

## Supporting information available

### Aminolysis of iridium thiophene compounds

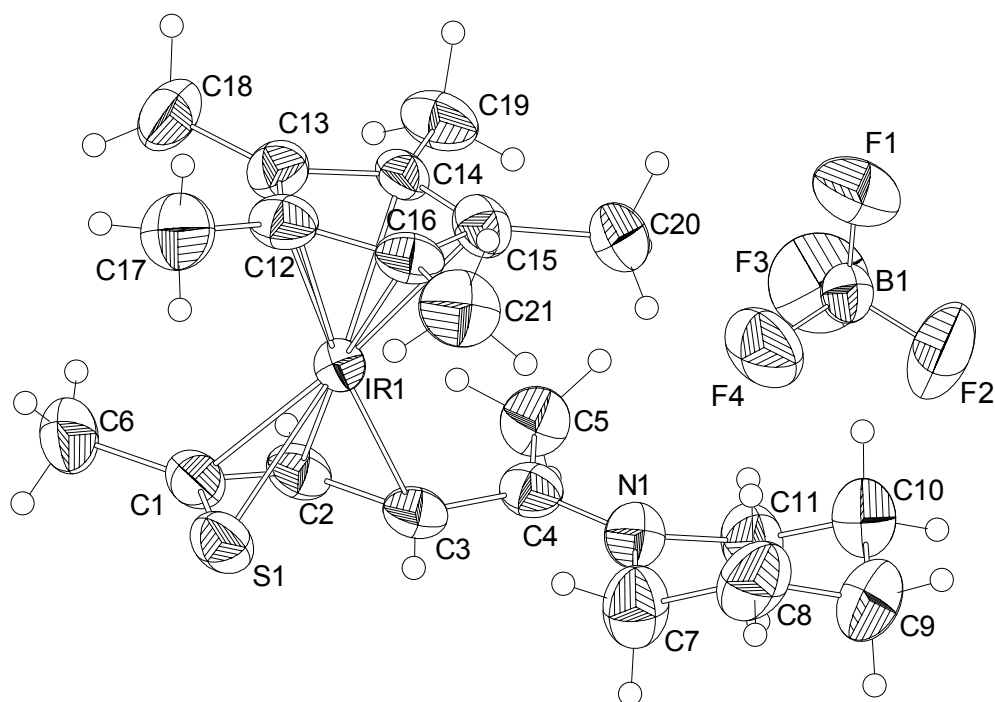
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**Dedicated to Professor Rosalinda Contreras, friend and colleague, on the occasion of her 60<sup>th</sup> birthday in recognition of her outstanding contribution to the main group chemistry**

#### Supporting information for crystal structures of compounds:

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| Atomic coordinates and equivalent isotropic displacement parameters | 1  | 1  | 1  |
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| Selected torsion angles   | 5  | 5  | 5  |
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DEPARTAMENTO DE QUIMICA  
CinvestavORTEP drawing of Compound **12**Formula:  $C_{21}H_{33}BF_4IrNS$ 

Authors : Marisol Cervantes Vásquez y Ma. de los Ángeles Paz Sandoval.

Tab. 1 Crystal data and data collection

Tab. 2 Solution and refinement

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**Table 1.** Crystal data and data collection

|                              |   |               |
|------------------------------|---|---------------|
| Identification code          | Compound <b>12</b>                                      |               |
| Operator                     | MLR and BPM   |               |
| Empirical formula            | C <sub>21</sub> H <sub>33</sub> B F <sub>4</sub> Ir N S |               |
| Formula weight               | 610.55  |               |
| Crystal size                 | 0.56 x 0.45 x 0.33 mm                                   |               |
| Crystal color and habit      | red prism   |               |
| Crystal system               | Monoclinic  |               |
| Space group                  | P2/c  |               |
| Unit cell dimensions         | a = 13.450(3) Å   | α = 90.00(2)° |
|                              | b = 7.877(3) Å  | β = 91.34(2)° |
|                              | c = 21.840(3) Å   | γ = 90.00(2)° |
| Volume                       | 2313.2(11) Å <sup>3</sup>                               |               |
| Z                            | 4   |               |
| Density (calculated)         | 1.753 Mg/m <sup>3</sup>                                 |               |
| Absorption coefficient       | 5.901 mm <sup>-1</sup>                                  |               |
| F(000)                       | 1200  |               |
| Diffractometer used          | Enraf-Nonius CAD4                                       |               |
| Radiation and wavelength     | MoKα with λ=0.71073 Å                                   |               |
| Scan type                    | ω/2θ  |               |
| Temperature                  | 270(2) K  |               |
| 2θ range for data collection | 5.18 to 51.92°  |               |
| Index ranges                 | -16 ≤ h ≤ 16   0 ≤ k ≤ 9   0 ≤ l ≤ 26                   |               |
| Reflections collected        | 4527  |               |
| Independent reflections      | 4527 (R <sub>int</sub> = 0.0000)                        |               |
| Observed reflections         | 3635 (F > 4σ(F))  |               |
| Absorption correction        | Semi-empirical  |               |
| Max. and min. transmission   | 0.2462 and 0.1367                                       |               |

**Experimental details :**

|   |                                 |
|---|---------------------------------|
| Scan speed  | Variable; 16.1 to 60 °/min in ω |
| Scan range (ω)  | 0.7 °                           |
| Background measurement : Moving crystal and moving counter at the beginning and end of scan, each for 25% of total scan area. |                                 |
| Crystal mounted in perfluoropolyether oil   |                                 |

**Table 2.** Solution and refinement

|                                   |   |
|-----------------------------------|---|
| Structure solution program        | SHELXS-97(Sheldrick 1990)   |
| Solution                          | heavy-atom-method   |
| Refinement method                 | Full-matrix Least-Squares on F <sup>2</sup>                               |
| Hydrogen atoms                    | mixed   |
| Weighting scheme                  | $w^{-1} = \sigma^2 F_o^2 + (P)^2 + P$<br>where $P = (F_o^2 + 2F_c^2) / 3$ |
| Data / restraints / parameters    | 4527 / 0 / 277  |
| Data-to-parameter-ratio           | 16.3 : 1 (13.1 : 1 [F > 4σ(F)])   |
| Final R indices [F > 4σ(F)]       | R1 = 0.0282, wR2 = 0.0801   |
| R indices (all data)              | R1 = 0.0416, wR2 = 0.0846   |
| Goodness-of-Fit on F <sup>2</sup> | 1.081   |
| Largest and mean Δ/σ              | 0.012 0.002   |
| Largest difference peak           | 0.888 eÅ <sup>-3</sup>  |
| Largest difference hole           | -1.013 eÅ <sup>-3</sup>   |

**Refinement details :**

|                     |                            |
|---------------------|----------------------------|
| Program used        | SHELXL-97 (Sheldrick 1997) |
| CifRtf version used | 2.0                        |

**Table 3.** Atomic coordinates (  $\times 10^4$  ) and equivalent isotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ ) for compound **12**. U(eq) is defined as one third of the trace of the orthogonalized  $U_{ij}$  tensor

|       | x          | y          | z          | U(eq)     |
|-------|------------|------------|------------|-----------|
| C(1)  | 1666(4)    | -174(6)    | 3618(2)    | 46.6(12)  |
| C(2)  | 1364(3)    | 385(6)     | 4202(2)    | 43.1(11)  |
| C(3)  | 2099(4)    | 461(6)     | 4681(2)    | 42.6(11)  |
| C(4)  | 1956(3)    | 1284(6)    | 5260(2)    | 41.8(10)  |
| C(5)  | 991(4)     | 2112(7)    | 5393(3)    | 55.7(14)  |
| C(6)  | 956(4)     | -259(8)    | 3079(3)    | 64.5(15)  |
| C(7)  | 3627(4)    | 398(9)     | 5586(3)    | 65.9(16)  |
| C(8)  | 4484(4)    | 1350(10)   | 5912(3)    | 72.7(17)  |
| C(9)  | 4285(6)    | 1628(10)   | 6584(3)    | 85(2)     |
| C(10) | 3334(6)    | 2589(9)    | 6638(4)    | 75.8(19)  |
| C(11) | 2462(5)    | 1696(8)    | 6342(3)    | 63.1(16)  |
| C(12) | 3108(4)    | 3675(6)    | 3197(2)    | 47.4(12)  |
| C(13) | 2124(4)    | 4241(6)    | 3319(2)    | 46.4(11)  |
| C(14) | 2117(3)    | 4763(5)    | 3957(2)    | 36.9(10)  |
| C(15) | 3086(4)    | 4547(6)    | 4211(2)    | 45.0(11)  |
| C(16) | 3699(3)    | 3844(5)    | 3751(2)    | 43.8(11)  |
| C(17) | 3468(5)    | 3083(7)    | 2590(3)    | 69.3(18)  |
| C(18) | 1271(5)    | 4378(8)    | 2885(3)    | 68.6(16)  |
| C(19) | 1240(4)    | 5565(6)    | 4264(3)    | 57.4(14)  |
| C(20) | 3412(4)    | 5074(7)    | 4843(3)    | 59.5(14)  |
| C(21) | 4797(4)    | 3516(8)    | 3820(3)    | 68.1(16)  |
| B(1)  | 8540(6)    | 2888(8)    | 3870(3)    | 53.2(15)  |
| F(1)  | 8833(4)    | 4474(5)    | 3712(2)    | 112.9(16) |
| F(2)  | 8994(5)    | 2349(7)    | 4390(2)    | 126(2)    |
| F(3)  | 7528(3)    | 2908(9)    | 3944(2)    | 127(3)    |
| F(4)  | 8723(4)    | 1791(6)    | 3412(2)    | 105.0(16) |
| Ir(1) | 2515.8(1)  | 2043.3(2)  | 3907.7(1)  | 33.9(1)   |
| N(1)  | 2652(3)    | 1239(6)    | 5691.3(19) | 48.8(10)  |
| S(1)  | 2913.2(10) | -765.5(16) | 3555.4(6)  | 50.2(3)   |

**Table 4.** Bond lengths [Å] and angles [°] for compound **12**

|                   |          |                   |            |
|-------------------|----------|-------------------|------------|
| C(1)-C(2)         | 1.417(7) | C(1)-C(6)         | 1.500(7)   |
| C(1)-S(1)         | 1.750(5) | C(1)-Ir(1)        | 2.173(5)   |
| C(2)-C(3)         | 1.423(7) | C(2)-Ir(1)        | 2.137(4)   |
| C(2)-H(2)         | 0.95(4)  | C(3)-C(4)         | 1.438(7)   |
| C(3)-Ir(1)        | 2.182(5) | C(3)-H(3)         | 0.67(4)    |
| C(4)-N(1)         | 1.313(6) | C(4)-C(5)         | 1.488(7)   |
| C(7)-N(1)         | 1.491(6) | C(7)-C(8)         | 1.537(8)   |
| C(8)-C(9)         | 1.514(9) | C(9)-C(10)        | 1.493(11)  |
| C(10)-C(11)       | 1.501(9) | C(11)-N(1)        | 1.494(7)   |
| C(12)-C(13)       | 1.428(7) | C(12)-C(16)       | 1.439(7)   |
| C(12)-C(17)       | 1.497(7) | C(12)-Ir(1)       | 2.179(5)   |
| C(13)-C(14)       | 1.452(7) | C(13)-C(18)       | 1.476(7)   |
| C(13)-Ir(1)       | 2.212(5) | C(14)-C(15)       | 1.415(7)   |
| C(14)-C(19)       | 1.509(6) | C(14)-Ir(1)       | 2.212(4)   |
| C(15)-C(16)       | 1.426(6) | C(15)-C(20)       | 1.499(7)   |
| C(15)-Ir(1)       | 2.211(5) | C(16)-C(21)       | 1.503(7)   |
| C(16)-Ir(1)       | 2.165(4) | Ir(1)-S(1)        | 2.4066(15) |
| B(1)-F(2)         | 1.345(8) | B(1)-F(4)         | 1.349(8)   |
| B(1)-F(1)         | 1.358(8) | B(1)-F(3)         | 1.374(8)   |
| C(2)-C(1)-C(6)    | 122.0(5) | C(2)-C(1)-S(1)    | 116.7(4)   |
| C(6)-C(1)-S(1)    | 121.3(4) | C(2)-C(1)-Ir(1)   | 69.4(3)    |
| C(6)-C(1)-Ir(1)   | 125.8(4) | S(1)-C(1)-Ir(1)   | 74.80(18)  |
| C(1)-C(2)-C(3)    | 117.7(4) | C(1)-C(2)-Ir(1)   | 72.2(3)    |
| C(3)-C(2)-Ir(1)   | 72.5(3)  | C(1)-C(2)-H(2)    | 117(3)     |
| C(3)-C(2)-H(2)    | 125(3)   | Ir(1)-C(2)-H(2)   | 116(3)     |
| C(2)-C(3)-C(4)    | 124.0(5) | C(2)-C(3)-Ir(1)   | 69.1(3)    |
| C(4)-C(3)-Ir(1)   | 117.7(3) | C(2)-C(3)-H(3)    | 115(4)     |
| C(4)-C(3)-H(3)    | 111(4)   | Ir(1)-C(3)-H(3)   | 115(4)     |
| N(1)-C(4)-C(3)    | 120.8(4) | N(1)-C(4)-C(5)    | 118.8(5)   |
| C(3)-C(4)-C(5)    | 120.3(5) | C(4)-C(5)-H(5A)   | 109.5      |
| N(1)-C(7)-C(8)    | 111.3(5) | C(9)-C(8)-C(7)    | 111.8(6)   |
| C(10)-C(9)-C(8)   | 108.8(6) | C(11)-C(10)-C(9)  | 113.0(6)   |
| N(1)-C(11)-C(10)  | 111.9(6) | C(13)-C(12)-C(16) | 108.1(4)   |
| C(13)-C(12)-C(17) | 125.7(5) | C(16)-C(12)-C(17) | 126.1(5)   |
| C(13)-C(12)-Ir(1) | 72.3(3)  | C(16)-C(12)-Ir(1) | 70.1(3)    |
| C(17)-C(12)-Ir(1) | 125.3(4) | C(12)-C(13)-C(14) | 107.1(4)   |
| C(12)-C(13)-C(18) | 127.8(5) | C(14)-C(13)-C(18) | 125.0(5)   |
| C(12)-C(13)-Ir(1) | 69.8(3)  | C(14)-C(13)-Ir(1) | 70.8(3)    |
| C(18)-C(13)-Ir(1) | 127.0(4) | C(15)-C(14)-C(13) | 108.4(4)   |

|                   |            |                   |            |
|-------------------|------------|-------------------|------------|
| C(15)-C(14)-C(19) | 126.8(4)   | C(13)-C(14)-C(19) | 124.6(5)   |
| C(15)-C(14)-Ir(1) | 71.3(3)    | C(13)-C(14)-Ir(1) | 70.9(3)    |
| C(19)-C(14)-Ir(1) | 128.4(3)   | C(14)-C(15)-C(16) | 108.3(4)   |
| C(14)-C(15)-C(20) | 125.1(5)   | C(16)-C(15)-C(20) | 126.5(5)   |
| C(14)-C(15)-Ir(1) | 71.4(2)    | C(16)-C(15)-Ir(1) | 69.2(3)    |
| C(20)-C(15)-Ir(1) | 128.0(4)   | C(15)-C(16)-C(12) | 108.0(4)   |
| C(15)-C(16)-C(21) | 125.4(5)   | C(12)-C(16)-C(21) | 126.2(5)   |
| C(15)-C(16)-Ir(1) | 72.8(3)    | C(12)-C(16)-Ir(1) | 71.2(3)    |
| C(21)-C(16)-Ir(1) | 126.6(3)   | F(2)-B(1)-F(4)    | 109.7(6)   |
| F(2)-B(1)-F(1)    | 112.1(6)   | F(4)-B(1)-F(1)    | 110.0(6)   |
| F(2)-B(1)-F(3)    | 109.7(6)   | F(4)-B(1)-F(3)    | 107.0(6)   |
| F(1)-B(1)-F(3)    | 108.3(6)   | C(2)-Ir(1)-C(16)  | 171.38(19) |
| C(2)-Ir(1)-C(1)   | 38.39(19)  | C(16)-Ir(1)-C(1)  | 149.83(19) |
| C(2)-Ir(1)-C(12)  | 148.7(2)   | C(16)-Ir(1)-C(12) | 38.69(19)  |
| C(1)-Ir(1)-C(12)  | 117.7(2)   | C(2)-Ir(1)-C(3)   | 38.44(19)  |
| C(16)-Ir(1)-C(3)  | 134.37(19) | C(1)-Ir(1)-C(3)   | 67.84(19)  |
| C(12)-Ir(1)-C(3)  | 172.8(2)   | C(2)-Ir(1)-C(13)  | 119.23(19) |
| C(16)-Ir(1)-C(13) | 64.03(18)  | C(1)-Ir(1)-C(13)  | 110.1(2)   |
| C(12)-Ir(1)-C(13) | 37.93(18)  | C(3)-Ir(1)-C(13)  | 146.53(19) |
| C(2)-Ir(1)-C(14)  | 113.50(17) | C(16)-Ir(1)-C(14) | 63.47(17)  |
| C(1)-Ir(1)-C(14)  | 131.74(17) | C(12)-Ir(1)-C(14) | 63.68(17)  |
| C(3)-Ir(1)-C(14)  | 116.66(18) | C(13)-Ir(1)-C(14) | 38.32(18)  |
| C(2)-Ir(1)-C(15)  | 134.70(18) | C(16)-Ir(1)-C(15) | 38.00(17)  |
| C(1)-Ir(1)-C(15)  | 168.50(18) | C(12)-Ir(1)-C(15) | 63.73(19)  |
| C(3)-Ir(1)-C(15)  | 111.80(19) | C(13)-Ir(1)-C(15) | 63.4(2)    |
| C(14)-Ir(1)-C(15) | 37.31(18)  | C(2)-Ir(1)-S(1)   | 72.69(14)  |
| C(16)-Ir(1)-S(1)  | 112.48(13) | C(1)-Ir(1)-S(1)   | 44.57(13)  |
| C(12)-Ir(1)-S(1)  | 103.13(13) | C(3)-Ir(1)-S(1)   | 77.59(15)  |
| C(13)-Ir(1)-S(1)  | 125.84(14) | C(14)-Ir(1)-S(1)  | 164.15(13) |
| C(15)-Ir(1)-S(1)  | 146.87(13) | C(4)-N(1)-C(7)    | 121.0(4)   |
| C(4)-N(1)-C(11)   | 122.8(5)   | C(7)-N(1)-C(11)   | 115.2(5)   |
| C(1)-S(1)-Ir(1)   | 60.63(16)  |                   |            |

**Table 5.** Anisotropic displacement parameters [ $\text{\AA}^2 \times 10^3$ ]

|       | U11     | U22      | U33     | U23      | U13     | U12      |
|-------|---------|----------|---------|----------|---------|----------|
| C(1)  | 48(3)   | 37(2)    | 54(3)   | -6(2)    | -5(2)   | 0(2)     |
| C(2)  | 37(2)   | 34(2)    | 58(3)   | 0(2)     | 3(2)    | -8.6(19) |
| C(3)  | 44(3)   | 31(2)    | 53(3)   | 4(2)     | 4(2)    | -4(2)    |
| C(4)  | 44(2)   | 37(2)    | 45(3)   | 4(2)     | 9(2)    | -3(2)    |
| C(5)  | 47(3)   | 62(3)    | 58(4)   | -1(3)    | 4(3)    | 5(2)     |
| C(6)  | 66(4)   | 65(3)    | 62(4)   | -12(3)   | -18(3)  | -5(3)    |
| C(7)  | 50(3)   | 83(4)    | 64(4)   | -13(3)   | -7(3)   | 8(3)     |
| C(8)  | 55(3)   | 90(4)    | 72(4)   | 5(4)     | -16(3)  | 13(3)    |
| C(9)  | 95(5)   | 92(5)    | 67(5)   | 0(4)     | -34(4)  | 7(4)     |
| C(10) | 92(6)   | 73(4)    | 62(4)   | -8(4)    | -17(4)  | -6(4)    |
| C(11) | 79(4)   | 64(3)    | 47(3)   | -8(3)    | 5(3)    | -10(3)   |
| C(12) | 53(3)   | 38(2)    | 51(3)   | 8(2)     | 11(2)   | -3(2)    |
| C(13) | 50(3)   | 46(3)    | 43(3)   | 6(2)     | 1(2)    | -1(2)    |
| C(14) | 49(2)   | 26.5(19) | 36(2)   | -1.2(18) | 5(2)    | -7.9(19) |
| C(15) | 46(3)   | 42(2)    | 47(3)   | -3(2)    | 5(2)    | -13(2)   |
| C(16) | 43(2)   | 31(2)    | 58(3)   | 7(2)     | 7(2)    | -5(2)    |
| C(17) | 86(5)   | 75(4)    | 47(4)   | -2(3)    | 20(3)   | 10(3)    |
| C(18) | 80(4)   | 76(4)    | 49(3)   | 4(3)     | -20(3)  | 18(3)    |
| C(19) | 64(3)   | 40(3)    | 69(4)   | -5(3)    | 17(3)   | 11(2)    |
| C(20) | 68(3)   | 50(3)    | 60(3)   | -8(3)    | -11(3)  | -13(3)   |
| C(21) | 41(3)   | 70(3)    | 92(5)   | 3(4)     | 1(3)    | -11(3)   |
| B(1)  | 61(4)   | 57(4)    | 42(3)   | -4(3)    | 0(3)    | 1(3)     |
| F(1)  | 142(4)  | 67(2)    | 129(4)  | -6(3)    | -2(3)   | -19(3)   |
| F(2)  | 145(5)  | 163(5)   | 69(3)   | 17(3)    | -31(3)  | 13(4)    |
| F(3)  | 73(4)   | 159(8)   | 151(7)  | -5(3)    | 30(4)   | 7(3)     |
| F(4)  | 149(5)  | 89(3)    | 78(3)   | -25(2)   | 30(3)   | -2(3)    |
| Ir(1) | 33.8(1) | 34.0(1)  | 33.9(1) | -1.5(1)  | 0.9(1)  | -1.3(1)  |
| N(1)  | 48(2)   | 53(2)    | 46(2)   | -1(2)    | 1.3(18) | -2(2)    |
| S(1)  | 52.8(7) | 41.3(6)  | 56.4(8) | -10.6(6) | 2.0(6)  | 6.5(5)   |



**Table 6.** Hydrogen coordinates (  $\times 10^4$  ) and isotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ ) for compound **12**

|        | x        | y        | z        | U(eq)  |
|--------|----------|----------|----------|--------|
| H(5A)  | 628      | 1417     | 5671     | 84     |
| H(5B)  | 610      | 2248     | 5019     | 84     |
| H(5C)  | 1112     | 3205     | 5574     | 84     |
| H(6A)  | 671      | -1374    | 3053     | 97     |
| H(6B)  | 1307     | -19      | 2711     | 97     |
| H(6C)  | 437      | 563      | 3128     | 97     |
| H(7A)  | 3745     | 359      | 5149     | 79     |
| H(7B)  | 3605     | -761     | 5736     | 79     |
| H(8A)  | 5093     | 705      | 5873     | 87     |
| H(8B)  | 4577     | 2441     | 5715     | 87     |
| H(9A)  | 4236     | 543      | 6791     | 102    |
| H(9B)  | 4827     | 2264     | 6773     | 102    |
| H(10A) | 3202     | 2763     | 7068     | 91     |
| H(10B) | 3408     | 3696     | 6450     | 91     |
| H(11A) | 2320     | 672      | 6571     | 76     |
| H(11B) | 1882     | 2426     | 6356     | 76     |
| H(17A) | 2909     | 2777     | 2332     | 104    |
| H(17B) | 3892     | 2114     | 2649     | 104    |
| H(17C) | 3833     | 3979     | 2400     | 104    |
| H(18A) | 1212     | 5528     | 2743     | 103    |
| H(18B) | 672      | 4056     | 3086     | 103    |
| H(18C) | 1373     | 3639     | 2542     | 103    |
| H(19A) | 1303     | 5407     | 4699     | 86     |
| H(19B) | 636      | 5042     | 4115     | 86     |
| H(19C) | 1222     | 6757     | 4173     | 86     |
| H(20A) | 3659     | 6217     | 4833     | 89     |
| H(20B) | 3929     | 4328     | 4991     | 89     |
| H(20C) | 2857     | 5015     | 5111     | 89     |
| H(21A) | 5155     | 4548     | 3748     | 102    |
| H(21B) | 4990     | 2672     | 3529     | 102    |
| H(21C) | 4947     | 3118     | 4228     | 102    |
| H(2)   | 720(03)  | 880(06)  | 4220(02) | 40(12) |
| H(3)   | 2370(03) | -240(06) | 4720(02) | 28(14) |

**Table 7.** Crystal data and data collection parameters

| Compound                            | Compound <b>12</b>                                      |
|-------------------------------------|---|
| Chem. formula                       | C <sub>21</sub> H <sub>33</sub> B F <sub>4</sub> Ir N S |
| Form. wght.                         | 610.55  |
| Cryst. size [mm]                    | 0.33x0.45x0.56  |
| Cryst. system                       | Monoclinic  |
| Space group                         | P2/c  |
| a, [Å]                              | 13.450(3)   |
| b, [Å]                              | 7.877(3)  |
| c, [Å]                              | 21.840(3)   |
| α, [°]                              | 90.00(2)  |
| β, [°]                              | 91.34(2)  |
| γ, [°]                              | 90.00(2)  |
| V, [Å <sup>3</sup> ]                | 2313.2(11)  |
| Z                                   | 4   |
| ρ(calcd.), [Mg/m <sup>3</sup> ]     | 1.753   |
| μ [mm <sup>-1</sup> ]               | 5.901   |
| F(000)                              | 1200  |
| Index range                         | -16≤h≤160≤k≤90≤l≤26                                     |
| 2 θ [°]                             | 51.92   |
| Temp, [K]                           | 270(2)  |
| Refl. collected                     | 4527  |
| Refl. unique                        | 4527  |
| Refl. observed (4σ)                 | 3635  |
| R (int.)                            | 0.0000  |
| No. variables                       | 277   |
| Weighting scheme <sup>1</sup> x/y   | /   |
| GOOF                                | 1.081   |
| Final R (4σ)                        | 0.0282  |
| Final wR2                           | 0.0801  |
| Larg. res. peak [e/Å <sup>3</sup> ] | 0.888   |

$$^1 w^{-1} = \sigma^2 F_o^2 + (xP)^2 + yP; P = (F_o^2 + 2F_c^2)/3$$

**Selected torsion angles for compound 12**

-178.41 ( 0.46) C6 - C1 - C2 - C3  
1.56 ( 0.58) S1 - C1 - C2 - C3  
-58.31 ( 0.38) Ir1 - C1 - C2 - C3  
-120.10 ( 0.48) C6 - C1 - C2 - Ir1  
59.87 ( 0.28) S1 - C1 - C2 - Ir1  
168.15 ( 0.43) C1 - C2 - C3 - C4  
109.99 ( 0.46) Ir1 - C2 - C3 - C4  
58.16 ( 0.38) C1 - C2 - C3 - Ir1  
176.89 ( 0.45) C2 - C3 - C4 - N1  
-100.74 ( 0.50) Ir1 - C3 - C4 - N1  
0.67 ( 0.72) C2 - C3 - C4 - C5  
83.04 ( 0.50) Ir1 - C3 - C4 - C5  
52.56 ( 0.75) N1 - C7 - C8 - C9  
-57.67 ( 0.85) C7 - C8 - C9 - C10  
58.37 ( 0.90) C8 - C9 - C10 - C11  
-53.32 ( 0.83) C9 - C10 - C11 - N1  
0.03 ( 0.51) C16 - C12 - C13 - C14  
-177.49 ( 0.46) C17 - C12 - C13 - C14  
61.33 ( 0.31) Ir1 - C12 - C13 - C14  
176.95 ( 0.52) C16 - C12 - C13 - C18  
-0.57 ( 0.85) C17 - C12 - C13 - C18  
-121.75 ( 0.57) Ir1 - C12 - C13 - C18  
-61.30 ( 0.31) C16 - C12 - C13 - Ir1  
121.18 ( 0.50) C17 - C12 - C13 - Ir1  
1.16 ( 0.52) C12 - C13 - C14 - C15  
-175.87 ( 0.50) C18 - C13 - C14 - C15  
61.82 ( 0.31) Ir1 - C13 - C14 - C15  
175.39 ( 0.42) C12 - C13 - C14 - C19  
-1.64 ( 0.77) C18 - C13 - C14 - C19  
-123.95 ( 0.43) Ir1 - C13 - C14 - C19  
-60.66 ( 0.32) C12 - C13 - C14 - Ir1  
122.31 ( 0.53) C18 - C13 - C14 - Ir1  
-1.91 ( 0.52) C13 - C14 - C15 - C16  
-175.97 ( 0.42) C19 - C14 - C15 - C16  
59.61 ( 0.32) Ir1 - C14 - C15 - C16  
174.66 ( 0.44) C13 - C14 - C15 - C20  
0.60 ( 0.74) C19 - C14 - C15 - C20

-123.81 ( 0.48) Ir1 - C14 - C15 - C20  
-61.52 ( 0.31) C13 - C14 - C15 - Ir1  
124.42 ( 0.45) C19 - C14 - C15 - Ir1  
1.92 ( 0.51) C14 - C15 - C16 - C12  
-174.59 ( 0.46) C20 - C15 - C16 - C12  
62.87 ( 0.32) Ir1 - C15 - C16 - C12  
176.04 ( 0.44) C14 - C15 - C16 - C21  
-0.47 ( 0.79) C20 - C15 - C16 - C21  
-123.01 ( 0.48) Ir1 - C15 - C16 - C21  
-60.95 ( 0.31) C14 - C15 - C16 - Ir1  
122.53 ( 0.50) C20 - C15 - C16 - Ir1  
-1.19 ( 0.52) C13 - C12 - C16 - C15  
176.31 ( 0.47) C17 - C12 - C16 - C15  
-63.89 ( 0.32) Ir1 - C12 - C16 - C15  
-175.25 ( 0.45) C13 - C12 - C16 - C21  
2.25 ( 0.79) C17 - C12 - C16 - C21  
122.05 ( 0.48) Ir1 - C12 - C16 - C21  
62.69 ( 0.32) C13 - C12 - C16 - Ir1  
-119.80 ( 0.50) C17 - C12 - C16 - Ir1  
-164.08 ( 1.06) C1 - C2 - Ir1 - C16  
-36.26 ( 1.25) C3 - C2 - Ir1 - C16  
127.82 ( 0.43) C3 - C2 - Ir1 - C1  
50.80 ( 0.48) C1 - C2 - Ir1 - C12  
178.62 ( 0.33) C3 - C2 - Ir1 - C12  
-127.82 ( 0.43) C1 - C2 - Ir1 - C3  
85.82 ( 0.33) C1 - C2 - Ir1 - C13  
-146.36 ( 0.30) C3 - C2 - Ir1 - C13  
128.34 ( 0.28) C1 - C2 - Ir1 - C14  
-103.84 ( 0.31) C3 - C2 - Ir1 - C14  
166.16 ( 0.29) C1 - C2 - Ir1 - C15  
-66.03 ( 0.39) C3 - C2 - Ir1 - C15  
-36.07 ( 0.26) C1 - C2 - Ir1 - S1  
91.75 ( 0.29) C3 - C2 - Ir1 - S1  
-34.96 ( 1.25) C15 - C16 - Ir1 - C2  
-151.59 ( 1.08) C12 - C16 - Ir1 - C2  
86.76 ( 1.23) C21 - C16 - Ir1 - C2  
164.84 ( 0.35) C15 - C16 - Ir1 - C1  
48.22 ( 0.50) C12 - C16 - Ir1 - C1  
-73.43 ( 0.66) C21 - C16 - Ir1 - C1  
116.63 ( 0.42) C15 - C16 - Ir1 - C12

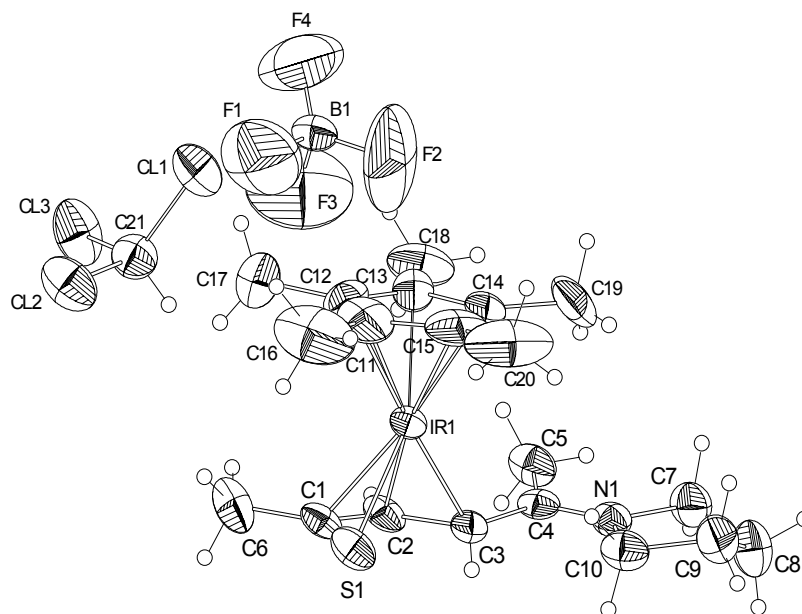
-121.65 ( 0.59) C21 - C16 - Ir1 - C12  
-65.92 ( 0.39) C15 - C16 - Ir1 - C3  
177.45 ( 0.28) C12 - C16 - Ir1 - C3  
55.80 ( 0.57) C21 - C16 - Ir1 - C3  
79.32 ( 0.32) C15 - C16 - Ir1 - C13  
-37.31 ( 0.27) C12 - C16 - Ir1 - C13  
-158.96 ( 0.54) C21 - C16 - Ir1 - C13  
36.39 ( 0.28) C15 - C16 - Ir1 - C14  
-80.24 ( 0.29) C12 - C16 - Ir1 - C14  
158.11 ( 0.55) C21 - C16 - Ir1 - C14  
-116.63 ( 0.42) C12 - C16 - Ir1 - C15  
121.72 ( 0.62) C21 - C16 - Ir1 - C15  
-160.47 ( 0.26) C15 - C16 - Ir1 - S1  
82.90 ( 0.27) C12 - C16 - Ir1 - S1  
-38.74 ( 0.52) C21 - C16 - Ir1 - S1  
115.33 ( 0.56) C6 - C1 - Ir1 - C2  
-126.77 ( 0.36) S1 - C1 - Ir1 - C2  
175.31 ( 0.33) C2 - C1 - Ir1 - C16  
-69.36 ( 0.61) C6 - C1 - Ir1 - C16  
48.53 ( 0.45) S1 - C1 - Ir1 - C16  
-152.91 ( 0.29) C2 - C1 - Ir1 - C12  
-37.59 ( 0.51) C6 - C1 - Ir1 - C12  
80.31 ( 0.23) S1 - C1 - Ir1 - C12  
32.03 ( 0.29) C2 - C1 - Ir1 - C3  
147.36 ( 0.51) C6 - C1 - Ir1 - C3  
-94.75 ( 0.23) S1 - C1 - Ir1 - C3  
-112.08 ( 0.30) C2 - C1 - Ir1 - C13  
3.25 ( 0.49) C6 - C1 - Ir1 - C13  
121.14 ( 0.20) S1 - C1 - Ir1 - C13  
-74.58 ( 0.36) C2 - C1 - Ir1 - C14  
40.75 ( 0.57) C6 - C1 - Ir1 - C14  
158.64 ( 0.18) S1 - C1 - Ir1 - C14  
-58.51 ( 1.01) C2 - C1 - Ir1 - C15  
56.81 ( 1.15) C6 - C1 - Ir1 - C15  
174.71 ( 0.82) S1 - C1 - Ir1 - C15  
126.77 ( 0.36) C2 - C1 - Ir1 - S1  
-117.90 ( 0.54) C6 - C1 - Ir1 - S1  
54.55 ( 0.48) C13 - C12 - Ir1 - C2  
172.12 ( 0.31) C16 - C12 - Ir1 - C2  
-67.07 ( 0.64) C17 - C12 - Ir1 - C2

-117.56 ( 0.41) C13 - C12 - Ir1 - C16  
120.82 ( 0.61) C17 - C12 - Ir1 - C16  
87.49 ( 0.33) C13 - C12 - Ir1 - C1  
-154.95 ( 0.27) C16 - C12 - Ir1 - C1  
-34.13 ( 0.55) C17 - C12 - Ir1 - C1  
-132.35 ( 1.45) C13 - C12 - Ir1 - C3  
-14.79 ( 1.61) C16 - C12 - Ir1 - C3  
106.03 ( 1.52) C17 - C12 - Ir1 - C3  
117.56 ( 0.41) C16 - C12 - Ir1 - C13  
-121.62 ( 0.64) C17 - C12 - Ir1 - C13  
-37.90 ( 0.29) C13 - C12 - Ir1 - C14  
79.67 ( 0.29) C16 - C12 - Ir1 - C14  
-159.52 ( 0.56) C17 - C12 - Ir1 - C14  
-79.70 ( 0.32) C13 - C12 - Ir1 - C15  
37.86 ( 0.27) C16 - C12 - Ir1 - C15  
158.68 ( 0.56) C17 - C12 - Ir1 - C15  
132.75 ( 0.27) C13 - C12 - Ir1 - S1  
-109.69 ( 0.25) C16 - C12 - Ir1 - S1  
11.13 ( 0.52) C17 - C12 - Ir1 - S1  
-118.41 ( 0.54) C4 - C3 - Ir1 - C2  
172.88 ( 0.26) C2 - C3 - Ir1 - C16  
54.46 ( 0.51) C4 - C3 - Ir1 - C16  
-31.98 ( 0.28) C2 - C3 - Ir1 - C1  
-150.40 ( 0.46) C4 - C3 - Ir1 - C1  
-174.23 ( 1.39) C2 - C3 - Ir1 - C12  
67.36 ( 1.64) C4 - C3 - Ir1 - C12  
61.24 ( 0.47) C2 - C3 - Ir1 - C13  
-57.18 ( 0.56) C4 - C3 - Ir1 - C13  
94.88 ( 0.31) C2 - C3 - Ir1 - C14  
-23.53 ( 0.46) C4 - C3 - Ir1 - C14  
135.62 ( 0.29) C2 - C3 - Ir1 - C15  
17.20 ( 0.46) C4 - C3 - Ir1 - C15  
-77.72 ( 0.28) C2 - C3 - Ir1 - S1  
163.87 ( 0.41) C4 - C3 - Ir1 - S1  
-150.96 ( 0.30) C12 - C13 - Ir1 - C2  
91.65 ( 0.30) C14 - C13 - Ir1 - C2  
-28.27 ( 0.54) C18 - C13 - Ir1 - C2  
38.05 ( 0.29) C12 - C13 - Ir1 - C16  
-79.35 ( 0.29) C14 - C13 - Ir1 - C16  
160.74 ( 0.53) C18 - C13 - Ir1 - C16

-109.70 ( 0.32) C12 - C13 - Ir1 - C1  
132.90 ( 0.29) C14 - C13 - Ir1 - C1  
12.99 ( 0.51) C18 - C13 - Ir1 - C1  
-117.40 ( 0.42) C14 - C13 - Ir1 - C12  
122.69 ( 0.61) C18 - C13 - Ir1 - C12  
170.39 ( 0.33) C12 - C13 - Ir1 - C3  
52.99 ( 0.46) C14 - C13 - Ir1 - C3  
-66.92 ( 0.62) C18 - C13 - Ir1 - C3  
117.40 ( 0.42) C12 - C13 - Ir1 - C14  
-119.91 ( 0.59) C18 - C13 - Ir1 - C14  
80.63 ( 0.32) C12 - C13 - Ir1 - C15  
-36.76 ( 0.26) C14 - C13 - Ir1 - C15  
-156.68 ( 0.54) C18 - C13 - Ir1 - C15  
-61.90 ( 0.33) C12 - C13 - Ir1 - S1  
-179.29 ( 0.21) C14 - C13 - Ir1 - S1  
60.79 ( 0.51) C18 - C13 - Ir1 - S1  
134.03 ( 0.29) C15 - C14 - Ir1 - C2  
-107.99 ( 0.29) C13 - C14 - Ir1 - C2  
11.43 ( 0.50) C19 - C14 - Ir1 - C2  
-37.06 ( 0.27) C15 - C14 - Ir1 - C16  
80.92 ( 0.30) C13 - C14 - Ir1 - C16  
-159.67 ( 0.52) C19 - C14 - Ir1 - C16  
174.78 ( 0.27) C15 - C14 - Ir1 - C1  
-67.24 ( 0.37) C13 - C14 - Ir1 - C1  
52.17 ( 0.53) C19 - C14 - Ir1 - C1  
-80.48 ( 0.31) C15 - C14 - Ir1 - C12  
37.51 ( 0.28) C13 - C14 - Ir1 - C12  
156.92 ( 0.50) C19 - C14 - Ir1 - C12  
91.54 ( 0.31) C15 - C14 - Ir1 - C3  
-150.48 ( 0.28) C13 - C14 - Ir1 - C3  
-31.07 ( 0.51) C19 - C14 - Ir1 - C3  
-117.99 ( 0.39) C15 - C14 - Ir1 - C13  
119.41 ( 0.57) C19 - C14 - Ir1 - C13  
117.99 ( 0.39) C13 - C14 - Ir1 - C15  
-122.60 ( 0.55) C19 - C14 - Ir1 - C15  
-115.89 ( 0.43) C15 - C14 - Ir1 - S1  
2.10 ( 0.62) C13 - C14 - Ir1 - S1  
121.51 ( 0.49) C19 - C14 - Ir1 - S1  
-68.08 ( 0.37) C14 - C15 - Ir1 - C2  
173.06 ( 0.28) C16 - C15 - Ir1 - C2

52.37 ( 0.58) C20 - C15 - Ir1 - C2  
118.86 ( 0.41) C14 - C15 - Ir1 - C16  
-120.68 ( 0.61) C20 - C15 - Ir1 - C16  
-19.92 ( 1.03) C14 - C15 - Ir1 - C1  
-138.78 ( 0.88) C16 - C15 - Ir1 - C1  
100.53 ( 1.02) C20 - C15 - Ir1 - C1  
80.32 ( 0.29) C14 - C15 - Ir1 - C12  
-38.54 ( 0.29) C16 - C15 - Ir1 - C12  
-159.23 ( 0.53) C20 - C15 - Ir1 - C12  
-105.80 ( 0.29) C14 - C15 - Ir1 - C3  
135.33 ( 0.30) C16 - C15 - Ir1 - C3  
14.65 ( 0.53) C20 - C15 - Ir1 - C3  
37.76 ( 0.27) C14 - C15 - Ir1 - C13  
-81.10 ( 0.31) C16 - C15 - Ir1 - C13  
158.21 ( 0.54) C20 - C15 - Ir1 - C13  
-118.86 ( 0.41) C16 - C15 - Ir1 - C14  
120.45 ( 0.57) C20 - C15 - Ir1 - C14  
153.28 ( 0.23) C14 - C15 - Ir1 - S1  
34.42 ( 0.43) C16 - C15 - Ir1 - S1  
-86.26 ( 0.50) C20 - C15 - Ir1 - S1  
2.64 ( 0.73) C3 - C4 - N1 - C7  
178.92 ( 0.47) C5 - C4 - N1 - C7  
-164.96 ( 0.46) C3 - C4 - N1 - C11  
11.31 ( 0.70) C5 - C4 - N1 - C11  
144.04 ( 0.52) C8 - C7 - N1 - C4  
-47.46 ( 0.67) C8 - C7 - N1 - C11  
-144.00 ( 0.58) C10 - C11 - N1 - C4  
47.73 ( 0.70) C10 - C11 - N1 - C7  
-57.04 ( 0.32) C2 - C1 - S1 - Ir1  
122.94 ( 0.47) C6 - C1 - S1 - Ir1  
31.40 ( 0.25) C2 - Ir1 - S1 - C1  
-155.95 ( 0.26) C16 - Ir1 - S1 - C1  
-116.38 ( 0.26) C12 - Ir1 - S1 - C1  
70.92 ( 0.25) C3 - Ir1 - S1 - C1  
-82.55 ( 0.28) C13 - Ir1 - S1 - C1  
-84.15 ( 0.47) C14 - Ir1 - S1 - C1  
-178.07 ( 0.30) C15 - Ir1 - S1 - C1



DEPARTAMENTO DE QUIMICA  
CinvestavORTEP drawing of Compound **13**Formula:  $C_{21}H_{32}BCl_3F_4IrNS$ 

Authors : Marisol Cervantes Vásquez y Ma. de los Ángeles Paz Sandoval.

Tab. 1 Crystal data and data collection

Tab. 2 Solution and refinement

Tab. 3 Atomic coordinates and equivalent isotropic displacement parameters

Tab. 4 Bond lengths and angles

Tab. 5 Anisotropic displacement parameters

Tab. 6 Hydrogen coordinates and isotropic displacement parameters



**Table 2.** Solution and refinement

|                                   |   |
|-----------------------------------|---|
| Structure solution program        | SHELXS-97(Sheldrick 1990)   |
| Solution                          | heavy-atom-method   |
| Refinement method                 | Full-matrix Least-Squares on F <sup>2</sup>                             |
| Hydrogen atoms                    | mixed   |
| Weighting scheme                  | $w^{-1} = \sigma^2 F_o^2 + (P)^2 + P$<br>where $P = (F_o^2 + 2F_c^2)/3$ |
| Data / restraints / parameters    | 2932 / 0 / 298  |
| Data-to-parameter-ratio           | 9.8 : 1 (9.8 : 1 [F > 4σ(F)])   |
| Final R indices [F > 4σ(F)]       | R1 = 0.0391, wR2 = 0.1037   |
| R indices (all data)              | R1 = 0.0391, wR2 = 0.1037   |
| Goodness-of-Fit on F <sup>2</sup> | 1.056   |
| Largest and mean Δ/σ              | 0.001 0.000   |
| Largest difference peak           | 1.590 eÅ <sup>-3</sup>  |
| Largest difference hole           | -1.158 eÅ <sup>-3</sup>   |

**Refinement details :**

|                     |                            |
|---------------------|----------------------------|
| Program used        | SHELXL-97 (Sheldrick 1997) |
| CifRtf version used | 2.0                        |

**Table 3.** Atomic coordinates (  $\times 10^4$  ) and equivalent isotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ ) for compound **13**. U(eq) is defined as one third of the trace of the orthogonalized  $U_{ij}$  tensor

|       | x         | y         | z         | U(eq)    |
|-------|-----------|-----------|-----------|----------|
| C(1)  | 4926(10)  | 7251(9)   | 7410(8)   | 36(3)    |
| C(2)  | 5699(11)  | 8270(7)   | 6472(8)   | 39(3)    |
| C(3)  | 5673(12)  | 8408(10)  | 5276(8)   | 39(3)    |
| C(4)  | 6631(10)  | 9232(7)   | 4253(7)   | 34(3)    |
| C(5)  | 7723(13)  | 9992(9)   | 4292(9)   | 49(3)    |
| C(6)  | 4925(15)  | 6984(10)  | 8653(8)   | 57(4)    |
| C(7)  | 7341(13)  | 10121(10) | 2081(9)   | 51(3)    |
| C(8)  | 6572(17)  | 9993(11)  | 1237(9)   | 60(4)    |
| C(9)  | 6018(16)  | 8719(11)  | 1785(9)   | 60(4)    |
| C(10) | 5560(13)  | 8520(9)   | 3097(9)   | 48(3)    |
| C(11) | 7356(17)  | 4871(10)  | 6848(12)  | 77(6)    |
| C(12) | 8092(14)  | 5617(10)  | 7090(9)   | 50(4)    |
| C(13) | 8838(11)  | 6506(10)  | 6103(11)  | 46(3)    |
| C(14) | 8530(13)  | 6311(10)  | 5093(8)   | 53(4)    |
| C(15) | 7602(15)  | 5312(12)  | 5558(13)  | 67(4)    |
| C(16) | 6420(02)  | 3772(12)  | 7628(18)  | 120(9)   |
| C(17) | 8200(02)  | 5428(18)  | 8320(11)  | 98(7)    |
| C(18) | 9814(15)  | 7466(13)  | 5935(14)  | 76(5)    |
| C(19) | 9185(19)  | 7055(17)  | 3811(11)  | 122(9)   |
| C(20) | 7116(19)  | 4759(17)  | 4857(19)  | 122(9)   |
| C(21) | 9468(14)  | 7396(10)  | 9943(10)  | 52(3)    |
| Ir(1) | 6531.6(4) | 6673.4(3) | 6191.0(3) | 29.5(1)  |
| S(1)  | 4006(3)   | 6358(2)   | 7016(2)   | 46.5(8)  |
| N(1)  | 6503(9)   | 9295(6)   | 3231(6)   | 39(2)    |
| Cl(1) | 10853(4)  | 6868(3)   | 9065(3)   | 63.9(10) |
| Cl(2) | 8258(4)   | 6228(3)   | 10924(3)  | 70.9(11) |
| Cl(3) | 10167(5)  | 8054(3)   | 10699(3)  | 76.6(13) |
| B(1)  | 8120(14)  | 96(12)    | 7757(11)  | 46(4)    |
| F(1)  | 7123(17)  | -803(14)  | 8556(13)  | 169(7)   |
| F(2)  | 7920(02)  | 360(18)   | 6693(10)  | 191(8)   |
| F(3)  | 7590(02)  | 1007(14)  | 7945(18)  | 206(9)   |
| F(4)  | 9262(15)  | -106(19)  | 7937(14)  | 234(13)  |

**Table 4.** Bond lengths [Å] and angles [°] for compound **13**

|                   |           |                   |           |
|-------------------|-----------|-------------------|-----------|
| C(1)-C(2)         | 1.422(14) | C(1)-C(6)         | 1.480(14) |
| C(1)-S(1)         | 1.734(11) | C(1)-Ir(1)        | 2.146(7)  |
| C(2)-C(3)         | 1.465(13) | C(2)-Ir(1)        | 2.138(7)  |
| C(3)-C(4)         | 1.426(14) |                   |           |
| C(3)-Ir(1)        | 2.178(9)  | C(3)-H(3)         | 0.78(11)  |
| C(4)-N(1)         | 1.306(12) | C(4)-C(5)         | 1.474(17) |
| C(7)-N(1)         | 1.464(12) | C(7)-C(8)         | 1.51(2)   |
| C(8)-C(9)         | 1.496(18) | C(9)-C(10)        | 1.525(15) |
| C(10)-N(1)        | 1.441(16) | C(11)-C(12)       | 1.36(2)   |
| C(11)-C(15)       | 1.465(19) | C(11)-C(16)       | 1.50(2)   |
| C(11)-Ir(1)       | 2.169(9)  | C(12)-C(13)       | 1.355(17) |
| C(12)-C(17)       | 1.516(17) | C(12)-Ir(1)       | 2.158(10) |
| C(13)-C(18)       | 1.47(2)   | C(13)-C(14)       | 1.493(16) |
| C(13)-Ir(1)       | 2.191(10) | C(14)-C(15)       | 1.394(19) |
| C(14)-C(19)       | 1.501(15) | C(14)-Ir(1)       | 2.168(8)  |
| C(15)-C(20)       | 1.478(18) | C(15)-Ir(1)       | 2.160(9)  |
| C(21)-Cl(1)       | 1.736(10) | C(21)-Cl(3)       | 1.739(14) |
| C(21)-Cl(2)       | 1.740(14) | Ir(1)-S(1)        | 2.372(3)  |
| B(1)-F(4)         | 1.17(2)   | B(1)-F(3)         | 1.250(17) |
| B(1)-F(2)         | 1.324(17) | B(1)-F(1)         | 1.39(2)   |
| C(2)-C(1)-C(6)    | 120.7(10) | C(2)-C(1)-S(1)    | 116.7(7)  |
| C(6)-C(1)-S(1)    | 122.6(9)  | C(2)-C(1)-Ir(1)   | 70.3(4)   |
| C(6)-C(1)-Ir(1)   | 125.0(7)  | S(1)-C(1)-Ir(1)   | 74.5(3)   |
| C(1)-C(2)-C(3)    | 115.6(10) | C(1)-C(2)-Ir(1)   | 70.9(4)   |
| C(3)-C(2)-Ir(1)   | 71.6(5)   | C(4)-C(3)-C(2)    | 121.2(12) |
| C(4)-C(3)-Ir(1)   | 117.2(6)  | C(2)-C(3)-Ir(1)   | 68.7(4)   |
| C(4)-C(3)-H(3)    | 115(6)    | C(2)-C(3)-H(3)    | 110(6)    |
| Ir(1)-C(3)-H(3)   | 117(7)    | N(1)-C(4)-C(3)    | 116.1(10) |
| N(1)-C(4)-C(5)    | 119.7(9)  | C(3)-C(4)-C(5)    | 124.2(9)  |
| N(1)-C(7)-C(8)    | 102.2(11) | C(9)-C(8)-C(7)    | 105.4(9)  |
| C(8)-C(9)-C(10)   | 101.5(10) | N(1)-C(10)-C(9)   | 105.2(10) |
| C(12)-C(11)-C(15) | 107.6(12) | C(12)-C(11)-C(16) | 132.4(16) |
| C(15)-C(11)-C(16) | 120.1(18) | C(12)-C(11)-Ir(1) | 71.3(5)   |
| C(15)-C(11)-Ir(1) | 69.9(5)   | C(16)-C(11)-Ir(1) | 123.9(10) |
| C(13)-C(12)-C(11) | 113.1(11) | C(13)-C(12)-C(17) | 122.3(15) |
| C(11)-C(12)-C(17) | 124.4(14) | C(13)-C(12)-Ir(1) | 73.2(5)   |
| C(11)-C(12)-Ir(1) | 72.1(7)   | C(17)-C(12)-Ir(1) | 127.2(8)  |
| C(12)-C(13)-C(18) | 132.1(12) | C(12)-C(13)-C(14) | 105.1(11) |

|                    |           |                   |           |
|--------------------|-----------|-------------------|-----------|
| C(18)-C(13)-C(14)  | 122.8(12) | C(12)-C(13)-Ir(1) | 70.5(7)   |
| C(18)-C(13)-Ir(1)  | 126.1(7)  | C(14)-C(13)-Ir(1) | 69.2(6)   |
| C(15)-C(14)-C(13)  | 108.1(10) | C(15)-C(14)-C(19) | 127.7(15) |
| C(13)-C(14)-C(19)  | 124.2(15) | C(15)-C(14)-Ir(1) | 70.9(6)   |
| C(13)-C(14)-Ir(1)  | 70.8(5)   | C(19)-C(14)-Ir(1) | 126.0(7)  |
| C(14)-C(15)-C(11)  | 106.1(12) | C(14)-C(15)-C(20) | 125.5(15) |
| C(11)-C(15)-C(20)  | 128.1(17) | C(14)-C(15)-Ir(1) | 71.5(5)   |
| C(11)-C(15)-Ir(1)  | 70.5(5)   | C(20)-C(15)-Ir(1) | 127.5(8)  |
| C(11)-C(16)-H(16A) | 109.5     | Cl(1)-C(21)-Cl(3) | 110.2(8)  |
| Cl(1)-C(21)-Cl(2)  | 110.1(6)  | Cl(3)-C(21)-Cl(2) | 111.1(6)  |
| C(2)-Ir(1)-C(1)    | 38.8(4)   |                   |           |
| C(2)-Ir(1)-C(12)   | 120.5(4)  | C(1)-Ir(1)-C(12)  | 108.8(4)  |
| C(2)-Ir(1)-C(15)   | 168.4(5)  | C(1)-Ir(1)-C(15)  | 152.6(5)  |
| C(12)-Ir(1)-C(15)  | 63.7(5)   | C(2)-Ir(1)-C(14)  | 132.5(5)  |
| C(1)-Ir(1)-C(14)   | 165.1(5)  | C(12)-Ir(1)-C(14) | 63.1(4)   |
| C(15)-Ir(1)-C(14)  | 37.6(5)   | C(2)-Ir(1)-C(11)  | 150.0(5)  |
| C(1)-Ir(1)-C(11)   | 118.3(4)  | C(12)-Ir(1)-C(11) | 36.6(6)   |
| C(15)-Ir(1)-C(11)  | 39.6(5)   | C(14)-Ir(1)-C(11) | 63.6(4)   |
| C(2)-Ir(1)-C(3)    | 39.7(4)   | C(1)-Ir(1)-C(3)   | 68.8(3)   |
| C(12)-Ir(1)-C(3)   | 150.8(5)  | C(15)-Ir(1)-C(3)  | 131.3(4)  |
| C(14)-Ir(1)-C(3)   | 111.6(3)  | C(11)-Ir(1)-C(3)  | 170.1(5)  |
| C(2)-Ir(1)-C(13)   | 111.3(4)  | C(1)-Ir(1)-C(13)  | 126.0(4)  |
| C(12)-Ir(1)-C(13)  | 36.3(4)   | C(15)-Ir(1)-C(13) | 65.0(5)   |
| C(14)-Ir(1)-C(13)  | 40.0(4)   | C(11)-Ir(1)-C(13) | 62.6(6)   |
| C(3)-Ir(1)-C(13)   | 120.1(5)  | C(2)-Ir(1)-S(1)   | 73.1(3)   |
| C(1)-Ir(1)-S(1)    | 44.8(3)   | C(12)-Ir(1)-S(1)  | 122.8(3)  |
| C(15)-Ir(1)-S(1)   | 114.7(4)  | C(14)-Ir(1)-S(1)  | 149.9(4)  |
| C(11)-Ir(1)-S(1)   | 102.7(4)  | C(3)-Ir(1)-S(1)   | 77.2(3)   |
| C(13)-Ir(1)-S(1)   | 158.6(3)  | C(1)-S(1)-Ir(1)   | 60.7(3)   |
| C(4)-N(1)-C(10)    | 124.0(8)  | C(4)-N(1)-C(7)    | 124.4(10) |
| C(10)-N(1)-C(7)    | 111.5(9)  | F(4)-B(1)-F(3)    | 112(2)    |
| F(4)-B(1)-F(2)     | 122.1(16) | F(3)-B(1)-F(2)    | 99.4(14)  |
| F(4)-B(1)-F(1)     | 111.5(13) | F(3)-B(1)-F(1)    | 102.3(14) |
| F(2)-B(1)-F(1)     | 107.3(16) |                   |           |

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**Table 5.** Anisotropic displacement parameters [ $\text{\AA}^2 \times 10^3$ ]

|       | U11      | U22      | U33      | U23       | U13      | U12       |
|-------|----------|----------|----------|-----------|----------|-----------|
| C(1)  | 22(5)    | 47(5)    | 47(5)    | -29(4)    | 0(4)     | -5(5)     |
| C(2)  | 41(6)    | 27(4)    | 48(5)    | -22(4)    | 1(4)     | 9(5)      |
| C(3)  | 37(6)    | 39(5)    | 35(4)    | -13(4)    | -6(4)    | 14(6)     |
| C(4)  | 30(5)    | 18(3)    | 48(5)    | -11(3)    | -8(4)    | 7(4)      |
| C(5)  | 39(7)    | 37(5)    | 60(6)    | -13(4)    | -1(5)    | -4(6)     |
| C(6)  | 66(10)   | 50(5)    | 43(5)    | -19(4)    | 12(6)    | -13(7)    |
| C(7)  | 44(7)    | 45(5)    | 46(5)    | -5(4)     | -1(5)    | -6(7)     |
| C(8)  | 67(11)   | 60(7)    | 45(5)    | -15(5)    | -4(6)    | -15(9)    |
| C(9)  | 63(9)    | 64(7)    | 49(5)    | -22(5)    | -9(6)    | 8(8)      |
| C(10) | 36(7)    | 42(5)    | 62(6)    | -17(5)    | -8(5)    | -5(6)     |
| C(11) | 81(11)   | 30(5)    | 91(9)    | -8(5)     | -2(8)    | 27(7)     |
| C(12) | 57(8)    | 45(6)    | 48(5)    | -14(4)    | -23(5)   | 17(7)     |
| C(13) | 17(5)    | 56(6)    | 90(8)    | -45(6)    | -28(5)   | 8(6)      |
| C(14) | 49(7)    | 61(7)    | 45(5)    | -24(5)    | -8(5)    | 33(7)     |
| C(15) | 58(9)    | 62(7)    | 114(10)  | -67(8)    | -38(8)   | 34(8)     |
| C(16) | 91(15)   | 36(6)    | 171(17)  | -2(8)     | 10(13)   | 1(10)     |
| C(17) | 98(14)   | 139(15)  | 56(7)    | -34(9)    | -31(8)   | 34(14)    |
| C(18) | 39(8)    | 86(10)   | 120(11)  | -56(9)    | -24(8)   | 6(9)      |
| C(19) | 106(14)  | 149(16)  | 54(7)    | -22(8)    | 21(8)    | 73(14)    |
| C(20) | 96(15)   | 168(18)  | 230(02)  | -179(18)  | -100(15) | 70(15)    |
| C(21) | 55(8)    | 45(5)    | 58(6)    | -23(5)    | -14(6)   | 5(7)      |
| Ir(1) | 27.5(2)  | 26.6(1)  | 37.1(1)  | -16.4(1)  | -6.3(1)  | 1.2(1)    |
| S(1)  | 33.2(16) | 46.4(13) | 53.2(13) | -20.2(11) | 5.5(12)  | -11.9(15) |
| N(1)  | 36(5)    | 30(3)    | 44(4)    | -12(3)    | -7(4)    | 7(4)      |
| Cl(1) | 59(2)    | 68.8(17) | 66.3(16) | -38.3(14) | 0.6(15)  | -5(2)     |
| Cl(2) | 62(2)    | 58.1(16) | 83(2)    | -31.1(15) | 5.1(18)  | -11(2)    |
| Cl(3) | 87(3)    | 86(2)    | 67.2(17) | -45.9(16) | -1(2)    | -27(3)    |
| B(1)  | 30(7)    | 49(6)    | 57(6)    | -21(5)    | -8(6)    | 3(7)      |
| F(1)  | 106(13)  | 165(12)  | 174(12)  | -16(10)   | 2(10)    | -29(13)   |
| F(2)  | 190(02)  | 330(02)  | 82(7)    | -69(10)   | -53(10)  | -100(02)  |
| F(3)  | 210(02)  | 150(12)  | 320(02)  | -148(15)  | -87(18)  | 40(15)    |
| F(4)  | 86(10)   | 260(02)  | 183(13)  | 78(13)    | -21(9)   | 39(14)    |

**Table 6.** Hydrogen coordinates (  $\times 10^4$  ) and isotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ ) for compound **13**

|        | x         | y        | z        | U(eq)  |
|--------|-----------|----------|----------|--------|
| H(2)   | 6289      | 8798     | 6611     | 47     |
| H(5A)  | 8391      | 9492     | 4704     | 80     |
| H(5B)  | 7250      | 10504    | 4699     | 80     |
| H(5C)  | 8234      | 10478    | 3499     | 80     |
| H(6A)  | 4928      | 6130     | 9082     | 80     |
| H(6B)  | 4101      | 7320     | 9042     | 80     |
| H(6C)  | 5790      | 7344     | 8630     | 80     |
| H(7A)  | 8325      | 9876     | 1917     | 80     |
| H(7B)  | 7329      | 10932    | 2047     | 80     |
| H(8A)  | 7256      | 10141    | 484      | 80     |
| H(8B)  | 5812      | 10563    | 1130     | 80     |
| H(9A)  | 6739      | 8160     | 1629     | 80     |
| H(9B)  | 5202      | 8648     | 1515     | 80     |
| H(10A) | 5664      | 7694     | 3582     | 80     |
| H(10B) | 4572      | 8734     | 3323     | 80     |
| H(16A) | 6917      | 3226     | 8181     | 179    |
| H(16B) | 6221      | 3384     | 7149     | 179    |
| H(16C) | 5536      | 4002     | 8052     | 179    |
| H(17A) | 8840      | 4781     | 8568     | 147    |
| H(17B) | 7260      | 5232     | 8865     | 147    |
| H(17C) | 8562      | 6153     | 8306     | 147    |
| H(18A) | 9270      | 8050     | 6213     | 114    |
| H(18B) | 10289     | 7847     | 5108     | 114    |
| H(18C) | 10521     | 7128     | 6373     | 114    |
| H(19A) | 8728      | 6845     | 3326     | 183    |
| H(19B) | 10198     | 6902     | 3612     | 183    |
| H(19C) | 9048      | 7893     | 3676     | 183    |
| H(20A) | 6873      | 5381     | 4188     | 183    |
| H(20B) | 6283      | 4252     | 5343     | 183    |
| H(20C) | 7875      | 4286     | 4586     | 183    |
| H(21)  | 8966      | 7996     | 9435     | 80     |
| H(3)   | 4880(101) | 8410(08) | 5240(06) | 10(02) |



**Table 7.** Crystal data and data collection parameters

| Compound                            | Compound <b>13</b>  |
|-------------------------------------|---|
| Chem. formula                       | C <sub>21</sub> H <sub>32</sub> B Cl <sub>3</sub> F <sub>4</sub> Ir N S |
| Form. wght.                         | 715.90  |
| Cryst. size [mm]                    | 0.33x0.45x0.56  |
| Cryst. system                       | Triclinic   |
| Space group                         | P -1  |
| a, [Å]                              | 9.6363(10)  |
| b, [Å]                              | 12.0380(10)   |
| c, [Å]                              | 12.6614(10)   |
| α, [°]                              | 67.321(10)  |
| β, [°]                              | 74.241(10)  |
| γ, [°]                              | 85.072(10)  |
| V, [Å <sup>3</sup> ]                | 1304.0(2)   |
| Z                                   | 2   |
| ρ(calcd.), [Mg/m <sup>3</sup> ]     | 1.823   |
| μ [mm <sup>-1</sup> ]               | 5.546   |
| F(000)                              | 700   |
| Index range                         | -11≤h≤11-13≤k≤140≤l≤15  |
| 2 θ [°]                             | 52.48   |
| Temp, [K]                           | 293(2)  |
| Refl. collected                     | 2932  |
| Refl. unique                        | 2932  |
| Refl. observed (4σ)                 | 2932  |
| R (int.)                            | 0.0000  |
| No. variables                       | 298   |
| Weighting scheme <sup>1</sup> x/y   | /   |
| GOOF                                | 1.056   |
| Final R (4σ)                        | 0.0391  |
| Final wR2                           | 0.1037  |
| Larg. res. peak [e/Å <sup>3</sup> ] | 1.590   |

$$^1 w^{-1} = \sigma^2 F_o^2 + (xP)^2 + yP; P = (F_o^2 + 2F_c^2)/3$$

**Selected torsion angles for compound 13**

177.25 ( 0.80) C6 - C1 - C2 - C3  
-2.37 ( 0.95) S1 - C1 - C2 - C3  
57.76 ( 0.60) Ir1 - C1 - C2 - C3  
119.49 ( 0.84) C6 - C1 - C2 - Ir1  
-60.13 ( 0.48) S1 - C1 - C2 - Ir1  
-166.97 ( 0.70) C1 - C2 - C3 - C4  
-109.60 ( 0.71) Ir1 - C2 - C3 - C4  
-57.37 ( 0.60) C1 - C2 - C3 - Ir1  
-179.32 ( 0.66) C2 - C3 - C4 - N1  
100.46 ( 0.85) Ir1 - C3 - C4 - N1  
0.65 ( 1.15) C2 - C3 - C4 - C5  
-79.57 ( 0.98) Ir1 - C3 - C4 - C5  
-32.04 ( 1.07) N1 - C7 - C8 - C9  
37.72 ( 1.21) C7 - C8 - C9 - C10  
-28.92 ( 1.13) C8 - C9 - C10 - N1  
-176.62 ( 0.97) C16 - C11 - C12 - C13  
0.46 ( 1.06) C15 - C11 - C12 - C13  
61.43 ( 0.81) Ir1 - C11 - C12 - C13  
-1.95 ( 1.55) C16 - C11 - C12 - C17  
175.14 ( 0.92) C15 - C11 - C12 - C17  
-123.89 ( 0.97) Ir1 - C11 - C12 - C17  
121.94 ( 0.99) C16 - C11 - C12 - Ir1  
-60.97 ( 0.53) C15 - C11 - C12 - Ir1  
-1.17 ( 1.20) C11 - C12 - C13 - C14  
-175.65 ( 0.98) C17 - C12 - C13 - C14  
60.86 ( 0.69) Ir1 - C12 - C13 - C14  
177.81 ( 1.15) C11 - C12 - C13 - C18  
3.33 ( 2.00) C17 - C12 - C13 - C18  
-120.17 ( 1.35) Ir1 - C12 - C13 - C18  
-62.02 ( 0.71) C11 - C12 - C13 - Ir1  
123.49 ( 1.09) C17 - C12 - C13 - Ir1  
1.40 ( 1.11) C12 - C13 - C14 - C15  
-177.72 ( 1.01) C18 - C13 - C14 - C15  
63.16 ( 0.63) Ir1 - C13 - C14 - C15  
175.05 ( 1.05) C12 - C13 - C14 - C19  
-4.06 ( 1.72) C18 - C13 - C14 - C19  
-123.19 ( 1.15) Ir1 - C13 - C14 - C19  
-61.76 ( 0.71) C12 - C13 - C14 - Ir1

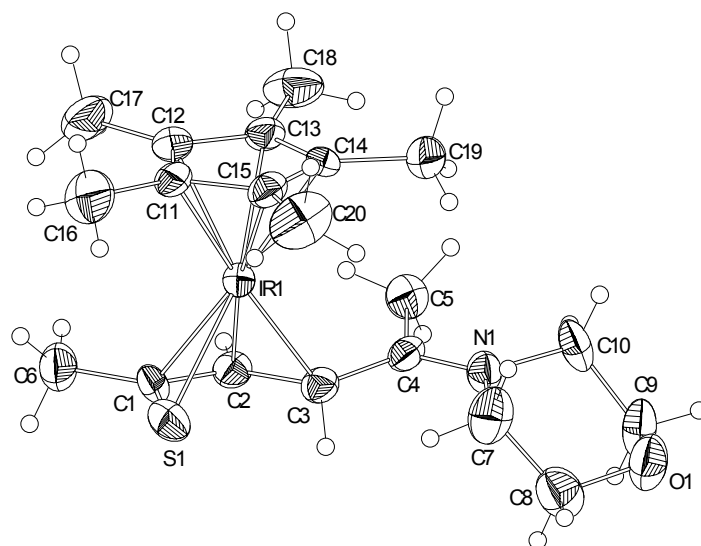
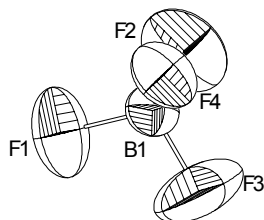
119.12 ( 1.15) C18 - C13 - C14 - Ir1  
-1.12 ( 0.95) C13 - C14 - C15 - C11  
-174.88 ( 0.94) C19 - C14 - C15 - C11  
61.33 ( 0.55) Ir1 - C14 - C15 - C11  
175.69 ( 0.92) C13 - C14 - C15 - C20  
1.92 ( 1.62) C19 - C14 - C15 - C20  
-121.86 ( 0.97) Ir1 - C14 - C15 - C20  
-62.45 ( 0.63) C13 - C14 - C15 - Ir1  
123.79 ( 1.09) C19 - C14 - C15 - Ir1  
0.47 ( 0.91) C12 - C11 - C15 - C14  
177.85 ( 0.83) C16 - C11 - C15 - C14  
-61.34 ( 0.62) Ir1 - C11 - C15 - C14  
-176.49 ( 0.87) C12 - C11 - C15 - C20  
0.89 ( 1.29) C16 - C11 - C15 - C20  
121.70 ( 0.86) Ir1 - C11 - C15 - C20  
61.81 ( 0.59) C12 - C11 - C15 - Ir1  
-120.81 ( 0.84) C16 - C11 - C15 - Ir1  
-126.50 ( 0.99) C3 - C2 - Ir1 - C1  
-82.33 ( 0.77) C1 - C2 - Ir1 - C12  
151.17 ( 0.71) C3 - C2 - Ir1 - C12  
169.81 ( 2.02) C1 - C2 - Ir1 - C14  
43.31 ( 2.44) C3 - C2 - Ir1 - C14  
-49.93 ( 1.32) C1 - C2 - Ir1 - C13  
-176.43 ( 0.99) C3 - C2 - Ir1 - C13  
-162.24 ( 0.61) C1 - C2 - Ir1 - C15  
71.26 ( 0.87) C3 - C2 - Ir1 - C15  
126.50 ( 0.99) C1 - C2 - Ir1 - C3  
-121.67 ( 0.68) C1 - C2 - Ir1 - C11  
111.83 ( 0.73) C3 - C2 - Ir1 - C11  
36.28 ( 0.56) C1 - C2 - Ir1 - S1  
-90.22 ( 0.67) C3 - C2 - Ir1 - S1  
-114.33 ( 1.29) C6 - C1 - Ir1 - C2  
126.54 ( 0.73) S1 - C1 - Ir1 - C2  
115.50 ( 0.71) C2 - C1 - Ir1 - C12  
1.17 ( 1.15) C6 - C1 - Ir1 - C12  
-117.96 ( 0.47) S1 - C1 - Ir1 - C12  
-175.52 ( 0.86) C2 - C1 - Ir1 - C14  
70.16 ( 1.48) C6 - C1 - Ir1 - C14  
-48.97 ( 1.04) S1 - C1 - Ir1 - C14  
154.25 ( 0.79) C2 - C1 - Ir1 - C13

39.92 ( 1.23) C6 - C1 - Ir1 - C13  
-79.20 ( 0.66) S1 - C1 - Ir1 - C13  
60.69 ( 1.47) C2 - C1 - Ir1 - C15  
-53.63 ( 1.78) C6 - C1 - Ir1 - C15  
-172.76 ( 1.11) S1 - C1 - Ir1 - C15  
-33.43 ( 0.68) C2 - C1 - Ir1 - C3  
-147.76 ( 1.15) C6 - C1 - Ir1 - C3  
93.11 ( 0.51) S1 - C1 - Ir1 - C3  
78.92 ( 0.75) C2 - C1 - Ir1 - C11  
-35.41 ( 1.18) C6 - C1 - Ir1 - C11  
-154.54 ( 0.43) S1 - C1 - Ir1 - C11  
-126.54 ( 0.73) C2 - C1 - Ir1 - S1  
119.13 ( 1.10) C6 - C1 - Ir1 - S1  
153.29 ( 0.71) C13 - C12 - Ir1 - C2  
-84.76 ( 0.71) C11 - C12 - Ir1 - C2  
32.70 ( 1.59) C17 - C12 - Ir1 - C2  
112.34 ( 0.77) C13 - C12 - Ir1 - C1  
-125.71 ( 0.68) C11 - C12 - Ir1 - C1  
-8.26 ( 1.57) C17 - C12 - Ir1 - C1  
-39.13 ( 0.80) C13 - C12 - Ir1 - C14  
82.81 ( 0.80) C11 - C12 - Ir1 - C14  
-159.73 ( 1.64) C17 - C12 - Ir1 - C14  
121.94 ( 1.02) C11 - C12 - Ir1 - C13  
-120.60 ( 1.77) C17 - C12 - Ir1 - C13  
-81.36 ( 0.83) C13 - C12 - Ir1 - C15  
40.59 ( 0.70) C11 - C12 - Ir1 - C15  
158.04 ( 1.63) C17 - C12 - Ir1 - C15  
-167.61 ( 0.69) C13 - C12 - Ir1 - C3  
-45.66 ( 1.03) C11 - C12 - Ir1 - C3  
71.79 ( 1.67) C17 - C12 - Ir1 - C3  
-121.94 ( 1.02) C13 - C12 - Ir1 - C11  
117.46 ( 1.74) C17 - C12 - Ir1 - C11  
64.54 ( 0.79) C13 - C12 - Ir1 - S1  
-173.52 ( 0.53) C11 - C12 - Ir1 - S1  
-56.06 ( 1.56) C17 - C12 - Ir1 - S1  
34.58 ( 2.51) C15 - C14 - Ir1 - C2  
149.93 ( 2.04) C13 - C14 - Ir1 - C2  
-87.28 ( 2.87) C19 - C14 - Ir1 - C2  
-159.39 ( 0.79) C15 - C14 - Ir1 - C1  
-44.04 ( 1.44) C13 - C14 - Ir1 - C1

78.75 ( 1.92) C19 - C14 - Ir1 - C1  
-79.21 ( 0.77) C15 - C14 - Ir1 - C12  
36.14 ( 0.88) C13 - C14 - Ir1 - C12  
158.92 ( 1.83) C19 - C14 - Ir1 - C12  
-115.35 ( 1.18) C15 - C14 - Ir1 - C13  
122.78 ( 2.10) C19 - C14 - Ir1 - C13  
115.35 ( 1.18) C13 - C14 - Ir1 - C15  
-121.87 ( 1.94) C19 - C14 - Ir1 - C15  
70.23 ( 0.94) C15 - C14 - Ir1 - C3  
-174.42 ( 0.85) C13 - C14 - Ir1 - C3  
-51.64 ( 1.91) C19 - C14 - Ir1 - C3  
-38.67 ( 0.67) C15 - C14 - Ir1 - C11  
76.67 ( 0.93) C13 - C14 - Ir1 - C11  
-160.54 ( 1.82) C19 - C14 - Ir1 - C11  
164.79 ( 0.56) C15 - C14 - Ir1 - S1  
-79.86 ( 0.93) C13 - C14 - Ir1 - S1  
42.92 ( 1.74) C19 - C14 - Ir1 - S1  
-50.80 ( 1.35) C12 - C13 - Ir1 - C2  
-168.28 ( 0.85) C14 - C13 - Ir1 - C2  
77.74 ( 2.13) C18 - C13 - Ir1 - C2  
-83.75 ( 0.85) C12 - C13 - Ir1 - C1  
158.77 ( 0.80) C14 - C13 - Ir1 - C1  
44.79 ( 1.82) C18 - C13 - Ir1 - C1  
-117.48 ( 1.21) C14 - C13 - Ir1 - C12  
128.54 ( 1.92) C18 - C13 - Ir1 - C12  
117.48 ( 1.21) C12 - C13 - Ir1 - C14  
-113.98 ( 2.08) C18 - C13 - Ir1 - C14  
79.53 ( 0.83) C12 - C13 - Ir1 - C15  
-37.95 ( 0.82) C14 - C13 - Ir1 - C15  
-151.93 ( 1.84) C18 - C13 - Ir1 - C15  
142.56 ( 2.61) C12 - C13 - Ir1 - C3  
25.08 ( 3.29) C14 - C13 - Ir1 - C3  
-88.90 ( 2.89) C18 - C13 - Ir1 - C3  
34.54 ( 0.70) C12 - C13 - Ir1 - C11  
-82.94 ( 0.90) C14 - C13 - Ir1 - C11  
163.08 ( 1.80) C18 - C13 - Ir1 - C11  
-128.95 ( 0.69) C12 - C13 - Ir1 - S1  
113.57 ( 0.84) C14 - C13 - Ir1 - S1  
-0.41 ( 1.69) C18 - C13 - Ir1 - S1  
-171.04 ( 0.67) C14 - C15 - Ir1 - C2

70.85 ( 0.75) C11 - C15 - Ir1 - C2  
-47.80 ( 1.73) C20 - C15 - Ir1 - C2  
141.31 ( 1.35) C14 - C15 - Ir1 - C1  
23.20 ( 1.57) C11 - C15 - Ir1 - C1  
-95.45 ( 1.85) C20 - C15 - Ir1 - C1  
81.13 ( 0.80) C14 - C15 - Ir1 - C12  
-36.98 ( 0.66) C11 - C15 - Ir1 - C12  
-155.63 ( 1.72) C20 - C15 - Ir1 - C12  
-118.11 ( 0.94) C11 - C15 - Ir1 - C14  
123.24 ( 1.85) C20 - C15 - Ir1 - C14  
40.03 ( 0.83) C14 - C15 - Ir1 - C13  
-78.09 ( 0.79) C11 - C15 - Ir1 - C13  
163.26 ( 1.74) C20 - C15 - Ir1 - C13  
-130.46 ( 0.77) C14 - C15 - Ir1 - C3  
111.42 ( 0.70) C11 - C15 - Ir1 - C3  
-7.23 ( 1.67) C20 - C15 - Ir1 - C3  
118.11 ( 0.94) C14 - C15 - Ir1 - C11  
-118.65 ( 1.81) C20 - C15 - Ir1 - C11  
-28.46 ( 0.96) C14 - C15 - Ir1 - S1  
-146.58 ( 0.60) C11 - C15 - Ir1 - S1  
94.77 ( 1.60) C20 - C15 - Ir1 - S1  
114.96 ( 1.29) C4 - C3 - Ir1 - C2  
147.62 ( 1.07) C4 - C3 - Ir1 - C1  
32.66 ( 0.64) C2 - C3 - Ir1 - C1  
56.66 ( 1.20) C4 - C3 - Ir1 - C12  
-58.29 ( 0.98) C2 - C3 - Ir1 - C12  
-54.39 ( 1.27) C4 - C3 - Ir1 - C14  
-169.35 ( 0.70) C2 - C3 - Ir1 - C14  
-75.45 ( 3.18) C4 - C3 - Ir1 - C13  
169.59 ( 2.63) C2 - C3 - Ir1 - C13  
-16.30 ( 1.09) C4 - C3 - Ir1 - C15  
-131.26 ( 0.70) C2 - C3 - Ir1 - C15  
27.32 ( 1.06) C4 - C3 - Ir1 - C11  
-87.64 ( 0.69) C2 - C3 - Ir1 - C11  
-166.20 ( 0.95) C4 - C3 - Ir1 - S1  
78.84 ( 0.61) C2 - C3 - Ir1 - S1  
113.01 ( 0.69) C12 - C11 - Ir1 - C2  
-14.67 ( 1.23) C16 - C11 - Ir1 - C2  
-131.58 ( 0.62) C15 - C11 - Ir1 - C2  
71.82 ( 0.80) C12 - C11 - Ir1 - C1

-55.86 ( 1.29) C16 - C11 - Ir1 - C1  
-172.77 ( 0.56) C15 - C11 - Ir1 - C1  
-127.68 ( 1.44) C16 - C11 - Ir1 - C12  
115.41 ( 0.96) C15 - C11 - Ir1 - C12  
-79.00 ( 0.79) C12 - C11 - Ir1 - C14  
153.32 ( 1.32) C16 - C11 - Ir1 - C14  
36.41 ( 0.65) C15 - C11 - Ir1 - C14  
-34.69 ( 0.71) C12 - C11 - Ir1 - C13  
-162.37 ( 1.33) C16 - C11 - Ir1 - C13  
80.72 ( 0.75) C15 - C11 - Ir1 - C13  
-115.41 ( 0.96) C12 - C11 - Ir1 - C15  
116.91 ( 1.41) C16 - C11 - Ir1 - C15  
156.22 ( 0.66) C12 - C11 - Ir1 - C3  
28.53 ( 1.28) C16 - C11 - Ir1 - C3  
-88.37 ( 0.70) C15 - C11 - Ir1 - C3  
15.16 ( 1.23) C12 - C11 - Ir1 - S1  
-112.52 ( 1.21) C16 - C11 - Ir1 - S1  
130.57 ( 0.77) C15 - C11 - Ir1 - S1  
57.91 ( 0.54) C2 - C1 - S1 - Ir1  
-121.70 ( 0.83) C6 - C1 - S1 - Ir1  
-31.70 ( 0.44) C2 - Ir1 - S1 - C1  
84.09 ( 0.53) C12 - Ir1 - S1 - C1  
157.61 ( 0.56) C14 - Ir1 - S1 - C1  
117.56 ( 0.58) C13 - Ir1 - S1 - C1  
176.27 ( 0.55) C15 - Ir1 - S1 - C1  
-72.62 ( 0.47) C3 - Ir1 - S1 - C1  
73.44 ( 0.88) C11 - Ir1 - S1 - C1  
-5.53 ( 1.01) C3 - C4 - N1 - C10  
174.49 ( 0.73) C5 - C4 - N1 - C10  
177.22 ( 0.72) C3 - C4 - N1 - C7  
-2.76 ( 1.07) C5 - C4 - N1 - C7  
-167.61 ( 0.73) C9 - C10 - N1 - C4  
9.96 ( 0.94) C9 - C10 - N1 - C7  
-169.21 ( 0.77) C8 - C7 - N1 - C4  
13.24 ( 0.97) C8 - C7 - N1 - C10

DEPARTAMENTO DE QUIMICA  
CinvestavORTEP drawing of Compound **14**Formula Minima :  $C_{20}H_{31}BF_4IrNO_5S$ 

Autores : Marisol Cervantez Vásquez y Ángeles Paz Sandoval

Tab. 1 Crystal data and data collection

Tab. 2 Solution and refinement

Tab. 3 Atomic coordinates and equivalent isotropic displacement parameters

Tab. 4 Bond lengths and angles

Tab. 5 Anisotropic displacement parameters

Tab. 6 Hydrogen coordinates and isotropic displacement parameters





**Table 2.** Solution and refinement

|                                    |   |
|------------------------------------|---|
| Structure solution program         | SHELXS-97(Sheldrick 1990)   |
| Solution                           | heavy-atom-method   |
| Refinement method                  | Full-matrix Least-Squares on F <sup>2</sup>                             |
| Extinction correction              | SHELXL  |
| Extinction coefficients            | 0.0038(9)   |
| Hydrogen atoms                     | constr  |
| Weighting scheme                   | $w^{-1} = \sigma^2 F_o^2 + (P)^2 + P$<br>where $P = (F_o^2 + 2F_c^2)/3$ |
| Data / restraints / parameters     | 5058 / 0 / 270  |
| Data-to-parameter-ratio            | 18.7 : 1 (15.6 : 1 [F>4 $\sigma$ (F)])                                  |
| Final R indices [F>4 $\sigma$ (F)] | R1 = 0.0560, wR2 = 0.1254   |
| R indices (all data)               | R1 = 0.0698, wR2 = 0.1313   |
| Goodness-of-Fit on F <sup>2</sup>  | 1.071   |
| Largest and mean $\Delta/\sigma$   | 0.001 0.000   |
| Largest difference peak            | 2.854 eÅ <sup>-3</sup>  |
| Largest difference hole            | -1.367 eÅ <sup>-3</sup>   |

**Refinement details :**

|                     |                            |
|---------------------|----------------------------|
| Program used        | SHELXL-97 (Sheldrick 1997) |
| CifRtf version used | 2.0                        |

**Table 3.** Atomic coordinates (  $\times 10^4$  ) and equivalent isotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ ) for compound **14**. U(eq) is defined as one third of the trace of the orthogonalized  $U_{ij}$  tensor

|       | x         | y         | z         | U(eq)    |
|-------|-----------|-----------|-----------|----------|
| C(1)  | 10879(10) | 8930(7)   | 8074(7)   | 35.7(19) |
| C(2)  | 9689(10)  | 9088(7)   | 8629(7)   | 32.6(17) |
| C(3)  | 9156(10)  | 8077(7)   | 9124(7)   | 31.3(17) |
| C(4)  | 7681(10)  | 7975(7)   | 9484(7)   | 30.4(17) |
| C(5)  | 6549(12)  | 8905(8)   | 9280(8)   | 45(2)    |
| C(6)  | 11486(12) | 9894(8)   | 7496(8)   | 49(2)    |
| C(7)  | 8153(14)  | 6025(8)   | 10054(9)  | 53(3)    |
| C(8)  | 8621(12)  | 5726(9)   | 11249(10) | 55(3)    |
| C(9)  | 6461(13)  | 6593(8)   | 11583(9)  | 51(2)    |
| C(10) | 5832(11)  | 6930(9)   | 10396(9)  | 47(2)    |
| C(11) | 7981(11)  | 6868(8)   | 5516(7)   | 36.2(19) |
| C(12) | 7184(10)  | 7884(7)   | 5542(7)   | 34.8(18) |
| C(13) | 5990(10)  | 7674(7)   | 6112(7)   | 33.6(18) |
| C(14) | 6030(9)   | 6515(7)   | 6431(7)   | 31.2(17) |
| C(15) | 7256(11)  | 5992(7)   | 6063(7)   | 33.4(18) |
| C(16) | 9284(14)  | 6671(10)  | 4940(9)   | 61(3)    |
| C(17) | 7506(14)  | 8967(9)   | 4995(9)   | 63(3)    |
| C(18) | 4725(13)  | 8482(10)  | 6190(9)   | 57(3)    |
| C(19) | 4909(12)  | 5923(9)   | 7007(8)   | 52(2)    |
| C(20) | 7660(15)  | 4780(8)   | 6186(9)   | 66(3)    |
| B(1)  | 2886(17)  | 7942(10)  | 2102(12)  | 59(3)    |
| F(1)  | 4114(12)  | 8665(7)   | 1833(9)   | 116(3)   |
| F(2)  | 1457(11)  | 8394(8)   | 1942(10)  | 130(4)   |
| F(3)  | 3635(17)  | 7776(10)  | 3185(9)   | 169(5)   |
| F(4)  | 2481(9)   | 6820(6)   | 1448(7)   | 86(2)    |
| Ir(1) | 8597.5(3) | 7601.1(2) | 7286.0(2) | 26.3(1)  |
| N(1)  | 7284(9)   | 7074(6)   | 9964(6)   | 34.4(15) |
| O(1)  | 7160(9)   | 5533(6)   | 11600(6)  | 55.5(18) |
| S(1)  | 11614(3)  | 7566(2)   | 8061(2)   | 44.1(5)  |

**Table 4.** Bond lengths [Å] and angles [°] for compound **14**

|                   |           |                   |           |
|-------------------|-----------|-------------------|-----------|
| C(1)-C(2)         | 1.404(11) | C(1)-C(6)         | 1.505(11) |
| C(1)-S(1)         | 1.755(9)  | C(1)-Ir(1)        | 2.158(8)  |
| C(2)-C(3)         | 1.457(11) | C(2)-Ir(1)        | 2.145(7)  |
| C(3)-C(4)         | 1.436(11) | C(3)-Ir(1)        | 2.191(8)  |
| C(4)-N(1)         | 1.308(10) | C(4)-C(5)         | 1.502(12) |
| C(7)-N(1)         | 1.479(11) | C(7)-C(8)         | 1.507(14) |
| C(8)-O(1)         | 1.423(11) | C(9)-O(1)         | 1.415(11) |
| C(9)-C(10)        | 1.517(13) | C(10)-N(1)        | 1.477(10) |
| C(11)-C(12)       | 1.418(12) | C(11)-C(15)       | 1.449(11) |
| C(11)-C(16)       | 1.514(12) | C(11)-Ir(1)       | 2.158(8)  |
| C(12)-C(13)       | 1.413(11) | C(12)-C(17)       | 1.515(12) |
| C(12)-Ir(1)       | 2.200(8)  | C(13)-C(14)       | 1.430(11) |
| C(13)-C(18)       | 1.512(11) | C(13)-Ir(1)       | 2.213(8)  |
| C(14)-C(15)       | 1.423(11) | C(14)-C(19)       | 1.494(11) |
| C(14)-Ir(1)       | 2.201(7)  | C(15)-C(20)       | 1.486(11) |
| C(15)-Ir(1)       | 2.191(8)  | B(1)-F(2)         | 1.325(14) |
| B(1)-F(3)         | 1.342(16) | B(1)-F(1)         | 1.385(15) |
| B(1)-F(4)         | 1.393(14) | Ir(1)-S(1)        | 2.393(2)  |
| C(2)-C(1)-C(6)    | 121.0(8)  | C(2)-C(1)-S(1)    | 117.8(6)  |
| C(6)-C(1)-S(1)    | 121.2(7)  | C(2)-C(1)-Ir(1)   | 70.4(4)   |
| C(6)-C(1)-Ir(1)   | 124.9(6)  | S(1)-C(1)-Ir(1)   | 74.6(3)   |
| C(1)-C(2)-C(3)    | 116.0(7)  | C(1)-C(2)-Ir(1)   | 71.5(5)   |
| C(3)-C(2)-Ir(1)   | 72.1(4)   | C(4)-C(3)-C(2)    | 123.2(7)  |
| C(4)-C(3)-Ir(1)   | 115.4(6)  | C(2)-C(3)-Ir(1)   | 68.7(4)   |
| N(1)-C(4)-C(3)    | 121.3(7)  | N(1)-C(4)-C(5)    | 119.3(7)  |
| C(3)-C(4)-C(5)    | 119.5(7)  | N(1)-C(7)-C(8)    | 110.5(8)  |
| O(1)-C(8)-C(7)    | 111.9(8)  | O(1)-C(9)-C(10)   | 112.4(8)  |
| N(1)-C(10)-C(9)   | 108.8(7)  | C(12)-C(11)-C(15) | 109.0(7)  |
| C(12)-C(11)-C(16) | 126.3(8)  | C(15)-C(11)-C(16) | 124.5(8)  |
| C(12)-C(11)-Ir(1) | 72.6(5)   | C(15)-C(11)-Ir(1) | 71.7(4)   |
| C(16)-C(11)-Ir(1) | 125.2(6)  | C(13)-C(12)-C(11) | 107.8(7)  |
| C(13)-C(12)-C(17) | 126.8(8)  | C(11)-C(12)-C(17) | 125.4(8)  |
| C(13)-C(12)-Ir(1) | 71.8(5)   | C(11)-C(12)-Ir(1) | 69.4(4)   |
| C(17)-C(12)-Ir(1) | 126.6(6)  | C(12)-C(13)-C(14) | 108.3(7)  |
| C(12)-C(13)-C(18) | 124.0(8)  | C(14)-C(13)-C(18) | 127.2(8)  |
| C(12)-C(13)-Ir(1) | 70.8(5)   | C(14)-C(13)-Ir(1) | 70.7(4)   |
| C(18)-C(13)-Ir(1) | 131.2(6)  | C(15)-C(14)-C(13) | 109.0(7)  |
| C(15)-C(14)-C(19) | 125.4(8)  | C(13)-C(14)-C(19) | 125.6(8)  |

|                   |           |                   |           |
|-------------------|-----------|-------------------|-----------|
| C(15)-C(14)-Ir(1) | 70.7(4)   | C(13)-C(14)-Ir(1) | 71.5(4)   |
| C(19)-C(14)-Ir(1) | 126.1(6)  | C(14)-C(15)-C(11) | 106.0(7)  |
| C(14)-C(15)-C(20) | 126.6(8)  | C(11)-C(15)-C(20) | 127.4(8)  |
| C(14)-C(15)-Ir(1) | 71.5(4)   | C(11)-C(15)-Ir(1) | 69.3(4)   |
| C(20)-C(15)-Ir(1) | 125.2(6)  | F(2)-B(1)-F(3)    | 113.1(13) |
| F(2)-B(1)-F(1)    | 112.2(10) | F(3)-B(1)-F(1)    | 106.3(11) |
| F(2)-B(1)-F(4)    | 108.9(10) | F(3)-B(1)-F(4)    | 105.7(10) |
| F(1)-B(1)-F(4)    | 110.5(11) | C(2)-Ir(1)-C(11)  | 148.8(3)  |
| C(2)-Ir(1)-C(1)   | 38.1(3)   | C(11)-Ir(1)-C(1)  | 118.0(3)  |
| C(2)-Ir(1)-C(15)  | 171.6(3)  | C(11)-Ir(1)-C(15) | 38.9(3)   |
| C(1)-Ir(1)-C(15)  | 149.3(3)  | C(2)-Ir(1)-C(3)   | 39.3(3)   |
| C(11)-Ir(1)-C(3)  | 171.6(3)  | C(1)-Ir(1)-C(3)   | 67.8(3)   |
| C(15)-Ir(1)-C(3)  | 133.2(3)  | C(2)-Ir(1)-C(14)  | 135.8(3)  |
| C(11)-Ir(1)-C(14) | 63.5(3)   | C(1)-Ir(1)-C(14)  | 169.9(3)  |
| C(15)-Ir(1)-C(14) | 37.8(3)   | C(3)-Ir(1)-C(14)  | 112.0(3)  |
| C(2)-Ir(1)-C(12)  | 120.0(3)  | C(11)-Ir(1)-C(12) | 38.0(3)   |
| C(1)-Ir(1)-C(12)  | 111.2(3)  | C(15)-Ir(1)-C(12) | 64.2(3)   |
| C(3)-Ir(1)-C(12)  | 147.9(3)  | C(14)-Ir(1)-C(12) | 63.1(3)   |
| C(2)-Ir(1)-C(13)  | 114.7(3)  | C(11)-Ir(1)-C(13) | 63.1(3)   |
| C(1)-Ir(1)-C(13)  | 132.7(3)  | C(15)-Ir(1)-C(13) | 63.6(3)   |
| C(3)-Ir(1)-C(13)  | 118.3(3)  | C(14)-Ir(1)-C(13) | 37.8(3)   |
| C(12)-Ir(1)-C(13) | 37.4(3)   | C(2)-Ir(1)-S(1)   | 73.2(2)   |
| C(11)-Ir(1)-S(1)  | 102.3(2)  | C(1)-Ir(1)-S(1)   | 45.0(2)   |
| C(15)-Ir(1)-S(1)  | 110.8(2)  | C(3)-Ir(1)-S(1)   | 77.4(2)   |
| C(14)-Ir(1)-S(1)  | 145.1(2)  | C(12)-Ir(1)-S(1)  | 126.1(2)  |
| C(13)-Ir(1)-S(1)  | 163.3(2)  | C(4)-N(1)-C(10)   | 124.4(7)  |
| C(4)-N(1)-C(7)    | 123.8(7)  | C(10)-N(1)-C(7)   | 111.6(7)  |
| C(9)-O(1)-C(8)    | 108.1(7)  | C(1)-S(1)-Ir(1)   | 60.4(3)   |

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**Table 5.** Anisotropic displacement parameters [ $\text{\AA}^2 \times 10^3$ ]

|       | U11      | U22      | U33      | U23      | U13      | U12    |
|-------|----------|----------|----------|----------|----------|--------|
| C(1)  | 25(4)    | 43(5)    | 36(5)    | 5(4)     | 11(4)    | -8(3)  |
| C(2)  | 36(4)    | 29(4)    | 25(4)    | -2(3)    | 7(3)     | -6(3)  |
| C(3)  | 31(4)    | 39(4)    | 24(4)    | 3(3)     | 9(3)     | 8(3)   |
| C(4)  | 36(4)    | 31(4)    | 22(4)    | 5(3)     | 8(3)     | 4(3)   |
| C(5)  | 41(5)    | 51(5)    | 45(6)    | 10(4)    | 17(4)    | 9(4)   |
| C(6)  | 50(6)    | 52(6)    | 42(6)    | 7(4)     | 20(5)    | -9(4)  |
| C(7)  | 70(7)    | 36(5)    | 64(7)    | 21(4)    | 33(6)    | 17(5)  |
| C(8)  | 46(6)    | 48(6)    | 74(8)    | 17(5)    | 21(5)    | 8(4)   |
| C(9)  | 62(6)    | 49(5)    | 62(7)    | 22(5)    | 43(5)    | 13(5)  |
| C(10) | 33(5)    | 50(5)    | 67(7)    | 13(5)    | 30(5)    | 1(4)   |
| C(11) | 33(4)    | 56(5)    | 19(4)    | 4(4)     | 10(3)    | 5(4)   |
| C(12) | 30(4)    | 41(5)    | 33(5)    | 13(4)    | 6(4)     | 10(3)  |
| C(13) | 27(4)    | 45(5)    | 31(5)    | 13(4)    | 7(3)     | 15(3)  |
| C(14) | 20(4)    | 43(5)    | 24(4)    | 4(3)     | 3(3)     | -5(3)  |
| C(15) | 39(5)    | 32(4)    | 24(4)    | 4(3)     | 6(4)     | 2(3)   |
| C(16) | 57(7)    | 91(8)    | 51(7)    | 10(6)    | 34(6)    | 29(6)  |
| C(17) | 70(7)    | 59(6)    | 49(7)    | 30(5)    | 5(5)     | 2(5)   |
| C(18) | 49(6)    | 77(7)    | 42(6)    | 1(5)     | 6(5)     | 31(5)  |
| C(19) | 45(6)    | 64(6)    | 40(6)    | 15(5)    | 9(4)     | -12(4) |
| C(20) | 100(9)   | 28(5)    | 63(7)    | 3(4)     | 18(7)    | 15(5)  |
| B(1)  | 59(8)    | 41(6)    | 68(9)    | 9(6)     | 14(7)    | 2(6)   |
| F(1)  | 132(7)   | 64(5)    | 175(9)   | 24(5)    | 88(7)    | -4(4)  |
| F(2)  | 106(7)   | 95(6)    | 199(11)  | 12(6)    | 58(7)    | 51(5)  |
| F(3)  | 212(12)  | 141(9)   | 75(7)    | 39(6)    | -35(7)   | -48(8) |
| F(4)  | 76(5)    | 64(4)    | 111(6)   | -9(4)    | 32(4)    | 2(3)   |
| Ir(1) | 24.9(2)  | 28.3(2)  | 26.4(2)  | 6.9(1)   | 9.4(1)   | 3.5(1) |
| N(1)  | 33(4)    | 39(4)    | 36(4)    | 9(3)     | 18(3)    | 7(3)   |
| O(1)  | 63(4)    | 57(4)    | 65(5)    | 31(3)    | 37(4)    | 17(3)  |
| S(1)  | 27.4(11) | 54.2(14) | 51.9(14) | 11.0(10) | 14.2(10) | 7.5(9) |

**Table 6.** Hydrogen coordinates (  $\times 10^4$  ) and isotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ ) for compound **14**

|        | x     | y     | z     | U(eq) |
|--------|-------|-------|-------|-------|
| H(2)   | 9175  | 9808  | 8628  | 39    |
| H(3)   | 10113 | 7740  | 9600  | 38    |
| H(5A)  | 6821  | 9446  | 9975  | 67    |
| H(5B)  | 5366  | 8530  | 9036  | 67    |
| H(5C)  | 6737  | 9332  | 8699  | 67    |
| H(6A)  | 12608 | 10302 | 7975  | 73    |
| H(6B)  | 10697 | 10447 | 7363  | 73    |
| H(6C)  | 11540 | 9549  | 6780  | 73    |
| H(7A)  | 9185  | 6188  | 9859  | 63    |
| H(7B)  | 7396  | 5352  | 9518  | 63    |
| H(8A)  | 9142  | 5016  | 11283 | 66    |
| H(8B)  | 9462  | 6370  | 11773 | 66    |
| H(9A)  | 7331  | 7234  | 12090 | 61    |
| H(9B)  | 5509  | 6493  | 11869 | 61    |
| H(10A) | 4915  | 6316  | 9890  | 57    |
| H(10B) | 5384  | 7667  | 10423 | 57    |
| H(16A) | 10083 | 6216  | 5371  | 92    |
| H(16B) | 9895  | 7425  | 4903  | 92    |
| H(16C) | 8702  | 6248  | 4181  | 92    |
| H(17A) | 8667  | 9080  | 4996  | 94    |
| H(17B) | 7332  | 9655  | 5419  | 94    |
| H(17C) | 6723  | 8853  | 4222  | 94    |
| H(18A) | 5308  | 9291  | 6400  | 86    |
| H(18B) | 4239  | 8275  | 6757  | 86    |
| H(18C) | 3824  | 8393  | 5463  | 86    |
| H(19A) | 4655  | 6512  | 7501  | 78    |
| H(19B) | 5494  | 5372  | 7450  | 78    |
| H(19C) | 3857  | 5506  | 6442  | 78    |
| H(20A) | 7585  | 4577  | 6895  | 99    |
| H(20B) | 8803  | 4760  | 6178  | 99    |
| H(20C) | 6852  | 4219  | 5564  | 99    |

**Table 7.** Crystal data and data collection parameters:

| Compound                            | Compound <b>14</b>  |
|-------------------------------------|---|
| Chem. formula                       | C <sub>20</sub> H <sub>31</sub> B F <sub>4</sub> Ir N O S |
| Form. wght.                         | 612.53  |
| Cryst. size [mm]                    | 0.10x0.23x0.25  |
| Cryst. system                       | Triclinic   |
| Space group                         | Triclinic   |
| a, [Å]                              | 8.3432(2)   |
| b, [Å]                              | 11.4873(3)  |
| c, [Å]                              | 12.5263(4)  |
| α, [°]                              | 95.8270(10)   |
| β, [°]                              | 108.9380(9)   |
| γ, [°]                              | 97.3639(10)   |
| V, [Å <sup>3</sup> ]                | 1113.00(5)  |
| Z                                   | 2   |
| ρ(calcd.), [Mg/m <sup>3</sup> ]     | 1.828   |
| μ [mm <sup>-1</sup> ]               | 6.136   |
| F(000)                              | 600   |
| Index range                         | -10≤h≤10-12≤k≤14-16≤l≤14                                  |
| 2 θ [°]                             | 55.08   |
| Temp, [K]                           | 298(2)  |
| Refl. collected                     | 10670   |
| Refl. unique                        | 5058  |
| Refl. observed (4σ)                 | 4204  |
| R (int.)                            | 0.0729  |
| No. variables                       | 270   |
| Weighting scheme <sup>1</sup> x/y   | /   |
| GOOF                                | 1.071   |
| Final R (4σ)                        | 0.0560  |
| Final wR2                           | 0.1254  |
| Larg. res. peak [e/Å <sup>3</sup> ] | 2.854   |

$$^1 w^{-1} = \sigma^2 F_o^2 + (xP)^2 + yP; P = (F_o^2 + 2F_c^2)/3$$



**Selected torsion angles of compound 14**

177.99 ( 0.74) C6 - C1 - C2 - C3  
-1.19 ( 1.02) S1 - C1 - C2 - C3  
58.40 ( 0.62) Ir1 - C1 - C2 - C3  
119.60 ( 0.79) C6 - C1 - C2 - Ir1  
-59.59 ( 0.52) S1 - C1 - C2 - Ir1  
-165.17 ( 0.75) C1 - C2 - C3 - C4  
-107.09 ( 0.76) Ir1 - C2 - C3 - C4  
-58.08 ( 0.63) C1 - C2 - C3 - Ir1  
-177.35 ( 0.75) C2 - C3 - C4 - N1  
102.52 ( 0.81) Ir1 - C3 - C4 - N1  
4.10 ( 1.19) C2 - C3 - C4 - C5  
-76.04 ( 0.84) Ir1 - C3 - C4 - C5  
56.41 ( 1.07) N1 - C7 - C8 - O1  
-58.47 ( 1.00) O1 - C9 - C10 - N1  
1.01 ( 0.96) C15 - C11 - C12 - C13  
176.83 ( 0.86) C16 - C11 - C12 - C13  
-61.92 ( 0.61) Ir1 - C11 - C12 - C13  
-176.08 ( 0.83) C15 - C11 - C12 - C17  
-0.26 ( 1.45) C16 - C11 - C12 - C17  
120.99 ( 0.88) Ir1 - C11 - C12 - C17  
62.93 ( 0.56) C15 - C11 - C12 - Ir1  
-121.25 ( 0.93) C16 - C11 - C12 - Ir1  
-0.69 ( 0.96) C11 - C12 - C13 - C14  
176.35 ( 0.85) C17 - C12 - C13 - C14  
-61.09 ( 0.56) Ir1 - C12 - C13 - C14  
-172.25 ( 0.80) C11 - C12 - C13 - C18  
4.78 ( 1.43) C17 - C12 - C13 - C18  
127.34 ( 0.86) Ir1 - C12 - C13 - C18  
60.40 ( 0.58) C11 - C12 - C13 - Ir1  
-122.56 ( 0.92) C17 - C12 - C13 - Ir1  
0.11 ( 0.93) C12 - C13 - C14 - C15  
171.33 ( 0.81) C18 - C13 - C14 - C15  
-61.09 ( 0.54) Ir1 - C13 - C14 - C15  
-177.19 ( 0.80) C12 - C13 - C14 - C19  
-5.97 ( 1.40) C18 - C13 - C14 - C19  
121.61 ( 0.84) Ir1 - C13 - C14 - C19  
61.20 ( 0.59) C12 - C13 - C14 - Ir1  
-127.58 ( 0.89) C18 - C13 - C14 - Ir1

0.50 ( 0.88) C13 - C14 - C15 - C11  
177.80 ( 0.78) C19 - C14 - C15 - C11  
-61.12 ( 0.53) Ir1 - C14 - C15 - C11  
-177.80 ( 0.86) C13 - C14 - C15 - C20  
-0.50 ( 1.37) C19 - C14 - C15 - C20  
120.58 ( 0.90) Ir1 - C14 - C15 - C20  
61.62 ( 0.56) C13 - C14 - C15 - Ir1  
-121.08 ( 0.82) C19 - C14 - C15 - Ir1  
-0.93 ( 0.90) C12 - C11 - C15 - C14  
-176.85 ( 0.83) C16 - C11 - C15 - C14  
62.56 ( 0.53) Ir1 - C11 - C15 - C14  
177.35 ( 0.88) C12 - C11 - C15 - C20  
1.43 ( 1.44) C16 - C11 - C15 - C20  
-119.16 ( 0.91) Ir1 - C11 - C15 - C20  
-63.49 ( 0.58) C12 - C11 - C15 - Ir1  
120.59 ( 0.88) C16 - C11 - C15 - Ir1  
-50.15 ( 0.83) C1 - C2 - Ir1 - C11  
-176.55 ( 0.48) C3 - C2 - Ir1 - C11  
-126.40 ( 0.73) C3 - C2 - Ir1 - C1  
154.85 ( 1.76) C1 - C2 - Ir1 - C15  
28.45 ( 2.09) C3 - C2 - Ir1 - C15  
126.40 ( 0.73) C1 - C2 - Ir1 - C3  
-167.71 ( 0.45) C1 - C2 - Ir1 - C14  
65.90 ( 0.63) C3 - C2 - Ir1 - C14  
-86.61 ( 0.57) C1 - C2 - Ir1 - C12  
146.99 ( 0.46) C3 - C2 - Ir1 - C12  
-128.47 ( 0.51) C1 - C2 - Ir1 - C13  
105.13 ( 0.49) C3 - C2 - Ir1 - C13  
35.76 ( 0.46) C1 - C2 - Ir1 - S1  
-90.64 ( 0.44) C3 - C2 - Ir1 - S1  
-56.78 ( 0.80) C12 - C11 - Ir1 - C2  
-174.35 ( 0.51) C15 - C11 - Ir1 - C2  
65.85 ( 1.07) C16 - C11 - Ir1 - C2  
-89.21 ( 0.54) C12 - C11 - Ir1 - C1  
153.21 ( 0.48) C15 - C11 - Ir1 - C1  
33.41 ( 0.94) C16 - C11 - Ir1 - C1  
117.58 ( 0.69) C12 - C11 - Ir1 - C15  
-119.80 ( 1.02) C16 - C11 - Ir1 - C15  
138.42 ( 1.73) C12 - C11 - Ir1 - C3  
20.85 ( 2.01) C15 - C11 - Ir1 - C3

-98.95 ( 1.94) C16 - C11 - Ir1 - C3  
79.52 ( 0.52) C12 - C11 - Ir1 - C14  
-38.06 ( 0.46) C15 - C11 - Ir1 - C14  
-157.86 ( 0.93) C16 - C11 - Ir1 - C14  
-117.58 ( 0.69) C15 - C11 - Ir1 - C12  
122.62 ( 1.03) C16 - C11 - Ir1 - C12  
37.00 ( 0.47) C12 - C11 - Ir1 - C13  
-80.58 ( 0.51) C15 - C11 - Ir1 - C13  
159.62 ( 0.94) C16 - C11 - Ir1 - C13  
-134.47 ( 0.45) C12 - C11 - Ir1 - S1  
107.96 ( 0.44) C15 - C11 - Ir1 - S1  
-11.84 ( 0.85) C16 - C11 - Ir1 - S1  
-114.75 ( 0.96) C6 - C1 - Ir1 - C2  
127.71 ( 0.62) S1 - C1 - Ir1 - C2  
153.28 ( 0.49) C2 - C1 - Ir1 - C11  
38.52 ( 0.86) C6 - C1 - Ir1 - C11  
-79.01 ( 0.39) S1 - C1 - Ir1 - C11  
-173.00 ( 0.50) C2 - C1 - Ir1 - C15  
72.25 ( 0.99) C6 - C1 - Ir1 - C15  
-45.29 ( 0.70) S1 - C1 - Ir1 - C15  
-33.38 ( 0.49) C2 - C1 - Ir1 - C3  
-148.13 ( 0.83) C6 - C1 - Ir1 - C3  
94.33 ( 0.35) S1 - C1 - Ir1 - C3  
57.67 ( 1.76) C2 - C1 - Ir1 - C14  
-57.08 ( 1.93) C6 - C1 - Ir1 - C14  
-174.62 ( 1.44) S1 - C1 - Ir1 - C14  
112.00 ( 0.52) C2 - C1 - Ir1 - C12  
-2.76 ( 0.84) C6 - C1 - Ir1 - C12  
-120.29 ( 0.32) S1 - C1 - Ir1 - C12  
75.27 ( 0.62) C2 - C1 - Ir1 - C13  
-39.48 ( 0.93) C6 - C1 - Ir1 - C13  
-157.02 ( 0.33) S1 - C1 - Ir1 - C13  
-127.71 ( 0.62) C2 - C1 - Ir1 - S1  
117.54 ( 0.85) C6 - C1 - Ir1 - S1  
43.75 ( 2.04) C14 - C15 - Ir1 - C2  
159.64 ( 1.79) C11 - C15 - Ir1 - C2  
-78.49 ( 2.15) C20 - C15 - Ir1 - C2  
-115.88 ( 0.66) C14 - C15 - Ir1 - C11  
121.87 ( 1.02) C20 - C15 - Ir1 - C11  
-167.19 ( 0.53) C14 - C15 - Ir1 - C1

-51.31 ( 0.79) C11 - C15 - Ir1 - C1  
70.56 ( 1.04) C20 - C15 - Ir1 - C1  
68.18 ( 0.59) C14 - C15 - Ir1 - C3  
-175.93 ( 0.40) C11 - C15 - Ir1 - C3  
-54.06 ( 0.97) C20 - C15 - Ir1 - C3  
115.88 ( 0.66) C11 - C15 - Ir1 - C14  
-122.25 ( 1.01) C20 - C15 - Ir1 - C14  
-78.63 ( 0.50) C14 - C15 - Ir1 - C12  
37.25 ( 0.47) C11 - C15 - Ir1 - C12  
159.12 ( 0.92) C20 - C15 - Ir1 - C12  
-36.87 ( 0.46) C14 - C15 - Ir1 - C13  
79.01 ( 0.50) C11 - C15 - Ir1 - C13  
-159.12 ( 0.92) C20 - C15 - Ir1 - C13  
160.31 ( 0.40) C14 - C15 - Ir1 - S1  
-83.81 ( 0.46) C11 - C15 - Ir1 - S1  
38.06 ( 0.86) C20 - C15 - Ir1 - S1  
117.70 ( 0.78) C4 - C3 - Ir1 - C2  
-74.67 ( 1.95) C4 - C3 - Ir1 - C11  
167.63 ( 1.71) C2 - C3 - Ir1 - C11  
150.14 ( 0.67) C4 - C3 - Ir1 - C1  
32.44 ( 0.48) C2 - C3 - Ir1 - C1  
-56.81 ( 0.73) C4 - C3 - Ir1 - C15  
-174.52 ( 0.42) C2 - C3 - Ir1 - C15  
-18.94 ( 0.67) C4 - C3 - Ir1 - C14  
-136.64 ( 0.48) C2 - C3 - Ir1 - C14  
55.18 ( 0.88) C4 - C3 - Ir1 - C12  
-62.52 ( 0.75) C2 - C3 - Ir1 - C12  
22.44 ( 0.69) C4 - C3 - Ir1 - C13  
-95.27 ( 0.52) C2 - C3 - Ir1 - C13  
-163.62 ( 0.61) C4 - C3 - Ir1 - S1  
78.67 ( 0.44) C2 - C3 - Ir1 - S1  
-171.66 ( 0.43) C15 - C14 - Ir1 - C2  
69.66 ( 0.62) C13 - C14 - Ir1 - C2  
-51.37 ( 0.93) C19 - C14 - Ir1 - C2  
39.17 ( 0.46) C15 - C14 - Ir1 - C11  
-79.51 ( 0.52) C13 - C14 - Ir1 - C11  
159.47 ( 0.88) C19 - C14 - Ir1 - C11  
139.95 ( 1.52) C15 - C14 - Ir1 - C1  
21.27 ( 1.79) C13 - C14 - Ir1 - C1  
-99.76 ( 1.68) C19 - C14 - Ir1 - C1

-118.68 ( 0.68) C13 - C14 - Ir1 - C15  
120.29 ( 0.96) C19 - C14 - Ir1 - C15  
-133.12 ( 0.47) C15 - C14 - Ir1 - C3  
108.20 ( 0.49) C13 - C14 - Ir1 - C3  
-12.83 ( 0.85) C19 - C14 - Ir1 - C3  
81.86 ( 0.51) C15 - C14 - Ir1 - C12  
-36.82 ( 0.46) C13 - C14 - Ir1 - C12  
-157.85 ( 0.88) C19 - C14 - Ir1 - C12  
118.68 ( 0.68) C15 - C14 - Ir1 - C13  
-121.03 ( 0.97) C19 - C14 - Ir1 - C13  
-33.39 ( 0.66) C15 - C14 - Ir1 - S1  
-152.07 ( 0.40) C13 - C14 - Ir1 - S1  
86.90 ( 0.84) C19 - C14 - Ir1 - S1  
-92.17 ( 0.54) C13 - C12 - Ir1 - C2  
150.01 ( 0.49) C11 - C12 - Ir1 - C2  
30.57 ( 0.92) C17 - C12 - Ir1 - C2  
117.82 ( 0.71) C13 - C12 - Ir1 - C11  
-119.44 ( 1.02) C17 - C12 - Ir1 - C11  
-133.54 ( 0.50) C13 - C12 - Ir1 - C1  
108.64 ( 0.52) C11 - C12 - Ir1 - C1  
-10.80 ( 0.91) C17 - C12 - Ir1 - C1  
79.62 ( 0.52) C13 - C12 - Ir1 - C15  
-38.19 ( 0.48) C11 - C12 - Ir1 - C15  
-157.64 ( 0.92) C17 - C12 - Ir1 - C15  
-51.74 ( 0.78) C13 - C12 - Ir1 - C3  
-169.56 ( 0.51) C11 - C12 - Ir1 - C3  
71.00 ( 1.04) C17 - C12 - Ir1 - C3  
37.25 ( 0.47) C13 - C12 - Ir1 - C14  
-80.57 ( 0.53) C11 - C12 - Ir1 - C14  
159.99 ( 0.93) C17 - C12 - Ir1 - C14  
-117.82 ( 0.71) C11 - C12 - Ir1 - C13  
122.74 ( 1.02) C17 - C12 - Ir1 - C13  
177.43 ( 0.38) C13 - C12 - Ir1 - S1  
59.61 ( 0.54) C11 - C12 - Ir1 - S1  
-59.83 ( 0.88) C17 - C12 - Ir1 - S1  
107.79 ( 0.51) C12 - C13 - Ir1 - C2  
-133.97 ( 0.48) C14 - C13 - Ir1 - C2  
-11.04 ( 0.95) C18 - C13 - Ir1 - C2  
-37.59 ( 0.49) C12 - C13 - Ir1 - C11  
80.65 ( 0.52) C14 - C13 - Ir1 - C11

-156.42 ( 0.96) C18 - C13 - Ir1 - C11  
66.73 ( 0.63) C12 - C13 - Ir1 - C1  
-175.03 ( 0.43) C14 - C13 - Ir1 - C1  
-52.10 ( 0.99) C18 - C13 - Ir1 - C1  
-81.35 ( 0.52) C12 - C13 - Ir1 - C15  
36.89 ( 0.47) C14 - C13 - Ir1 - C15  
159.82 ( 0.95) C18 - C13 - Ir1 - C15  
151.71 ( 0.47) C12 - C13 - Ir1 - C3  
-90.05 ( 0.52) C14 - C13 - Ir1 - C3  
32.88 ( 0.94) C18 - C13 - Ir1 - C3  
-118.24 ( 0.69) C12 - C13 - Ir1 - C14  
122.93 ( 1.04) C18 - C13 - Ir1 - C14  
118.24 ( 0.69) C14 - C13 - Ir1 - C12  
-118.83 ( 1.04) C18 - C13 - Ir1 - C12  
-7.26 ( 1.07) C12 - C13 - Ir1 - S1  
110.98 ( 0.78) C14 - C13 - Ir1 - S1  
-126.09 ( 0.86) C18 - C13 - Ir1 - S1  
177.55 ( 0.79) C3 - C4 - N1 - C10  
-3.89 ( 1.28) C5 - C4 - N1 - C10  
-7.72 ( 1.28) C3 - C4 - N1 - C7  
170.85 ( 0.83) C5 - C4 - N1 - C7  
-132.73 ( 0.87) C9 - C10 - N1 - C4  
51.97 ( 1.00) C9 - C10 - N1 - C7  
132.89 ( 0.88) C8 - C7 - N1 - C4  
-51.78 ( 1.06) C8 - C7 - N1 - C10  
62.55 ( 1.06) C10 - C9 - O1 - C8  
-61.00 ( 1.05) C7 - C8 - O1 - C9  
57.43 ( 0.58) C2 - C1 - S1 - Ir1  
-121.75 ( 0.78) C6 - C1 - S1 - Ir1  
-30.67 ( 0.40) C2 - Ir1 - S1 - C1  
117.46 ( 0.41) C11 - Ir1 - S1 - C1  
157.19 ( 0.40) C15 - Ir1 - S1 - C1  
-71.08 ( 0.39) C3 - Ir1 - S1 - C1  
178.35 ( 0.44) C14 - Ir1 - S1 - C1  
84.58 ( 0.43) C12 - Ir1 - S1 - C1  
90.02 ( 0.83) C13 - Ir1 - S1 - C1