

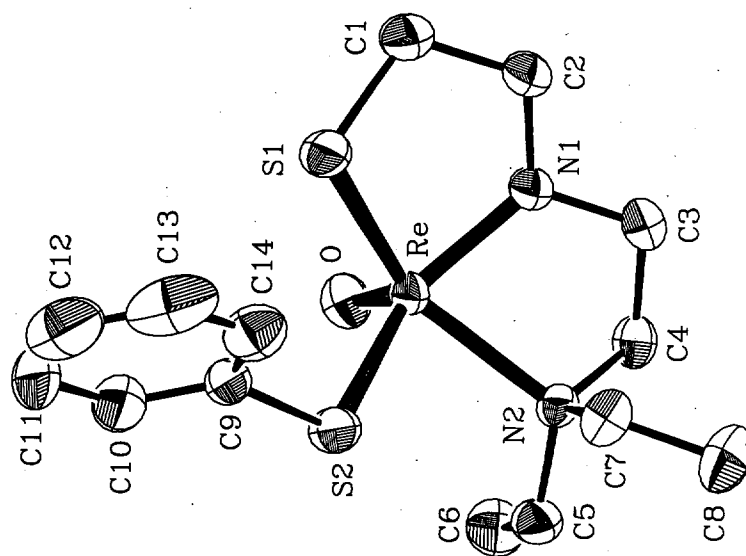


Table 1. Summary of Crystal, Intensity Collection and Refinement Data

|                                                                |                                                              |
|----------------------------------------------------------------|--------------------------------------------------------------|
| Empirical formula                                              | $C_{14}H_{23}N_2OReS_2$                                      |
| Formula weight                                                 | 485.66                                                       |
| Temperature, K                                                 | 298                                                          |
| Wavelength                                                     | Mo K $\alpha$ 0.71070                                        |
| Space group                                                    | P2 <sub>1</sub> /n                                           |
| a (Å)                                                          | 6.888(2)                                                     |
| b (Å)                                                          | 28.080(8)                                                    |
| c (Å)                                                          | 8.694(2)                                                     |
| $\beta$ , deg                                                  | 100.064(7)                                                   |
| V, (Å <sup>3</sup> )                                           | 1655.6(8)                                                    |
| Z                                                              | 4                                                            |
| Dcalcd/Dmeasd (Mg m <sup>-3</sup> )                            | 1.948/1.93                                                   |
| abs coeff(_O5), mm <sup>-1</sup>                               | 7.589                                                        |
| Abs.correction mode Tmin/Tmax                                  | 0.41/1.00                                                    |
| Scan mode / speed (deg/min)                                    | $\theta$ -2 $\theta$ /4.0                                    |
| Scan range (2 $\theta$ deg)                                    | 2.5 + $\alpha_1\alpha_2$ separation                          |
| range(deg)                                                     | 2.49 to 26.50                                                |
| Reflections collected                                          | 3704                                                         |
| Independent reflections                                        | 3426 [R(int) = 0.0232]                                       |
| Range of h,k,l                                                 | -8 $\rightarrow$ 0, 0 $\rightarrow$ 35, -10 $\rightarrow$ 10 |
| F(000)                                                         | 944                                                          |
| $[\Delta/\sigma]_{\max}$                                       | 0.173                                                        |
| $W^{(1)}$                                                      | a=0.0757 b=3.6391                                            |
| $[\Delta\rho]_{\max}/[\Delta\rho]_{\min}$ (e/ Å <sup>3</sup> ) | 0.735 and -0.625                                             |
| Refinement method                                              | Full-matrix least-squares on F <sup>2</sup>                  |
| Data / restraints / parameters                                 | 3425 / 0 / 237                                               |
| Goodness-of-fit on F <sup>2</sup>                              | 1.121                                                        |
| R indices [3106 refs I>2 $\sigma$ (I)] <sup>(2)</sup>          | R1 = 0.0408, wR2 = 0.1138                                    |
| R indices (all data)                                           | R1 = 0.0460, wR2 = 0.1204                                    |

(1)  $W=1/[\sigma^2(F_o^2)+(a*P)^2+b*P]$  and  $P=(\text{Max}(F_o^2,0)+2*F_c^2)/3$ (2) (2) R1 based on F's, wR2 based on F<sup>2</sup> Extinction coefficient 0.0107(7)

Table 2. Positional ( $\times 10^4$ ) and equivalent thermal parameters ( $\times 10^3$ ) of the non-H atoms.E.s.d.'s in parentheses. U(eq) is defined as one third of the trace of the orthogonalized  $U_{ij}$  tensor.

|       | x         | y       | z         | U(eq) |
|-------|-----------|---------|-----------|-------|
| Re    | 445(1)    | 851(1)  | 2891(1)   | 28(1) |
| S(1)  | -482(3)   | 893(1)  | 249(2)    | 43(1) |
| S(2)  | 2029(3)   | 1590(1) | 2969(2)   | 47(1) |
| O     | 2175(7)   | 422(2)  | 3280(6)   | 42(1) |
| N(1)  | -2173(8)  | 586(2)  | 2781(6)   | 34(1) |
| N(2)  | -343(8)   | 1072(2) | 5158(6)   | 30(1) |
| C(1)  | -2382(11) | 430(3)  | 4(8)      | 46(2) |
| C(2)  | -3549(11) | 483(3)  | 1321(8)   | 41(2) |
| C(3)  | -3097(11) | 554(3)  | 4163(9)   | 42(2) |
| C(4)  | -1520(10) | 654(2)  | 5556(7)   | 37(1) |
| C(5)  | 1377(11)  | 1179(3) | 6452(8)   | 42(2) |
| C(6)  | 2770(13)  | 766(3)  | 6955(10)  | 53(2) |
| C(7)  | -1620(12) | 1513(3) | 4923(8)   | 40(2) |
| C(8)  | -2815(14) | 1616(3) | 6210(10)  | 57(2) |
| C(9)  | 2346(11)  | 1821(2) | 1103(8)   | 39(2) |
| C(10) | 4151(14)  | 1792(3) | 659(11)   | 56(2) |
| C(11) | 4459(20)  | 2032(5) | -717(13)  | 83(4) |
| C(12) | 2943(25)  | 2271(4) | -1613(13) | 83(4) |
| C(13) | 1174(21)  | 2291(3) | -1173(11) | 77(4) |
| C(14) | 842(14)   | 2071(3) | 185(10)   | 54(2) |

Table 5. Bond lengths [Å] and angles [deg]

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|              |           |
|--------------|-----------|
| Re-O         | 1.687(5)  |
| Re-N(1)      | 1.938(5)  |
| Re-N(2)      | 2.223(5)  |
| Re-S(1)      | 2.277(2)  |
| Re-S(2)      | 2.339(2)  |
| S(1)-C(1)    | 1.831(8)  |
| S(2)-C(9)    | 1.796(7)  |
| N(1)-C(3)    | 1.457(9)  |
| N(1)-C(2)    | 1.474(9)  |
| N(2)-C(4)    | 1.502(8)  |
| N(2)-C(7)    | 1.511(9)  |
| N(2)-C(5)    | 1.515(9)  |
| C(1)-C(2)    | 1.516(10) |
| C(3)-C(4)    | 1.505(10) |
| C(5)-C(6)    | 1.520(11) |
| C(7)-C(8)    | 1.529(10) |
| C(9)-C(10)   | 1.367(12) |
| C(9)-C(14)   | 1.384(12) |
| C(10)-C(11)  | 1.420(14) |
| C(11)-C(12)  | 1.37(2)   |
| C(12)-C(13)  | 1.34(2)   |
| C(13)-C(14)  | 1.385(13) |
| O-Re-N(1)    | 110.7(2)  |
| O-Re-N(2)    | 107.2(2)  |
| N(1)-Re-N(2) | 77.4(2)   |
| O-Re-S(1)    | 107.6(2)  |
| N(1)-Re-S(1) | 82.9(2)   |
| N(2)-Re-S(1) | 144.2(2)  |
| O-Re-S(2)    | 108.6(2)  |
| N(1)-Re-S(2) | 140.1(2)  |

|                   |           |
|-------------------|-----------|
| N(2)-Re-S(2)      | 84.92(14) |
| S(1)-Re-S(2)      | 91.74(6)  |
| C(1)-S(1)-Re      | 98.6(2)   |
| C(9)-S(2)-Re      | 115.1(2)  |
| C(3)-N(1)-C(2)    | 113.0(5)  |
| C(3)-N(1)-Re      | 121.3(4)  |
| C(2)-N(1)-Re      | 124.8(4)  |
| C(4)-N(2)-C(7)    | 110.3(5)  |
| C(4)-N(2)-C(5)    | 111.6(5)  |
| C(7)-N(2)-C(5)    | 107.4(5)  |
| C(4)-N(2)-Re      | 102.2(4)  |
| C(7)-N(2)-Re      | 109.6(4)  |
| C(5)-N(2)-Re      | 115.7(4)  |
| C(2)-C(1)-S(1)    | 107.8(5)  |
| N(1)-C(2)-C(1)    | 108.9(6)  |
| N(1)-C(3)-C(4)    | 107.1(5)  |
| N(2)-C(4)-C(3)    | 107.6(5)  |
| N(2)-C(5)-C(6)    | 115.8(6)  |
| N(2)-C(7)-C(8)    | 115.5(6)  |
| C(10)-C(9)-C(14)  | 119.5(8)  |
| C(10)-C(9)-S(2)   | 119.6(6)  |
| C(14)-C(9)-S(2)   | 120.5(6)  |
| C(9)-C(10)-C(11)  | 119.2(10) |
| C(12)-C(11)-C(10) | 120.1(10) |
| C(13)-C(12)-C(11) | 120.0(10) |
| C(12)-C(13)-C(14) | 121.2(11) |
| C(9)-C(14)-C(13)  | 120.0(10) |

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Table 3. Anisotropic thermal parameters ( $\times 10^3$ ) of the non-H atoms.

E.s.d.'s in parentheses

|       | U11     | U22     | U33   | U23    | U13   | U12    |
|-------|---------|---------|-------|--------|-------|--------|
| Re    | 28(1)   | 32(1)   | 25(1) | 1(1)   | 6(1)  | -5(1)  |
| S(1)  | 49(1)   | 52(1)   | 27(1) | 3(1)   | 6(1)  | -13(1) |
| S(2)  | 60(1)   | 50(1)   | 32(1) | -3(1)  | 13(1) | -25(1) |
| O     | 36(2)   | 43(3)   | 45(3) | 3(2)   | 8(2)  | 1(2)   |
| N(1)  | 30(3)   | 44(3)   | 28(3) | -1(2)  | 4(2)  | -6(2)  |
| N(2)  | 35(3)   | 32(3)   | 24(2) | 2(2)   | 8(2)  | -3(2)  |
| C(1)  | 46(4)   | 56(5)   | 34(3) | -5(3)  | 2(3)  | -9(3)  |
| C(2)  | 32(3)   | 53(4)   | 37(4) | -2(3)  | 2(3)  | -5(3)  |
| C(3)  | 34(3)   | 51(4)   | 44(4) | -1(3)  | 12(3) | -7(3)  |
| C(4)  | 43(4)   | 41(4)   | 29(3) | 8(3)   | 13(3) | -7(3)  |
| C(5)  | 44(4)   | 51(4)   | 29(3) | -1(3)  | 4(3)  | -14(3) |
| C(6)  | 38(4)   | 77(6)   | 40(4) | 7(4)   | -5(3) | 6(4)   |
| C(7)  | 52(4)   | 34(4)   | 37(4) | 5(3)   | 12(3) | 10(3)  |
| C(8)  | 63(5)   | 64(5)   | 47(4) | 2(4)   | 20(4) | 20(4)  |
| C(9)  | 47(4)   | 37(3)   | 34(3) | -3(3)  | 14(3) | -14(3) |
| C(10) | 59(5)   | 58(5)   | 53(5) | -7(4)  | 19(4) | -9(4)  |
| C(11) | 85(8)   | 114(10) | 62(6) | -24(6) | 47(6) | -51(7) |
| C(12) | 140(12) | 59(6)   | 53(6) | -5(5)  | 29(7) | -45(7) |
| C(13) | 132(11) | 41(5)   | 50(5) | 4(4)   | -9(6) | -5(6)  |
| C(14) | 68(5)   | 41(4)   | 54(5) | -5(3)  | 10(4) | 0(4)   |

Table 4. Positional ( $\times 10^4$ ) and isotropic thermal parameters ( $\times 10^3$ ) of the refined hydrogen atoms. E.s.d.'s in parentheses

|       | x          | y        | z          | U(eq)  |
|-------|------------|----------|------------|--------|
| H(2A) | -4548(128) | 756(27)  | 1157(98)   | 34(19) |
| H(2B) | -4487(118) | 171(29)  | 1515(90)   | 40(20) |
| H(3A) | -4069(125) | 808(23)  | 4073(97)   | 26(18) |
| H(3B) | -3508(119) | 219(30)  | 4336(93)   | 43(21) |
| H(4A) | -2182(83)  | 745(19)  | 6521(65)   | 6(12)  |
| H(4B) | -25(147)   | 280(34)  | 5594(117)  | 70(29) |
| H(7A) | -755(111)  | 1745(29) | 4797(91)   | 36(20) |
| H(7B) | -2462(89)  | 1484(21) | 3900(75)   | 15(14) |
| H(10) | 4879(121)  | 1583(29) | 1190(97)   | 36(22) |
| H(11) | 5810(149)  | 2019(33) | -954(113)  | 62(26) |
| H(12) | 3449(176)  | 2416(38) | -2586(141) | 81(34) |
| H(14) | -548(166)  | 2050(37) | 593(131)   | 81(32) |

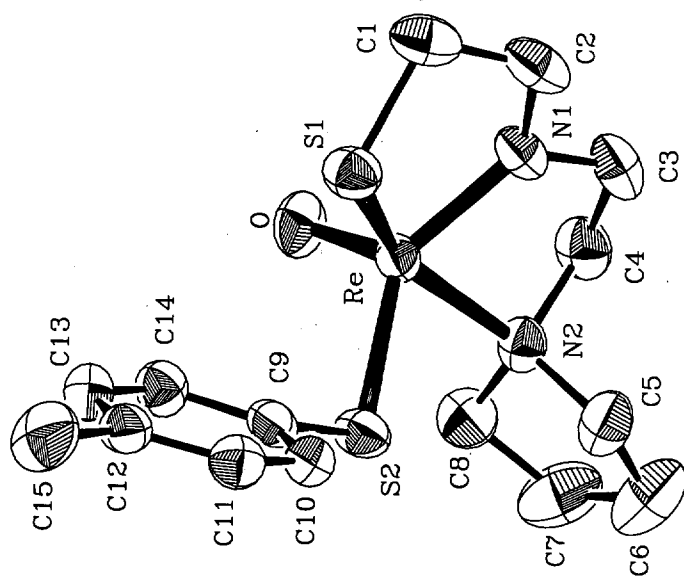


Table 1. Summary of Crystal, Intensity Collection and Refinement Data

|                                                                                      |                                                                  |
|--------------------------------------------------------------------------------------|------------------------------------------------------------------|
| Empirical formula                                                                    | C <sub>15</sub> H <sub>23</sub> N <sub>2</sub> OReS <sub>2</sub> |
| Formula weight                                                                       | 497.67                                                           |
| Temperature, K                                                                       | 298                                                              |
| Wavelength                                                                           | Mo K $\alpha$ 0.71073                                            |
| Space group                                                                          | P2 <sub>1</sub> /n                                               |
| a (Å)                                                                                | 10.231(6)                                                        |
| b (Å)                                                                                | 9.277(5)                                                         |
| c (Å)                                                                                | 18.316(9)                                                        |
| $\beta$ , deg                                                                        | 97.60(1)                                                         |
| V, (Å <sup>3</sup> )                                                                 | 1723.2(2)                                                        |
| Z                                                                                    | 4                                                                |
| D <sub>calcd</sub> /D <sub>measd</sub> (Mg m <sup>-3</sup> )                         | 1.918/1.89                                                       |
| abs coeff( $\mu$ ), mm <sup>-1</sup>                                                 | 7.294                                                            |
| Abs.correction mode Tmin/Tmax                                                        | 0.41/1.00                                                        |
| Scan mode / speed (deg/min)                                                          | $\theta$ -2 $\theta$ /3.5                                        |
| Scan range (2 $\theta$ deg)                                                          | 2.5 + $\alpha_1\alpha_2$ separation                              |
| $\theta$ range(deg)                                                                  | 2.17 to 26.98                                                    |
| Reflections collected                                                                | 3871                                                             |
| Independent reflections                                                              | 3749 [R(int) = 0.0092]                                           |
| Range of h,k,l                                                                       | -13 $\rightarrow$ 12, -11 $\rightarrow$ 0, 0 $\rightarrow$ 23    |
| F(000)                                                                               | 968                                                              |
| [ $\Delta/\sigma$ ] <sub>max</sub>                                                   | 0.018                                                            |
| W <sup>(1)</sup>                                                                     | a=0.0646 b=0.8592                                                |
| [ $\Delta\rho$ ] <sub>max</sub> /[ $\Delta\rho$ ] <sub>min</sub> (e/Å <sup>3</sup> ) | 0.741 and -0.942                                                 |
| Refinement method                                                                    | Full-matrix least-squares on F <sup>2</sup>                      |
| Data / restraints / parameters                                                       | 3746 / 0 / 273                                                   |
| Goodness-of-fit on F <sup>2</sup>                                                    | 1.086                                                            |
| R indices [3749 refs I > 2 $\sigma$ (I)] <sup>(2)</sup>                              | R1 = 0.0323, wR2 = 0.0929                                        |
| R indices (all data)                                                                 | R1 = 0.0349, wR2 = 0.0962                                        |

(1)  $W = 1/[\sigma^2 \langle (F_o^2) + (a \cdot P)^2 + b \cdot P \rangle]$  and  $P = (\text{Max}(F_o^2, 0) + 2 \cdot F_c^2)/3$

(2) R1 based on F's, wR2 based on F<sup>2</sup> Extinction coefficient 0.0138(7)

Table 2. Positional ( $\times 10^4$ ) and equivalent thermal parameters ( $\times 10^3$ ) of the non-H atoms. E.s.d.'s in parentheses.  $U(eq)$  is defined as one third of the trace of the orthogonalized  $U_{ij}$  tensor.

|       | x        | y        | z       | $U(eq)$ |
|-------|----------|----------|---------|---------|
| Re    | 889(1)   | 750(1)   | 1705(1) | 34(1)   |
| O     | 1122(4)  | 705(4)   | 2640(2) | 53(2)   |
| S(1)  | -1311(1) | 555(1)   | 1290(1) | 47(1)   |
| S(2)  | 865(1)   | 3113(1)  | 1286(1) | 45(1)   |
| N(1)  | 956(4)   | -1130(4) | 1258(2) | 45(2)   |
| N(2)  | 2952(4)  | 660(4)   | 1491(2) | 40(2)   |
| C(1)  | -1407(6) | -1422(6) | 1232(4) | 59(3)   |
| C(2)  | -203(6)  | -1954(5) | 922(3)  | 59(3)   |
| C(3)  | 2203(5)  | -1759(6) | 1126(3) | 56(3)   |
| C(4)  | 3279(6)  | -911(5)  | 1574(3) | 51(3)   |
| C(5)  | 3285(6)  | 1176(9)  | 759(3)  | 55(3)   |
| C(6)  | 4776(7)  | 1462(10) | 889(4)  | 83(3)   |
| C(7)  | 5132(6)  | 1722(10) | 1702(4) | 79(3)   |
| C(8)  | 3855(5)  | 1555(6)  | 2036(3) | 53(2)   |
| C(9)  | -752(4)  | 3869(5)  | 1185(3) | 39(2)   |
| C(10) | -1341(5) | 4343(4)  | 494(3)  | 43(2)   |
| C(11) | -2546(4) | 5036(5)  | 429(3)  | 46(2)   |
| C(12) | -3181(4) | 5313(5)  | 1039(3) | 44(2)   |
| C(13) | -2599(5) | 4842(5)  | 1721(3) | 48(2)   |
| C(14) | -1402(5) | 4114(5)  | 1792(3) | 46(3)   |
| C(15) | -4456(6) | 6153(8)  | 968(4)  | 72(3)   |

Table 5. Bond lengths [Å] and angles [deg]

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|              |           |
|--------------|-----------|
| Re-O         | 1.699(4)  |
| Re-N(1)      | 1.931(4)  |
| Re-N(2)      | 2.198(5)  |
| Re-S(1)      | 2.286(2)  |
| Re-S(2)      | 2.321(2)  |
| S(1)-C(1)    | 1.839(6)  |
| S(2)-C(9)    | 1.784(5)  |
| N(1)-C(3)    | 1.452(7)  |
| N(1)-C(2)    | 1.475(6)  |
| N(2)-C(4)    | 1.498(6)  |
| N(2)-C(5)    | 1.505(7)  |
| N(2)-C(8)    | 1.516(7)  |
| C(1)-C(2)    | 1.506(9)  |
| C(3)-C(4)    | 1.506(8)  |
| C(5)-C(6)    | 1.535(9)  |
| C(6)-C(7)    | 1.504(11) |
| C(7)-C(8)    | 1.523(8)  |
| C(9)-C(14)   | 1.387(7)  |
| C(9)-C(10)   | 1.399(7)  |
| C(10)-C(11)  | 1.381(7)  |
| C(11)-C(12)  | 1.387(7)  |
| C(12)-C(13)  | 1.382(7)  |
| C(12)-C(15)  | 1.510(7)  |
| C(13)-C(14)  | 1.389(7)  |
| O-Re-N(1)    | 113.4(2)  |
| O-Re-N(2)    | 99.7(2)   |
| N(1)-Re-N(2) | 78.6(2)   |
| O-Re-S(1)    | 109.5(2)  |
| N(1)-Re-S(1) | 82.87(14) |

|                   |            |
|-------------------|------------|
| N(2)-Re-S(1)      | 149.81(12) |
| O-Re-S(2)         | 110.54(13) |
| N(1)-Re-S(2)      | 135.42(14) |
| N(2)-Re-S(2)      | 86.91(10)  |
| S(1)-Re-S(2)      | 89.88(7)   |
| C(1)-S(1)-Re      | 98.1(2)    |
| C(9)-S(2)-Re      | 112.0(2)   |
| C(3)-N(1)-C(2)    | 113.4(4)   |
| C(3)-N(1)-Re      | 121.0(3)   |
| C(2)-N(1)-Re      | 125.0(3)   |
| C(4)-N(2)-C(5)    | 109.0(4)   |
| C(4)-N(2)-C(8)    | 111.0(4)   |
| C(5)-N(2)-C(8)    | 102.9(4)   |
| C(4)-N(2)-Re      | 103.0(3)   |
| C(5)-N(2)-Re      | 118.9(3)   |
| C(8)-N(2)-Re      | 112.2(3)   |
| C(2)-C(1)-S(1)    | 108.0(4)   |
| N(1)-C(2)-C(1)    | 108.9(4)   |
| N(1)-C(3)-C(4)    | 107.1(4)   |
| N(2)-C(4)-C(3)    | 108.4(4)   |
| N(2)-C(5)-C(6)    | 104.7(5)   |
| C(7)-C(6)-C(5)    | 106.7(5)   |
| C(6)-C(7)-C(8)    | 105.9(5)   |
| N(2)-C(8)-C(7)    | 105.7(5)   |
| C(14)-C(9)-C(10)  | 118.6(4)   |
| C(14)-C(9)-S(2)   | 121.2(4)   |
| C(10)-C(9)-S(2)   | 120.0(4)   |
| C(11)-C(10)-C(9)  | 119.8(5)   |
| C(10)-C(11)-C(12) | 121.6(5)   |
| C(13)-C(12)-C(11) | 118.5(4)   |
| C(13)-C(12)-C(15) | 120.3(5)   |
| C(11)-C(12)-C(15) | 121.1(5)   |
| C(12)-C(13)-C(14) | 120.5(4)   |

C(9)-C(14)-C(13)

120.9(5)

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Table 3. Anisotropic thermal parameters ( $\times 10^3$ ) of the non-H atoms. E.s.d.'s in parentheses

|       | U <sub>11</sub> | U <sub>22</sub> | U <sub>33</sub> | U <sub>23</sub> | U <sub>13</sub> | U <sub>12</sub> |
|-------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| Re    | 38(1)           | 32(1)           | 33(1)           | -3(1)           | 5(1)            | 0(1)            |
| O     | 60(2)           | 69(3)           | 29(2)           | -7(1)           | 5(2)            | -1(1)           |
| S(1)  | 40(1)           | 42(1)           | 58(1)           | -1(1)           | 4(1)            | -4(1)           |
| S(2)  | 38(1)           | 33(1)           | 64(1)           | 0(1)            | 8(1)            | 1(1)            |
| N(1)  | 56(2)           | 34(2)           | 43(2)           | -8(2)           | 1(2)            | 1(2)            |
| N(2)  | 41(2)           | 42(2)           | 37(2)           | 3(1)            | 6(2)            | 8(1)            |
| C(1)  | 61(3)           | 47(3)           | 66(3)           | 0(2)            | -5(3)           | -16(2)          |
| C(2)  | 75(3)           | 37(2)           | 59(3)           | -10(2)          | -5(3)           | -6(2)           |
| C(3)  | 69(3)           | 42(2)           | 57(3)           | -12(2)          | 6(2)            | 14(2)           |
| C(4)  | 51(3)           | 45(3)           | 55(3)           | 4(2)            | 5(2)            | 15(2)           |
| C(5)  | 56(3)           | 64(3)           | 49(3)           | 12(3)           | 17(2)           | 17(3)           |
| C(6)  | 62(3)           | 114(6)          | 77(4)           | 28(4)           | 27(3)           | 4(4)            |
| C(7)  | 49(3)           | 95(5)           | 92(5)           | 23(4)           | 8(3)            | -13(3)          |
| C(8)  | 42(2)           | 56(3)           | 58(3)           | -2(2)           | 3(2)            | -1(2)           |
| C(9)  | 42(2)           | 28(2)           | 48(2)           | -4(2)           | 7(2)            | -1(2)           |
| C(10) | 43(2)           | 40(2)           | 47(3)           | -3(2)           | 11(2)           | -2(2)           |
| C(11) | 46(2)           | 46(2)           | 46(2)           | 1(2)            | 4(2)            | -1(2)           |
| C(12) | 39(2)           | 35(2)           | 60(3)           | 2(2)            | 14(2)           | 2(2)            |
| C(13) | 52(2)           | 48(3)           | 46(2)           | -3(2)           | 14(2)           | 3(2)            |
| C(14) | 52(3)           | 46(2)           | 39(2)           | -1(2)           | 5(2)            | 2(2)            |
| C(15) | 51(3)           | 72(3)           | 99(5)           | 16(4)           | 28(3)           | 18(3)           |

Table 4. Positional ( $\times 10^4$ ) and isotropic thermal parameters ( $\times 10^3$ ) of the refined hydrogen atoms.  
E.s.d.'s in parentheses

|       | x         | y         | z        | U(eq)   |
|-------|-----------|-----------|----------|---------|
| H(1A) | -1445(52) | -1845(62) | 1775(32) | 48(13)  |
| H(1B) | -2153(69) | -1693(79) | 960(37)  | 70(18)  |
| H(2A) | -86(59)   | -3098(71) | 1099(33) | 65(16)  |
| H(2B) | -345(52)  | -1717(63) | 382(31)  | 51(14)  |
| H(3A) | 2262(46)  | -2632(50) | 1401(25) | 32(11)  |
| H(3B) | 2122(71)  | -1727(86) | 615(44)  | 86(22)  |
| H(4A) | 3358(55)  | -1143(68) | 2099(35) | 43(14)  |
| H(4B) | 4116(59)  | -1142(63) | 1426(33) | 53(14)  |
| H(5A) | 3042(89)  | 1696(106) | 753(50)  | 90(32)  |
| H(5B) | 2940(61)  | 496(68)   | 323(38)  | 55(16)  |
| H(6A) | 5312(99)  | 586(97)   | 601(57)  | 107(32) |
| H(6B) | 5024(124) | 2187(145) | 469(69)  | 160(46) |
| H(7A) | 5286(120) | 2756(118) | 1751(57) | 130(35) |
| H(7B) | 5714(85)  | 757(85)   | 1751(44) | 83(34)  |
| H(8A) | 3968(61)  | 985(62)   | 2543(38) | 54(16)  |
| H(8B) | 3481(63)  | 2516(73)  | 2031(35) | 58(17)  |
| H(10) | -903(51)  | 4251(41)  | 41(30)   | 31(12)  |
| H(11) | -2866(73) | 5295(92)  | 11(42)   | 76(20)  |
| H(13) | -3027(62) | 5128(75)  | 2187(35) | 63(16)  |
| H(14) | -993(62)  | 3804(74)  | 2269(37) | 65(17)  |

fig 5 complex 7

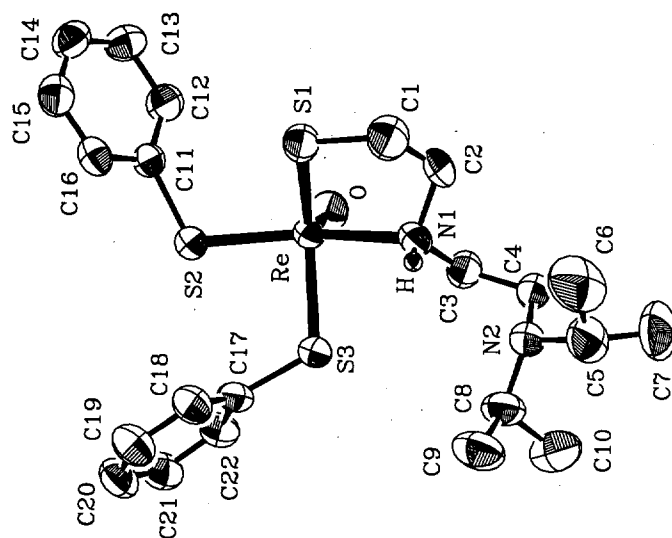


Table 1. Summary of Crystal, Intensity Collection and Refinement Data

|                                                                |                                                                  |
|----------------------------------------------------------------|------------------------------------------------------------------|
| Empirical formula                                              | C <sub>22</sub> H <sub>33</sub> N <sub>2</sub> OReS <sub>3</sub> |
| Formula weight                                                 | 623.88                                                           |
| Temperature, K                                                 | 298                                                              |
| Wavelength                                                     | Cu K $\alpha$ 1.54180                                            |
| Space group                                                    | P2 <sub>1</sub> /n                                               |
| a (Å)                                                          | 9.500(1)                                                         |
| b (Å)                                                          | 16.385(1)                                                        |
| c (Å)                                                          | 16.254(1)                                                        |
| $\beta$ , deg                                                  | 95.905(2)                                                        |
| V, (Å <sup>3</sup> )                                           | 2516.7(3)                                                        |
| Z                                                              | 4                                                                |
| D <sub>calc</sub> /D <sub>meas</sub> (Mg m <sup>-3</sup> )     | 1.647/1.62                                                       |
| abs coeff( $\mu$ ), mm <sup>-1</sup>                           | 11.882                                                           |
| Abs. correction mode Tmin/Tmax                                 | 0.74/1.00                                                        |
| Scan mode / speed (deg/min)                                    | $\theta$ -2 $\theta$ /3.0                                        |
| Scan range (2 $\theta$ deg)                                    | 2.4 + $\alpha_1\alpha_2$ separation                              |
| $\theta$ range(deg)                                            | 3.84 to 64.99                                                    |
| Reflections collected                                          | 4534                                                             |
| Independent reflections                                        | 4261 [R(int) = 0.0495]                                           |
| Range of h,k,l                                                 | -11 $\rightarrow$ 0, -19 $\rightarrow$ 0, -19 $\rightarrow$ 19   |
| F(000)                                                         | 1240                                                             |
| $[\Delta/\sigma]_{\max}$                                       | 0.610                                                            |
| W <sup>(1)</sup>                                               | a=0.0745 b=1.7078                                                |
| $[\Delta\rho]_{\max}/[\Delta\rho]_{\min}$ (e/ Å <sup>3</sup> ) | 0.728 and -0.895                                                 |
| Refinement method                                              | Full-matrix least-squares on F <sup>2</sup>                      |
| Data / restraints / parameters                                 | 4261 / 0 / 348                                                   |
| Goodness-of-fit on F <sup>2</sup>                              | 1.109                                                            |
| R indices [3834 refs I $\geq$ 2 $\sigma$ (I)] <sup>(2)</sup>   | R1 = 0.0387, wR2 = 0.1100                                        |
| R indices (all data)                                           | R1 = 0.0427, wR2 = 0.1145                                        |

(1)  $W=1/[\sigma^2(F_o^2)+(a*P)^2+b*P]$  and  $P=(\text{Max}(F_o^2,0)+2*F_c^2)/3$

(2) R1 based on F's, wR2 based on F<sup>2</sup> Extinction coefficient 0.0022(1)

Table 2. Positional ( $\times 10^4$ ) and equivalent thermal parameters ( $\times 10^3$ ) of the non-H atoms. E.s.d.'s in parentheses. U(eq) is defined as one third of the trace of the orthogonalized  $U_{ij}$  tensor.

|       | x        | y       | z        | U(eq)  |
|-------|----------|---------|----------|--------|
| Re    | 747(1)   | 2424(1) | 674(1)   | 40(1)  |
| S(1)  | -882(2)  | 2410(1) | 1606(1)  | 63(1)  |
| S(2)  | -1079(2) | 2364(1) | -363(1)  | 52(1)  |
| S(3)  | 1785(1)  | 1362(1) | 34(1)    | 51(1)  |
| O     | 1555(4)  | 3324(2) | 567(2)   | 59(1)  |
| N(1)  | 2147(5)  | 1989(3) | 1736(2)  | 50(1)  |
| C(1)  | 136(8)   | 2090(7) | 2563(4)  | 91(2)  |
| C(2)  | 1673(9)  | 2308(6) | 2524(4)  | 83(2)  |
| C(3)  | 3664(6)  | 2208(4) | 1679(4)  | 57(1)  |
| C(4)  | 4710(6)  | 1748(4) | 2273(4)  | 59(1)  |
| N(2)  | 4559(5)  | 866(3)  | 2199(3)  | 56(1)  |
| C(5)  | 5111(9)  | 425(5)  | 2956(5)  | 84(2)  |
| C(6)  | 3966(13) | 334(7)  | 3520(5)  | 123(4) |
| C(7)  | 6470(11) | 772(7)  | 3417(6)  | 122(4) |
| C(8)  | 5037(7)  | 548(4)  | 1414(4)  | 66(2)  |
| C(9)  | 4504(8)  | -306(5) | 1235(5)  | 92(2)  |
| C(10) | 6626(8)  | 599(5)  | 1344(6)  | 91(2)  |
| C(11) | -2614(5) | 2891(3) | -105(3)  | 43(1)  |
| C(12) | -2562(6) | 3680(4) | 208(3)   | 55(1)  |
| C(13) | -3815(7) | 4088(4) | 319(4)   | 61(1)  |
| C(14) | -5093(6) | 3724(4) | 122(4)   | 64(2)  |
| C(15) | -5147(6) | 2952(4) | -183(3)  | 60(1)  |
| C(16) | -3910(7) | 2526(3) | -297(4)  | 52(1)  |
| C(17) | 1020(5)  | 1124(3) | -986(3)  | 45(1)  |
| C(18) | -87(6)   | 571(4)  | -1108(3) | 56(1)  |
| C(19) | -583(8)  | 344(4)  | -1908(4) | 69(2)  |
| C(20) | 17(7)    | 649(4)  | -2568(4) | 64(2)  |
| C(21) | 1122(7)  | 1198(4) | -2452(3) | 61(1)  |
| C(22) | 1613(6)  | 1442(4) | -1662(3) | 55(1)  |

Table 5. Bond lengths [ $\text{\AA}$ ] and angles [deg]

|             |            |
|-------------|------------|
| Re-O        | 1.679(4)   |
| Re-N(1)     | 2.187(4)   |
| Re-S(1)     | 2.273(2)   |
| Re-S(2)     | 2.293(2)   |
| Re-S(3)     | 2.3012(12) |
| S(1)-C(1)   | 1.823(7)   |
| S(2)-C(11)  | 1.780(5)   |
| S(3)-C(17)  | 1.784(5)   |
| N(1)-C(3)   | 1.497(8)   |
| N(1)-C(2)   | 1.496(8)   |
| C(1)-C(2)   | 1.511(12)  |
| C(3)-C(4)   | 1.513(8)   |
| C(4)-N(2)   | 1.456(7)   |
| N(2)-C(5)   | 1.476(8)   |
| N(2)-C(8)   | 1.490(7)   |
| C(5)-C(6)   | 1.500(13)  |
| C(5)-C(7)   | 1.533(12)  |
| C(8)-C(9)   | 1.507(9)   |
| C(8)-C(10)  | 1.527(9)   |
| C(11)-C(16) | 1.375(8)   |
| C(11)-C(12) | 1.389(7)   |
| C(12)-C(13) | 1.393(8)   |
| C(13)-C(14) | 1.361(9)   |
| C(14)-C(15) | 1.357(9)   |
| C(15)-C(16) | 1.395(9)   |
| C(17)-C(22) | 1.386(7)   |
| C(17)-C(18) | 1.387(7)   |
| C(18)-C(19) | 1.388(9)   |
| C(19)-C(20) | 1.360(9)   |
| C(20)-C(21) | 1.380(9)   |
| C(21)-C(22) | 1.380(8)   |

|                   |            |
|-------------------|------------|
| O-Re-N(1)         | 96.9(2)    |
| O-Re-S(1)         | 115.32(14) |
| N(1)-Re-S(1)      | 82.64(13)  |
| O-Re-S(2)         | 106.29(14) |
| N(1)-Re-S(2)      | 156.73(14) |
| S(1)-Re-S(2)      | 88.48(6)   |
| O-Re-S(3)         | 113.27(14) |
| N(1)-Re-S(3)      | 81.73(12)  |
| S(1)-Re-S(3)      | 130.26(5)  |
| S(2)-Re-S(3)      | 87.84(5)   |
| C(1)-S(1)-Re      | 103.5(2)   |
| C(11)-S(2)-Re     | 112.5(2)   |
| C(17)-S(3)-Re     | 115.7(2)   |
| C(3)-N(1)-C(2)    | 110.2(5)   |
| C(3)-N(1)-Re      | 112.6(4)   |
| C(2)-N(1)-Re      | 110.5(4)   |
| C(2)-C(1)-S(1)    | 109.1(6)   |
| N(1)-C(2)-C(1)    | 109.4(6)   |
| N(1)-C(3)-C(4)    | 114.5(5)   |
| N(2)-C(4)-C(3)    | 112.9(5)   |
| C(4)-N(2)-C(5)    | 113.1(5)   |
| C(4)-N(2)-C(8)    | 112.4(5)   |
| C(5)-N(2)-C(8)    | 115.4(5)   |
| N(2)-C(5)-C(6)    | 109.9(7)   |
| N(2)-C(5)-C(7)    | 115.8(7)   |
| C(6)-C(5)-C(7)    | 111.4(8)   |
| N(2)-C(8)-C(9)    | 111.4(6)   |
| N(2)-C(8)-C(10)   | 115.8(6)   |
| C(9)-C(8)-C(10)   | 110.5(6)   |
| C(16)-C(11)-C(12) | 119.0(5)   |
| C(16)-C(11)-S(2)  | 118.2(4)   |
| C(12)-C(11)-S(2)  | 122.6(4)   |
| C(11)-C(12)-C(13) | 119.7(5)   |

|                   |          |
|-------------------|----------|
| C(14)-C(13)-C(12) | 120.8(6) |
| C(15)-C(14)-C(13) | 119.6(6) |
| C(14)-C(15)-C(16) | 120.9(6) |
| C(11)-C(16)-C(15) | 120.0(5) |
| C(22)-C(17)-C(18) | 119.8(5) |
| C(22)-C(17)-S(3)  | 119.6(4) |
| C(18)-C(17)-S(3)  | 120.4(4) |
| C(17)-C(18)-C(19) | 119.2(5) |
| C(20)-C(19)-C(18) | 120.8(6) |
| C(19)-C(20)-C(21) | 120.4(6) |
| C(22)-C(21)-C(20) | 119.8(5) |
| C(21)-C(22)-C(17) | 120.1(5) |

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Table 3. Anisotropic thermal parameters ( $\times 10^3$ ) of the non-H atoms. E.s.d.'s in parentheses

|       | U <sub>11</sub> | U <sub>22</sub> | U <sub>33</sub> | U <sub>23</sub> | U <sub>13</sub> | U <sub>12</sub> |
|-------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| Re    | 43(1)           | 43(1)           | 35(1)           | -1(1)           | 5(1)            | 3(1)            |
| S(1)  | 50(1)           | 99(1)           | 42(1)           | 1(1)            | 10(1)           | 9(1)            |
| S(2)  | 52(1)           | 64(1)           | 40(1)           | -6(1)           | 0(1)            | 13(1)           |
| S(3)  | 53(1)           | 54(1)           | 43(1)           | -8(1)           | 1(1)            | 10(1)           |
| O     | 60(2)           | 49(2)           | 67(2)           | 1(2)            | 2(2)            | -5(2)           |
| N(1)  | 58(2)           | 53(3)           | 38(2)           | -6(2)           | 1(2)            | 4(2)            |
| C(1)  | 78(5)           | 154(8)          | 43(3)           | 10(4)           | 17(3)           | 23(5)           |
| C(2)  | 79(5)           | 130(7)          | 35(3)           | -25(4)          | -14(3)          | 33(5)           |
| C(3)  | 52(3)           | 48(3)           | 68(4)           | -8(3)           | -5(3)           | 3(2)            |
| C(4)  | 54(3)           | 55(3)           | 63(3)           | -11(3)          | -10(3)          | 10(2)           |
| N(2)  | 59(3)           | 52(3)           | 55(3)           | -9(2)           | -2(2)           | 8(2)            |
| C(5)  | 113(6)          | 66(4)           | 70(4)           | 5(3)            | -10(4)          | 22(4)           |
| C(6)  | 169(10)         | 132(9)          | 70(5)           | 25(5)           | 23(6)           | 6(8)            |
| C(7)  | 134(8)          | 125(8)          | 92(6)           | 0(5)            | -53(6)          | 34(6)           |
| C(8)  | 70(4)           | 56(3)           | 72(4)           | -18(3)          | 11(3)           | 6(3)            |
| C(9)  | 102(5)          | 78(5)           | 98(5)           | -36(4)          | 21(4)           | -12(4)          |
| C(10) | 78(5)           | 81(5)           | 117(6)          | -14(4)          | 25(4)           | 6(4)            |
| C(11) | 47(3)           | 48(3)           | 35(2)           | 3(2)            | 4(2)            | 6(2)            |
| C(12) | 52(3)           | 54(3)           | 59(3)           | -2(2)           | 6(2)            | 0(2)            |
| C(13) | 65(4)           | 52(3)           | 67(4)           | 3(3)            | 13(3)           | 8(3)            |
| C(14) | 53(3)           | 83(4)           | 59(3)           | 8(3)            | 12(3)           | 16(3)           |
| C(15) | 50(3)           | 86(5)           | 45(3)           | 8(3)            | 9(2)            | -3(3)           |
| C(16) | 57(4)           | 59(4)           | 40(3)           | -2(2)           | 3(3)            | -6(2)           |
| C(17) | 56(3)           | 40(2)           | 41(2)           | -4(2)           | 10(2)           | 6(2)            |
| C(18) | 65(3)           | 56(3)           | 48(3)           | 0(2)            | 12(2)           | -7(2)           |
| C(19) | 82(5)           | 65(4)           | 61(4)           | -7(3)           | 6(3)            | -16(3)          |
| C(20) | 81(4)           | 71(4)           | 40(3)           | -6(3)           | 4(3)            | 2(3)            |
| C(21) | 75(4)           | 65(4)           | 45(3)           | 2(3)            | 17(3)           | 2(3)            |
| C(22) | 60(3)           | 55(3)           | 51(3)           | -1(2)           | 12(2)           | -1(2)           |

Table 4. Positional ( $\times 10^4$ ) and isotropic thermal parameters ( $\times 10^3$ ) of the refined hydrogen atoms.

E.s.d.'s in parentheses

|       | x          | y        | z         | U(eq)   |
|-------|------------|----------|-----------|---------|
| HN1   | 2031(55)   | 1493(34) | 1738(32)  | 44(15)  |
| H(2A) | 2144(43)   | 2132(23) | 2845(26)  | 6(9)    |
| H(2B) | 1997(58)   | 2910(35) | 2739(35)  | 47(14)  |
| H(3A) | 3886(60)   | 2093(39) | 1038(37)  | 60(16)  |
| H(3B) | 3742(67)   | 2827(42) | 1785(40)  | 61(17)  |
| H(4A) | 4629(46)   | 1906(31) | 2874(30)  | 38(12)  |
| H(4B) | 5709(67)   | 1906(42) | 2082(37)  | 67(17)  |
| H(5)  | 5416(75)   | -165(51) | 2799(45)  | 97(23)  |
| H(8)  | 4578(80)   | 1033(52) | 900(52)   | 114(26) |
| H(12) | -1564(75)  | 3917(48) | 378(44)   | 98(23)  |
| H(13) | -3705(88)  | 4540(53) | 517(52)   | 103(29) |
| H(14) | -5975(64)  | 3951(38) | 220(38)   | 62(17)  |
| H(15) | -6165(108) | 2868(72) | -474(64)  | 144(36) |
| H(16) | -3883(53)  | 1960(36) | -483(30)  | 45(14)  |
| H(18) | -603(50)   | 406(39)  | -654(31)  | 48(15)  |
| H(19) | -1321(80)  | 88(50)   | -1995(46) | 84(24)  |
| H(20) | -434(67)   | 440(42)  | -3126(43) | 76(19)  |
| H(21) | 1455(67)   | 1477(41) | -2869(43) | 73(19)  |
| H(22) | 2346(64)   | 1860(43) | -1589(37) | 72(19)  |

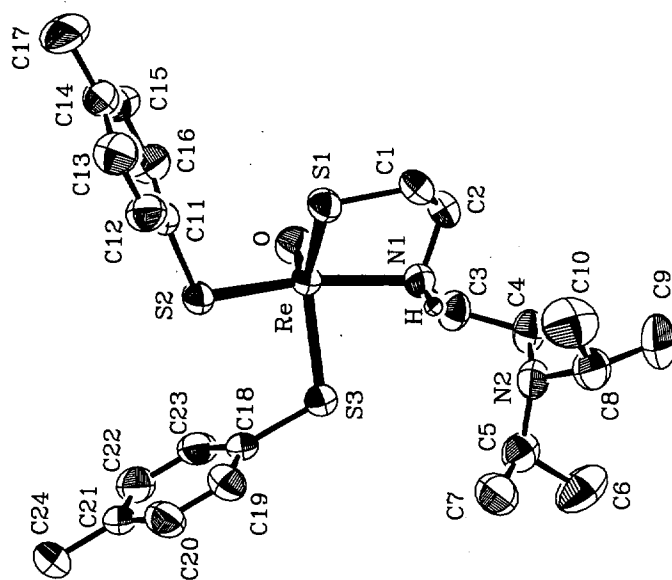


Table 1. Summary of Crystal, Intensity Collection and Refinement Data

|                                                                                       |                                                                  |
|---------------------------------------------------------------------------------------|------------------------------------------------------------------|
| Empirical formula                                                                     | C <sub>24</sub> H <sub>37</sub> N <sub>2</sub> OReS <sub>3</sub> |
| Formula weight                                                                        | 651.94                                                           |
| Temperature, K                                                                        | 298                                                              |
| Wavelength                                                                            | Mo K $\alpha$ 0.71073                                            |
| Space group                                                                           | P2 <sub>1</sub> /n                                               |
| a (Å)                                                                                 | 11.687(7)                                                        |
| b (Å)                                                                                 | 14.462(7)                                                        |
| c (Å)                                                                                 | 16.273(9)                                                        |
| $\beta$ , deg                                                                         | 92.11(2)                                                         |
| V, (Å <sup>3</sup> )                                                                  | 2748.6(2)                                                        |
| Z                                                                                     | 4                                                                |
| D <sub>calc</sub> /D <sub>meas</sub> (Mg m <sup>-3</sup> )                            | 1.575/1.55                                                       |
| abs coeff( $\mu$ ), mm <sup>-1</sup>                                                  | 4.667                                                            |
| Abs.correction mode Tmin/Tmax                                                         | 0.61/1.00                                                        |
| Scan mode / speed (deg/min)                                                           | $\theta$ -2 $\theta$ /1.00                                       |
| Scan range (2 $\theta$ deg)                                                           | 2.5 + $\alpha_1\alpha_2$ separation                              |
| $\theta$ range(deg)                                                                   | 1.88 to 25.00                                                    |
| Reflections collected                                                                 | 5062                                                             |
| Independent reflections                                                               | 4814 [R(int) = 0.0299]                                           |
| Range of h,k,l                                                                        | -13 $\rightarrow$ 0, 0 $\rightarrow$ 17, -19 $\rightarrow$ 19    |
| F(000)                                                                                | 1304                                                             |
| [ $\Delta/\sigma$ ] <sub>max</sub>                                                    | 0.293                                                            |
| W <sup>(1)</sup>                                                                      | a=0.0192 b=0.7153                                                |
| [ $\Delta\rho$ ] <sub>max</sub> /[ $\Delta\rho$ ] <sub>min</sub> (e/ Å <sup>3</sup> ) | 0.561 and -0.559                                                 |
| Refinement method                                                                     | Full-matrix least-squares on F <sup>2</sup>                      |
| Data / restraints / parameters                                                        | 4814 / 0 / 420                                                   |
| Goodness-of-fit on F <sup>2</sup>                                                     | 1.100                                                            |
| R indices [4282 refs I>2 $\sigma$ (I)] <sup>(2)</sup>                                 | R1 = 0.0240, wR2 = 0.0608                                        |
| R indices (all data)                                                                  | R1 = 0.0292, wR2 = 0.0636                                        |

(1)  $W=1/[\sigma^2(F_o^2)+(a*P)^2+b*P]$  and  $P=(\text{Max}(F_o^2,0)+2*F_c^2)/3$

(2) R1 based on F's, wR2 based on F<sup>2</sup>

Table 2. Positional ( $\times 10^4$ ) and equivalent thermal parameters ( $\times 10^3$ ) of the non-H atoms. E.s.d.'s in parentheses. U(eq) is defined as one third of the trace of the orthogonalized  $U_{ij}$  tensor.

|       | x       | y        | z        | U(eq)  |
|-------|---------|----------|----------|--------|
| Re    | 2122(1) | 1763(1)  | 1834(1)  | 39(1)  |
| O     | 3441(2) | 2060(2)  | 2201(2)  | 57(1)  |
| S(1)  | 1308(1) | 486(1)   | 2406(1)  | 51(1)  |
| S(2)  | 844(1)  | 2634(1)  | 2551(1)  | 50(1)  |
| S(3)  | 1568(1) | 2568(1)  | 667(1)   | 50(1)  |
| N(1)  | 2656(3) | 720(2)   | 947(2)   | 45(1)  |
| C(1)  | 1930(4) | -476(3)  | 1840(3)  | 60(1)  |
| C(2)  | 2955(4) | -145(3)  | 1400(3)  | 58(1)  |
| C(3)  | 3577(3) | 1009(3)  | 385(2)   | 52(1)  |
| C(4)  | 3596(4) | 393(3)   | -375(3)  | 58(1)  |
| N(2)  | 2484(3) | 374(2)   | -804(2)  | 50(1)  |
| C(5)  | 2293(4) | 1164(3)  | -1360(3) | 62(1)  |
| C(6)  | 2916(7) | 1101(6)  | -2180(4) | 95(2)  |
| C(7)  | 1031(5) | 1345(4)  | -1519(4) | 82(2)  |
| C(8)  | 2111(5) | -528(3)  | -1149(3) | 71(1)  |
| C(9)  | 3073(9) | -1158(5) | -1449(5) | 108(2) |
| C(10) | 1376(7) | -1033(5) | -545(5)  | 102(2) |
| C(11) | 639(3)  | 2132(2)  | 3541(2)  | 46(1)  |
| C(12) | -442(4) | 1852(3)  | 3762(3)  | 62(1)  |
| C(13) | -619(4) | 1524(4)  | 4542(3)  | 71(1)  |
| C(14) | 257(4)  | 1481(3)  | 5133(3)  | 65(1)  |
| C(15) | 1333(5) | 1768(3)  | 4909(3)  | 70(1)  |
| C(16) | 1519(4) | 2093(3)  | 4120(3)  | 63(1)  |
| C(17) | 52(6)   | 1144(5)  | 6003(3)  | 102(2) |
| C(18) | 813(3)  | 3612(2)  | 878(2)   | 45(1)  |
| C(19) | -339(4) | 3683(3)  | 703(3)   | 57(1)  |
| C(20) | -907(4) | 4516(3)  | 858(3)   | 65(1)  |
| C(21) | -325(5) | 5266(3)  | 1186(2)  | 65(1)  |
| C(22) | 838(5)  | 5187(3)  | 1344(3)  | 72(1)  |

|       |          |         |         |        |
|-------|----------|---------|---------|--------|
| C(23) | 1415(4)  | 4371(3) | 1183(2) | 60(1)  |
| C(24) | -946(11) | 6162(5) | 1357(4) | 103(3) |

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Table 5. Bond lengths [Å] and angles [deg]

|             |            |
|-------------|------------|
| Re-O        | 1.688(3)   |
| Re-N(1)     | 2.195(3)   |
| Re-S(1)     | 2.2896(12) |
| Re-S(3)     | 2.2998(13) |
| Re-S(2)     | 2.3044(12) |
| S(1)-C(1)   | 1.834(4)   |
| S(2)-C(11)  | 1.791(3)   |
| S(3)-C(18)  | 1.789(3)   |
| N(1)-C(2)   | 1.487(5)   |
| N(1)-C(3)   | 1.496(5)   |
| C(1)-C(2)   | 1.498(6)   |
| C(3)-C(4)   | 1.525(5)   |
| C(4)-N(2)   | 1.453(5)   |
| N(2)-C(5)   | 1.469(5)   |
| N(2)-C(8)   | 1.479(5)   |
| C(5)-C(7)   | 1.510(7)   |
| C(5)-C(6)   | 1.546(7)   |
| C(8)-C(10)  | 1.517(8)   |
| C(8)-C(9)   | 1.541(9)   |
| C(11)-C(16) | 1.370(6)   |
| C(11)-C(12) | 1.387(6)   |
| C(12)-C(13) | 1.378(7)   |
| C(13)-C(14) | 1.380(7)   |
| C(14)-C(15) | 1.386(7)   |
| C(14)-C(17) | 1.524(6)   |
| C(15)-C(16) | 1.392(7)   |
| C(18)-C(19) | 1.369(5)   |
| C(18)-C(23) | 1.385(5)   |
| C(19)-C(20) | 1.403(6)   |
| C(20)-C(21) | 1.377(7)   |
| C(21)-C(22) | 1.379(7)   |
| C(21)-C(24) | 1.517(6)   |

|                   |            |
|-------------------|------------|
| C(22)-C(23)       | 1.389(6)   |
| O-Re-N(1)         | 97.41(13)  |
| O-Re-S(1)         | 116.73(10) |
| N(1)-Re-S(1)      | 81.14(9)   |
| O-Re-S(3)         | 112.86(10) |
| N(1)-Re-S(3)      | 83.26(9)   |
| S(1)-Re-S(3)      | 129.39(4)  |
| O-Re-S(2)         | 106.42(11) |
| N(1)-Re-S(2)      | 156.14(9)  |
| S(1)-Re-S(2)      | 87.06(5)   |
| S(3)-Re-S(2)      | 88.45(5)   |
| C(1)-S(1)-Re      | 103.3(2)   |
| C(11)-S(2)-Re     | 110.10(12) |
| C(18)-S(3)-Re     | 113.27(11) |
| C(2)-N(1)-C(3)    | 112.1(3)   |
| C(2)-N(1)-Re      | 108.6(2)   |
| C(3)-N(1)-Re      | 116.0(2)   |
| C(2)-C(1)-S(1)    | 109.6(3)   |
| N(1)-C(2)-C(1)    | 109.2(3)   |
| N(1)-C(3)-C(4)    | 111.4(3)   |
| N(2)-C(4)-C(3)    | 111.2(3)   |
| C(4)-N(2)-C(5)    | 113.3(3)   |
| C(4)-N(2)-C(8)    | 116.4(3)   |
| C(5)-N(2)-C(8)    | 114.6(3)   |
| N(2)-C(5)-C(7)    | 111.4(4)   |
| N(2)-C(5)-C(6)    | 114.8(4)   |
| C(7)-C(5)-C(6)    | 110.6(5)   |
| N(2)-C(8)-C(10)   | 110.2(4)   |
| N(2)-C(8)-C(9)    | 115.7(5)   |
| C(10)-C(8)-C(9)   | 111.0(6)   |
| C(16)-C(11)-C(12) | 118.5(4)   |
| C(16)-C(11)-S(2)  | 120.9(3)   |

|                   |          |
|-------------------|----------|
| C(12)-C(11)-S(2)  | 120.3(3) |
| C(13)-C(12)-C(11) | 120.6(5) |
| C(12)-C(13)-C(14) | 121.6(5) |
| C(13)-C(14)-C(15) | 117.4(4) |
| C(13)-C(14)-C(17) | 121.6(5) |
| C(15)-C(14)-C(17) | 121.0(5) |
| C(14)-C(15)-C(16) | 121.2(5) |
| C(11)-C(16)-C(15) | 120.7(5) |
| C(19)-C(18)-C(23) | 119.8(4) |
| C(19)-C(18)-S(3)  | 120.8(3) |
| C(23)-C(18)-S(3)  | 119.3(3) |
| C(18)-C(19)-C(20) | 119.7(4) |
| C(21)-C(20)-C(19) | 121.0(4) |
| C(20)-C(21)-C(22) | 118.4(4) |
| C(20)-C(21)-C(24) | 120.7(6) |
| C(22)-C(21)-C(24) | 120.8(6) |
| C(21)-C(22)-C(23) | 121.2(4) |
| C(18)-C(23)-C(22) | 119.8(4) |

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Table 3. Anisotropic thermal parameters ( $\times 10^3$ ) of the non-H atoms. E.s.d.'s in parentheses

|       | U <sub>11</sub> | U <sub>22</sub> | U <sub>33</sub> | U <sub>23</sub> | U <sub>13</sub> | U <sub>12</sub> |
|-------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| Re    | 44(1)           | 36(1)           | 36(1)           | 2(1)            | 4(1)            | 3(1)            |
| O     | 52(2)           | 59(2)           | 61(2)           | 1(1)            | -3(1)           | -2(1)           |
| S(1)  | 63(1)           | 39(1)           | 51(1)           | 6(1)            | 11(1)           | 2(1)            |
| S(2)  | 66(1)           | 42(1)           | 44(1)           | 6(1)            | 15(1)           | 12(1)           |
| S(3)  | 65(1)           | 47(1)           | 38(1)           | 5(1)            | 7(1)            | 12(1)           |
| N(1)  | 45(2)           | 46(2)           | 43(2)           | -4(1)           | 2(1)            | 7(1)            |
| C(1)  | 80(3)           | 37(2)           | 62(3)           | 3(2)            | 3(2)            | 11(2)           |
| C(2)  | 68(3)           | 47(2)           | 60(2)           | -4(2)           | 4(2)            | 20(2)           |
| C(3)  | 43(2)           | 63(2)           | 50(2)           | -7(2)           | 7(2)            | 4(2)            |
| C(4)  | 53(2)           | 65(3)           | 55(2)           | -13(2)          | 15(2)           | 6(2)            |
| N(2)  | 57(2)           | 46(2)           | 46(2)           | -7(1)           | 8(1)            | -3(1)           |
| C(5)  | 81(3)           | 49(2)           | 55(2)           | -4(2)           | 0(2)            | -13(2)          |
| C(6)  | 121(6)          | 109(5)          | 56(3)           | 9(3)            | 11(3)           | -39(4)          |
| C(7)  | 94(4)           | 65(3)           | 84(4)           | -9(3)           | -24(3)          | 13(3)           |
| C(8)  | 99(4)           | 49(2)           | 64(3)           | -8(2)           | -6(3)           | -5(2)           |
| C(9)  | 157(7)          | 72(4)           | 95(5)           | -32(4)          | 15(5)           | 17(4)           |
| C(10) | 119(5)          | 71(4)           | 115(5)          | 13(4)           | -9(4)           | -41(4)          |
| C(11) | 59(2)           | 39(2)           | 41(2)           | 2(1)            | 12(2)           | 8(2)            |
| C(12) | 56(2)           | 77(3)           | 54(2)           | 8(2)            | 7(2)            | 12(2)           |
| C(13) | 61(3)           | 84(3)           | 71(3)           | 17(2)           | 25(2)           | 7(2)            |
| C(14) | 84(3)           | 61(2)           | 50(2)           | 9(2)            | 21(2)           | 19(2)           |
| C(15) | 79(3)           | 82(3)           | 48(2)           | 8(2)            | -3(2)           | 8(2)            |
| C(16) | 66(3)           | 71(3)           | 53(2)           | 4(2)            | 3(2)            | 0(2)            |
| C(17) | 126(5)          | 127(5)          | 55(3)           | 28(3)           | 27(3)           | 27(5)           |
| C(18) | 62(2)           | 39(2)           | 32(2)           | 8(1)            | 5(2)            | 7(2)            |
| C(19) | 60(2)           | 44(2)           | 65(2)           | 6(2)            | 3(2)            | 2(2)            |
| C(20) | 61(3)           | 65(3)           | 68(3)           | 13(2)           | 10(2)           | 18(2)           |
| C(21) | 113(4)          | 46(2)           | 35(2)           | 8(2)            | 6(2)            | 25(2)           |
| C(22) | 117(4)          | 45(2)           | 53(2)           | -4(2)           | -19(2)          | -1(2)           |
| C(23) | 79(3)           | 51(2)           | 50(2)           | 5(2)            | -16(2)          | -1(2)           |
| C(24) | 178(8)          | 67(4)           | 65(3)           | 6(3)            | 5(5)            | 60(4)           |

Table 4. Positional ( $\times 10^4$ ) and isotropic thermal parameters ( $\times 10^3$ ) of the refined hydrogen atoms.

E.s.d.'s in parentheses

|        | x         | y         | z         | U(eq)   |
|--------|-----------|-----------|-----------|---------|
| HN1    | 2204(31)  | 646(25)   | 620(23)   | 36(10)  |
| H(1A)  | 2191(35)  | -929(31)  | 2192(27)  | 59(12)  |
| H(1B)  | 1310(42)  | -722(32)  | 1513(29)  | 75(14)  |
| H(2A)  | 3567(42)  | -30(33)   | 1806(31)  | 81(15)  |
| H(2B)  | 3151(40)  | -556(34)  | 1023(30)  | 78(15)  |
| H(3A)  | 4278(33)  | 968(24)   | 688(22)   | 44(9)   |
| H(3B)  | 3432(38)  | 1634(28)  | 209(27)   | 55(12)  |
| H(4A)  | 4157(31)  | 653(23)   | -683(22)  | 41(9)   |
| H(4B)  | 3707(36)  | -289(34)  | -149(26)  | 66(12)  |
| H(5)   | 2513(54)  | 1703(33)  | -989(39)  | 94(19)  |
| H(6A)  | 2591(40)  | 697(32)   | -2530(30) | 72(15)  |
| H(6B)  | 3724(59)  | 972(44)   | -2022(38) | 113(23) |
| H(6C)  | 2690(75)  | 1820(54)  | -2365(59) | 156(33) |
| H(7A)  | 600(47)   | 1401(38)  | -966(36)  | 91(17)  |
| H(7B)  | 618(51)   | 814(44)   | -1765(38) | 108(19) |
| H(7C)  | 925(50)   | 1863(37)  | -1932(37) | 95(18)  |
| H(8)   | 1618(42)  | -406(35)  | -1562(31) | 80(15)  |
| H(9A)  | 3355(56)  | -801(45)  | -1892(40) | 110(23) |
| H(9B)  | 3511(45)  | -1352(37) | -984(35)  | 78(16)  |
| H(9C)  | 2419(84)  | -1684(55) | -1590(57) | 163(34) |
| H(10A) | 1899(45)  | -1220(36) | -63(34)   | 81(16)  |
| H(10B) | 1048(56)  | -1552(45) | -817(40)  | 99(19)  |
| H(10C) | 871(84)   | -509(71)  | -627(61)  | 203(44) |
| H(12)  | -1002(45) | 1946(33)  | 3418(32)  | 77(15)  |
| H(13)  | -1265(53) | 1276(43)  | 4694(37)  | 101(19) |
| H(15)  | 2006(48)  | 1710(30)  | 5251(34)  | 79(16)  |
| H(16)  | 2254(61)  | 2284(50)  | 4025(43)  | 139(26) |
| H(19)  | -702(42)  | 3192(29)  | 472(29)   | 64(14)  |
| H(20)  | -1651(45) | 4547(34)  | 753(31)   | 80(15)  |
| H(22)  | 1178(46)  | 5726(39)  | 1572(34)  | 99(17)  |
| H(23)  | 2342(37)  | 4334(28)  | 1320(26)  | 63(12)  |
| H(24A) | -484(57)  | 6534(46)  | 1840(47)  | 107(21) |
| H(24B) | -1565(55) | 6145(48)  | 1070(42)  | 104(25) |
| H(24C) | -677(96)  | 6570(63)  | 1161(70)  | 171(46) |