# Crystal data and structure refinement of compound 1a

## *Crystal data*

$C_{25}H_{28}O_6$
$M_r$ = 424.493
Monoclinic
$P2_1$
a = 8.7040 (6)Å
b = 15.983 (2)Å
c = 9.2734 (9)Å
$\alpha$ = 90.00°
$\beta$ = 114.978 (5)°
$\gamma$ = 90.00°
V = 1169.4 (2)Å$^3$
Z = 2

$D_x$ = 1.206 Mg m$^{-3}$
Density measured by: not measured
fine-focus sealed tube
Mo $K\alpha$ radiation
$\lambda$ = 0.71073 Å
Cell parameters from 1601 reflections
$\theta$ = 1.018—23.257 °
$\mu$ = 0.08 mm$^{-1}$
T = 298 K
Plate
Red
Crystal source: *Chaetomium cupreum*

## *Data collection*

KappaCCD
Absorption correction: none
5691 measured reflections
1736 independent reflections
1505 observed reflections
Criterion: >2sigma(I)

$\theta_{max}$ = 23.38 °
h = 0 $\rightarrow$9
k = 0 $\rightarrow$17
l = -10 $\rightarrow$9

## *Refinement*

Refinement on $F^2$
Full-matrix least squares refinement
R(all) = 0.0723
R(gt) = 0.0625
wR(ref) = 0.1869
wR(gt)= 0.1738
S(ref) = 1.052
1736 reflections
280 parameters
3 restraints

$\Delta/\sigma_{max}$ = 0.039
$\Delta\rho_{max}$ = 0.305eÅ$^3$
$\Delta\rho_{min}$ = -0.274eÅ$^3$
Extinction correction: none
Atomic scattering factors from International Tables Vol C Tables 4.2.6.8 and 6.1.1.4

Data collection: KappaCCD
Cell refinement: HKL Scalepack (Otwinowski & Minor 1997)
Data reduction: Denzo and Scalepak (Otwinowski & Minor, 1997)
Program(s) used to solve structure: *SIR*97 (Cascarano al.,*Acta Cryst*.,1996,A52,C-79)
Program(s) used to refine structure: *SHELXL*-97 (Sheldrick, 1997)

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters (Å$^2$)*

$U_{eq} = 1/3\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \boldsymbol{a}_i.\boldsymbol{a}_j$ .

| | x | y | z | $U_{eq}$ | Occ |
|---|---|---|---|---|---|
| O9 | -0.1026 (5) | 0.6752 (3) | 0.7122 (4) | 0.0845 (10) | 1 |
| O2 | -0.3714 (5) | 0.6112 (3) | 0.9712 (5) | 0.0896 (11) | 1 |
| O8 | 0.1252 (5) | 0.5468 (3) | 0.6760 (4) | 0.0912 (12) | 1 |
| C3 | -0.3542 (7) | 0.5354 (4) | 1.0427 (7) | 0.0824 (15) | 1 |
| C10 | -0.4556 (8) | 0.5256 (4) | 1.1294 (7) | 0.0892 (16) | 1 |
| C9 | -0.0363 (7) | 0.6044 (4) | 0.8147 (6) | 0.0737 (13) | 1 |
| C5 | -0.0356 (7) | 0.4362 (4) | 0.9296 (7) | 0.0840 (15) | 1 |
| C4 | -0.2456 (8) | 0.4782 (4) | 1.0314 (7) | 0.0863 (15) | 1 |
| C1 | -0.2690 (7) | 0.6314 (4) | 0.8990 (7) | 0.0837 (15) | 1 |
| O7 | 0.3543 (7) | 0.4803 (4) | 0.6875 (6) | 0.1162 (16) | 1 |
| C17 | 0.2910 (8) | 0.3536 (4) | 0.9118 (8) | 0.0910 (16) | 1 |
| C6 | 0.1925 (8) | 0.4257 (4) | 0.8247 (7) | 0.0855 (15) | 1 |
| O17 | 0.2648 (7) | 0.3236 (3) | 1.0231 (6) | 0.1117 (14) | 1 |
| C12 | -0.6448 (7) | 0.5819 (4) | 1.2473 (7) | 0.0852 (16) | 1 |
| C11 | -0.5537 (7) | 0.5858 (4) | 1.1475 (7) | 0.0837 (15) | 1 |
| C19 | -0.6580 (9) | 0.4995 (4) | 1.3184 (8) | 0.0982 (18) | 1 |
| O22 | 0.1122 (8) | 0.7558 (3) | 0.8700 (7) | 0.1204 (17) | 1 |
| C22 | -0.0173 (9) | 0.7479 (4) | 0.7524 (9) | 0.0866 (16) | 1 |
| C7 | 0.2378 (9) | 0.4819 (4) | 0.7254 (7) | 0.0921 (17) | 1 |
| C13 | -0.7129 (8) | 0.6529 (5) | 1.2734 (8) | 0.0991 (19) | 1 |
| C23 | -0.1035 (11) | 0.8148 (5) | 0.6375 (10) | 0.113 (2) | 1 |
| C14 | -0.7971 (10) | 0.6670 (5) | 1.3778 (8) | 0.108 (2) | 1 |
| C18 | 0.4250 (10) | 0.3174 (5) | 0.8710 (11) | 0.117 (2) | 1 |
| C21 | -0.1697 (9) | 0.5092 (5) | 0.5752 (7) | 0.106 (2) | 1 |
| C15 | -0.768 (2) | 0.7481 (15) | 1.466 (2) | 0.275 (12) | 1 |
| C20 | -0.9767 (12) | 0.6803 (12) | 1.2981 (12) | 0.198 (7) | 1 |
| C16 | -0.5926 (18) | 0.7620 (15) | 1.559 (3) | 0.260 (10) | 1 |
| C5A | 0.0515 (7) | 0.4581 (3) | 0.8355 (6) | 0.0756 (14) | 1 |
| C4A | -0.1442 (7) | 0.4938 (4) | 0.9475 (6) | 0.0780 (14) | 1 |
| C1A | -0.1588 (7) | 0.5781 (3) | 0.8851 (6) | 0.0761 (14) | 1 |
| C8A | -0.0098 (7) | 0.5309 (4) | 0.7241 (6) | 0.0808 (14) | 1 |
| H10 | -0.4564 | 0.4729 | 1.1788 | 0.107 | 1 |
| H9 | 0.0702 | 0.6191 | 0.9000 | 0.088 | 1 |
| H5 | -0.0170 | 0.3826 | 0.9807 | 0.101 | 1 |
| H4 | -0.2348 | 0.4246 | 1.0816 | 0.104 | 1 |
| H1 | -0.2765 | 0.6858 | 0.8527 | 0.100 | 1 |
| H11 | -0.5664 | 0.6377 | 1.0916 | 0.100 | 1 |
| H19A | -0.7218 | 0.5052 | 1.3807 | 0.118 | 1 |
| H19B | -0.5461 | 0.4797 | 1.3848 | 0.118 | 1 |
| H19C | -0.7136 | 0.4604 | 1.2338 | 0.118 | 1 |
| H13 | -0.7137 | 0.7024 | 1.2145 | 0.119 | 1 |
| H23A | -0.0393 | 0.8657 | 0.6693 | 0.135 | 1 |
| H23B | -0.2142 | 0.8235 | 0.6339 | 0.135 | 1 |
| H23C | -0.1141 | 0.7986 | 0.5340 | 0.135 | 1 |
| H14 | -0.7631 | 0.6216 | 1.4525 | 0.130 | 1 |
| H18A | 0.4768 | 0.2706 | 0.9394 | 0.140 | 1 |
| H18B | 0.5093 | 0.3593 | 0.8854 | 0.140 | 1 |
| H18C | 0.3759 | 0.2991 | 0.7622 | 0.140 | 1 |
| H21A | -0.2616 | 0.4989 | 0.6043 | 0.127 | 1 |
| H21B | -0.1496 | 0.4600 | 0.5264 | 0.127 | 1 |
| H21C | -0.1983 | 0.5550 | 0.5016 | 0.127 | 1 |
| H15A | -0.8112 | 0.7923 | 1.3888 | 0.330 | 1 |
| H15B | -0.8235 | 0.7494 | 1.5356 | 0.330 | 1 |

| | | | | | |
|---|---|---|---|---|---|
| H20A | -1.0231 | 0.6894 | 1.3740 | 0.238 | 1 |
| H20B | -1.0275 | 0.6315 | 1.2358 | 0.238 | 1 |
| H20C | -0.9997 | 0.7281 | 1.2293 | 0.238 | 1 |
| H16A | -0.5732 | 0.8151 | 1.6122 | 0.312 | 1 |
| H16B | -0.5364 | 0.7610 | 1.4892 | 0.312 | 1 |
| H16C | -0.5487 | 0.7181 | 1.6360 | 0.312 | 1 |

Table 2. *Anisotropic displacement parameters (Å$^2$)*

| | $U_{11}$ | $U_{12}$ | $U_{13}$ | $U_{22}$ | $U_{23}$ | $U_{33}$ |
|---|---|---|---|---|---|---|
| O9 | 0.099 (2) | 0.005 (2) | 0.0359 (19) | 0.064 (2) | 0.0106 (19) | 0.087 (2) |
| O2 | 0.095 (2) | 0.0041 (19) | 0.052 (2) | 0.073 (3) | 0.011 (2) | 0.109 (3) |
| O8 | 0.117 (3) | 0.014 (2) | 0.052 (2) | 0.083 (3) | 0.014 (2) | 0.084 (2) |
| C3 | 0.086 (3) | -0.007 (3) | 0.040 (3) | 0.067 (3) | 0.008 (3) | 0.095 (4) |
| C10 | 0.095 (3) | -0.013 (3) | 0.036 (3) | 0.068 (4) | 0.011 (3) | 0.100 (4) |
| C9 | 0.087 (3) | 0.000 (2) | 0.031 (2) | 0.062 (3) | 0.003 (2) | 0.070 (3) |
| C5 | 0.100 (4) | 0.002 (3) | 0.030 (3) | 0.058 (3) | 0.008 (3) | 0.085 (3) |
| C4 | 0.097 (4) | -0.003 (3) | 0.044 (3) | 0.068 (3) | 0.008 (3) | 0.098 (4) |
| C1 | 0.103 (4) | 0.003 (3) | 0.049 (3) | 0.063 (3) | 0.009 (3) | 0.094 (4) |
| O7 | 0.138 (4) | 0.027 (3) | 0.080 (3) | 0.120 (4) | 0.021 (3) | 0.118 (3) |
| C17 | 0.099 (4) | 0.001 (3) | 0.035 (3) | 0.063 (3) | -0.002 (3) | 0.102 (4) |
| C6 | 0.099 (4) | 0.004 (3) | 0.032 (3) | 0.075 (4) | -0.003 (3) | 0.077 (3) |
| O17 | 0.138 (4) | 0.021 (3) | 0.051 (3) | 0.075 (3) | 0.018 (3) | 0.118 (3) |
| C12 | 0.077 (3) | -0.012 (3) | 0.030 (3) | 0.086 (4) | 0.002 (3) | 0.087 (3) |
| C11 | 0.086 (3) | -0.006 (3) | 0.042 (3) | 0.074 (4) | 0.008 (3) | 0.095 (3) |
| C19 | 0.109 (4) | -0.007 (3) | 0.054 (4) | 0.083 (4) | 0.012 (3) | 0.110 (4) |
| O22 | 0.135 (4) | -0.021 (3) | 0.037 (3) | 0.081 (3) | -0.002 (3) | 0.126 (4) |
| C22 | 0.108 (4) | 0.005 (3) | 0.059 (4) | 0.061 (4) | 0.006 (3) | 0.106 (4) |
| C7 | 0.115 (4) | 0.013 (4) | 0.046 (3) | 0.080 (4) | 0.004 (3) | 0.085 (3) |
| C13 | 0.113 (4) | -0.004 (4) | 0.054 (4) | 0.085 (5) | 0.008 (3) | 0.107 (4) |
| C23 | 0.148 (6) | 0.021 (4) | 0.077 (5) | 0.075 (4) | 0.019 (4) | 0.133 (6) |
| C14 | 0.143 (6) | 0.005 (4) | 0.056 (4) | 0.091 (5) | 0.004 (4) | 0.097 (4) |
| C18 | 0.111 (5) | 0.017 (4) | 0.054 (4) | 0.084 (5) | -0.004 (4) | 0.154 (6) |
| C21 | 0.121 (5) | 0.005 (4) | 0.016 (3) | 0.094 (5) | -0.008 (3) | 0.077 (3) |
| C15 | 0.39 (3) | -0.13 (2) | 0.125 (18) | 0.26 (2) | -0.096 (16) | 0.186 (14) |
| C20 | 0.131 (7) | 0.082 (11) | 0.054 (6) | 0.35 (2) | 0.019 (10) | 0.118 (6) |
| C16 | 0.182 (11) | -0.039 (14) | 0.055 (14) | 0.25 (2) | -0.077 (19) | 0.30 (2) |
| C5A | 0.084 (3) | -0.002 (2) | 0.026 (3) | 0.059 (3) | 0.000 (2) | 0.076 (3) |
| C4A | 0.087 (3) | -0.004 (3) | 0.032 (3) | 0.060 (3) | 0.005 (2) | 0.083 (3) |
| C1A | 0.082 (3) | -0.001 (3) | 0.028 (2) | 0.062 (3) | 0.000 (2) | 0.079 (3) |
| C8A | 0.094 (3) | 0.006 (3) | 0.032 (3) | 0.068 (3) | 0.004 (3) | 0.076 (3) |

Table 3 . *Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| O9—C22 | 1.344 (8) | C5A—C8A | 1.498 (8) |
| O9—C9 | 1.433 (7) | C4A—C1A | 1.450 (8) |
| O2—C3 | 1.359 (8) | C10—H10 | 0.9600 |
| O2—C1 | 1.360 (6) | C9—H9 | 0.9600 |
| O8—C7 | 1.367 (8) | C5—H5 | 0.9599 |
| O8—C8A | 1.444 (7) | C4—H4 | 0.9599 |
| C3—C4 | 1.351 (9) | C1—H1 | 0.9599 |
| C3—C10 | 1.431 (9) | C11—H11 | 0.9601 |
| C10—C11 | 1.344 (9) | C19—H19A | 0.9600 |
| C9—C8A | 1.517 (8) | C19—H19B | 0.9600 |
| C9—C1A | 1.526 (8) | C19—H19C | 0.9600 |
| C5—C4A | 1.378 (8) | C13—H13 | 0.9601 |
| C5—C5A | 1.420 (8) | C23—H23A | 0.9601 |
| C4—C4A | 1.423 (8) | C23—H23B | 0.9600 |
| C1—C1A | 1.330 (8) | C23—H23C | 0.9600 |
| O7—C7 | 1.205 (7) | C14—H14 | 0.9599 |
| C17—O17 | 1.242 (8) | C18—H18A | 0.9601 |
| C17—C6 | 1.461 (9) | C18—H18B | 0.9600 |
| C17—C18 | 1.487 (9) | C18—H18C | 0.9600 |
| C6—C5A | 1.374 (9) | C21—H21A | 0.9599 |
| C6—C7 | 1.454 (9) | C21—H21B | 0.9601 |
| C12—C13 | 1.349 (10) | C21—H21C | 0.9600 |
| C12—C11 | 1.452 (8) | C15—H15A | 0.9601 |
| C12—C19 | 1.498 (9) | C15—H15B | 0.9600 |
| O22—C22 | 1.199 (8) | C20—H20A | 0.9599 |
| C22—C23 | 1.472 (10) | C20—H20B | 0.9600 |
| C13—C14 | 1.458 (9) | C20—H20C | 0.9600 |
| C14—C20 | 1.435 (12) | C16—H16A | 0.9601 |
| C14—C15 | 1.49 (2) | C16—H16B | 0.9600 |
| C21—C8A | 1.530 (9) | C16—H16C | 0.9600 |
| C15—C16 | 1.418 (13) | | |
| | | | |
| C22—O9—C9 | 118.6 (4) | O22—C22—O9 | 122.7 (6) |
| C3—O2—C1 | 119.5 (5) | O22—C22—C23 | 125.3 (7) |
| C7—O8—C8A | 108.9 (5) | O9—C22—C23 | 112.0 (6) |
| C4—C3—O2 | 120.1 (5) | O7—C7—O8 | 119.4 (6) |
| C4—C3—C10 | 125.9 (6) | O7—C7—C6 | 131.1 (6) |
| O2—C3—C10 | 113.9 (5) | O8—C7—C6 | 109.4 (5) |
| C11—C10—C3 | 124.5 (6) | C12—C13—C14 | 129.2 (7) |
| O9—C9—C8A | 110.9 (4) | C20—C14—C13 | 115.1 (7) |
| O9—C9—C1A | 110.1 (4) | C20—C14—C15 | 92.9 (11) |
| C8A—C9—C1A | 108.7 (5) | C13—C14—C15 | 118.6 (10) |
| C4A—C5—C5A | 118.7 (5) | C16—C15—C14 | 111.1 (19) |
| C3—C4—C4A | 122.6 (6) | C6—C5A—C5 | 132.7 (5) |
| C1A—C1—O2 | 123.0 (5) | C6—C5A—C8A | 108.1 (5) |
| O17—C17—C6 | 119.5 (6) | C5—C5A—C8A | 119.2 (5) |
| O17—C17—C18 | 120.0 (6) | C5—C4A—C4 | 124.2 (5) |
| C6—C17—C18 | 120.4 (6) | C5—C4A—C1A | 121.4 (5) |
| C5A—C6—C7 | 107.7 (6) | C4—C4A—C1A | 114.3 (5) |
| C5A—C6—C17 | 126.2 (6) | C1—C1A—C4A | 120.1 (5) |
| C7—C6—C17 | 125.8 (6) | C1—C1A—C9 | 121.9 (5) |
| C13—C12—C11 | 118.5 (6) | C4A—C1A—C9 | 117.9 (5) |
| C13—C12—C19 | 122.6 (5) | O8—C8A—C5A | 104.5 (4) |
| C11—C12—C19 | 118.8 (6) | O8—C8A—C9 | 111.6 (5) |
| C10—C11—C12 | 126.6 (6) | C5A—C8A—C9 | 108.3 (4) |

| | | | |
|---|---|---|---|
| O8—C8A—C21 | 108.1 (5) | C13—C14—H14 | 105.9 |
| C5A—C8A—C21 | 111.6 (5) | C15—C14—H14 | 109.5 |
| C9—C8A—C21 | 112.5 (5) | C17—C18—H18A | 109.5 |
| C11—C10—H10 | 115.8 | C17—C18—H18B | 109.2 |
| C3—C10—H10 | 119.7 | H18A—C18—H18B | 109.5 |
| O9—C9—H9 | 109.4 | C17—C18—H18C | 109.7 |
| C8A—C9—H9 | 109.0 | H18A—C18—H18C | 109.5 |
| C1A—C9—H9 | 108.7 | H18B—C18—H18C | 109.5 |
| C4A—C5—H5 | 121.3 | C8A—C21—H21A | 109.5 |
| C5A—C5—H5 | 120.0 | C8A—C21—H21B | 109.5 |
| C3—C4—H4 | 119.6 | H21A—C21—H21B | 109.5 |
| C4A—C4—H4 | 117.8 | C8A—C21—H21C | 109.4 |
| C1A—C1—H1 | 116.9 | H21A—C21—H21C | 109.5 |
| O2—C1—H1 | 120.0 | H21B—C21—H21C | 109.5 |
| C10—C11—H11 | 119.0 | C16—C15—H15A | 108.7 |
| C12—C11—H11 | 114.4 | C14—C15—H15A | 108.1 |
| C12—C19—H19A | 110.7 | C16—C15—H15B | 107.7 |
| C12—C19—H19B | 109.1 | C14—C15—H15B | 111.6 |
| H19A—C19—H19B | 109.5 | H15A—C15—H15B | 109.5 |
| C12—C19—H19C | 108.6 | C14—C20—H20A | 110.4 |
| H19A—C19—H19C | 109.5 | C14—C20—H20B | 108.3 |
| H19B—C19—H19C | 109.5 | H20A—C20—H20B | 109.5 |
| C12—C13—H13 | 119.3 | C14—C20—H20C | 109.7 |
| C14—C13—H13 | 111.4 | H20A—C20—H20C | 109.5 |
| C22—C23—H23A | 109.9 | H20B—C20—H20C | 109.5 |
| C22—C23—H23B | 109.1 | C15—C16—H16A | 111.3 |
| H23A—C23—H23B | 109.5 | C15—C16—H16B | 108.1 |
| C22—C23—H23C | 109.4 | H16A—C16—H16B | 109.5 |
| H23A—C23—H23C | 109.5 | C15—C16—H16C | 109.1 |
| H23B—C23—H23C | 109.5 | H16A—C16—H16C | 109.5 |
| C20—C14—H14 | 114.9 | H16B—C16—H16C | 109.5 |