# Electronic Supplementary Information (ESI)

# Synthesis of the core ring system of the stemona alkaloids by cascade condensation, cyclization, intramolecular cycloaddition

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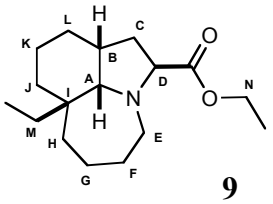
## Contents

## 1. General experimental

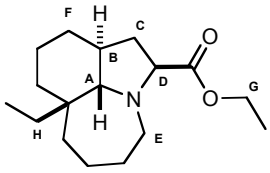
All reagents were obtained from commercial suppliers and were used without further purification unless otherwise specified. Solvents were purified using a Grubbs dry solvent system (model SPS-200-6). Thin layer chromatography was performed on silica plates and visualised by UV irradiation at 254 nm or by staining with an alkaline $KMnO_4$ dip. Column chromatography was performed using silica gel (40–63 micron mesh). Infrared spectra were recorded on Perkin Elmer Spectrum RX Fourier Transform IR System. Only selected peaks are reported and absorption maxima are given in $cm^{-1}$. $^1H$ NMR spectra were recorded on a Bruker AC400 (400 MHz) instrument. Chemical shifts are reported in ppm with respect to the residual solvent peaks, with multiplicities given as s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet. Coupling constants, *J*, are quoted to the nearest 0.5 Hz. $^{13}C$ NMR were recorded on the above instrument at 100 MHz; $^1H$–$^1H$ and $^1H$–$^{13}C$ correlation spectra were run to confirm the assignment of peaks. Low and high resolution (accurate mass) mass spectra were recorded on a Walters LCT instrument for Electro–Spray (ES).

## 2. Procedures and spectroscopic data for the cyclization–cycloaddition cascade using aldehydes 7, 11 and 16

*Using aldehyde **7** (to give cycloadducts **9** and **10**):*



**9**



**10**

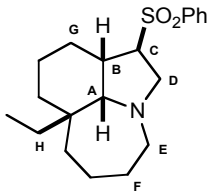
The aldehyde **7** (515 mg, 1.6 mmol) (prepared as shown in Scheme 2), glycine ethyl ester hydrochloride (338 mg, 2.4 mmol) and *N*,*N*-diisopropylethylamine (0.84 mL, 4.8 mmol) in PhMe (15 mL) were heated under reflux. After 36 h, the mixture was allowed to cool to room temperature and the solvent was evaporated. Purification by column chromatography, eluting with petrol–EtOAc (97:3 to 4:1), gave the cycloadduct **9** (124 mg, 28%) as an oil and the cycloadduct **10** (103 mg, 23%) as an oil.

Data for **9**; $R_f$ 0.36 [petrol–EtOAc (19:1)]; $\nu_{max}/cm^{-1}$ 2930, 2860, 1730, 1450; $^1H$ NMR (500 MHz, $CDCl_3$) δ = 4.12 (2H, q, *J* 7 Hz, 2 × $CH^N$), 3.90 (1H, dd, *J* 8, 6 Hz, $CH^D$), 2.76–2.66 (3H, m, $CH^A$ and 2 × $CH^E$), 2.34–2.27 (1H, m, $CH^B$), 1.87 (1H, ddd, *J* 12.5, 7, 6 Hz, $CH^C$), 1.72 (1H, ddd, *J* 12.5, 8, 3.5 Hz, $CH^C$), 1.66–1.27 (12H, m, 2 × $CH^F$, 2 x $CH^G$, 2 x $CH^H$, 2 x $CH^J$, $CH^K$, 2 x $CH^L$ and $CH^M$), 1.25 (3H, t, *J* 7 Hz, $OCH_2CH_3$), 1.23–1.15 (1H, m, $CH^M$), 1.13–1.07 (1H, m, $CH^K$), 0.77 (3H, t, *J* 7.5 Hz, $CH_3$); $^{13}C$ NMR (125 MHz, $CDCl_3$) δ = 175.2 (C=O), 70.9 ($CH^A$), 66.1 ($CH^D$), 60.0 ($CH_2^N$), 50.0 ($CH_2^E$), 38.0 (C), 36.1 ($CH^B$), 35.0 ($CH_2^C$), 34.5 ($CH_2$), 30.4 ($CH_2^M$), 28.9 ($CH_2^F$), 27.7 ($CH_2^L$), 27.2 ($CH_2^K$), 20.8 ($CH_2$), 19.7 ($CH_2$), 14.4 ($CH_3$), 7.5 ($CH_3$); HRMS (ES) Found: $MH^+$, 280.2273. $C_{17}H_{30}NO_2$ requires $MH^+$, 280.2277; LRMS *m/z* (ES) 280 (100%, $MH^+$).

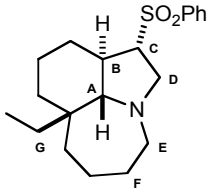
Data for **10**; $R_f$ 0.36 [petrol–EtOAc (5:1)]; $\nu_{max}/cm^{-1}$ 2930, 2860, 1735, 1445; $^1H$ NMR (500 MHz, $CDCl_3$) δ = 4.13 (2H, q, *J* 7 Hz, 2 x $CH^G$), 3.54 (1H, dd, *J* 10, 7 Hz,

$CH^D$), 3.06 (1H, dd, *J* 15, 10 Hz, $CH^E$), 2.89 (1H, dd, *J* 15, 6 Hz, $CH^E$), 2.60 (1H, d, *J* 11 Hz, $CH^A$), 2.19–2.13 (1H, m, $CH^C$), 1.94–1.73 (4H, m, $CH^B$, $CH^H$ and 2 × CH), 1.65–1.59 (1H, m, CH), 1.55–1.30 (9H, m, $CH^C$, 2 × $CH^F$, $CH^H$ and 5 × CH), 1.23 (3H, t, *J* 7 Hz, $OCH_2CH_3$), 1.15–1.08 (1H, m, CH), 0.95 (1H, qd, *J* 12.0, 4.5 Hz, CH), 0.84 (3H, t, *J* 7.5 Hz, $CH_3$); $^{13}C$ NMR (125 MHz, $CDCl_3$) δ = 175.7 (C=O), 73.9 ($CH^A$), 65.5 ($CH^D$), 60.3 ($CH_2{}^G$), 52.9 ($CH_2{}^E$), 40.6 (C), 40.1 ($CH^B$), 38.3 ($CH_2$), 35.7 ($CH_2{}^C$), 31.7 ($CH_2$), 31.3 ($CH_2$), 30.5 ($CH_2$), 27.8 ($CH_2$), 25.1 ($CH_2$), 21.4 ($CH_2$), 14.2 ($CH_3$), 7.0 ($CH_3$); HRMS (ES) Found: $MH^+$, 280.2280. $C_{17}H_{30}NO_2$ requires $MH^+$, 280.2277; LRMS *m/z* (ES) 280 (100%, $MH^+$).

*Using aldehyde **11** (to give cycloadducts **12** and **13**):*




**12**




**13**

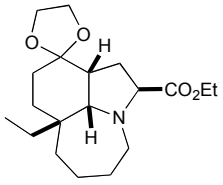
The aldehyde **11** (400 mg, 0.87 mmol) (prepared as shown in Scheme 4) and glycine (260 mg, 3.46 mmol) in xylenes (9 mL) were heated under reflux. After 5 h, further glycine (260 mg, 3.46 mmol) was added and the mixture heated under reflux. After 18 h, the mixture was allowed to cool to room temperature, filtered. washed with $CH_2Cl_2$ (20 mL) and evaporated. Purification by column chromatography, eluting with $CH_2Cl_2$–MeOH–$NH_3$ (99:1:0.1 to 95:5:0.1), gave the amine **12** (145 mg, 48%) as a solid, which recrystallised from $CH_2Cl_2$–petrol as needles, and the amine **13** (54 mg, 18%) as an oil, which solidified on standing to give a gum.

Data for **12**; m.p. 144–146 °C; $R_f$ 0.34 [$CH_2Cl_2$–MeOH–$NH_3$ (97:3:0.1)]; $\nu_{max}/cm^{-1}$ 2930, 2860, 2815, 1465, 1445, 1300, 1140; $^1H$ NMR (500 MHz, $CDCl_3$) δ = 7.90−7.87 (2H, m, ArH), 7.64 (1H, tt, *J* 7.5, 1.5 Hz, ArH), 7.58−7.53 (2H, m, ArH), 3.48 (1H, dd, *J* 12, 8 Hz, $CH^D$), 3.08 (1H, ddd, *J* 8, 5, 1 Hz, $CH^C$), 2.90 (1H, dd, *J* 12, 5 Hz, $CH^D$), 2.89−2.84 (1H, m, $CH^E$), 2.71−2.65 (1H, m, $CH^B$), 2.58−2.51 (1H, m,

$CH^E$), 2.38 (1H, d, *J* 5.5 Hz, $CH^A$), 1.62−1.49 (4H, 2 × $CH^F$ and 2 × CH), 1.48−1.31 (5H, m, $CH^G$, $CH^H$ and 3 × CH), 1.30−1.13 (5H, m, $CH^G$, $CH^H$ and 3 × CH), 0.75 (3H, t, *J* 7.5 Hz, $CH_3$); $^{13}$C NMR (125 MHz, $CDCl_3$) δ = 138.5 (ArC), 133.6 (ArCH), 129.3 (ArCH), 128.5 (ArCH), 74.0 ($CH^A$), 68.4 ($CH^C$), 57.2 ($CH_2^E$), 56.1 ($CH_2^D$), 39.4 ($CH^B$), 37.4 (C), 35.6 ($CH_2$), 30.2 ($CH_2$), 29.6, 29.4, 29.2 (3 x $CH_2$), 20.3 ($CH_2$), 20.0 ($CH_2$), 7.3 ($CH_3$); HRMS (ES) Found: $MH^+$, 348.1982. $C_{20}H_{30}NO_2S$ requires $MH^+$, 348.1997; LRMS *m/z* (ES) 348 (100%, $MH^+$); Anal. Calcd. for $C_{20}H_{29}NO_2S$: C, 69.12; H, 8.41; N, 4.03; S, 9.23. Found: C, 69.06; H, 8.47; N, 3.76; S, 9.25 %.

Data for **13**; $R_f$ 0.34 [$CH_2Cl_2$–MeOH–$NH_3$ (95:5:0.1)]; $\nu_{max}/cm^{-1}$ 2930, 2860, 1445, 1305, 1145; $^1$H NMR (500 MHz, $CDCl_3$) δ = 7.84−7.81 (2H, m, ArH), 7.64−7.60 (1H, m, ArH), 7.57−7.52 (2H, m, ArH), 3.25 (1H, dd, *J* 13, 9.5 Hz, $CH^D$), 3.18−3.10 (2H, m, $CH^C$ and $CH^D$), 2.86 (1H, ddd, *J* 13.5, 9, 1 Hz, $CH^E$), 2.53 (1H, ddd, *J* 13.5, 7.5, 1 Hz, $CH^E$), 2.43−2.34 (1H, m, $CH^B$), 2.22 (1H, d, *J* 11.5 Hz, $CH^A$), 1.93−1.77 (2H, m, $CH^G$ and CH), 1.77−1.61 (2H, m, 2 × $CH^F$), 1.60−1.20 (8H, m, $CH^G$ and 7 × CH), 1.00 (1H, td, *J* 13, 4 Hz, CH), 0.88 (1H, t, *J* 7 Hz, CH), 0.81 (3H, t, *J* 7.5 Hz, $CH_3$); $^{13}$C NMR (125 MHz, $CDCl_3$) δ = 139.2 (ArC), 133.6 (ArCH), 129.3 (ArCH), 128.2 (ArCH), 76.8 ($CH^A$), 68.4 ($CH^C$), 55.8 ($CH_2^D$), 52.7 ($CH_2^E$), 39.6 (C), 37.9 ($CH^B$), 37.4 ($CH_2$), 31.5 ($CH_2^G$), 30.2 ($CH_2$), 28.4 ($CH_2$), 27.3 ($CH_2^F$), 22.3 ($CH_2$), 20.7 ($CH_2$), 7.4 ($CH_3$); HRMS (ES) Found: $MH^+$, 348.2002. $C_{20}H_{30}NO_2S$ requires $MH^+$, 348.1997; LRMS *m/z* (ES) 348 (100%, $MH^+$).

*Using aldehyde **16** (to give cycloadduct **17**):*



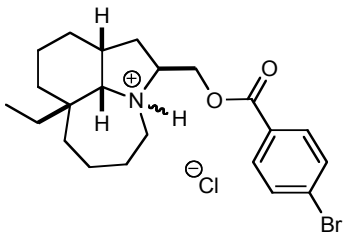

**17**

The aldehyde **16** (300 mg, 0.79 mmol) (prepared as shown in Scheme 5), glycine ethyl ester hydrochloride (160 mg, 1.18 mmol), *N*,*N*-diisopropylethylamine (0.41 mL, 2.37 mmol) and camphor sulfonic acid (1 mg, 0.04 mmol) in xylenes (10 mL) were

heated under reflux. After 16 h, the mixture was allowed to cool to room temperature and the solvent was evaporated. Purification by column chromatography, eluting with petrol–EtOAc (19:1–9:1), gave the cycloadduct **17** (116 mg, 49%) as an oil; $\nu_{max}/cm^{-1}$ 2930, 1725, 1460; $^{1}H$ NMR (500 MHz, $CDCl_3$) δ = 4.11 (2H, q, *J* 7 Hz, $OCH_2$), 3.99–3.91 (4H, m, 2 × $CH_2$), 3.90–3.86 (1H, m, CH), 2.92 (1H, d, *J* 6 Hz, CH), 2.84–2.78 (1H, m, CH), 2.67–2.60 (2H, m, 2 × CH), 2.10–2.03 (1H, m, CH), 1.83–1.74 (1H, m, CH), 1.73–1.32 (11H, m, 5 × $CH_2$, CH), 1.24 (3H, t, *J* 7 Hz, $CH_3$), 1.16 (1H, m, CH), 0.77 (3H, t, *J* 7.5 Hz, $CH_3$); $^{13}C$ NMR (125 MHz, $CDCl_3$) δ = 175.2, 109.7, 72.1, 66.9, 65.1, 63.6, 60.0, 50.8, 44.3, 37.8, 33.8, 29.7, 29.5, 29.5, 28.5, 25.8, 20.4, 14.3, 7.5; HRMS (ES) Found: $MH^+$, 338.2331. $C_{19}H_{32}NO_4$ requires $MH^+$, 338.2341. LRMS *m/z* (ES) 338 ($MH^+$, 100%).

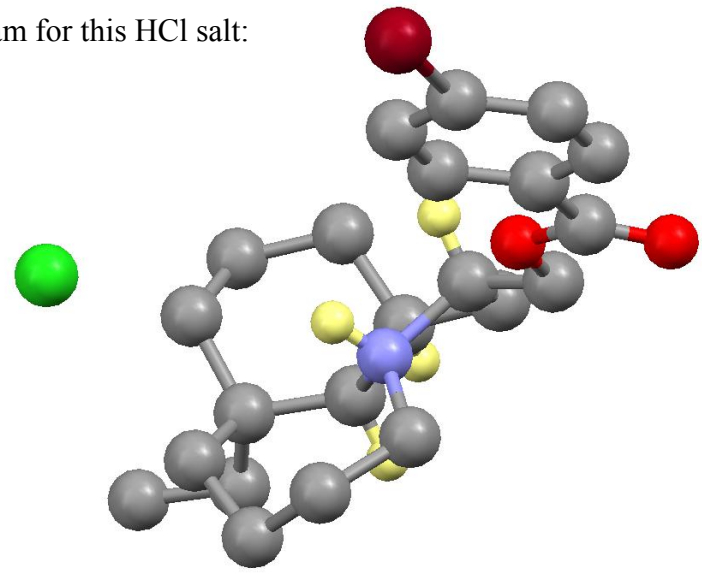
## 3. ORTEP diagram and CIF data for the *p*-bromobenzoate obtained from 9

Compound **9** was converted to 4-bromobenzoic acid (2SR,7aSR,10aRS,10bSR)-7a-ethyldodecahydroazepino[3,2,1-*hi*]indol-2-ylmethyl ester hydrochloride salt using $LiAlH_4$ (2 equiv.) in THF; the crude alcohol was treated with 4-bromobenzoyl chloride (2 equiv.) and $Et_3N$ (3 equiv.) in $CH_2Cl_2$ and the ester, after purification by column chromatography, was treated with HCl (1 M solution in $Et_2O$).




This HCl salt was recrystallised from $CH_2Cl_2$–$Et_2O$–petrol as cubes; m.p. 243 °C (dec); $\nu_{max}/cm^{-1}$ 2925, 2865, 2475, 1725, 1265; $^1H$ NMR (250 MHz, $CDCl_3$, 9:1 mixture of diastereomeric ammonium salts) δ = 12.27 (0.1H, br s, NH), 11.80 (0.9H, br s, NH), 7.99 (2H, d, *J* 8.5 Hz, ArH), 7.59 (2H, d, *J* 8.5 Hz, ArH), 5.30–5.19 (0.1H, m, CH), 4.89–4.70 (0.9H, m, CH), 4.69–4.51 (1H, m, CH), 4.11–4.03 (0.1H, m, CH), 3.80–3.63 (0.9H, m, CH), 3.59–3.41 (0.1H, m, CH), 3.09–2.97 (0.9H, m, CH), 2.87–2.20 (3H, m, 3 × CH), 2.09–1.10 (16H, m, 16 × CH), 0.78 (3H, t, *J* 7.0 Hz, $CH_2CH_3$); $^{13}C$ NMR (100 MHz, $CDCl_3$, only peaks of major diastereomeric HCl observed) δ = 165.2 (C=O), 132.2 (ArCH), 131.6 (ArCH), 129.0 (ArC), 127.8 (ArC), 80.1 (CH), 64.1 (CH), 61.8 ($CH_2$), 55.3 ($CH_2$), 37.3 ($CH_2$), 34.9 ($CH_2$), 34.8 (CH), 34.3 (C), 33.2 ($CH_2$), 29.9 ($CH_2$), 27.5 ($CH_2$), 27.1 ($CH_2$), 20.2 ($CH_2$), 19.1 ($CH_2$), 7.2 ($CH_3$); HRMS (ES) Found: $M^+$–Cl, 420.1533. $C_{22}H_{31}NO_2{}^{79}Br$ requires $M^+$–Cl, 420.1538; LRMS *m/z* (ES) 422 (100%, $M^+$–Cl ($^{81}Br$)), 420 (100, $M^+$–Cl ($^{79}Br$)); Anal. Calcd for $C_{22}H_{31}BrClNO_2$: C, 57.84; H, 6.84; N, 3.07; Br, 17.49; Cl, 7.76. Found: C, 57.66; H, 6.86; N, 2.94; Br, 17.70; Cl, 7.90.

Ortep diagram for this HCl salt:



Results are deposited at the Cambridge Crystallographic Data Centre.

Structure Number CCDC-782368.

Table 1. Crystal data and structure refinement for this HCl salt (reference number **oic66_0m**).

| | | |
|---|---|---|
| Identification code | oic66_0m | |
| Empirical formula | C22 H31 Br Cl N O2 | |
| Formula weight | 456.84 | |
| Temperature | 296(2) K | |
| Wavelength | 0.71073 Å | |
| Crystal system | Monoclinic | |
| Space group | $P2_1/n$ | |
| Unit cell dimensions | a = 7.8562(4) Å | α= 90°. |
| | b = 11.9575(6) Å | β= 95.316(3)°. |
| | c = 23.0397(11) Å | γ = 90°. |
| Volume | 2155.05(19) $Å^3$ | |
| Z | 4 | |
| Density (calculated) | 1.408 $Mg/m^3$ | |
| Absorption coefficient | 2.048 $mm^{-1}$ | |
| F(000) | 952 | |
| Crystal size | 0.32 x 0.21 x 0.21 $mm^3$ | |
| Theta range for data collection | 1.78 to 29.85°. | |
| Index ranges | -10<=h<=10, -16<=k<=16, -32<=l<=32 | |
| Reflections collected | 63019 | |

| | |
|---|---|
| Independent reflections | 6158 [R(int) = 0.0281] |
| Completeness to theta = 29.85° | 99.5 % |
| Absorption correction | Semi-empirical from equivalents |
| Max. and min. transmission | 0.6730 and 0.5602 |
| Refinement method | Full-matrix least-squares on $F^2$ |
| Data / restraints / parameters | 6158 / 0 / 245 |
| Goodness-of-fit on $F^2$ | 1.030 |
| Final R indices [I>2sigma(I)] | R1 = 0.0433, wR2 = 0.1096 |
| R indices (all data) | R1 = 0.0754, wR2 = 0.1262 |
| Largest diff. peak and hole | 0.827 and -0.806 e.$Å^{-3}$ |

Table 2. Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters ($Å^2$x $10^3$) for oic66_0m. U(eq) is defined as one third of the trace of the orthogonalized $U^{ij}$ tensor.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| Br(1) | 2331(1) | 2930(1) | 10482(1) | 86(1) |
| Cl(1) | 4896(1) | 5899(1) | 2266(1) | 56(1) |
| O(1) | 3640(2) | 6696(1) | 8393(1) | 47(1) |
| O(2) | 2775(4) | 7940(2) | 9025(1) | 84(1) |
| N(1) | 5958(2) | 6488(1) | 7389(1) | 32(1) |
| C(1) | 4178(3) | 7021(2) | 7411(1) | 38(1) |
| C(2) | 4010(3) | 7820(2) | 6897(1) | 49(1) |
| C(3) | 4896(3) | 7206(2) | 6435(1) | 47(1) |
| C(4) | 3789(3) | 6282(3) | 6138(1) | 62(1) |
| C(5) | 4813(4) | 5478(3) | 5797(1) | 69(1) |
| C(6) | 6240(3) | 4980(2) | 6206(1) | 57(1) |
| C(7) | 7499(3) | 5853(2) | 6490(1) | 41(1) |
| C(8) | 8804(3) | 5246(2) | 6918(1) | 50(1) |
| C(9) | 9785(3) | 5935(2) | 7394(1) | 50(1) |
| C(10) | 8830(3) | 6088(2) | 7932(1) | 53(1) |
| C(11) | 7261(3) | 6836(2) | 7874(1) | 43(1) |
| C(12) | 6528(3) | 6777(2) | 6786(1) | 36(1) |
| C(13) | 3799(3) | 7569(2) | 7971(1) | 47(1) |
| C(14) | 3093(3) | 6995(2) | 8905(1) | 50(1) |
| C(15) | 2939(3) | 6005(2) | 9285(1) | 48(1) |
| C(16) | 3447(4) | 4953(2) | 9123(1) | 56(1) |

| | | | | |
|---|---|---|---|---|
| C(17) | 3285(4) | 4046(2) | 9485(1) | 61(1) |
| C(18) | 2609(4) | 4196(2) | 10006(1) | 59(1) |
| C(19) | 2120(4) | 5241(3) | 10182(1) | 74(1) |
| C(20) | 2288(4) | 6146(3) | 9821(1) | 65(1) |
| C(21) | 8471(3) | 6478(2) | 6029(1) | 53(1) |
| C(22) | 9226(4) | 5753(3) | 5572(1) | 72(1) |

Table 3. Bond lengths [Å] and angles [°] for oic66_0m.

| | |
|---|---|
| Br(1)-C(18) | 1.894(3) |
| O(1)-C(14) | 1.340(3) |
| O(1)-C(13) | 1.440(3) |
| O(2)-C(14) | 1.196(3) |
| N(1)-C(11) | 1.503(3) |
| N(1)-C(12) | 1.537(3) |
| N(1)-C(1) | 1.542(2) |
| N(1)-H(1A) | 0.9100 |
| C(1)-C(13) | 1.501(3) |
| C(1)-C(2) | 1.519(3) |
| C(1)-H(1) | 0.9800 |
| C(2)-C(3) | 1.515(3) |
| C(2)-H(2A) | 0.9700 |
| C(2)-H(2B) | 0.9700 |
| C(3)-C(4) | 1.528(4) |
| C(3)-C(12) | 1.540(3) |
| C(3)-H(3) | 0.9800 |
| C(4)-C(5) | 1.518(4) |
| C(4)-H(4A) | 0.9700 |
| C(4)-H(4B) | 0.9700 |
| C(5)-C(6) | 1.517(4) |
| C(5)-H(5A) | 0.9700 |
| C(5)-H(5B) | 0.9700 |
| C(6)-C(7) | 1.541(3) |
| C(6)-H(6A) | 0.9700 |
| C(6)-H(6B) | 0.9700 |
| C(7)-C(8) | 1.538(3) |
| C(7)-C(12) | 1.538(3) |

| | |
|---|---|
| C(7)-C(21) | 1.555(3) |
| C(8)-C(9) | 1.523(4) |
| C(8)-H(8A) | 0.9700 |
| C(8)-H(8B) | 0.9700 |
| C(9)-C(10) | 1.518(4) |
| C(9)-H(9A) | 0.9700 |
| C(9)-H(9B) | 0.9700 |
| C(10)-C(11) | 1.519(3) |
| C(10)-H(10A) | 0.9700 |
| C(10)-H(10B) | 0.9700 |
| C(11)-H(11A) | 0.9700 |
| C(11)-H(11B) | 0.9700 |
| C(12)-H(12) | 0.9800 |
| C(13)-H(13A) | 0.9700 |
| C(13)-H(13B) | 0.9700 |
| C(14)-C(15) | 1.484(4) |
| C(15)-C(16) | 1.382(3) |
| C(15)-C(20) | 1.390(4) |
| C(16)-C(17) | 1.381(4) |
| C(16)-H(16) | 0.9300 |
| C(17)-C(18) | 1.370(4) |
| C(17)-H(17) | 0.9300 |
| C(18)-C(19) | 1.380(4) |
| C(19)-C(20) | 1.379(4) |
| C(19)-H(19) | 0.9300 |
| C(20)-H(20) | 0.9300 |
| C(21)-C(22) | 1.527(3) |
| C(21)-H(21A) | 0.9700 |
| C(21)-H(21B) | 0.9700 |
| C(22)-H(22A) | 0.9600 |
| C(22)-H(22B) | 0.9600 |
| C(22)-H(22C) | 0.9600 |

| | |
|---|---|
| C(14)-O(1)-C(13) | 117.10(18) |
| C(11)-N(1)-C(12) | 111.98(15) |
| C(11)-N(1)-C(1) | 114.70(16) |
| C(12)-N(1)-C(1) | 106.19(15) |
| C(11)-N(1)-H(1A) | 107.9 |

| | |
|---|---|
| C(12)-N(1)-H(1A) | 107.9 |
| C(1)-N(1)-H(1A) | 107.9 |
| C(13)-C(1)-C(2) | 112.71(18) |
| C(13)-C(1)-N(1) | 117.53(18) |
| C(2)-C(1)-N(1) | 104.30(16) |
| C(13)-C(1)-H(1) | 107.3 |
| C(2)-C(1)-H(1) | 107.3 |
| N(1)-C(1)-H(1) | 107.3 |
| C(3)-C(2)-C(1) | 103.38(17) |
| C(3)-C(2)-H(2A) | 111.1 |
| C(1)-C(2)-H(2A) | 111.1 |
| C(3)-C(2)-H(2B) | 111.1 |
| C(1)-C(2)-H(2B) | 111.1 |
| H(2A)-C(2)-H(2B) | 109.1 |
| C(2)-C(3)-C(4) | 112.7(2) |
| C(2)-C(3)-C(12) | 101.89(18) |
| C(4)-C(3)-C(12) | 113.97(19) |
| C(2)-C(3)-H(3) | 109.4 |
| C(4)-C(3)-H(3) | 109.4 |
| C(12)-C(3)-H(3) | 109.4 |
| C(5)-C(4)-C(3) | 112.5(2) |
| C(5)-C(4)-H(4A) | 109.1 |
| C(3)-C(4)-H(4A) | 109.1 |
| C(5)-C(4)-H(4B) | 109.1 |
| C(3)-C(4)-H(4B) | 109.1 |
| H(4A)-C(4)-H(4B) | 107.8 |
| C(6)-C(5)-C(4) | 108.9(2) |
| C(6)-C(5)-H(5A) | 109.9 |
| C(4)-C(5)-H(5A) | 109.9 |
| C(6)-C(5)-H(5B) | 109.9 |
| C(4)-C(5)-H(5B) | 109.9 |
| H(5A)-C(5)-H(5B) | 108.3 |
| C(5)-C(6)-C(7) | 113.9(2) |
| C(5)-C(6)-H(6A) | 108.8 |
| C(7)-C(6)-H(6A) | 108.8 |
| C(5)-C(6)-H(6B) | 108.8 |
| C(7)-C(6)-H(6B) | 108.8 |
| H(6A)-C(6)-H(6B) | 107.7 |

| | |
|---|---|
| C(8)-C(7)-C(12) | 112.49(18) |
| C(8)-C(7)-C(6) | 108.45(19) |
| C(12)-C(7)-C(6) | 110.48(18) |
| C(8)-C(7)-C(21) | 108.93(19) |
| C(12)-C(7)-C(21) | 104.61(17) |
| C(6)-C(7)-C(21) | 111.9(2) |
| C(9)-C(8)-C(7) | 118.00(18) |
| C(9)-C(8)-H(8A) | 107.8 |
| C(7)-C(8)-H(8A) | 107.8 |
| C(9)-C(8)-H(8B) | 107.8 |
| C(7)-C(8)-H(8B) | 107.8 |
| H(8A)-C(8)-H(8B) | 107.1 |
| C(10)-C(9)-C(8) | 113.45(19) |
| C(10)-C(9)-H(9A) | 108.9 |
| C(8)-C(9)-H(9A) | 108.9 |
| C(10)-C(9)-H(9B) | 108.9 |
| C(8)-C(9)-H(9B) | 108.9 |
| H(9A)-C(9)-H(9B) | 107.7 |
| C(9)-C(10)-C(11) | 117.2(2) |
| C(9)-C(10)-H(10A) | 108.0 |
| C(11)-C(10)-H(10A) | 108.0 |
| C(9)-C(10)-H(10B) | 108.0 |
| C(11)-C(10)-H(10B) | 108.0 |
| H(10A)-C(10)-H(10B) | 107.2 |
| N(1)-C(11)-C(10) | 113.18(18) |
| N(1)-C(11)-H(11A) | 108.9 |
| C(10)-C(11)-H(11A) | 108.9 |
| N(1)-C(11)-H(11B) | 108.9 |
| C(10)-C(11)-H(11B) | 108.9 |
| H(11A)-C(11)-H(11B) | 107.8 |
| N(1)-C(12)-C(7) | 115.98(16) |
| N(1)-C(12)-C(3) | 104.60(16) |
| C(7)-C(12)-C(3) | 115.39(18) |
| N(1)-C(12)-H(12) | 106.8 |
| C(7)-C(12)-H(12) | 106.8 |
| C(3)-C(12)-H(12) | 106.8 |
| O(1)-C(13)-C(1) | 107.44(17) |
| O(1)-C(13)-H(13A) | 110.2 |

| | |
|---|---|
| C(1)-C(13)-H(13A) | 110.2 |
| O(1)-C(13)-H(13B) | 110.2 |
| C(1)-C(13)-H(13B) | 110.2 |
| H(13A)-C(13)-H(13B) | 108.5 |
| O(2)-C(14)-O(1) | 123.1(2) |
| O(2)-C(14)-C(15) | 125.9(2) |
| O(1)-C(14)-C(15) | 110.9(2) |
| C(16)-C(15)-C(20) | 119.4(2) |
| C(16)-C(15)-C(14) | 121.6(2) |
| C(20)-C(15)-C(14) | 119.0(2) |
| C(17)-C(16)-C(15) | 120.4(2) |
| C(17)-C(16)-H(16) | 119.8 |
| C(15)-C(16)-H(16) | 119.8 |
| C(18)-C(17)-C(16) | 119.5(3) |
| C(18)-C(17)-H(17) | 120.3 |
| C(16)-C(17)-H(17) | 120.3 |
| C(17)-C(18)-C(19) | 121.2(3) |
| C(17)-C(18)-Br(1) | 118.6(2) |
| C(19)-C(18)-Br(1) | 120.2(2) |
| C(20)-C(19)-C(18) | 119.2(2) |
| C(20)-C(19)-H(19) | 120.4 |
| C(18)-C(19)-H(19) | 120.4 |
| C(19)-C(20)-C(15) | 120.3(3) |
| C(19)-C(20)-H(20) | 119.8 |
| C(15)-C(20)-H(20) | 119.8 |
| C(22)-C(21)-C(7) | 116.3(2) |
| C(22)-C(21)-H(21A) | 108.2 |
| C(7)-C(21)-H(21A) | 108.2 |
| C(22)-C(21)-H(21B) | 108.2 |
| C(7)-C(21)-H(21B) | 108.2 |
| H(21A)-C(21)-H(21B) | 107.4 |
| C(21)-C(22)-H(22A) | 109.5 |
| C(21)-C(22)-H(22B) | 109.5 |
| H(22A)-C(22)-H(22B) | 109.5 |
| C(21)-C(22)-H(22C) | 109.5 |
| H(22A)-C(22)-H(22C) | 109.5 |
| H(22B)-C(22)-H(22C) | 109.5 |

Table 4. Anisotropic displacement parameters ($Å^2$x $10^3$) for oic66_0m. The anisotropic displacement factor exponent takes the form: $-2\pi^2[ h^2a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12} ]$

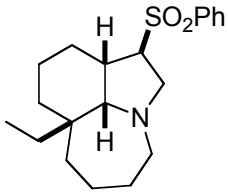
| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{23}$ | $U^{13}$ | $U^{12}$ |
|---|---|---|---|---|---|---|
| Br(1) | 113(1) | 92(1) | 55(1) | 20(1) | 7(1) | -16(1) |
| Cl(1) | 64(1) | 32(1) | 77(1) | -1(1) | 30(1) | -7(1) |
| O(1) | 57(1) | 40(1) | 48(1) | -4(1) | 20(1) | 0(1) |
| O(2) | 133(2) | 54(1) | 70(1) | -11(1) | 38(1) | 20(1) |
| N(1) | 33(1) | 25(1) | 39(1) | 0(1) | 7(1) | -1(1) |
| C(1) | 31(1) | 37(1) | 48(1) | -1(1) | 8(1) | 3(1) |
| C(2) | 48(1) | 45(1) | 56(1) | 7(1) | 10(1) | 13(1) |
| C(3) | 50(1) | 47(1) | 43(1) | 6(1) | 6(1) | 8(1) |
| C(4) | 54(1) | 77(2) | 53(2) | -7(1) | -8(1) | 3(1) |
| C(5) | 75(2) | 78(2) | 52(2) | -19(1) | 1(1) | -13(2) |
| C(6) | 67(2) | 44(1) | 60(2) | -17(1) | 18(1) | -5(1) |
| C(7) | 45(1) | 33(1) | 47(1) | -4(1) | 14(1) | 1(1) |
| C(8) | 50(1) | 36(1) | 66(2) | 5(1) | 21(1) | 8(1) |
| C(9) | 33(1) | 47(1) | 72(2) | 13(1) | 9(1) | 2(1) |
| C(10) | 38(1) | 63(2) | 56(1) | 6(1) | -4(1) | -3(1) |
| C(11) | 39(1) | 47(1) | 42(1) | -4(1) | 3(1) | -6(1) |
| C(12) | 39(1) | 30(1) | 39(1) | 2(1) | 10(1) | -1(1) |
| C(13) | 50(1) | 35(1) | 57(1) | -2(1) | 19(1) | 1(1) |
| C(14) | 50(1) | 53(1) | 47(1) | -13(1) | 11(1) | 0(1) |
| C(15) | 48(1) | 55(1) | 41(1) | -10(1) | 8(1) | -5(1) |
| C(16) | 72(2) | 54(1) | 46(1) | -7(1) | 19(1) | -4(1) |
| C(17) | 78(2) | 54(2) | 53(2) | -5(1) | 13(1) | -7(1) |
| C(18) | 67(2) | 69(2) | 40(1) | 2(1) | 1(1) | -13(1) |
| C(19) | 92(2) | 90(2) | 41(1) | -6(1) | 21(1) | -2(2) |
| C(20) | 80(2) | 68(2) | 49(1) | -12(1) | 18(1) | 3(1) |
| C(21) | 59(1) | 54(1) | 50(1) | 1(1) | 22(1) | 3(1) |
| C(22) | 76(2) | 85(2) | 61(2) | -13(2) | 33(2) | 0(2) |

Table 5. Hydrogen coordinates ( x $10^4$) and isotropic displacement parameters (Å$^2$x $10^3$) for oic66_0m.

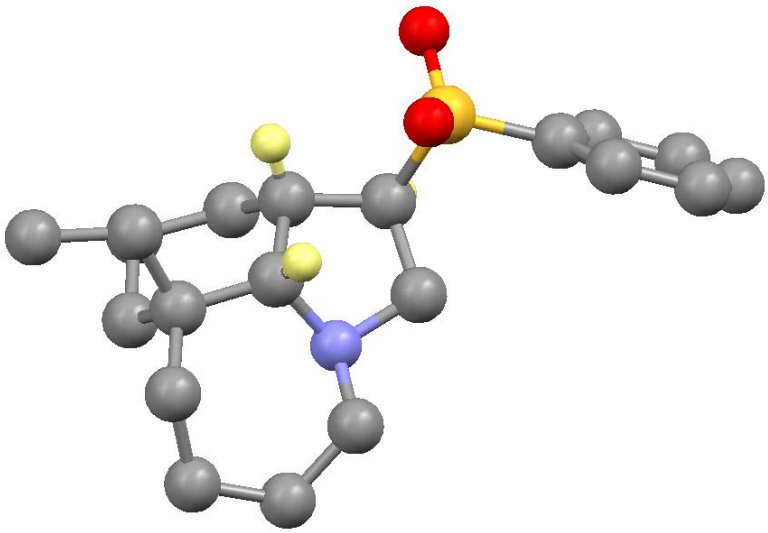
| | x | y | z | U(eq) |
|---|---|---|---|---|
| H(1A) | 5834 | 5733 | 7408 | 39 |
| H(1) | 3329 | 6427 | 7329 | 46 |
| H(2A) | 2819 | 7959 | 6767 | 59 |
| H(2B) | 4570 | 8527 | 6996 | 59 |
| H(3) | 5210 | 7741 | 6140 | 56 |
| H(4A) | 3243 | 5868 | 6432 | 74 |
| H(4B) | 2897 | 6619 | 5875 | 74 |
| H(5A) | 4075 | 4887 | 5630 | 82 |
| H(5B) | 5290 | 5872 | 5482 | 82 |
| H(6A) | 6872 | 4450 | 5989 | 68 |
| H(6B) | 5739 | 4570 | 6511 | 68 |
| H(8A) | 8205 | 4658 | 7106 | 59 |
| H(8B) | 9632 | 4886 | 6692 | 59 |
| H(9A) | 10869 | 5572 | 7507 | 60 |
| H(9B) | 10027 | 6666 | 7238 | 60 |
| H(10A) | 9625 | 6388 | 8240 | 63 |
| H(10B) | 8482 | 5355 | 8057 | 63 |
| H(11A) | 6735 | 6826 | 8239 | 51 |
| H(11B) | 7616 | 7598 | 7805 | 51 |
| H(12) | 7307 | 7416 | 6841 | 43 |
| H(13A) | 4717 | 8076 | 8105 | 56 |
| H(13B) | 2744 | 7994 | 7913 | 56 |
| H(16) | 3901 | 4855 | 8767 | 68 |
| H(17) | 3631 | 3339 | 9375 | 74 |
| H(19) | 1681 | 5334 | 10541 | 88 |
| H(20) | 1965 | 6854 | 9937 | 78 |
| H(21A) | 7691 | 7010 | 5829 | 64 |
| H(21B) | 9392 | 6903 | 6234 | 64 |
| H(22A) | 9975 | 5204 | 5763 | 108 |
| H(22B) | 9858 | 6215 | 5328 | 108 |
| H(22C) | 8319 | 5383 | 5339 | 108 |

## 4. ORTEP diagram and CIF data for the sulfone 12

Compound **12** was prepared as described above.




Ortep diagram for **12**:



Results are deposited at the Cambridge Crystallographic Data Centre.

Structure Number CCDC-782369.

Table 1. Crystal data and structure refinement for **12** (reference number **oic70_0m**).

| | |
|---|---|
| Identification code | oic70_0m |
| Empirical formula | C20 H29 N O2 S |
| Formula weight | 347.50 |
| Temperature | 296(2) K |
| Wavelength | 0.71073 Å |
| Crystal system | Monoclinic |
| Space group | $P2_1/c$ |

| | | |
|---|---|---|
| Unit cell dimensions | a = 13.1393(7) Å | α= 90°. |
| | b = 7.7782(4) Å | β= 102.927(3)°. |
| | c = 17.9896(9) Å | γ = 90°. |
| Volume | 1791.94(16) $Å^3$ | |
| Z | 4 | |
| Density (calculated) | 1.288 $Mg/m^3$ | |
| Absorption coefficient | 0.193 $mm^{-1}$ | |
| F(000) | 752 | |
| Crystal size | 0.38 x 0.11 x 0.04 $mm^3$ | |
| Theta range for data collection | 1.59 to 27.65°. | |
| Index ranges | -17<=h<=17, -10<=k<=10, -23<=l<=23 | |
| Reflections collected | 40116 | |
| Independent reflections | 4150 [R(int) = 0.0738] | |
| Completeness to theta = 25.00° | 100.0 % | |
| Absorption correction | Semi-empirical from equivalents | |
| Max. and min. transmission | 0.9923 and 0.9303 | |
| Refinement method | Full-matrix least-squares on $F^2$ | |
| Data / restraints / parameters | 4150 / 0 / 218 | |
| Goodness-of-fit on $F^2$ | 1.067 | |
| Final R indices [I>2sigma(I)] | R1 = 0.0421, wR2 = 0.0981 | |
| R indices (all data) | R1 = 0.0615, wR2 = 0.1058 | |
| Largest diff. peak and hole | 0.302 and -0.352 e.$Å^{-3}$ | |

Table 2. Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters ($Å^2$x $10^3$) for oic70_0m. U(eq) is defined as one third of the trace of the orthogonalized $U^{ij}$ tensor.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| S(1) | 1085(1) | 9188(1) | 1737(1) | 24(1) |
| N(1) | 2406(1) | 5119(2) | 2680(1) | 22(1) |
| O(1) | 2016(1) | 9391(2) | 1454(1) | 31(1) |
| O(2) | 680(1) | 10674(2) | 2056(1) | 31(1) |
| C(1) | 1504(1) | 5796(2) | 2126(1) | 25(1) |
| C(2) | 1274(1) | 7541(2) | 2443(1) | 22(1) |
| C(3) | 2238(1) | 7923(2) | 3090(1) | 22(1) |
| C(4) | 1985(1) | 7600(2) | 3870(1) | 27(1) |
| C(5) | 2962(1) | 7438(2) | 4508(1) | 30(1) |

| | | | | |
|---|---|---|---|---|
| C(6) | 3683(1) | 6054(2) | 4315(1) | 26(1) |
| C(7) | 4027(1) | 6429(2) | 3571(1) | 22(1) |
| C(8) | 4764(1) | 5026(2) | 3384(1) | 28(1) |
| C(9) | 4490(2) | 3144(2) | 3455(1) | 35(1) |
| C(10) | 3492(1) | 2472(2) | 2921(1) | 29(1) |
| C(11) | 2891(1) | 3723(2) | 2337(1) | 26(1) |
| C(12) | 3056(1) | 6658(2) | 2914(1) | 21(1) |
| C(13) | 96(1) | 8323(2) | 997(1) | 23(1) |
| C(14) | 329(1) | 7924(2) | 303(1) | 32(1) |
| C(15) | -439(2) | 7220(3) | -270(1) | 36(1) |
| C(16) | -1418(2) | 6909(3) | -147(1) | 34(1) |
| C(17) | -1645(1) | 7310(2) | 547(1) | 32(1) |
| C(18) | -891(1) | 8026(2) | 1124(1) | 29(1) |
| C(19) | 4636(1) | 8153(2) | 3622(1) | 26(1) |
| C(20) | 5554(1) | 8373(2) | 4304(1) | 33(1) |

Table 3. Bond lengths [Å] and angles [°] for oic70_0m.

| | |
|---|---|
| S(1)-O(1) | 1.4357(12) |
| S(1)-O(2) | 1.4430(12) |
| S(1)-C(13) | 1.7720(17) |
| S(1)-C(2) | 1.7811(16) |
| N(1)-C(11) | 1.464(2) |
| N(1)-C(1) | 1.465(2) |
| N(1)-C(12) | 1.476(2) |
| C(1)-C(2) | 1.529(2) |
| C(1)-H(1A) | 0.9700 |
| C(1)-H(1B) | 0.9700 |
| C(2)-C(3) | 1.546(2) |
| C(2)-H(2) | 0.9800 |
| C(3)-C(4) | 1.533(2) |
| C(3)-C(12) | 1.541(2) |
| C(3)-H(3) | 0.9800 |
| C(4)-C(5) | 1.525(2) |
| C(4)-H(4A) | 0.9700 |
| C(4)-H(4B) | 0.9700 |

| | |
|---|---|
| C(5)-C(6) | 1.524(2) |
| C(5)-H(5A) | 0.9700 |
| C(5)-H(5B) | 0.9700 |
| C(6)-C(7) | 1.534(2) |
| C(6)-H(6A) | 0.9700 |
| C(6)-H(6B) | 0.9700 |
| C(7)-C(12) | 1.543(2) |
| C(7)-C(8) | 1.544(2) |
| C(7)-C(19) | 1.554(2) |
| C(8)-C(9) | 1.520(3) |
| C(8)-H(8A) | 0.9700 |
| C(8)-H(8B) | 0.9700 |
| C(9)-C(10) | 1.534(2) |
| C(9)-H(9A) | 0.9700 |
| C(9)-H(9B) | 0.9700 |
| C(10)-C(11) | 1.517(2) |
| C(10)-H(10A) | 0.9700 |
| C(10)-H(10B) | 0.9700 |
| C(11)-H(11A) | 0.9700 |
| C(11)-H(11B) | 0.9700 |
| C(12)-H(12) | 0.9800 |
| C(13)-C(18) | 1.386(2) |
| C(13)-C(14) | 1.386(2) |
| C(14)-C(15) | 1.384(3) |
| C(14)-H(14) | 0.9300 |
| C(15)-C(16) | 1.374(3) |
| C(15)-H(15) | 0.9300 |
| C(16)-C(17) | 1.384(2) |
| C(16)-H(16) | 0.9300 |
| C(17)-C(18) | 1.382(2) |
| C(17)-H(17) | 0.9300 |
| C(18)-H(18) | 0.9300 |
| C(19)-C(20) | 1.526(2) |
| C(19)-H(19A) | 0.9700 |
| C(19)-H(19B) | 0.9700 |
| C(20)-H(20A) | 0.9600 |
| C(20)-H(20B) | 0.9600 |
| C(20)-H(20C) | 0.9600 |

| | |
|---|---|
| O(1)-S(1)-O(2) | 118.09(7) |
| O(1)-S(1)-C(13) | 108.37(8) |
| O(2)-S(1)-C(13) | 109.23(7) |
| O(1)-S(1)-C(2) | 109.76(8) |
| O(2)-S(1)-C(2) | 107.35(7) |
| C(13)-S(1)-C(2) | 103.00(8) |
| C(11)-N(1)-C(1) | 109.96(12) |
| C(11)-N(1)-C(12) | 116.32(13) |
| C(1)-N(1)-C(12) | 103.37(12) |
| N(1)-C(1)-C(2) | 105.21(12) |
| N(1)-C(1)-H(1A) | 110.7 |
| C(2)-C(1)-H(1A) | 110.7 |
| N(1)-C(1)-H(1B) | 110.7 |
| C(2)-C(1)-H(1B) | 110.7 |
| H(1A)-C(1)-H(1B) | 108.8 |
| C(1)-C(2)-C(3) | 104.70(13) |
| C(1)-C(2)-S(1) | 112.33(11) |
| C(3)-C(2)-S(1) | 111.04(11) |
| C(1)-C(2)-H(2) | 109.6 |
| C(3)-C(2)-H(2) | 109.6 |
| S(1)-C(2)-H(2) | 109.6 |
| C(4)-C(3)-C(12) | 112.83(13) |
| C(4)-C(3)-C(2) | 110.35(13) |
| C(12)-C(3)-C(2) | 102.39(12) |
| C(4)-C(3)-H(3) | 110.3 |
| C(12)-C(3)-H(3) | 110.3 |
| C(2)-C(3)-H(3) | 110.3 |
| C(5)-C(4)-C(3) | 112.56(14) |
| C(5)-C(4)-H(4A) | 109.1 |
| C(3)-C(4)-H(4A) | 109.1 |
| C(5)-C(4)-H(4B) | 109.1 |
| C(3)-C(4)-H(4B) | 109.1 |
| H(4A)-C(4)-H(4B) | 107.8 |
| C(6)-C(5)-C(4) | 110.39(13) |
| C(6)-C(5)-H(5A) | 109.6 |
| C(4)-C(5)-H(5A) | 109.6 |
| C(6)-C(5)-H(5B) | 109.6 |

| | |
|---|---|
| C(4)-C(5)-H(5B) | 109.6 |
| H(5A)-C(5)-H(5B) | 108.1 |
| C(5)-C(6)-C(7) | 112.61(14) |
| C(5)-C(6)-H(6A) | 109.1 |
| C(7)-C(6)-H(6A) | 109.1 |
| C(5)-C(6)-H(6B) | 109.1 |
| C(7)-C(6)-H(6B) | 109.1 |
| H(6A)-C(6)-H(6B) | 107.8 |
| C(6)-C(7)-C(12) | 109.60(13) |
| C(6)-C(7)-C(8) | 112.17(14) |
| C(12)-C(7)-C(8) | 111.47(13) |
| C(6)-C(7)-C(19) | 111.18(13) |
| C(12)-C(7)-C(19) | 105.79(13) |
| C(8)-C(7)-C(19) | 106.42(13) |
| C(9)-C(8)-C(7) | 119.39(15) |
| C(9)-C(8)-H(8A) | 107.5 |
| C(7)-C(8)-H(8A) | 107.5 |
| C(9)-C(8)-H(8B) | 107.5 |
| C(7)-C(8)-H(8B) | 107.5 |
| H(8A)-C(8)-H(8B) | 107.0 |
| C(8)-C(9)-C(10) | 117.47(14) |
| C(8)-C(9)-H(9A) | 107.9 |
| C(10)-C(9)-H(9A) | 107.9 |
| C(8)-C(9)-H(9B) | 107.9 |
| C(10)-C(9)-H(9B) | 107.9 |
| H(9A)-C(9)-H(9B) | 107.2 |
| C(11)-C(10)-C(9) | 117.04(15) |
| C(11)-C(10)-H(10A) | 108.0 |
| C(9)-C(10)-H(10A) | 108.0 |
| C(11)-C(10)-H(10B) | 108.0 |
| C(9)-C(10)-H(10B) | 108.0 |
| H(10A)-C(10)-H(10B) | 107.3 |
| N(1)-C(11)-C(10) | 113.09(13) |
| N(1)-C(11)-H(11A) | 109.0 |
| C(10)-C(11)-H(11A) | 109.0 |
| N(1)-C(11)-H(11B) | 109.0 |
| C(10)-C(11)-H(11B) | 109.0 |
| H(11A)-C(11)-H(11B) | 107.8 |

| | |
|---|---|
| N(1)-C(12)-C(3) | 101.00(12) |
| N(1)-C(12)-C(7) | 116.83(13) |
| C(3)-C(12)-C(7) | 114.29(13) |
| N(1)-C(12)-H(12) | 108.1 |
| C(3)-C(12)-H(12) | 108.1 |
| C(7)-C(12)-H(12) | 108.1 |
| C(18)-C(13)-C(14) | 121.02(16) |
| C(18)-C(13)-S(1) | 119.66(12) |
| C(14)-C(13)-S(1) | 119.31(13) |
| C(15)-C(14)-C(13) | 119.22(17) |
| C(15)-C(14)-H(14) | 120.4 |
| C(13)-C(14)-H(14) | 120.4 |
| C(16)-C(15)-C(14) | 120.19(16) |
| C(16)-C(15)-H(15) | 119.9 |
| C(14)-C(15)-H(15) | 119.9 |
| C(15)-C(16)-C(17) | 120.28(17) |
| C(15)-C(16)-H(16) | 119.9 |
| C(17)-C(16)-H(16) | 119.9 |
| C(18)-C(17)-C(16) | 120.39(17) |
| C(18)-C(17)-H(17) | 119.8 |
| C(16)-C(17)-H(17) | 119.8 |
| C(17)-C(18)-C(13) | 118.90(16) |
| C(17)-C(18)-H(18) | 120.5 |
| C(13)-C(18)-H(18) | 120.5 |
| C(20)-C(19)-C(7) | 116.60(14) |
| C(20)-C(19)-H(19A) | 108.1 |
| C(7)-C(19)-H(19A) | 108.1 |
| C(20)-C(19)-H(19B) | 108.1 |
| C(7)-C(19)-H(19B) | 108.1 |
| H(19A)-C(19)-H(19B) | 107.3 |
| C(19)-C(20)-H(20A) | 109.5 |
| C(19)-C(20)-H(20B) | 109.5 |
| H(20A)-C(20)-H(20B) | 109.5 |
| C(19)-C(20)-H(20C) | 109.5 |
| H(20A)-C(20)-H(20C) | 109.5 |
| H(20B)-C(20)-H(20C) | 109.5 |

Table 4. Anisotropic displacement parameters ($Å^2$x $10^3$) for oic70_0m. The anisotropic displacement factor exponent takes the form: $-2\pi^2[ h^2a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12} ]$

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{23}$ | $U^{13}$ | $U^{12}$ |
|---|---|---|---|---|---|---|
| S(1) | 22(1) | 23(1) | 25(1) | 2(1) | 2(1) | 1(1) |
| N(1) | 21(1) | 19(1) | 23(1) | 0(1) | 1(1) | -1(1) |
| O(1) | 24(1) | 36(1) | 32(1) | 5(1) | 6(1) | -2(1) |
| O(2) | 32(1) | 25(1) | 34(1) | -2(1) | 3(1) | 5(1) |
| C(1) | 26(1) | 22(1) | 25(1) | 0(1) | 0(1) | -1(1) |
| C(2) | 20(1) | 24(1) | 22(1) | 2(1) | 3(1) | -2(1) |
| C(3) | 21(1) | 21(1) | 23(1) | 0(1) | 2(1) | -1(1) |
| C(4) | 25(1) | 32(1) | 24(1) | -5(1) | 5(1) | -1(1) |
| C(5) | 29(1) | 40(1) | 20(1) | -3(1) | 6(1) | -3(1) |
| C(6) | 25(1) | 32(1) | 21(1) | 5(1) | 2(1) | -2(1) |
| C(7) | 20(1) | 24(1) | 22(1) | 2(1) | 3(1) | -1(1) |
| C(8) | 22(1) | 30(1) | 31(1) | 0(1) | 0(1) | 2(1) |
| C(9) | 41(1) | 27(1) | 32(1) | 2(1) | -1(1) | 9(1) |
| C(10) | 34(1) | 22(1) | 33(1) | 2(1) | 9(1) | 4(1) |
| C(11) | 29(1) | 25(1) | 24(1) | -3(1) | 6(1) | 1(1) |
| C(12) | 22(1) | 20(1) | 21(1) | 0(1) | 5(1) | -2(1) |
| C(13) | 24(1) | 22(1) | 23(1) | 3(1) | 2(1) | 4(1) |
| C(14) | 27(1) | 41(1) | 28(1) | 1(1) | 7(1) | 2(1) |
| C(15) | 36(1) | 49(1) | 23(1) | -4(1) | 6(1) | 2(1) |
| C(16) | 31(1) | 41(1) | 27(1) | -4(1) | -4(1) | 2(1) |
| C(17) | 21(1) | 40(1) | 34(1) | -2(1) | 4(1) | 0(1) |
| C(18) | 26(1) | 36(1) | 24(1) | -3(1) | 5(1) | 3(1) |
| C(19) | 22(1) | 27(1) | 26(1) | 1(1) | 3(1) | -5(1) |
| C(20) | 29(1) | 34(1) | 32(1) | 0(1) | -1(1) | -7(1) |

Table 5. Hydrogen coordinates ( x $10^4$) and isotropic displacement parameters ($Å^2$x $10^3$) for oic70_0m.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| H(1A) | 909 | 5032 | 2078 | 30 |
| H(1B) | 1667 | 5927 | 1629 | 30 |
| H(2) | 651 | 7451 | 2656 | 27 |
| H(3) | 2471 | 9111 | 3055 | 26 |
| H(4A) | 1561 | 8540 | 3986 | 32 |
| H(4B) | 1578 | 6552 | 3845 | 32 |
| H(5A) | 3327 | 8531 | 4579 | 36 |
| H(5B) | 2765 | 7145 | 4981 | 36 |
| H(6A) | 3325 | 4955 | 4270 | 32 |
| H(6B) | 4297 | 5967 | 4729 | 32 |
| H(8A) | 4832 | 5214 | 2864 | 34 |
| H(8B) | 5448 | 5216 | 3713 | 34 |
| H(9A) | 4432 | 2942 | 3975 | 42 |
| H(9B) | 5071 | 2458 | 3371 | 42 |
| H(10A) | 3677 | 1484 | 2649 | 35 |
| H(10B) | 3026 | 2069 | 3233 | 35 |
| H(11A) | 3363 | 4211 | 2049 | 31 |
| H(11B) | 2351 | 3100 | 1983 | 31 |
| H(12) | 3289 | 7088 | 2468 | 25 |
| H(14) | 994 | 8126 | 223 | 38 |
| H(15) | -293 | 6957 | -740 | 43 |
| H(16) | -1930 | 6427 | -533 | 41 |
| H(17) | -2310 | 7098 | 626 | 38 |
| H(18) | -1043 | 8303 | 1591 | 34 |
| H(19A) | 4896 | 8278 | 3160 | 31 |
| H(19B) | 4148 | 9084 | 3629 | 31 |
| H(20A) | 5300 | 8411 | 4765 | 49 |
| H(20B) | 5914 | 9424 | 4251 | 49 |
| H(20C) | 6025 | 7421 | 4326 | 49 |