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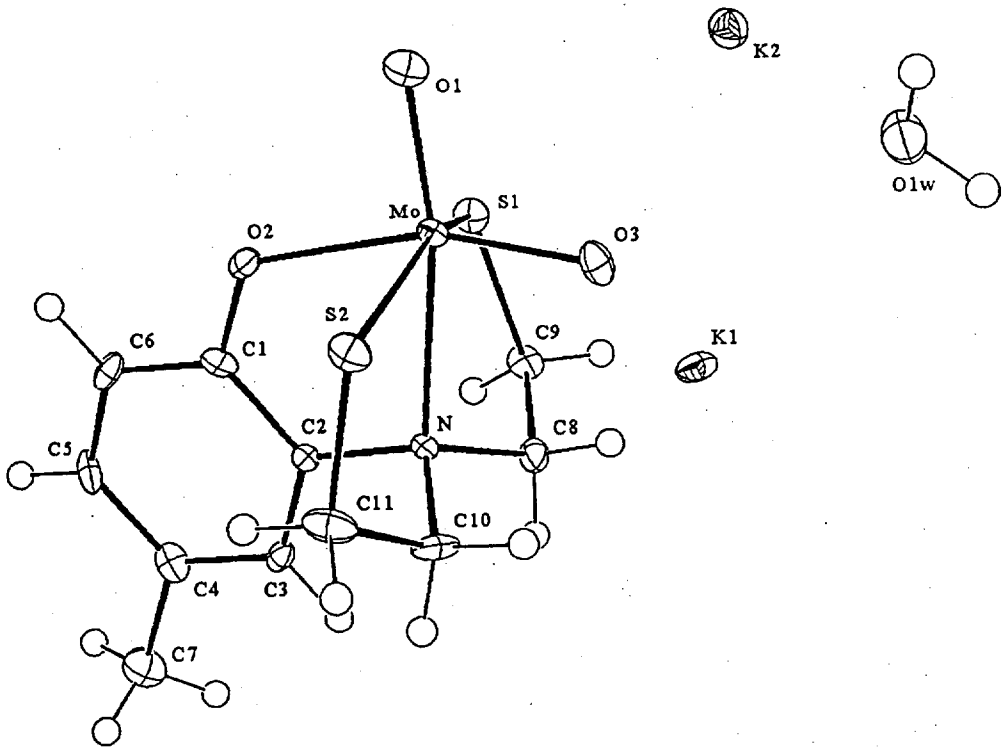
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**Figure S1.** ORTEP drawing of **1** with full numbering scheme.



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**Figure S2.** ORTEP drawing of **2** with full numbering scheme.

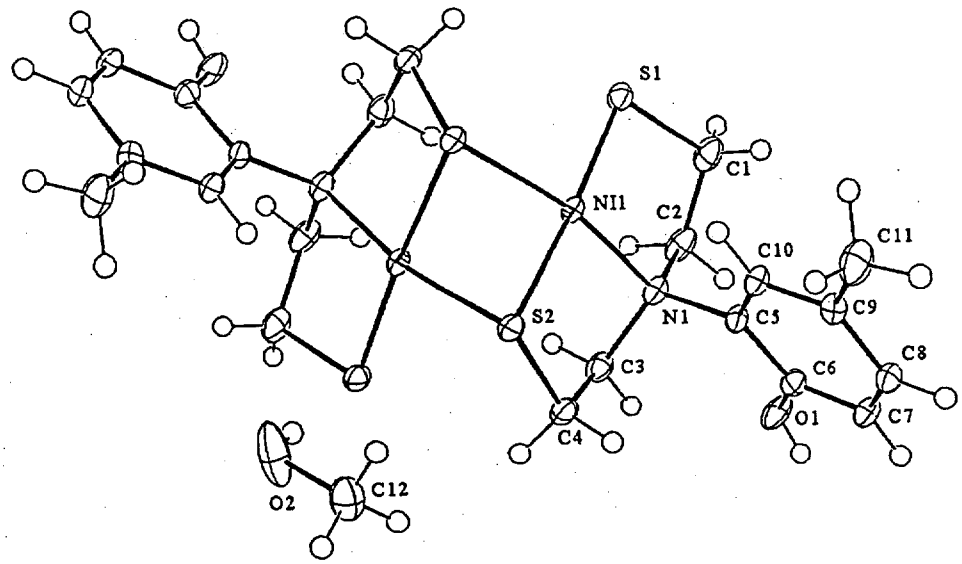


**Table 1.** Final atomic parameters x,y,z and $B_{iso}$ for **1**.

| | x | y | z | Biso |
|---|---|---|---|---|
| Mo | .03717( 8) | .33761( 4) | .176184(15) | .76( 2) |
| S1 | -.1026 ( 2) | .43579(12) | .12138 ( 5) | 1.16( 6) |
| S2 | .2556 ( 2) | .21073(12) | .20586 ( 4) | 1.11( 6) |
| O1 | -.1673 ( 7) | .3380 ( 3) | .20777 (12) | 1.38(17) |
| O2 | -.0694 ( 6) | .2217 ( 3) | .14189 (12) | 1.02(16) |
| O3 | .1931 ( 6) | .4296 ( 3) | .19428 (12) | 1.21(16) |
| N | .2715 ( 7) | .3172 ( 4) | .12209 (15) | .89(19) |
| C1 | .0066 ( 9) | .2037 ( 5) | .10494 (17) | .9 ( 2) |
| C2 | .1814 ( 9) | .2507 ( 4) | .09254 (17) | .8 ( 2) |
| C3 | .2673 ( 9) | .2313 ( 4) | .05512 (18) | .9 ( 2) |
| C4 | .1799 ( 9) | .1646 ( 5) | .02895 (15) | 1.3 ( 2) |
| C5 | .0043 (10) | .1191 ( 4) | .04052 (18) | 1.3 ( 3) |
| C6 | -.0848 ( 9) | .1380 ( 4) | .07821 (19) | 1.1 ( 2) |
| C7 | .2817 (10) | .1382 ( 5) | -.01160 (16) | 2.0 ( 3) |
| C8 | .3076 ( 9) | .4171 ( 5) | .10544 (18) | 1.2 ( 2) |
| C9 | .1151 (10) | .4624 ( 5) | .08913 (17) | 1.2 ( 2) |
| C10 | .4622 ( 9) | .2768 ( 5) | .13837 (18) | 1.2 ( 2) |
| C11 | .4230 (10) | .1865 ( 5) | .1629 ( 2) | 1.6 ( 3) |
| K1 | 1/2 | .39468(14) | 1/4 | .99(7) |
| K2 | 0 | .53984(14) | 1/4 | 1.27(8) |
| O1w | .3475(7) | .5483(4) | .29829(14) | 1.9(2) |
| H1wa | .454(7) | .584(5) | .2982(17) | 2.6 |
| H1wb | .317(9) | .538(5) | .3232(5) | 2.7 |
| H3 | .389 | .264 | .047 | 1.7 |
| H5 | -.059 | .074 | .022 | 2.1 |
| H6 | -.207 | .106 | .086 | 1.9 |
| H7a | .1817(18) | .117(3) | -.0307(5) | 2.9 |
| H7b | .377(5) | .087(2) | -.0073(4) | 2.9 |
| H7c | .350(6) | .1943(12) | -.0221(8) | 2.9 |
| H8a | .359 | .458 | .127 | 2.0 |
| H8b | .404 | .413 | .084 | 2.0 |
| H9a | .132 | .532 | .087 | 2.0 |
| H9b | .090 | .437 | .063 | 2.0 |
| H10a | .551 | .262 | .116 | 2.0 |
| H10b | .524 | .325 | .156 | 2.0 |
| H11a | .362 | .139 | .146 | 2.4 |
| H11b | -.048 | .662 | .173 | 2.4 |

Biso is the Mean of the Principal Axes of the Thermal Ellipsoid

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**Tabel S2.** Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters ($A^2$ x $10^3$) for **2**. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| Ni(1) | 5779(1) | 2337(1) | 6935(1) | 15(1) |
| S(1) | 7347(1) | 3044(1) | 7047(1) | 20(1) |
| S(2) | 4153(1) | 1655(1) | 6906(1) | 17(1) |
| N(1) | 5396(1) | 3033(1) | 5946(1) | 17(1) |
| O(1) | 4732(1) | 3722(1) | 4421(1) | 26(1) |
| O(2) | 8469(2) | 1053(1) | 6863(1) | 51(1) |
| C(1) | 7282(2) | 3661(2) | 6098(1) | 25(1) |
| C(2) | 6089(2) | 3900(1) | 5931(1) | 24(1) |
| C(3) | 4189(2) | 3277(2) | 6075(1) | 22(1) |
| C(4) | 3507(2) | 2412(2) | 6165(1) | 22(1) |
| C(5) | 5536(2) | 2511(1) | 5199(1) | 16(1) |
| C(6) | 5171(2) | 2853(1) | 4461(1) | 19(1) |
| C(7) | 5276(2) | 2301(2) | 3785(1) | 22(1) |
| C(8) | 5748(2) | 1431(2) | 3833(1) | 21(1) |
| C(9) | 6152(2) | 1092(1) | 4551(1) | 19(1) |
| C(10) | 6036(2) | 1644(1) | 5228(1) | 17(1) |
| C(11) | 6686(2) | 156(2) | 4611(1) | 28(1) |
| C(12) | 8461(2) | 607(2) | 7610(2) | 31(1) |
| | | | | |
| H(1A) | 7713(21) | 4208(18) | 6156(14) | 29(7) |
| H(1B) | 7600(19) | 3266(16) | 5692(14) | 19(6) |
| H(2A) | 5772(19) | 4313(16) | 6360(15) | 24(6) |
| H(2B) | 5984(19) | 4197(16) | 5423(14) | 19(6) |
| H(3A) | 4163(18) | 3662(16) | 6564(14) | 19(6) |
| H(3B) | 3919(18) | 3654(15) | 5645(13) | 16(5) |
| H(4A) | 3473(19) | 2028(16) | 5669(15) | 25(6) |
| H(4B) | 2784(20) | 2538(15) | 6338(14) | 20(6) |
| H(7) | 5000(20) | 2504(15) | 3329(14) | 21(6) |
| H(8) | 5831(19) | 1060(16) | 3365(15) | 24(6) |
| H(10) | 6314(17) | 1418(14) | 5721(13) | 13(5) |
| H(11A) | 6243(24) | -272(19) | 4867(17) | 41(8) |
| H(11B) | 7362(24) | 180(18) | 4936(17) | 41(8) |
| H(11C) | 6868(25) | -74(22) | 4122(20) | 56(9) |
| H(1O) | 4415(22) | 3768(19) | 4043(16) | 31(8) |
| H(12A) | 9039(28) | 158(24) | 7639(20) | 66(10) |
| H(12B) | 7788(29) | 284(22) | 7657(20) | 63(10) |
| H(12C) | 8469(29) | 958(24) | 8057(21) | 66(11) |
| H(2O) | 8186(31) | 1472(25) | 6881(22) | 65(12) |

**Table S3.** Bond distances and angles for **1**.

| | | | |
|---|---|---|---|
| Mo-S1 | 2.4406(16) | K1-S2c | 3.341(2) |
| Mo-S2 | 2.4729(16) | K1-O1d | 2.731(4) |
| Mo-O1 | 1.715(4) | K1-O1e | 2.731(4) |
| Mo-O2 | 2.079(4) | K1-O3c | 2.790(4) |
| Mo-O3 | 1.742(4) | K1-K2d | 3.8818(15) |
| Mo-N | 2.387(5) | K1-K2 | 3.8818(15) |
| Mo-K1 | 4.0058(7) | K1-K2a | 4.877(3) |
| Mo-K2 | 3.7038(15) | K1-O1w | 2.833(5) |
| S1-C9 | 1.835(6) | K1-O1wc | 2.833(5) |
| S2-C11 | 1.834(7) | K1-H1wa | 3.06(6) |
| S2-K1 | 3.341(2) | K1-H1wac | 3.06(6) |
| S2-K2a | 3.207(2) | K1-H1wb | 3.35(5) |
| O1-K1b | 2.731(4) | K1-H1wbc | 3.35(5) |
| O1-K2 | 3.299(5) | K1-H10b | 3.2665(6) |
| O2-C1 | 1.343(7) | K1-H10bc | 3.2665(6) |
| O3-K1 | 2.790(4) | K2-Moe | 3.7038(15) |
| O3-K2 | 2.707(4) | K2-S2f | 3.207(2) |
| N-C2 | 1.466(8) | K2-S2g | 3.207(2) |
| N-C8 | 1.498(8) | K2-O1e | 3.299(5) |
| N-C10 | 1.487(8) | K2-O3e | 2.707(4) |
| C1-C2 | 1.393(9) | K2-C11f | 3.548(7) |
| C1-C6 | 1.401(9) | K2-C11g | 3.548(7) |
| C2-C3 | 1.387(8) | K2-K1b | 3.8818(15) |
| C3-C4 | 1.388(8) | K2-K1f | 4.877(3) |
| C3-H3 | .960(6) | K2-O1w | 2.812(5) |
| C4-C5 | 1.380(9) | K2-O1we | 2.812(5) |
| C4-C7 | 1.543(8) | K2-H1wa | 3.47(5) |
| C5-C6 | 1.402(9) | K2-H1wae | 3.47(5) |
| C5-H5 | .960(6) | K2-H1wb | 3.21(4) |
| C6-H6 | .960(6) | K2-H1wbe | 3.21(4) |
| C7-H7a | .96(2) | K2-H11b | 3.0601(11) |
| C7-H7b | .96(4) | K2-H11be | 3.0601(11) |
| C7-H7c | .96(3) | O1w-H1wa | .86(5) |
| C8-C9 | 1.523(9) | O1w-H1wb | .86(3) |
| C8-H8a | .960(6) | H1wa-H1wb | 1.38(8) |
| C8-H8b | .960(6) | H7a-H7b | 1.57(4) |
| C9-H9a | .960(7) | H7a-H7c | 1.57(5) |
| C9-H9b | .960(6) | H7b-H7c | 1.57(4) |
| C10-C11 | 1.504(9) | H8a-H8b | 1.56763(8) |
| C10-H10a | .960(6) | H9a-H9b | 1.56763(8) |
| C10-H10b | .960(6) | H10a-H10b | 1.56763(7) |
| C11-K2a | 3.548(7) | H11a-H11ba | 1.56975(14) |
| C11-H11a | .960(7) | H11b-C11f | .962(6) |
| C11-H11ba | .962(6) | H11b-H11af | 1.56975(14) |
| K1-Moc | 4.0058(7) | | |

| | | | |
|---|---|---|---|
| S1-Mo-S2 | 155.17(6) | O1wc-K1-H1wa | 72.3(11) |
| S1-Mo-O1 | 98.38(15) | O1wc-K1-H1wac | 16.2(9) |
| S1-Mo-O2 | 83.71(12) | O1wc-K1-H1wb | 95.6(11) |
| S1-Mo-O3 | 94.58(14) | O1wc-K1-H1wbc | 12.8(10) |
| S1-Mo-N | 76.13(13) | O1wc-K1-H10b | 70.47(10) |
| S1-Mo-K1 | 129.46(5) | O1wc-K1-H10bc | 140.70(11) |
| S1-Mo-K2 | 92.67(4) | H1wa-K1-H1wac | 63.8(15) |
| S2-Mo-O1 | 103.21(15) | H1wa-K1-H1wb | 24.4(15) |
| S2-Mo-O2 | 82.86(12) | H1wa-K1-H1wbc | 84.9(15) |
| S2-Mo-O3 | 91.47(14) | H1wa-K1-H10b | 138.6(11) |
| S2-Mo-N | 80.14(13) | H1wa-K1-H10bc | 75.5(11) |

**Table S3.** Bond distances and angles for **1**. (continued)

| | |
|---|---|
| S2-Mo-K1 | 56.28(5) |
| S2-Mo-K2 | 107.95(4) |
| O1-Mo-O2 | 93.54(19) |
| O1-Mo-O3 | 105.3(2) |
| O1-Mo-N | 166.86(19) |
| O1-Mo-K1 | 103.95(14) |
| O1-Mo-K2 | 62.95(15) |
| O2-Mo-O3 | 161.16(18) |
| O2-Mo-N | 74.13(16) |
| O2-Mo-K1 | 138.04(12) |
| O2-Mo-K2 | 155.52(11) |
| O3-Mo-N | 87.23(18) |
| O3-Mo-K1 | 35.97(14) |
| O3-Mo-K2 | 43.09(14) |
| N-Mo-K1 | 88.54(12) |
| N-Mo-K2 | 128.55(12) |
| K1-Mo-K2 | 60.31(2) |
| Mo-S1-C9 | 103.8(2) |
| Mo-S2-C11 | 100.4(2) |
| Mo-S2-K1 | 85.72(5) |
| Mo-S2-K2a | 174.09(7) |
| C11-S2-K1 | 100.3(2) |
| C11-S2-K2a | 84.8(2) |
| K1-S2-K2a | 96.26(5) |
| Mo-O1-K1b | 162.8(2) |
| Mo-O1-K2 | 89.47(17) |
| K1b-O1-K2 | 79.54(11) |
| Mo-O2-C1 | 120.4(4) |
| Mo-O3-K1 | 122.5(2) |
| Mo-O3-K2 | 110.8(2) |
| K1-O3-K2 | 89.82(12) |
| Mo-N-C2 | 107.6(3) |
| Mo-N-C8 | 105.8(3) |
| Mo-N-C10 | 109.4(3) |
| C2-N-C8 | 113.2(5) |
| C2-N-C10 | 110.9(5) |
| C8-N-C10 | 109.7(5) |
| O2-C1-C2 | 119.7(5) |
| O2-C1-C6 | 121.8(5) |
| C2-C1-C6 | 118.5(5) |
| N-C2-C1 | 115.9(5) |
| N-C2-C3 | 122.9(5) |
| C1-C2-C3 | 121.2(6) |
| C2-C3-C4 | 120.5(6) |
| C2-C3-H3 | 119.4(6) |
| C4-C3-H3 | 120.1(6) |
| C3-C4-C5 | 118.9(5) |
| C3-C4-C7 | 120.7(6) |
| C5-C4-C7 | 120.4(5) |
| C4-C5-C6 | 121.4(5) |
| C4-C5-H5 | 119.3(6) |
| C6-C5-H5 | 119.3(6) |
| C1-C6-C5 | 119.6(5) |
| C1-C6-H6 | 119.9(6) |
| C5-C6-H6 | 120.5(6) |
| C4-C7-H7a | 109.5(11) |
| C4-C7-H7b | 109.5(10) |
| C4-C7-H7c | 109.5(16) |
| H7a-C7-H7b | 109(3) |
| H7a-C7-H7c | 109(3) |
| H1wac-K1-H1wb | 84.9(15) |
| H1wac-K1-H1wbc | 24.4(15) |
| H1wac-K1-H10b | 75.5(11) |
| H1wac-K1-H10bc | 138.6(11) |
| H1wb-K1-H1wbc | 107.9(15) |
| H1wb-K1-H10b | 151.6(9) |
| H1wb-K1-H10bc | 57.8(9) |
| H1wbc-K1-H10b | 57.8(9) |
| H1wbc-K1-H10bc | 151.6(9) |
| H10b-K1-H10bc | 145.71(7) |
| Mo-K2-Moe | 82.74(4) |
| Mo-K2-S2f | 102.54(3) |
| Mo-K2-S2g | 151.89(3) |
| Mo-K2-O1 | 27.58(7) |
| Mo-K2-O1e | 67.91(8) |
| Mo-K2-O3 | 26.08(9) |
| Mo-K2-O3e | 93.34(10) |
| Mo-K2-C11f | 83.34(11) |
| Mo-K2-C11g | 165.63(12) |
| Mo-K2-K1 | 63.70(3) |
| Mo-K2-K1b | 70.83(3) |
| Mo-K2-K1f | 138.63(2) |
| Mo-K2-O1w | 110.40(11) |
| Mo-K2-O1we | 73.36(10) |
| Mo-K2-H1wa | 112.0(10) |
| Mo-K2-H1wae | 83.6(10) |
| Mo-K2-H1wb | 116.5(12) |
| Mo-K2-H1wbe | 62.8(12) |
| Mo-K2-H11b | 82.647(14) |
| Moe-K2-S2f | 151.89(3) |
| Moe-K2-S2g | 102.54(3) |
| Moe-K2-O1 | 67.91(8) |
| Moe-K2-O1e | 27.58(7) |
| Moe-K2-O3 | 93.34(10) |
| Moe-K2-O3e | 26.08(9) |
| Moe-K2-C11f | 165.63(12) |
| Moe-K2-C11g | 83.34(11) |
| Moe-K2-K1 | 70.83(3) |
| Moe-K2-K1b | 63.70(3) |
| Moe-K2-K1f | 138.63(2) |
| Moe-K2-O1w | 73.36(10) |
| Moe-K2-O1we | 110.40(11) |
| Moe-K2-H1wa | 83.6(10) |
| Moe-K2-H1wae | 112.0(10) |
| Moe-K2-H1wb | 62.8(12) |
| Moe-K2-H1wbe | 116.5(12) |
| Moe-K2-H11b | 161.74(4) |
| S2f-K2-S2g | 85.84(6) |
| S2f-K2-O1 | 126.56(8) |
| S2f-K2-O1e | 129.52(8) |
| S2f-K2-O3 | 81.97(10) |
| S2f-K2-O3e | 163.59(10) |
| S2f-K2-C11f | 30.99(11) |
| S2f-K2-C11g | 91.44(13) |
| S2f-K2-K1 | 86.62(4) |
| S2f-K2-K1b | 144.26(4) |
| S2f-K2-K1f | 42.92(4) |
| S2f-K2-O1w | 79.00(11) |
| S2f-K2-O1we | 97.47(11) |
| S2f-K2-H1wa | 68.8(10) |

**Table S3.** Bond distances and angles for **1**. (continued)

| | | | |
|---|---|---|---|
| H7b-C7-H7c | 109(3) | S2f-K2-H1wae | 96.1(10) |
| N-C8-C9 | 111.6(5) | S2f-K2-H1wb | 90.8(11) |
| N-C8-H8a | 109.0(5) | S2f-K2-H1wbe | 89.8(11) |
| N-C8-H8b | 108.8(5) | S2f-K2-H11b | 43.58(3) |
| C9-C8-H8a | 108.7(6) | S2g-K2-O1 | 129.52(8) |
| C9-C8-H8b | 109.2(5) | S2g-K2-O1e | 126.56(8) |
| H8a-C8-H8b | 109.5(6) | S2g-K2-O3 | 163.59(10) |
| S1-C9-C8 | 112.3(4) | S2g-K2-O3e | 81.97(10) |
| S1-C9-H9a | 108.9(5) | S2g-K2-C11f | 91.44(13) |
| S1-C9-H9b | 108.6(5) | S2g-K2-C11g | 30.99(11) |
| C8-C9-H9a | 109.1(6) | S2g-K2-K1 | 144.26(4) |
| C8-C9-H9b | 108.5(6) | S2g-K2-K1b | 86.62(4) |
| H9a-C9-H9b | 109.5(6) | S2g-K2-K1f | 42.92(4) |
| N-C10-C11 | 110.8(5) | S2g-K2-O1w | 97.47(11) |
| N-C10-H10a | 109.3(5) | S2g-K2-O1we | 79.00(11) |
| N-C10-H10b | 108.8(6) | S2g-K2-H1wa | 96.1(10) |
| C11-C10-H10a | 109.6(6) | S2g-K2-H1wae | 68.8(10) |
| C11-C10-H10b | 108.8(5) | S2g-K2-H1wb | 89.8(11) |
| H10a-C10-H10b | 109.5(6) | S2g-K2-H1wbe | 90.8(11) |
| S2-C11-C10 | 111.8(4) | S2g-K2-H11b | 85.56(5) |
| S2-C11-K2a | 64.2(2) | O1-K2-O1e | 65.48(11) |
| S2-C11-H11a | 108.9(5) | O1-K2-O3 | 53.35(12) |
| S2-C11-H11ba | 108.9(5) | O1-K2-O3e | 69.85(12) |
| C10-C11-K2a | 151.6(4) | O1-K2-C11f | 100.64(13) |
| C10-C11-H11a | 108.5(6) | O1-K2-C11g | 140.47(14) |
| C10-C11-H11ba | 109.2(6) | O1-K2-K1 | 81.80(9) |
| K2a-C11-H11a | 98.9(4) | O1-K2-K1b | 43.77(8) |
| K2a-C11-H11ba | 52.6(3) | O1-K2-K1f | 147.26(8) |
| H11a-C11-H11ba | 109.6(6) | O1-K2-O1w | 123.51(14) |
| Mo-K1-Moc | 157.41(6) | O1-K2-O1we | 61.16(13) |
| Mo-K1-S2 | 38.00(3) | O1-K2-H1wa | 129.4(11) |
| Mo-K1-S2c | 119.45(6) | O1-K2-H1wae | 70.1(11) |
| Mo-K1-O1d | 104.96(9) | O1-K2-H1wb | 122.3(13) |
| Mo-K1-O1e | 68.28(9) | O1-K2-H1wbe | 56.9(13) |
| Mo-K1-O3 | 21.51(9) | O1-K2-H11b | 94.26(7) |
| Mo-K1-O3c | 176.15(9) | O1e-K2-O3 | 69.85(12) |
| Mo-K1-K2d | 139.52(3) | O1e-K2-O3e | 53.35(12) |
| Mo-K1-K2 | 55.99(2) | O1e-K2-C11f | 140.47(14) |
| Mo-K1-K2a | 78.71(3) | O1e-K2-C11g | 100.64(13) |
| Mo-K1-O1w | 102.24(10) | O1e-K2-K1 | 43.77(8) |
| Mo-K1-O1wc | 94.58(10) | O1e-K2-K1b | 81.80(9) |
| Mo-K1-H1wa | 113.8(9) | O1e-K2-K1f | 147.26(8) |
| Mo-K1-H1wac | 85.9(9) | O1e-K2-O1w | 61.16(13) |
| Mo-K1-H1wb | 105.9(10) | O1e-K2-O1we | 123.51(14) |
| Mo-K1-H1wbc | 87.6(9) | O1e-K2-H1wa | 70.1(11) |
| Mo-K1-H10b | 53.116(11) | O1e-K2-H1wae | 129.4(11) |
| Mo-K1-H10bc | 118.99(3) | O1e-K2-H1wb | 56.9(13) |
| Moc-K1-S2 | 119.45(6) | O1e-K2-H1wbe | 122.3(13) |
| Moc-K1-S2c | 38.00(3) | O1e-K2-H11b | 147.85(8) |
| Moc-K1-O1d | 68.28(9) | O3-K2-O3e | 111.94(15) |
| Moc-K1-O1e | 104.96(9) | O3-K2-C11f | 72.49(15) |
| Moc-K1-O3 | 176.15(9) | O3-K2-C11g | 159.51(14) |
| Moc-K1-O3c | 21.51(9) | O3-K2-K1 | 45.96(9) |
| Moc-K1-K2d | 55.99(2) | O3-K2-K1b | 96.89(10) |
| Moc-K1-K2 | 139.52(3) | O3-K2-K1f | 124.03(10) |
| Moc-K1-K2a | 78.71(3) | O3-K2-O1w | 90.97(13) |
| Moc-K1-O1w | 94.58(10) | O3-K2-O1we | 91.69(13) |
| Moc-K1-O1wc | 102.24(10) | O3-K2-H1wa | 89.7(9) |
| Moc-K1-H1wa | 85.9(9) | O3-K2-H1wae | 101.6(10) |
| Moc-K1-H1wac | 113.8(9) | O3-K2-H1wb | 101.3(10) |

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**Table S3.** Bond distances and angles for **1**. (continued)

| | | | |
|---|---|---|---|
| Moc-K1-H1wb | 87.6(9) | O3-K2-H1wbe | 78.3(10) |
| Moc-K1-H1wbc | 105.9(10) | O3-K2-H11b | 78.03(9) |
| Moc-K1-H10b | 118.99(3) | O3e-K2-C11f | 159.51(14) |
| Moc-K1-H10bc | 53.116(11) | O3e-K2-C11g | 72.49(15) |
| S2-K1-S2c | 81.65(6) | O3e-K2-K1 | 96.89(10) |
| S2-K1-O1d | 87.53(11) | O3e-K2-K1b | 45.96(9) |
| S2-K1-O1e | 67.11(10) | O3e-K2-K1f | 124.03(10) |
| S2-K1-O3 | 59.09(9) | O3e-K2-O1w | 91.69(13) |
| S2-K1-O3c | 140.73(11) | O3e-K2-O1we | 90.97(13) |
| S2-K1-K2d | 143.79(4) | O3e-K2-H1wa | 101.6(10) |
| S2-K1-K2 | 88.34(4) | O3e-K2-H1wae | 89.7(9) |
| S2-K1-K2a | 40.82(3) | O3e-K2-H1wb | 78.3(10) |
| S2-K1-O1w | 129.38(10) | O3e-K2-H1wbe | 101.3(10) |
| S2-K1-O1wc | 119.57(10) | O3e-K2-H11b | 145.09(9) |
| S2-K1-H1wa | 145.0(9) | C11f-K2-C11g | 110.75(17) |
| S2-K1-H1wac | 117.7(10) | C11f-K2-K1 | 99.68(11) |
| S2-K1-H1wb | 125.6(11) | C11f-K2-K1b | 114.58(10) |
| S2-K1-H1wbc | 108.0(9) | C11f-K2-K1f | 55.38(11) |
| S2-K1-H10b | 52.03(3) | C11f-K2-O1w | 108.45(15) |
| S2-K1-H10bc | 99.73(5) | C11f-K2-O1we | 68.68(14) |
| S2c-K1-O1d | 67.11(10) | C11f-K2-H1wa | 98.4(10) |
| S2c-K1-O1e | 87.53(11) | C11f-K2-H1wae | 69.8(9) |
| S2c-K1-O3 | 140.73(11) | C11f-K2-H1wb | 121.3(11) |
| S2c-K1-O3c | 59.09(9) | C11f-K2-H1wbe | 59.2(11) |
| S2c-K1-K2d | 88.34(4) | C11f-K2-H11b | 14.45(11) |
| S2c-K1-K2 | 143.79(4) | C11g-K2-K1 | 114.58(10) |
| S2c-K1-K2a | 40.82(3) | C11g-K2-K1b | 99.68(11) |
| S2c-K1-O1w | 119.57(10) | C11g-K2-K1f | 55.38(11) |
| S2c-K1-O1wc | 129.38(10) | C11g-K2-O1w | 68.68(14) |
| S2c-K1-H1wa | 117.7(10) | C11g-K2-O1we | 108.45(15) |
| S2c-K1-H1wac | 145.0(9) | C11g-K2-H1wa | 69.8(9) |
| S2c-K1-H1wb | 108.0(9) | C11g-K2-H1wae | 98.4(10) |
| S2c-K1-H1wbc | 125.6(11) | C11g-K2-H1wb | 59.2(11) |
| S2c-K1-H10b | 99.73(5) | C11g-K2-H1wbe | 121.3(11) |
| S2c-K1-H10bc | 52.03(3) | C11g-K2-H11b | 110.26(12) |
| O1d-K1-O1e | 146.83(16) | K1-K2-K1b | 118.14(6) |
| O1d-K1-O3 | 107.89(12) | K1-K2-K1f | 120.93(4) |
| O1d-K1-O3c | 77.94(13) | K1-K2-O1w | 46.79(10) |
| O1d-K1-K2d | 56.69(10) | K1-K2-O1we | 136.66(11) |
| O1d-K1-K2 | 147.42(9) | K1-K2-H1wa | 48.9(10) |
| O1d-K1-K2a | 73.41(10) | K1-K2-H1wae | 146.8(10) |
| O1d-K1-O1w | 142.22(15) | K1-K2-H1wb | 55.4(10) |
| O1d-K1-O1wc | 68.60(14) | K1-K2-H1wbe | 124.1(11) |
| O1d-K1-H1wa | 126.1(9) | K1-K2-H11b | 111.822(11) |
| O1d-K1-H1wac | 84.0(10) | K1b-K2-K1f | 120.93(4) |
| O1d-K1-H1wb | 146.3(11) | K1b-K2-O1w | 136.66(11) |
| O1d-K1-H1wbc | 60.3(11) | K1b-K2-O1we | 46.79(10) |
| O1d-K1-H10b | 52.35(9) | K1b-K2-H1wa | 146.8(10) |
| O1d-K1-H10bc | 116.26(10) | K1b-K2-H1wae | 48.9(10) |
| O1e-K1-O3 | 77.94(13) | K1b-K2-H1wb | 124.1(11) |
| O1e-K1-O3c | 107.89(12) | K1b-K2-H1wbe | 55.4(10) |
| O1e-K1-K2d | 147.42(9) | K1b-K2-H11b | 101.013(14) |
| O1e-K1-K2 | 56.69(10) | K1f-K2-O1w | 87.62(11) |
| O1e-K1-K2a | 73.41(10) | K1f-K2-O1we | 87.62(11) |
| O1e-K1-O1w | 68.60(14) | K1f-K2-H1wa | 79.9(11) |
| O1e-K1-O1wc | 142.22(15) | K1f-K2-H1wae | 79.9(11) |
| O1e-K1-H1wa | 84.0(10) | K1f-K2-H1wb | 90.4(13) |
| O1e-K1-H1wac | 126.1(9) | K1f-K2-H1wbe | 90.4(13) |
| O1e-K1-H1wb | 60.3(11) | K1f-K2-H11b | 56.81(3) |
| O1e-K1-H1wbc | 146.3(11) | O1w-K2-O1we | 175.25(16) |

**Table S3.** Bond distances and angles for **1**. (continued)

| | |
|---|---|
| O1e-K1-H10b | 116.26(10) |
| O1e-K1-H10bc | 52.35(9) |
| O3-K1-O3c | 160.18(15) |
| O3-K1-K2d | 122.67(10) |
| O3-K1-K2 | 44.22(9) |
| O3-K1-K2a | 99.91(10) |
| O3-K1-O1w | 88.83(13) |
| O3-K1-O1wc | 76.34(14) |
| O3-K1-H1wa | 97.0(10) |
| O3-K1-H1wac | 65.5(9) |
| O3-K1-H1wb | 96.1(9) |
| O3-K1-H1wbc | 72.0(10) |
| O3-K1-H10b | 57.18(9) |
| O3-K1-H10bc | 130.06(9) |
| O3c-K1-K2d | 44.22(9) |
| O3c-K1-K2 | 122.67(10) |
| O3c-K1-K2a | 99.91(10) |
| O3c-K1-O1w | 76.34(14) |
| O3c-K1-O1wc | 88.83(13) |
| O3c-K1-H1wa | 65.5(9) |
| O3c-K1-H1wac | 97.0(10) |
| O3c-K1-H1wb | 72.0(10) |
| O3c-K1-H1wbc | 96.1(9) |
| O3c-K1-H10b | 130.06(9) |
| O3c-K1-H10bc | 57.18(9) |
| K2d-K1-K2 | 118.14(6) |
| K2d-K1-K2a | 120.93(4) |
| K2d-K1-O1w | 85.66(11) |
| K2d-K1-O1wc | 46.33(10) |
| K2d-K1-H1wa | 69.5(9) |
| K2d-K1-H1wac | 58.5(9) |
| K2d-K1-H1wb | 90.6(11) |
| K2d-K1-H1wbc | 52.0(9) |
| K2d-K1-H10b | 96.287(12) |
| K2d-K1-H10bc | 101.161(12) |
| K2-K1-K2a | 120.93(4) |
| K2-K1-O1w | 46.33(10) |
| K2-K1-O1wc | 85.66(11) |
| K2-K1-H1wa | 58.5(9) |
| K2-K1-H1wac | 69.5(9) |
| K2-K1-H1wb | 52.0(9) |
| K2-K1-H1wbc | 90.6(11) |
| K2-K1-H10b | 101.161(12) |
| K2-K1-H10bc | 96.287(12) |
| K2a-K1-O1w | 138.18(10) |
| K2a-K1-O1wc | 138.18(10) |
| K2a-K1-H1wa | 148.1(11) |
| K2a-K1-H1wac | 148.1(11) |
| K2a-K1-H1wb | 126.1(11) |
| K2a-K1-H1wbc | 126.1(11) |
| K2a-K1-H10b | 72.85(3) |
| K2a-K1-H10bc | 72.85(3) |
| O1w-K1-O1wc | 83.63(15) |
| O1w-K1-H1wa | 16.2(9) |
| O1w-K1-H1wac | 72.3(11) |
| O1w-K1-H1wb | 12.8(10) |
| O1w-K1-H1wbc | 95.6(11) |
| O1w-K1-H10b | 140.70(11) |
| O1w-K1-H10bc | 70.47(10) |
| O1w-K2-H1wa | 10.2(10) |
| O1w-K2-H1wae | 165.8(10) |
| O1w-K2-H1wb | 14.6(9) |
| O1w-K2-H1wbe | 165.5(9) |
| O1w-K2-H11b | 122.30(11) |
| O1we-K2-H1wa | 165.8(10) |
| O1we-K2-H1wae | 10.2(10) |
| O1we-K2-H1wb | 165.5(9) |
| O1we-K2-H1wbe | 14.6(9) |
| O1we-K2-H11b | 54.57(10) |
| H1wa-K2-H1wae | 159.9(15) |
| H1wa-K2-H1wb | 23.5(13) |
| H1wa-K2-H1wbe | 156.9(13) |
| H1wa-K2-H11b | 112.1(10) |
| H1wae-K2-H1wb | 156.9(13) |
| H1wae-K2-H1wbe | 23.5(13) |
| H1wae-K2-H11b | 55.4(9) |
| H1wb-K2-H1wbe | 179.2(18) |
| H1wb-K2-H11b | 134.3(11) |
| H1wbe-K2-H11b | 46.3(11) |
| K1-O1w-K2 | 86.88(14) |
| K1-O1w-H1wa | 97(4) |
| K1-O1w-H1wb | 120(5) |
| K2-O1w-H1wa | 134(4) |
| K2-O1w-H1wb | 109(4) |
| H1wa-O1w-H1wb | 107(6) |
| K1-H1wa-K2 | 72.6(12) |
| K1-H1wa-O1w | 66(4) |
| K1-H1wa-H1wb | 89(4) |
| K2-H1wa-O1w | 35(3) |
| K2-H1wa-H1wb | 67(3) |
| O1w-H1wa-H1wb | 36(3) |
| K1-H1wb-K2 | 72.6(7) |
| K1-H1wb-O1w | 46(4) |
| K1-H1wb-H1wa | 66(3) |
| K2-H1wb-O1w | 55(3) |
| K2-H1wb-H1wa | 88(3) |
| O1w-H1wb-H1wa | 36(3) |
| C7-H7a-H7b | 35.3(16) |
| C7-H7a-H7c | 35.3(17) |
| H7b-H7a-H7c | 60.0(17) |
| C7-H7b-H7a | 35(2) |
| C7-H7b-H7c | 35(2) |
| H7a-H7b-H7c | 60(2) |
| C7-H7c-H7a | 35.3(16) |
| C7-H7c-H7b | 35.3(16) |
| H7a-H7c-H7b | 60(2) |
| C8-H8a-H8b | 35.3(4) |
| C8-H8b-H8a | 35.3(4) |
| C9-H9a-H9b | 35.3(3) |
| C9-H9b-H9a | 35.3(4) |
| C10-H10a-H10b | 35.3(4) |
| C10-H10b-K1 | 138.0(4) |
| C10-H10b-H10a | 35.3(4) |
| K1-H10b-H10a | 163.08(3) |
| C11-H11a-H11ba | 35.3(4) |
| C11f-H11b-K2 | 113.0(4) |
| C11f-H11b-H11af | 35.2(4) |
| K2-H11b-H11af | 106.543(14) |

**Table S4.** Bond distances and angles for **2**.

| | | | |
|---|---|---|---|
| Ni(1)-N(1) | 1.997(2) | O(1)-C(6) | 1.367(2) |
| Ni(1)-S(1) | 2.1619(6) | O(2)-C(12) | 1.409(3) |
| Ni(1)-S(2)#1 | 2.1824(5) | C(1)-C(2) | 1.509(3) |
| Ni(1)-S(2) | 2.2006(5) | C(3)-C(4) | 1.508(3) |
| Ni(1)-Ni(1)#1 | 2.6733(5) | C(5)-C(10) | 1.396(3) |
| S(1)-C(1) | 1.828(2) | C(5)-C(6) | 1.405(3) |
| S(2)-C(4) | 1.832(2) | C(6)-C(7) | 1.394(3) |
| S(2)-Ni(1)#1 | 2.1823(5) | C(7)-C(8) | 1.386(3) |
| N(1)-C(5) | 1.474(2) | C(8)-C(9) | 1.389(3) |
| N(1)-C(2) | 1.510(3) | C(9)-C(10) | 1.396(3) |
| N(1)-C(3) | 1.516(3) | C(9)-C(11) | 1.506(3) |

| | | | |
|---|---|---|---|
| N(1)-Ni(1)-S(1) | 92.07(5) | C(2)-N(1)-Ni(1) | 107.79(12) |
| N(1)-Ni(1)-S(2)#1 | 167.91(5) | C(3)-N(1)-Ni(1) | 102.83(12) |
| S(1)-Ni(1)-S(2)#1 | 95.98(2) | C(2)-C(1)-S(1) | 108.4(2) |
| N(1)-Ni(1)-S(2) | 90.08(5) | C(1)-C(2)-N(1) | 109.6(2) |
| S(1)-Ni(1)-S(2) | 175.86(2) | C(4)-C(3)-N(1) | 110.3(2) |
| S(2)#1-Ni(1)-S(2) | 81.36(2) | C(3)-C(4)-S(2) | 109.44(14) |
| N(1)-Ni(1)-Ni(1)#1 | 115.19(5) | C(10)-C(5)-C(6) | 119.1(2) |
| S(1)-Ni(1)-Ni(1)#1 | 123.75(2) | C(10)-C(5)-N(1) | 118.9(2) |
| S(2)#1-Ni(1)-Ni(1)#1 | 52.73(2) | C(6)-C(5)-N(1) | 122.1(2) |
| S(2)-Ni(1)-Ni(1)#1 | 52.106(14) | O(1)-C(6)-C(7) | 121.7(2) |
| C(1)-S(1)-Ni(1) | 96.82(8) | O(1)-C(6)-C(5) | 119.3(2) |
| C(4)-S(2)-Ni(1)#1 | 110.47(7) | C(7)-C(6)-C(5) | 119.0(2) |
| C(4)-S(2)-Ni(1) | 97.26(7) | C(8)-C(7)-C(6) | 120.8(2) |
| Ni(1)#1-S(2)-Ni(1) | 75.17(2) | C(7)-C(8)-C(9) | 121.1(2) |
| C(5)-N(1)-C(2) | 110.5(2) | C(8)-C(9)-C(10) | 117.9(2) |
| C(5)-N(1)-C(3) | 110.6(2) | C(8)-C(9)-C(11) | 121.8(2) |
| C(2)-N(1)-C(3) | 110.0(2) | C(10)-C(9)-C(11) | 120.3(2) |
| C(5)-N(1)-Ni(1) | 114.79(12) | C(5)-C(10)-C(9) | 122.0(2) |

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

**Table S5.** u(i,j) or U values *100. E.S.Ds. refer to the last digit printed.

| | u11(U) | u22 | u33 | u12 | u13 | u23 |
|---|---|---|---|---|---|---|
| Mo | .97( 3) | .94( 3) | .97(3) | .21( 2) | .04( 2) | -.07( 2) |
| S1 | 1.46( 8) | 1.38( 8) | 1.58(8) | .23( 6) | -.27( 6) | .19( 6) |
| S2 | 1.55( 8) | 1.35( 8) | 1.33(7) | .41( 7) | -.10( 6) | .19( 7) |
| O1 | 1.7 ( 2) | 2.0 ( 2) | 1.4 (2) | .3 ( 2) | .14(18) | -.1 ( 2) |
| O2 | 1.1 ( 2) | 1.4 ( 2) | 1.4 (2) | -.39(19) | .34(17) | -.17(18) |
| O3 | 1.9 ( 2) | 1.3 ( 2) | 1.4 (2) | .2 ( 2) | -.52(18) | -.26(18) |
| N | .9 ( 3) | 1.0 ( 3) | 1.5 (2) | .0 ( 2) | .2 ( 2) | -.4 ( 2) |
| C1 | 1.0 ( 3) | 1.2 ( 3) | 1.1 (3) | .6 ( 2) | -.3 ( 2) | .1 ( 2) |
| C2 | 1.0 ( 3) | .9 ( 3) | 1.4 (3) | .1 ( 3) | .1 ( 2) | -.1 ( 3) |
| C3 | .9 ( 3) | .9 ( 3) | 1.7 (3) | -.1 ( 3) | -.1 ( 3) | .4 ( 2) |
| C4 | 2.3 ( 3) | 1.3 ( 3) | 1.3 (3) | .1 ( 3) | -.1 ( 3) | .0 ( 3) |
| C5 | 2.7 ( 4) | .7 ( 3) | 1.4 (3) | -.3 ( 3) | -.1 ( 3) | -.1 ( 2) |
| C6 | 1.4 ( 3) | 1.1 ( 3) | 1.7 (3) | -.5 ( 3) | -.1 ( 3) | .7 ( 3) |
| C7 | 2.7 ( 4) | 2.5 ( 4) | 2.6 (4) | .2 ( 3) | .6 ( 3) | -.4 ( 3) |
| C8 | 1.9 ( 3) | 1.2 ( 3) | 1.4 (3) | -.5 ( 3) | .5 ( 3) | -.2 ( 3) |
| C9 | 2.2 ( 3) | 1.6 ( 3) | .6 (3) | .0 ( 3) | .4 ( 3) | .2 ( 3) |
| C10 | .7 ( 3) | 2.7 ( 4) | 1.2 (3) | .3 ( 3) | .1 ( 3) | .0 ( 3) |
| C11 | 1.6 ( 4) | 2.4 ( 4) | 2.0 (3) | 1.1 ( 3) | .1 ( 3) | .3 ( 3) |
| K1 | .67(10) | 1.80(10) | 1.30(9) | .0 | .02( 7) | .0 |
| K2 | 1.84(12) | 1.39(10) | 1.60(9) | .0 | .18( 8) | .0 |
| O1w | 2.5 ( 3) | 2.0 ( 3) | 2.5 (2) | -.1 ( 2) | -.2 ( 2) | -.5 ( 2) |
| H1wa | 3.4 | | | | | |
| H1wb | 3.4 | | | | | |
| H3 | 2.2 | | | | | |
| H5 | 2.6 | | | | | |
| H6 | 2.4 | | | | | |
| H7a | 3.7 | | | | | |
| H7b | 3.7 | | | | | |
| H7c | 3.7 | | | | | |
| H8a | 2.5 | | | | | |
| H8b | 2.5 | | | | | |
| H9a | 2.5 | | | | | |
| H9b | 2.5 | | | | | |
| H10a | 2.5 | | | | | |
| H10b | 2.5 | | | | | |
| H11a | 3.0 | | | | | |
| H11b | 3.0 | | | | | |

Anisotropic Temperature Factors are of the form:
Temp=-2*Pi*Pi*(h*h*u11*astar*astar+---+2*h*k*u12*astar*bstar+---)

**Table S6.** Anisotropic displacement parameters (A^2 x 10^3) for **2**. The anisotropic displacement factor exponent takes the form:
-2 pi^2 [ h^2 a*^2 U11 + ... + 2 h k a* b* U12 ]

| | U11 | U22 | U33 | U23 | U13 | U12 |
|---|---|---|---|---|---|---|
| Ni(1) | 18(1) | 16(1) | 10(1) | 1(1) | -1(1) | 0(1) |
| S(1) | 21(1) | 23(1) | 15(1) | 4(1) | -1(1) | -3(1) |
| S(2) | 19(1) | 19(1) | 13(1) | -3(1) | -1(1) | 0(1) |
| N(1) | 24(1) | 16(1) | 12(1) | 1(1) | -1(1) | 2(1) |
| O(1) | 39(1) | 25(1) | 14(1) | 4(1) | -6(1) | 9(1) |
| O(2) | 66(1) | 34(1) | 52(1) | 19(1) | 38(1) | 21(1) |
| C(1) | 34(1) | 26(1) | 17(1) | 5(1) | 0(1) | -9(1) |
| C(2) | 39(1) | 18(1) | 16(1) | 4(1) | -4(1) | -4(1) |
| C(3) | 27(1) | 24(1) | 15(1) | 1(1) | -1(1) | 11(1) |
| C(4) | 21(1) | 31(1) | 14(1) | -4(1) | -2(1) | 6(1) |
| C(5) | 18(1) | 20(1) | 11(1) | 1(1) | 2(1) | -1(1) |
| C(6) | 20(1) | 21(1) | 15(1) | 3(1) | -1(1) | 2(1) |
| C(7) | 24(1) | 30(1) | 11(1) | 3(1) | -2(1) | 0(1) |
| C(8) | 21(1) | 26(1) | 14(1) | -4(1) | 1(1) | -1(1) |
| C(9) | 21(1) | 19(1) | 16(1) | -1(1) | 2(1) | -1(1) |
| C(10) | 21(1) | 19(1) | 12(1) | 2(1) | 0(1) | 0(1) |
| C(11) | 40(1) | 23(1) | 21(1) | -3(1) | 1(1) | 6(1) |
| C(12) | 34(1) | 30(1) | 30(1) | -2(1) | 7(1) | 5(1) |

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**Figure S3.** $^{1}H$ NMR of $K[MoO_2(btap)]$ in acetone-$d_6$. Asterisks denote residual solvent protons and minor impurities.

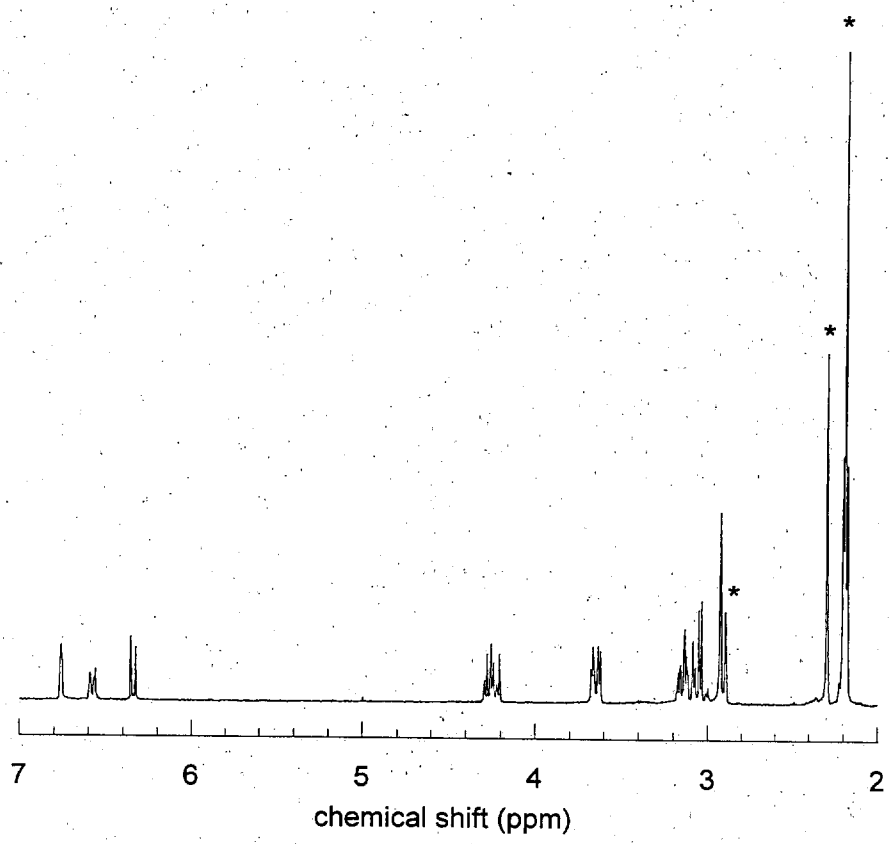


**Figure S4.** IR spectra of $H_3$btap, $MoO_2(btap)^{1-}$ and the *m*-CPBA-oxidation product of $MoO_2(btap)^{1-}$.

