-5-(hydroxy(o-tolyl)amino)-10-tosyl-4,5,10,10b-tetrahydropyrrolo[3,4-a]carbazole-1,3(2H,3aH)-dione

Table 1 Crystal data and structure refinement for mjh120026.

<table>
<thead>
<tr>
<th>Identification code</th>
<th>mjh120026</th>
</tr>
</thead>
<tbody>
<tr>
<td>Empirical formula</td>
<td>C_{29}H_{26}O_{5}SN_{3}Cl_{3}</td>
</tr>
<tr>
<td>Formula weight</td>
<td>634.94</td>
</tr>
<tr>
<td>Temperature/K</td>
<td>150.0</td>
</tr>
<tr>
<td>Crystal system</td>
<td>triclinic</td>
</tr>
<tr>
<td>Space group</td>
<td>P-1</td>
</tr>
<tr>
<td>a/Å</td>
<td>10.0614(5)</td>
</tr>
<tr>
<td>b/Å</td>
<td>10.2208(5)</td>
</tr>
<tr>
<td>c/Å</td>
<td>15.2649(7)</td>
</tr>
<tr>
<td>α/°</td>
<td>104.231(4)</td>
</tr>
<tr>
<td>β/°</td>
<td>95.071(4)</td>
</tr>
<tr>
<td>γ/°</td>
<td>109.240(4)</td>
</tr>
<tr>
<td>Volume/Å³</td>
<td>1411.79(12)</td>
</tr>
<tr>
<td>Z</td>
<td>2</td>
</tr>
<tr>
<td>ρ\text{calc}/g/cm³</td>
<td>1.494</td>
</tr>
<tr>
<td>μ/mm⁻¹</td>
<td>4.017</td>
</tr>
<tr>
<td>F(000)</td>
<td>656.0</td>
</tr>
<tr>
<td>Crystal size/mm³</td>
<td>0.15 × 0.13 × 0.12</td>
</tr>
<tr>
<td>Radiation</td>
<td>CuKα (λ = 1.54178)</td>
</tr>
<tr>
<td>2Θ range for data collection/°</td>
<td>6.078 to 133.94</td>
</tr>
<tr>
<td>Index ranges</td>
<td>-11 ≤ h ≤ 11, -11 ≤ k ≤ 12, -17 ≤ l ≤ 18</td>
</tr>
<tr>
<td>Reflections collected</td>
<td>13047</td>
</tr>
</tbody>
</table>
Independent reflections 4955 [$R_{int} = 0.0264$, $R_{sigma} = 0.0239$]
Data/restraints/parameters 4955/411/397
Goodness-of-fit on $F^2$ 1.050
Final R indexes [$I>=2\sigma (I)$] $R_1 = 0.0490$, $wR_2 = 0.1265$
Final R indexes [all data] $R_1 = 0.0537$, $wR_2 = 0.1296$
Largest diff. peak/hole / e Å$^{-3}$ 0.93/-0.69
**Table 1 Crystal data and structure refinement for mjh140002.**

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
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</thead>
<tbody>
<tr>
<td>Identification code</td>
<td>mjh140002</td>
</tr>
<tr>
<td>Empirical formula</td>
<td>C\textsubscript{33}H\textsubscript{25}N\textsubscript{7}O\textsubscript{6}S</td>
</tr>
<tr>
<td>Formula weight</td>
<td>647.66</td>
</tr>
<tr>
<td>Temperature/K</td>
<td>150.00(10)</td>
</tr>
<tr>
<td>Crystal system</td>
<td>monoclinic</td>
</tr>
<tr>
<td>Space group</td>
<td>P2\textsubscript{1}/c</td>
</tr>
<tr>
<td>a/Å</td>
<td>13.1236(3)</td>
</tr>
<tr>
<td>b/Å</td>
<td>20.8432(5)</td>
</tr>
<tr>
<td>c/Å</td>
<td>12.0391(2)</td>
</tr>
<tr>
<td>α/°</td>
<td>90</td>
</tr>
<tr>
<td>β/°</td>
<td>100.991(2)</td>
</tr>
<tr>
<td>γ/°</td>
<td>90</td>
</tr>
<tr>
<td>Volume/Å\textsuperscript{3}</td>
<td>3232.74(13)</td>
</tr>
<tr>
<td>Z</td>
<td>4</td>
</tr>
<tr>
<td>ρ\textsubscript{calc} mg/mm\textsuperscript{3}</td>
<td>1.331</td>
</tr>
<tr>
<td>m/mm\textsuperscript{-1}</td>
<td>1.360</td>
</tr>
<tr>
<td>F(000)</td>
<td>1344.0</td>
</tr>
<tr>
<td>Crystal size/mm\textsuperscript{3}</td>
<td>0.25 \times 0.1 \times 0.06</td>
</tr>
<tr>
<td>Radiation</td>
<td>CuKα (λ = 1.54184)</td>
</tr>
<tr>
<td>2Θ range for data collection</td>
<td>6.862 to 132.494°</td>
</tr>
<tr>
<td>Index ranges</td>
<td>-14 ≤ h ≤ 15, -24 ≤ k ≤ 24, -14 ≤ l ≤ 10</td>
</tr>
<tr>
<td>Reflections collected</td>
<td>23327</td>
</tr>
<tr>
<td>Independent reflections</td>
<td>5646 [R\textsubscript{int} = 0.0383, R\textsubscript{sigma} = 0.0297]</td>
</tr>
<tr>
<td>Data/restraints/parameters</td>
<td>5646/0/428</td>
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</tbody>
</table>
Goodness-of-fit on $F^2$ 1.036
Final R indexes [$I>2\sigma(I)$] $R_1 = 0.0357$, $wR_2 = 0.0863$
Final R indexes [all data] $R_1 = 0.0464$, $wR_2 = 0.0927$
Largest diff. peak/hole / e Å$^{-3}$ 0.35/-0.36
3u - (3a5*,5S*,10b5*)-5-((2,6-dibromophenyl)(hydroxy)amino)-N,N-dimethyl-1,3-dioxo-2-phenyl-1,3,3a,4,5,10b-hexahydropyrrolo[3,4-a]carbazole-10(2H)-sulfonamide

Table 1 Crystal data and structure refinement for mjh69.

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
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<tbody>
<tr>
<td>Identification code</td>
<td>mjh69</td>
</tr>
<tr>
<td>Empirical formula</td>
<td>C_{32}H_{34}O_{6}SBr_2N_4</td>
</tr>
<tr>
<td>Formula weight</td>
<td>762.51</td>
</tr>
<tr>
<td>Temperature/K</td>
<td>150.00(10)</td>
</tr>
<tr>
<td>Crystal system</td>
<td>triclinic</td>
</tr>
<tr>
<td>Space group</td>
<td>P-1</td>
</tr>
<tr>
<td>a/Å</td>
<td>10.0627(11)</td>
</tr>
<tr>
<td>b/Å</td>
<td>12.5064(17)</td>
</tr>
<tr>
<td>c/Å</td>
<td>13.7858(16)</td>
</tr>
<tr>
<td>α/°</td>
<td>87.321(10)</td>
</tr>
<tr>
<td>β/°</td>
<td>78.605(10)</td>
</tr>
<tr>
<td>γ/°</td>
<td>70.030(11)</td>
</tr>
<tr>
<td>Volume/Å³</td>
<td>1598.0(4)</td>
</tr>
<tr>
<td>Z</td>
<td>2</td>
</tr>
<tr>
<td>ρ_{calc}g/cm³</td>
<td>1.585</td>
</tr>
<tr>
<td>μ/mm⁻¹</td>
<td>2.653</td>
</tr>
<tr>
<td>F(000)</td>
<td>776.0</td>
</tr>
<tr>
<td>Crystal size/mm³</td>
<td>0.7113 × 0.156 × 0.1482</td>
</tr>
<tr>
<td>Radiation</td>
<td>MoKα (λ = 0.71073)</td>
</tr>
<tr>
<td>2Θ range for data collection/°</td>
<td>5.788 to 53.596</td>
</tr>
<tr>
<td>Index ranges</td>
<td>-12 ≤ h ≤ 12, -15 ≤ k ≤ 9, -17 ≤ l ≤ 15</td>
</tr>
<tr>
<td>Reflections collected</td>
<td>10012</td>
</tr>
</tbody>
</table>
Independent reflections 6056 [R_{int} = 0.0263, R_{sigma} = 0.0504]
Data/restraints/parameters 6056/366/414
Goodness-of-fit on F^2 1.037
Final R indexes [I>=2\sigma (I)] R_1 = 0.0604, wR_2 = 0.1457
Final R indexes [all data] R_1 = 0.0755, wR_2 = 0.1595
Largest diff. peak/hole / e Å^{-3} 3.75/-0.92
3v - (3aS*,5S*,10bS*)-5-((S*)-hydroxy(perfluorophenyl)methyl)-N,N-dimethyl-1,3-dioxo-1,3,3a,4,5,10b-hexahydropyrrolo[3,4-a]carbazole-10(2H)-sulfonamide

Table 1 Crystal data and structure refinement for mjh120042.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Identification code</td>
<td>mjh120042</td>
</tr>
<tr>
<td>Empirical formula</td>
<td>C_{24}H_{20}N_{3}O_{5}F_{5}S</td>
</tr>
<tr>
<td>Formula weight</td>
<td>557.49</td>
</tr>
<tr>
<td>Temperature/K</td>
<td>150.00</td>
</tr>
<tr>
<td>Crystal system</td>
<td>monoclinic</td>
</tr>
<tr>
<td>Space group</td>
<td>P2_1/n</td>
</tr>
<tr>
<td>a/Å</td>
<td>7.9070(4)</td>
</tr>
<tr>
<td>b/Å</td>
<td>14.2862(7)</td>
</tr>
<tr>
<td>c/Å</td>
<td>20.6839(10)</td>
</tr>
<tr>
<td>α/°</td>
<td>90</td>
</tr>
<tr>
<td>β/°</td>
<td>101.016(5)</td>
</tr>
<tr>
<td>γ/°</td>
<td>90</td>
</tr>
<tr>
<td>Volume/Å³</td>
<td>2293.4(2)</td>
</tr>
<tr>
<td>Z</td>
<td>4</td>
</tr>
<tr>
<td>ρ calc /g/cm³</td>
<td>1.615</td>
</tr>
<tr>
<td>μ /mm⁻¹</td>
<td>0.227</td>
</tr>
<tr>
<td>F(000)</td>
<td>1144.0</td>
</tr>
<tr>
<td>Crystal size/mm³</td>
<td>0.15 × 0.1 × 0.1</td>
</tr>
<tr>
<td>Radiation</td>
<td>MoKα (λ = 0.71073)</td>
</tr>
<tr>
<td>2Θ range for data collection/°</td>
<td>5.704 to 57.17</td>
</tr>
<tr>
<td>Index ranges</td>
<td>-10 ≤ h ≤ 10, -18 ≤ k ≤ 18, -26 ≤ l ≤ 24</td>
</tr>
<tr>
<td>Reflections collected</td>
<td>23890</td>
</tr>
<tr>
<td>Independent reflections</td>
<td>5125 [R_{int} = 0.0596, R_{sigma} = 0.0600]</td>
</tr>
<tr>
<td>Data/restraints/parameters</td>
<td>5125/0/350</td>
</tr>
<tr>
<td>Goodness-of-fit on F²</td>
<td>1.027</td>
</tr>
<tr>
<td>Final R indexes [I≥2σ (I)]</td>
<td>R₁ = 0.0492, wR₂ = 0.0954</td>
</tr>
<tr>
<td>Final R indexes [all data]</td>
<td>R₁ = 0.0864, wR₂ = 0.1108</td>
</tr>
<tr>
<td>Largest diff. peak/hole / e Å⁻³</td>
<td>0.34/-0.37</td>
</tr>
</tbody>
</table>

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